PESTICIDE ANALYTICAL MANUAL

VOLUME I: Multiresidue Methods

U.S. Department of Health and Human Services • Public Health Service
Food and Drug Administration

PESTICIDE ANALYTICAL MANUAL VOLUME I

3rd Edition, 1994

Revised, September, 1996
Revised, October, 1997
Revised, October 1999

Design and Format: R.O.W. Sciences, Inc.
under contract to U.S. Food and Drug Administration

Transmittal No. 2000-1 (10/1999)
Form FDA 2905a (6/92)

PESTICIDE ANALYTICAL MANUAL VOLUME I

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PESTICIDE ANALYTICAL MANUAL

INTRODUCTION

The Food and Drug Administration (FDA) is responsible under the Federal Food, Drug, and Cosmetic Act for enforcing tolerances established by the Environmental Protection Agency (EPA) for amounts of pesticide residues that may legally remain on food (including animal feed). In meeting this responsibility, FDA collects and analyzes food from commercial channels of trade for determining compliance with EPA tolerances. The residue data gathered under this regulatory monitoring program are also used for evaluating the extent and significance of pesticide residues in the food supply.

The Pesticide Analytical Manual (PAM) is published by FDA as a repository of the analytical methods used in FDA laboratories to examine food for pesticide residues for regulatory purposes.[1] The manual is organized according to the scope of the analytical methods:

Volume I contains multiresidue methods (MRMs) that are used by FDA on a routine basis, because of their efficiency and broad applicability, especially for analyzing foods of unknown pesticide treatment history.

Volume II contains methods designed for the analysis of commodities for residues of only a single compound (although some methods are capable of determining several related compounds). These methods are most often used when the likely residue is known to the chemist and/or when the residue of interest cannot be determined by common MRMs.

PAM is designed to be used by analysts experienced in trace residue analysis. All of the techniques employed are subject to potential interferences from reagents, apparatus, containers, contaminated air supply, and handling by personnel. The experienced analyst is alert for these possibilities and recognizes the need to confirm results by other techniques that measure different chemical or physical properties of the analyte.

Experienced residue analysts are aware that no report of validation in another laboratory can substitute for verification that the method does indeed work in the analyst's own laboratory. The analyst should verify method performance in each particular application by a trial of the method that includes examination of reagent and sample blanks and measurement of the recovery of added analyte. The editors invite analysts to report results of their experiences with PAM methods.

Revisions

Starting with transmittal 96-1 (9/96), revisions of PAM I have been issued in two ways: (1) changes in most manual sections will be distributed as hard (paper) copies, with symbols ▶ or ◀ marking lines that have been changed, and (2) updates to the tables

[1] 40 CFR 180.101 (c)

in Chapters 3 and 4, Appendix I, and the indices to methods, names, and CAS Registry numbers will be issued only *via* Internet. No hard copies will be distributed for the latter updated sections, but updates will be available more frequently than in the past.

As chapter tables of contents are revised, they will include the date on which each section within the chapter was transmitted; dates associated with those sections distributed only electronically will reflect the most recent version at the time the table of contents issued.

Internet Access to PAM I Files

PAM I is now available *via* Internet as Adobe Acrobat "portable document format" (pdf) files. Pdf format permits the user to read and print the document from any computer using appropriate free software.

▶ To obtain a copy of PAM I files, go to the World Wide Web site at: http://vm.cfsan.fda.gov/~frf/pami1.html. The resulting page describes PAM and provides links to currently available files. Follow the instructions for downloading.

Adobe Acrobat Reader is required to view and print pdf files. Download a copy of this free software from Adobe's web site at http://www.adobe.com/acrobat/readstep.html. A link to that site is provided on the PAM I page. Choose the version of Acrobat Reader appropriate to your own computer system.

PREFACE TO PAM I 3RD EDITION

The third edition of PAM I follows by 26 years the publication of the second edition. During that period, 29 revisions were made, reflecting new or revised methods, new technologies, and periodic updates of tables describing the capabilities of PAM I methods. Preparation of PAM I 3rd edition was motivated by three major deficiencies in the oft-revised 2nd edition: outdated material, obsolete organization of methods, and lack of consistent style.

Changes in multiresidue methods (MRMs) over the past 26 years have been significant. Among the most notable changes are those related to instrumental determinative techniques. Capillary columns and improved detectors have enhanced GLC applications; HPLC, with its various operating modes, has extended multiresidue capabilities to pesticides not amenable to GLC determination; and mass spectrometry, in the form of compact, automated instruments readily combined with GLC, has replaced many cumbersome, less sensitive, and less definitive techniques. PAM I 3rd edition attempts to provide a more up-to-date picture of the status of instrumentation currently used in FDA pesticide laboratories.

Despite advances in instrumentation, the basic approach to determination of trace level residues has not departed dramatically from that used in the 1960s. Residues are still extracted from the food commodity, isolated from co-extracted materials, and determined by instrumental techniques that separate residues from one another. While these analytical steps continue to be part of any MRM, methods research, coupled with advances in analytical technologies, has produced MRMs capable of determining a greater number of widely different types of pesticide residues in a single extract, *i.e.*, "multiclass MRMs." Research has also produced other MRMs that determine multiple residues of chemically related pesticides, such as N-methylcarbamates; these types of methods are known as "selective MRMs."

PAM I 2nd edition organized methods according to the chemical class of the targeted residues, an organization that does not conform to modern methodology. A major change in the PAM I 3rd edition is its grouping of methods into multiclass MRMs (Chapter 3) and selective MRMs (Chapter 4).

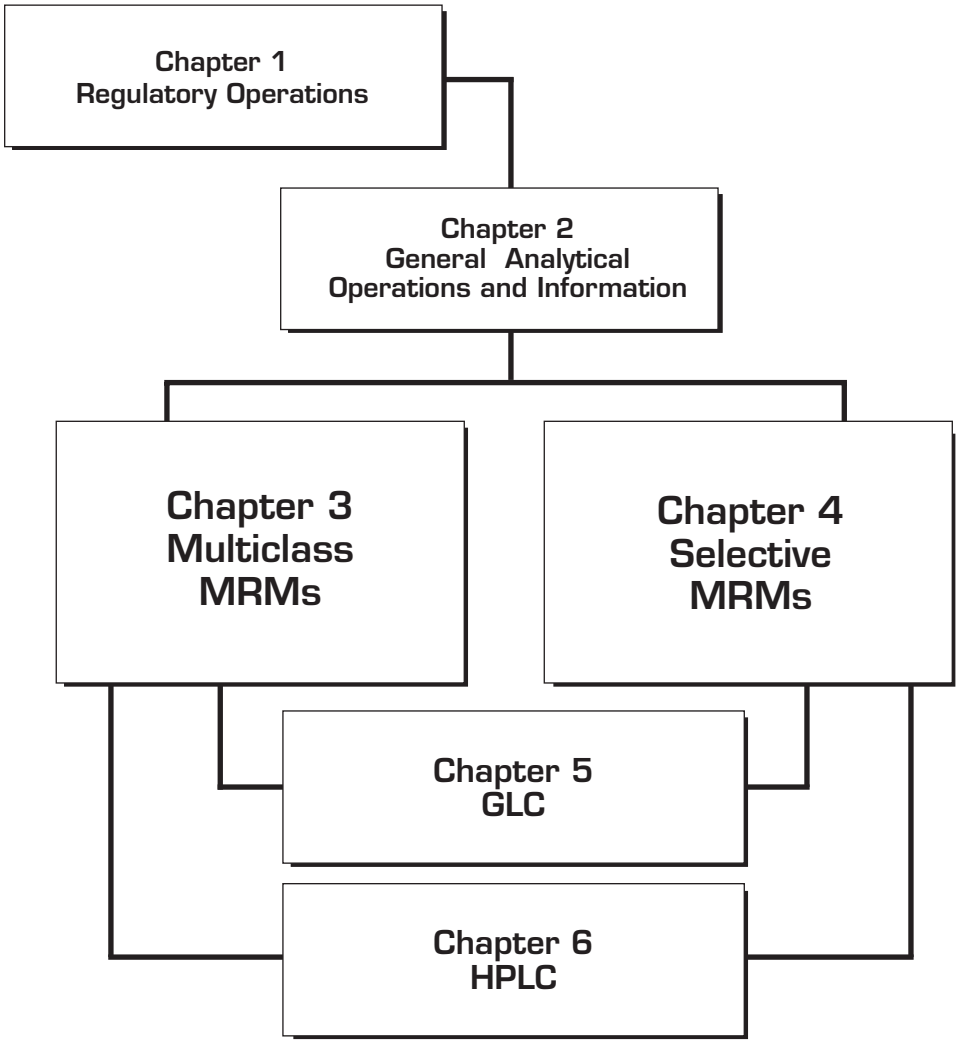
Another change in method descriptions accommodates the choices available to the experienced residue analyst. Typically, the residue laboratory chooses from among several validated options within basic methodology; choices are dictated by the particular commodity, available instrumentation, and targeted residues. Thus, PAM I 3rd edition method descriptions consist of individual extraction, cleanup, and determinative step modules, with indications of which combinations are validated. This organization permits easier reference to the precise combination of steps used in an analysis and facilitates sharing particular methods with colleagues. Future addition or revision of methods will be simplified by adding or replacing only the necessary sections or modules. The numbering system used in Chapters 3 and 4 is explained in the Guide to PAM I.

Finally, PAM I 3rd edition incorporates a new and consistent design. A new numbering system is used, in which chapter and subsection numbers avoid the restrictive 2nd edition decimal system. Pages are numbered within major subsections. Four indices are included: (1) to methods applicable for individual residues, (2) to preferred names for pesticides, (3) to Chemical Abstracts Service (CAS) Registry Numbers for the chemicals, and (4) to subjects by key word. An introductory Guide to PAM I, on the following pages, explains the organization of chapters

and the most useful path for finding pertinent information within the volume. The user is urged to take advantage of these tools and to offer comments or improvements that would make them more useful. PAM I remains, as always, a loose-leaf volume, designed for continuing update.

The Editors acknowledge the continuing cooperation and support of the pesticide chemists in FDA District and Regional Laboratories, District Research Centers, and Division of Pesticides and Industrial Chemicals; those who contributed substantially to 3rd edition preparation are included as technical advisors on the title page. Many of these advisors drafted or reviewed individual sections of the 3rd edition, and all FDA chemists responded repeatedly to requests for information about the applications of analytical methodology for pesticide residues. The editors also acknowledge the preparation of specialized sections by Mark Wirtz (QA/QC and GLC Quantitation by Electronic Integration) and Ann Stack (Safety), numerous editorial reviews performed by Norma Yess, secretarial assistance provided by Joan Duy, and comments on portions of the chapters on GLC and HPLC by Dr. Colin Poole. Without the assistance of all these individuals, the 3rd edition would not have been possible, and we are grateful to them.

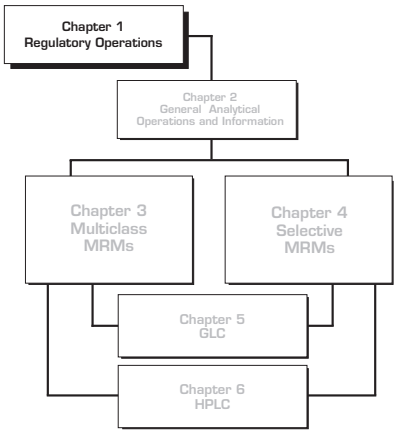
December, 1993



GUIDE TO PAM I

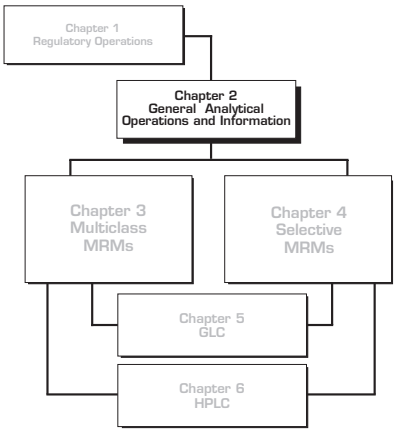
Each chapter in Volume I covers a different topic. Effective use of PAM I requires an understanding of the reasons that specific information is included in the chapter in which it appears.

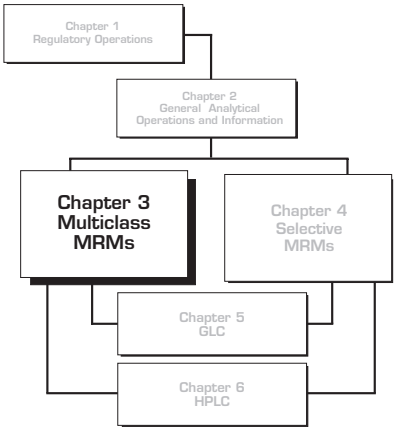
The user is advised to: (1) become familiar with the information in this manual and where it is located, as explained on pages x–xi; (2) understand how to choose and find appropriate methods, as detailed on pages xii–xiii; (3) review pages xiv–xvi for other information about Volume I and comparison to the 2nd edition; and (4) learn to use the indices provided.



Chapter 1 provides information and directions that reflect FDA regulatory policy. PAM I is not the authoritative source for publication of FDA policy, but policy decisions that directly affect the application of pesticide analytical methodology are included here as a service to the manual user.

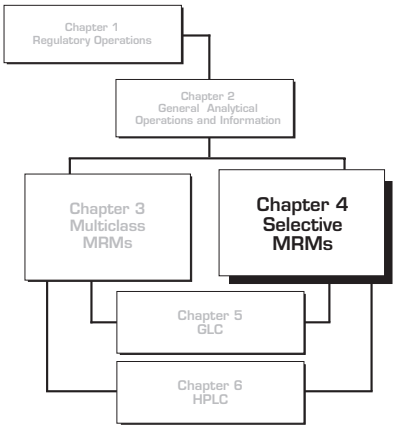
Chapter 2 is a collection of data and directions on a variety of unrelated topics, each of which provides background information needed to perform methods of Chapters 3 and 4. Where information in Chapters 1 and 2 appears to overlap (*e.g.*, Sections 102, Preparation of Analytical Samples; and 203, Equipment and Procedures for Comminuting), the material in Chapter 1 reflects the agency policy that must be followed for enforcement of regulations, and Chapter 2 provides information and hints found useful by FDA pesticide chemists.





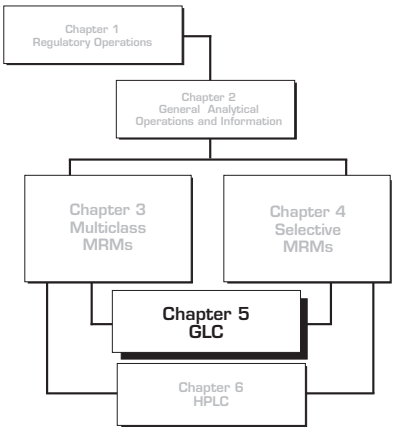
Chapter 3 includes multiclass multiresidue methods (MRMs), those that are capable of determining pesticide residues of many chemical types. The actual number and identity of the residues determinable by the methods are usually dependent on the number and variety of different determinative steps used to examine the extract. Each method in this chapter is presented as a series of modules for the extraction, cleanup, and determinative steps; a complete method is defined as a combination of one or more modules from each step. Complete methods that have been validated in interlaboratory studies, including collaborative studies performed under the auspices of AOAC International, are listed; these are sanctioned for use in regulatory analyses. Other combinations of modules must be treated as experimental methodology; additional supporting data for the validity of the analysis are required when such combinations are used in regulatory analyses.

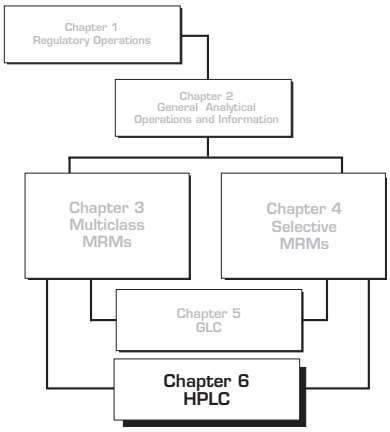
Chapter 4 includes descriptions of selective MRMs, *i.e.*, methods designed to determine a limited number of residues related by chemical structure. Selective MRMs target residues that are not amenable to determination by the multiclass MRMs of Chapter 3. In actual practice, FDA laboratories often examine an extract from a multiclass MRM with determinative step(s) from selective MRMs to broaden the scope of the analysis. When such uses have been validated in interlaboratory studies, the determinative steps are included as modules in Chapter 3 methodology. When no interlaboratory validation has occurred, such combinations are treated as experimental methodology, with the same requirements noted above for use in regulatory analyses.



Tables of data are provided for each method in Chapters 3 and 4 to describe the analytical behavior of each chemical tested through the method. Data are available only for methods sanctioned for use by virtue of previous interlaboratory studies.

Chapter 5, GLC, and Chapter 6, HPLC, provide background information about the two major determinative steps used in MRMs. Basic information about the techniques is included, as well as specific directions for implementing use of the instruments based on experiences in FDA laboratories. Manual users attempting to employ the determinative steps defined in Chapters 3 and 4 may need to refer to Chapters 5 and 6 for additional information and advice.





Appendix I combines information on GLC behavior of particular chemicals with recoveries of the chemicals through Chapter 3 methods. Appendix II defines the steps of protocols that were used to develop such method behavior data, for use in continuing testing.

STEP-BY-STEP SAMPLE ANALYSIS

PAM I provides information and/or directions to facilitate performance of each step in sample analysis. Use these sections for information needed at each step:

REVIEW PROCEDURES	Secs. 101, 103	Appropriate procedures for regulatory analyses
PREPARE SAMPLE	Sec. 102	Portion of commodity to include in analytical sample, regulatory requirements for subsamples
	Sec. 203	How to comminute or homogenize various commodities
CHOOSE METHOD		Based on commodity type: _____ Based on targeted residues: _____
REVIEW BACKGROUND INFORMATION (as needed)	Sec. 201	Percentage fat, water, and sugars in commodity
	Sec. 202	Detailed directions for column chromatography, solvent concentration
	Sec. 204	Directions for preparing frequently used reagents
	Sec. 205	Reference standards
	Sec. 206	Quality assurance/quality control
	Sec. 207	Safety
	Sec. 208	Hazardous waste disposal
	Chap. 5	Operation of GLC systems
	Chap. 6	Operation of HPLC systems
PERFORM ANALYSIS	Chap. 3 Chap. 4	Method directions
IDENTIFY RESIDUES	Tables, Appendix I	Details of behavior of chemicals tested through methods, for tentative identification
QUANTITATE RESIDUES	Secs. 504, 606	Quantitation for GLC, HPLC
CONFIRM RESIDUES	Sec. 103	Approach to confirmation of residue identity
REPORT RESIDUES	Secs. 104, 105	FDA procedures

Transmittal No. 94-1 (1/94)
Form FDA 2905a (6/92)

CHOICE OF METHOD BASED ON COMMODITY TYPE

PRODUCTS >2% FAT (dairy, meat, fish, oilseeds, *etc.*)	Sec. 304	Appropriate extraction for product; relatively nonpolar residues; actual residues covered depends on determinative step(s) included
	Sec. 401	N-methylcarbamate residues
	Sec. 402	Acidic and phenolic residues
NONFATTY PRODUCTS (<2% fat) (fruits and vegetables, grains, *etc.*)	Sec. 302	Nonpolar and polar residues, if no cleanup is used; actual residues covered depends on determinative step(s) included
	Sec. 303	Relatively nonpolar residues; actual residues covered depends on determinative step(s) included
	Sec. 401	N-methylcarbamate residues
	Sec. 402	Acidic and phenolic residues
	Sec. 403	Phenylurea herbicide residues
	Sec. 404	Benomyl (as MBC), thiophanate-methyl, allophanate, and thiabendazole
EGGS, EGG WHITES	Sec. 303	E2; relatively nonpolar residues
DRIED EGG WHITES	Sec. 303	E3; relatively nonpolar residues

CHOICE OF METHOD BASED ON TARGETED RESIDUES

SPECIFIC RESIDUE(S)		Use Index to Methods to find method(s) applicable to targeted residue(s).
N-METHYL-CARBAMATES	Sec. 302	Use appropriate extraction + C3 or C4 + DL1. Confirm residues with Sec. 401.
ACIDS, PHENOLS	Sec. 402	Confirm residues by use of additional appropriate GLC systems.
PHENYLUREAS	Sec. 403	Confirm residues as directed in method.
BENZIMIDAZOLES	Sec. 404	Confirm residues as directed in method.
NO TARGET	Sec. 301	Use scheme for multiclass MRM, Figure 301-a.

NOTES ON TERMINOLOGY

Within PAM I, abbreviations are explained the first time they are used within any major subsection of a chapter (Section 101, 204, *etc.*). Subsequent use within that major subsection is not explained.

Certain common abbreviations are used without explanation throughout the volume; these include units of length, weight, volume, time, and concentration.

PAM I alphabetized tables of data use the following sequence: [space] ! " # $ % & ' ( ) * + , - . / 0 1 2 3 4 5 6 7 8 9 : ; < = > ? @ A B C D E F G H I J K L M N O P Q R S T U V W X Y Z [\]. Because commas precede hyphens in this sequence, chemical names that start with combinations of numbers, hyphens, and commas may not appear where expected, *e.g.*, 2,4,5,-T precedes 2-chloroethyl caprate.

"LIB" references refer to FDA's in-house Laboratory Information Bulletins, issued by Office of Regulatory Affairs, Division of Field Science, 5600 Fishers Lane, Rockville, MD 20857. LIB references are used only for material that was not subsequently published in the scientific literature; the latter reference is used whenever available.

NUMBERING OF METHODS MODULES

Methods described in Chapters 3 and 4 of this volume are presented as a series of extraction, cleanup, and determination modules. This organization offers flexible combination of modules as appropriate to the commodity being analyzed and/or the residues being targeted. The analyst and laboratory are responsible for assuring that the combination is valid.

Each method in Chapters 3 and 4 is treated as a major subsection, *i.e.*, it is numbered consecutively with a whole number: 301, 302, *etc.*, 401, 402, *etc.* Within those subsections, modules are numbered according to the following scheme:

Extraction — **E**, followed by a number.

"E" module numbers are repeated in different methods (*i.e.*, E1 of Section 302 is not the same as E1 of Section 303) so both the section number and the module number must be included in a reference. For example, 302 E1 defines an extraction step, but E1 does not.

Cleanup — **C**, followed by a number.

As above, "C" module numbers are repeated in different methods and both the section number and the module number must be referenced.

Determination — **DG**, followed by a number, for GLC determinative steps.
DL, followed by a number, for HPLC determinative steps.

Unlike E and C numbers, there is no repetition of DG or DL numbers, because the same determinative steps are used to examine cleaned-up extracts from many different methods. DG1 always refers to the same GLC system, no matter what section of Chapter 3 (or 4) it is combined with.

COMPARISON TO SECOND EDITION

Where PAM I 2nd edition material appears in the 3rd edition, section numbers are different. The following list of equivalent references applies:

2nd Edition	Description	3rd Edition
Methods		
211.13a-k/231.1	(Mills) method for fatty foods extractions	304 E1-E5
211.14a	Petroleum ether-acetonitrile partitioning	304 C1-C4
211.14b	Petroleum ether-acetonitrile "backwash"	not in 3rd ed
211.14c	Partition chromatography	not in 3rd ed
211.14d	Florisil cleanup with ethyl ether/petr ether	304 C1, C3
211.15a-c	Supplemental cleanups	not in 3rd ed
211.15d	Alkaline hydrolysis supplemental cleanup	part of 304 C7
212.13a-d/232.1	(MOG) method for nonfatty foods extractions	303 E1-E5
212.14	Cross-reference to Florisil with ethers	303 C1
212.2	(Luke) method for nonfatty foods with Florisil cleanup	302 E1+C5
221.1	(Hopper) method for chlorophenoxy acids, phenols	402
232.2	Sweep co-distillation method for OPs	not in 3rd ed
232.3	(Storherr) method for OPs	not in 3rd ed
232.4/242.1	(Luke) method for nonfatty foods, no cleanup	302 E1
242.2	(Krause) method for N-methylcarbamates	401
242.3	(PICRC) method for benzimidazoles	404
242.4	(Luchtefeld) method for substituted ureas	403
251.1	Silicic acid separation of PCBs and pesticides	not in 3rd ed
251.2	Derivatization and separation of PCBs, pesticides	not in 3rd ed
252.1	Florisil elution with methylene chloride	303 C2, 304 C2, C4
253	Exhaustive extraction of organochlorine residues	not in 3rd ed

Gas Chromatography (Chapter on GLC has been revised extensively; references to equivalent sections reflect the same topic but not necessarily the same information.)

300	Application of GLC to pesticide residue analysis	501, 504
301	Columns	502
310	Detectors	503
311	Electron capture detector	503 B
312	Microcoulometric detector	not in 3rd ed
313	Potassium chloride thermionic detector	not in 3rd ed
314	Flame photometric detector	503 C
315	Electrolytic conductivity detector	503 D
316	N/P detector	503 E
320	Multiple detectors	not in 3rd ed
330	GLC parameters and data tables	302 DG1-DG23, Appendix I

HPLC (Chapter on HPLC has undergone only minor revision, but chapter and sections are renumbered.)

500	General information	601, 608
510	HPLC columns	602
520	Mobile phase selection, preparation, delivery	603

530	Injection systems	604
540	Detectors	605
550	Data handling	606
560	Quality assurance and troubleshooting	607

Tables

201	Chemicals tested through specific methods	tables follow method sections
202	Percentage Fat, Water, and Sugar in Foods	201
Appendix I	PESTDATA lists of GLC data and recoveries	Appendix I

Other Information

110, 120	[Combined] lists of apparatus and reagents	not in 3rd ed
130	Standards	205
141-142	Preparation of analytical portion, compositing	102, 203
143.1	Reporting analytical results	104 B
143.2	Analytical limit of quantitation	105
230	Organophosphate residues	not in 3rd ed
Chapter 4	TLC	not in 3rd ed
Chapter 6	Confirmation	not in 3rd ed, except 103 E

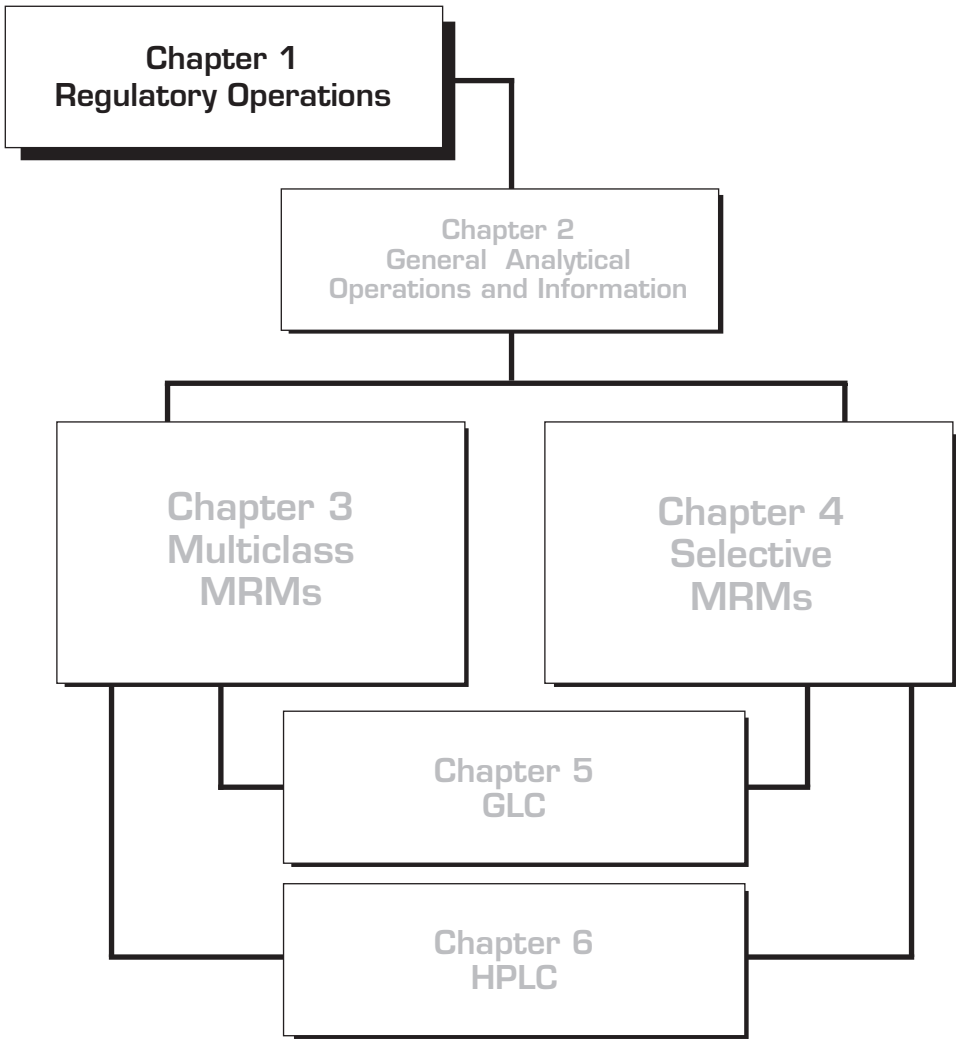


Table of Contents

101: REGULATORY POLICY

The expressed purpose of the Pesticide Analytical Manual is to publish analytical methodology used by the FDA in enforcing pesticide tolerances. To understand FDA's application of methodology published in the manual, it is important to understand pesticide tolerance regulations and related FDA regulatory operations. Material in Chapter 1 reflects FDA regulatory policies that affect its application of analytical methodology.

101 A: REGULATORY AUTHORITY

Information related to and the characteristics of pesticide tolerances include the following:

A tolerance is the maximum concentration of a pesticide residue that is legally permitted to remain in a food. The tolerance is not expected to be exceeded if the pesticide's registered use directions are followed.

The statutory authority for the Environmental Protection Agency's (EPA's) establishment of tolerances is provided by sections 408 and 409 of the Federal Food, Drug, and Cosmetic Act.

Tolerances established by EPA are set forth in Title 40 of the *Code of Federal Regulations* (CFR), Part 180 for raw agricultural commodities, Part 185 for processed food, and Part 186 for animal feed. The concentration of tolerances listed in 40 CFR 180, 185, and 186 is expressed in terms of ppm (*i.e.*, mg pesticide residue/kg food). In addition, certain pesticides are exempted from the need for tolerances; such exemptions are listed in 40 CFR 180.1001.

A tolerance for a pesticide residue on a raw agricultural commodity, *e.g.*, tomatoes, also applies to processed forms of that commodity, *e.g.*, canned tomatoes. In cases where processing may concentrate the residue, a food additive regulation may be issued in 40 CFR Part 185 to establish a higher tolerance on that processed commodity, *e.g.*, tomato paste.

A tolerance regulation specifies the composition of pesticide residue for which the limit applies; *i.e.*, a tolerance can apply to the parent form of the active ingredient only, parent compound plus one or more metabolites and/or degradation products, one or more metabolites and/or degradation products only, or some chemical moiety that can be measured analytically for calculating the pesticide residue. A chemical entity not specified by the tolerance regulation is not included in the residue for tolerance enforcement purposes (Section 104).

A tolerance regulation also specifies each individual food (*e.g.*, apples) or food group (*e.g.*, citrus fruit) to which the limit applies. No tolerance exists for a residue on a commodity unless the commodity itself or the group to which it belongs is specified.

In the examination of a food lot to determine whether it complies with tolerances, it is necessary to provide a sample for analysis that is representative of the lot in order to determine the average pesticide residue content

of the lot. Tolerances apply to that sample or a representative portion of that sample.

Unless otherwise specified in a tolerance regulation, each tolerance applies to the whole portion of a food commodity that moves in commerce. In practice, however, some food commodities (mainly raw agricultural commodities) require further definition as to the portion of commodity to which a tolerance applies and which is to be analyzed.

In summary, a tolerance provides a means of ascertaining that a pesticide was properly used. If a pesticide residue is found to exceed a tolerance or be present in a food for which there is no tolerance, then the pesticide was not used in a manner consistent with the tolerance. Under Sections 402 (a) (2) (B) or (C) of the Federal Food, Drug, and Cosmetic Act, this constitutes a violation of the law; *i.e.*, the food commodity is adulterated because it contains an "unsafe" or illegal pesticide residue.

101 B: REGULATORY OPERATIONS

To fulfill its responsibility to enforce regulations on pesticide residues in foods, FDA maintains a comprehensive pesticide program, including the analysis of food for enforcement of pesticide tolerances. Although the majority of samples analyzed contain no violative residues, sample handling must be consistent for all analyzed samples, because it is impossible to know in advance which samples will contain violative residues.

Accordingly, the following procedures must be followed by FDA laboratories to establish that a product contains illegal pesticide residue(s):

1) A laboratory sample of food must be collected from a consignment in accordance with agency sample collection instructions [1]; this laboratory sample is then considered representative of the food consignment.

2) The portion of food taken from the laboratory sample (whole product or specified parts of product) must be in accordance with agency instructions, and that portion must be appropriately composited and comminuted (Section 102). From that resulting test sample, a test portion is taken for analysis; test portion size is dictated by requirements of the analytical method. Residues found in the test portion are considered representative of the average residue content of the original consignment.

   (Note that terminology related to food products in this chapter, *i.e.*, "consignment," "laboratory sample," "test sample," "test portion," reflects recommendations of IUPAC Analytical Chemistry Division, Commission on Analytical Nomenclature [2]. Common usage, however, usually refers to the test portion as "sample," and this convention is used throughout most of PAM I.)

3) The test portion must be analyzed by a published, official analytical method or one that has been validated for the specific pesticide/commodity portion, and findings of residues must be confirmed (Section 103). For FDA monitoring purposes, analytical methods must be capable of accurately

measuring pesticide residues as defined by the tolerance regulation at not only the tolerance limit but also the lower limit of quantitation (Section 105).

4) If the residue level found in the original analysis exceeds an established tolerance, or if no tolerance exists for the residue in that commodity, another analysis of a second test portion of the same composited test sample must be conducted by a second analyst (normally a senior analyst); the second analysis is referred to as a "check analysis."

5) If check analysis verifies that the residue violates a regulation, *i.e.*, the results of both original and check analyses exceed a tolerance and are in close agreement or are in close agreement for pesticide residues for which there is no tolerance, the analytical findings will support enforcement action against the food consignment. If the check analysis result is below a tolerance or if the results of the original and check analyses are widely divergent, enforcement action cannot be supported. Additional analyses may be required to resolve widely divergent analytical results.

101 C: MONITORING

The FDA pesticide program has two main objectives: (1) to enforce residue tolerances and (2) to determine incidence and level of pesticide residues in the food supply. The section above defines operations established to enforce regulations. Monitoring aspects of the programs can be accomplished simultaneously, because levels of all residues found are calculated and recorded, whether or not they support enforcement action. Section 104 provides information about reporting residues for monitoring purposes, as well as determining compliance with regulations.

References

[1] Investigations Operation Manual, Sample Schedule Chart 3, FDA, Rockville, MD

[2] Horwitz, W. (1990) *Pure Appl. Chem.* **62**, 1193-1208

102: PREPARATION OF ANALYTICAL SAMPLES

102 A: INTRODUCTION

This section contains directions for preparation of test samples of food from laboratory samples collected for pesticide residue analysis. The following topics are considered, but not all are pertinent to every sample situation: (1) instructions for portion of commodity to be analyzed for pesticide residues, (2) directions for compositing and comminuting food items, (3) procedures for samples that are to undergo special analyses, and (4) requirements for retention of reserve portions of test samples.

102 B: PORTION OF FOOD COMMODITY TO BE ANALYZED

As a general approach, the "portion of commodity" composited to create the test sample consists of the entire food commodity (*e.g.*, whole cantaloupe). For many raw and processed foods, however, only specific portions of the food are included in the composite for the test sample. To ensure uniformity and consistency in tolerance enforcement and related monitoring, it is necessary to adhere to the following instructions on the portion of commodity to be analyzed.

Raw Agricultural Commodities

EPA regulations [1] specify that a raw agricultural commodity examined for compliance with a pesticide tolerance consist of the "whole raw agricultural commodity." The regulations contain some specific instructions on what constitutes the whole raw agricultural commodity; *e.g.*, "caps (hulls) shall be removed and discarded from strawberries before examination for pesticide residues." Such instructions are provided for only nine individual food commodities (*e.g.*, bananas) and crop group commodities (*e.g.*, root vegetables).

Recognizing the limitation of these regulations, FDA developed directions for additional commodities, taking into account practical considerations of sample preparation (*e.g.*, removal of stones from peaches to facilitate preparation of a homogenate). Table 102-a is a compilation of EPA regulations and FDA directions. (An EPA rulemaking is expected to be initiated that would amend the above existing regulation to incorporate FDA's more complete instructions on the portion of commodity to which a tolerance applies and that is to be analyzed.)

In some instances, a pesticide tolerance regulation specifies an exception to directions in Table 102-a. For example, the tolerance for mevinphos residues on melons states that compliance with the tolerance is to be "determined on the edible portion with rind removed," [2] even though the tolerances for most other pesticides on melons apply to the whole commodity including the rind.

Follow these directions to prepare test samples of raw agricultural commodities:

- Use the entire raw agricultural commodity, as specified in Table 102-a.
- When a pesticide residue is found in a commodity for which the tolerance applies to a portion different from that specified in Table 102-a, prepare a new test sample in accordance with the pesticide's tolerance regulation.

Table 102-a: Portion of Raw Agricultural Commodity to be Analyzed for Pesticide Residues

Root and tuber vegetables group[1]	Where separate tolerances are established for root or tuber, analyze whole commodity after removing adhering soil by lightly rinsing in running water. Where a tolerance is established on a root vegetable including tops and/or with tops, and tops and roots are marketed together, analyze tops and roots separately. Neither the pesticide residue on the roots nor the pesticide residue on the tops shall exceed the tolerance level. For carrots, parsnips, and rutabagas, remove and discard tops.
Bulb vegetables (green or dry) group	Whole commodity after removing and discarding roots. Remove adhering soil by lightly rinsing in running water. In the case of dry bulb onions and garlic, remove and discard stems and outer sheaths (husk or parchment skin) that are easily removed.
Leafy vegetables (except Brassica vegetables) group	Whole commodity after removing and discarding obviously decomposed or withered leaves. In the case of rhubarb, analyze only the stem without leaves. Remove adhering soil from celery by lightly rinsing in running water.
Brassica (cole) leafy vegetables group	Whole commodity after removing and discarding obviously decomposed or withered leaves, except remove and discard all leaves from cauliflower and headed broccoli and use sprouts only from brussels sprouts.
Legume vegetables (succulent or dried) group	Whole commodity, including pods for succulent and without pods for dry.
Fruiting vegetables (except cucurbits) group	Whole commodity after removing and discarding stems and husks.
Cucurbit vegetables group	Whole commodity after removing and discarding stems.
Citrus fruits group	Whole commodity.
Pome fruits group	Whole commodity after removing and discarding stems.
Stone fruits group	Whole commodity after removing and discarding stems and stones.
Small fruits and berries group	Whole commodity after removing and discarding caps and stems, except for currants, where the stems are to be included.

[1] Members of food groups are listed in 40 CFR 180.34 (f) (9).

Peanuts	Whole peanut meat (kernel) after removing hulls.
Peanut hulls	Whole commodity after removing peanut meat.
Dates and olives	Whole commodity after removing and discarding stems and stones or pits.
Pineapples	Whole commodity after removing and discarding crowns (leaves at top of fruit).
Avocados and mangoes	Whole commodity after removing and discarding stones.
Bananas	Whole commodity including peel after removing and discarding crown tissue and stalk.
Miscellaneous raw fruits and vegetables not previously included	Whole commodity after removing and discarding obviously decomposed or withered leaves, stems, stones or pits, shells or husks; if commodity has adhering amounts of soil, remove by lightly rinsing in running water.
Almond hulls	Whole commodity after removing shell and nutmeat.
Cereal grains group	Whole commodity (grain) except for fresh corn (including sweet corn). Include kernels plus cob after removing and discarding husk.
Eggs	Whole commodity after removing and discarding shells.
Fish	Edible portion of the commodity after removing and discarding heads, tails, scales, fins, viscera, bones (if inedible), and skin (if inedible).
Crab (hard shell)	Edible portion of commodity after removing and discarding shells, gills, and viscera.
Crab (soft shell)	Edible portion of commodity after removing and discarding gills.
Shrimp and crayfish	Edible portion of commodity after removing and discarding heads, shells, and inedible tails of shrimp.
Lobster	Edible portion of commodity including tomalley (liver) after removing and discarding shells and stomachs (hard sac near head).
Oyster, clam, and other shellfish	Edible portion of commodity including the liquor, after removing and discarding shells.
Rabbits and other game	Edible portion of commodity after removing and discarding bones.

Processed Foods

In the absence of EPA regulations, FDA also developed the instructions listed in Table 102-b on the portion of processed food to be analyzed for tolerance enforcement purposes. These instructions, like the ones for raw agricultural commodities, ensure uniformity and consistency in FDA analysis of processed food for pesticide residues. The instructions take a practical approach for sample preparation of processed food; *e.g.*, fruit juice concentrates that are normally reconstituted before consumption are also reconstituted prior to analysis for pesticide residues. Therefore:

- Follow the directions in Table 102-b to prepare test samples of processed foods.

Table 102-b: Portion of Processed Food to be Analyzed for Pesticide Residues

Processed food consisting of one ingredient and sold in a ready-to-eat form (*e.g.*, canned fruits packed in syrup or their own juice, canned vegetables packed in water or brine, or frozen fruits or vegetables, dried fruits, single-strength juices, catsup)	Analyze the whole processed commodity including any liquid or other edible media in which the commodity is packed. Discard inedible media, *e.g.*, brine.
Processed food consisting primarily of one ingredient and sold in a form requiring further preparation before it is ready to eat (*e.g.*, fruit juice concentrates, dehydrated vegetables, and powdered potatoes)	Analyze the whole processed commodity after compensating for or reconstituting to the commodity's normal moisture content.
Processed food in a form not ready to eat, used as an ingredient or component of other food (*e.g.*, flour, tomato concentrates such as paste, and citrus oils)	Analyze the whole processed commodity on an "as is" basis.
Cheese	Analyze the whole commodity including natural cheese rind after removing and discarding waxed or oiled rinds.
Frozen seafood (*e.g.*, fish or shrimp)	Analyze the edible portion after thawing; discard water.
Canned seafood	Analyze the edible portion including edible liquor and media, such as oil, broth, or sauces in which commodity is packed. Discard media that is not edible.
Frog legs	Analyze the edible portion of commodity after removing and discarding bones.

102 C: COMPOSITING AND COMMINUTING THE LABORATORY SAMPLE

Laboratory samples are comminuted or homogenized so that the relatively small (25-100 g) test portion taken for analysis is representative of the entire sample. Meaningful residue data can only be obtained when sample representation is preserved. Specialized equipment is employed to provide as much homogeneity as possible for the particular commodity.

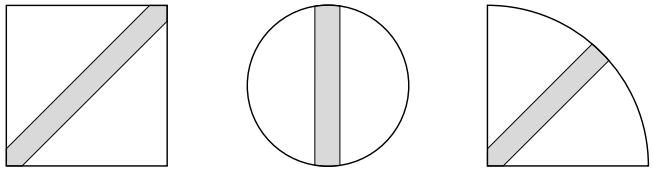
The general procedure for comminuting the commodity is:

- Comminute the test sample, prepared according to Section 102 B directions.
- Use comminuting operations (grinding, chopping, *etc.*) that produce the best possible homogenate for that commodity. Chopping procedures adequate for fruits and vegetables are often inadequate for homogenizing commodities such as dried hays and fish. See Section 203 for useful information about equipment and procedures.
- After comminuting, handle the homogenate carefully to minimize loss of residue by volatilization and concentration of residue through physical separation of product.

Exceptions to the general rule occur in several situations related to commodity type and special directions for the particular analysis. For example, removal of pits or caps from large quantities of small units can be too time consuming; similarly, melting, dicing, shredding, or blending a large unit of a food commodity such as butter or cheese is impractical. The following procedures are to be used in these situations:

- When the commodity consists of small units (*e.g.*, grains, cherries, nuts, dried peas and beans), mix and reduce by quartering to approximately 4 lb or 4 qt. Prepare the portion of commodity from this smaller amount, according to the appropriate directions in Table 102-a or 102-b. Chop or grind that material to obtain >1 lb or >1 qt comminuted sample.
- When the laboratory sample consists of large units of commodities of homogeneous nature (*e.g.*, butter, cheese), prepare the test sample by taking equal portions from each packaged unit. Select the appropriate portion of commodity (for cheese, see Table 102-b) and comminute by dicing, shredding, or blending.
- When the laboratory sample consists of large blocks, wedges, or wheels of cheese, take fraction shown as shaded area in Figure 102-a. Select the appropriate portion of commodity for cheese (Table 102-b) and comminute by dicing, shredding, or blending.

Figure 102-a
Fraction of Cheese Units to Take for Compositing



102 D: HANDLING SAMPLES FOR SPECIAL ANALYSES

Analyses for residues of ethylenebisdithiocarbamates (EBDCs) require special handling of the laboratory sample. EBDCs decompose rapidly as soon as the crop surface is broken and residues contact water, enzymes, and sugars [3]. Recoveries continue to decrease with time of contact in aqueous crop solution. Samples for EBDC analysis must either be analyzed immediately or frozen for storage.

Follow these directions for samples to be analyzed for EBDCs:

- Select representative units for EBDC analysis prior to chopping, grinding, or blending the laboratory sample.
- If the individual units are small and free flowing (*e.g.*, grains, beans, berries), mix well and take whole units for analysis.
- If the individual units are large, take wedges from each unit. Analyze immediately or freeze immediately after cutting.
- If the commodity contains free juices (*e.g.*, tomatoes, apples, oranges) and also requires cutting in pieces to fit into the digestion apparatus, freeze representative whole units before cutting. Dice frozen units without allowing them to thaw; mix and take sample for analysis.

102 E: RETENTION OF RESERVE PORTIONS

The following directions apply to all test samples (*i.e.*, comminuted material prepared from appropriate portion of commodity):

- Select three 1 qt portions from the total sample homogenate; identify them, respectively, as: "original analysis," "check analysis," and "reserve."
- Take analytical test portions from the "original analysis" and "check analysis," as appropriate, and analyze. Store the "reserve" portion in a freezer to provide to the claimant if requested.
- In addition to their sample homogenate, remaining fractions of large samples (*e.g.*, blocks of cheese) must be retained in a manner that prevents decomposition of product and/or residue. This requires that all products be frozen until findings of original analysis have been verified. The amount of commodity retained is governed by the extent of analysis required on the sample. However, in no case should portions be <1 qt each (or for products of high density, <1 lb) for original, check, and reserve.

References

[1] 40 CFR 180.1(j)

[2] 40 CFR 180.157

[3] Cullen, T.E. (1964) *Anal. Chem.* **36**, 221-224

103: METHOD APPLICATION IN REGULATORY ANALYSIS

103 A: INTRODUCTION

The Pesticide Analytical Manual (PAM) is published to provide analytical methodology for determination of pesticide residues in foods. Method application in a tolerance enforcement program imposes certain requirements and restrictions, and this section provides information about FDA operations concerning choice of method, identification, quantitation, and confirmation of residues(s), and documentation of analysis. Procedures established to support enforcement also apply to analyses that result only in monitoring data (*i.e.*, nonviolative samples), to the degree required in a program that fulfills both needs. (When PAM methods are used by other organizations for different purposes, *e.g.*, environmental monitoring without regulatory consequences, application requirements may vary from those expressed here.)

103 B: CHOICE OF METHOD

To support enforcement action against a commodity, original, check, and any additional analyses must be performed using official methods or methods shown by the analyst to support the validity of the result. The following minimum evidence is required to demonstrate that a method is valid for a particular analyte in a particular commodity:

1) Reagent blank analysis performed using reagents only (no commodity) shows no detector responses that could be mistaken for the analyte.

2) Historical or concurrent analyses of a residue-free lot of the same or similar commodity show no interfering detector responses.

3) Recovery of the analyte, added to a residue-free sample at or near the level of residue in the violative sample, is in the range 80-110%. If a residue-free lot is not readily available, recovery determination may be performed on another portion of the sample of interest by fortifying it with at least twice the level of residue found (*e.g.*, add 2 ppm if original analysis is 1 ppm).

Validation tests may be performed by either the original or check analyst and must be carried out concurrently with analysis of the sample of interest. The method used to determine any illegal residue found in a commodity for the first time by a laboratory must be validated in this way.

Analytical methods may be taken from the following sources, in decreasing order of preference:

PAM

Analytical methods from the PAM are suitable for regulatory analysis. As specified by regulation in the CFR, PAM contains the methods FDA uses for determining compliance with pesticide tolerances [1]. Volume I methods are capable of determining more than one residue (multiresidue methods, MRMs) and are most often used for routine analysis.

PAM I MRMs are presented as choices of several extraction, cleanup, and determinative modules. At the beginning of each method section in Chapters 3 and 4, method combinations that have undergone interlaboratory validation are listed. Absence of a particular combination of modules from this list does not prevent its use in regulatory analysis, but the analyst must provide suitable supporting evidence, as described above, that the combination is indeed valid for the particular commodity and residue combination.

PAM II methods are designed to determine residues of a single pesticide (single residue methods, SRMs). SRMs are published in PAM II for residues of all pesticides subject to EPA tolerances. These methods, usually from the pesticide manufacturer that petitioned for the tolerances, may be used by FDA to target residues not determined by MRMs. SRMs are also useful for check analyses when a residue has been determined first by an MRM, especially when a residue determined by an MRM is known to represent only part of the expected residue of a particular pesticide. For example, it may be known that the MRM does not completely recover the residue, or that the tolerance definition of the pesticide residue includes metabolites not detectable by the MRM. In these cases, MRMs may detect the presence of a residue but further analysis must be performed using a PAM II SRM to determine the total residue.

Other "Official" Methods

AOAC International maintains a system of interlaboratory testing through which methods passing the requirements are designated "AOAC Official"; many PAM I methods have undergone this process and are AOAC Official for certain residue/commodity combinations. Other AOAC official methods are not included in PAM but are acceptable for use in FDA regulatory analyses; AOAC methods have also been designated in the CFR as methods suitable for use [2]. In some cases, the AOAC method is preferable; *e.g.*, AOAC method 977.19 [3], official for hexachlorobenzene and mirex in adipose tissue, is preferred to PAM I method 304, through which the two chemicals involved are incompletely recovered.

Other Published Methods

When investigational evidence suggests that a commodity may contain a residue for which no PAM I or other official method is acceptable, published methods from the scientific literature must be sought. Use of such a method must be supported by documented evidence of its applicability to the residue and commodity in question, in the hands of the analyst who performed the regulatory analysis. The requirements described above are the minimum acceptable as supporting evidence.

Liquid-liquid partitioning and column chromatography cleanup steps that vary from established versions only in the use of proportionately smaller amounts of reagents are usually considered equivalent to the original; such methods are often referred to as "scaled-down" or "miniaturized." Miniaturized procedures that are published, validated, and used routinely in FDA are included as method modules in PAM I, *e.g.*, the 4 g Florisil column of Section 302 C1, developed as an alternative to the 20 g column. Certain other miniaturized versions of PAM I method modules have not undergone validation, but analyses performed in this way are considered adequate to support enforcement action and to assume equivalent coverage of residues for monitoring purposes. Extraction steps that involve smaller sample weight and extractant volume have not yet been studied sufficiently to

endorse, however; it is recommended that miniaturization be applied only to steps subsequent to filtration of the original extract.

103 C: TENTATIVE IDENTIFICATION

Application of methods in this manual results in tentative identification of residues based on the analyte's behavior matching that of a reference standard. "Behavior" of the analyte refers to its recovery through methods, including the eluate(s) in which it elutes from cleanup columns, its GLC or HPLC retention time, and the response it elicits from selective detectors or detection systems.

PAM I tables list test results of chemical behavior through various analytical procedures. The tables are provided to supply analytical chemists with information useful in residue identification. Typically, a sample is analyzed by a particular method and the extract examined by one or more determinative steps. When a GLC or HPLC response is recorded, the analyst measures the retention time of the response, calculates a relative retention time, and refers to the appropriate PAM I table (*e.g.*, Appendix I, PESTDATA; Table 403-a) to find chemicals that elute at that approximate time. Detector response data and molecular formulas included in PESTDATA offer further clues about which residue is likely to have caused response by the GLC element-selective detector used. Other details of chemical behavior through methods provide the analyst with means to eliminate certain candidate chemicals from consideration and strengthen the case for others; *e.g.*, if the method included a column chromatographic cleanup step and more than one eluate was used, only those chemicals known to elute in the pertinent eluate should be considered further.

In most cases, this preliminary evaluation provides the analyst with a limited number of potential candidates for residue identification. A reference standard solution of each likely chemical is then chromatographed on the appropriate GLC or HPLC system for direct comparison with the residue. In no circumstance is a table of data alone adequate for residue identification.

The expertise of the residue chemist is most critical during the determinative step of the analysis. The chemist's knowledge of pesticide usage and the chemistry and metabolism of pesticides is invaluable to the correct interpretation of evidence. Familiarity with commonly encountered artifacts from commodities, reagents, and environmental contaminants helps avoid incorrect conclusions.

103 D: RESIDUE QUANTITATION

Quantitation of residues is considered appropriate when:

1) The level of residue in a commodity is quantitated according to standard practices of GLC (Section 504) and HPLC (Section 606). In addition:

   a) Peak sizes of sample and standard match within ±25%.

   b) Time between sample and standard injection is ≤1 hr; chromatographic sequence ends with a standard.

   c) Replicate injections of standard and sample have been made when determining difficult-to-chromatograph residues.

2) Directions from Section 104 have been followed for summing levels of related residues for purposes of determining compliance with regulations.

3) Determination occurred at conditions that provided a limit of quantitation as directed in Section 105.

103 E: CONFIRMATION OF IDENTITY

Because analytical processes are subject to possible error in interpretation or measurement, confirmatory evidence must be developed to increase confidence in the tentative residue identification (Section 103 C). Attempting to define minimum confirmation requirements that are adequate for every situation is impractical. Instead, a philosophy of confirmatory analyses and discussion of certain minimum expectations are presented.

The extent of confirmatory effort will be influenced by the significance of the sample, nature and level of the residue, sample history, purpose of the analysis, and practical considerations such as time, cost, number of other samples, *etc.* The choice of confirmatory procedures depends on the tentative identity of the pesticide, amount of residue available for testing, sample type, and availability of instrumentation required for confirmatory tests.

The logic of most confirmatory schemes relies upon presumptive evidence; *i.e.*, if the behavior of an unknown in a particular analytical technique is the same as that of a reference standard, it is presumed that they are the same chemical. Any analytical technique that measures a single property of an analyte may err in distinguishing between two chemicals that behave the same; *e.g.*, two different chemicals may have the same GLC retention time. To avoid this potential error, either the analytical technique must inherently measure more than one property of the analyte or more than one analytical technique must be employed, each of which measures a different property. Following this logic, FDA laboratories confirm most residues by one of two approaches:

1) FDA requires that mass spectrometry (MS) be used to confirm the identity of any residue found for the first time. Modern laboratories usually have access to compact, highly automated mass spectrometers, configured as either mass-selective or ion trap detectors for GLC. Such instruments are capable, in some instances, of simultaneously detecting, quantitating, and confirming the residue, especially when reference standards are available. Errors may still occur, however, if GLC-MS is operated in the single ion monitor mode, which is not adequate to distinguish between two chemicals that elute from the column at the same time and are detected at the same m/z. For identification to be confirmed, GLC-MS must be operated by monitoring at least three ions [4].

   Full spectrum MS on a high resolution instrument may be required for full structural elucidation of previously unidentified chemicals. Such analyses usually require research MS instruments, operated by specialists, that offer several different modes of ionization that may be needed for unambiguous identification.

2) Residues previously reported may be confirmed by less rigorous techniques, such as additional chromatographic analysis (GLC or HPLC), with different

columns, mobile phases, and/or detectors. Confirmation of identity requires an accumulation of corroborating evidence sufficient to prove that the residue and reference standard must in fact be identical because they behave the same way in different tests. Such evidence is provided by measurement of a different chemical or physical property in each test used.

Selective MRMs (Chapter 4) are designed to be applicable to residues of a single chemical type; the steps of the method provide some built-in confirmation because only residues with that chemistry are recovered and determined. Other available confirmatory analyses are referenced as part of these method descriptions.

The GLC determinative steps included with multiclass MRMs (Section 302 DG1-DG23) offer a series of alternatives that can be used as confirmatory analyses. To the degree that a detector is selective to a single element or group, its use inherently provides some confirmatory evidence during the original tentative identification. Additional chromatography using other element-selective detectors is ideal for confirming a residue that contains appropriate elements. For example, many chemicals contain both nitrogen and phosphorus, others both phosphorus and sulfur. Complementary evidence from phosphorus and sulfur mode flame photometric (FPD-P and FPD-S), N/P, and electrolytic conductivity nitrogen mode (ElCD-N) detectors, as shown in Table 103-a, can provide excellent confirmatory information.

Table 103-a: Information Provided by Use of Element-Selective Detectors

Detector	Response	Conclusion
Example 1		
N/P[1]	Measurable	Either N or P in molecule
and		
FPD-P[2]	None	N in molecule
Example 2		
N/P	None	No N or P in molecule
and		
FPD-P[2]	Measurable	Large amount of S in molecule (verify with FPD-S[3])
Example 3		
N/P	Measurable	Either N or P in molecule
and		
FPD-P	Measurable	P in molecule; N also possible (verify N with E1CD-N[4])

[1] responds to both N and P
[2] responds to P; large amount of S can also cause response
[3] responds to S; presence of P can also cause response
[4] responds to N only

No detector is completely element-specific, however, and the analyst must be aware of the potential for false-positive responses. Replacement of the relatively nonselective electron capture detector with element-selective detectors encouraged development of methods with minimal cleanup. These methods are popular because they reduce analytical time and solvent volumes, and because they permit determination of residues that are removed by traditional column chromatographic cleanup steps (Section 301). However, extracts from such methods contain relatively large amounts of co-extractives, to which even selective detectors may respond. Columns of dissimilar polarity should be used with the various element-selective detectors to strengthen confirmation.

Special precautions are necessary when determining residues in which nitrogen is the only element to which element-selective detectors respond. Because many nonpesticidal organonitrogen chemicals occur naturally in foods, chromatograms from nitrogen-selective detectors often display a pattern of responses related to the commodity being analyzed. Additional columns and/or cleanup steps should be employed to confirm identification of residues found with nitrogen detectors.

Other techniques are available for confirmation if chromatography with element-selective detectors is not applicable or available. Chemical or photolytic derivatization and thin layer chromatography are among those most frequently employed.

103 F: DOCUMENTATION

FDA regulatory operations require that analytical results be adequate to support enforcement actions, if necessary, in a court of law. The analytical package accompanying a recommendation for enforcement action must demonstrate that the requirements described above for sampling, sample handling, and analysis have been met. Descriptions of analytical operations must be documented in a format readily understandable to another residue chemist and explainable to nonscientists. To that end, FDA established the following minimum requirements for documenting analyses that support recommendations for enforcement action against food or feeds because of the presence of violative pesticide residues.

Analytical Reports

Analytical reports for both original and check analyses must provide complete information on the sample and on sample handling steps used, as well as the following analytical information:

1) Method reference or memorandum of analysis, including description of any modifications made to the referenced method

2) Measured weights and volumes used for sample weight calculations

3) Volume of final sample solution or eluate

4) Details of residue determination record

   a) Sample solution/eluate identity (*e.g.*, 6% ethyl ether/petroleum ether)

   b) Sample weight per unit volume

c) Volume and sample weight equivalent injected

d) Retention time or distance and retention time relative to the marker compound appropriate to the system

e) Peak size; area or height for sample(s) and standard(s)

5) Calculation of results; may also appear on chromatograms

6) Column and detector used for each injection

Chromatograms

Each chromatogram must be labeled with identity of solution, volume and weight injected, date, analyst's initials, and time of injection. Time is not required if a continuous chromatogram is submitted. The following chromatograms must be submitted:

1) Marker compound. For each column-detector combination used, a chromatogram of the reference standard used as a marker compound for that combination; it is preferable for additional reference standards also to be included in a mixed standard solution appropriate to the determinative step.

2) Sample and standard chromatograms used for quantitation

3) Any other chromatograms (for both sample and standard) associated with additional tests to confirm identity of the residue

Chromatographic Data

The following information must be included on the chromatograms. (If a data collection system is used, much of the required information can be automatically entered into the chromatographic report.)

1) Brand name and model of chromatograph

2) Column: type, size, liquid phase, plus solid support and percentage loading for GLC packed column; coating identification, film thickness, length and internal diameter for GLC wide bore or capillary column; packing identification, length, and internal diameter for HPLC column

3) Temperatures: column, detector, injector, transfer lines, furnaces, *etc.*

4) Description of injector and/or inlet system

5) Gas flow rates and identities for carrier, fuel, purge and make-up gases (GLC); mobile phase, gradient if applicable, flow rate (HPLC)

6) Detector type, including design, mode of operation, and specifics related to operation of the particular detector, such as ion exchange resin, electrolyte, and electrolyte flow rate of the electrolytic conductivity detector

7) Range settings of electrometer, integrator, linearizer, *etc.*, along with pertinent detector voltages

8) Parameters for signal measurement: recorder span and speed, integrator settings, *etc.* (recommended chart speed is 1 cm/min or 0.5"/min)

References

[1] 40 CFR 180.101(c)

[2] 21 CFR 2.19

[3] *Official Methods of Analysis* (1990)15th ed., Association of Official Analytical Chemists, Inc., Arlington, VA

[4] Sphon, J.A. (1978) *J. Assoc. Off. Chem.* **61**, 1247-1252

104: ANALYTICAL RESULTS

104 A: INTRODUCTION

Three separate but related responsibilities must be managed within any regulatory program of analyses for pesticide residues: (1) determination of residue identity and calculation of level, (2) reporting residue identity and level, and (3) judging whether the presence of that level of residue is in compliance with regulations or whether it warrants enforcement action.

The first responsibility, residue identification and calculation, is a scientific endeavor treated in the remaining chapters of this manual; regulatory requirements for these operations were discussed in Section 103. Regardless of the purpose of the analysis, the basic instructions remain the same. Appropriate application of Chapters 3 or 4 methods, combined with the precepts of accurate quantitation described in Sections 504 and 606, will permit the chemist to ascertain the presence or absence of particular residues and to calculate the quantity of each residue present in the sample.

Beyond the scientific endeavor, the use to which analytical results are put is within the province of the organization sponsoring the work. In this section, current FDA procedures for reporting residues and determining compliance with prevailing regulations are noted. Operations are often dictated by the needs of the dual-purpose FDA pesticide program: to monitor incidence and levels of pesticide residues in foods and to enforce regulations concerning permissibility of these residues.

104 B: REPORTING

In this context, reporting the presence and levels of pesticides refers to laboratory entry of information into a computerized data base. FDA has monitored residue trends for more than 30 years by reporting results of all analyses, including those in which no residues were found, into a data system. Pertinent information deduced from accumulated data includes the decline in residue levels of persistent chlorinated hydrocarbons in the years following their ban and the identity of terminal residues in different commodity types. One goal of the residue reporting system is to retain all pertinent information obtained during the analysis; of special interest is the precise identity of the residue. Accumulated reports of residue analyses are used to answer many questions about the prevalence of residues in the food supply; most prominently, the data are used to prepare an annual report of residue findings [1].

The following reporting practices have evolved over the years to produce the most meaningful possible data for agency interpretation:

Portion of Commodity

The exact portion of food that is analyzed for pesticide residues is dictated by the purpose of the analysis. Section 102 reflects agency procedures for the portions of particular commodities used in FDA regulatory monitoring. Once analyzed, residue levels are calculated and reported based on the exact portion of food taken for analysis as indicated in Table 104-a.

Table 104-a: Portion of Commodity for Calculation and Reporting of Residue Levels

Commodity	Report Results On:
Raw agricultural commodities	Whole commodity as prepared for analysis
Milk products[1]	Whole product
Juice concentrates or powders	Reconstituted basis
Concentrated/dehydrated products consumed "as is" or used as ingredients	Whole product, "as is"
Dehydrated vegetables intended for use *after* reconstitution	Calculated equivalent weight of original product before dehydration
Other processed foods	Whole product

[1] Includes whole, low fat, skim, and other milk products. Note that reporting residues in whole milk on the whole basis (effective 10/1/91) does not change the way of determining compliance with regulations for residues whose tolerances in milk are on the fat basis.

Nature of the Residue

Whenever possible, pesticide residues in foods are identified and calculated as individual chemicals (*i.e.*, parent compound, metabolites, and degradation products). Although many pesticides are formulated and marketed as technical mixtures of related chemicals, their residues are calculated separately whenever chromatographic conditions and availability of separate reference standards permit.

Residues are reported as the individual isomer or congener that is identified and calculated during the determination. If the residue can be identified and its level calculated only by comparison to a mixed or technical reference standard, the residue may be reported as such in the data system.

Residues of polychlorinated biphenyls (PCBs) are calculated by comparison to commercial mixtures known as Aroclors (Section 504 D). Levels are reported in terms of the particular Aroclor(s) used as reference standard.

Residues Measured from Derivative or Breakdown Product

Some analytical methods convert the residue(s) to a derivative or breakdown product so that common determinative steps can be used. In these cases, some convention must be established for reporting the residues. Specific instances of this situation are:

Acids and Phenols. Residues of acids and phenols are converted to respective methyl esters/ethers by Section 402 method, to produce chemicals that can be measured by GLC. Reference standard solutions are prepared from standards of the ester/ether, if available, or from standards of the acid/phenol carried through the procedure. Levels of residue are calculated and reported as the acid or phenol.

Benomyl, Thiophanate-Methyl, and Carbendazim (MBC). Residues of these benzimidazole pesticides and related residues are determined by Section 404. MBC, the most common residue, may result from use of benomyl, which converts rapidly to MBC; thiophanate-methyl, which converts slowly; or carbendazim, a fungicide that is the same chemical as MBC. Residues are reported according to assumptions made about the source of MBC found.

In absence of evidence to the contrary, a residue of MBC is assumed to result from use of benomyl. MBC residues are quantitated by comparison to a reference standard of MBC and the level converted to the equivalent benomyl level, which is reported.

However, if MBC and thiophanate-methyl are both found in the sample, or if investigatory evidence indicates the commodity was treated with thiophanate-methyl, the quantitated level of MBC is converted to equivalent thiophanate-methyl and reported as such.

EBDCs. Tolerances for EBDCs are established in terms of parts per million (ppm) zineb, one of the EBDCs. Residues of EBDCs are determined by methods that convert these chemicals to carbon disulfide, which is measured and calculated as zineb. This analytical approach precludes identification of which EBDC was present. By convention, FDA laboratories report levels found as "EBDC (identity unknown)," unless investigatory evidence suggests which of the EBDCs was used on the product.

Because registrations for zineb have been cancelled, supplies of the chemical may not be available for use as a reference standard. If necessary, another EBDC analytical standard may be used and appropriate molecular weight conversion made to permit reporting residues in terms of ppm zineb.

Significant Figures

The level of each residue that appears at or above its limit of quantitation for the method is calculated (Section 105 discusses limit of quantitation). Residues are calculated and reported in ppm. Unless the quality of the chromatography or other factors necessitates fewer significant figures, levels are reported as follows:

≥100 ppm	to nearest ppm
10 to 99.9 ppm	to nearest 0.1 ppm
1 to 9.99 ppm	to nearest 0.01 ppm
0.010 to 0.999 ppm	to nearest 0.001 ppm

Trace

Residues that are detectable by the method but present at less than the limit of quantitation are reported as "Trace."

Confirmation

Identities of residues are confirmed before reporting, according to the principles discussed in Section 103. Confirmation of nonviolative levels of frequently found residues do not require confirmation in every sample; frequency of confirmation is at the discretion of the laboratory.

104 C: DETERMINING COMPLIANCE WITH REGULATIONS

For monitoring purposes, all residues of the same pesticide are calculated and reported as individual chemicals. For tolerance enforcement purposes, however, the residue definition as stated in the particular regulation applies; in some situations, not all residues are included in the total residue for determining compliance with the tolerance. FDA's Compliance Policy Guide [2] provides criteria that must be met to initiate an enforcement action for violative pesticide residues found in a food commodity. Directions included in this section assume that quantitation has been accurately performed, according to directions in Sections 504 and 606, and that individual residues have been reported into the data system.

General Rule for Multicomponent Residues

Residues that consist of more than one isomer of a technical pesticide, or that consist of parent and degradation products, are added together to determine compliance with existing tolerances, insofar as the degradation products are included in the tolerance expressions of 40 CFR Parts 180, 185, or 186 [3].

Special Situations

BHC. Determining compliance of residues of BHC is complicated by the fact that γ-BHC, also known as lindane, is marketed as a separate pesticide and is also an isomeric component of technical BHC, which may have up to six different isomers. At one time separate tolerances for BHC and for lindane were established, and the possibility existed that both might be used on the same commodity. Currently, U.S. tolerances for BHC have been revoked, but residues are still found in domestic and imported commodities; U.S. tolerances for lindane remain for several commodities.

When residues of BHC isomers are found in a commodity, the quantity of each isomer that is present is calculated against an individual reference standard, according to the general principle; quantities of α, β, and δ isomers are then added together. If the amount of γ-BHC is $<1/3$ the total of α+β+δ, the total of the four isomers is considered to be a residue of BHC. If the γ-isomer is $>1/3$ the total of α+β+δ, the amount in excess of $1/3$ (α+β+δ) is considered to be lindane and the remainder of the γ plus α, β, and δ is considered BHC. Appropriate regulatory action is decided based on these calculations.

Chlordane, Heptachlor, Heptachlor Epoxide. Two factors complicate the residue situation for chlordane: (1) chlordane is a multicomponent mixture whose terminal residue pattern varies considerably, and (2) one component of technical chlordane is heptachlor, which was marketed as a separate pesticide. At the time when both chlordane and heptachlor were registered for food use, the possibility existed that they might be used on the same product. U.S. tolerances for both chlordane

and heptachlor are now revoked, and most residues now occur in fish as a result of lingering environmental contamination, but the procedures developed during the previous period still apply if needed.

Section 504 outlines the prescribed method for quantitating chlordane residues, either against a technical standard or against individual reference standards, depending on the residue pattern. It also specifies that peaks at the retention times of heptachlor and heptachlor epoxide can be included in quantitation of chlordane against the technical standard, if those peaks are relatively small.

If chlordane residues are calculated as individual terminal residues, they are added together to determine total chlordane. If measured against a technical chlordane reference standard, the calculated value is considered total chlordane. In either case, heptachlor and heptachlor epoxide peaks are included as part of total chlordane if they are relatively small and in reasonable proportion to the rest of the residue. If heptachlor and/or heptachlor epoxide are much out of proportion, as shown in Figure 504-d, they are considered as separate residues. Appropriate regulatory action is decided based on these calculations.

PCBs. Regulations related to PCB residues establish tolerances for "PCBs," so compliance is based on total PCBs calculated, regardless of which Aroclor(s) was used as reference standard.

Residues of More Than One Pesticide. In certain cases, pesticides "that cause related pharmacological effects [are] regarded, in the absence of evidence to the contrary, as having an additive deleterious action" [3]. Special rules for adding together residues in these categories may apply.

Residues Calculated From a Derivative. As described above under reporting, some residues can be quantitated only by methods that form a derivative prior to the determinative step. Compliance with regulations in these cases depends on the precise statement of the regulation and on investigatory evidence related to the particular sample. For regulatory purposes, the residue level must be calculated in terms of the chemical(s) specified in the tolerance; if necessary, a conversion is made.

References

[1] Food and Drug Administration (1993) *J. AOAC Int.* **76**, 127A-148A, and previous annual reports

[2] *Compliance Policy Guide*, Section 7141.02, Food and Drug Administration, Rockville, MD

[3] 40 CFR 180.3

105: ANALYTICAL LIMITS OF QUANTITATION

105 A: DEFINITION

FDA defines limit of quantitation (Lq) as the lowest level of residue that can be quantitated by a given method and whose identity can be confirmed in regulatory laboratories operating under routine conditions. Levels less than the Lq are defined as trace.

When MRMs are used, a separate Lq applies to each residue determined by the method because each represents a different analytical situation.

The following factors must be specified in order to define the analytical situation; only then can an Lq be calculated:

1) Analytical method used

2) Sample (matrix) type

3) Sample weight equivalent introduced to the determinative step

4) Sensitivity of the determinative step to the analyte; sensitivity is dependent on the following instrumental conditions:

   a) Determinative technique (In MRMs, the determinative step is usually GLC or HPLC; operational parameters must be defined as part of the method description.)

   b) Range of analyte weight that produces a linear detector response

   c) Overall condition of the system

   d) Amplification and/or attenuation of the detector signal

   e) Characteristics of the signal processing or recording device

   f) Chromatographic elution characteristics of the analyte

105 B: CALCULATION

FDA Lqs for each method are arrived at by (1) specifying a sample weight equivalent to be examined by the determinative step (the amount chosen must be compatible with long-term instrument stability); (2) establishing a recommended determinative step sensitivity that is stable, reproducible, and achievable by all laboratories; and (3) establishing a response equivalent to 10% of full scale deflection (FSD) on the signal-processing device as the minimum considered quantifiable and confirmable. FDA methods applied according to these guidelines are capable of analyzing for most residues at levels well below established tolerances.

Determinative step sensitivity is established by reference to a "marker compound"; *i.e.*, the instrumental parameters are adjusted to cause a specified response to a specified quantity of the marker compound. This approach makes it possible for different laboratories to achieve approximately the same Lq even though the instrument settings may be different for each. Lq for the marker compound can then be calculated with the formula below for any particular method. Lqs for all other compounds recovered through the method will vary according to the determinative step sensitivities for each.

▶ With these guidelines established, Lq for a method is calculated thus:

$$\text{ng 50\% FSD} = \text{ng analyte injected} \times \frac{\text{ng marker specified}}{\text{ng marker injected}} \times \frac{\text{marker peak height}}{\text{analyte peak height}}$$

$$\text{ng 10\% FSD} = \text{ng 50\% FSD}/5$$

$$\text{Lq} = (\text{ng 10\% FSD})/(\text{mg sample injected})$$

▶ Round the Lq result following the guidance for significant figures and reporting analytical results in Section 104, page 104-3. For general purposes, results at or below 0.010 ppm are deemed to have an Lq of 0.010 ppm.

105 C: IMPLEMENTATION

Guidelines for applying analytical methods are required to provide consistency among laboratories performing regulatory analyses. Otherwise, variations in the amount of sample equivalent injected and/or the sensitivity of the determinative step can cause different Lqs in different laboratories. Lqs that result from following FDA guidelines are adequate for the enforcement of tolerances and, in most cases, are sufficient to determine residues below the tolerance level so that data on incidence and levels of residues in foods and feeds can be collected.

The following rules are established to maintain consistent Lqs among FDA laboratories:

- ▶ Establish the sensitivity recommended in each determinative step method module (*e.g.*, Section 302 DG1-DG12, Section 401 DL1). Note that the requirement for GC determinations to be based on columns of 100% methyl siloxane is in effect as of FY'98 (October 1, 1997); prior to that time, other DG modules may have been used to calculate Lq.

- Inject a volume of extract containing the equivalent sample weight recommended for each method (*e.g.*, Section 302, Determination).

- If one of the recommended specifications above cannot be achieved, or if changing one is advisable for any reason, adjust the other parameter to maintain the targeted limit of quantitation. Section 105 D describes factors that may cause problems in specific situations.

Table 105-a lists examples of Lqs that can be calculated from the recommended sample weight equivalent and determinative step sensitivity for particular PAM I methods. The list is not exhaustive but does illustrate the way in which the Lq for any method in PAM I can be calculated.

105 D: FACTORS AFFECTING TARGET LIMITS OF QUANTITATION

The following factors, individually or in combination, may reduce the certainty of quantitation and/or identification of a residue in any specific analytical situation. They may also cause the Lq to differ from the recommended limit defined by the formula above and by Table 105-a. Measures taken to compensate for one factor may trigger the influence of another.

1) Determinative step sensitivity to any particular residue. A distinct Lq applies to each residue determinable by a particular MRM, because the sensitivity of the determinative step to each compound may be different.

2) Limited detector sensitivity. Not all individual detectors are capable of reaching the sensitivity specified; in such cases, the Lq will be higher than targeted.

3) Greater detector sensitivity. Directions here recommend sensitivity at which detectors should be operated, even though some are capable of greater sensitivity. However, operation at conditions that produce recommended sensitivity may sometimes be precluded by other disadvantages in detector performance. For example, many models of ^{63}Ni electron capture detectors are not linear at conditions that produce sensitivity of 50% FSD to 1.5 ng chlorpyrifos, as is recommended for other detectors; most are linear, however, at conditions that produce 50% FSD to 0.15 ng chlorpyrifos. The rules in Section 105 C specify that, in this situation, the laboratory should operate at the greater sensitivity in order to work in a linear range, then proportionately reduce the weight of sample equivalent injected in order to maintain Lqs consistent with those achieved by other laboratories.

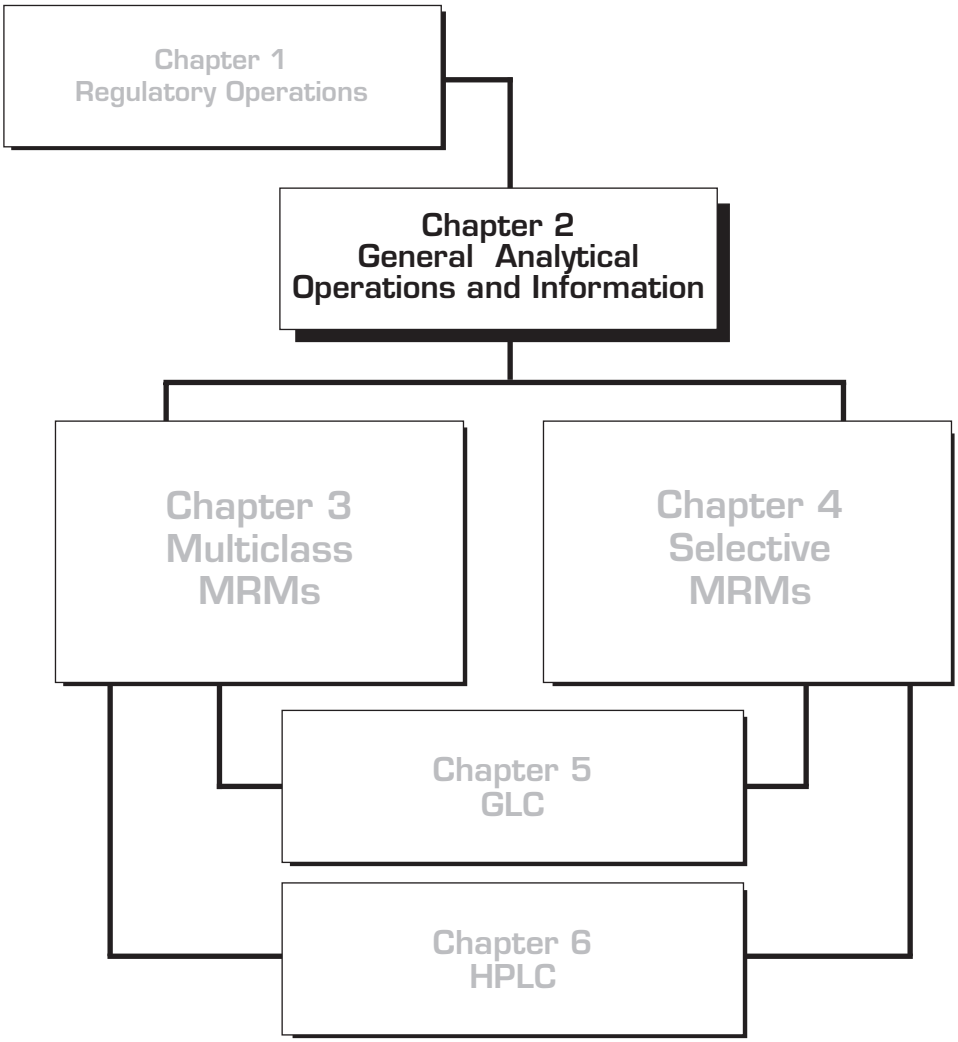
4) Other improvements that affect determinative step. Wide bore capillary GLC columns (Section 502 C) permit analytes to elute in a tighter band than was possible with packed column chromatography. When detector response is measured in terms of peak height, use of capillary columns results in an apparent improvement of response. Injection of a smaller amount of equivalent sample, as directed in Section 105 C, is appropriate and, at the same time, beneficial to the longevity of the column.

5) Excessive interferences from sample co-extractives. Interferences from sample co-extractives raise the Lq of a method by masking the detector response to the residue or by preventing injection of the specified sample equivalent without undesirable damage to the system. Additional procedures to clean up the sample extract prior to determination may improve the Lq by removing these interferences.

Table 105-a: Examples of Method Specifications Used to Calculate Lqs

PAM I Method[1]		Recommended Mg Injected	Recommended Sensitivity[2]	Lq (marker compound)[3]
302E1+DG2 (FPD-P)		20mg	1.5ng chlorpyrifos	0.015ppm chlorpyrifos
302E3+C1+DG3 (E1CD-X)		20mg	1.5ng chlorpyrifos	0.015ppm chlorpyrifos
302+E1+C3+DL1		116mg	10ng carbofuran	0.017ppm carbofuran
303E1+C1+DG1 (EC)	OR	20mg 2mg	1.5ng chlorpyrifos 0.15ng chlorpyrifos	0.015ppm chlorpyrifos 0.015ppm chlorpyrifos
304E4+C2+DG1 (EC)		10mg (cheese with 30% fat)	1.5ng chlorpyrifos	0.03ppm chlorpyrifos, whole product basis
401E1+C1+DL1		200mg	10ng carbofuran	0.01ppm carbofuran
402E1+C1+DG3 (fatty foods)		5mg Eluate 1	1.5ng chlorpyrifos (0.2ng PCP methylether)	0.008ppm PCP methylether
		10mg Eluate 2	1.5ng chlorpyrifos (0.5ng 2,4,5-T methylester)	0.01ppm 2,4,5-T methylester
402E2+C1+DG3 (nonfatty foods)		10mg Eluate 1	1.5ng chlorpyrifos (0.2ng PCP methylether)	0.004ppm PCP methylether
		20mg Eluate 2	1.5ng chlorpyrifos (0.5ng 2,4,5-T methylester)	0.005ppm 2,4,5-T methylester
403E1+C1+DL3		800mg	40ng diuron	0.01ppm diuron
404E1+DL5		125mg	62.5ng MBC	0.1ppm MBC
404E1+DL7		125mg	6.25ng thiabendazole (fluorescence detector)	0.01ppm thiabendazole

[1] Parenthetical codes indicate the detector used in the GLC determinative step.
[2] Ng marker compound that causes detector response of 50% FSD; where residues targeted by the method are different from the marker compound, weight of example target that caused 50% FSD is also listed.
[3] Calculated by formula in Section 105 B; note that sensitivity is divided by 5 to produce ng causing 10% FSD.



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201: PERCENTAGE FAT, WATER, AND SUGARS IN FOODS

Methods in Chapters 3 and 4 are usually designated as applicable to either fatty (>2%) or nonfatty (<2%) foods. In addition, some methods for nonfatty foods include alternative extraction steps, choice of which is dictated by the percentage water or sugars in the product. To facilitate proper application of these methods, this table provides percentage fat, water, and sugars for raw agricultural commodities and some processed foods.

Data were obtained from the following sources:

USDA Nutrient Data Base for Standard Reference, Release 8 and Release 9, U.S. Department of Agriculture, Washington, DC

Home Economics Research Report No. 48, "Sugar Content of Selected Foods: Individual and Total Sugars," Tables 1, 3, and 4, Stock No. 001-000-04515-8, Government Printing Office, Washington, DC 20402; obtained by download from bulletin board

Pehrsson, P. (Jan. 1994), private communication

Smith, J.S., *et al.* (1986) *J. Food Sci.* **51**, 1373-1375

Notes on the information in this table:

For the convenience of FDA field personnel, Office of Regulatory Affairs (ORA) product codes are included for most commodities, and commodities are grouped by the same categories used to create the codes. (Product codes are those used in the FDA reporting system; these are not the same codes used by USDA to identify commodities in the Nutrient Data Base.) Within each subcategory, items are sorted alphabetically by name of the commodity.

Names of commodities, including abbreviations, are those used by USDA Nutrient Data Base.

Percentage sugars represents the sum of one or more mono- and disaccharides. Data are not available for all commodities, but the table will be updated in the future as data become available.

For some commodities, sum of percent fat, water, and sugars may exceed 100%; this is caused by the fact that data were developed at different times on different samples.

Product Code	Commodity	% Fat	% Water	% Sugars
02: GRAINS				
A: whole grain				
02A-02	barley, pearled, raw	1.16	10.09	*
02A-99	buckwheat groats, roasted, dry	2.71	8.41	*
02A-01	corn, dry	2.08	10	*
02A-99	couscous, dry	0.64	8.56	*
02A-03	oat bran, raw	7.03	6.55	1.4
02A-03	oats	6.9	8.22	*
02A-04	popcorn: unpopped	4.7	9.8	*
02A-07	rye	2.5	10.95	*
02A-08	sorghum	3.3	9.2	*
02A-06	wild rice, raw	1.08	7.76	2.5
B: corn meal & milled corn products				
02B-01	corn flour, masa, enriched	3.78	9.03	*
02B-01	corn flour, whole-grain, white	3.86	10.91	*
02B-01	corn flour, whole-grain, yellow	3.86	10.91	*
C: rice flour				
02C-01	rice flour, brown	2.78	11.97	*
02C-01	rice flour, white	1.42	11.89	1
D: processed rice & milled rice products				
02D-99	rice bran, crude	20.85	6.13	0.9
02D-01	rice, brown, long-grain, raw	2.92	10.37	0.7
02D-01	rice, brown, medium-grain, raw	2.68	12.37	0.7
02D-10	rice, white, glutinous, raw	0.55	10.46	*
02D-03	rice, white, long-grain, parboiled, dry, enriched	0.56	10.16	*
02D-03	rice, white, long-grain, precooked or instant, enriched, dry	0.29	8.14	*
02D-03	rice, white, long-grain, regular, raw, enriched	0.66	11.62	0.5
02D-02	rice, white, medium-grain, cooked, enriched	0.21	68.61	0.2
02D-03	rice, white, medium-grain, raw, enriched	0.58	12.89	0.5
02D-02	rice, white, medium-grain, raw, unenriched	0.58	12.89	0.5
02D-03	rice, white, short-grain, raw, enriched	0.52	13.29	*
F: milled wheat product				
02F-04	semolina, enriched	1.05	12.67	*
G: other flour/milled products				
02G-02	buckwheat	3.4	9.75	*
02G-02	buckwheat flour, whole-groat	3.1	11.15	*
02G-10	rye flour, dark	2.69	11.07	3.5
02G-10	rye flour, light	1.36	8.78	3.5
02G-10	rye flour, medium	1.77	9.85	3.5

* Percent sugars not available.

Product Code	Commodity	% Fat	% Water	% Sugars
H: starch products				
02H-99	arrowroot flour	0.1	11.37	*
02H-02	cornstarch	0.05	8.32	*
09: DAIRY				
A: butter products				
09A-01	butter, wo/salt	81.11	17.94	*
09A-01	butter: w/salt	81.11	15.87	*
09A-01	butter: whipped	81.11	15.87	*
C: milk/cream				
09C-07	cream: fluid, half & half, cream and milk	11.5	80.57	*
09C-13	cream: fluid, heavy whipping	37	57.71	2.8
09C-13	cream: fluid, light whipping	30.91	63.5	2.8
09C-13	cream: fluid, light, coffee or table	19.31	73.75	*
09C-13	cream: fluid, medium, 25% fat	25	68.5	*
09C-12	cream: sour half & half, cultured	12	80.14	*
09C-04	eggnog	7.48	74.37	*
09C-03	milk: cow, cnd, condensed, sweetened	8.7	27.16	*
09C-03	milk: cow, cnd, evaporated, unsweetened, w/added Vit. A	7.56	74.04	*
09C-16	milk: cow, dry, whole	26.71	2.47	35.9
09C-09	milk: cow, whole, past and raw, fluid, 3.3% fat	3.34	87.99	4.9
09C-09	milk: cow, whole, past and raw, fluid, 3.7% fat	3.66	87.69	4.9
09C-12	sour cream	20.96	70.95	*
D: low fat milk product				
09D-03	milk: cow, cnd, evaporated, skim	0.2	79.4	*
09D-16	milk: cow, dry, skim, calcium reduced	0.2	4.9	*
09D-16	milk: cow, dry, skim, nonfat solids, instant, w/added Vit. A	0.72	3.96	50.4
09D-09	milk: cow, lowfat, past & raw, fluid, 1% fat	1.06	90.08	*
09D-09	milk: cow, lowfat, past & raw, fluid, 2% fat	1.92	89.21	*
E: non-fat milk product				
09E-01	milk: buttermilk, fluid, cultured, from skim milk	0.88	90.13	4.8
09E-16	milk: cow, dry, skim, nonfat solids, regular, w/added Vit. A	0.77	3.16	*
09E-11	milk: cow, skim, past & raw, fluid, w/added Vit. A	0.18	90.8	4.9
12: CHEESE				
A: standard cheese				
12A-02	cheese: natural, blue	28.74	42.41	*
12A-03	cheese: natural, brick	29.68	41.11	*
12A-56	cheese: natural, brie	27.68	48.42	*
12A-56	cheese: natural, camembert, domestic	24.26	51.8	*
12A-57	cheese: natural, caraway	29.2	39.28	*
12A-05	cheese: natural, cheddar, American domestic	33.14	36.75	1.8
12A-06	cheese: natural, colby	32.11	38.2	*

Product Code	Commodity	% Fat	% Water	% Sugars
12A-11	cheese: natural, cottage, creamed, large or small curd	4.51	78.96	0.6
12A-62	cheese: natural, cottage, low fat, 1% fat	1.02	82.48	*
12A-62	cheese: natural, cottage, low fat, 2% fat	1.93	79.31	*
12A-09	cheese: natural, cottage, uncreamed, dry, large or small curd	0.42	79.77	*
12A-10	cheese: natural, cream	34.87	53.75	1.7
12A-12	cheese: natural, edam	27.8	41.56	*
12A-16	cheese: natural, gouda	27.44	41.46	*
12A-18	cheese: natural, gruyere	32.34	33.19	*
12A-37	cheese: natural, limburger	27.25	48.42	*
12A-38	cheese: natural, monterey	30.28	41.01	*
12A-39	cheese: natural, muenster	30.04	41.77	*
12A-40	cheese: natural, neufchatel	23.43	62.21	1
12A-42	cheese: natural, Parmesan, grated	30.02	17.66	*
12A-52	cheese: natural, port de salut	28.2	45.45	*
12A-44	cheese: natural, provolone	26.62	40.95	*
12A-47	cheese: natural, romano	26.94	30.91	*
12A-49	cheese: natural, Roquefort	30.64	39.38	*
12A-60	cheese: natural, Swiss, domestic	27.45	37.21	1.2
12A-52	cheese: natural, tilsit, whole milk	25.98	42.86	*
B: standard cheese products				
12B-01	cheese food: cold pack, American	24.46	43.12	*
12B-09	cheese food: pasteurized processed, Swiss	24.14	43.67	*
12B-09	cheese: pasteurized processed, American, w/di Na phos	31.25	39.16	*
12B-13	cheese: pasteurized processed, Swiss, w/di Na phos	25.01	42.31	*
C: non-standard cheese products				
12C-99	cheese: natural, cheshire	30.6	37.65	*
12C-12	cheese: natural, feta	21.28	55.22	*
12C-04	cheese: natural, fontina	31.14	37.92	*
12C-99	cheese: natural, gjetost	29.51	13.44	*
12C-06	cheese: natural, mozzarella, part skim milk	15.92	53.78	*
12C-06	cheese: natural, mozzarella, part skim milk, low moisture	17.12	48.57	*
12C-06	cheese: natural, mozzarella, whole milk	21.6	54.14	*
12C-06	cheese: natural, mozzarella, whole milk, low moisture	24.64	48.38	*
12C-11	cheese: natural, ricotta, part skim milk	7.91	74.41	1.4
12C-11	cheese: natural, ricotta, whole milk	12.98	71.7	1.5
13: ICE CREAM, ETC.				
A: ice cream				
13A-02	ice cream: French, vanilla, soft serve	13.02	59.76	*
13A-03	ice cream: vanilla, regular, appx 10% fat	10.77	60.8	17.5
13A-02	ice cream: vanilla, rich, appx 16% fat	16	58.87	17.5
C: ice milk				
13C-01	ice milk: vanilla, hardened	4.3	68.62	*

Product Code	Commodity	% Fat	% Water	% Sugars
13C-99	ice milk: vanilla, soft serve	2.64	69.64	*
D: sherbet				
13D-01	sherbet: orange	1.98	66.07	*
14: MILK PRODUCTS				
B: imitation milk products				
14B-99	cream substitute: nondairy, liquid, w/hydr veg oil & soy protein	9.97	77.27	*
14B-99	cream substitute: nondairy, liquid, w/lauric acid oil & Na casn	9.97	77.27	*
14B-06	cream substitute: nondairy, powdered	35.48	2.21	*
15: EGGS AND EGG PRODUCTS				
A: shell eggs				
15A-01	eggs: chicken, white, raw, fresh and frozen	0	87.81	*
15A-01	eggs: chicken, whole, raw, fresh, and frozen	10.02	75.33	*
15A-03	eggs: duck, whole, fresh, raw	13.77	70.83	*
15A-99	eggs: goose, whole, fresh, raw	13.27	70.43	*
B: shelled egg products				
15B-02	eggs: chicken, yolk, dried	61.28	4.65	*
15B-02	eggs: chicken, yolk, raw, fresh	30.87	48.81	*
15B-02	eggs: chicken, yolk, raw, frozen	26.01	55	*
E: imitation/substitute egg products				
15E-03	egg substitute: frozen	11.11	73.1	*
15E-99	egg substitute: liquid	3.31	82.75	*
15E-99	egg substitute: powder	13	3.86	*
16: FISH				
A: fish				
16A-01	fish/shellfish: anchovy, European, raw	4.84	73.37	0.0
16A-03	fish/shellfish: bass, freshwater, mixed species, raw	3.69	75.66	0.0
16A-03	fish/shellfish: bass, striped, raw	2.33	79.22	0.0
16A-05	fish/shellfish: bluefish, raw	4.24	70.86	0.0
16A-99	fish/shellfish: burbot, raw	0.81	79.26	0.0
16A-08	fish/shellfish: butterfish, raw	8.02	74.13	0.0
16A-09	fish/shellfish: carp, raw	5.6	76.31	0.0
16A-10	fish/shellfish: catfish, channel, raw	4.26	76.39	0.0
16A-48	fish/shellfish: cisco, raw	1.91	78.93	0.0
16A-12	fish/shellfish: cod, Atlantic, raw	0.67	81.22	0.0
16A-12	fish/shellfish: cod, Pacific, raw	0.63	81.28	0.0
16A-13	fish/shellfish: croaker, Atlantic, raw	3.17	78.03	0.0
16A-14	fish/shellfish: cusk, raw	0.69	76.35	0.0
16A-51	fish/shellfish: dolphinfish, raw	0.7	77.55	0.0
16A-13	fish/shellfish: drum, freshwater, raw	4.93	77.33	0.0
16A-15	fish/shellfish: eel, mixed species, raw	11.66	68.26	0.0

Product Code	Commodity	% Fat	% Water	% Sugars
16A-16	fish/shellfish: flatfish (flounder and sole species), raw	1.19	79.06	0.0
16A-17	fish/shellfish: grouper, mixed species, raw	1.02	79.22	0.0
16A-18	fish/shellfish: haddock, raw	0.72	79.92	0.0
16A-20	fish/shellfish: halibut, Atlantic and Pacific, raw	2.29	77.92	0.0
16A-20	fish/shellfish: halibut, Greenland, raw	13.84	70.27	0.0
16A-21	fish/shellfish: herring, Atlantic, raw	9.04	72.05	0.0
16A-21	fish/shellfish: herring, Pacific, raw	13.88	71.52	0.0
16A-99	fish/shellfish: ling, raw	0.64	79.63	0.0
16A-99	fish/shellfish: lingcod, raw	1.06	81.03	0.0
16A-22	fish/shellfish: mackerel, Atlantic, raw	13.89	63.55	0.0
16A-22	fish/shellfish: mackerel, king, raw	2	75.85	0.0
16A-22	fish/shellfish: mackerel, Pacific and jack, mixed species, raw	7.89	70.15	0.0
16A-22	fish/shellfish: mackerel, Spanish, raw	6.3	71.67	0.0
16A-53	fish/shellfish: milkfish, raw	6.73	70.85	0.0
16A-99	fish/shellfish: monkfish, raw	1.52	83.24	0.0
16A-24	fish/shellfish: ocean perch, Atlantic, raw	1.63	78.7	0.0
16A-25	fish/shellfish: pike, northern, raw	0.69	78.92	0.0
16A-25	fish/shellfish: pike, walleye, raw	1.22	79.31	0.0
16A-28	fish/shellfish: pollock, Atlantic, raw	0.98	78.18	0.0
16A-28	fish/shellfish: pollock, walleye, raw	0.8	81.56	0.0
16A-29	fish/shellfish: pompano, Florida, raw	9.47	71.12	0.0
16A-99	fish/shellfish: pout, ocean, raw	0.91	81.36	0.0
16A-24	fish/shellfish: rockfish, Pacific, mixed species, raw	1.57	79.26	0.0
16A-50	fish/shellfish: roughy, orange, raw	7	75.9	0.0
16A-31	fish/shellfish: sablefish, raw	15.3	71.02	0.0
16A-32	fish/shellfish: salmon, Atlantic, raw	6.34	68.5	0.0
16A-32	fish/shellfish: salmon, chinook, raw	10.44	73.17	0.0
16A-32	fish/shellfish: salmon, chum, raw	3.77	75.38	0.0
16A-32	fish/shellfish: salmon, coho, raw	5.95	72.63	0.0
16A-32	fish/shellfish: salmon, pink, raw	3.45	76.35	0.0
16A-32	fish/shellfish: salmon, sockeye, raw	8.56	70.24	0.0
16A-99	fish/shellfish: scup, raw	2.73	75.37	0.0
16A-04	fish/shellfish: sea bass, mixed species, raw	2	78.27	0.0
16A-47	fish/shellfish: seatrout, mixed species, raw	3.61	78.09	0.0
16A-21	fish/shellfish: shad, American, raw	13.77	68.19	0.0
16A-35	fish/shellfish: shark, mixed species, raw	4.51	73.58	0.0
16A-99	fish/shellfish: sheepshead, raw	2.41	77.97	0.0
16A-36	fish/shellfish: smelt, rainbow, raw	2.42	78.77	0.0
16A-99	fish/shellfish: snapper, mixed species, raw	1.34	76.87	0.0
16A-39	fish/shellfish: spot, raw	4.9	75.95	0.0
16A-40	fish/shellfish: sturgeon, mixed species, raw	4.04	76.55	0.0
16A-41	fish/shellfish: sucker, white, raw	2.32	79.71	0.0
16A-99	fish/shellfish: sunfish, pumpkinseed, raw	0.7	79.5	0.0
16A-42	fish/shellfish: swordfish, raw	4.01	75.62	0.0
16A-99	fish/shellfish: tilefish, raw	2.31	78.9	0.0
16A-44	fish/shellfish: trout, mixed species, raw	6.61	71.42	0.0
16A-44	fish/shellfish: trout, rainbow, raw	3.36	71.48	0.0

Product Code	Commodity	% Fat	% Water	% Sugars
16A-45	fish/shellfish: tuna, fresh, bluefin, raw	4.9	68.09	0.0
16A-45	fish/shellfish: tuna, fresh, skipjack, raw	1.01	70.58	0.0
16A-45	fish/shellfish: tuna, fresh, yellowfin, raw	0.95	70.99	0.0
16A-46	fish/shellfish: turbot, European, raw	2.95	76.95	0.0
16A-48	fish/shellfish: whitefish, mixed species, raw	5.86	72.77	0.0
16A-49	fish/shellfish: whiting, mixed species, raw	1.31	80.27	0.0
16A-99	fish/shellfish: wolffish, Atlantic, raw	2.39	79.9	0.0
16A-99	fish/shellfish: yellowtail, mixed species, raw	5.24	74.52	0.0
16A-02	fish: barracuda, Pacific, raw	2.6	75.4	0.0
E: shellfish				
16E-01	fish/shellfish: abalone, mixed species, raw	0.76	74.56	*
16E-02	fish/shellfish: clam, mixed species, raw	0.97	81.82	*
16E-04	fish/shellfish: mussel, blue, raw	2.24	80.58	*
16E-03	fish/shellfish: oyster, eastern, raw	2.47	85.14	*
16E-03	fish/shellfish: oyster, Pacific, raw	2.3	82.06	*
16E-05	fish/shellfish: scallop, mixed species, raw	0.76	78.57	*
16E-99	fish/shellfish: whelk, unspecified, raw	0.4	66	*
J: crustaceans				
16J-01	fish/shellfish: crab, Alaska king, raw	0.6	79.57	0.0
16J-01	fish/shellfish: crab, blue, raw	1.08	79.02	*
16J-01	fish/shellfish: crab, dungeness, raw	0.97	79.18	*
16J-01	fish/shellfish: crab, queen, raw	1.18	80.58	0.0
16J-02	fish/shellfish: crayfish, mixed species, raw	1.06	80.79	0.0
16J-04	fish/shellfish: lobster, northern, raw	0.9	76.76	*
16J-05	fish/shellfish: shrimp, mixed species, raw	1.73	75.86	*
16J-03	fish/shellfish: spiny lobster, mixed species, raw	1.51	74.07	*
M: other aquatic animals				
16M-07	fish/shellfish: cuttlefish, mixed species, raw	0.7	80.56	*
16M-09	fish/shellfish: octopus, common, raw	1.04	80.25	*
16M-03	fish/shellfish: squid, mixed species, raw	1.38	78.55	*
16M-01	frog legs: raw	0.3	81.9	0.0
16M-05	seafood: terrapin, (diamond back), raw	3.5	77	0.0
16M-05	seafood: turtle, green, raw	0.5	78.5	0.0
16M-06	seafood: whale meat, raw	7.5	70.9	*
R: engineered seafood				
16R-01	fish/shellfish: surimi	0.9	76.34	*
Y: fishery products				
16Y-04	fish/shellfish: roe, mixed species, raw	6.42	67.73	*

17: MEAT AND POULTRY

A: red meat products				
17A-01	frankfurters: raw, beef	28.54	54.71	*

Product Code	Commodity	% Fat	% Water	% Sugars
17A-99	frankfurters: raw, beef & pork	29.15	53.87	2
17A-99	goat: raw	2.31	75.84	*
B: poultry/poultry products				
17B-99	frankfurter: chicken	19.48	57.53	*
17B-06	frankfurter: turkey	17.7	62.99	*
20: FRUITS				
A: berries				
20A-01	blackberries: raw	0.39	85.64	7.9
20A-02	blueberries: raw	0.38	84.61	7.3
20A-04	cranberries: raw	0.2	86.54	*
20A-05	currants: European black, raw	0.41	81.96	8
20A-05	currants: red and white, raw	0.2	83.95	8
20A-07	elderberries: raw	0.5	79.8	*
20A-08	gooseberries: raw	0.58	87.87	*
20A-09	grapes: American type (slip skin), raw	0.35	81.3	16.4
20A-09	grapes: European type (adherent skin), raw	0.58	80.56	18.1
20A-99	groundcherries: (cape-gooseberries or poha), raw	0.7	85.4	*
20A-99	mulberries: raw	0.39	87.68	*
20A-99	oheloberries: raw	0.22	92.3	*
20A-10	raisins: golden seedless	0.46	14.97	*
20A-10	raisins: seeded	0.54	16.57	*
20A-10	raisins: seedless	0.46	15.42	61.7
20A-13	raspberries: raw	0.55	86.57	*
20A-14	strawberries: raw	0.37	91.57	5.7
D: berry juice				
20D-09	grape juice: canned or bottled, unsweetened	0.08	84.12	14.2
G: citrus fruit				
20G-02	grapefruit: raw, pink/red/white, all areas	0.1	90.89	6.2
20G-03	kumquats: raw	0.1	81.7	*
20G-04	lemon peel: raw	0.3	81.6	*
20G-04	lemons: raw, w/peel	0.3	87.4	*
20G-05	limes: raw	0.2	88.26	0.4
20G-06	oranges: raw, all commercial varieties	0.12	86.75	8.9
20G-06	oranges: raw, California, navels	0.09	86.81	*
20G-06	oranges: raw, California, valencias	0.3	86.34	*
20G-06	oranges: raw, Florida	0.21	87.14	8.9
20G-07	pummelo: raw	0.04	89.1	*
20G-09	tangerines: (mandarin orange), raw	0.19	87.6	*
K: citrus fruit juices				
20K-02	grapefruit juice: pink/red/white, all varieties, raw	0.1	90	7.5
20K-04	lemon juice: canned or bottled	0.29	92.46	2.4
20K-04	lemon juice: raw	0	90.73	2.4

Product Code	Commodity	% Fat	% Water	% Sugars
20K-05	lime juice: canned or bottled, unsweetened	0.23	92.52	*
20K-05	lime juice: raw	0.1	90.21	*
20K-06	orange juice: canned, unsweetened	0.14	89.01	9.8
20K-06	orange juice: raw	0.2	88.3	10.2
N: core fruit				
20N-01	apples: raw, w/skin	0.36	83.93	11.5
20N-02	crabapples: raw	0.3	78.94	*
20N-99	mammy-apple: (mamey), raw	0.5	86.2	*
20N-03	pears: raw	0.4	83.81	10.5
20N-05	pricklypears: raw	0.51	87.55	*
20N-04	quinces: raw	0.1	83.8	*
20N-99	rose-apples: raw	0.3	93	*
Q: core fruit, dried/paste				
20Q-01	apples: dehydrated (low moisture), sulfured, uncooked	0.58	3	*
20Q-01	apples: dried, sulfured, uncooked	0.32	31.76	*
20Q-01	applesauce: canned, sweetened, w/salt	0.18	79.58	16.5
S: core fruit juice				
20S-01	apple juice: canned or bottled, unsweetened, w/added asc acid	0.11	87.93	10.9

21: FRUIT PRODUCTS

G: pit fruit				
21G-01	apricots: raw	0.39	86.35	9.3
21G-02	avocados: raw, California	17.33	72.56	0.9
21G-02	avocados: raw, Florida	8.87	79.73	*
21G-03	cherries: sour, red, raw	0.3	86.13	8.1
21G-03	cherries: sweet, raw	0.96	80.76	14.6
21G-05	dates: domestic, natural and dry	0.45	22.5	64.2
21G-99	java-plum: (jambolan), raw	0.23	83.13	*
21G-16	jujube: raw	0.2	77.86	*
21G-08	loquats: raw	0.2	86.73	*
21G-07	nectarines: raw	0.46	86.28	8.5
21G-13	peaches: raw	0.09	87.66	8.7
21G-12	pitanga: (surinam-cherry), raw	0.4	90.81	*
21G-14	plums: raw	0.62	85.2	7.5
21G-99	sapodilla: raw	1.1	78	*
H: pit fruit dried/paste				
21H-01	apricots: dehydrated (low-moisture), sulfured, uncooked	0.62	7.5	*
21H-01	apricots: dried, sulfured, uncooked	0.46	31.09	38.9
K: pit fruit juice				
21K-01	apricot nectar: canned, w/added asc acid	0.09	84.87	13.5

Product Code	Commodity	% Fat	% Water	% Sugars
S: sub/tropical fruit				
21S-99	balsam-pear: leafy tips, raw	0.69	89.25	0.8
21S-99	balsam-pear: pods, raw	0.17	94.03	0.8
21S-02	bananas: raw	0.48	74.26	18.4
21S-02	bananas: red, raw	0.2	74.4	*
21S-20	carambola: (starfruit), raw	0.35	90.92	7.1
21S-99	cherimoya: raw	0.4	73.5	*
21S-99	custard-apple: (bullock's-heart), raw	0.6	71.5	*
21S-03	figs: raw	0.3	79.11	6.9
21S-04	guavas: common, raw	0.6	86.1	6
21S-04	guavas: strawberry, raw	0.6	80.66	*
21S-10	jackfruit: raw	0.3	73.23	18.4
21S-11	kiwifruit: (Chinese gooseberries), fresh, raw	0.44	83.05	8.9
21S-19	litchis: raw	0.44	81.76	*
21S-18	longans: raw	0.1	82.75	*
21S-05	mangoes: raw	0.27	81.71	14.8
21S-06	papayas: raw	0.14	88.83	5.9
21S-12	passion-fruit: (granadilla), purple, raw	0.7	72.93	11.2
21S-07	pineapple: raw	0.43	86.5	11.9
21S-08	plantain: raw	0.37	65.28	*
21S-16	sapotes: (marmalade plum), raw	0.6	62.43	*
21S-09	seeds: breadfruit seeds, boiled	2.3	59.3	*
21S-09	seeds: breadfruit seeds, raw	5.59	56.27	*
21S-14	tamarinds: raw	0.6	31.4	*
T: sub/tropical fruit dried/paste				
21T-19	litchis: dried	1.2	22.3	*
21T-18	longans: dried	0.4	17.6	*
V: sub/tropical fruit juice/milk,creme/nect				
21V-01	acerola juice: raw	0.3	94.3	*

22: VINE FRUITS (MELONS) AND PRODUCTS

A: vine fruit				
22A-01	melons: cantaloupe, raw	0.28	89.78	8.1
22A-02	melons: casaba, raw	0.1	92	*
22A-03	melons: honeydew, raw	0.1	89.66	*
22A-04	watermelon: raw	0.43	91.51	9
G: other fruit products				
22G-02	persimmons: Japanese, raw	0.19	80.32	*
22G-02	persimmons: native, raw	0.4	64.4	*
22G-01	pomegranate: raw	0.3	80.97	8.9
22G-99	roselle: raw	0.64	86.58	*
22G-04	soursop: raw	0.3	81.16	*
22G-05	sugar-apples: (sweetsop), raw	0.29	73.23	*

Product Code	Commodity	% Fat	% Water	% Sugars
23: NUTS, EDIBLE SEEDS, AND PRODUCTS				
A: nut in shell				
23A-04	nuts: chestnuts, European, raw, unpeeled	2.26	48.65	10.6
B: nut shelled				
23B-99	nuts: acorns, raw	23.86	27.9	*
23B-04	nuts: chestnuts, Chinese, raw	1.11	43.95	*
23B-04	nuts: chestnuts, European, raw, peeled	1.25	52	11.3
23B-04	nuts: chestnuts, Japanese, raw	0.53	61.41	*
23B-05	nuts: coconut meat, raw	33.49	46.99	3.5
23B-15	nuts: ginkgo nuts, raw	1.68	55.2	*
23B-07	peanuts: all types, raw	49.24	6.5	4.3
23B-07	peanuts: Spanish, raw	49.6	6.39	*
23B-07	peanuts: Valencia, raw	47.58	4.26	*
23B-07	peanuts: Virginia, raw	48.75	6.91	4.3
C: nut butter				
23C-07	nuts: peanut butter, w/salt added	49.98	1.42	*
23C-07	peanut butter: chunk style, w/salt	49.94	1.13	7.8
23C-07	peanut butter: chunk style, wo/salt	49.94	1.13	*
23C-07	peanut butter: smooth style, w/salt	49.98	1.42	7.8
23C-07	peanut butter: smooth style, wo/salt	49.98	1.42	7.8
K: edible seed				
23K-08	millet, raw	4.22	8.67	1.2
23K-09	quinoa	5.8	9.3	*
23K-06	seeds: lotus seeds, raw	0.53	77	*
23K-03	soybeans: green, raw	6.8	67.5	*
24: BEANS, PEAS, CORN, AND FRUITING VEGETABLES				
A: bean/pea/corn				
24A-01	alfalfa seeds: sprouted, raw	0.69	91.14	0.2
24A-99	beans: adzuki, mature seeds, raw	0.53	13.44	*
24A-99	beans: black turtle soup, mature seeds, raw	0.9	11	*
24A-16	beans: black, mature seeds, raw	1.42	11.02	*
24A-04	beans: cranberry (Roman), mature seeds, canned	0.28	77.56	*
24A-99	beans: French, mature seeds, raw	2.02	10.77	*
24A-10	beans: great northern, mature seeds, raw	1.14	10.7	*
24A-99	beans: hyacinth, mature seeds, raw	1.69	9.38	*
24A-07	beans: kidney, all types, mature seeds, raw	0.83	11.75	*
24A-07	beans: kidney, California red, mature seeds, raw	0.25	11.75	*
24A-07	beans: kidney, red, mature seeds, raw	1.06	11.75	*
24A-07	beans: kidney, royal red, mature seeds, raw	0.45	11.9	*
24A-08	beans: lima, large, mature seeds, raw	0.69	10.17	8.5
24A-09	beans: mung, mature seeds, raw	1.15	9.05	6.6
24A-01	beans: mung, mature seeds, sprouted, raw	0.18	90.4	1.7
24A-99	beans: mungo, mature seeds, raw	1.83	8.58	*

Product Code	Commodity	% Fat	% Water	% Sugars
24A-10	beans: navy, mature seeds, raw	1.28	12.36	*
24A-99	beans: pink, mature seeds, raw	1.13	10.06	*
24A-11	beans: pinto, mature seeds, raw	1.13	10.95	*
24A-99	beans: small white, mature seeds, raw	1.18	11.71	*
24A-14	beans: snap, green var, raw	0.12	90.27	2.6
24A-15	beans: snap, yellow var, raw	0.12	90.27	*
24A-10	beans: white, mature seeds, raw	0.85	11.32	*
24A-99	beans: winged, mature seeds, raw	16.32	8.34	7
24A-14	beans: yardlong, mature seeds, raw	1.31	8.43	*
24A-99	beans: yellow, mature seeds, raw	2.6	11.1	*
24A-05	broadbeans (fava beans): mature seeds, raw	1.53	10.98	5.7
24A-06	chickpeas (garbanzo beans, bengal gram): mature seeds, raw	6.04	11.53	3.8
24A-60	corn, white	4.74	10.37	*
24A-60	corn, yellow	4.74	10.37	*
24A-60	corn: sweet, white, raw	1.18	75.96	*
24A-60	corn: sweet, yellow, raw	1.18	75.96	5.4
24A-99	cowpeas: catjang, mature seeds, raw	2.07	11.05	3.0
24A-99	hyacinth-beans: immature seeds, raw	0.2	87.87	*
24A-70	lentils: mature seeds, raw	0.96	11.19	2.5
24A-99	lupins: mature seeds, raw	9.74	10.44	*
24A-99	mothbeans: mature seeds, raw	1.61	9.68	*
24A-99	natto	11	55.02	*
24A-51	peas: edible-podded, raw	0.2	88.89	4
24A-51	peas: green, raw	0.4	78.86	4.5
24A-99	peas: split, mature seeds, raw	1.16	11.27	*
24A-17	pigeon peas (red gram): mature seeds, raw	1.49	10.59	2.8
24A-13	soybeans: mature seeds, raw	19.94	8.54	6.6
24A-99	tempeh	7.68	54.95	1.3
24A-13	tofu: raw, firm	8.72	69.83	0.4
24A-13	tofu: raw, regular	4.78	84.55	0.4
24A-99	winged bean: leaves, raw	1.1	76.85	*
24A-99	winged bean: tuber, raw	0.9	57.4	*
24A-14	yardlong bean: raw	0.4	87.85	*
F: fruit (vegetable)				
24F-99	chayote: fruit, raw (pear-shaped veg, squash family)	0.3	93	*
24F-20	cucumber: not pared, raw	0.13	96.05	2.3
24F-01	eggplant: raw	0.1	91.93	3.4
24F-09	gourd: dishcloth (towelgourd), raw	0.2	93.85	*
24F-09	gourd: white-flowered (calabash), raw	0.02	95.54	*
24F-02	okra: raw	0.1	89.58	2.4
24F-08	peppers: hot chili, green, raw	0.2	87.74	*
24F-08	peppers: hot chili, red, raw	0.2	87.74	*
24F-07	peppers: sweet, green, raw	0.19	92.19	2.5
24F-07	peppers: sweet, red, raw	0.19	92.19	*
24F-05	pumpkin: raw	0.1	91.6	4.4
24F-06	squash: summer, all varieties, raw	0.21	93.68	2.2

Product Code	Commodity	% Fat	% Water	% Sugars
24F-06	squash: summer, crookneck and straightneck, raw	0.24	94.2	2.2
24F-06	squash: summer, scallop, raw	0.2	94.18	2.2
24F-06	squash: summer, zucchini, incl skin, raw	0.14	95.28	2.2
24F-06	squash: winter, acorn, raw	0.1	87.78	2.2
24F-06	squash: winter, all varieties, raw	0.23	88.72	2.2
24F-06	squash: winter, butternut, raw	0.1	86.41	2.2
24F-06	squash: winter, hubbard, raw	0.5	88	2.2
24F-06	squash: winter, spaghetti, raw	0.57	91.6	2.2
24F-50	tomatoes: green, raw	0.2	93	2.7
24F-50	tomatoes: red, ripe, raw, yr round average	0.33	93.76	3.0
24F-09	waxgourd: (Chinese preserving melon), raw	0.2	96.1	1
T: leaf/stem vegetable				
24T-99	amaranth	6.51	9.84	1.9
24T-01	artichokes: (globe or French), raw	0.15	84.94	2.2
24T-02	asparagus: raw	0.22	92.25	2.1
24T-03	bamboo shoots: raw	0.3	91	*
24T-04	beet greens: raw	0.06	92.15	*
24T-99	borage: raw	0.7	93	0.9
24T-05	broccoli: raw	0.35	90.69	1.6
24T-07	brussels sprouts: raw	0.3	86	2.2
24T-99	butterbur: (fuki), raw	0.04	94.5	*
24T-33	cabbage: Chinese (pak-choi), raw	0.2	95.32	1
24T-12	cabbage: Chinese (pe-tsai), raw	0.2	94.39	1.3
24T-08	cabbage: common (Danish, domestic, and pointed types), raw	0.18	92.52	2.7
24T-08	cabbage: red, raw	0.26	91.55	5.4
24T-08	cabbage: savoy, raw	0.1	91	2.9
24T-99	cardoon: raw	0.1	94	1.7
24T-10	cauliflower: raw	0.18	92.26	2.2
24T-44	celeriac: raw	0.3	88	2
24T-11	celery: raw	0.14	94.64	1
24T-99	celtuce: raw	0.3	94.5	1.7
24T-26	chard: Swiss, raw	0.2	92.66	1.1
24T-34	chicory: greens, raw	0.3	92	0.9
24T-34	chicory: witloof, raw	0.1	95.1	*
24T-13	collards: raw	0.22	90.55	*
24T-35	cornsalad: raw	0.4	92.8	*
24T-99	cowpeas: leafy tips, raw	0.25	89.78	*
24T-99	cress: garden, raw	0.7	89.4	*
24T-14	dandelion greens: raw	0.7	85.6	2.4
24T-99	dock: raw	0.7	93	*
24T-30	endive: raw	0.2	93.79	1.2
24T-99	horseradish-tree: leafy tips, raw	1.4	78.66	*
24T-99	jute, potherb: raw	0.25	87.72	*
24T-18	kale, Scotch: raw	0.6	87	2.2
24T-18	kale: raw	0.7	84.46	2.2
24T-99	lambsquarters: raw	0.8	84.3	*

Product Code	Commodity	% Fat	% Water	% Sugars
24T-31	lettuce: butterhead (includes Boston and bibb types), raw	0.22	95.58	*
24T-32	lettuce: cos or romaine, raw	0.2	94.91	2
24T-31	lettuce: iceberg (includes crisphead types), raw	0.19	95.89	1.8
24T-32	lettuce: looseleaf, raw	0.3	94	*
24T-20	mustard greens: raw	0.2	90.8	0.8
24T-99	mustard spinach: (tendergreen), raw	0.3	92.2	*
24T-25	New Zealand spinach: raw	0.2	94	*
24T-21	parsley: raw	0.3	88.31	1.1
24T-43	pokeberry shoots: (poke), raw	0.4	91.6	*
24T-99	purslane: raw	0.1	93.92	*
24T-24	rhubarb: raw	0.2	93.61	0.9
24T-29	seaweed: agar, raw	0.03	91.32	*
24T-29	seaweed: dulse, raw	3.2	16.6	*
24T-29	seaweed: irishmoss, raw	0.16	81.34	*
24T-29	seaweed: kelp, raw	0.56	81.58	*
24T-29	seaweed: laver, raw	0.28	85.03	*
24T-29	seaweed: spirulina, raw	0.39	90.67	*
24T-29	seaweed: wakame, raw	0.64	79.99	*
24T-99	sesbania flower: raw	0.04	91.58	*
24T-21	spices: parsley, dried	4.431	9.02	*
24T-25	spinach: raw	0.35	91.58	0.4
24T-42	swamp cabbage: (skunk cabbage), raw	0.2	92.47	*
24T-27	turnip greens: raw	0.3	91.07	1
24T-99	vinespinach: (basella), raw	0.3	93.1	*
24T-28	watercress: raw	0.1	95.11	0.4
25: VEGETABLES AND PRODUCTS				
J: root/tuber vegetable				
25J-28	arrowhead: raw	0.29	72.48	*
25J-08	beets: raw	0.14	87.32	5.9
25J-22	burdock root: raw	0.15	80.09	*
25J-01	carrots: raw	0.19	87.79	6.6
25J-16	cassava: raw	0.39	68.51	1.2
25J-20	chicory: roots, raw	0.2	80	2.4
25J-21	garlic: raw	0.5	58.58	1
25J-24	ginger root: raw	0.73	81.67	*
25J-02	horseradish-tree: pods, raw	0.2	88.2	*
25J-02	horseradish: raw	0.3	74.6	1.8
25J-29	Jerusalem-artichokes: raw	0.01	78.01	2.5
25J-99	kohlrabi: (thickened bulb-like stems): raw	0.1	91	4.5
25J-03	leeks: (bulb and lower leaf-portion), raw	0.3	83	3.9
25J-18	lotus root: raw	0.1	79.1	*
25J-12	mountain yam: Hawaii, raw	0.1	81.44	*
25J-25	onions: raw	0.16	89.68	4.1
25J-04	onions: spring (includes tops and bulb), raw	0.19	89.83	3.2
25J-25	onions: Welsh, raw	0.4	90.5	*
25J-05	parsnips: raw	0.3	79.53	4.8

Product Code	Commodity	% Fat	% Water	% Sugars
25J-06	potatoes: raw, flesh	0.1	78.96	1.0
25J-06	potatoes: raw, skin	0.1	83.29	0.6
25J-26	radishes: oriental, raw	0.1	94.62	2.5
25J-07	radishes: raw	0.54	94.84	2.7
25J-07	radishes: white icicle, raw	0.1	95.37	2.5
25J-09	rutabagas: raw	0.2	89.66	5.6
25J-10	salsify: (vegetable oyster), raw	0.2	77	2.9
25J-11	shallots, raw	0.1	79.8	3.2
25J-12	sweet potato leaves: raw	0.3	87.96	*
25J-12	sweet potatoes: raw	0.3	72.84	5.0
25J-23	taro, Tahitian: raw	0.97	87.96	*
25J-23	taro: leaves, raw	0.74	85.66	*
25J-23	taro: raw	0.2	70.64	0.8
25J-23	taro: shoots, raw	0.09	95.82	*
25J-14	turnips: raw	0.1	91.87	3.8
25J-15	water chestnuts: Chinese, (matai), raw	0.1	73.46	4.8
25J-12	yam: raw	0.17	69.6	0.5
25J-17	yambean: raw	0.2	89.15	*
L: root/tuber vegetable dried/paste/spread/f				
25L-23	poi	0.14	71.64	*
P: fungi, mushrooms, truffles; whole (button				
25P-01	mushrooms: raw	0.42	91.81	1.8
25P-04	mushrooms: shiitake, dried	0.99	9.5	*
S: fungi, mushroom, truffle products, not el				
25S-99	jew's ear: (pepeao), raw	0.04	92.59	*

28: SPICES, FLAVORS, AND SALTS

A: whole spices				
28A-99	chervil: raw	0.9	80.7	*
28A-99	chives: raw	0.6	92	1
28A-15	coriander: raw	0.59	92.8	*
28A-18	fennel: leaves, raw	0.4	90	*
28A-03	spices: anise seed	15.901	9.535	*
28A-09	spices: caraway seed	14.593	9.875	*
28A-12	spices: celery seed	25.271	6.037	*
28A-15	spices: coriander seed	17.77	8.861	*
28A-16	spices: cumin seed	22.27	8.063	*
28A-17	spices: dill seed	14.535	7.701	*
28A-18	spices: fennel seed	14.868	8.813	*
28A-56	spices: fenugreek seed	6.408	8.843	*
28A-29	spices: mustard seed, yellow	28.759	6.858	*
28A-37	spices: poppy seed	44.704	6.782	*
28A-40	spices: saffron	5.852	11.898	*

Product Code	Commodity	% Fat	% Water	% Sugars
B: ground/cracked spices				
28B-43	seeds: sesame butter, tahini, from raw and stone ground kernels	48	3	*
28B-01	spices: allspice, ground	8.685	8.459	*
28B-04	spices: basil, ground	3.977	6.432	*
28B-05	spices: bay leaf, crumbled	8.362	5.436	*
28B-10	spices: cardamom, ground	6.699	8.282	*
28B-99	spices: chervil, dried	3.9	7.2	*
28B-08	spices: chili powder	16.76	7.79	*
28B-13	spices: cinnamon, ground	3.185	9.52	*
28B-14	spices: cloves, ground	20.066	6.857	*
28B-15	spices: coriander leaf, dried	4.778	7.3	*
28B-17	spices: dill weed, dried	4.36	7.3	*
28B-11	spices: garlic powder	0.759	6.446	*
28B-19	spices: ginger, ground	5.949	9.377	*
28B-24	spices: mace, ground	32.382	8.172	*
28B-27	spices: marjoram, dried	7.036	7.641	*
28B-30	spices: nutmeg, ground	36.307	6.228	*
28B-31	spices: onion powder	1.052	5.005	*
28B-27	spices: oregano, ground	10.25	7.164	*
28B-33	spices: paprika	12.953	9.536	*
28B-54	spices: pepper, black	3.26	10.508	*
28B-08	spices: pepper, red or cayenne	15.668	8.047	*
28B-55	spices: pepper, white	2.12	11.42	*
28B-53	spices: rosemary, dried	15.22	9.306	*
28B-41	spices: sage, ground	12.745	7.955	*
28B-42	spices: savory, ground	5.907	9.003	*
28B-47	spices: tarragon, ground	7.242	7.739	*
28B-48	spices: thyme, ground	7.425	7.79	*
28B-49	spices: turmeric, ground	9.876	11.356	*
F: ground/cracked mix spice/season w/o salt				
28F-04	spices: curry powder	13.81	9.52	*
36: SWEETENERS (NUTRITIVE)				
C: honey				
36C-02	honey: strained or extracted	0	17.2	81.9
38: SOUPS				
A: soup				
38A-33	miso	6.07	41.45	*
40: BABY FOODS				
A: baked goods (baby)				
40A-01	babyfood: cookies, arrowroot	14.3	5.6	23
40A-02	babyfood: teething biscuits	4.2	6.4	24
40A-02	zwieback	9.7	4.5	12.9

Product Code	Commodity	% Fat	% Water	% Sugars
B: cereal (baby)				
40B-01	babyfood: cereal, barley, dry	3.4	6.8	9.79
40B-30	babyfood: cereal, egg yolks and bacon, str	5.2	84.9	*
40B-03	babyfood: cereal, hi-prot, dry	5.9	6.1	3.89
40B-10	babyfood: cereal, mixed, dry	4.4	6.7	7.75
40B-20	babyfood: cereal, mixed, w/applesauce & bananas, junior	0.4	79.6	11
40B-20	babyfood: cereal, mixed, w/applesauce & bananas, strained	0.5	80	11
40B-20	babyfood: cereal, mixed, w/bananas, dry	4.6	4.5	20.9
40B-04	babyfood: cereal, oatmeal, dry	7.8	6.2	9.79
40B-20	babyfood: cereal, oatmeal, w/applesauce & bananas, junior	0.7	81.8	10.4
40B-20	babyfood: cereal, oatmeal, w/applesauce & bananas, strained	0.7	82.2	10.4
40B-20	babyfood: cereal, oatmeal, w/bananas, dry	6	4.7	22.4
40B-05	babyfood: cereal, rice, dry	4.9	6.7	9.75
40B-20	babyfood: cereal, rice, w/applesauce & bananas, strained	0.4	81	8.91
40B-20	babyfood: cereal, rice, w/bananas, dry	4.2	4.7	19.4
40B-30	babyfood: cereal, w/egg yolks, junior	1.8	88.7	0.68
40B-30	babyfood: cereal, w/egg yolks, strained	1.8	88.8	0.68
D: vegetables (baby)				
40D-06	babyfood: vegetables, carrots, jnr	0.2	91	3.07
40D-06	babyfood: vegetables, carrots, str	0.1	92.3	3.07
40D-02	babyfood: vegetables, corn, creamed, junior	0.4	81.4	1.18
40D-02	babyfood: vegetables, corn, creamed, strained	0.4	83.6	1.18
40D-01	babyfood: vegetables, green beans, buttered, jnr	0.9	91.2	*
40D-01	babyfood: vegetables, green beans, buttered, str	0.8	90.8	*
40D-01	babyfood: vegetables, green beans, jnr	0.1	92.5	*
40D-05	babyfood: vegetables, mix veg, jnr	0.4	89.4	1.36
40D-05	babyfood: vegetables, mix veg, str	0.5	89.8	1.36
40D-01	babyfood: vegetables, peas, creamed, strained	1.9	86.5	1.52
40D-04	babyfood: vegetables, spinach, creamed, jnr	1.4	88.2	1.94
40D-04	babyfood: vegetables, spinach, creamed, str	1.3	89.6	1.94
40D-03	babyfood: vegetables, squash, jnr	0.2	92.8	2.18
40D-03	babyfood: vegetables, squash, str	0.2	92.7	2.18
E: fruit/juice/drink (baby)				
40E-20	babyfood: fruit, apple & blueberry, junior	0.2	82.8	8.9
40E-20	babyfood: fruit, apple & blueberry, strained	0.2	83.1	8.9
40E-20	babyfood: fruit, applesauce & apricots, jnr	0.2	86.9	10.6
40E-20	babyfood: fruit, applesauce & apricots, str	0.2	87.7	10.6
40E-20	babyfood: fruit, applesauce & pineapple, junior	0.1	89.1	11.8
40E-20	babyfood: fruit, applesauce & pineapple, strained	0.1	89.5	11.8
40E-20	babyfood: fruit, pears & pineapple, jnr	0.2	87.8	8.58
40E-20	babyfood: fruit, pears & pineapple, str	0.1	88.5	8.58
40E-10	babyfood: fruit, pears, jnr	0.1	87.8	10.6
40E-10	babyfood: fruit, pears, str	0.2	88.4	10.6
40E-03	babyfood: juice, apple	0.1	88	14
40E-30	babyfood: juice, apple & grape	0.2	88.1	13.3

Product Code	Commodity	% Fat	% Water	% Sugars
40E-30	babyfood: juice, apple & peach	0.1	89	11.4
40E-30	babyfood: juice, apple & prune	0.1	81.3	13.1
40E-30	babyfood: juice, mixed fruit	0.1	87.9	*
40E-02	babyfood: juice, orange	0.3	88.5	*
40E-30	babyfood: juice, orange & apple	0.2	88.9	*
40E-30	babyfood: juice, orange & apple & banana	0.1	87.6	12.3
40E-30	babyfood: juice, orange & pineapple	0.1	87.3	12
F: meat products/comb meat dinner (baby)				
40F-51	babyfood: dinner, beef noodle, jnr	1.9	87.8	*
40F-50	babyfood: dinner, macaroni & tomato & beef, jnr	1.1	86.7	1.44
40F-50	babyfood: dinner, macaroni & tomato & beef, str	1.1	87.3	1.44
40F-53	babyfood: dinner, spaghetti & tomato & meat, jnr	1.3	85.5	1.21
40F-53	babyfood: dinner, spaghetti & tomato & meat, toddler	1	81.6	*
40F-55	babyfood: dinner, veg & bacon, jnr	3.9	86.2	1.71
40F-55	babyfood: dinner, veg & bacon, str	3.3	85.9	1.71
40F-57	babyfood: dinner, veg & ham, jnr	1.7	88.4	1.26
40F-57	babyfood: dinner, veg & ham, str	1.7	89.2	1.26
40F-57	babyfood: dinner, veg & ham, toddler	3	83.6	1.26
40F-58	babyfood: dinner, veg & lamb, jnr	1.7	88.6	1.08
40F-58	babyfood: dinner, veg & lamb, str	2	88.6	1.08
40F-59	babyfood: dinner, veg & liver, jnr	0.6	88.9	1.03
40F-59	babyfood: dinner, veg & liver, str	0.4	90	1.03
40F-01	babyfood: hi-mt dinner, beef & all veg, str	4.2	85.4	0.71
40F-01	babyfood: hi-mt dinner, beef & veg, jnr	4.6	83.2	0.71
40F-01	babyfood: meat, beef with beef heart, strained	4.4	82.5	*
40F-01	babyfood: meat, beef, junior	4.9	79.9	*
40F-01	babyfood: meat, beef, strained	5.4	80.6	*
40F-99	babyfood: meat, ham, jnr	6.7	78.5	*
40F-99	babyfood: meat, ham, str	5.8	79.4	*
40F-02	babyfood: meat, lamb, junior	5.2	79.6	*
40F-02	babyfood: meat, lamb, strained	4.7	80.3	*
40F-03	babyfood: meat, liver, strained	3.8	79.3	*
40F-54	babyfood: meat, meat sticks, junior	14.6	69.5	*
40F-99	babyfood: meat, pork, strained	7.1	78.4	*
40F-04	babyfood: meat, veal, junior	5	79.8	*
40F-04	babyfood: meat, veal, strained	4.8	80.9	*
G: poultry product/comb poultry dinner (baby)				
40G-40	babyfood: dinner, veg & chicken, jnr	1.1	88.2	1.17
40G-40	babyfood: dinner, veg & chicken, str	1.1	90	1.17
40G-40	babyfood: hi-mt dinner, chicken & veg, jnr	5.5	82.7	0.53
40G-40	babyfood: hi-mt dinner, chicken & veg, str	3.6	83.7	*
40G-50	babyfood: hi-mt dinner, turkey & veg, jnr	5	82.5	0.71
40G-50	babyfood: hi-mt dinner, turkey & veg, str	4.8	83	*
40G-20	babyfood: meat, chicken sticks, junior	14.4	68.3	*
40G-01	babyfood: meat, chicken, jnr	9.6	76	*

Product Code	Commodity	% Fat	% Water	% Sugars
40G-01	babyfood: meat, chicken, str	7.9	77.5	*
40G-20	babyfood: meat, turkey sticks, junior	14.2	69.8	*
40G-02	babyfood: meat, turkey, junior	7.1	77.5	*
40G-02	babyfood: meat, turkey, strained	5.8	78.9	*
H: high meat dinner/cheese food (baby)				
40H-30	babyfood: dinner, macaroni & cheese, junior	2	86.5	1.2
40H-30	babyfood: dinner, macaroni & cheese, strained	2.1	87.1	1.2
40H-30	babyfood: hi-mt dinner, cottage cheese w/pineapple, strained	2.2	72	*
J: egg products (baby)				
40J-01	babyfood: egg yolks, strained	17.3	70.6	*
K: pudding/custard (baby)				
40K-99	babyfood: dessert, caramel pudding, junior	0.9	80.4	*
40K-99	babyfood: dessert, cherry vanilla pudding, junior	0.2	81	10.6
40K-09	babyfood: dessert, custard pudding, vanilla, jnr	2.3	79.4	*
40K-99	babyfood: fruit, apricot w/tapioca, jnr	0	82.1	12.6
40K-99	babyfood: fruit, apricot w/tapioca, str	0	83.1	12.6
40K-99	babyfood: fruit, bananas & pineapple with tapioca, jnr	0.1	81.1	6.67
40K-99	babyfood: fruit, bananas & pineapple with tapioca, str	0	81.7	6.67
40K-99	babyfood: fruit, bananas with tapioca, jnr	0.2	81.5	11.6
40K-99	babyfood: fruit, bananas with tapioca, strained	0.1	84	11.6
L: soups/soup mix (baby)				
40L-02	babyfood: dinner, chicken soup, strained	1.7	89.1	0.67
Y: baby food, not elsewhere classified				
40Y-99	babyfood: dessert, dutch apple, junior	1	82.1	12.3
40Y-99	babyfood: dessert, dutch apple, strained	0.9	82.2	12.3
40Y-99	babyfood: dessert, peach cobbler, junior	0	81.2	14.1
40Y-99	babyfood: dessert, peach cobbler, strained	0	81.8	14.1

202: BASIC ANALYTICAL TECHNIQUES

202 A: INTRODUCTION

Most residue analytical methods follow the same patterns and use the same techniques. This section provides step-by-step recommended operating procedures for certain commonly used analytical techniques that appear repeatedly in the methods described in Chapters 3 and 4 of this manual. These procedure descriptions are not aimed at experienced analysts but may be useful to the relatively inexperienced. Justifications for certain steps are provided to offer guidance to the analyst troubleshooting an operation that did not perform as expected.

The techniques described in this section will not be described in detail elsewhere in PAM I. Where these techniques are used in the methods of Chapters 3 and 4, only the information particular to the method will be prescribed. Where alternative techniques appear in this section, the one most appropriate to a particular method is described in Chapters 3 and 4; the analyst is responsible for validating its replacement with an alternative.

202 B: COLUMN CHROMATOGRAPHY

Column chromatography is used to "clean up" extracts, *i.e.*, to remove extraneous materials that were co-extracted from the commodity with the residues. Ideally, co-extractives such as fat and chlorophyll are more strongly retained by the column adsorbent than are the residues. If so, solvent can be passed through the column, dissolving and removing ("eluting") residues, while leaving co-extractives attached to the adsorbent. Sequential elution of the column with different mixtures of solvents may also be used to separate groups of residues from one another.

The ability of a particular column chromatographic system to remove co-extractives and separate residues is determined empirically during method development. Successful duplication of results by other analysts requires adherence to both recommended operating procedures and specific directions in the method. This section provides terminology, recommended operating procedures, and suggestions for dealing with common problems encountered in the use of column chromatography.

Terminology

adsorbent: a finely sieved solid material, usually of prescribed mesh size, to which dissolved substances will preferentially attach and thus be removed from solutions.

column: either an empty glass tube intended to hold an adsorbent or that tube filled with adsorbent. Residue analytical methods most often require columns with fritted glass discs at the bottom, to hold the adsorbent in place, and Teflon stopcocks to control the rate at which eluant passes through the adsorbent; column length and diameter vary. Columns without stopcocks are suitable only for drying extracts through anhydrous sodium sulfate. If a column has no fritted glass disc, a plug of glass wool is placed in the bottom to retain the adsorbent. Solvent reservoirs incorporated at the top of columns are an optional convenience.

eluant: solvent or mixture of solvents that is passed through the column to remove ("elute") adsorbed residues; also known as eluting solvent.

eluate: solvent or mixture of solvents that has passed through the column. The eluate is the cleaned up extract.

Recommended Operating Procedure

- If adsorbent is stored in oven, remove and allow to reach room temperature in desiccator before use.
- Rinse empty glass column with suitable solvent to remove contaminants.
- Depending on method, either weigh appropriate amount of adsorbent and pour it into empty glass column or pour enough adsorbent into column to reach prescribed height.
- Gently tap side of column to settle adsorbent. Most laboratories make use of empty cardboard tube, length of rubber hose, or some other homemade device for this purpose.
- If directions specify layer of anhydrous sodium sulfate or glass wool plug, add on top of adsorbent layer. Gently tap again.
- Open stopcock completely and pour prescribed volume of rinse solvent through adsorbent; tap again gently during rinsing. Close stopcock when solvent level is still slightly above adsorbent. Discard rinse solvent.
- Place receiving vessel prescribed by method under column tip. Open stopcock part way and immediately transfer sample extract to top of column. As solvent drips from bottom, rinse vessel from which extract was poured with several small volumes of same solvent in which extract is dissolved; add rinses to column.
- As last rinse approaches top of adsorbent layer, add prescribed volume of eluant to column. Adjust stopcock to create prescribed flow rate of eluant through column. Allow elution to proceed; do not use pressure or vacuum to speed elution unless directed by method.
- If method requires more than one eluant, close stopcock when first eluant is still somewhat above top of adsorbent layer. Change receiving vessel, open stopcock part way, and immediately add prescribed amount of next eluant to top. Adjust flow rate as before, and repeat as needed.

Common Problems

Variation in Adsorptivity. Column chromatography is useful for cleanup because of the adsorbent's capacity to retain materials; this "adsorptivity" is dependent on both physical and chemical characteristics. Adsorptivity of a material may vary from one batch to another, with variations affecting both adequacy of cleanup and recovery of residues. To minimize problems caused by variations in adsorptivity, follow these rules:

- Always purchase exact material specified by method. Although relatively few materials are commonly used as adsorbents, many brands and manufacturing treatments are available. Methods are developed using specific adsorbents, and substitutes should not be used.

- Carefully follow any adsorbent handling and storage procedures specified to recreate conditions used during method development.

- If method requires heating adsorbent, always cool in desiccator before use to prevent absorption of moisture from air, which will change adsorptivity.

- If available, use calibration steps provided in method to measure, verify, and/or compensate for variations in adsorptivity.

Physical Disruption of Adsorbent Column. Because adsorption occurs while dissolved materials are in contact with the surface of the adsorbent, cleanup and separations may be adversely affected when physical disruption of the column occurs. Improper handling can cause "pockets" or "channels" to develop in the adsorbent column; if these exist, solutions will flow preferentially through those spaces rather than spread evenly throughout the adsorbent, thus decreasing solute exposure to the adsorbent surface area. Reduction of cleanup capacity and change in elution patterns will likely occur.

To minimize problems caused by channeling, follow the recommended operating procedure, especially:

- Cool adsorbent before use in column; solvents may boil in presence of hot adsorbent and leave pockets within column.

- Tap adsorbent to settle it properly.

- Do not let column go dry; always close stopcock when eluant level is still somewhat above top of adsorbent layer.

Contribution of Interferences. Any material used in residue analysis is a potential source of interference during the determinative steps of the analysis. Routine performance of "reagent blanks," *i.e.*, complete method with no sample present, will notify the analyst if adsorbents or other reagents used in the method are contributing contaminants that interfere. Once detected, the source of contamination must be eliminated. To minimize interferences from adsorbents:

- Always rinse adsorbent as directed and discard rinsings.

- Follow any method directions concerning prewashing adsorbent.

- Store adsorbent in manner that prevents contamination; *e.g.*, if stored without stopper, at least keep vessel covered with loose layer of aluminum foil.

202 C: SOLVENT EVAPORATION

Essentially all residue analytical methods require removal of solvent at some point in order to increase the concentration of the analyte(s) in solution. Several different concentration/evaporation techniques are available, each with advantages and disadvantages. The best technique in a particular situation depends on the physical and chemical characteristics of the analyte and the solvent that must be evaporated.

Each method in Chapters 3 and 4 directs use of a specific concentration/evaporation technique. The method developer chose the particular technique for its applicability to the analyte and solvent involved, so attempts by the user to substitute a different technique will be successful only if the substitute is also applicable. This section provides information on applicability of several techniques, directions for correct operation, and cautions about common problems.

One evaporation technique not described in this section is evaporation in an open vessel, with or without gas flow impinging upon the solvent. Evaporating solvent in this way is not recommended for quantitative residue analysis, because loss of residues is likely [1], opportunities for contamination are increased, and safety and hazardous waste concerns are increased compared to other techniques.

Kuderna-Danish Concentrators

Use of Kuderna-Danish (K-D) concentrators for evaporation of solvents was established in FDA pesticide residue analyses during the 1960s when studies proved its value in reducing large volumes of solvent quickly and without loss of analytes [2]. Most methods in Chapter 3 specify K-D evaporation, because this technique was the most reliable at the time the methods underwent interlaboratory validation.

Application. Evaporation in K-D is most suitable for solvents with relatively low boiling points, such as ethyl ether (b.p. 34.6° C), methylene chloride (40.5°), and acetone (56.5°), or for solvent mixtures that form low boiling azeotropes. Analytes must be able to withstand steam bath temperatures for concentration by K-D.

Equipment. Basic K-D equipment is available from most major suppliers of laboratory equipment; certain sizes may be available only from specialty glassware suppliers.

Complete K-D apparatus consists of three pieces:

1) receiving flask, 10-50 mL volume, ground glass joint. Flasks may be straight sided tubes, most useful for concentration of extracts that will be transferred to other containers, or volumetric or graduated flasks, useful for concentration of final, cleaned up extract. No transfer is necessary if volume of final solution can be reliably measured in receiving flask. Ground glass joint at top of receiving flask must match that at bottom of concentrator.

2) concentrator, available in 125, 250, and 500 mL sizes, with ground glass joints at bottom and top. Concentrator holds most of the solvent during evaporation and is empty at end of procedure.

3) Snyder column, specially designed 30 cm long condensation column containing three balls; permits escape of solvent in gas phase without loss of higher boiling analytes. Micro-Snyder columns, with two balls, and micro-Vigreaux columns, with no balls, are also available for use in evaporating solvent in the receiving flask only, without use of concentrator.

Boiling chips facilitate solvent evaporation by providing a surface area for bubbles to form as boiling starts; use of 20-30 mesh carborundum chips minimizes volume displaced by boiling chip.

Recommended Operating Procedure.

- Connect concentrator and receiving flask and secure with springs or other means of preventing separation.

- Collect solutions from extraction or cleanup steps in connected concentrator/receiving flask.

- Turn on steam in steam bath.

- Add boiling chip to receiving flask, place Snyder column on top of concentrator, and gently lower receiving flask into opening in steam bath. Until boiling is well established, hold in place and tap side of Snyder column to facilitate pressure release. (Excess pressure buildup will result in loss of sample if sudden release occurs.)

- Support concentrator on steam bath. Adjust steam as needed to ensure appropriate evaporation rate. Once started, boiling should cause balls in Snyder column to rattle vigorously as escaping gases move past them. Do not allow boiling to be so vigorous that Snyder column is flooded with liquid.

- When balls in Snyder column stop rattling, remove K-D from steam bath and allow to cool, with Snyder column in place, until all fluid has drained back into receiving flask. Volume can be reduced to ≥5 mL in this way.

- Follow method directions for reconcentration in presence of additional solvent volume or different solvent, as sometimes required for complete removal of particular solvent.

- To reduce volume to <5 mL, remove receiving flask from concentrator and add fresh boiling chip. Connect micro-Snyder column directly to receiving flask. (To avoid flooding two-ball micro-Snyder column during evaporation, use micro-Vigreaux or other micro reflux column without balls for solvents with boiling point >65° C.)

- Hold receiving flask with spring test tube holder, and place tip into steam. Position tip of receiving flask carefully to avoid "bumping" of solvent as it boils. Allow to boil until volume is slightly less than desired.

- Allow apparatus to cool and fluid to drain into receiving flask before removing column. Minimum attainable volume is about 0.2-0.4 mL.

Rotary Evaporator

Vacuum and heat combine to reduce the quantity of solvent in a rotary evaporator. Solution in a round-bottom (r-b) flask is simultaneously rotated and heated by a water bath, while a vacuum pulled from the end of a condenser increases the rate of evaporation. Solvent evaporated from the solution is collected in a second flask attached to condenser.

Application. Evaporation of solvent using rotary evaporation is recommended for heat-labile residues, because temperature of the water bath used is much lower than that of a steam bath. Vacuum withdrawal of vapors, combined with water bath temperature of about 30° C, adequately removes solvents such as methylene chloride (b.p. 40.5° C) without damaging analytes. Evaporation in a rotary evaporator is more rapid than in a K-D, but is limited to only one solution at a time.

Equipment.

1) r-b flasks of appropriate volume. Flask that contains solution being evaporated has ground glass joint; flask that collects condensate has ball-and-socket joint.

2) rotary evaporator, including condenser and variable speed motor that rotates shaft to which r-b flask is attached. Use of glass trap connected to shaft between motor and r-b flask is recommended to protect motor. Electronically controlled motor for constant torque is preferred to maintain constant rotation throughout evaporation. Variable transformer for controlling speed is recommended only for evaporator with AC/DC motor; use of variable transformer on evaporator with AC motor will result in burning out evaporator motor.

3) constant temperature water bath

4) vacuum pump

5) needle valve to control vacuum, positioned between condenser and pump. Controlling vacuum with stopcock-type valve on condenser is also possible but is less precise than needle valve and requires more analyst attention. Use of vacuum gauge (preferred) or manometer is also recommended.

6) (optional) additional traps between condenser and vacuum pump, to protect pump from effects of corrosive vapors. Refrigerated condensation traps and/or chemical traps that collect vapors on disposable cartridges are available.

Recommended Operating Procedure.

- Circulate cold water through condenser.
- Heat water in water bath to temperature specified in method.
- Attach r-b flask for collecting condensate to condenser with clip supplied with rotary evaporator.

- When temperatures have equilibrated, attach r-b flask containing solution to rotary evaporator shaft (or to trap on shaft) and lower r-b flask into water bath. (Directions in some methods specify placing flask into water bath *after* applying vacuum.)

- Turn on motor and adjust rotation to about 70 rpm.

- Turn needle valve to disconnect vacuum pump from apparatus, turn on pump, then apply vacuum to system gradually by adjusting valve to minimize frothing or rapid boiling of solution.

- When solution is evaporated to dryness, reverse these steps to shut down system.

Rotary Evaporator with Circulating Chilled Liquid

Rotary evaporation can be made applicable to higher boiling solvents by addition of a refrigeration unit and pump to chill and circulate coolant through condenser and through bath for collecting condensate.

Application. Rotary evaporation with circulating chilled liquid is specified when solvent with higher boiling points, such as methanol (b.p. 64.7° C), acetonitrile (81.6°), and toluene (110.6°), must be removed from heat-labile analytes. Evaporation is facilitated by simultaneous use of warm water bath, vacuum, and condensation and collection of evaporated solvent at the low temperature provided by circulating refrigerated coolant.

Equipment. Figure 401-a (Section 401) illustrates arrangement of the following basic equipment.

1) r-b flasks, as above

2) rotary evaporator, as above. Condenser should be insulated (*e.g.*, with Styrofoam rings) to maintain low temperature for efficient condensation.

3) constant temperature water bath

4) vacuum pump, with needle valve control and vacuum gauge, as above

3) system capable of chilling coolant such as ethylene glycol (antifreeze) and circulating it through coil of insulated condenser; coolant also circulates through bath in which receiving flask is placed. Several commercial units are available.

5) optional additional traps, as above

Recommended Operating Procedure.

- Ensure that coolant level meets manufacturer's recommendation, then turn on refrigeration and circulating unit; follow manufacturer's directions for equilibration time needed to reach temperature specified in method.

- Follow directions specified for rotary evaporator, above, starting with "Heat water in water bath to temperature specified in method."

Turbo-Vap

This microprocessor-controlled apparatus from Zymark Corp. permits automated evaporation of small volumes of solvent using a patented gas vortex shearing action and mild thermal conditions. Solutions in 200 mL tubes are held in a temperature-controlled water bath while a flow of nitrogen creates a helical flow vortex within the liquid. An exhaust fan moves solvent vapors into a hose for delivery to any suitable hood or other vent. The system can be programmed to shut off when liquid level reaches specified point or after a specified time; this permits solvent evaporation during unattended operation. Up to six samples can be evaporated simultaneously.

No recommended operating procedure for Turbo-Vap is provided in this section, because it is not yet specified by any PAM I method; experiments with the apparatus are included in current method development projects.

Common Problems

Loss of Analyte. Evaporation steps are potential sources of analyte loss. Predictably, losses increase with analyte volatility and with decreasing final solution volume. Any evaporation of solution to dryness may cause analyte loss; even the presence of co-extractives may not prevent losses of the most volatile residues. Studies using open vessel evaporation showed no correlation between amount of co-extractives and losses that occurred during evaporation, and evidence suggested that the nature of co-extractives is more important than the amount [1]. More recent studies showed higher losses of α-BHC, a volatile residue, when evaporating extracts to dryness twice in a Turbo-Vap than when concentrating (never <2 mL) in a K-D [3]. Purified extracts (free of plant extractives and fat) are evaporated to dryness only when absolutely necessary, *e.g.*, when all traces of a solvent like methylene chloride must be removed to prevent interference in a determinative step.

References

[1] Chiba, M., and Morley, H.V. (1968) *J. Assoc. Off. Anal. Chem.* **51**, 55-62

[2] Burke, J.A., *et al.* (1966) *J. Assoc. Off. Anal. Chem.* **49**, 999-1003

[3] Parfitt, C.H., Jr. (Nov. 1991) "Miniaturized Multiresidue Approach to Determine Pesticide Residues in Fresh Fruits and Vegetables," LIB 3616, FDA, Rockville, MD

203: EQUIPMENT AND PROCEDURES FOR COMMINUTING SAMPLES

Section 102 C describes regulatory requirements related to compositing and comminuting test samples so that the test portion removed for analysis can be considered representative of the original consignment. This section provides descriptions of equipment and procedures that FDA laboratories have found effective in comminuting specific commodities.

203 A: EQUIPMENT

Distinctions among various pieces of equipment often relate to the type of sample for which each is most effective. Some equipment is designed to work best with samples that are inherently liquid and thus easily mixed, while other devices provide the power necessary to cut dry products into small pieces for subsequent mixing and homogenization. The following categories of equipment are defined by their mode of operation and the commodity types for which they are most useful.

Blenders and Homogenizers

Devices labelled "blenders" or "homogenizers" usually include blades that are capable of high speed movement but are small relative to the total volume of the container. The container ("blender jar") is designed to propel the material being mixed into a vortex, so that it repeatedly comes into contact with the blades. Such devices are most effective with liquids or materials that liquefy readily when blended. Blenders and homogenizers are most often used in residue analysis for extraction of residues into a solvent that has been added to already chopped sample, but the same devices can also be used for homogenizing commodities that are primarily liquid.

Commercially available devices used for this purpose include Waring, Lourdes, Polytron, and Omni-Mixer models. At least one published study [1] showed that results obtained from using the first three of these were equivalent for practical purposes.

Choppers and Food Processors

Large capacity (20-40 qt) choppers, with blades designed to cut and mix simultaneously, are the traditional equipment used to comminute solid raw agricultural commodities, such as fruits and vegetables, into a homogenate from which test portions are taken. Depending on the water content of the commodity, the final chopped product will consist of a totally homogeneous slurry or a mixture of small pieces. A typical example of this equipment is the Hobart vertical cutter-mixer, originally designed for use in large volume food preparation industries.

Modern food processors, in commercial sizes, are also capable of chopping such commodities into sufficiently fine pieces to provide homogeneity. Because even commercial size food processors are smaller than the 20 qt Hobart cutter-mixers, processing of several batches, followed by thorough mixing, may be necessary.

Grinders

The presence of skin and cartilage in commodities such as raw meat and fish make homogenization difficult. Meat grinders, which force the product through openings in a plate, provide a better mechanism for homogenizing such products. Some choppers (above) can be equipped with grinder attachments so that the single device serves two purposes. Food processors (above) may also comminute these commodities to a suitably homogeneous state. In either case, freezing pieces of the product before grinding improves homogeneity of final mix.

Mills

Dry, hard commodities, such as grains, consist of small individual units, but need further comminuting to expose all parts of the product to the solvent used in the analytical method extraction step. Several types of mills have been found suitable for reducing commodities to particles of <20 mesh.

Mills such as the Wiley mill or Hammer mill grind the commodity with a shearing action created by metal blades rotating at high speed against metal cutting surfaces; a sieve ensures that only particles ground to less than a specified size are able to pass through and be collected. These devices have several drawbacks, including excessive heat buildup and difficult cleaning procedures. Most important, use of mills can cause commodity components to separate from one another and result in a nonhomogeneous final product, because soft, starchy components are reduced to smaller particles than are harder germ and coatings.

High power centrifugal mills are better than these traditional mills for grinding hard materials, including those with high oil content such as soybeans. Centrifugal mills operate by grinding the product with a multiple blade rotor while ground particles are moved through a sieve by centrifugal force. Most models offer a variety of blades and sieves, and the mill can be readily disassembled for cleaning. Capacity of these mills is limited, so processing of small batches and subsequent mixing is usually necessary, but operation of the mill is sufficiently fast that the effort is minimal.

203 B: PROCEDURES FOR SPECIFIC COMMODITIES

The following recommendations for comminuting or homogenizing specific commodities are based on FDA experiences. Once homogeneous material is prepared, a portion is removed and analyzed. If analysis is performed after the homogenate has been frozen, the homogenate must first be thawed completely and mixed thoroughly before a test portion can be taken for analysis; liquid that has separated during freezing and thawing must be re-incorporated.

Crabs and Crayfish

Crabs and crayfish that are marketed live must be sacrificed in order to remove inedible parts (specified in Section 102). Freeze, cook, or autoclave crabs to loosen meat from inedible parts; then prepare a homogenate of edible portion by grinding with a meat grinder, food processor, or chopper.

Eggs

Blend eggs in a Waring or other blender at low speed for ≥5 min or until composite is homogeneous. Low speed blending will minimize foaming or "whipping" of sample.

Fish

Preparation of homogeneous samples of fish depends on whether skin and/or bones are considered edible for the particular species and product. In all cases, preparation must meet the requirements of regulatory policies on what portion of the commodity to include in the composite (Section 102). Skin is removed from species whose skin is considered inedible (*e.g.*, catfish), as are other inedible portions, such as heads, tails, scales, fins, viscera, and inedible bones (Table 102-a). Water that results from thawing frozen fish should be discarded.

Grind the remaining composite three times in a meat grinder, food processor, or food cutter equipped with a grinder attachment [2]. If skin is included in the edible portion, freeze portions of suitable size before introduction into the grinder; this causes the skin to be more brittle and minimizes clogging of the grinder.

Fruits and Vegetables

Chop ≥20 lb dense commodities (*e.g.*, potatoes, beets, carrots) or ≥1/2 bushel loosely formed products (*e.g.*, cabbage, lettuce, greens) in a Hobart vertical cutter mixer ≥5 min; stop machine and scrape material by hand into bottom of mixer at least once during operation. This technique was found to produce adequate particle size and distribution in tests with several agricultural products at several time intervals [3].

Hays, Straws, and Dry, Low Fat Feed Ingredients

Grind samples to fine (about 20) mesh in a centrifugal mill using a 1 mm sieve. Collect ground material in the 500-800 g capacity collecting pan and thoroughly mix several batches as necessary to provide appropriate composite from which to take the test portion [4]. Some materials, including hay, may require grinding through a Wiley mill prior to final grinding through the centrifugal mill.

If a centrifugal mill is not available, grind samples through a Wiley mill or equivalent, taking care to prevent physical separation of the product in the mill. A stepwise grinding procedure, with finer grind produced at each step, may be necessary with some products. At each step, grind sample, divide ground material into four sections, reset mill to produce finer particles, and regrind material from two opposite quarters, until final portion is ≤20 mesh.

If the product is to be analyzed for volatile residues, which may be lost because of the heat generated during grinding, cool the mill prior to grinding the sample by grinding dry ice in it.

Oilseeds

Oilseeds are usually hard, as well as high in oil, so special care is required during initial grinding to prevent excessive heat buildup or separation of oil from the rest of the commodity.

Grind well mixed sample in centrifugal mill equipped with 1 mm sieve ring to produce ground product of ≤20 mesh. If noticeable heat builds up, alternatively grind without sieve ring or use sieve with larger openings (*e.g.*, 3 mm), then regrind using 1 or 0.5 mm sieve. Maintain mill rotor speed at 20,000 rpm to aid in cooling.

Dry Products (Pasta, Dry Beans, Grains, *etc.*)

Products such as dry pasta should be treated as described for oilseeds.

References

[1] Wheeler, W.B., *et al.* (1979) *Bull. Environ. Contam. Toxicol.* **23**, 387-390

[2] Thompson, F.D. (Feb. 1976) "Preparation of Fish Sample Composite by Grinding in Frozen State," LIB 1860, FDA, Rockville, MD

[3] More, C.A. (June 1966) "Sample Preparation Using the Hobart Vertical Cutter Mixer," LIB 402, FDA, Rockville, MD

[4] Sawyer, L.D. (Jan. 1977) "A New Mill for Grinding Difficult Samples," LIB 2023, FDA, Rockville, MD

204: SPECIAL REAGENT PREPARATION

204 A: INTRODUCTION

Reagents used in trace analytical chemistry must be carefully chosen and handled to ensure their purity. Impurities in reagents can cause degradation of residues during the analytical process or can cause determinative step responses that interfere with determination of residues. Each laboratory's quality assurance program plan and standard operating procedures (SOPs) (Section 206) should address the way in which reagents are tested for purity, purified if necessary, and stored in a manner that ensures continued purity.

This section contains general tests for reagent and solvent purity. It also provides directions for handling and purifying certain reagents that are common to many methods. Handling and purification of any reagent used in only one method are described as part of that method.

204 B: PAM I CONVENTIONS FOR REAGENTS

Throughout PAM I, the following conventions are used to describe reagents:

1) Lists of reagents for each method specify the grade that should be used; subsequent directions refer to the reagent by name only, unless more than one grade is used in the method.

2) Cross-reference to this section is included in method description reagents lists (Chapters 3 and 4) whenever the method uses a common reagent for which special directions are included here.

3) In almost all cases, solvents must be distilled in all-glass apparatus; in some cases (*e.g.*, HPLC mobile phases), even greater purification is required or recommended.

4) Unless otherwise specified, "water" means distilled water, except where the water does not mix with the determination, as in "water bath." In the latter case, tap water is acceptable.

5) "Ultrapure water," often required for HPLC, refers to the product prepared using the Milli-Q water purification system or its equivalent.

204 C: GENERAL TESTS FOR REAGENT PURITY

Test for Substances Causing Determinative Step Interference

All reagents used in a method should be checked by performing all steps of the method with no sample present. This "reagent blank" should accompany use of any method being used for the first time, or after a period of inactivity, and periodically thereafter. If performance of a reagent blank indicates the presence of an interference, individual reagents (and apparatus) should be examined separately to locate and eliminate it.

Solvents may be checked separately by concentrating 300 mL to 5 mL in a Kuderna-Danish (K-D) concentrator with Snyder column and calibrated receiving flask, each previously rinsed with solvent. Examination of 5 µL concentrated solvent by the determinative step used in the analysis should result in no recorder deflection >1 mm from baseline for 2-60 min after injection.

Other reagents and apparatus used in a method should be checked by rinsing them with solvents that are used in the method, concentrating rinse solvents if appropriate to the method, and checking for responses by the appropriate determinative step. No responses should be detected.

Test for Substances Causing Pesticide Degradation

Substances that degrade residues may also be present in reagents, but these will not be identified by the previous test unless they also cause determinative step response. To detect such impurities, known amounts of chemicals subject to degradation should be added to the extracting solvent (no commodity) and the whole method performed. Noticeable losses of sensitive chemicals indicate the presence of unacceptable contaminants in the reagents. Common contaminants of this type include oxidants that may be present in solvents; these cause degradation of organophosphorus pesticides, especially carbophenothion, particularly during evaporation.

204 D: TESTS AND PURIFICATION PROCESSES FOR SPECIFIC REAGENTS

Acetonitrile

PAM I methods specify use of acetonitrile distilled from all-glass apparatus. To make use of reagent grade acetonitrile, test for impurities by holding moistened litmus paper over mouth of storage container. If litmus paper turns blue, purify 4 L acetonitrile by adding 1 mL 85% phosphoric acid, 30 g phosphorus pentoxide, and boiling chips, then allowing to stand overnight. Distill from all glass apparatus at 81-82° C, discarding first and last 10% of distillate; do not exceed 82° C.

Ethyl Ether

PAM I methods specify use of ethyl ether distilled from all-glass apparatus and assume the presence of 2% ethanol added as a "stabilizer" to prevent formation of peroxides. Practical shelf life is limited, however, even when alcohol has been added; peroxides form readily. Test for peroxides using "Peroxid Test" paper.

Glass Wool

PAM I methods specify use of Pyrex glass wool, which can have contaminants that interfere with determination. If reagent blank tests indicate that glass wool is contaminated, rinse it with solvent and air-dry or heat 1 hr at 400° C.

Some PAM I methods specify silanized glass wool, which may be purchased. To silanize glass wool in laboratory, soak 10 min in 10% dimethyldichlorosilane, rinse with toluene, and soak another 10 min in methanol. Rinse with methanol and allow to air-dry.

Sodium Sulfate

PAM I methods specify use of anhydrous, granular, reagent grade sodium sulfate. To remove phthalate esters that interfere in determinations using electron capture detectors, heat sodium sulfate 4 hr in muffle furnace at 600° C. Store in glass containers; if plastic lids are used, separate them from sodium sulfate with layer of foil.

If reagent blank tests indicate that sodium sulfate is contributing interferences to other determinations, rinse several times with acetone and ethanol, then dry.

Florisil

Florisil, a synthetic magnesium silicate long used as an adsorbent in FDA methodology, is subject to variations in adsorptivity common to most analytical grade adsorbents. Years of experience in using Florisil have led to establishment of procedures for purchasing, handling, and testing the material to optimize and standardize its application. PR Grade Florisil (U.S. Silica, Berkeley Springs, WV 25411) is specified because other grades available from chemical supply companies may be prepared differently by the manufacturer and may exhibit drastically different adsorptivity from what is required for PAM I methods. Handling directions are designed to prevent contamination that may interfere with subsequent analyses and to ensure consistent adsorptivity throughout use of a particular lot of Florisil.

Purchasing and Handling. Observe these procedures for handling Florisil:

- Use PR Grade Florisil, 60-100 mesh, calcined (heated) 3 hr at 1250° F (677° C), for all PAM I methods that require Florisil. Other grades may not be capable of providing the elution patterns required for successful application of the methods.

- Immediately after opening bulk lots of Florisil, transfer to glass containers (preferably amber) that are glass-stoppered or have Teflon-lined or foil-lined screw caps; store in dark. Activate each portion by heating at 130° C for 168 hr (1 wk) before use. Store at 130° C in foil-covered bottles. Florisil may be heated in bulk in pint glass bottles or in individual column amounts in 50 mL Erlenmeyer flasks. Cover containers with foil to prevent contamination, and use in rotation to avoid lengthy storage time. Alternatively, store stoppered container of activated Florisil in desiccator at room temperature and reheat at 130° C after 2 days.

- If entire lot of Florisil is purchased, perform tests below on composite of four-five subsamples taken from each drum with grain trier. Combine subsamples, mix well, and activate mix at 130° C for 168 hr before testing.

Testing. Each lot of activated Florisil must be tested before use to determine whether adjustments in column size are needed to ensure proper elution and quantitative recovery of pesticides. Florisil column size is decreased or increased to adjust for over-retentive or under-retentive Florisil. Two tests should be performed: the "lauric acid test," which measures general adsorptivity, and an elution test that confirms the appropriate elution of pesticides.

LAURIC ACID TEST

Reference

Mills, P.A. (1968) *J. Assoc. Off. Anal. Chem.* **51**, 29-32

Principles

Adsorptivity capacity of Florisil is measured by exposing weighed amount to excess of lauric acid in hexane solution. Amount of lauric acid not adsorbed is measured by titration with alkali. Weight of lauric acid adsorbed ("LA Value") is subsequently used to calculate appropriate weight of that lot of Florisil equivalent to standardized Florisil (LA Value 110).

Apparatus

buret, 25 mL with 0.1 mL graduations, Class A

Erlenmeyer flasks, 125 mL narrow mouth and 25 mL T

GLC, equipped with ^{63}Ni electron capture (EC) and flame photometric, phosphorus mode (FPD-P) detectors (Section 302 DG1, DG2)

pipets, 10 and 20 mL transfer, Class A

volumetric flasks, 500 mL, Class A

Reagents

ethanol, USP or absolute, neutralized to phenolphthalein

hexane, distilled from all-glass apparatus

lauric acid, purified, CP

lauric acid solution, 10.000 g lauric acid/500 mL hexane (1 mL solution = 20 mg lauric acid)

phenolphthalein indicator, 1 g/100 mL ethanol

sodium hydroxide, pellets, reagent grade

sodium hydroxide solution, 0.05 N. Make 1 N solution (20 g/500 mL water), and dilute 25 mL to 500 mL with water. Standardize by weighing 100-200 mg lauric acid into 125 mL Erlenmeyer flask. Add 50 mL neutralized ethanol and 3 drops phenolphthalein indicator; titrate to permanent end point. Calculate mg lauric acid/mL 0.05 N sodium hydroxide (about 10 mg/mL).

Directions

Calculate LA Value for each Florisil lot by performing the following test in triplicate. When method directions in Chapters 3 and 4 require adjustment of Florisil weight for LA Value, calculate as follows: 110/LA Value × weight specified.

- Transfer 2.000 g Florisil to 25 mL T Erlenmeyer flask. Cover loosely with aluminum foil and heat overnight at 130° C.
- Stopper, cool to room temperature, add 20.0 mL lauric acid solution (400 mg), stopper, and shake occasionally 15 min.
- Let adsorbent settle and pipet 10.0 mL supernatant into 125 mL Erlenmeyer flask. Avoid inclusion of any Florisil.
- Add 50 mL neutral alcohol and 3 drops phenolphthalein indicator solution. Titrate with 0.05 N sodium hydroxide to permanent end point.
- Calculate LA Value (mg lauric acid/g Florisil):

$$\text{LA Value} = 200 - \frac{\text{mL required for titration} \times \text{mg lauric acid}}{\text{mL 0.05 N sodium hydroxide}}$$

ELUTION TEST

Reference

Bong, R.L. (1991) Minneapolis District SOPs for Florisil, FDA, private communication

Principles

Solutions of pesticides and butterfat are eluted from Florisil columns, adjusted for LA Value, by eluants from PAM I methods. Appropriate elution of pesticides and weight of butterfat verify that elution pattern and cleanup capacity are adequate. Pesticides are chosen to provide indicators of improper elution, poor Florisil, and impure reagents.

Apparatus

chromatographic column, 22 mm id × 300 mm, Teflon stopcock, coarse porosity fritted disc

K-D concentrator, 500 mL, with Snyder column, two-ball micro-Snyder column, volumetric receiving flask

Reagents

acetonitrile, distilled from all-glass apparatus, see above

ethyl ether, distilled from all-glass apparatus, with 2% ethanol as preservative, peroxide free (see above)

hexane, distilled from all-glass apparatus

methylene chloride, distilled from all-glass apparatus

petroleum ether, distilled from all-glass apparatus

eluants: 6% (v/v) ethyl ether/petroleum ether
15% (v/v) ethyl ether/petroleum ether
50% (v/v) ethyl ether/petroleum ether

eluant 1—20% methylene chloride/hexane (v/v). Dilute 200 mL methylene chloride with hexane. Allow mixture to reach room temperature, and adjust volume to 1 L with hexane.

eluant 2—50% methylene chloride/0.35% acetonitrile/49.65% hexane (v/v/v). Pipet 3.5 mL acetonitrile into 500 mL methylene chloride and dilute with hexane. Allow mixture to reach room temperature and adjust to 1 L with hexane.

eluant 3—50% methylene chloride/1.5% acetonitrile/48.5% hexane (v/v/v). Pipet 15 mL acetonitrile into 500 mL methylene chloride and dilute with hexane. Allow mixture to reach room temperature and adjust to 1 L with hexane.

Pesticide standard solutions, each mL containing these approximate concentrations:

A: 1.0 μg heptachlor, 3.0 μg chlorpyrifos, 2.0 μg heptachlor epoxide, 2.0 μg dieldrin, 3.0 μg endosulfan I, 3.0 μg endosulfan II, 10.0 μg endosulfan sulfate

B: 4.0 μg malathion, 2.0 μg parathion-methyl, 4.0 μg fonofos, 4.0 μg pirimiphos-methyl

C: 20.0 μg Aroclor 1254, 200.0 mg butterfat

D: 1.0 μg α-BHC, 3.0 μg chlorpyrifos, 1.0 μg heptachlor, 2.0 μg heptachlor epoxide, 2.0 μg dieldrin, 2.0 μg endrin, 4.0 μg malathion, 2.0 μg parathion-methyl

Directions

- Prepare three Florisil columns to contain, respectively: 110/LA Value × 20 g, 2 g more than that, and 2 g less than that.
- Rinse columns with 50 mL petroleum ether, discarding rinses. Place K-D with 10 mL volumetric flask under each column.
- Pipet 1.0 mL each standard solutions A and B onto each column. Rinse sides of column with two 3 mL portions petroleum ether, then rinse column with 50 mL petroleum ether.

- Elute each column with 200 mL 6% ethyl ether/petroleum ether. (Collect rinses with this eluate.)

- Change receivers; elute each column with 200 mL 15% ethyl ether/petroleum ether.

- Change receivers; elute each column with 200 mL 50% ethyl ether/petroleum ether.

- Concentrate each eluate, dilute to volume with hexane, and inject about 5 µL into appropriate GLC systems to determine recoveries. Dilute 1.0 mL each standard solutions A and B to 10 mL and use diluted solution as GLC reference standard.

- Consider Florisil lot acceptable if one of three columns permits complete recovery of test compounds and exhibits proper elution pattern (heptachlor, heptachlor epoxide, chlorpyrifos, and fonofos in 6% eluate; dieldrin, endosulfan I, parathion-methyl, and pirimiphos-methyl in 15% eluate; malathion and endosulfan sulfate in 50% eluate; and endosulfan II in both 15 and 50% eluates). Acceptable recovery is >80% for all compounds except heptachlor, and 60-90% for heptachlor. In subsequent use of lot of Florisil, use same weight as that in column with acceptable elution.

- If none of the three columns exhibits proper elution but a consistent relationship exists between weight and elution, test additional columns of weights 3 g above or 3 g below that calculated using LA Value. If these columns also do not exhibit proper elution, it is best to use a different lot of Florisil.

If acceptable weight of Florisil is determined, test that column size further with following procedures:

- Repeat elution tests above, using 1.0 mL each solutions C and D. Elute column with 250 mL petroleum ether, followed by 6, 15, and 50% ethyl ether/petroleum ether eluants; collect each eluate separately. Determine recoveries of pesticides and verify accuracy of elution pattern using gas chromatographic measurement.

- Transfer each eluate quantitatively to separate tared 20 mL beaker. Evaporate solvent on steam bath or hot plate until constant weight is attained to measure amount of butterfat recovered in each eluate. Acceptable lots of Florisil typically permit about 0.3 mg (range 0-1.7 mg) butterfat to elute in petroleum ether eluate, 0.1 (0-0.4) mg in 6% ethyl ether/petroleum ether, 82 (40-135) mg in 15%, and 105 (60-172) mg in 50%.

- Repeat elution tests above, using 1.0 mL each solutions A and B and eluting with Eluants 1, 2, and 3 instead of ethyl ether/petroleum ether eluants.

It is acceptable, once the Florisil lot has been tested and appropriate weight of Florisil determined, to measure and record height of column produced by specified weight; subsequent columns may then be prepared by measuring height rather than weight.

205: REFERENCE STANDARDS

The purity of reference standards and use of appropriate preparation and storage techniques for standard solutions significantly affect analytical results. Reliable and accurate data can be obtained only if correct analytical standard solutions are used for identification and quantitation. Each laboratory's quality assurance program plan (Section 206) should include an element on reference standards and standard solutions. Standard operating procedures (SOPs) should include protocols for obtaining, labeling, storing, and handling standards. This section provides rudimentary information that may be incorporated, as appropriate, into such documentation.

205 A: SOURCES

Reference standards are currently available from several commercial sources, including companies that supply only reference standards, suppliers of specialty laboratory chemicals, and suppliers of chromatographic equipment. Each company publishes lists of reference standards for pesticides, related metabolites, and certain industrial chemicals. Eligible laboratories, mainly Federal Government laboratories, may also obtain reference standards for some chemicals from a repository maintained, under contract, by EPA; eligibility is determined by EPA.

Reference standards in "neat" (undiluted) form, preferably certified by EPA, should be used whenever possible. If neat standards are not available, certified solutions of standards may be used.

205 B: EQUIPMENT AND SOLVENTS

Equipment

Equipment used for preparation and storage of reference standards and solutions includes the following essential, but not all-inclusive, items:

1) analytical balance calibrated for accuracy of ±0.05 mg

2) explosion-resistant refrigerator/freezer, used only to store standards

3) standard solution storage containers:

   a) amber colored, screw-cap bottles, 1 and 2 oz

   b) Teflon-lined caps for bottles

   c) vials for working standards

4) desiccators to store reference standards. Larger vials containing desiccant can be used as individual desiccators for vials of standards.

5) appropriate volumetric glassware, pipets, or microliter syringes

Solvents

Pesticide residue quality solvents are essential for preparation of reference standard solutions. Solvents should be checked before use for the presence of interfering substances by injecting the solvent into the determinative system(s) to be used.

Choice of solvent is sometimes restricted by solubility and stability of the particular chemical. The following solvents, in order of preference, should be used to prepare standard solutions, if suitable for the particular chemical: isooctane (2,2,4-trimethylpentane), hexane, acetone, isopropanol, and toluene.

205 C: STORAGE

Reference standards must be stored properly to prevent undesirable reactions, such as oxidation, re-arrangement, or hydrolysis. Improper storage can lead to loss of integrity of previously acceptable standards. Storage conditions must also prevent the possibility of external contamination. Storage requirements are dependent on the chemical and physical properties of the chemical of interest and are much more stringent for volatile, reactive, or unstable compounds. Review the physical and chemical properties of each compound to determine which storage conditions are appropriate. Minimum requirements for long term storage of analytical reference standards follow:

- If at all possible, store reference standards in tightly sealed containers under desiccation in a freezer.
- Store more stable compounds, such as organochlorine pesticides, in a refrigerator if freezer is not available.

Reference standards that have been stored in refrigerators or freezers must be brought to room temperature in a desiccator prior to weighing.

205 D: PURITY

The analyst is responsible for knowing the purity of the reference standard used to obtain reported data. Follow these rules for recording information about reference standard purity:

- Standards with known purity ≥99%: weight may be recorded as measured; it is not necessary to correct for purity.
- Standards with purity <99%: apply appropriate correction factor to measured weights.
- Technical standards with unknown purity (use only if this is the only available reference standard): record weight as measured, do not correct for purity, but include a note on the source and unknown purity of this standard with the results of any analysis whose results rely on this standard.

205 E: STANDARD SOLUTIONS

Use of inaccurate standard solutions leads to correspondingly incorrect data even if excellent technique and instrumentation are employed. Many analysts consider problems associated with standards and standard solutions as the greatest single source of error in trace residue analysis. Standard solution accuracy is dependent on accurate weighing, correct choice of solvent, chemical stability, appropriate storage conditions, and accuracy in recording the information about solution preparation.

Definitions

The following definitions are used in discussions of standard solutions:

standard "stock" solutions: initial solution from which other dilute solutions are prepared

standard "working" solutions: prepared from stock solutions and appropriately diluted for use in quantitation

Protocols for Preparing Standard Solutions

The following basic requirements are recommended for inclusion in the laboratory's protocol:

Weighing Standards.

- Use only suitable and calibrated analytical balances to weigh standards.
- Weigh solids not affected by moisture on analytical balances with pans open to atmosphere.
- Weigh semisolid standards or liquid standards by technique appropriate to physical and chemical properties of compound. For example, add material to tared volumetric flask, then immediately stopper and reweigh flask.
- Measure appropriate volume of volatile liquid standard in microliter syringe and introduce below surface of solvent in volumetric flask. Dilute to volume. Calculate concentration using volume and specific gravity of liquid standard.

Preparing Solutions.

- Rinse volumetric glassware with solvent in which standard will be dissolved.
- Use pipets, volumetric glassware, or accurate microliter syringes for dilution.
- Dissolve reference standard in solvent in which it is known to be completely soluble. Be aware of any solids remaining in the solution, and ensure that dissolution is complete before using.

- Use solvents of lowest volatility, lowest reactivity, and lowest toxicity possible. If necessary to use a less desirable solvent in order to completely dissolve weighed standard, use minimum amount necessary for complete dissolution, then dilute with solvent of choice. Check for precipitation that may be caused by addition of diluent.

- If possible, verify solution identity and concentration by comparing determinative system data for new solution to data previously reported for that chemical.

Storage of Solutions.

- Be aware that special storage conditions may be necessary for solutions prepared from chemicals that are unstable, reactive, or volatile.

- Prepare new working standard solutions frequently, at least every 6 mo.

- Verify accuracy of concentration of working solutions as needed.

Record-keeping.

- Use similar formats in all types of record-keeping for similar type work. (This approach is also recommended to laboratories within the same organization but at various locations.)

- Record all raw data, such as physical appearance, weight of material dissolved, and dilutions; have all calculations checked by a second analyst.

- Label all solutions with identification number, compound name, concentration, date prepared, analyst initials, and solvent(s) used.

205 F: SAFETY

Procedures for safe handling of reference standards and solutions must be included among the SOPs of each laboratory's Chemical Hygiene Plan (Section 207). Information from the Material Safety Data Sheet supplied with each reference standard and other sources should be used in developing procedures for handling and weighing reference materials. Particular attention should be given to compound toxicity and likelihood of analyst exposure to the compound during handling.

205 G: DISPOSAL OF REFERENCE STANDARDS AND SOLUTIONS

Pesticide reference standards, including primary or working solutions, are classified as hazardous waste under the requirements of the Resource Conservation and Recovery Act. As such, provision for appropriate collection, storage, and disposal of outdated reference standards must be included in each laboratory's hazardous waste disposal plans (Section 208).

206: QUALITY ASSURANCE AND QUALITY CONTROL

206 A: GENERAL PRINCIPLES

Quality assurance (QA) and quality control (QC) should be integral parts of any pesticide residue program in order to ensure the accuracy and appropriate documentation of data generated by the program. The QA process consists of management review and oversight at the planning, implementation, and completion stages of the data collection activity to ensure that data are of the quality required. The QC process includes those activities required during data collection to produce the data quality desired and to document the quality of the collected data. QA activities ensure that the QC system is functioning effectively and that any deficiencies are corrected.

QA/QC programs related to pesticide residue testing emphasize the importance of accuracy and reliability of data. Regulatory action is based on such analyses, as are long term decisions such as banning pesticides. A well-functioning QA/QC program benefits the laboratory and the regulatory program by:

1) ensuring that data are scientifically sound and legally defensible

2) preserving data integrity, validity, and usability

3) ensuring that analytical measurement systems are maintained in an acceptable state of stability and reproducibility

4) establishing the continuing need for training

5) recognizing problems through data assessment

6) establishing corrective action procedures that keep the analytical process reliable

Each laboratory should establish a QA/QC program to ensure reliable analytical data and to document its reliability. This program should include QC procedures, any necessary corrective action, and all documentation required during data collection. The laboratory should prepare, maintain, and support both a written QA Program Plan and standard operating procedures (SOPs). The written QA Program Plan and SOPs should reflect activities as they are currently performed in the laboratory.

Differences between a QA Program Plan and SOPs are subtle. A QA Program Plan provides, in general terms, the QA/QC activities, policies, organization, objectives, and functional guidelines, while SOPs provide detailed step-by-step procedures for operations, analysis, and action. The writing voice for the two types of documents is different, with the QA Program Plans usually written in indicative style and SOPs in imperative style. Written documents (both QA Program Plans and SOPs) that describe procedures performed to accomplish the goals of the program should relate to results rather than to specific activities or procedures. Goals should be achievable and measurable so that the program's success can be evaluated.

Computer applications form a subset of topics within laboratory QA/QC as computers increasingly replace many manual procedures related to laboratory operations and data collection. Computers now manage operations, interface with laboratory equipment, and generate scientific/technical reports. They are increasingly used in laboratories to process, store, and retrieve data; schedule and monitor work throughput; generate test reports; capture data directly from instruments; control critical environmental conditions; and process and display laboratory quality control data. QA Program Plan elements and SOPs should be written to cover computers and their applications wherever appropriate to the laboratory's operation.

This section provides suggestions for a QA Program Plan as well as guidance for preparing SOPs. These materials are suggestions only; their appearance in this manual does not establish them as requirements within FDA or elsewhere. Each laboratory and organization is responsible for preparation of materials appropriate to its own work and required by programs in which it participates.

206 B: QA PROGRAM PLAN

The elements of a written QA Program Plan, outlined below, may be presented in any order. Consideration given to each element will differ among laboratories, depending upon laboratory setting, function, and quality of the measurements deemed essential. A QA Program Plan should fit the needs of the laboratory's program and is not limited to these elements.

Quality Control Points

A. Organizational chart with reporting relationships
B. Responsibilities
   1. Assignments of QC and QA
   2. Management of the quality system

Quality of Equipment

A. Performance criteria for each type of equipment, including computers—minimum standards
B. Responsible person for:
   1. Performance checks
   2. Evaluating performance check results
C. Frequency of performance checks
D. Corrective action—equipment failure
E. Equipment performance log books
F. Equipment maintenance log books—accurate and up-to-date

Quality of Standards and Reagents

A. Preparation, labeling, and documentation of reagents and standards
B. Standards
   1. Identification of primary *vs* secondary standards
   2. Verification of secondary standards (purity, potency, and viability)
   3. Documentation of verification

4. Frequency of verification
5. Handling and storage of standard materials

C. Reagents/media/solvents
1. Handling and storage
2. Procurement procedures to ensure supply and quality
3. Criteria for laboratory water—verification and frequency

Environmental Control/Facility

A. Environmental conditions—documentation
1. Samples
2. Instruments/equipment
3. Computers
4. Personnel

B. Facility
1. Security
a. Laboratory
b. Computer
2. Air handling system—maintenance documentation
3. Sample handling and storage

Quality of Analytical Work

A. Method validation
B. Quality control
1. Responsibility designation
2. Specification of intervals for internal QC techniques
a. Fortified sample
b. Analysis of standard reference material
c. Duplicate analysis requirements
d. Split samples
3. Reference material analysis
4. Corrective action—QC failure
C. Sample analysis procedures
D. Calibration procedure and frequency
E. Corrective action—analysis/calibration failure
1. Decision processes
2. Responsibility for initiation
3. Procedure for correction

Quality of Analytical Documentation

A. Data generation, manual or computerized
1. Data collection procedures
2. Data reduction procedures
3. Data validation procedures

4. Data reporting and approval procedures
   a. Supervisory review of analytical worksheets
   b. Internal laboratory audit of worksheets
   c. Oral review of worksheets
5. Data maintenance (storage, retrieval, and retention)

B. Laboratory notebook and log book policy

Audits

A. Performance audits—internal/external
B. System audits—internal/external

Sample Accountability

A. Sample receipt
   1. Sample custody
   2. Sample tracking
B. Sample storage and handling
C. Sample scheduling
D. Sample disposal/archiving

Quality of Administrative Systems

A. Training program
B. QA reports to management
C. Corrective action procedures

206 C: SOPs

In order to obtain reliable and documented results, adherence to prescribed analytical methodology is imperative. In any operation performed on a repetitive basis, reproducibility is best accomplished through use of SOPs. The SOP describes the commonly accepted method(s) for performing certain routine or repetitive tasks. Adherence to SOPs ensures that analytical results are reliable, reproducible, and properly documented and thus support data quality.

SOPs prepared by laboratories should be up-to-date, comprehensive, clear, and sufficiently detailed to permit duplication of results by qualified analysts. In addition, all SOPs should be:

1) amenable to documentation that is sufficiently complete to record performance of all tasks required by the procedure

2) consistent with current guidelines, regulations, and other requirements

3) consistent with instrument manufacturers' specific instruction manuals

4) inclusive of corrective measures and feedback mechanisms utilized when analytical results do not meet procedural requirements

5) reviewed regularly and updated as necessary when facility or procedural modifications are made

6) capable of demonstrating validity of data reported by the laboratory and explaining the cause of missing or inconsistent results

7) subject to a document control procedure that precludes the use of outdated or inappropriate SOPs

8) available at appropriate work stations

9) archived for future reference or evidentiary situations

SOPs should be written in a format prescribed by the operational QA Program Plan; establishment of a format promotes consistency among SOPs and simplifies the writing process. SOPs are usually written in imperative mode.

Typical topics for SOPs include the following:

1) General laboratory techniques; *e.g.*, use of glassware; glassware cleaning; pipetting techniques; analytical balances, calibration and use

2) Reagents and standard preparation, including source, concentration, storage, and labeling (*e.g.*, see Sections 204, Special Reagent Preparation, and 205, Reference Standards)

3) Sample management; *e.g.*, receipt, handling, and custody; scheduling; shipping requirements; and sample storage

4) Instrument and computer calibration and maintenance; *e.g.*, maintenance logs, procedure and schedule, service arrangements, spare parts

5) Laboratory test methods, including sample preparation and analysis (analyte and matrix specific), test-specific QC, instrument standardization, and quantitation and reporting limits

6) Procedures for data reduction; includes data validation, reviewing, and reporting; verifying electronic data input; and electronic reporting

7) Records management; *e.g.*, generating, controlling, and archiving project-specific and operations records; backup and recovery of computer data; defining raw data

8) Laboratory QC; *e.g.*, procedures for determining method quantitation limits, acceptance/rejection criteria for blanks, matrix-specific quantitation limit, methods precision and bias, matrix-specific bias, matrix-specific precision, control limits for precision and bias, historical performance

9) Laboratory records handling, including review, approval, and revision; computer data entry; and security

10) Waste disposal; includes disposal of samples and waste material

11) QA review; includes requirements for internal, external, and on-site assessment

206 D: BIBLIOGRAPHY

The following references are recommended for guidance in establishing QA Plans and SOPs:

Dux, J.P. (1986) *Handbook of Quality Assurance for the Analytical Chemistry Laboratory*, Van Nostrand Reinhold Company Inc., New York, NY

Garfield, F.M. (1991) *Quality Assurance Principles for Analytical Laboratories*, AOAC International, Arlington, VA

General Requirements for the Competence of Calibration and Testing Laboratories, 3rd ed. (1990) Guide 25, International Standards Organization, Geneva, Switzerland

Good Automated Laboratory Practices—EPA's Recommendations for Ensuring Data Integrity in Automated Laboratory Operations with Implementation Guidance (in press, 1994) Environmental Protection Agency, Washington, DC

Quality Assurance Program (1988) Food and Drug Administration, Office of Regulatory Affairs, Rockville, MD

Standard Guide for Establishing a Quality Assurance Program for Analytical Chemistry Laboratories Within the Nuclear Industry (1983) Document C 1009, American Society for Testing and Materials, Philadelphia, PA

Taylor, J.K. (1987) *Quality Assurance of Chemical Measurements*, Lewis Publishers, Chelsea, MI

207: SAFETY

207 A: INTRODUCTION

Chemists analyzing foods for pesticide residues are affected by safety issues, as are all persons working in or managing chemical laboratories. There are many challenges and obstacles to ensuring a safe workplace, especially the costs of upgrading facilities and providing appropriate training, but by developing an effective plan and extensive training of all employees, safe and healthy work conditions are attainable.

It is beyond the scope of the PAM to provide details of an adequate laboratory safety program. Instead, this section is included to provide background information on laws and regulations related to laboratory safety and to stress the need for every laboratory to have a safety plan and for every employee to adhere to that plan. Because the PAM is largely directed toward and used by laboratory chemists, responsibilities of the individual chemist for laboratory safety are emphasized.

207 B: LAWS AND REGULATIONS RELATED TO SAFETY

While it has always been in the best interest of employers to ensure that safety is a primary concern to reduce injuries, property damage, liability, and lost time of employees from the job, occupational safety and employee health issues have drastically changed in the last 20 years as a result of regulatory oversight. The following laws and regulations form the basis for safety requirements in the United States:

1) Williams-Steiger Occupational Safety and Health Act, 1970, which established the Occupational Safety and Health Administration (OSHA) and authorized it to regulate safety and health issues for all employees

2) Executive Order 12196, 1980, which requires each Federal agency to have an occupational safety and health program

3) 29 Code of Federal Regulations (CFR) 1910, which includes OSHA's regulations for toxic and hazardous substances (listed in Subpart Z Sections 1910.1000-1910.1500) that affect laboratory personnel. 29 CFR 1910 includes the following specific regulations:

   a) 1910.1000 establishes, for specific chemicals, limits on the levels to which employees may be exposed. Depending on the hazard of the chemical, a limit is defined either as a permissible exposure limit (PEL), measured in a time-weighted average (TWA) over an 8 hr day, or as a short term exposure limit (STEL), measured in a 15 min TWA exposure, not to be exceeded at any time. Chemicals for which PELs, TWAs, and STELs have been established are listed in Table Z of Section 1910.1000, along with the limits. Implementation of these regulations requires engineering or administrative controls to protect laboratory employees from excess exposure or, in the absence or inefficiency of these controls, use of personal protective equipment (PPE).

b) 1910.1200, also known as the Hazard Communication Standard, requires that manufacturers and importers of hazardous materials protect users from potential dangers by providing written notification of the hazards, in the form of a Material Safety Data Sheet (MSDS) for each chemical. This section also requires manufacturers to provide hazard information on chemical container labels.

c) 1910.1450 establishes the Standard for Occupational Exposure to Hazardous Chemicals in Laboratories ("Lab Standard"). The Lab Standard was issued by OSHA, in recognition of unique characteristics of chemical laboratories, to protect employees associated with laboratory operations. Among other requirements, the Lab Standard requires employers to prepare a written Chemical Hygiene Plan (CHP) to establish work practices, procedures, and policies that reduce the potential for employee exposure to hazardous chemicals. The Lab Standard also establishes requirements for appropriate employee training in safety practices, for monitoring, for appropriate PPE to be worn when hazards cannot be otherwise controlled, and for medical surveillance.

It is FDA policy to select operational strategies that foster a safe and healthful environment for all employees and for those communities in which FDA operates. FDA complies with OSHA regulations, including ensuring that its laboratories operate according to the Lab Standard. To that end, each FDA laboratory has prepared a CHP. FDA's Safety and Occupational Health Management Program is described in the FDA Staff Manual Guide, 2130.1 through 2130.7.

207 C: MATERIAL SAFETY DATA SHEETS

An MSDS, required by OSHA's Hazard Communication Standard, provides precautionary information to the user on physical and health hazards of an individual chemical. Its availability enables chemists to make hazard determinations for materials handled in the laboratory and to identify unsafe conditions that may exist. The recognized source of an MSDS for any hazardous material is the actual manufacturer. If asked, manufacturers usually will provide MSDSs for chemicals purchased prior to the Standard. OSHA also recognizes generic MSDSs as a substitute for those of the actual manufacturer when the original MSDS is not available; these may be substituted for older materials manufactured by a company that has gone out of business.

MSDSs are required to provide the following information in brief:

1) label name of the material; manufacturer's name, address, and telephone numbers for emergencies; and further information

2) list of all hazardous ingredients, by chemical and common names, percentage concentration of each hazardous component, and established PELs and/or threshold limit values (TLVs), established by the American Conference of Governmental Industrial Hygienists

3) various physical and chemical characteristics, including boiling point, specific gravity, appearance and odor, melting point, solubility, vapor pressure, evaporation rate, odor threshold, *etc.*

4) fire and explosion hazard data for fire fighters, with such information as flash point, hazardous fire decomposition products, special fire-fighting procedures, flammable limits, and upper and lower explosion limit concentrations

5) reactivity data for stability, incompatibility, hazardous decomposition products, polymerization potential, and conditions to avoid

6) health hazard information with specific information on acute and chronic health effects expected for each route of entry into the body, whether a carcinogen is present, what signs and symptoms can be expected if exposed, what medical conditions could be aggravated if exposed, and emergency/first aid to be administered if exposed

7) precautions for safe handling and use of the material, including procedures for spill cleanup and waste disposal, conditions to avoid when storing the material, and any other precautions for handling the material

8) recommended control measures, such as what PPE should be used when handling the material, whether special ventilation is needed when handling the material, what glove and eye protection is needed, whether any other protective equipment is needed, and what other work or hygienic practices should be followed when handling the material

MSDSs provide the laboratory employee with a ready source of information about chemical hazard, much of which can be used during development of SOPs (below). MSDSs can also be used to accompany shipments of hazardous materials to fulfill the Department of Transportation requirements for information on spill remediation.

Manufacturers often recommend extensive control methods for use of their hazardous materials to avoid any liability, even when the controls address the concentrated form of the material and normal usage involves a diluted product. MSDSs often do not give specific information for waste disposal and other highly regulated areas, because such requirements vary by geographic location. Specific information is usually lacking for exactly which PPE should be used to avoid specification of trade name products or because the manufacturer has not always thoroughly tested various PPEs with the product. The information presented in MSDSs is usually very general, but technical, in nature. OSHA has expressed concern over the quantity and quality of information present in the MSDSs; manufacturers are required to complete each section, even if the only information conveyed is that inadequate testing has been done.

207 D: DEVELOPMENT OF A CHEMICAL HYGIENE PLAN

As required by OSHA's Lab Standard, each laboratory must have a CHP that provides written statements of work practices, procedures, and policies intended to reduce the potential for employee exposure to hazardous chemicals. The CHP must be made available to all employees. The Lab Standard is a performance-oriented standard that can be readily changed to address the current needs of the laboratory personnel; such flexibility facilitates compliance, despite the complexity and diversity of tasks performed in the laboratory. The CHP must identify:

1) standard operating procedures (SOPs), relevant to safety and health, for activities involving use of hazardous chemicals. Each SOP must identify: the exact nature of the hazard, what safety procedures are established to eliminate or reduce the hazard, what personal protective clothing and equipment are needed to protect the employee, what immediate steps will be taken in the event of an emergency or spill, and what steps will be taken to remedy the situation afterwards, such as decontamination of a spill. SOPs should provide procedures for: handling highly specific reactive chemicals; operating equipment whose use poses potential hazards, malfunction, or repair; handling ionizing and nonionizing radiation; handling compressed and high pressure gases; and working with extremely low or high temperatures.

2) criteria used to determine when additional controls are needed to reduce potential personnel exposure, particularly when highly toxic materials are used. Provision must be made for additional protection when a project involves use of highly toxic or hazardous components, reproductive toxins, or carcinogens. Such protection can include isolating the work area, limiting personnel assigned to the project, providing special PPE, requiring decontamination steps, outlining special waste considerations, and providing special emergency safety equipment and containment devices.

3) circumstances under which certain laboratory procedures require prior approval of the supervisor

4) procedures for medical surveillance and consultation when an exposure is suspected, including an exposure assessment

5) procedures for monitoring for any substance regulated by OSHA, if there is reason to believe that exposure levels exceed the action level, or, in the absence of an action level, the PEL level

6) provisions for maintaining individual employee records of exposure monitoring, medical consultations, and evaluations

7) provisions for personnel training and information

8) procedures for evaluating engineering controls, such as fume hoods, to verify proper functioning

9) identification and maintenance of emergency handling equipment, such as fire extinguishers, eye washes, safety showers, fire alarms, *etc.*

10) measures taken to ensure PPE is functioning correctly

11) emergency response and remediation procedures

12) the Chemical Hygiene Officer who will develop and implement the CHP, and other individuals responsible for implementing any portion of it, including phone numbers

13) procedures for storing materials safely. Issues addressed in this section should include classes of chemicals and segregation to ensure compatibility, expected lifetime of chemicals in general, special cases where degradation is common (*e.g.*, picric acid and peroxide formers), and flammability storage issues. Under the Lab Standard, MSDSs must be maintained and labels on incoming hazardous materials must not be removed or defaced.

14) any other issues, such as individual work practices, attire, electrical hazards, and housekeeping, that affect safety in the workplace

There are many references available to assist chemists in preparing SOPs for the CHP. Information in MSDSs is particularly useful for identifying hazards and safe levels of exposure. Many hazardous materials listed in 29 CFR 1910.1000, Table Z, are routinely used in pesticide analytical laboratories. The levels and standards in Subpart Z are an excellent reference for laboratory personnel for preparing SOPs. A comparison of the odor threshold to the TLV or PEL values is invaluable information in indicating quickly what practices and controls must be in place when working with certain hazardous materials. Often information presented in an MSDS has legal authority and accordingly is an excellent reference. However, according to 29 CFR 1910.1450 (a)(2)(c), the Lab Standard supersedes the Hazard Communication Standard for laboratory operations; more restrictive limits of exposure may be established in the CHP than are established in the MSDS.

Monitoring is conducted to establish the level to which employees are exposed. The Lab Standard requires that engineering or administrative controls be in place to protect chemists from excessive exposure to hazardous materials. For example, one of the most effective engineering controls is proper ventilation and fume hoods. If necessary, controls may extend to the temporary re-assignment of personnel outside the hazard area. In the case of absence or inefficiency of these controls, PPE must be used to protect the chemists; examples include respirators, special gloves, goggles, nonpermeable lab coats, *etc.*

The bibliography in Section 207 G lists government documents and sources, as well as other publications on safety.

207 E: RESPONSIBILITIES OF THE INDIVIDUAL CHEMIST

Every employee working in a laboratory must comply with the agency/company CHP. Each employee must be familiar with safety requirements for working in the laboratory and must adhere to them; each must handle or process all chemicals safely; and each must wear any safety gear and PPE needed to perform laboratory operations safely. Laboratory employees are ultimately responsible for:

1) identifying unsafe or unhealthy situations that exist in the laboratory and reporting such to a supervisor and to the person responsible for the safety program

2) complying with any safety standards applicable to the employee's job performance

3) developing an awareness of activities that may affect the safety of self, fellow workers, and the general public

4) reporting all accidents, injuries, unsafe incidents, or property damage that occur in the workplace

In order to identify unsafe conditions in the laboratory, employees must make hazard determinations for all materials handled there, based upon available scientific information and information found in MSDSs. The ultimate responsibility for a safe working environment rests with each employee.

207 F: ROLE OF TRAINING

OSHA stresses employee training in all their standards as the key to reducing hazards in the workplace. To be effective, laboratory safety training should be tailored to the specific needs of each laboratory. Situations often vary from one facility to the next, and even among the various laboratory operations within the same facility.

Supervisors are often the most knowledgeable about activities within each laboratory and are responsible for training all employees on proper work practices to safely perform laboratory tasks. Safety and health professionals can train laboratory personnel in selection of the proper PPE for each task, maintenance of such equipment, availability of employee services for prevention and treatment of exposures, procedures to follow in an emergency, and conditions to meet and procedures to follow to improve workplace safety and prevent environmental contamination. Employees must be advised about possible sources of exposure, what adverse health effects may result from exposure, what laboratory practices and engineering controls can reduce hazard and prevent contamination of the environment, availability of medical surveillance and environmental monitoring, and their specific responsibilities. An employee who is trained to recognize hazards, and who understands what work practices, PPE, and controls must be implemented, ensures a safer laboratory environment.

207 G: BIBLIOGRAPHY

Government or Organizational Materials

"Pocket Guide to Chemical Hazards," Department of Health and Human Services, National Institute of Occupational Safety and Health (NIOSH) Publication No. 90-117, U.S. Government Printing Office, Washington, DC

For other NIOSH documents, write to NIOSH, 4676 Columbia Parkway, Cincinnati, OH 45226, or call (513) 553-8287. For information on other occupational safety and health problems, call (800) 35-NIOSH.

For National Safety Council occupational safety and health data sheets on specific hazardous materials, call (708) 285-1121; for a list of these data sheets, call (800) 621-7619.

To purchase safety standards for laboratory operations, contact National Fire Protection Association (NFPA), Quincy, MA 02269, (800) 344-3555, or American National Standards Institute (ANSI), New York, NY, (212) 642-4900.

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208: HAZARDOUS WASTE DISPOSAL

All waste generated in pesticide residue analyses must be disposed of properly to comply with local, state, and Federal statutes. Because each locale is subject to different statutes, each laboratory must develop an individual program. Spent solvents and unused reagents generally constitute most laboratory wastes, but other materials that must be discarded may also be considered hazardous; examples of the latter include broken mercury thermometers, unused samples containing a listed or characteristic waste, and contaminated glassware.

It is beyond the purview of the PAM to provide complete directions for hazardous waste programs; the bibliography in Section 208 G is provided to offer guidance beyond the bare outline presented here. Each laboratory is encouraged to assign individuals as Hazardous Waste Managers and to provide appropriate training to those assigned. The complexities and responsibilities associated with hazardous waste management demand no less.

The following outline recommends an approach to developing a program suitable for proper handling and disposal of hazardous waste. In all cases, it must be supplemented by specific local statutes, be configured to the needs of the individual laboratory, and be managed by trained personnel.

208 A: IDENTIFICATION OF WASTE

The first step in developing a hazardous waste disposal program is to determine what wastes the laboratory discards and which of these are classified as hazardous. Under the Resource Conservation and Recovery Act (RCRA), the Environmental Protection Agency (EPA) establishes regulations for determination of hazards and publishes them in Code of Federal Regulations Title 40, Section 261 (40 CFR 261). Those regulations establish that a material is a hazardous waste if:

1) it is not specifically excluded under 40 CFR 261.4.

2) it is listed in 40 CFR 261.30 Subpart D.

3) it exhibits any of the characteristics of a hazardous waste.

4) it is part of a mixture that includes hazardous waste.

The following provides some additional information about these hazardous waste categories; consult 40 CFR 261 for full details.

Waste Specifically Excluded

Certain categories of waste are specifically excluded from being considered hazardous; examples include domestic sewage and household waste.

Chemicals Listed in 40 CFR 261.30 Subpart D

The following lists are published by EPA in 40 CFR 261.30 Subpart D to define those chemicals it classifies as hazardous waste:

F List: Hazardous wastes from nonspecific sources, *i.e.*, generically named wastes. Many F List solvents are used in pesticide analytical laboratories, *e.g.*, "spent halogenated solvents" like methylene chloride and "spent nonhalogenated solvents" like acetone and ethyl ether.

K List: Hazardous waste from specific sources, *e.g.*, "still bottoms from the distillation of benzyl chloride." This list does not contain any laboratory chemicals.

P List: Acute hazardous wastes, specifically named chemicals, *e.g.*, carbon disulfide and fluorine

U List: Commercial chemical products, specifically named chemicals, *e.g.*, acetone and chloroform

P and U Lists contain waste solvents and chemicals and include many solvents used in residue analyses. Any residue remaining in a container or in an inner lining removed from a container that previously held the listed waste is also classified as a hazardous waste, with certain exceptions. All regulated residues, plus any soil, water, or other debris from spill cleanups, are also treated as hazardous, as are any formulations in which a chemical from U or P Lists appears as the sole or active ingredient. The latter category includes primary or working analytical solutions. Some pesticides are on the P List; *e.g.*, aldicarb, aldrin, dieldrin, dinoseb, endrin, parathion, and toxaphene are included on the P List and thus classified as acutely hazardous waste.

Chemicals Exhibiting Hazardous Waste Characteristics

The following characteristics are sufficient to categorize a chemical as hazardous waste, according to 40 CFR 261.20 Subpart C:

Ignitability. EPA defines any solid, liquid, or gas as ignitable waste if:

1) it is a liquid, other than an aqueous solution containing <24% alcohol by volume, and has a flash point <60° C (140° F).

2) it is not a liquid and is capable, under standard temperature and pressure, of causing fire through friction, absorption of moisture, or spontaneous chemical changes, or, when ignited, burns so vigorously and persistently that it creates a hazard.

3) it is an ignitable compressed gas.

4) it is an oxidizer as defined in 49 CFR 173.151.

Corrosivity. A solid waste exhibits corrosivity if it is an aqueous waste with a pH <2 or ≥12.5. Nonaqueous wastes are subject to a steel corrosion test to determine corrosivity.

Reactivity. Solid wastes are considered reactive based on extreme instability and the tendency to react violently or explode; they are considered to pose a problem at all stages of the waste management process.

Toxicity. Hazardous waste is classified as having a "toxicity characteristic" if any of 40 specific contaminants can be extracted at levels greater than or equal to those specified in Table I, 40 CFR 261.24 (b), using an extraction procedure known as the Toxicity Characteristic Leaching Procedure (TCLP). Table I contains mostly pesticides, solvents, and heavy metals that EPA considers potentially leachable into soils or groundwater as a result of improper management. Items such as analytical samples, column packing, and extraction solvents are classified as TCLP waste if any material in Table I is extractable at the regulated levels of concentration. Examples of pesticides in Table I include 2,4-D, 2,4,5-T, chlordane, endrin, heptachlor, lindane, methoxychlor, silvex, and toxaphene.

Hazardous Waste Mixtures

In general, mixing hazardous waste with nonhazardous waste causes the entire volume to be regulated as hazardous waste. Similarly, mixing acutely hazardous waste with hazardous waste may cause the mixture to be regulated as acutely hazardous waste.

208 B: CATEGORIZATION OF WASTE GENERATOR

Once wastes are categorized, a determination must be made of what generator classification the facility meets. The following generator classifications, as defined by EPA, are based on quantity and categories of waste generated:

1) Large quantity generators, *i.e.*, facilities that generate >1000 kg hazardous waste or >1 kg acutely hazardous waste per month, or that accumulate ≥1000 kg hazardous waste on-site

2) Small quantity generators, *i.e.*, those facilities that produce >100 kg but <1000 kg hazardous waste or <1 kg acutely hazardous waste per month, or that accumulate <6000 kg hazardous waste or <1 kg acutely hazardous waste at any one time. Most pesticide analytical laboratories are classified by EPA as small quantity generators because they meet the first criterion. If the quantity of waste from the P List (acutely hazardous waste) is >1 kg per month, then the laboratory becomes a large quantity generator.

3) Conditionally exempt small quantity generators, *i.e.*, those that generate <100 kg hazardous waste or <1 kg acutely hazardous waste per month, or that accumulate <1000 kg hazardous waste or <1 kg acutely hazardous waste at any one time. Generator status varies from state to state; *i.e.*, each state may set its own threshold for generator status. It is therefore necessary to contact the state environmental agency to obtain copies of pertinent regulations.

208 C: OBTAINING APPROPRIATE ID NUMBERS

Once the waste generator category is established, the laboratory should contact the environmental agency of the state in which it is located to obtain both state and EPA ID numbers.

208 D: WASTE COLLECTION AND STORAGE PROCEDURES

Laboratory policy should be established to minimize the amount of waste generated. Where practical and safe, solvents should be recycled by distillation and chemicals should be shared with other laboratories. Analytical procedures should be miniaturized when possible to reduce amounts of solvent and chemicals required. To handle the waste that is generated, laboratory procedures for collection, segregation, and storage of different categories of waste must be established and rigorously enforced. Typical operations include:

1) Arrangement for locations and containers in the laboratory. Hazardous and nonhazardous wastes should be kept segregated during storage to avoid increasing the volume of waste considered hazardous. Waste chemicals from the P List should be separated from other hazardous waste to minimize the amount categorized as acutely toxic. Other segregation should be established for recyclable materials and for wastes with different modes of disposal.

2) Disposal of waste in drains. If local statutes permit, some water-soluble waste, *e.g.*, ethanol and methanol, may be poured down a drain. City pollution control and/or local sanitary sewer district must be contacted to determine local regulations.

3) Waste storage prior to disposal. Maximum storage times for waste are dependent on generator classification and distance between laboratory and waste disposal company. Small quantity generators are allowed to store hazardous waste for 180 days on-site without a permit. Generators accumulating waste in containers must comply with Subpart I of 40 CFR 265.170. All storage vessels must be in good condition and labeled "hazardous waste," and the accumulation start date must be marked on each container.

208 E: ARRANGEMENT FOR WASTE DISPOSAL

Numerous firms specialize in waste disposal. The regional EPA office and/or the state environmental office may provide useful information about local waste disposal firms. Some disposal firms provide consulting services that include preparation of manifests and proper labeling of shipping containers, but the waste generator is ultimately responsible for verifying accuracy of all labeling and shipping information. Department of Transportation shipping data and regulations are published in 49 CFR.

208 F: ADDITIONAL MANAGEMENT REQUIREMENTS

Accurate records must be kept of all waste collection and disposal activities, including manifests and biennial waste analysis and exception reports, when required.

All necessary safety procedures (Section 207) must be followed in handling any hazardous waste.

208 G: BIBLIOGRAPHY

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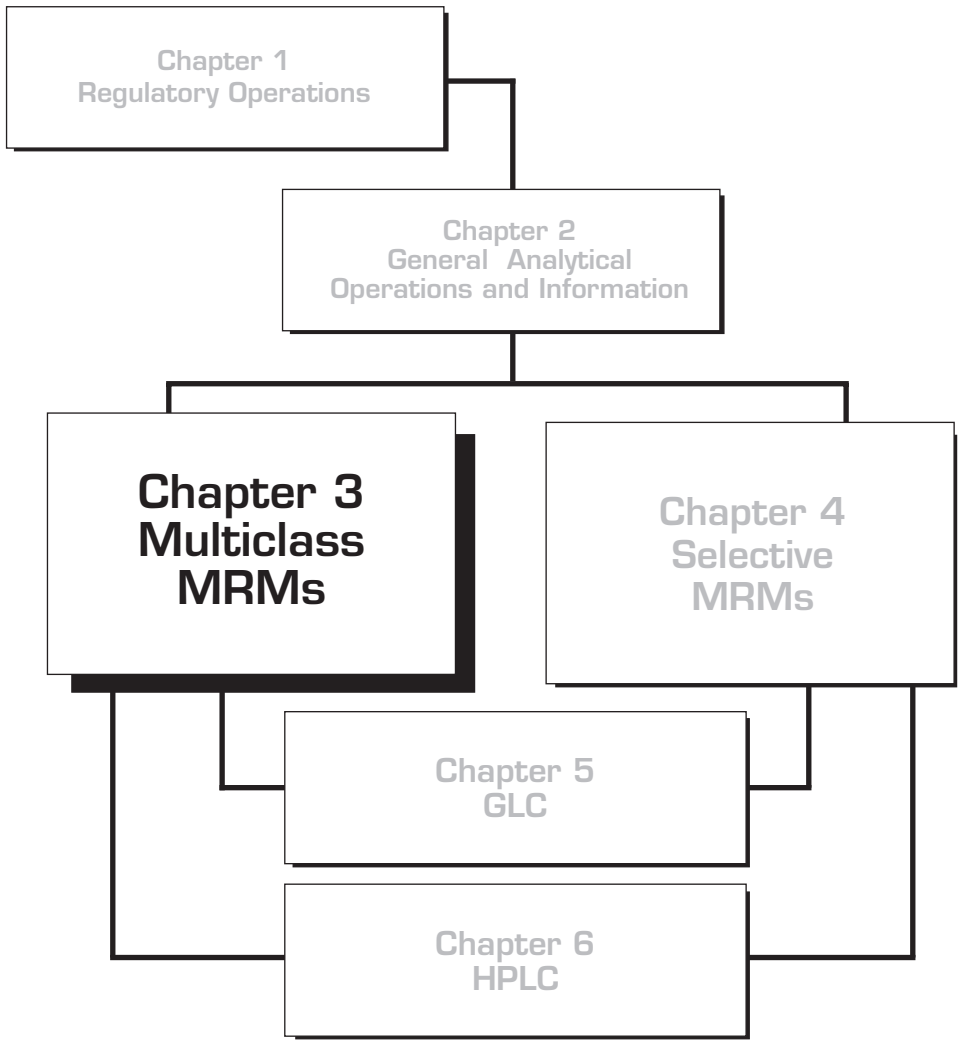

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301: MULTICLASS MRMS: CONCEPT AND APPLICATION

Pesticide multiresidue methods (MRMs) are capable of simultaneously determining more than one residue in a single analysis; this multiresidue capability is provided by a GLC or HPLC determinative step that separates residues from one another before detection. The MRM concept is raised to a higher dimension when a single extract is examined with more than one chromatographic determinative step, each providing coverage of residues in a different class, *e.g.*, chlorinated hydrocarbons, organophosphates, and carbamates. PAM I refers to these broad scope methods as "multiclass MRMs."

A multiclass MRM is potentially capable of determining any residue extracted by its extraction step; PAM I multiclass MRMs extract residues with organic solvents known to remove most nonionic residues from food commodities. Each determinative step in a multiclass MRM provides coverage for a particular group of residues in the extract, and each cleanup step is designed to purify the extract sufficiently to permit accurate determination. A multiclass MRM scheme can be expanded continually as new technologies are developed and adapted.

This introductory section presents a recommended approach to application of multiclass MRMs and background information with which any analyst using such methods should be familiar.

301 A: RECOMMENDED APPLICATION OF MULTICLASS MRMS

Whenever a sample of unknown pesticide treatment history is analyzed, and no residue(s) is targeted, a multiclass MRM should be used to provide the broadest coverage of potential residues; Figure 301-a displays the recommended multiclass MRM for each commodity category. The more detailed scheme provided with each method (Figures 302-a, 303-a, 304-a) directs the user to recommended module(s) for particular commodities. The user may choose as many or as few of these modules as time and resources permit; once residues are extracted, each determinative step extends coverage of the analysis to additional compounds.

Follow these directions to maximize coverage of residues without sacrificing quantitative accuracy:

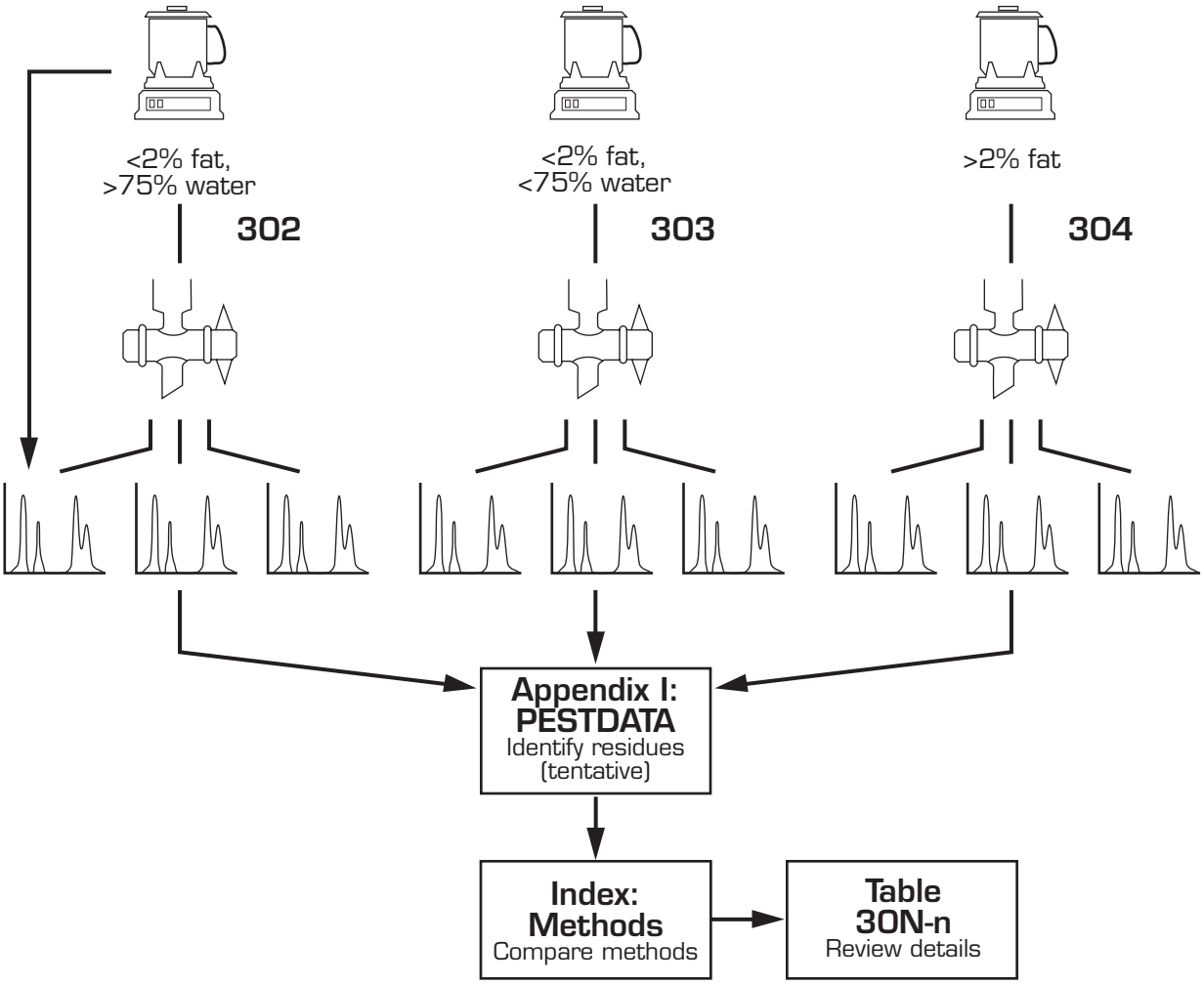
- For broadest coverage of potential residues, examine the uncleaned extract by determinative steps that are sufficiently selective to permit residue identification and quantitation in the presence of co-extractives.

- Following determination by selective determinative steps, clean up the extract as needed to permit additional determinations; these may include determinative steps designed for specific groups of residues (*e.g.*, from Chapter 4 methods).

- When a peak appears in the chromatogram of the extract, use the following PAM I tables to tentatively identify the residue and to choose the additional analyses needed for optimum identification, quantitation, and/or confirmation:

    1) PESTDATA (Appendix I). Compare GLC relative retention time (rrt) of the residue to lists of rrts on several common GLC columns

Figure 301-a
Recommended Approach to Analysis of Foods



for the first clue to residue identity. Review method behavior information for additional clues about which potential candidate(s) behaves in the same way as the residue. Inject solutions of appropriate reference standard(s) for comparison to the residue peak.

If retention times of residue and reference standard match, use PESTDATA information on the chemical's molecular formula and its rrts on other columns as a guide to selecting other determinations that will provide confirmatory evidence. If additional analyses are needed, choose appropriate other methods from PESTDATA listings of recoveries, Index to Methods, and tables related to specific methods, below.

PESTDATA rrts are for GLC systems only. Retention times from the HPLC determinative steps of Sections 401, 403, and 404 are included in the tables that accompany those methods. Use those tables to tentatively identify residues found.

2) Index to Methods. Use this summary as a guide to other method(s) available for a tentatively identified residue. Review method tables, below, for additional details.

3) Tables 302, 303, 304, 401, 402, 403, and 404. When a residue is tentatively identified, review method tables for details about special situations that may diminish recoveries, opportunities to improve recoveries, need for particular determinative step(s), *etc.* Decide what additional analyses are necessary based on this information.

- When tabulated information about behavior of the tentatively identified residue indicates that the method used provided only incomplete recovery, re-analyze the commodity with another method capable of complete recovery of the residue.

  (The analyst should be aware that all data in PAM I tables reflect the best information available but do not guarantee that results will be identical in every situation. Data have been collected for 30 years from many sources, including original method development studies, recovery studies by FDA laboratories, recovery studies by pesticide registrants and/or their contract laboratories, and collaborative and validation studies conducted under the auspices of AOAC International. Particular results may represent many analyses or only one, may have been performed with or without sample present, through complete methods or through individual procedures of a method, and with or without use of lauric acid adsorption value for Florisil column weight adjustment.)

- When the method used has not been previously validated for the residue/commodity combination, develop the necessary validation data.

Inherent in this approach to residue analysis is the acknowledgment that no multiclass MRM is quantitatively valid for all residues it is capable of detecting. Thus, re-analysis by other method(s) is required when a residue(s) is identified by a method known to be incapable of confirmatory identification and/or quantitative accuracy. Demonstration of method validity for any residue/commodity combination that is reported is the responsibility of the analyst using the method.

301 B: CAPABILITIES AND LIMITATIONS OF MRMS

Several aspects of an MRM influence its scope as a multiclass method: (1) thoroughness with which the extraction solvent and physical procedure are capable of extracting residues from the sample, (2) ability of subsequent cleanup techniques to remove sample co-extractives without removing residues, and (3) the number of different determinative steps used to examine the extract. During method development, a researcher evaluates each step of a method and makes choices based on optimum performance. Subsequent interlaboratory validation verifies that the method produces accurate results when performed as written.

This edition of PAM I presents MRMs as a series of modules, in recognition of standard practices in laboratories required to analyze many different commodities for many different potential residues. Modules presented within the same section in this manual were not necessarily developed at the same time or by the same researcher. Module combinations that have undergone interlaboratory validation are listed and recommended, but analysts may find it necessary to combine other modules to meet a particular need. Any such combination must be supported by data that validate its use in the situation. Steps of an MRM must be compatible with one another for the whole method to be applied in a valid manner.

This section provides background information to assist the analyst in making valid choices and avoiding potential pitfalls. Included here are discussions about the overall influence solvents have on method performance and important information about each category of method modules (extraction, cleanup, and determinative steps). Analysts combining method modules must be aware of the following concerns and take precautions to ensure that only appropriate combinations are used.

Influence of Solvents on Methodology

Choice of solvent(s) is among the most important decisions made by a researcher developing an analytical method. Analysts using these methods must also be aware of the following considerations related to solvents used in individual modules:

Availability of Pure Solvent. Solvent purity is essential to avoid potential interferences in the determinative step; impurities are usually concentrated during the evaporation steps included in most residue methods. Higher purity solvents invariably cost more, and it may be possible to use less expensive, lower purity materials if a solvent reagent blank examined by appropriate determinative step(s) (Section 204) supports their acceptability.

Detector Response to Solvent. GLC detectors used in residue determinations are usually selective for an element in the analyte molecule, so the final extract must not be dissolved in a solvent containing element(s) to which the detector(s) respond. For example, no trace of acetonitrile can be present when a nitrogen-selective detector is used, and no methylene chloride when a halogen-selective detector is used. HPLC detectors commonly used in residue determination preclude use of solvents that absorb UV light or fluoresce at the wavelength used during determination.

Solvents can adversely affect detectors in other ways, such as the deleterious but poorly defined effect acetonitrile has on electroconductivity detectors.

Experiences with such effects are usually noted in a method so use of particular solvents can be avoided.

Polarity. Increasing the polarity of an extraction solvent may improve a method's ability to extract particular residues, but it usually also increases the amount of co-extractives. The presence of polar solvents may also affect subsequent cleanup steps, so residues may need to be transferred to a different solvent before the next step of the method is performed.

Boiling Point. Solvents with a low boiling point are preferred, if evaporation to accommodate detector compatibility or appropriate polarity is necessary. In some cases, a solvent with a relatively high boiling point can be evaporated at a lower temperature if an azeotrope is first formed by addition of another solvent. Several types of evaporation apparatus exist (Section 202 C), and choice of which to use is often related to the boiling point of a particular solvent.

Toxicity. Solvents vary in toxicity, and laboratories should choose the least toxic among equivalent choices. Certain solvents (benzene, carbon tetrachloride) should no longer be used in residue analysis. Concentration and evaporation steps must be performed in an adequately ventilated hood, and other standard safety precautions must be followed (Section 207).

Extraction

The necessity of using water-miscible solvents to extract pesticide residues from high moisture products has long been established, as has the necessity of a "blending type" extraction process [1-4]. Acetone (Section 302), acetonitrile (Section 303), and methanol (Sections 401, 403) are used in PAM I multiclass and selective MRMs to extract nonionic residues from fruits and vegetables. Variations in polarity may affect the degree to which each can extract any particular residue [5-8].

Because extraction capabilities of these solvents are similar, other characteristics affect which solvent a developer chooses to use in a method. For example, developers of the method in Section 302 used acetone as extractant instead of acetonitrile (Section 303) because it is less toxic, has a lower boiling point (57° C *vs.* 82° C), does not affect detectors adversely, and does not form a two-phase system with water during analysis of fruit, as acetonitrile does [9].

Liquid-liquid partitioning of residues from initial extractant to nonaqueous solvent is a step common to most MRMs. Nature of the solvent(s) used in this step affects the degree of transfer of both residues and co-extractives. For example, in Section 302 E1, petroleum ether is included in the separator with aqueous acetone and methylene chloride to reduce the amount of polar plant constituents that partition into the organic phase. However, in a method variation targeted at the highly polar methamidophos, petroleum ether is replaced with acetone to improve partitioning of methamidophos from the aqueous to the organic layer [10].

Any MRM is applied with the understanding that certain residues are particularly difficult to extract, *e.g.*, the polar residue methamidophos, above. In such cases, notation of partial recovery is made in the table(s) of data that accompany the method description. Tentative identification of a residue known to be incompletely extracted by the method in use should then lead to re-analysis by another method or variation.

Certain commodities also present greater challenges to the extraction process, and methods may include special steps as an accommodation. Dry products are extracted with combinations of organic solvent and water to make up for the absence of water in the commodity itself. Several studies support the use of water/acetonitrile (Section 303 E3) for this purpose [11-13]. Water/acetone (Section 302 E4) is also used but has been found in some cases to extract less residue than water/acetonitrile [14, 15]; the two methods should be used to check one another when a residue has been identified that can be determined by both methods.

Extraction of residues from fatty products (*e.g.*, Section 304 E1-E5) has traditionally been aimed at nonpolar, lipophilic residues, which are readily extracted from the product when the fat itself is extracted. Currently, no method is available in this manual for quantitative determination of polar residues in fatty products.

Some residues absorbed from soil by plants, *e.g.*, dieldrin in potatoes, have been shown to be incompletely extracted by methods such as Section 303 [13]; other root-absorbed residues (*e.g.*, dieldrin and DDT in carrots) have been extracted completely by the same procedure [16, 17]. Laboratories analyzing root crops must be aware that the method may not be extracting all the residue present. Other, more exhaustive processes, such as use of a Soxhlet extractor [18], may be necessary if the residue or commodity warrants.

Cleanup

Cleanup steps are designed to purify extracts to permit more definitive identification of residues at lower limits of quantitation, and to minimize adverse effects on determinative step instrumentation. However, almost all cleanup steps adsorb, destroy, or otherwise remove at least some residues from the extract. Thus, cleanup may reduce the number of detectable residues in the final extract.

Schemes for multiclass MRMs attempt to determine as many residues as possible by examining uncleaned extracts with selective detectors, *e.g.*, flame photometric and electrolytic conductivity (GLC) and fluorescence (HPLC). Cleanup can subsequently be performed on the extract to permit determination with less selective detectors, *e.g.*, electron capture (GLC) or UV (HPLC). Use of several cleanup steps, each on a separate aliquot of extract, permits examination of each aliquot with a different determinative step. This approach provides coverage for the maximum number of residues, excluding only those not recovered from any cleanup step and also not determined by initial selective detectors.

Residues can often be detected but not reliably quantitated in an uncleaned extract; quantitation may be possible once the extract is cleaned up using a technique known to recover the particular residue. Other residues can be quantitatively measured only by re-analysis with a different extraction step. Tables of recovery data for each method provide the analyst with information to guide the choice of an appropriate cleanup technique or alternative method.

Many cleanup steps involve chromatography of the extract solution on a column or cartridge. Choices of the column/cartridge material and eluting solvent(s) dictate what chemicals can be recovered; *e.g.*, columns of the adsorbent Florisil provide suitable cleanup of relatively nonpolar residues (Sections 302 C1, 303 C1, *etc.*). Increasing the polarity of the eluant permits recovery of more polar residues but decreases the degree of cleanup, because more co-extractives are also eluted.

Very polar residues usually cannot be eluted from Florisil no matter how polar an eluant is used. Instead, charcoal columns are often used for cleaning up extracts containing polar residues, *e.g.*, Sections 302 C2 and C3.

The nature of the solvent in which the extract is dissolved when placed on a cleanup column may affect which residues elute from the column. Recovery data associated with a method are valid only when the extract is in the specified solvent. When combining method modules, the extract added to a cleanup column may be in a solvent different from that originally specified; in such cases, recovery data may not be applicable. To make use of existing tables of data related to chemicals recovered through a method, it may be necessary to change the extract solvent by evaporation or azeotroping.

Determinative Steps

Use of minimal cleanup in an MRM reduces analysis time and reagent costs, but it can jeopardize determinative step reliability by introducing co-extractives that interfere with the determination or cause physical damage to the system. Presence of materials to which the detector responds can cause (1) false reports of residues not actually present, (2) inaccurate quantitation of residues, or (3) complete masking of residues. Risk of chromatographic degradation is increased by repetitive injection of an uncleaned extract.

The analyst using methods from PAM I is responsible for ensuring that extract injected into any determinative step system does not contain potential interferences or materials that adversely affect chromatographic performance. Sections 501 C and 601 E provide recommendations related to determinations with GLC and HPLC systems, respectively. Analytical accuracy and minimal disruption of laboratory operations will result if reasonable use of cleanup steps and regular maintenance of instruments are both employed.

In certain cases, special precautions are needed to detect particular residues. For example, thiometon is known to break down while standing in the extract solution of Section 302 E1; examination of the extract soon after its preparation permits determination of thiometon residues that would not otherwise be detectable. Notes are included in the method tables of data to provide such advice.

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[18] Mumma, R.O., *et al.* (1966) *Science* 152, 530-531

302: METHOD I FOR NONFATTY FOODS

BASIC REFERENCES

Luke, M.A., *et al.* (1975) *J. Assoc. Off. Anal. Chem.* **58**, 1020-1026

Luke, M.A., *et al.* (1981) *J. Assoc. Off. Anal. Chem.* **64**, 1187-1195

GENERAL PRINCIPLES

Residues are extracted from nonfatty foods by blending with acetone or water/acetone, then transferred from the filtered aqueous extract into organic solvent. The extract is cleaned up if necessary and examined by various determinative steps; the amount of cleanup necessary is dictated by the determinative step(s) to be used and by the type of commodity being analyzed.

APPLICABILITY

Consult Guide to PAM I for additional information pertinent to the appropriate application of multiresidue methodology.

Method is applicable to nonionic residues in nonfatty foods. Cleanup steps may be needed for particularly dirty extracts or for examination by less selective detectors; some residues may be lost during cleanup. Extract is amenable to examination by many determinative steps, and the residues covered by a particular analysis are dependent on the number of different determinative steps used. See Tables 302-a and 302-b, following the method description, for results of recovery tests.

METHOD MODULES

Choose from these method modules, using Figure 302-a for guidance:

Extraction (E)			Recommended Use	
E1	(p. 302-7)	Extraction with acetone, liquid-liquid partitioning with petroleum ether/methylene chloride	nonfatty, high moisture commodities	
E2	(p. 302-9)	Extraction with acetone, removal of water with 40 g Hydromatrix	nonfatty, high moisture commodities	
E3	(p. 302-11)	Extraction with acetone, removal of water with 25 g Hydromatrix	alternative to E2 for reduction in solvent use	
E4	(p. 302-13)	Extraction with water/acetone, liquid-liquid partitioning with petroleum ether/methylene chloride	nonfatty, low moisture commodities	
E5	(p. 302-15)	Extraction with acetone, liquid-liquid partitioning with acetone/methylene chloride	alternative to E1 for relatively polar residues	◀
E6	(p. 302-16)	Extraction with water/acetone, liquid-liquid partitioning with acetone/methylene chloride	alternative to E4 for relatively polar residues	◀
E7	(p. 302-17)	Extraction with acetone and solid phase extraction cartridges, liquid-liquid partitioning	nonfatty, high moisture commodities for relatively polar residues	◀

Cleanup (C)

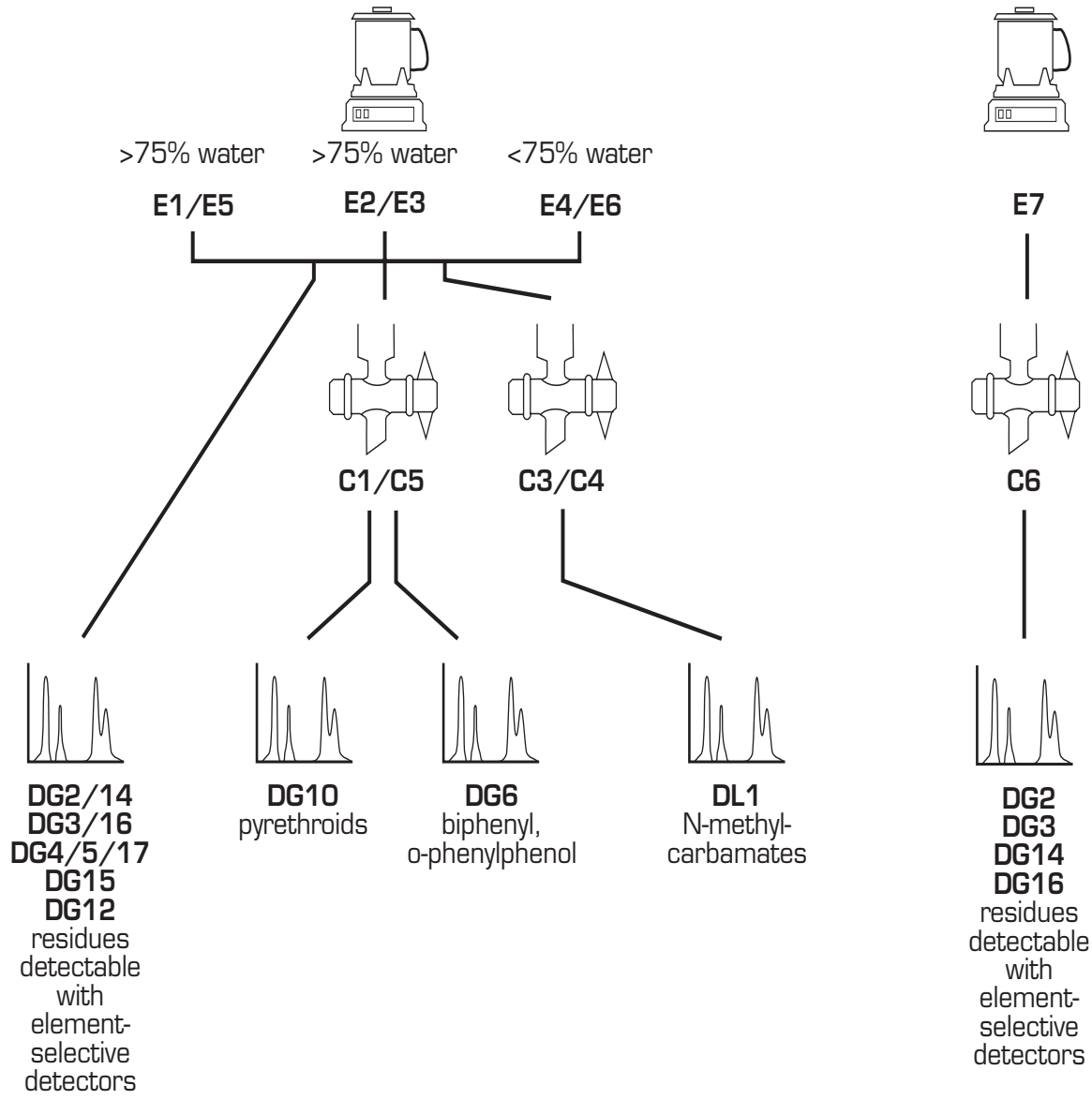
C1	(p. 302-21)	Florisil column (4 g) cleanup, with one methylene chloride eluant	relatively nonpolar residues
C2	(p. 302-23)	Charcoal/Celite/magnesium oxide column cleanup	polar residues
C3	(p. 302-25)	Charcoal/silanized Celite column cleanup	before HPLC determination for N-methylcarbamates
C4	(p. 302-27)	C-18 cartridge cleanup	before HPLC determination for N-methylcarbamates
C5	(p. 302-29)	Florisil column cleanup, with mixed ether eluants	relatively nonpolar residues
▶ **C6**	(p. 302-31)	SAX/PSA cartridge cleanup	polar and nonpolar residues

Determination (D)		**Recommended Use**	
DG 1	(p. 302-33)	GLC, 100% methyl siloxane column, 200°, EC detector	residues with halogen. sulfur, other moities
DG 2	(p. 302-35)	GLC, 100% methyl siloxane column, 200°, FPD-P	residues with phosphorus
DG 3	(p. 302-37)	GLC, 100% methyl siloxane column, 200°, ELCD-X	residues with halogen
DG 4	(p. 302-39)	GLC, 100% methyl siloxane column, 200°, ELCD-N	residues with nitrogen
DG 5	(p. 302-41)	GLC, 100% methyl siloxane column, 200°, N/P detector	residues with nitrogen or phosphorus
DG 6	(p. 302-43)	GLC, 100% methyl siloxane column, 160°, FID	biphenyl, o-phenylphenol
DG 7	(p. 302-45)	GLC, 100% methyl siloxane column, 130°, EC detector	early eluting residues with halogen, sulfur, other moieties
DG 8	(p. 302-47)	GLC, 100% methyl siloxane column, 130°, FPD-P	early eluting residues with phosphorus
DG 9	(p. 302-49)	GLC, 100% methyl siloxane column, 130°, ELCD-X	early eluting residues with halogen
DG10	(p. 302-51)	GLC, 100% methyl siloxane column, 230°, EC detector other moieties	late eluting residues with halogen, sulfur,
DG11	(p. 302-53)	GLC, 100% methyl siloxane column, 230°, FPD-P	late eluting residues with phosphorus
DG12	(p. 302-55)	GLC, 100% methyl siloxane column, 230°, ELCD-X	late eluting residues with halogen
DG13	(p. 302-57)	GLC, 50% phenyl, 50% methyl siloxane column, 200°, EC detector	residues with halogen, sulfur, other moieties

DG14 (p. 302-59)	GLC, 50% phenyl, 50% methyl siloxane column, 200°, FPD-P	residues with phosphorus
DG15 (p. 302-61)	GLC, 50% phenyl, 50% methyl siloxane column, 200°, FPD-S	residues with sulfur
DG16 (p. 302-63)	GLC, 50% phenyl, 50% methyl siloxane column, 200°, ELCD-X	residues with halogen
DG17 (p. 302-65)	GLC, 50% phenyl, 50% methyl siloxane column, 200°, N/P detector	residues with nitrogen or phosphorus
DG18 (p. 302-67)	GLC, 50% cyanopropylphenyl, 50% methyl siloxane column, 200°, EC detector	residues with halogen, sulfur, other moieties
DG19 (p. 302-69)	GLC, 50% cyanopropylphenyl, 50% methyl siloxane column, 200°, FPD-P	residues with phosphorus

►

Figure 302
Recommended Approach: Nonfatty Foods



VALIDATION

Many combinations of method modules are possible. The following combinations have undergone interlaboratory validation and are recommended for use:

E1 + DG2, DG3

Validation report:

> Sawyer, L.D. (1985) *J. Assoc. Off. Anal. Chem.* **68**, 64-71. Collaborative study lead-ing to AOAC official final action status for acephate, a-BHC, chlorpyrifos, dieldrin, monocrotophos, and omethoate in lettuce, strawberries, and tomatoes.
>
> AOAC official method reference: *Official Methods of Analysis of the AOAC* (1990) 15th ed., 985.22.

E1 + C3 + DL1

Validation report:

> Pardue, J.R. (April 1987) "Recoveries of N-Methyl Carbamates Using a Combination of the Luke (PAM I, 232.4) and Krause (PAM I, 242.24b, 242.25) Procedures," LIB 3138, FDA, Rockville, MD

E2 + C1 + [temperature programmed GLC systems equivalent to] DG1, DG7, DG10, and DG16

Validation report:

> Griffitt, K.R., and Szorik, M.M. (Sept 1989) "The Analysis of 127 Total Diet Items for Chlorinated Residues Using Luke/Solid Phase Extracts," LIB 3366, FDA, Rockville, MD

E1 EXTRACTION WITH ACETONE, LIQUID-LIQUID PARTITIONING WITH PETROLEUM ETHER/METHYLENE CHLORIDE

References

Luke, M.A., *et al.* (1975) *J. Assoc. Off. Anal. Chem.* **58**, 1020-1026

Luke, M.A., *et al.* (1981) *J. Assoc. Off. Anal. Chem.* **64**, 1187-1195

Principles

Nonfatty sample is blended with acetone and filtered. Most nonionic residues are extracted into aqueous acetone solution. Residues are transferred from aqueous acetone to methylene chloride/petroleum ether by partitioning, with salt added to aqueous layer after the first partitioning to aid transfer. Concentration step is repeated in the presence of petroleum ether to remove all traces of methylene chloride, then repeated again to produce final extract in acetone solution.

Apparatus

blender, high speed; explosion-proof Waring Blendor, 1 qt jar

Büchner funnel (Büchner), porcelain, 12 cm diameter

filter paper, Shark Skin®, to fit Büchner

long-stemmed funnel, glass, 4" diameter

Kuderna-Danish concentrator (K-D), 500 mL, with Snyder column, two-ball micro-Snyder column, graduated receiving flask

separatory funnel (separator), 1 L

Reagents

acetone, distilled from all-glass apparatus

boiling chips, 20-30 mesh carborundum

glass wool, Pyrex, see Section 204 for handling directions

methylene chloride, distilled from all-glass apparatus

petroleum ether, distilled from all-glass apparatus

sodium chloride, reagent grade

sodium sulfate, anhydrous, granular, reagent grade; see Section 204 for handling directions

Directions

- Prewash filter paper with acetone to remove contaminants.
- Weigh 100 g chopped or blended sample into blender jar, add 200 mL acetone, and blend 2 min at high speed.
- Filter with suction through 12 cm Büchner fitted with Shark Skin® paper; collect extract in 500 mL suction flask. Filtration is normally complete in <1 min. Continuation of vacuum for excessive period can reduce volume of extract and cause error in calculation.
- Place 80 mL sample extract in 1 L separator, and add 100 mL petroleum ether and 100 mL methylene chloride. Shake vigorously 1 min.
- Transfer lower aqueous layer to second 1 L separator.

- Dry upper layer of first separator by passing through about 1.5" sodium sulfate supported on washed glass wool in 4" funnel, collecting in K-D. (If extract will be cleaned up directly with C3, charcoal/Celite column, collect in vacuum rotary evaporator flask.)
- To separator with aqueous phase, add 7 g sodium chloride and shake vigorously 30 sec until most of the sodium chloride is dissolved.
- Add 100 mL methylene chloride, shake 1 min, and dry lower organic phase through same sodium sulfate.
- Extract aqueous phase with additional 100 mL methylene chloride and dry as above. Rinse sodium sulfate with about 50 mL methylene chloride.

  (If extract will be cleaned up directly with C3, proceed to concentration step described there instead of evaporating in K-D as follows.)
- Add boiling chips to K-D and concentrate solvent in K-D; start evaporation slowly by placing only receiver tube into steam. After 100-150 mL has evaporated, concentrator may be exposed to more steam. When liquid level in hot concentrator tube is about 2 mL, add 100 mL petroleum ether through Snyder column and reconcentrate to about 2 mL. Add 50 mL petroleum ether and repeat concentration step. Add 20 mL acetone, and reconcentrate to about 2 mL. Do not allow solution to go to dryness during any of the concentration steps. Adjust volume of extract to suitable definite volume with acetone.
- Calculate equivalent sample weight in final solution:

$$\frac{\text{mg sample equivalent}}{\mu\text{L final extract}} = 100 \times \frac{80}{200 + W - 10} \times \frac{1}{\text{mL final volume}}$$

where:

100 = g sample analyzed

80 = mL filtered extract taken for liquid-liquid partitioning

200 = mL acetone blended with 100 g sample

W = amount (mL) of water present in sample (Section 201; if data are not available for particular raw agricultural commodity, use 85%)

10 = adjustment for water/acetone volume contraction.

Thus, when sample contains 85% water (85 mL/100 g) and final extract volume is 7 mL, each µL contains:

$$100 \times \frac{80}{200 + 85 - 10} \times \frac{1}{7} = \frac{4.15 \text{ mg sample equivalent}}{\mu\text{L final extract}}$$

- Extract may be suitable, as is, for determination by GLC with selective detectors (*e.g.*, DG2, DG3). If co-extractives interfere with determination or adversely affect chromatography, clean up extract with C1, C2, or C5 prior to determination.
- Clean up extract with C1 or C5 prior to determination by electron capture (DG1, DG7, *etc.*) or flame ionization detectors (DG6). Clean up extract with C3 or C4 prior to determination by DL1 for N-methylcarbamates.

E2 EXTRACTION WITH ACETONE, REMOVAL OF WATER WITH 40 G HYDROMATRIX

References

Luke, M.A., *et al.* (1975) *J. Assoc. Off. Anal. Chem.* **58**, 1020-1026

Luke, M.A., *et al.* (1981) *J. Assoc. Off. Anal. Chem.* **64**, 1187-1195

Hopper, M.L. (1988) *J. Assoc. Off. Anal. Chem.* **71**, 731-734

Principles

Nonfatty sample is blended with acetone and filtered. Most nonionic residues are extracted from nonfatty foods into aqueous acetone solution. Water is removed from aqueous acetone solution by passing it through a column of specially treated diatomaceous earth (Hydromatrix). Residues are eluted from column with methylene chloride. Up to 13.3 mL water, from 40 mL aqueous acetone extractant, is adsorbed by the column, which is re-usable.

Apparatus

blender, high speed; explosion-proof Waring Blendor, 1 qt jar

Büchner funnel (Büchner), porcelain, 12 cm diameter

filter paper, Shark Skin®, to fit Büchner

chromatographic column, 25 mm id × 500 mm, Teflon stopcock

long-stemmed funnel, glass, 4" diameter

powder funnel, glass, 4" diameter

Kuderna-Danish concentrator (K-D), 500 mL, with Snyder column, two-ball micro-Snyder column, graduated receiving flask

sieve, No. 30

Reagents

acetone, distilled from all-glass apparatus

buffer solution: 0.1 M (13.6 g/L) potassium phosphate monobasic (KH_2PO_4) in water

Hydromatrix material (pelletized diatomaceous earth), Part No. 0019-8003, Analytichem International, Harbor City, CA; also available through Varian

methylene chloride, distilled from all-glass apparatus

potassium phosphate monobasic, certified ACS grade

wire gauze, 40 mesh stainless steel

Directions

- Prepare Hydromatrix column:
  - Cut two pieces stainless steel gauze into circles of diameter slightly larger than chromatographic column id. Place one circle in bottom of column.
  - Place 50 g Hydromatrix material on No. 30 sieve and sieve thoroughly to remove fines.

  - Pour 40 g sieved Hydromatrix material into column with aid of powder funnel. Tap end of column lightly on benchtop to settle material. Place second stainless steel gauze circle on top of material in column.
  - With stopcock fully open, wash column with 150 mL buffer solution.
  - After buffer solution has passed into column and flow has slowed to 3-5 mL/min, wash column with 300 mL acetone. Adjust flow to 50-60 mL/min after first 100 mL acetone has eluted.
  - Wash column with 300 mL methylene chloride. Re-adjust flow to 50-60 mL/min after first 100 mL methylene chloride has eluted.

- Prewash filter paper with acetone to remove artifacts.
- Weigh 100 g chopped or blended sample into blender jar, add 200 mL acetone, and blend 2 min at high speed.
- Filter with suction through 12 cm Büchner fitted with Shark Skin® paper; collect extract in 500 mL suction flask. Filtration is normally complete in <1 min. Continuation of vacuum for excessive period can reduce volume of extract and cause error in calculation.
- Prewash Hydromatrix column with 200 mL acetone followed by 200 mL methylene chloride immediately before each use. Discard wash solvents.
- Place K-D under column. (If extract will be cleaned up directly with C3, charcoal/Celite column, collect in vacuum rotary evaporator flask.) Trans fer 40 mL filtered acetone extract to top of column. Let extract pass into column until flow rate has slowed to <1 mL/min. Let column equilibrate 3 min at <1 mL/min.
- Add 50 mL methylene chloride to column. After that has passed into column, add another 50 mL methylene chloride. After that has passed into column, add another 200 mL methylene chloride.
- Collect eluate until flow rate has decreased to slow drip (about 1 mL/min). Total elution time is 6-8 min.

  (If extract will be cleaned up directly with C3, proceed to concentration step described there instead of evaporating in K-D as follows.)
- Add boiling chips to K-D and concentrate solvent in K-D; start evaporation slowly by placing only receiver tube into steam. After 100-150 mL has evaporated, concentrator may be exposed to more steam. When liquid level in hot concentrator tube is about 2 mL, add 100 mL petroleum ether through Snyder column and reconcentrate to about 2 mL. Add 50 mL petroleum ether and repeat concentration step. Add 20 mL acetone, and reconcentrate to about 2 mL. Do not allow solution to go to dryness during any of the concentration steps. Adjust volume of extract to suitable definite volume with acetone.
- If extract will be cleaned up directly with C1, Florisil column, it is not necessary to reconcentrate repeatedly (as above) to remove all traces of methylene chloride. Instead, add boiling chips and concentrate solvent in K-D to <5 mL. Without allowing K-D to cool, add 50 mL acetone through Snyder column, and reconcentrate to suitable definite volume; allow to cool.

- Calculate equivalent sample weight in final solution:

$$\frac{\text{mg sample equivalent}}{\mu\text{L final extract}} = 100 \times \frac{40}{200 + W - 10} \times \frac{1}{\text{mL final volume}}$$

where:

100 = g sample analyzed

40 = mL filtered extract taken for Hydromatrix partitioning

200 = mL acetone blended with 100 g sample

W = amount (mL) of water present in sample (Section 201; if data are not available for particular raw agricultural commodity, use 85%)

10 = adjustment for water/acetone volume contraction.

Thus, when sample contains 85% water (85 mL/100 g) and final extract volume is 5 mL, each µL contains:

$$100 \times \frac{40}{200 + 85 - 10} \times \frac{1}{5} = \frac{2.9 \text{ mg sample equivalent}}{\mu\text{L final extract}}$$

- Extract may be suitable, as is, for determination by GLC with selective detectors (*e.g.*, DG2, DG3). If co-extractives interfere with determination or adversely affect chromatography, clean up extract with C1, C2, or C5 prior to determination.
- Clean up extract with C1 or C5 prior to determination by electron capture (DG1, DG7, *etc.*) or flame ionization detectors (DG6). Clean up extract with C3 or C4 prior to determination by DL1 for N-methyl-carbamates.
- Re-use Hydromatrix column without further rinsing, unless any adsorbed color elutes from column (after about 20 uses). When this occurs, restore column as follows:
  - Do not change stopcock setting. Flow rate will change due to different solvent densities, but this is of no consequence.
  - Wash column with 200 mL acetone, followed by sufficient volume (200-300 mL) buffer solution to remove any color left on column. Once color has been removed, elute with 300 mL acetone followed by 200 mL methylene chloride. Column is now ready for re-use.

ALTERNATIVE:

E3 EXTRACTION WITH ACETONE, REMOVAL OF WATER WITH 25 G HYDROMATRIX

Reference

Palmer, R.E., and Hopper, M.L. (Nov. 1991) "Miniaturized Solid Phase Partition Column for Determination of Organochlorine and Organophosphate Pesticides with PAM I 232.4 (Luke procedure) Acetone Filtrate," LIB 3613, FDA, Rockville, MD

Principles

Smaller size column of Hydromatrix reduces solvent use by 40% over E2, while still removing water from same amount of extract. However, solution eluting from 25 g Hydromatrix column may be cloudy, probably from a small amount of water; this disappears during concentration. The 25 g column may also have a shorter lifetime than the 40 g column. Results using the 25 g column may be somewhat less reliable for certain chemicals; *e.g.*, p,p'-dicofol and dicloran are recovered less reproducibly, and >0.4 ppm methamidophos may be only partially recovered; elution with 300 mL methylene chloride permits complete recovery of the latter.

Directions

- Follow directions of E2, except:
  - Prepare Hydromatrix column from 25 g material instead of 40 g.
  - Prewash Hydromatrix column with 100 mL acetone followed by 100 mL methylene chloride immediately before each use.
  - After transferring 40 mL filtered acetone extract to top of column, elute with 25, 25, and 150 mL methylene chloride, instead of volumes used in E2.
  - Because amount of original sample and amount of filtered acetone extract transferred to Hydromatrix column are the same as in E2, mg sample equivalent is the same as E2.

E4 EXTRACTION WITH WATER/ACETONE, LIQUID-LIQUID PARTITIONING WITH PETROLEUM ETHER/METHYLENE CHLORIDE

Reference

Luke, M.A., and Doose, G.M. (1983) *Bull. Environ. Contam. Toxicol.* **30**, 110-116

Principles

Low moisture nonfatty sample is blended with 35% water/acetone and filtered; the presence of water in the extractant facilitates extraction of residues from the dry product and dilutes co-extractives. Most nonionic residues are extracted into aqueous acetone solution. Residues are transferred from aqueous acetone to organic solvent methylene chloride/petroleum ether by partitioning, with salt added to the aqueous layer after the first partitioning to aid transfer.

Apparatus

blender, high speed; explosion-proof Waring Blendor, 1 qt jar

Büchner funnel (Büchner), porcelain, 12 cm diameter

filter paper, Shark Skin®, to fit Büchner

long-stemmed funnel, glass, 4" diameter

grinder, suitable for reducing dry products to <20 mesh

Kuderna-Danish concentrator (K-D), 500 mL, with Snyder column, two-ball micro-Snyder column, graduated receiving flask

separatory funnel (separator), 1 L

Reagents

acetone, distilled from all-glass apparatus

boiling chips, 20-30 mesh carborundum (optional)

glass wool, Pyrex; see Section 204 for handling directions

methylene chloride, distilled from all-glass apparatus

petroleum ether, distilled from all-glass apparatus

sodium chloride, reagent grade

sodium sulfate, anhydrous, granular, reagent grade; see Section 204 for handling directions

35% (v/v) water/acetone

Directions

- Prewash filter paper with acetone to remove artifacts.
- Grind sample containing <10% fat or oil to <20 mesh.
- Weigh 15 g ground sample into blender jar, add 350 mL 35% water/acetone, and blend 2 min at high speed.
- Filter with suction through 12 cm Büchner fitted with Shark Skin® paper; collect extract in 500 mL suction flask. Filtration is normally complete in <1 min. Continuation of vacuum for excessive period can reduce volume of extract and cause error in calculation.
- Place 80 mL sample extract in 1 L separator containing 100 mL methylene chloride. Add 100 mL petroleum ether and shake vigorously 1 min.

- Transfer lower aqueous layer to second 1 L separator.
- Dry upper organic layer of first separator by passing through about 1.5" sodium sulfate supported on washed glass wool in 4" funnel, collecting in K-D. (If extract will be cleaned up directly with C3, charcoal/Celite column, collect in vacuum rotary evaporator flask.)
- To separator with aqueous phase, add 7 g sodium chloride and shake vigorously 30 sec until most of the sodium chloride is dissolved.
- Add 100 mL methylene chloride, shake 1 min, and dry lower organic phase through same sodium sulfate.
- Extract aqueous phase with additional 100 mL methylene chloride and dry as above. Rinse sodium sulfate with about 50 mL methylene chloride.

  (If extract will be cleaned up directly with C3, proceed to concentration step described there instead of evaporating in K-D as follows.)
- Add boiling chips to K-D and concentrate solvent in K-D; start evaporation slowly by placing only receiver tube into steam. After 100-150 mL has evaporated, concentrator may be exposed to more steam. When liquid level in hot concentrator tube is about 2 mL, add 100 mL petroleum ether through Snyder column and reconcentrate to about 2 mL. Add 50 mL petroleum ether and repeat concentration step. Add 20 mL acetone, and reconcentrate to about 2 mL. Do not allow solution to go to dryness during any of the concentration steps. Adjust volume of extract to suitable definite volume with acetone.
- Calculate equivalent sample weight in final solution:

$$\frac{\text{mg sample equivalent}}{\mu\text{L final extract}} = 15 \times \frac{80}{350} \times \frac{1}{\text{mL final volume}}$$

  where:

  15 = g sample analyzed

  80 = mL filtered extract taken for liquid-liquid partitioning

  Thus, when final extract volume is 2 mL, each µL contains:

$$15 \times \frac{80}{350} \times \frac{1}{2} = \frac{1.7 \text{ mg sample equivalent}}{\mu\text{L final extract}}$$

- Extract may be suitable, as is, for determination by GLC with selective detectors (*e.g.*, DG2, DG3). If co-extractives interfere with determination or adversely affect chromatography, clean up extract with C1, C2, or C5 prior to determination.
- Clean up extract with C1 or C5 prior to determination by electron capture (DG1, DG7, *etc.*) or flame ionization detectors (DG6). Clean up extract with C3 or C4 prior to determination by DL1 for N-methylcarbamates.

ALTERNATIVE: ◀

E5 EXTRACTION WITH ACETONE, LIQUID-LIQUID PARTITIONING WITH ACETONE/METHYLENE CHLORIDE

Reference

Luke, M. A., and Doose, G. M. (1983) *Bull. Environ. Contam. Toxicol.* **30**, 110-116

Principle

Polar pesticides such as methamidophos exhibit variable recoveries when petroleum ether/dichloromethane is used in partitioning. Better recoveries are obtained when acetone is substituted for petroleum ether. Transfer of polar pesticides from the aqueous phase to the organic layer is further facilitated by adding sodium chloride before, rather than after, the first partitioning step.

Directions

- Follow directions of E1 through blending and filtering. Then:
  - Place 80 mL sample extract in 1 L separator, and add 100 mL acetone, 100 mL methylene chloride, and 7 g sodium chloride. Shake vigorously 1 min.
  - Transfer lower aqueous layer to second 1 L separator.
  - Dry upper organic layer of first separator by passing through about 1.5" sodium sulfate supported on washed glass wool in 4" funnel, collecting in K-D. (If extract will be cleaned up directly with C3, charcoal/ silanized Celite column, collect in vacuum rotary evaporator flask.)
  - Add 100 mL methylene chloride, shake 1 min, and dry lower organic phase through same sodium sulfate.
- Continue as in E1, "Extract aqueous phase with additional 100 mL methylene chloride..."

ALTERNATIVE: ◀

E6 EXTRACTION WITH WATER/ACETONE, LIQUID-LIQUID PARTITIONING WITH ACETONE/METHYLENE CHLORIDE

Reference

Luke, M. A., and Doose, G. M. (1983) *Bull. Environ. Contam. Toxicol.* **30**, 110-116

Principle

Polar pesticides such as methamidophos exhibit variable recoveries when petroleum ether/methylene chloride is used in partitioning. Better recoveries are obtained when acetone is substituted for petroleum ether. Transfer of polar pesticides from the aqueous phase to the organic layer is further facilitated by adding sodium chloride before, rather than after, the first partitioning step.

Directions

- Follow directions of E4 through blending and filtering. Then:
  - Place 80 mL sample extract in 1 L separator containing 100 mL methylene chloride. Add 100 mL acetone and 7 g sodium chloride and shake vigorously 1 min.
  - Transfer lower aqueous layer to second 1 L separator.
  - Dry upper organic layer of first separator by passing through about 1.5" sodium sulfate supported on washed glass wool in 4" funnel, collecting in K-D. (If extract will be cleaned up directly with C3, charcoal/silanized Celite column, collect in vacuum rotary evaporator flask.)
  - Add 100 mL methylene chloride, shake 1 min, and dry lower organic phase through same sodium sulfate.
- Continue as in E4, "Extract aqueous phase with additional 100 mL methyl ene chloride..."

E7 EXTRACTION WITH ACETONE AND SOLID PHASE EXTRACTION CARTRIDGES, LIQUID-LIQUID PARTITIONING

Reference

Luke, M. A., *et al.* (Sept. 1994) "An Improved Variation of the Luke Multiresidue Pesticide Procedure for the Analysis of Fruits and Vegetables Using Solid Phase Extraction Cartridges and Element Selective Gas Chromatographic Detectors," LIB 3896, FDA, Rockville, MD

Apparatus

blender, high speed; explosion-proof Waring Blendor, 1 qt jar

Büchner funnel (Büchner), porcelain, 12 cm diameter

filter paper, Shark Skin®, to fit Büchner

500 mL suction flask

long-stemmed funnel, glass, 4" diameter

Kuderna-Danish concentrator (K-D), 500 mL, with Snyder column, two-ball micro-Snyder column, graduated receiving flask

separatory funnel (separator), 1 L

75 mL Bond Elut reservoir or equivalent

25 mm syringe filter, 0.45 µm Nylon 66, with 1 µm prefilter

tC-18 Solid Phase Extraction (SPE) cartridge, 500 mg

Reagents

acetone, distilled from all-glass apparatus

boiling chips, 20-30 mesh carborundum

eluant, water/acetone, 30% (v/v)

glass wool, Pyrex, see Section 204 for handling directions

methylene chloride, distilled from all-glass apparatus

petroleum ether, distilled from all-glass apparatus

sodium chloride, reagent grade

sodium sulfate, anhydrous, granular, reagent grade; see Section 204 for handling directions

Directions

- Prewash filter paper with acetone to remove contaminants.
- Weigh 100 g chopped or blended sample into blender jar, add 200 mL acetone, and blend 2 min at high speed.
- Filter with suction through 12 cm Büchner fitted with Shark Skin® paper; collect extract in 500 mL suction flask. Continuation of vacuum for excessive period can reduce volume of extract and cause error in calculation.
- Attach 0.45 µm Nylon cartridge filter to bottom of 75 mL reservoir; attach tC-18 SPE cartridge to outlet of cartridge filter.
- Wash system with 40 mL acetone, followed by 10 mL eluant. Discard washes.

- Measure 40 mL sample extract and place into reservoir. Elute extract at 3 to 5 mL/min, with air pressure, into 1 L separatory funnel; do not allow level of extract to go below bottom of reservoir.
- Rinse graduated cylinder used for transfer with 10 mL 30% water/acetone; place rinse into reservoir and elute to column dryness.
- Add 50 mL acetone and 100 mL methylene chloride to separatory funnel and shake vigorously 1 min. Let separator stand 5-10 min to allow layers to separate.
- Dry lower organic layer of first separator by passing through about 1.5" sodium sulfate supported on washed glass wool in 4" funnel, collecting in K-D.
- Add 100 mL acetone and 100 mL methylene chloride to separator and repeat shaking. Let separator stand 5-10 min.
- Drain lower organic layer through sodium sulfate into separator. (Sugar content of fruit samples may result in aqueous phase's being the lower layer. In that case, add 5-10 mL methylene chloride and repeat shaking.) Rinse sodium sulfate with about 50 mL methylene chloride.
- Add boiling chips to K-D and concentrate solvent in K-D; start evaporation slowly by placing only receiver tube into steam. After 100-150 mL has evaporated, concentrator may be exposed to more steam. Concentrate solvent to 2-3 mL. After cooling, remove tube from K-D and adjust volume to 5 mL with acetone.
- Calculate equivalent sample weight in final solution:

$$\frac{\text{mg sample equivalent}}{\mu\text{L final extract}} = 100 \times \frac{40}{200 + W - 10} \times \frac{1}{\text{mL final volume}}$$

where:

100 = g sample analyzed

40 = mL filtered extract taken for liquid-liquid partitioning

200 = mL acetone blended with 100 g sample

W = amount (mL) of water present in sample (Section 201; if data are not available for particular raw agricultural commodity, use 85%)

10 = adjustment for water/acetone volume contraction.

Thus, when sample contains 85% water (85 mL/100 g) and final extract volume is 5 mL, each uL contains:

$$100 \times \frac{40}{200 + 85 - 10} \times \frac{1}{5} = \frac{2.9 \text{ mg sample equivalent}}{\mu\text{L final extract}}$$

- Clean up extract with C6 prior to determination.

C1 FLORISIL COLUMN (4 G) CLEANUP, WITH ONE METHYLENE CHLORIDE ELUANT

References

Griffitt, K.R., *et al.* (July 1983) "Miniaturized Florisil Column Cleanup of Chlorinated and Organophosphate Eluates in Total Diet Samples," LIB 2722, FDA, Rockville, MD

Griffitt, K.R., and Szorik, M.M. (Sept. 1989) "The Analysis of 127 Total Diet Items for Chlorinated Residues Using Luke/Solid Phase Extracts," LIB 3366, FDA, Rockville, MD

Principle

Residues in solution are separated from sample co-extractives on a small column of Florisil adsorbent, eluting with a single eluant.

Apparatus

chromatographic column, 10 mm id × 300 mm, Teflon stopcock, coarse porosity fritted disc

Kuderna-Danish concentrator (K-D), 125 or 250 mL, with Snyder column, two-ball micro-Snyder column, graduated or volumetric receiving flask

Reagents

acetonitrile, distilled from all-glass apparatus; see Section 204 for distillation directions

Florisil, PR grade; see Section 204 for handling and testing directions and calculation of lauric acid value

hexane, distilled from all-glass apparatus

sodium sulfate, anhydrous, granular, reagent grade; see Section 204 for handling directions

eluant: 50% methylene chloride/1.5% acetonitrile/48.5% hexane (v/v/v). Pipet 15 mL acetonitrile into 500 mL methylene chloride and dilute with hexane. Allow mixture to reach room temperature and adjust to 1 L with hexane.

Directions

- Place activated Florisil (weight = 110/lauric acid value × 4 g) in 10 mm chromatographic column; add about 2 cm sodium sulfate. Completely open stopcock and tap column to settle adsorbent. Prewet column with 15 mL hexane. Do not allow column to go dry. Place K-D with volumetric or graduated receiving flask under column to receive eluate.
- Dilute extract with hexane to produce solution of 10% acetone/hexane. Volumes depend on concentration of extract, volume taken for cleanup; *e.g.*, dilute 1 mL E1 extract, previously concentrated to 7 mL acetone, to 10 mL with hexane.
- Transfer solution to Florisil column, letting it pass through at about 5 mL/min. Rinse container with two 3 mL portions hexane, transfer rinsings to column, and rinse walls of chromatographic tube with additional small portions hexane.
- Elute column at about 5 mL/min with 50 mL eluant.

- Add boiling chip to K-D and concentrate eluate to suitable definite volume. For example, if 1 mL E1 extract (equivalent to 4.15 mg/mL) was cleaned up, concentrate Florisil eluate to 1 mL for same final concentration.

  When volume <5 mL is needed, use two-ball micro-Snyder or micro-Vigreaux column during evaporation.

- Use appropriate determinative steps, such as DG1 or DG13, DG6, DG7, and DG10, to identify and measure residues.

C2 CHARCOAL/CELITE/MAGNESIUM OXIDE COLUMN CLEANUP

References

Luke, M.A., and Doose, G.M. (1983) *Bull. Environ. Contam. Toxicol.* **30**, 110-116

Hardy, R.P. (Fall 1984) "Recoveries of Organophosphorus Compounds Through the Modified Storherr Method Using Charcoal Columns With and Without Magnesium Oxide," LIB 2860, FDA, Rockville, MD

Principles

Polar residues in solution are separated from sample co-extractives on a column of charcoal/Celite/magnesium oxide; cleanup may be necessary for subsequent examination of extract with selective detectors. Aromatic residues are not eluted with this system and must be determined in extract cleaned up by C1, Florisil column. Magnesium oxide may be eliminated to prevent destruction of sensitive residues (*e.g.*, acephate) without diminishing recoveries of other residues normally eluted.

Apparatus

chromatographic column, 22 mm id × 300 mm, Teflon stopcock, coarse porosity fritted disc

Kuderna-Danish concentrator (K-D), 500 mL, with Snyder column, two-ball micro-Snyder column, graduated or volumetric receiving flask

Reagents

acetone, distilled from all-glass apparatus

adsorbent mixture, 1:4:2 (w/w/w) charcoal/Celite 545/magnesium oxide or 1:4 (w/w) charcoal/Celite 545

Celite 545. To prepare, slurry about 500 g with distilled water, heat on steam bath about 30 min, and filter with suction. Dry overnight at l05-l30° C and pulverize to pass No. 60 sieve. Store in closed jar.

charcoal, Darco G60 or Norite S.G. Extra

glass wool, Pyrex; see Section 204 for handling directions

magnesium oxide, 200 mesh, adsorptive grade (optional)

methylene chloride, distilled from all-glass apparatus

eluant: 2:1 (v/v) acetone/methylene chloride

Directions

- Place about 1" Celite 545 in column, then add 6 g adsorbent mixture, and top with large plug glass wool.
- Tamp column down firmly and add about 25 mL methylene chloride. Force solvent through column with air pressure until top of solvent reaches top of column. Discard solvent.
- Transfer sample extract quantitatively to column with small portions methylene chloride and force solvent through as before, collecting in K-D.
- Elute with 200 mL 2:1 acetone/methylene chloride; force through as before.

- Mix contents of K-D, add boiling chips, and concentrate solvent; start evaporation slowly by placing only receiver tube into steam. After 100-150 mL has evaporated, concentrator may be exposed to more steam. When liquid level in hot concentrator tube is about 2 mL, add 100 mL petroleum ether through Snyder column and reconcentrate to about 2 mL. Add 50 mL petroleum ether and repeat concentration step. Add 20 mL acetone, and reconcentrate to about 2 mL. Do not allow solution to go to dryness during any of the concentration steps. Adjust volume of extract to suitable definite volume with acetone.
- If magnesium oxide is not used, a white precipitate may form if extract is concentrated to <2 mL; this should not affect GLC.
- Use appropriate determinative steps or confirmatory steps, such as GLC with mass spectrometric detection.

C3 CHARCOAL/SILANIZED CELITE COLUMN CLEANUP

References

Krause, R.T. (1980) *J. Assoc. Off. Anal. Chem.* **63**, 1114-1124

Pardue, J.R. (May 1987) "Recoveries of N-Methyl Carbamates Using a Combination of the Luke (PAM I, 232.4) and Krause (PAM I, 242.24b, 242.25) Procedures," LIB 3138, FDA, Rockville, MD

Principle

Residues in solution are separated from sample co-extractives on a column of charcoal and Celite, cleaning up the extract sufficiently for subsequent determination by HPLC system DL1.

Apparatus

chromatographic column, 22 mm id × 300 mm, Teflon stopcock, coarse porosity fritted disc

evaporator, vacuum rotary, as described in Section 401 E1

flasks, round-bottom (r-b), 250 and 500 mL, 1 L

magnetic stirrer, star, 10 mm diameter × 8 mm

vacuum adapter, side arm, with Ts bottom joint to fit in 500 mL r-b flask

Reagents

acetonitrile, distilled from all-glass apparatus; see Section 204 for distillation directions

Celite 545, silanized and prepared for use as directed in Section 401 C1

charcoal (Nuchar S-N), produced by Westvaco Corp. and available from Eastman Kodak, Cat. No. 118 0454, purified as directed in Section 401 C1

glass wool, Pyrex; see Section 204 for handling directions

methanol, distilled from all-glass apparatus

methylene chloride, distilled from all-glass apparatus

toluene, distilled from all-glass apparatus

eluant: 25% (v/v) toluene/acetonitrile

Directions

- Test charcoal/silanized Celite column as described in Section 401 C1.
- To the extract in r-b flask, add star magnetic stirrer. Place 250 mL T 24/40 trap on 1 L r-b flask and attach to vacuum rotary evaporator.
- Circulate refrigerated (-15° C) 1+1 water/ethylene glycol through evaporator condensing coils; maintain receiving flask at -15° C by immersion in refrigerated bath.
- Apply vacuum slowly to minimize frothing by regulating with needle valve. After full vacuum is applied, slowly place flask in 35° C water bath.
- Remove r-b flask from evaporator immediately after last traces of solution have evaporated and add 10 mL methylene chloride to r-b flask.

- Fit one-hole No. 5 rubber stopper onto tip of chromatographic column, add side arm vacuum adapter and 500 mL r-b flask, open stopcock, and connect apparatus to vacuum line.
- Place 0.5 g silanized Celite 545 in chromatographic column, tamp, add 5 g charcoal/Celite 545 (1+4) mixture, and tamp again. Add 1-2 cm glass wool plug on top of adsorbent.
- Prewash column with 50 mL 25% toluene/acetonitrile eluant. Close stopcock when prewash solution is about 0.5 cm from top of glass wool.
- Disconnect vacuum, discard solution in r-b flask, and reconnect flask to apparatus.
- Transfer 10 mL methylene chloride extract to column and let pass through column at 5 mL/min.
- Wash 1 L r-b flask with 10 mL methylene chloride and then with 25 mL eluant. Transfer each separately to column and elute each to top of glass wool before adding next solution.
- Add 100 mL eluant and elute column at 5 mL/min. Turn off stopcock when top of eluant reaches top of glass wool.
- Evaporate solution in 500 mL r-b flask just to dryness using vacuum evaporator as above. Remove flask from evaporator immediately after all solution has evaporated.
- Immediately pipet 5 mL methanol into 500 mL r-b flask to dissolve residue. Cleaned up extract contains concentration of sample equivalent (mg/µL) equal to amount of sample in extract taken for cleanup, divided by 5. For example, if entire E1 extract of commodity with 85% water is used, 29 g sample equivalent is cleaned up, *i.e.*, $100\,g \times 80/(200+85-10)$; final concentration of cleaned up extract is 5.8 mg/µL (29 g/5 mL).
- Use determinative step DL1 or DL2 (Section 401) to determine N-methylcarbamates, except use 20 µL injection loop instead of 10 µL loop specified.

C4 C-18 CARTRIDGE CLEANUP

Reference

Sharp, K.B., and Bramlett, C.L. (Dec. 1983) "Analysis for Carbamate Residues in Fresh Produce," LIB 2778, FDA, Rockville, MD

Principle

Residues in solution are separated from sample co-extractives on a C-18 solid phase extraction cartridge, cleaning up the extract sufficiently for subsequent determination by HPLC system DL1.

Apparatus

Kuderna-Danish concentrator (K-D), 500 mL, with Snyder column, two-ball micro-Snyder column, graduated receiving flask

volumetric flask, 5 mL

Reagents

cartridge (solid phase extraction type), C-18, 2.8 mL

methanol, distilled from all-glass apparatus

Directions

- Concentrate extract in K-D to 2 mL. Evaporate almost to dryness (about 0.1 mL) under current of nitrogen.
- Prewet C-18 cartridge with methanol and discard solvent.
- Dissolve residue in receiving flask with 2 mL methanol and transfer quantitatively onto prewet C-18 cartridge. Collect eluate from cartridge in 5 mL volumetric flask.
- Elute cartridge with additional methanol until collected volume is almost 5 mL; add methanol to make volume 5.0 mL. Cleaned up extract contains concentration of sample equivalent (mg/µL) equal to amount of sample in extract taken for cleanup, divided by 5. For example, if entire E1 extract of commodity with 85% water is used, 29 g sample equivalent is cleaned up, *i.e.*, 100 g × 80/(200 + 85 - 10); final concentration of cleaned up extract is 5.8 mg/µL (29 g/5 mL).
- Use determinative step DL1 or DL2 (Section 401) to determine N-methylcarbamates, except use 20 µL injection loop instead of 10 µL loop specified.

C5 FLORISIL COLUMN CLEANUP, WITH MIXED ETHER ELUANTS

Reference

Luke, M.A., *et al.* (1975) *J. Assoc. Off. Anal. Chem.* **58**, 1020-1026

Principles

Residues in solution are separated from sample co-extractives on a column of Florisil adsorbent; cleanup is usually necessary for subsequent examination of extract with DG1, electron capture detector.

Apparatus

chromatographic column, 22 mm id × 300 mm, Teflon stopcock, coarse porosity fritted disc

graduated cylinder (graduate), glass-stoppered (g-s), 100 mL

Kuderna-Danish concentrator (K-D), 500 mL, with Snyder column, two-ball micro-Snyder column, volumetric or graduated receiving flask

Reagents

boiling chips, 20-30 mesh carborundum

eluants: 15% (v/v) ethyl ether/petroleum ether
50% (v/v) ethyl ether/petroleum ether

ethyl ether, distilled from all-glass apparatus, with 2% ethanol as preservative; see Section 204 for peroxide test

Florisil, PR grade; see Section 204 for handling and testing directions and calculation of lauric acid value

petroleum ether, distilled from all-glass apparatus

sodium sulfate, anhydrous, granular, reagent grade; see Section 204 for handling directions

Directions

- Place activated Florisil (4" or weight determined by lauric acid value) in 22 mm id column; add about 0.5" sodium sulfate. Prewet column with 40-50 mL petroleum ether. Place K-D with volumetric or graduated receiving flask under column to receive eluate.
- Dilute concentrated extract to 10 mL with acetone and transfer to 100 mL g-s graduate, using petroleum ether to rinse. Dilute to 100 mL with petroleum ether; stopper and mix well.
- Transfer diluted extract solution to column letting it pass through at about 5 mL/min.
- Elute column at about 5 mL/min with 200 mL 15% ethyl ether/petroleum ether eluant.
- Change K-Ds and elute at about 5 mL/min with 200 mL 50% ethyl ether/petroleum ether eluant.
- Add boiling chips to K-Ds and concentrate to suitable definite volume. For example, if entire E1 extract of commodity with 85% water is used, and final volume is 5 mL, final concentration of cleaned up extract is 5.8 mg/µL, *i.e.*, 100 g × 80/(200 + 85 - 10) = 29 g; 29 g/5 mL = 5.8 mg/µL.

- When volume <5 mL is needed, use two-ball micro-Snyder or micro-Vigreaux column during final evaporation in receiving flask.
- Use appropriate determinative steps, such as DG1 or DG13, DG6, DG7, and DG10, to identify and measure residues.

C6 SAX/PSA CARTRIDGE CLEANUP

Reference

Luke, M. A., *et al.* (Sept. 1994) "An Improved Variation of the Luke Multiresidue Pesticide Procedure for the Analysis of Fruits and Vegetables Using Solid Phase Extraction Cartridges and Element Selective Gas Chromatographic Detectors," LIB 3896, FDA, Rockville, MD

Principle

SAX and PSA cartridges provide the improved cleanup required for determination with capillary and megabore GC columns; both polar and nonpolar residues can be recovered.

Apparatus

75 mL Bond Elut reservoir or equivalent

25 mm syringe filter, 0.45 µm Nylon 66 with 1 µm prefilter

SAX SPE cartridge or equivalent, 500 mg

PSA SPE cartridge or equivalent, 500 mg

Kuderna-Danish concentrator (K-D), 500 mL, with Snyder column, two-ball micro-Snyder column, graduated or volumetric receiving flask

Reagents

acetone, distilled from all-glass apparatus

petroleum ether, distilled from all-glass apparatus

acetone+petroleum ether, 1+2

Directions

- Attach 0.45 µm filter to bottom of 75 mL reservoir. Attach SAX or equivalent cartridge to filter, and attach PSA or equivalent cartridge to first cartridge.
- Wash cartridges with 40 mL acetone; follow with 10 mL acetone+petroleum ether. Discard washes.
- Dilute the 5.0 mL concentrated acetone extract from E7 with 10 mL petroleum ether and mix. Transfer to reservoir, and elute dropwise with air pressure.
- Rinse tube with five 10 mL portions acetone+petroleum ether. Elute each rinse when the previous solvent has reached top of column.
- Mix contents of K-D, add boiling chips, and concentrate solvent; start evaporation slowly by placing only receiver tube into steam. When liquid level in hot concentrator tube is about 2 mL, add 100 mL petroleum ether through Snyder column and reconcentrate to about 2 mL. Add 50 mL petroleum ether and repeat concentration step. Carefully add 25 mL acetone and reconcentrate to about 2 mL. Do not allow solution to go to dryness during any of the concentration steps. Adjust volume of extract to suitable definite volume with acetone.
- Use appropriate determinative steps, such as DG2, DG3, DG14, or DG16, to identify and measure residues.

DETERMINATION

Inject concentrated extract equivalent to 20 mg (whole high moisture product) into the following GLC systems for determination of residues. (Although AOAC collaborative study for this method involved injection of 12 mg sample equivalent, experience since then has proven that GLC systems can tolerate routine injections equivalent to 20 mg of most nonfatty foods.)

Extract not cleaned up prior to determination:

DG2 or DG14	organophosphorus residues; large amounts of sulfur may interfere
DG3 or DG16	organohalogen residues
DG4 or	organonitrogen residues; selective to nitrogen, but co-extractives may contain nitrogen
DG5 or DG17	organonitrogen and organophosphorus residues
DG15	organosulfur residues; large amounts of phosphorus may interfere
DG12	late eluting organohalogen residues, especially pyrethroids

Additional recommended determinations:

Extract not cleaned up prior to determination:

DG8	early eluting organophosphorus residues
DG11	late eluting organophosphorus residues
DG9	early eluting organohalogen residues

Extract cleaned up on Florisil column, C1 or C5:

DG1 or DG13	residues with halogen, sulfur, or other moieties
DG7	early eluting residues with halogen, sulfur, or other moieties
DG10	late eluting residues, especially synthetic pyrethroids
DG6	o-phenylphenol and biphenyl

Inject concentrated extract equivalent to about 58-116 mg (whole high moisture product) cleaned up by C3 (charcoal/Celite column) or C4 (C-18 cartridge) into following HPLC system:

DL1	N-methylcarbamates (determinative step described in Section 401)

For accurate quantitation, reference standards should be dissolved in same solvent as concentrated extract, only peaks >10% FSD should be measured, and peak sizes of residue and reference standard should match within ±25%.

See Chapter 5 for additional information about operation of GLC systems; Section 504 provides information about quantitation of residues.

See Chapter 6 for additional information about operation of HPLC systems; Section 606 provides information about quantitation of residues.

See Section 205 for additional information about reference standards.

See Section 104 for additional information about reporting residues and determining compliance with regulations.

See Section 105 for additional information about analytical limits of quantitation.

CONFIRMATION

After residues have been tentatively identified and quantitated by comparison to appropriate reference standards, confirm identity according to principles discussed in Section 103. Use appropriate tables of data (PESTDATA, tables accompanying each method, Index to Methods) to choose the most appropriate determinative steps and/or alternative methods for confirmation.

DG1 GLC, 100% METHYL SILOXANE, 200° C, EC

Applicability

Determinative step is applicable to residues containing halogen, sulfur, or other electrophilic moieties. It is a general purpose system, but subject to interferences by nonpesticides.

Column

Wide bore capillary, 30 m × 0.53 mm id, coated with 100% methyl substituted polysiloxane liquid phase, 1-1.5 µm film thickness, bonded and cross-linked. For example, DB-1; see Table 502-a for equivalents. See Section 502 C, Recommended Operating Procedure for Wide Bore Columns.

Column Operating Conditions:

200° C isothermal; if necessary, adjust temperature so that relative retention time to chlorpyrifos (rrt_c) of p,p'-DDT is 3.1 ± 0.06.

Carrier gas: helium; adjust flow rate so that chlorpyrifos elutes in about 4.0 ± 0.5 min (about 20 mL/min).

Injector temperature: 220-250° C

Detector

Electron Capture (EC)

Detector Operating Conditions:

350° C

Make-up gas: nitrogen or argon/methane (95:5), at 30 mL/min

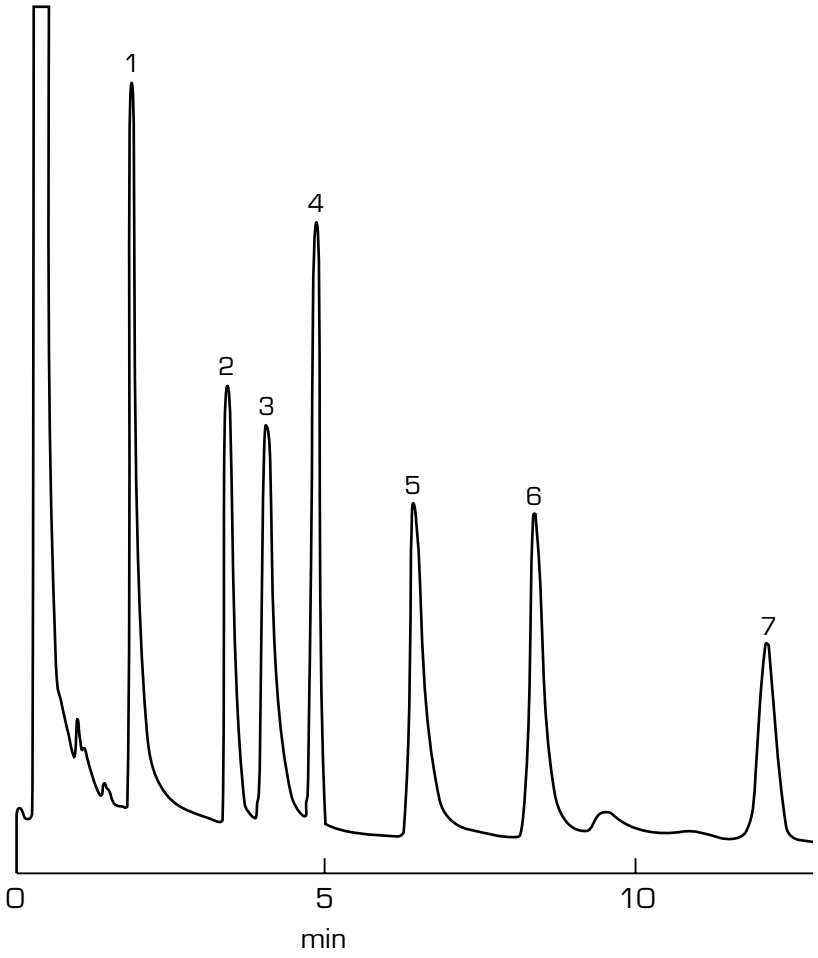
See Section 503 B for other information about EC detector operation.

Set detector electronics (amplification, attenuation) so that response to 0.15 ng chlorpyrifos (or an amount within the detector's linear range) is 50% full scale deflection (FSD).

Other Considerations

Rrt_cs and ng required to cause 50% FSD response are listed in Appendix I, PESTDATA (many data in PESTDATA were collected using equivalent packed column). Response data in Appendix I are based on detector sensitivity of 50% FSD to 1.5 ng chlorpyrifos.

Example chromatogram is on next page. Also see Figures 504-c, d, e, and f.



DG1

Chromatogram of: 1) 0.15 ng dicloran, 2) 0.10 ng heptachlor, 3) 0.19 ng chlorpyrifos, 4) 0.31 ng captan, 5) 0.14 ng endosulfan I, 6) 0.18 ng endrin, and 7) 0.20 ng p,p′-DDT at the conditions described.

DG2 GLC, 100% METHYL SILOXANE, 200° C, FPD-P

Applicability

Determinative step is applicable to residues containing phosphorus. It is particularly useful for residues such as organophosphate pesticides.

Column

Wide bore capillary, 30 m × 0.53 mm id, coated with 100% methyl substituted polysiloxane liquid phase, 1-1.5 µm film thickness, bonded and cross-linked. For example, DB-1; see Table 502-a for equivalents. See Section 502 C, Recommended Operating Procedure for Wide Bore Columns.

Column Operating Conditions:

200° C isothermal; if necessary, adjust temperature so that relative retention time to chlorpyrifos (rrt_c) of ethion is 2.56 ± 0.05.

Carrier gas: helium; adjust flow rate so that chlorpyrifos elutes in about 4.0 ± 0.5 min (about 20 mL/min).

Injector temperature: 220-250° C

Detector

Flame photometric, phosphorus mode (FPD-P)

Detector Operating Conditions:

225-250° C

See Section 503 C for other information about FPD operation.

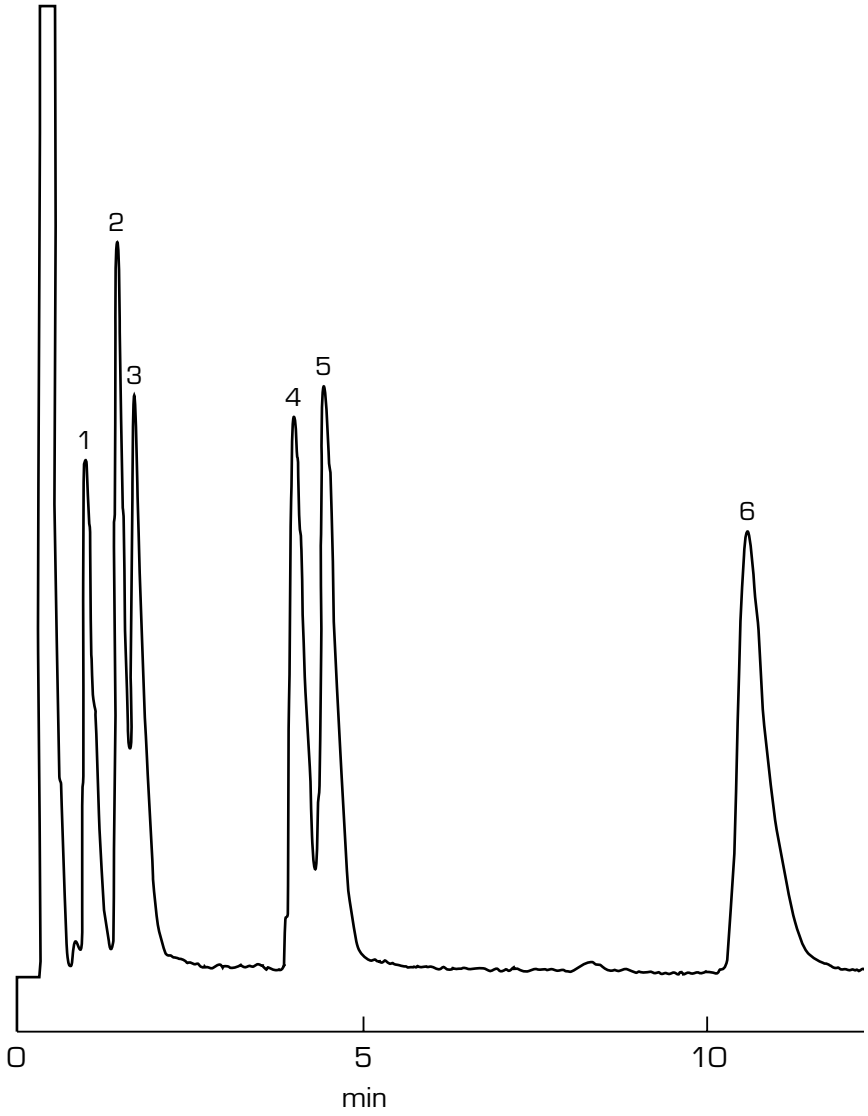
Set detector electronics (amplification, attenuation) so that response to 1.5 ng chlorpyrifos is 50% full scale deflection (FSD).

A properly functioning system will give a response to 1.5 ng omethoate of ≥50% FSD.

Other Considerations

Rrt_cs and ng required to cause 50% FSD response are listed in Appendix I, PESTDATA (many data in PESTDATA were collected using equivalent packed column).

Example chromatogram is on next page.



DG2

Chromatogram of: 1) 0.85 ng acephate, 2) 1.73 ng omethoate, 3) 0.68 ng monocrotophos, 4) 1.30 ng malathion, 5) 1.27 ng chlorpyrifos, and 6) 1.26 ng ethion at the conditions described; helium carrier gas flow was 15 mL/min, with 15 mL/min make-up gas being added before the detector. Detector gas flows: 100 mL/min hydrogen, 130 mL/min air.

DG3 GLC, 100% METHYL SILOXANE, 200° C, ELCD-X

Applicability

Determinative step is applicable to residues containing halogen. It is particularly useful for residues such as chlorinated hydrocarbon pesticides and polychlorinated biphenyls.

Column

Wide bore capillary, 30 m × 0.53 mm id, coated with 100% methyl substituted polysiloxane liquid phase, 1-1.5 µm film thickness, bonded and cross-linked. For example, DB-1; see Table 502-a for equivalents. See Section 502 C, Recommended Operating Procedure for Wide Bore Columns.

Column Operating Conditions:

200° C isothermal; if necessary, adjust temperature so that relative retention time to chlorpyrifos (rrt_c) of p,p'-DDT is 3.1 ± 0.06.

Carrier gas: helium; adjust flow rate so that chlorpyrifos elutes in about 4.0 ± 0.5 min (about 20 mL/min).

Injector temperature: 220-250° C

Detector

Electroconductivity, halogen mode (ELCD-X)

Detector Operating Conditions:

Base temperature 250° C; furnace temperature 900° C (or as specified in operating manual)

Reactor gas: hydrogen, flow as required by specific detector model; see operating manual

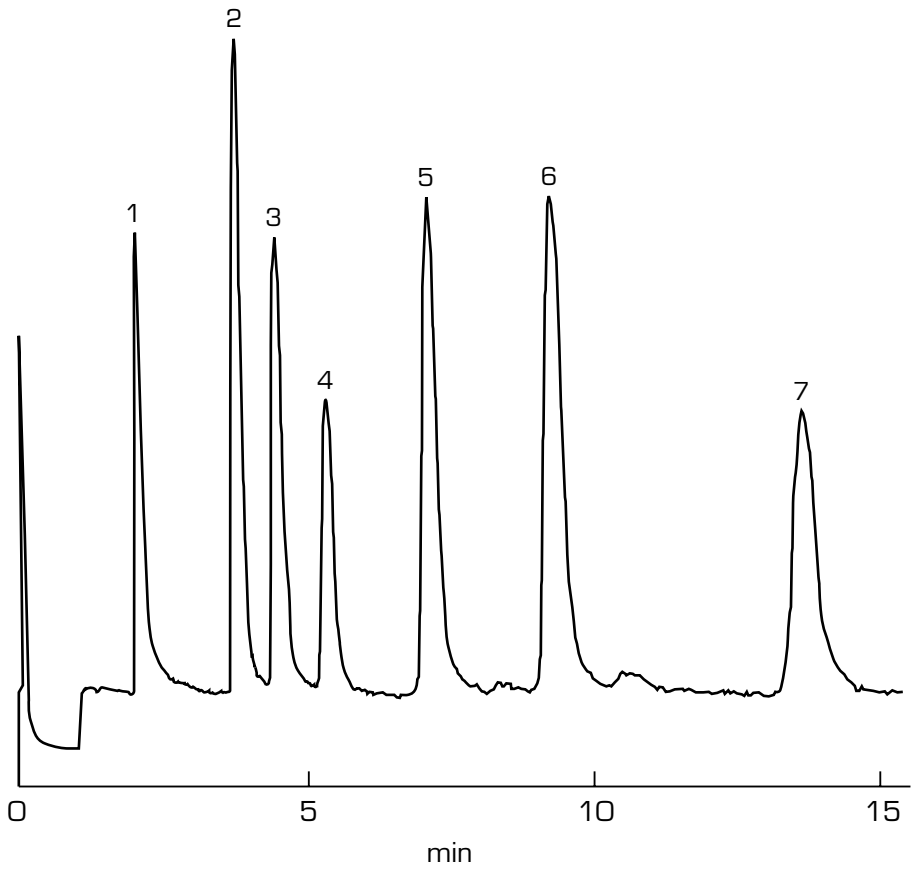
See Section 503 D for other information about ELCD operation.

Set detector electronics (amplification, attenuation) so that response to 1.5 ng chlorpyrifos is 50% full scale deflection (FSD).

Other Considerations

Rrt_cs and ng required to cause 50% FSD response are listed in Appendix I, PESTDATA (many data in PESTDATA were collected using equivalent packed column).

Example chromatogram is on next page.



DG3

Chromatogram of: 1) 1.44 ng dicloran, 2) 0.98 ng heptachlor, 3) 1.87 ng chlorpyrifos, 4) 2.99 ng captan, 5) 1.37 ng endosulfan I, 6) 1.77 ng endrin, and 7) 1.91 ng p,p'-DDT at the conditions described. Hydrogen reactor gas flow: 40 mL/min, n-propanol electrolyte: 0.3 mL/min.

DG4 GLC, 100% METHYL SILOXANE, 200° C, ELCD-N

Applicability

Determinative step is applicable to residues containing nitrogen. It may be useful for confirmation of residues such as triazines (atrazine, simazine, *etc.*) and triazoles (propiconazole, diclobutrazole, *etc.*).

Column

Wide bore capillary, 30 m × 0.53 mm id, coated with 100% methyl substituted polysiloxane liquid phase, 1-1.5 µm film thickness, bonded and cross-linked. For example, DB-1; see Table 502-a for equivalents. See Section 502 C, Recommended Operating Procedure for Wide Bore Columns.

Column Operating Conditions:

200° C isothermal

Carrier gas: helium; adjust flow rate so that chlorpyrifos elutes in about 4.0 ± 0.5 min (about 20 mL/min).

Injector temperature: 220-250° C

Detector

Electroconductivity, nitrogen mode (ELCD-N)

Detector Operating Conditions:

Base temperature 250° C; furnace temperature 900° C (or as specified in operating manual)

Reactor gas: hydrogen, flow as required by specific detector model; see operating manual

See Section 503 D for other information about ELCD operation.

Set detector electronics (amplification, attenuation) so that response to 1.5 ng chlorpyrifos is 50% full scale deflection (FSD).

Other Considerations

Rrt_cs and ng required to cause 50% FSD response are listed in Appendix I, PESTDATA (many data in PESTDATA were collected using equivalent packed column).

No chromatogram currently available.

DG5 GLC, 100% METHYL SILOXANE, 200° C, N/P

Applicability

Determinative step is applicable to residues containing nitrogen. It is particularly useful for residues such as triazines and triazoles.

Column:

Wide bore capillary, 30 m × 0.53 mm id, coated with 100% methyl substituted polysiloxane liquid phase, 1-1.5 µm film thickness, bonded and cross-linked. For example, DB-1; see Table 502-a for equivalents. See Section 502 C, Recommended Operating Procedure for Wide Bore Columns.

Column Operating Conditions:

200° C isothermal; if necessary, adjust temperature so that relative retention time to chlorpyrifos (rrt_c) of ethion is 2.56 ± 0.05.

Carrier gas: helium; adjust flow rate so that chlorpyrifos elutes in about 4.0 ± 0.5 min (about 20 mL/min).

Injector temperature: 220-250° C

Detector

Alkali bead detector, nitrogen selective (N/P)

Detector Operating Conditions:

250° C

See Section 503 E for other information about N/P detector operation.

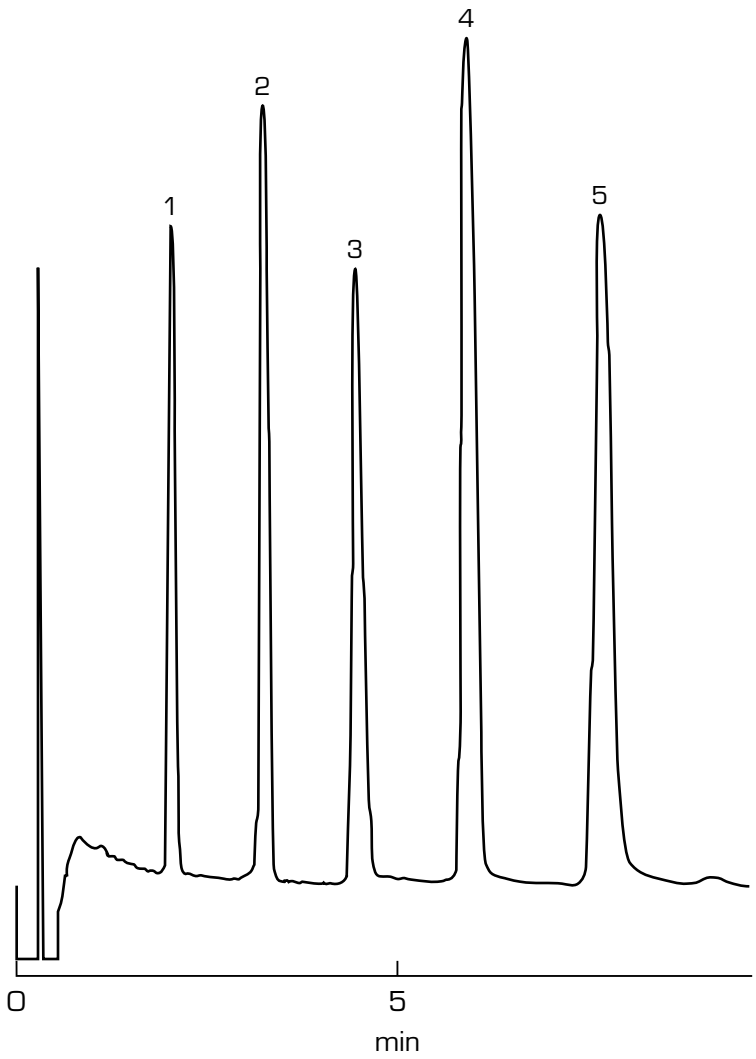
Set detector electronics (amplification, attenuation) so that response to 1.5 ng chlorpyrifos is 50% full scale deflection (FSD).

A properly functioning system will give a response to 1.5 ng omethoate of ≥50% FSD.

Other Considerations

Rrt_cs and ng required to cause 50% FSD response are listed in Appendix I, PESTDATA (many data in PESTDATA were collected using equivalent packed column).

Example chromatogram is on next page.



DG5

Chromatogram of: 1) 1.0 ng atrazine, 2) 7.5 ng carbaryl, 3) 1.5 ng chlorpyrifos, 4) 2.5 ng procyazine, and 5) 5.0 ng imazalil at the conditions described.

DG6 GLC, 100% METHYL SILOXANE, 130° C, FID

Applicability

Determinative step is applicable to residues containing no elements to which element-selective detectors respond. It is particularly useful for residues such as biphenyl and o-phenylphenol.

Column

Wide bore capillary, 30 m ¥ 0.53 mm id, coated with 100% methyl substituted polysiloxane liquid phase, 1-1.5 µm film thickness, bonded and cross-linked. For example, DB-1; see Table 502-a for equivalents. See Section 502 C, Recommended Operating Procedure for Wide Bore Columns.

Column Operating Conditions:

130° C isothermal

Carrier gas: helium, about 20 mL/min. At these conditions, chlorpyrifos elutes in about 16 min, and biphenyl and o-phenylphenol elute in <2 min.

Injector temperature: 220-250° C

Detector

Flame ionization detector (FID)

Detector Operating Conditions:

300° C

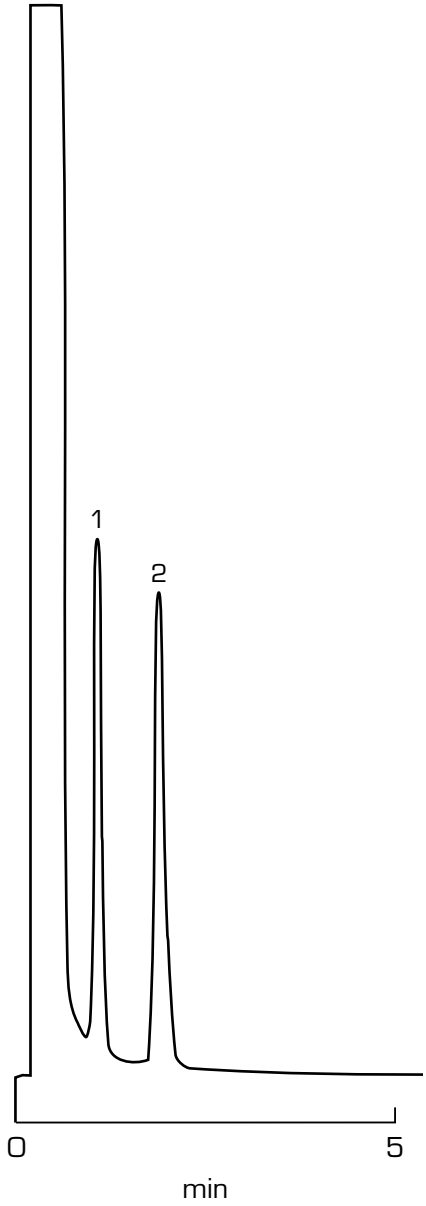
detector gases: hydrogen, 30 mL/min, air, 300 mL/min

set detector electronics (amplification, attenuation) so that response to 50 ng o-phenylphenol is 50% full scale deflection (FSD).

Other Considerations

FID is nonselective and will respond to large quantities of any co-extractive.

Example chromatogram is on next page.



DG6

Chromatogram of: 1) 20 ng biphenyl and 2) 53 ng o-phenylphenol at the conditions described.

DG7 GLC, 100% METHYL SILOXANE, 130° C, EC

Applicability

Determinative step is applicable to residues of high volatility (early elution) and containing halogen, sulfur, or other electrophilic moieties. It is particularly useful for residues such as benfluralin and sulfallate.

Column

Wide bore capillary, 30 m × 0.53 mm id, coated with 100% methyl substituted polysiloxane liquid phase, 1-1.5 µm film thickness, bonded and cross-linked. For example, DB-1; see Table 502-a for equivalents. See Section 502 C, Recommended Operating Procedure for Wide Bore Columns.

Column Operating Conditions:

130° C isothermal

Carrier gas: helium; adjust flow rate while column temperature is 200° C so that chlorpyrifos elutes in about 4.0 ± 0.5 min; then change column temperature without changing flow controller.

Injector temperature: 220-250° C

Detector

Electron Capture (EC)

Detector Operating Conditions:

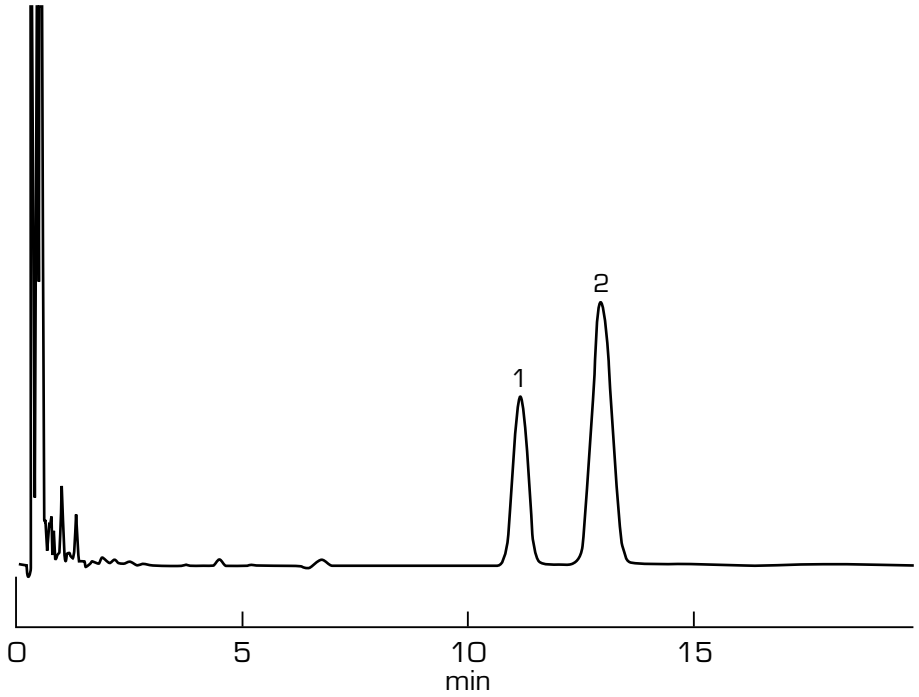
350° C

Make-up gas: nitrogen or argon/methane (95:5), at 30 mL/min

See Section 503 B for other information about EC detector operation.

While column temperature is 200° C, set detector electronics (amplification, attenuation) so that response to 0.15 ng chlorpyrifos (or an amount within the detector's linear range) is 50% full scale deflection; then change column temperature without changing electronics.

Example chromatogram is on next page.



DG7

Chromatogram of: 1) 0.18 ng benfluralin and 2) 0.09 ng sulfallate at the conditions described.

DG8 GLC, 100% METHYL SILOXANE, 130° C, FPD-P

Applicability

Determinative step is applicable to residues of high volatility (early elution) and containing phosphorus. It is particularly useful for residues such as mevinphos, acephate, demeton, and dicrotophos.

Column

Wide bore capillary, 30 m × 0.53 mm id, coated with 100% methyl substituted polysiloxane liquid phase, 1-1.5 µm film thickness, bonded and cross-linked. For example, DB-1; see Table 502-a for equivalents. See Section 502 C, Recommended Operating Procedure for Wide Bore Columns.

Column Operating Conditions:

130° C isothermal

Carrier gas: helium; adjust flow rate while column temperature is 200° C so that chlorpyrifos elutes in about 4.0 ± 0.5 min; then change column temperature without changing flow controller.

Injector temperature: 220-250° C

Detector

Flame photometric, phosphorus mode (FPD-P)

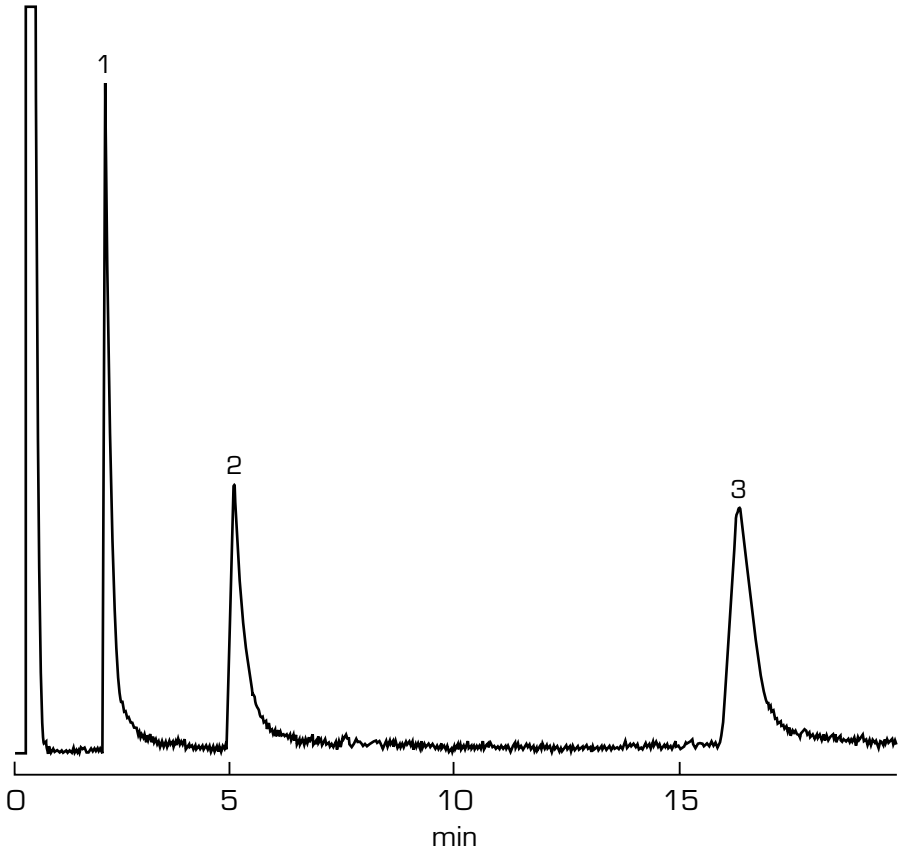
Detector Operating Conditions:

225-250° C

See Section 503 C for other information about FPD operation.

While column temperature is 200° C, set detector electronics (amplification, attenuation) so that response to 1.5 ng chlorpyrifos is 50% full scale deflection; then change column temperature without changing electronics.

Example chromatogram is on next page.



DG8

Chromatogram of: 1) 2.0 ng methamidophos, 2) 2.0 ng acephate, and 3) 4.0 ng dicrotophos at the conditions described.

DG9 GLC, 100% METHYL SILOXANE, 130° C, ELCD-X

Applicability

Determinative step is applicable to residues of high volatility (early elution) and containing halogen. It is particularly useful for residues such as the methyl esters of dicamba, MCPA, mecoprop, dichlorprop, and silvex.

Column

Wide bore capillary, 30 m × 0.53 mm id, coated with 100% methyl substituted polysiloxane liquid phase, 1-1.5 µm film thickness, bonded and cross-linked. For example, DB-1; see Table 502-a for equivalents. See Section 502 C, Recommended Operating Procedure for Wide Bore Columns.

Column Operating Conditions:

130° C isothermal

Carrier gas: helium, about 20 mL/min

Injector temperature: 220-250° C

Detector

Electroconductivity, halogen mode (ELCD-X)

Detector Operating Conditions:

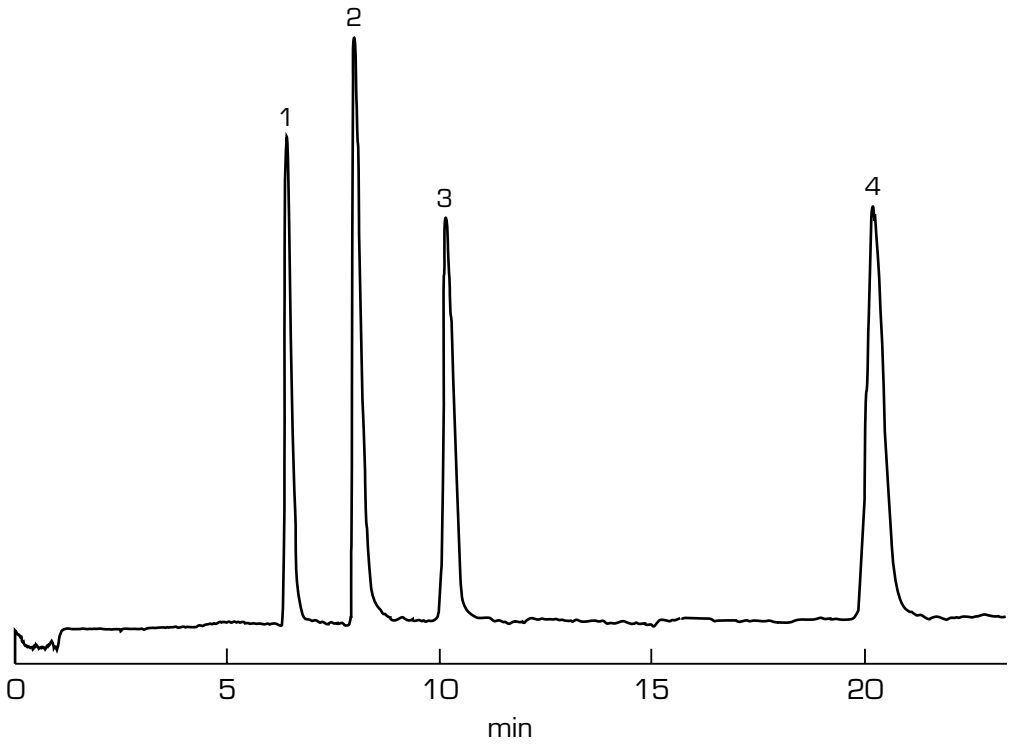
Base temperature 250° C; furnace temperature 900° C (or as specified in operating manual)

Reactor gas: hydrogen, flow as required by specific detector model; see operating manual

See Section 503 D for other information about ELCD operation.

Set detector electronics (amplification, attenuation) so that response to 0.5 ng pentachlorobenzene is 50% full scale deflection.

Example chromatogram is on next page.



DG9

Chromatogram of: 1) 1.0 ng dicamba methyl ester, 2) 3.0 ng MCPA methyl ester, 3) 1.5 ng dichlorprop methyl ester, and 4) 2.0 ng silvex methyl ester at the conditions described, except that carrier gas was hydrogen at 25 mL/min. Hydrogen reactor gas flow: 35 mL/min, n-propanol electrolyte 0.5 mL/min. Pentachlorobenzene eluted in 6.9 min at these conditions, and 0.3 ng pentachlorobenzene caused 40% FSD detector response.

DG10 GLC, 100% METHYL SILOXANE, 230° C, EC

Applicability

Determinative step is applicable to residues of low volatility (late elution) and containing halogen, sulfur, or other electrophilic moieties. It is particularly useful for residues such as pyrethroids, with halogen (permethrin, fenvalerate, deltamethrin) or without halogen (tetramethrin).

Column

Wide bore capillary, 30 m × 0.53 mm id, coated with 100% methyl substituted polysiloxane liquid phase, 1-1.5 µm film thickness, bonded and cross-linked. For example, DB-1; see Table 502-a for equivalents. See Section 502 C, Recommended Operating Procedure for Wide Bore Columns.

Column Operating Conditions:

230° C isothermal; if necessary, adjust temperature so that relative retention time (rrt) to phosalone of cis permethrin is about 1.55.

Carrier gas: helium; adjust flow rate so that phosalone elutes in about 8 min (about 18 mL/min).

Injector temperature: 250° C

Detector

Electron Capture (EC)

Detector Operating Conditions:

350° C

Make-up gas: nitrogen or argon/methane (95:5), at 30 mL/min

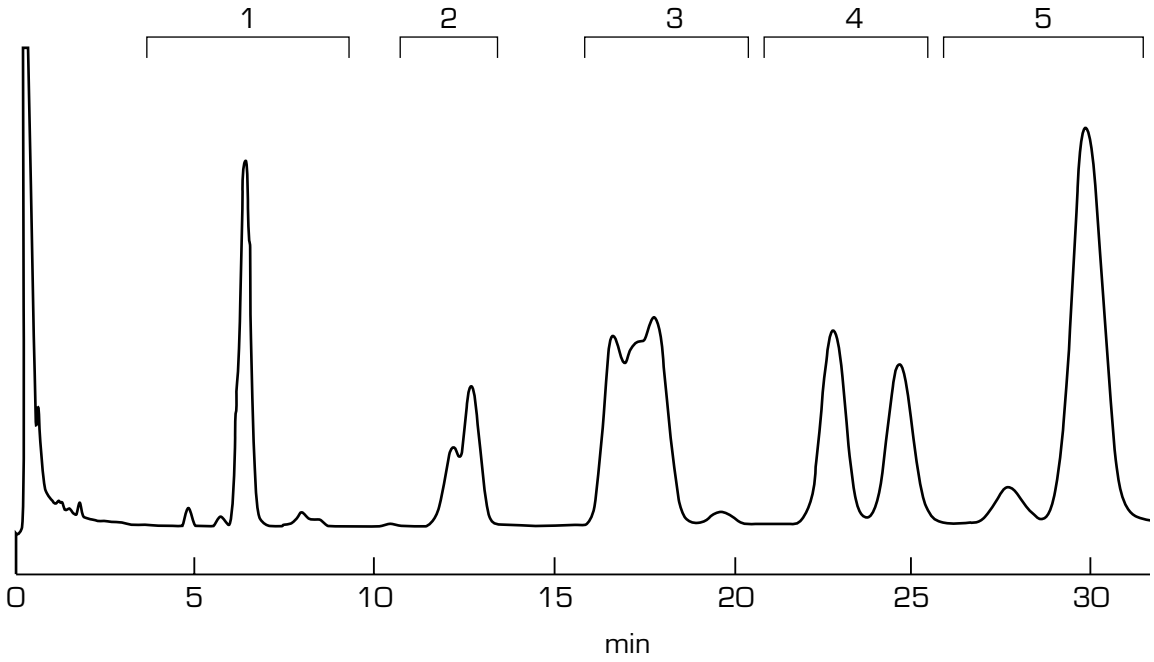
See Section 503 B for other information about EC detector operation.

Set detector electronics (amplification, attenuation) so that response to 0.5 ng phosalone is 50% full scale deflection (FSD).

Other Considerations

Detector sensitivity must be sufficient to measure residues of pyrethroids at ≤0.1 ppm, where some tolerances are set.

Example chromatogram is on next page.



DG10

Chromatogram of: 1) 3.5 ng tetramethrin, 2) 2.3 ng permethrin, 3) 2.1 ng cypermethrin, 4) 1.9 ng fenvalerate, and 5) 2.2 ng deltamethrin at the conditions described.

DG11 GLC, 100% METHYL SILOXANE, 230° C, FPD-P

Applicability

Determinative step is applicable to residues of low volatility (late elution) and containing phosphorus. It is particularly useful for residues such as some organophosphorus pesticides, their oxygen analog sulfones and sulfoxides, and aryl phosphate industrial chemicals.

Column

Wide bore capillary, 30 m × 0.53 mm id, coated with 100% methyl substituted polysiloxane liquid phase, 1-1.5 µm film thickness, bonded and cross-linked. For example, DB-1; see Table 502-a for equivalents. See Section 502 C, Recommended Operating Procedure for Wide Bore Columns.

Column Operating Conditions:

230° C isothermal; if necessary, adjust temperature so that relative retention time (rrt) to phosalone of coumaphos is about 1.56.

Carrier gas: helium; adjust flow rate so that phosalone elutes in about 8.5 min (about 18 mL/min).

Injector temperature: 250° C

Detector

Flame photometric, phosphorus mode (FPD-P)

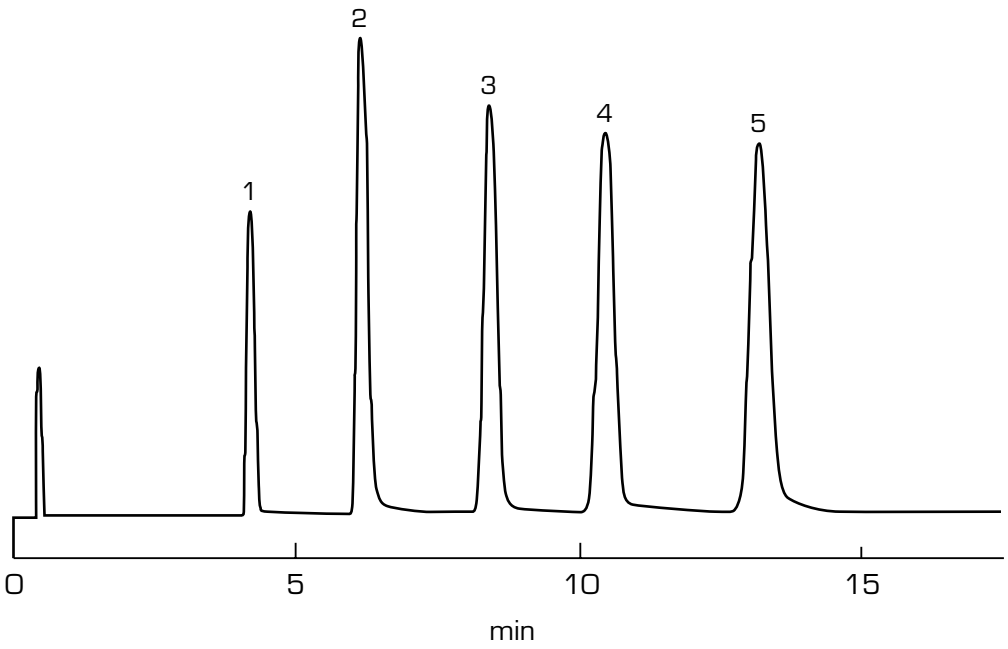
Detector Operating Conditions:

225-250° C

See Section 503 C for other information about FPD operation.

Set detector electronics (amplification, attenuation) so that response to 7.5 ng phosalone is 50% full scale deflection (FSD).

Example chromatogram is on next page.



DG11

Chromatogram of: 1) 1.38 ng ethion, 2) 20.8 ng azinphos-methyl oxygen analog, 3) 7.28 ng phosalone, 4) 7.79 ng pyrazophos, and 5) 10.1 ng coumaphos at the conditions described.

DG12 GLC, 100% METHYL SILOXANE, 230° C, ELCD-X

Applicability

Determinative step is applicable to residues of low volatility (late elution) and containing halogen. It is particularly useful for residues such as halogenated pyrethroids (cyfluthrin, alpha-cypermethrin).

Column

Wide bore capillary, 30 m × 0.53 mm id, coated with 100% methyl substituted polysiloxane liquid phase, 1-1.5 µm film thickness, bonded and cross-linked. For example, DB-1; see Table 502-a for equivalents. See Section 502 C, Recommended Operating Procedure for Wide Bore Columns.

Column Operating Conditions:

230° C isothermal; if necessary, adjust temperature so that relative retention time (rrt) to phosalone of cis permethrin is about 1.55.

Carrier gas: helium or hydrogen; adjust flow rate so that phosalone elutes in about 8 min.

Injector temperature: 250° C

Detector

Electroconductivity, halogen mode (ELCD-X)

Detector Operating Conditions:

Base temperature 250° C; furnace temperature 900° C (or as specified in operating manual)

Reactor gas: hydrogen, flow as required by specific detector model; see operating manual

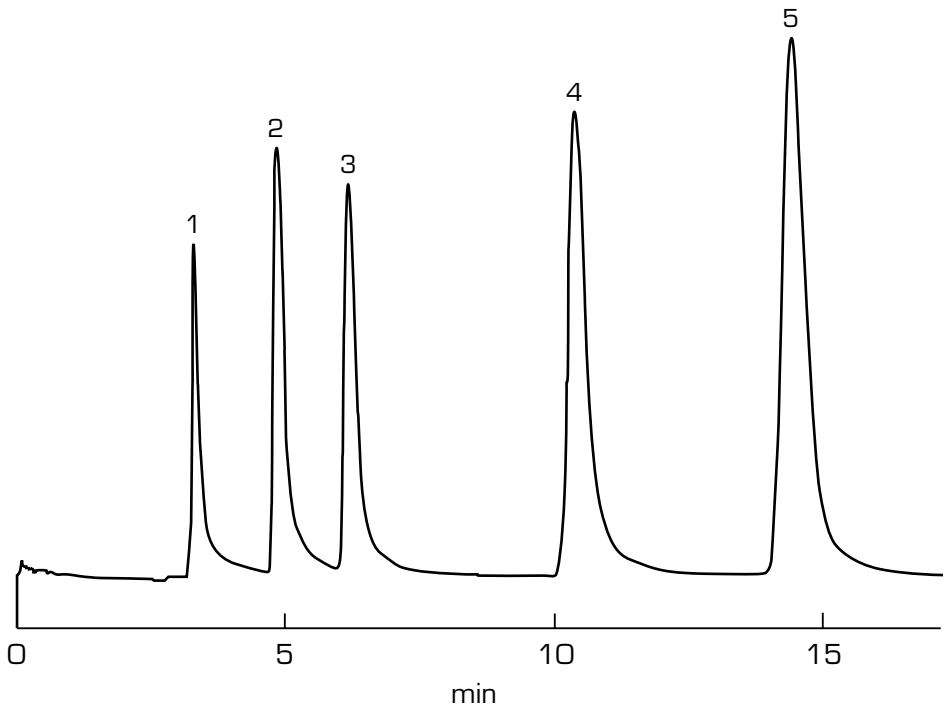
See Section 503 D for other information about ELCD operation.

Set detector electronics (amplification, attenuation) so that response to 18 ng phosalone is 50% full scale defection (FSD).

Other Considerations

Detector sensitivity can probably not be increased to match that of DG10, for the same residues.

Example chromatogram is on next page.



DG12

Chromatogram of: 1) 8.72 ng ofurace, 2) 9.96 ng iprodione, 3) 17.86 ng phosalone, 4) 11.01 ng prochloraz, and 5) 21.06 ng alpha-cypermethrin at the conditions described.

DG13 GLC, 50% PHENYL, 50% METHYL SILOXANE, 200° C, EC

Applicability

Determinative step is applicable to residues containing halogen, sulfur, or other electrophilic moieties. It is a general purpose system, but subject to interferences by nonpesticides.

Column

Wide bore capillary, 30 m m × 0.53 mm id, coated with 50% phenyl, 50% methyl substituted polysiloxane liquid phase, 1-1.5 µm film thickness, bonded and cross-linked. For example, DB-17; see Table 502-a for equivalents. See Section 502 C, Recommedned Operating Procedure for Wide Bore Columns.

Column Operating Conditions:

200° C, isothermal; if necessary, adjust temperature so that relative retention time to chlorpyrifos (rrt_c) of p,p'-DDT is 3.5 ± 0.07 or rrt_c of ethion is 3.36 ± 0.07.

Carrier gas: helium; adjust flow rate so that chlorpyrifos elutes in about 4 ± 0.5 min (about 20 mL/min).

Injector temperature: 250° C

Detector

Electron Capture (EC)

Detector Operating Conditions:

350° C

Make-up gas: nitrogen or argon/methane (95:5), at 30 mL/min

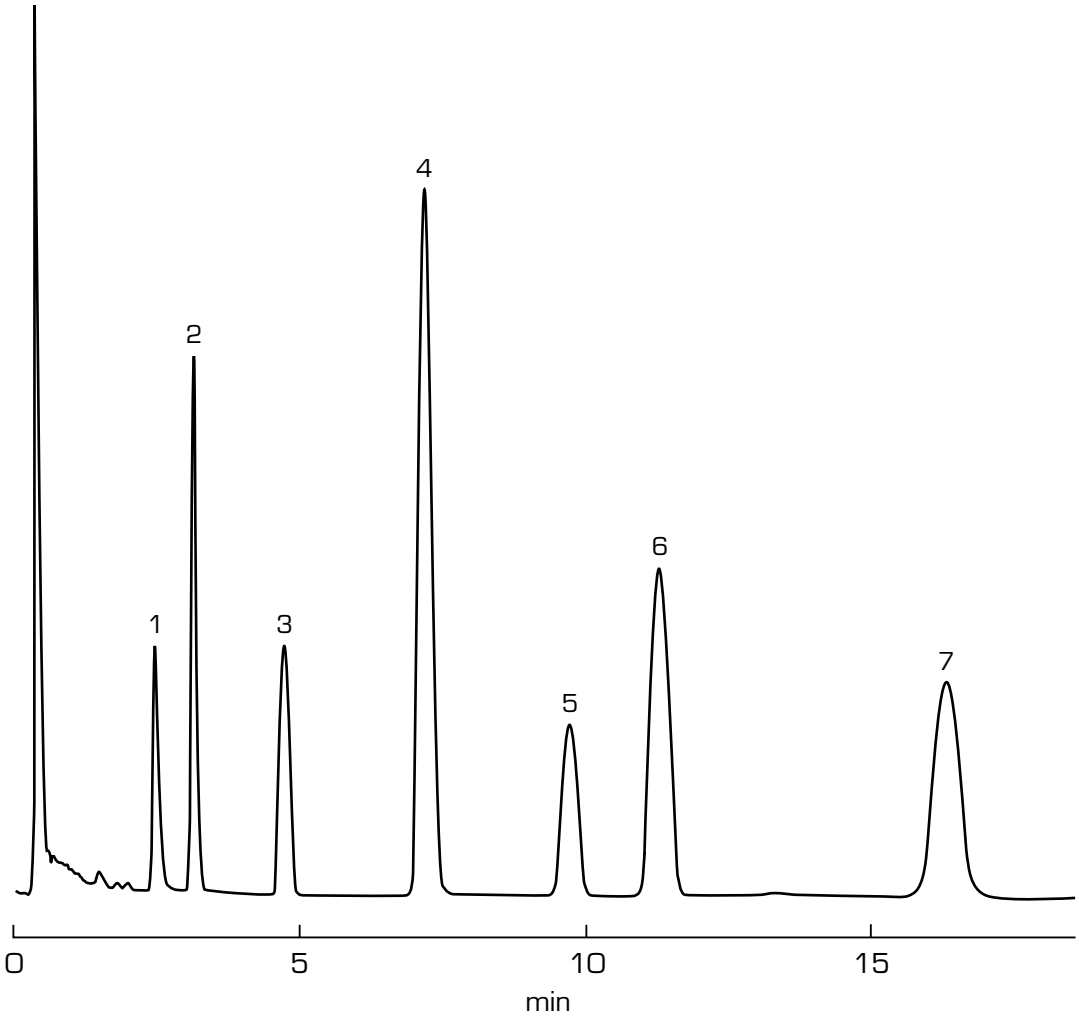
See Section 503 B for other information about EC detector operation.

Set detector electronics (amplification, attenuation) so that response to 0.15 ng chlorpyrifos (on an amount within the detector's linear range) is 50% full scale deflection (FSD).

Other Considerations

Rrt_cs and ng required to cause 50% FSD response are listed in Appendix I, PESTDATA (many data in PESTDATA were collected using equivalent packed column). Response data in Appendix I are based on detector sensitivity of 50% FSD to 1.5 ng chlorpyrifos.

Example chromatogram is on next page.



DG13

Chromatogram of: 1) 0.048 ng dicloran, 2) 0.049 ng heptachlor, 3) 0.15 ng chlorpyrifos, 4) 0.23 ng endosulfan I, 5) 0.22 ng captan, 6) 0.24 ng endrin, and 7) 0.24 ng p,p′-DDT at the conditions described.

DG14 GLC, 50% PHENYL, 50% METHYL SILOXANE, 200° C, FPD-P

Applicability

Determinative step is applicable to residues containing phosphorus. It is particularly useful for residues such as organophosphate pesticides.

Column

Wide bore capillary, 30 mm × 0.53 mm id, coated with 50% phenyl, 50% methyl substituted polysiloxane liquid phase, 1-1.5 µm film thickness, bonded and cross-linked. For example, DB-17; see Table 502-a for equivalents. See Section 502 C, Recommended Operating Procedure for Wide Bore Columns.

Column Operating Conditions:

200° C, isothermal; if necessary, adjust temperature so that relative retention time to chlorpyrifos (rrt_c) of ethion is 3.36 ± 0.07.

Carrier gas: helium; adjust flow rate so that chlorpyrifos elutes in about 4 ± 0.5 min (about 20 mL/min).

Injector temperature: 250° C

Detector

Flame photometric, phosphorus mode (FPD-P)

Detector Operating Conditions:

225-250° C

See Section 503 C for other information about FPD operation.

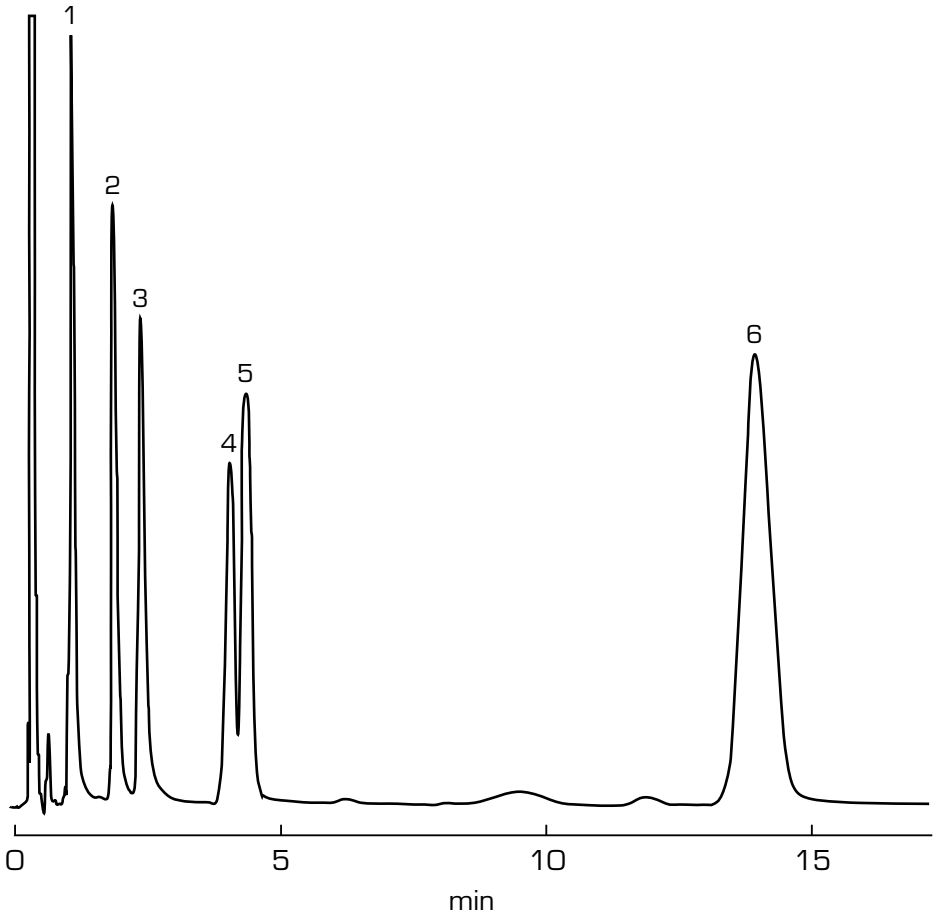
Set detector electronics (amplification, attenuation) so that response to 1.5 ng chlorpyrifos is 50% full scale deflection (FSD).

A properly functioning system will give a response to 1.5 ng omethoate of ≥50% FSD.

Other Considerations

Rrt_cs and ng required to cause 50% FSD response are listed in Appendix I, PESTDATA (many data in PESTDATA were collected using equivalent packed column).

Example chromatogram is on next page.



DG14

Chromatogram of: 1) 1.0 ng acephate, 2) 1.5 ng omethoate, 3) 1.0 ng monocrotophos, 4) 1.0 ng pirimiphos-methyl, 5) 1.0 ng chlorpyrifos, and 6) 3.0 ng ethion at the conditions described.

DG15 GLC, 50% PHENYL, 50% METHYL SILOXANE, 230° C, FPD-S

Applicability

Determinative step is applicable to residues containing sulfur. It is particularly useful for residues such as propargite, thiabendazole, and ethofumesate.

Column

Wide bore capillary, 30 m m × 0.53 mm id, coated with 50% phenyl, 50% methyl substituted polysiloxane liquid phase, 1-1.5 µm film thickness, bonded and cross-linked. For example, DB-17; see Table 502-a for equivalents. See Section 502 C, Recommended Operating Procedure for Wide Bore Columns.

Column Operating Conditions:

200° C, isothermal; if necessary, adjust temperature so that relative retention time to chlorpyrifos (rrt_c) of ethion is 3.36 ± 0.07.

Carrier gas: helium; adjust flow rate so that chlorpyrifos elutes in about 4 ± 0.5 min (about 20 mL/min).

Injector temperature: 250° C

Detector

Flame photometric, sulfur mode (FPD-S)

Detector Operating Conditions:

225-250° C

See Section 503 C for other information about FPD operation.

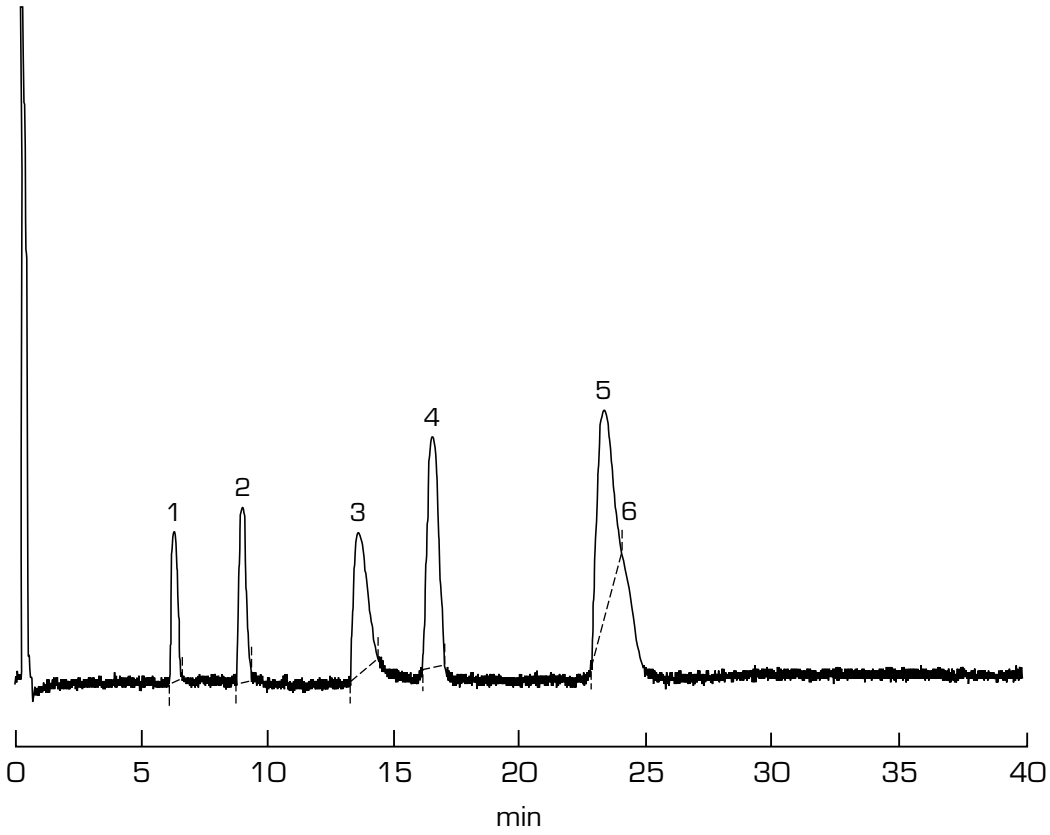
Set detector electronics (amplification, attenuation) to produce greatest possible response (50% full scale deflection [FSD]) to 15 ng chlorpyrifos is reasonable).

Other Considerations

Detector is not linear; quantitation of residues may be calculated from calibration curve (response vs amount injected).

Rrt_cs and ng required to cause 50% FSD response are listed in Appendix I, PESTDATA (many data in PESTDATA were collected using equivalent packed column).

Example chromatogram is on next page.



DG15

Chromatogram of: 1) 2.5 ng ethofumesate, 2) 5.0 ng endosulfan I, 3) 12.5 ng thiabendazole, 4) 10.0 ng endosulfan II, 5) 15.0 ng propargite, and 6) 15.0 ng endosulfan sulfate at the conditions described. Using this system, 5.0 ng chlorpyrifos caused about 50% FSD response.

DG16 GLC, 50% PHENYL, 50% METHYL SILOXANE, 200° C, ELCD-X

Applicability

Determinative step is applicable to residues containing halogen. It is particularly useful for residues such as chlorinated hydrocarbon pesticides and polychlorinated biphenyls.

Column

Wide bore capillary, 30 m m × 0.53 mm id, coated with 50% phenyl, 50% methyl substituted polysiloxane liquid phase, 1-1.5 µm film thickness, bonded and cross-linked. For example, DB-17; see Table 502-a for equivalents. See Section 502 C, Recommended Operating Procedure for Wide Bore Columns.

Column Operating Conditions:

200° C, isothermal; if necessary, adjust temperature so that relative retention time to chlorpyrifos (rrt_c) of p,p¢-DDT is 3.5 ± 0.07.

Carrier gas: helium; adjust flow rate so that chlorpyrifos elutes in about 4 ± 0.5 min (about 20 mL/min).

Injector temperature: 250° C

Detector

Electroconductivity, halogen mode (ELCD-X)

Detector Operating Conditions:

Base temperature 250° C; furnace temperature 900° C (or as specified in operating manual)

Reactor gas: hydrogen, flow as required by specific detector model; see operating manual

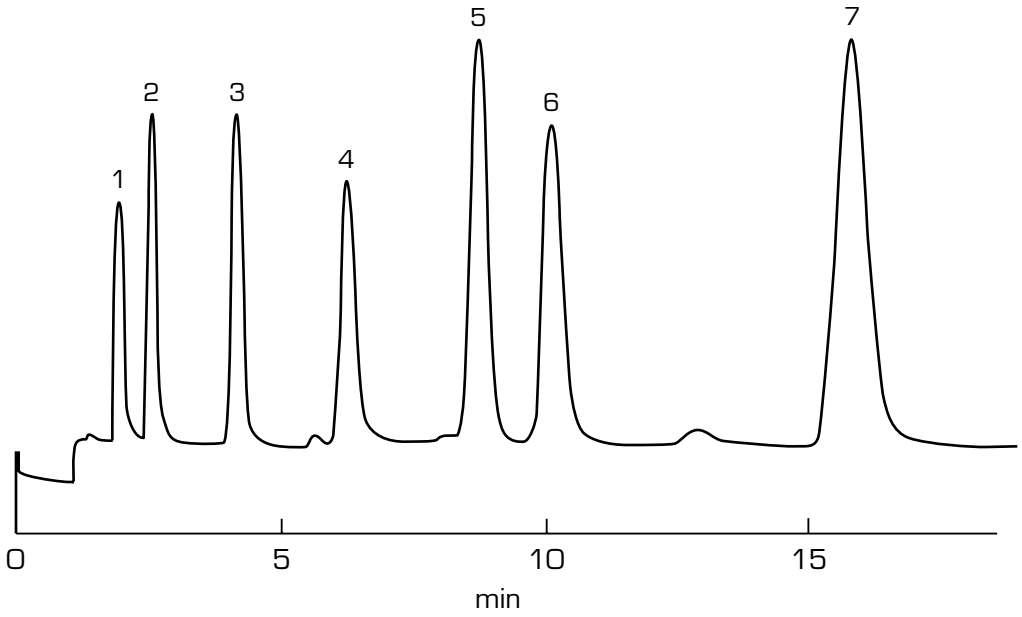
See Section 503 D for other information about ELCD operation.

Set detector electronics (amplification, attenuation) so that response to 1.5 ng chlorpyrifos is 50% full scale deflection (FSD).

Other Considerations

Rrt_cs and ng required to cause 50% FSD response are listed in Appendix I, PESTDATA (many data in PESTDATA were collected using equivalent packed column).

Example chromatogram is on next page.



DG16

Chromatogram of: 1) 0.85 ng dicloran, 2) 0.58 ng heptachlor, 3) 1.65 ng chlorpyrifos, 4) 1.01 ng endosulfan I, 5) 4.58 ng captan, 6) 1.56 ng endrin, and 7) 3.56 ng p,p'-DDT at the conditions described.

DG17 GLC, 50% PHENYL, 50% METHYL SILOXANE, 200° C, N/P

Applicability

Determinative step is applicable to residues containing nitrogen. It is particularly useful for residues such as triazines, triazoles, and THPI (captan metabolite).

Column

Wide bore capillary, 30 m m × 0.53 mm id, coated with 50% phenyl, 50% methyl substituted polysiloxane liquid phase, 1-1.5 µm film thickness, bonded and cross-linked. For example, DB-17; see Table 502-a for equivalents. See Section 502 C, Recommended Operating Procedure for Wide Bore Columns.

Column Operating Conditions:

200° C, isothermal; if necessary, adjust temperature so that relative retention time to chlorpyrifos (rrt_c) of ethion is 3.36 ± 0.07.

Carrier gas: helium; adjust flow rate so that chlorpyrifos elutes in about 4 ± 0.5 min (about 20 mL/min).

Injector temperature: 250° C

Detector

Alkali bead detector, nitrogen selective (N/P)

Detector Operating Conditions:

250° C

3.7 ± 0.1 mL/min hydrogen and 110 mL/min air

See Section 503 E for other information about N/P detector operation.

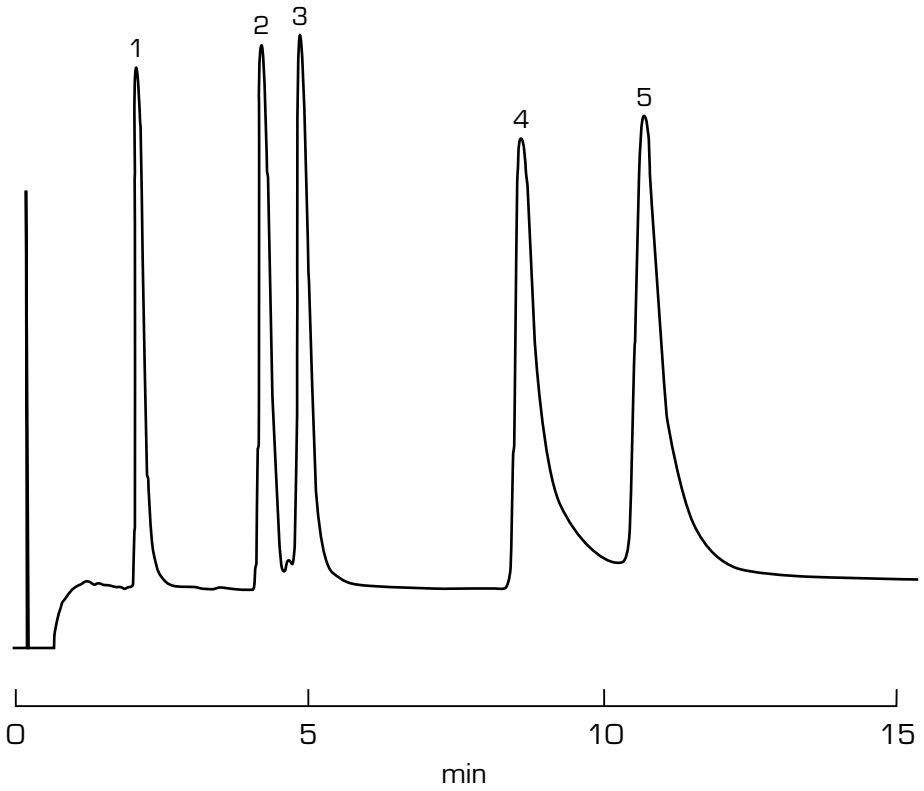
Set detector electronics (amplification, attenuation) so that response to 1.5 ng chlorpyrifos is 50% full scale deflection (FSD).

A properly functioning system will give a response to 1.5 ng omethoate of ≥50% FSD.

Other Considerations

Rrt_cs and ng required to cause 50% FSD response are listed in Appendix I, PESTDATA (many data in PESTDATA were collected using equivalent packed column).

Example chromatogram is on next page.



DG17

Chromatogram of: 1) 1.5 ng atrazine, 2) 1.5 ng chlorpyrifos, 3) 15.0 ng carbaryl, 4) 10.0 ng imazalil, and 5) 5.0 ng procyazine at the conditions described.

DG18 GLC, 50% CYANOPROPYLPHENYL, 50% METHYL SILOXANE, 200° C, EC

Applicability

Determinative step is applicable to residues containing halogen, sulfur, or other electrophilic moieties. It is a general purpose system, subject to interferences from nonpesticides; it is particularly useful for separating BHC isomers and hexachlorobenzene.

Column

Wide bore capillary, 30 m × 0.53 mm id, coated with 50% cyanopropylphenyl, 50% methyl substituted polysiloxane liquid phase, 1-1.5 µm film thickness, bonded and cross-linked. For example, DB-225; see Table 502-a for equivalents. See Section 502 C, Recommended Operating Procedure for Wide Bore Columns.

Column Operating Conditions:

200° C, isothermal; if necessary, adjust temperature so that relative retention time to chlorpyrifos (rrt_c) of lindane is 0.69 ± 0.02 and p,p¢-DDT is 3.6 ± 0.06 or rrt_c of ethion is 3.9 ± 0.1.

Carrier gas: helium; adjust flow rate so that chlorpyrifos elutes in about 5.5 ± 0.5 min (about 20 mL/min).

Injector temperature: 250° C

Detector

Electron Capture (EC)

Detector Operating Conditions:

350° C

Make-up gas: nitrogen or argon/methane (95:5), a 30 mL/min

See Section 503 B for other information about EC detector operation.

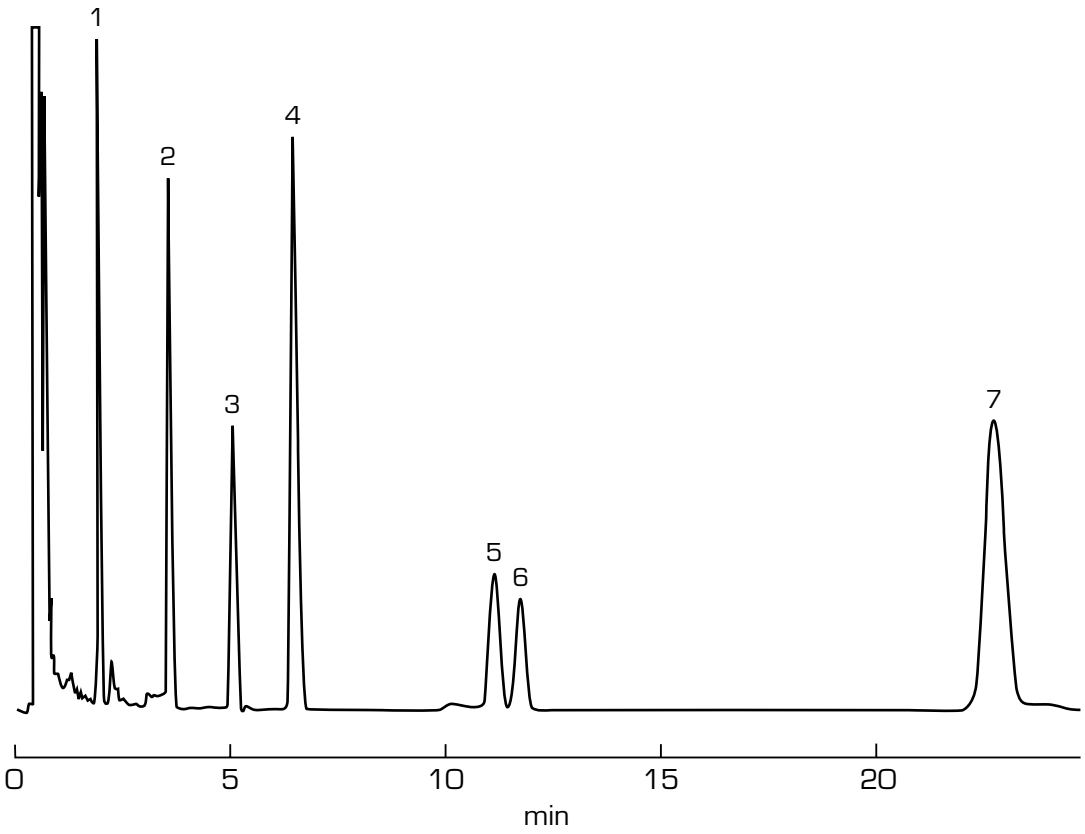
Set detector electronics (amplification, attenuation) so that response to 0.15 ng chlorpyrifos (on an amount within the detector's linear range) is 50% full scale deflection (FSD).

Other Considerations

Columns containing cyano moieties in the phase must not be connected to nitrogen selective or electrolytic conductivity detectors, so this column cannot be used with a different detector to confirm residues tentatively identified using this system.

Rrt_cs and ng required to cause 50% FSD response are listed in Appendix I, PESTDATA (many data in PESTDATA were collected using equivalent packed column). Response data in Appendix I are based on detector sensitivity of 50% FSD to 1.5 ng chlorpyrifos.

Example chromatogram is on next page.



DG18

Chromatogram of: 1) 0.032 ng hexachlorobenzene, 2) 0.049 ng α-BHC, 3) 0.056 lindane, 4) 0.15 ng chlorpyrifos, 5) 0.054 ng β-BHC, 6) 0.054 ng δ-BHC, and 7) 0.201 ng p,p′-DDT at the conditions described.

DG19 GLC, 50% CYANOPROPYLPHENYL, 50% METHYL SILOXANE, 200° C, FPD-P

Applicability

Determinative step is applicable to residues containing phosphorus. It is particularly useful for residues such as organophosphate pesticides.

Column

Wide bore capillary, 30 m × 0.53 mm id, coated with 50% cyanopropylphenyl, 50% methyl substituted polysiloxane liquid phase, 1-1.5 µm film thickness, bonded and cross-linked. For example, DB-225; see Table 502-a for equivalents. See Section 502 C, Recommended Operating Procedure for Wide Bore Columns.

Column Operating Conditions:

200° C, isothermal; if necessary, adjust temperature so that relative retention time to chlorpyrifos (rrt_c) of ethion is 3.9 ± 0.1.

Carrier gas: helium; adjust flow rate so that chlorpyrifos elutes in about 5.5 ± 0.5 min (about 20 mL/min).

Injector temperature: 250° C

Detector

Flame photometric, phosphorus mode (FPD-P)

Detector Operating Conditions:

225-250° C

See Section 503 C for other information about FPD operation.

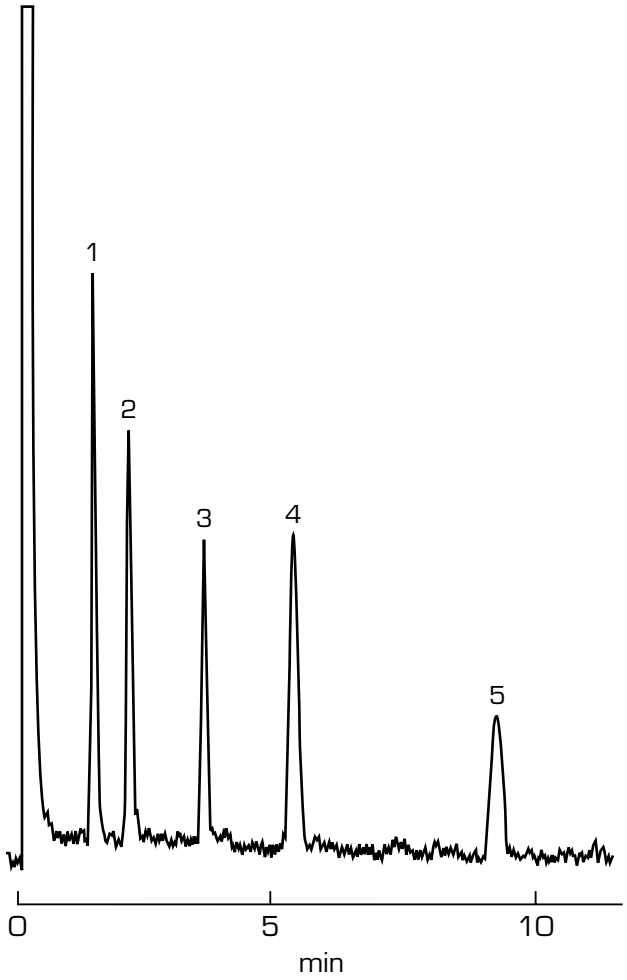
Set detector electronics (amplification, attenuation) so that response to 1.5 ng chlorpyrifos is 50% full scale deflection (FSD).

Other Considerations

Columns containing cyano moieties in the phase must not be connected to nitrogen selective or electrolytic conductivity detectors, so this column cannot be used with a different detector to confirm residues tentatively identified using this system.

Rrt_cs and ng required to cause 50% FSD response are listed in Appendix I, PESTDATA (many data in PESTDATA were collected using equivalent packed column).

Example chromatogram is on next page.



DG19

Chromatogram of: 1) 0.5 ng methamidophos, 2) 1.0 ng diazinon, 3) 1.0 ng acephate, 4) 1.5 ng chlorpyrifos, and 5) 1.0 ng monocrotophos at the conditions described.

303: METHOD II FOR NONFATTY FOODS

BASIC REFERENCE

Mills, P.A., *et al.* (1963) *J. Assoc. Off. Agric. Chem.* **46**, 186-191

GENERAL PRINCIPLES

Residues are extracted by blending with acetonitrile or water and acetonitrile, then transferred into petroleum ether by liquid-liquid partitioning. Subsequent cleanup of the extract with Florisil column chromatography results in an extract suitable for determination by GLC; two elution systems produce different elution patterns, useful in confirmatory or additional analyses.

The amount of sample represented in the final solution is calculated from the aliquot of acetonitrile extract used and the proportion of petroleum ether retrieved from the partitioning step; this calculation is valid only when the original filtered extract is homogeneous. Variations in the extraction step are used for products of high (>5%) sugar content to ensure homogeneity.

APPLICABILITY

Consult Guide to PAM I for additional information pertinent to the appropriate application of multiresidue methodology.

Method is generally applicable to relatively nonpolar residues in nonfatty commodities, i.e., fruits and vegetables containing ≤2 g fat in 100 g sample. Extraction E1 is applicable to products with high moisture (>75%) content; that extraction is also applicable to eggs if sample size is reduced (Extraction E2). Extraction E3 is applicable to dry products (<75% water), E4 to products with 5-15% sugar, and E5 to products with >15% sugar. See Section 201 for percentages fat, water, and sugar of many commodities. Florisil cleanup step prevents applicability to very polar residues. See Table 303-a, following the method description, for results of recovery tests.

METHOD MODULES

Choose from these method modules, using Figure 303-a for guidance:

Extraction (E)			**Recommended Use**
E1	(p. 303-7)	Extraction with acetonitrile, partition into petroleum ether with high moisture	fruits and vegetables (>75%), and low sugar (<5%), low fat (<2%)
E2	(p. 303-8)	Extraction from eggs with acetonitrile, partition into petroleum ether	whole eggs
E3	(p. 303-9)	Extraction with water/acetonitrile, partition into petroleum ether	dried egg whites, grains, and other foods with low moisture (<75%), low fat (<2%)
E4	(p. 303-9)	Extraction with acetonitrile and water, partition into petroleum ether	fruits and other foods with high sugar (5-15%)
E5	(p. 303-10)	Extraction with heated acetonitrile and water, partition into petroleum ether	fruits and other foods with very high sugar (>15%)

Cleanup (C)

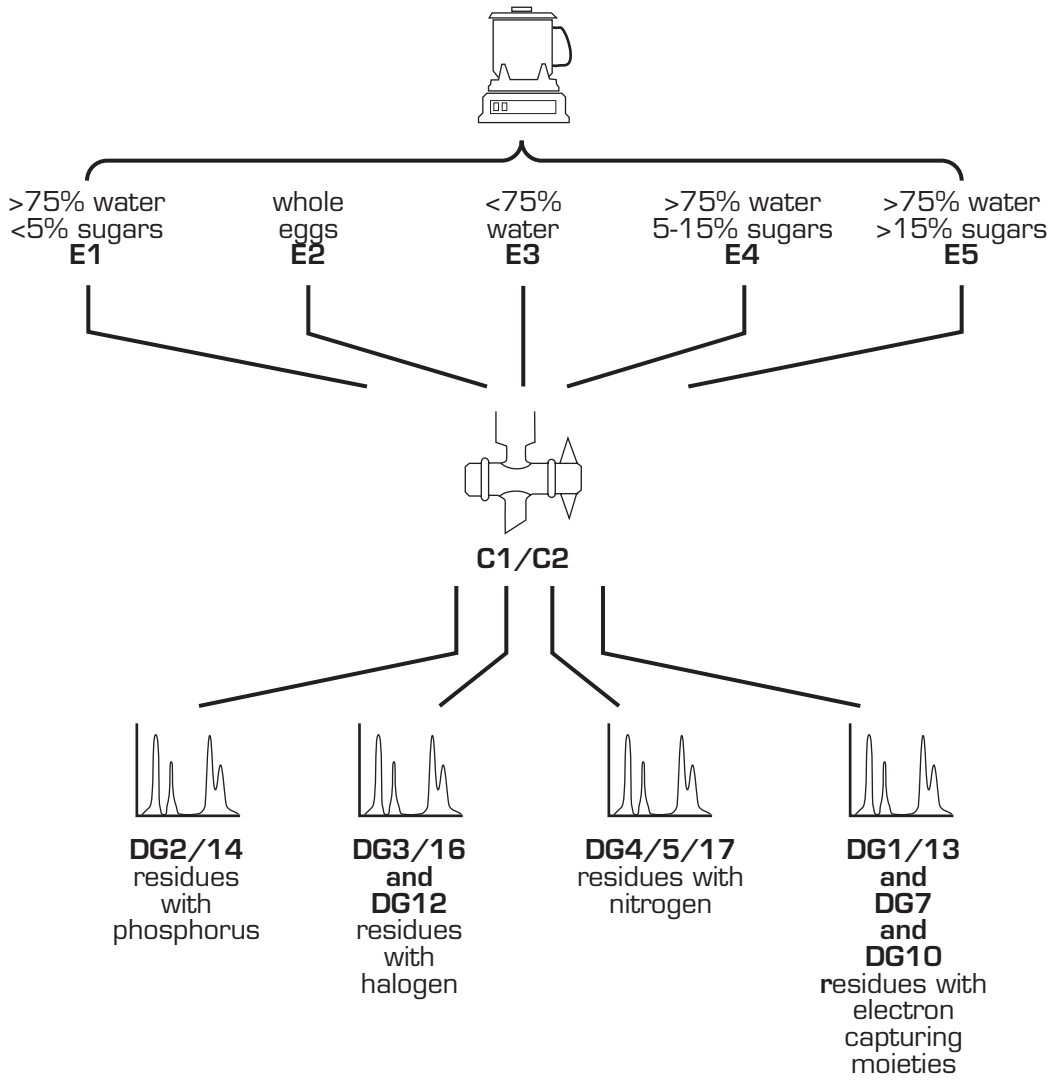
C1	(p. 303-13)	Florisil column cleanup, with three ethyl ether/petroleum ether eluants	for relatively nonpolar residues
C2	(p. 303-14)	Florisil column cleanup, with three methylene chloride eluants	alternative to C1, some additional residues recovered

Determination (D)

(See Section 302 for full details of GLC modules.)

DG 1	(p. 302-33)	GLC, 100% methyl siloxane column, 200°, EC detector	residues with halogen, sulfur, other moieties
DG 2	(p. 302-35)	GLC, 100% methyl siloxane column, 200°, FPD-P	residues with phosphorus
DG 3	(p. 302-37)	GLC, 100% methyl siloxane column, 200°, ELCD-X	residues with halogen
DG 4	(p. 302-39)	GLC, 100% methyl siloxane column, 200°, ELCD-N	residues with nitrogen
DG 5	(p. 302-41)	GLC, 100% methyl siloxane column, 200°, N/P detector	residues with nitrogen or phosphorus
DG 7	(p. 302-45)	GLC, 100% methyl siloxane column, 130°, EC detector	early eluting residues with halogen, sulfur, other moieties
DG10	(p. 302-51)	GLC, 100% methyl siloxane column, 230°, EC detector	late eluting residues with halogen, sulfur, other moieties
DG12	(p. 302-55)	GLC, 100% methyl siloxane column, 230°, ELCD-X	late eluting residues with halogen
DG13	(p. 302-57)	GLC, 50% phenyl, 50% methyl siloxane column, 200°, EC detector	residues with halogen, sulfur, other moieties
DG14	(p. 302-59)	GLC, 50% phenyl, 50% methyl siloxane column, 200°, FPD-P	residues with phosphorus
DG16	(p. 302-63)	GLC, 50% phenyl, 50% methyl siloxane column, 200°, ELCD-X	residues with halogen
DG17	(p. 302-65)	GLC, 50% phenyl, 50% methyl siloxane column, 200°, N/P detector	residues with nitrogen or phosphorus

Figure 303-a
Recommended Approach: Nonfatty Foods



VALIDATION

Many combinations of method modules are possible. The following combinations have undergone interlaboratory validation and are recommended for use:

E1 + C1 + DG1

Validation reports:

Krause, R.T. (1966) *J. Assoc. Off. Anal. Chem.* **49**, 460-463. Collaborative study leading to AOAC official final action status for aldrin, DDE, and methoxychlor in potatoes.

Gaul, J. (1966) *J. Assoc. Off. Anal. Chem.* **49**, 463-467. Collaborative study leading to AOAC official final action status for lindane, heptachlor, and TDE in endive and cauliflower.

Davidson, A.W. (1966) *J. Assoc. Off. Anal. Chem.* **49**, 468-472. Collaborative study leading to AOAC official final action status for BHC, p,p'-DDT, and endrin on apricots and strawberries.

Wells, C. (1967) *J. Assoc. Off. Anal. Chem.* **50**, 1205-1215. Interlaboratory study supporting validity of method for 32 residues in five groups of nonfatty commodities; studies supported extension of official status for previously collaborated resi dues in 13 additional commodities.

Burke, J.A. (1968) *J. Assoc. Off. Anal. Chem.* **51,** 311-314. Interlaboratory study supporting validity of method for nine residues in 21 nonfatty foods; studies supported extension of official status for previously collaborated residues to 15 additional commodities.

Krause, R.T. (1973) *J. Assoc. Off. Anal. Chem.* **56**, 721-727. Collaborative study leading to AOAC official final action status for dieldrin, heptachlor epoxide, mirex, and Perthane in apples and cauliflower.

E1 + C1 + (predecessor to) DG5

Validation reports:

Wessel, J.R. (1967) *J. Assoc. Off. Anal. Chem.* **50**, 430-439. Collaborative study lead ing to AOAC official final action status for diazinon, ethion, malathion, parathion, parathion-methyl, and ronnel in lettuce and apples.

Finsterwalder, C. W. (1976) *J. Assoc. Off. Anal. Chem.* **59**, 169-171. Collaborative study leading to AOAC official final action status for parathion in kale.

Wells, C. (1967) *J. Assoc. Off. Anal. Chem.* **50**, 1205-1215. Interlaboratory study supporting validity of method for 32 residues in five groups of nonfatty commodities; studies were later referenced (Burke, J.A. (1971) *J. Assoc. Off. Anal. Chem.* **54**, 325-327) as support for extension of official status for diazinon, ethion, malathion, parathion, parathion-methyl, and ronnel in barley, broccoli, cabbage, carrots, cauliflower, cucumbers, grapes, green peppers, mustard greens, oats, potatoes, squash, tomatoes, turnips, turnip greens, and wheat.

E2 + C1 + DG1

Validation report:

Finsterwalder, C. W. (1976) *J. Assoc. Off. Anal. Chem.* **59**, 169-171. Collaborative study leading to AOAC official final action status for o,p'-DDT, p,p'-DDT, and p,p'-DDE in kale and p,p'-DDE, dieldrin, lindane, and heptachlor epoxide in eggs.

E3 + C1 + DG1

Validation reports:

Burke, J.A. (1971) *J. Assoc. Off. Anal. Chem.* **54**, 325-327. Referenced the following publications in a recommendation to extend official status to extraction step E3 (for low moisture commodities) for previously collaborated residues on barley, corn meal, hay, oats, popcorn, and wheat:

Bertuzzi, P., *et al.* (1967) *J. Assoc. Off. Anal. Chem.* **50**, 623-627

Wilderman, M., and Shuman, H., (1968) *J. Assoc. Off. Anal. Chem.* **51**, 892-895

Burke, J.A., *et al.* (1971) *J. Assoc. Off. Anal. Chem.* **54**, 142-146. Evaluation of two extraction procedures.

E4 + C1 + DG1 and E5 + C1 + DG1

Validation report:

Burke, J.A. (1970) *J. Assoc. Off. Anal. Chem.* **53**, 355-357. Referenced the following publication in a recommendation to extend official status to extraction steps E4 and E5 (for commodities with high sugar) for previously collaborated residudues:

Porter, M.L., and Burke, J.A. (1969) *J. Assoc. Off. Anal. Chem.* **52**, 1280-1283. Description of way to accommodate commodities with high sugar.

For all combinations above, AOAC official method reference: Official Methods of Analysis of the AOAC (1990) 15th ed., 970.52 A, B, E, H, I, J, K, O, and R.

E1 + C2 + DG1

Validation reports:

Mitchell, L.E. (1976) *J. Assoc. Off. Anal. Chem.* **59**, 209-212. Collaborative study leading to AOAC official final action status for endosulfan, endosulfan sulfate, tetradifon, and tetrasul in apples and cucumbers.

McMahon, B.M. (1988) *J. Assoc. Off. Anal. Chem.* **71**, 94-97. Presentation of additional validation data for previously collaborated residues to 29 additional commodities.

AOAC official method reference: *Official Methods of Analysis of the AOAC* (1990) 15th ed., 976.23.

E1 EXTRACTION WITH ACETONITRILE, PARTITION INTO PETROLEUM ETHER

References

Mills, P.A., *et al.* (1963) *J. Assoc. Off. Agric. Chem.* **46**, 186-191
Porter, M., *et al.* (1967) *J. Assoc. Off. Anal. Chem.* **50**, 644-645

Principles

Residues are extracted from high moisture products by a single blending with acetonitrile, a solvent miscible with the water in high moisture products and also capable of dissolving organic residues. An aliquot of filtered extract is diluted with water and residues are transferred into petroleum ether by liquid-liquid partitioning; transfer to hydrocarbon solvent and removal of all traces of polar acetonitrile permit subsequent cleanup on Florisil. Amount of sample represented in final solution is calculated from aliquot of acetonitrile extract used and proportion of petroleum ether retrieved from partitioning step.

Apparatus

blender, high speed; explosion-proof Waring Blendor, 1 qt jar

Büchner funnel (Büchner), porcelain, 12 cm diameter

filter paper, Shark Skin®, to fit Büchner

graduated cylinders (graduates), glass-stoppered (g-s), 100 mL, and plain, 250 mL

Kuderna-Danish concentrator (K-D), 500 mL, with Snyder column, graduated or plain receiving flask

separatory funnel (separator), 1 L

vacuum filtration flask, 500 mL

Reagents

acetonitrile, distilled from all-glass apparatus; see Section 204 for distillation directions

boiling chips, 20-30 mesh carborundum

petroleum ether, distilled from all-glass apparatus

sodium sulfate, anhydrous, granular, reagent grade; see Section 204 for handling directions

Directions

- Chop or blend representative sample. Weigh 100 g sample into blender jar and add 200 mL acetonitrile (10 g Celite may be added as filter aid).
- Blend 2 min at high speed and filter with suction through Büchner fitted with filter paper into vacuum filtration flask.
- Transfer filtrate to 250 mL graduate and record volume (F).
- Transfer measured volume of filtrate to 1 L separator.
- Carefully measure 100 mL petroleum ether in same graduate used to measure filtered extract and pour into separator containing extract. Shake vigorously 1-2 min.

- Add 10 mL saturated sodium chloride solution and 600 mL water. Hold separator in horizontal position and mix vigorously 30-45 sec. (Inadequate mixing may lead to low recoveries of some pesticides, e.g., BHC, TDE.)
- Let layers separate, discard aqueous layer, and gently wash solvent layer with two 100 mL portions water. Discard washings, transfer solvent layer to 100 mL g-s graduate, and record volume (P).
- Add about 15 g sodium sulfate to graduate, stopper, and shake vigor ously. Do not let extract remain with sodium sulfate >1 hr or losses of organochlorine pesticides by adsorption may result.
- Transfer solution directly to Florisil column, C1 or C2, or concentrate to 5-10 mL in K-D for transfer.
- Calculate weight sample placed on Florisil column as:

$$g = S \times \frac{F}{T} \times \frac{P}{100}$$

where:

S = g sample extracted

F = volume of filtered acetonitrile extract

T = total volume (mL water in sample + mL acetonitrile added – correction in mL for volume contraction). 5 mL contraction volume is used for 80-95 mL water + 200 mL acetonitrile.

P = mL petroleum ether extract recovered

100 = mL petroleum ether into which residues were partitioned.

See Section 201 for percentage water in commodity; 85% may be assumed for most fruits and vegetables.

ALTERNATIVES:

E2 EXTRACTION FROM EGGS WITH ACETONITRILE, PARTITION INTO PETROLEUM ETHER

Additional Reference

Wessel, J.R. (1969) *J. Assoc. Off. Anal. Chem.* **52**, 172-175

Directions

- Blend combined yolks and whites of whole eggs at low speed at least 5 min or until sample is homogeneous. Low speed blending will minimize foaming or whipping of sample.
- Weigh ≤25g thoroughly mixed yolks and whites into blender jar and add 200 mL acetonitrile.
- Proceed as in E1, "Blend 2 min at high speed... ."
- Calculate g sample to be placed on Florisil column as in E1, except:

  T = 215 (15 mL water in 25 g whole eggs + 200 mL acetonitrile; contraction volume is negligible).

E3 EXTRACTION WITH 35% WATER/ACETONITRILE, PARTITION INTO PETROLEUM ETHER

Additional Reference

Bertuzzi, P.A., *et al.* (1967) *J. Assoc. Off. Anal. Chem.* **50**, 623-627

Principles

Residues are extracted from nonfatty, low moisture products by blending with 35% water/acetonitrile. Water in extractant is needed to obtain adequate extraction of residues from low moisture products. Aliquot (≤250 mL) of filtered extract is diluted with water and residues are transferred into petroleum ether by liquid-liquid partitioning; transfer to hydrocarbon solvent and removal of all traces of polar acetonitrile permit subsequent cleanup on Florisil. Water:acetonitrile dilution ratio of 4:1 is used for transfer of residues to petroleum ether; restricting volume of filtered extract to ≤250 mL allows extract, adequate water for dilution, and 100 mL petroleum ether to fit in 1 L separator.

Directions

- Grind sample to pass 20 mesh sieve.
- Weigh 20-25 g sample into blender jar, and add 350 mL 35% water/acetonitrile (10 g Celite may be added as filter aid). If larger sample is required, add sufficient additional extraction mixture to wet sample and permit thorough blending.
- Blend 5 min at high speed and filter with suction through Büchner fitted with filter paper into vacuum filtration flask.
- Take ≤250 mL filtered extract for analysis. Record volume (F).
- Continue as in E1, "Transfer measured volume of filtrate to 1 L separator…"
- Calculate g sample to be placed on Florisil column as in E1, except:

  T = mL water in sample + mL 35% water/acetonitrile. No correction for volume contraction is needed. If water content of sample is <10%, disregard it and use T = volume extracting mixture.

E4 EXTRACTION WITH ACETONITRILE AND WATER, PARTITION INTO PETROLEUM ETHER

Additional Reference

Porter, M.L., and Burke, J.A. (1969) *J. Assoc. Off. Anal. Chem.* **52**, 1280-1283

Principles

Residues are extracted from nonfatty, high moisture commodities that contain 5-15% sugar by addition of water and subsequent blending with acetonitrile. Water is added to the product to dilute the effect of sugar, which can cause separation of water and acetonitrile phases in the filtered extract and thus disrupt homogeneity of the extract solution. Aliquot (≤250 mL) of filtered extract is diluted with water and residues are transferred into petroleum ether by liquid-liquid partitioning; transfer to hydrocarbon solvent and removal of all traces of polar acetonitrile permit subsequent cleanup on Florisil.

Directions

- Weigh 100 g sample into blender jar and add 200 mL acetonitrile and 50 mL water.
- Blend 2 min at high speed and filter with suction through Büchner fitted with filter paper into vacuum filtration flask.
- Transfer ≤250 mL filtered extract to 250 mL graduate. Record volume (F).
- Continue as in E1, "Transfer measured volume to 1 L separator."
- Calculate g sample to be placed on Florisil column as in E1, except:

  T = mL water in sample + mL acetonitrile added + mL water added – correction in mL for volume contraction. When 50 mL water is added, T is 325 for foods of 85% water content. Contraction volumeof 5 mL is used for 80-95 mL water + 200 mL acetonitrile.

E5 EXTRACTION WITH HEATED ACETONITRILE AND WATER, PARTITION INTO PETROLEUM ETHER

Additional Reference

Porter, M.L., and Burke, J.A. (1969) *J. Assoc. Off. Anal. Chem.* **52**, 1280-1283

Principles

Residues are extracted from commodities with >15% sugar using a heated mixture of water and acetonitrile. Unheated water and acetonitrile (as in E4) is insufficient to prevent separation of water and acetonitrile phases in the filtered extract when sugar is >15%. Aliquot (≤250 mL) of filtered extract is diluted with water and residues are transferred into petroleum ether by liquid-liquid partitioning; transfer to hydrocarbon solvent and removal of all traces of polar acetonitrile permit subsequent cleanup on Florisil.

Directions

- Weigh 100 g sample into blender jar and add heated (75° C) mixture of 200 mL acetonitrile and 50 mL water.
- To analyze raisins, weigh 50 g. Heat 50 mL water and 200 mL acetonitrile separately to 75° C. Add 40-50 mL hot water to container in which raisins were weighed and stir or shake to disperse in water. Transfer to blender jar, using remaining water to rinse container into blender jar. Rinse container with hot acetonitrile and add to blender jar; add remaining hot acetonitrile to blender jar.
- Blend 2 min at high speed and filter with suction through Büchner fitted with filter paper into vacuum filtration flask.
- Before filtered extract cools, transfer ≤250 mL to 250 mL graduate. Record volume (F).
- Continue as in E1, "Transfer measured volume of filtrate to 1 L separator…"
- Calculate g sample to be placed on Florisil column as in E1, except:

  T = mL water in sample + mL acetonitrile added + mL water added – correction in mL for volume contraction. When 50 mL water added, T is 325 for foods of 85% water content. Contraction volume of 5 mL is used for 80-95 mL water + 200 mL acetonitrile.

C1 FLORISIL COLUMN CLEANUP, WITH THREE ETHYL ETHER/ PETROLEUM ETHER ELUANTS

Reference

Mills, P.A., *et al.* (1963) *J. Assoc. Off. Agric. Chem.* **46**, 186-191

Principles

Residues in solution are separated from sample co-extractives on a column of Florisil adsorbent; eluants of increasing polarity sequentially remove residues from the column.

Apparatus

chromatographic column, 22 mm id ¥ 300 mm, Teflon stopcock, coarse porosity fritted disc

Kuderna-Danish concentrator (K-D), 500 mL, with Snyder column, two-ball micro-Snyder column, volumetric or graduated receiving flask

Reagents

boiling chips, 20-30 mesh carborundum

ethyl ether, distilled from all-glass apparatus, with 2% ethanol as preservative; see Section 204 for peroxide test

Florisil, PR Grade; see Section 204 for handling and testing directions and calculation of lauric acid value

petroleum ether, distilled from all-glass apparatus

sodium sulfate, anhydrous, granular, reagent grade; see Section 204 for handling directions

eluants: 6% (v/v) ethyl ether/petroleum ether

15% (v/v) ethyl ether/petroleum ether

50% (v/v) ethyl ether/petroleum ether

Directions

- Place activated Florisil (4" or weight determined by lauric acid value) in 22 mm id column; add about 0.5" sodium sulfate. Prewet column with 40-50 mL petroleum ether. Place K-D with volumetric or graduated receiving flask under column to receive eluate.
- Transfer sample extract solution to column, letting it pass through at about 5 mL/min. Rinse container (and sodium sulfate if present) with two 5 mL portions petroleum ether, transfer rinsings to column, and rinse walls of chromatographic tube with additional small portions petroleum ether.
- Elute column at about 5 mL/min with 200 mL 6% ethyl ether/ petroleum ether eluant.
- Change K-Ds and elute at about 5 mL/min with 200 mL 15% ethyl ether/petroleum ether eluant.
- Change K-Ds and elute at about 5 mL/min with 200 mL 50% ethyl ether/petroleum ether eluant.

- Add boiling chips to K-Ds and concentrate each eluate to suitable definite volume. When volume <5 mL is needed, use two-ball micro-Snyder or micro-Vigreaux column during final evaporation in receiving flask.
- Use appropriate determinative steps, such as DG1 or DG13, DG7, and DG10, to identify and measure residues.

ALTERNATIVE:

C2 FLORISIL COLUMN CLEANUP, WITH THREE METHYLENE CHLORIDE ELUANTS

Additional Reference

Mills, P.A., *et al.* (1972) *J. Assoc. Off. Anal. Chem.* **55**, 39-43

Additional Reagents

acetonitrile, distilled from all-glass apparatus; see Section 204 for distillation directions

hexane, distilled from all-glass apparatus

methylene chloride, distilled from all-glass apparatus

eluants: 1—20% methylene chloride/hexane (v/v). Dilute 200 mL methylene chloride with hexane. Allow mixture to reach room temperature, and adjust volume to 1 L with hexane.

2—50% methylene chloride/0.35% acetonitrile/49.65% hexane (v/v/v). Pipet 3.5 mL acetonitrile into 500 mL methylene chloride and dilute with hexane. Allow mixture to reach room temperature and adjust to 1 L with hexane.

3—50% methylene chloride/1.5% acetonitrile/48.5% hexane (v/v/v). Pipet 15 mL acetonitrile into 500 mL methylene chloride and dilute with hexane. Allow mixture to reach room temperature and adjust to 1 L with hexane.

Directions

- Prepare Florisil column as in C1. Place K-D with volumetric or graduated receiving flask under column to receive eluate.
- Transfer sample extract solution to column, letting it pass through at about 5 mL/min. Rinse container (and sodium sulfate if present) with two 5 mL portions hexane, transfer rinsings to column, and rinse walls of chromatographic tube with additional small portions hexane.
- Elute column at about 5 mL/min with 200 mL eluant 1.
- Change K-Ds and elute with 200 mL eluant 2.
- Change K-Ds and elute with 200 mL eluant 3.
- Add boiling chips to K-Ds and concentrate each eluate to suitable definite volume. When volume <5 mL is needed, use two-ball micro-Snyder or micro-Vigreaux column during evaporation.
- Use appropriate determinative steps to identify and measure residues.

DETERMINATION

Inject concentrated extract equivalent to 20 mg (whole product) into the following GLC systems (Section 302) for determination of residues.

Minimum recommended determinations:

DG1 or DG13	residues with halogen, sulfur, or other moieties
or	
DG3 or DG16	organohalogen residues
DG10	late eluting residues, especially pyrethroids
DG12	late eluting organohalogen residues, especially pyrethroids
DG7	early eluting residues with halogen, sulfur, or other moietites
DG2 or DG14	organophosphorus residues; large amounts of sulfur may interfere
DG4	organonitrogen residues; selective to nitrogen, but coextractives may contain nitrogen
DG5 or DG17	organonitrogen and organophosphorus residues

For accurate quantitation, reference standards should be dissolved in same solvent as concentrated extract, only peaks >10% FSD should be measured, and peak sizes of residue and reference standard should match within ±25%.

See Chapter 5 for additional information about operation of GLC systems; Section 504 provides information about quantitation of residues.

See Section 205 for additional information about reference standards.

See Section 104 for additional information about reporting residues and determining compliance with regulations.

See Section 105 for additional information about analytical limits of quantitation.

CONFIRMATION

After residues have been tentatively identified and quantitated by comparison to appropriate reference standards, confirm identity according to principles discussed in Section 103. Use appropriate tables of data (PESTDATA, tables accompanying each method, Index to Methods) to choose most appropriate determinative steps and/or alternative methods for confirmation.

304: METHOD FOR FATTY FOODS

BASIC REFERENCE

Mills, P.A. (1959) *J. Assoc. Off. Agric. Chem.* **42,** 734-740

GENERAL PRINCIPLES

Fat and residues are extracted from fatty foods and dissolved in an organic solvent. Residues are separated from the extracted fat to produce a cleaned up extract solution suitable for determination by gas chromatography.

APPLICABILITY

Consult Guide to PAM I for additional information pertinent to the appropriate application of multiresidue methodology.

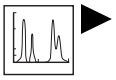
Method is applicable to moderately nonpolar residues in fatty foods. Residue polarity will affect recovery in Cleanups 1 and 2; neither very nonpolar nor very polar residues are recovered completely. See Table 304-a, following the method description, for results of recovery tests.

METHOD MODULES

Choose from these method modules, using Figure 304-a for guidance:

Extraction (E)		Recommended Use	
E1	(p. 304-5)	Extraction of fat with sodium sulfate, petroleum ether	animal tissues, fatty fish
E2	(p. 304-7)	Small scale extraction of fat with sodium sulfate, petroleum ether	animal tissues, fatty fish
E3	(p. 304-9)	Extraction of fat by filtering	butter, oils
E4	(p. 304-11)	Extraction of fat with solvents from denatured product	cheese, milk, egg yolks, dried whole eggs
E5	(p. 304-13)	Extraction of fat with solvents	oilseeds, high fat feeds or feed materials, grains, nuts
Cleanup (C)			
C1	(p. 304-15)	Acetonitrile-petroleum ether partitioning, Florisil column cleanup, three mixed ether eluants	for relatively few samples
C2	(p. 304-17)	Acetonitrile-petroleum ether partitioning, Florisil column cleanup, three methylene chloride eluants	for better cleanup than C1
C3	(p. 304-18)	Acetonitrile-petroleum ether partitioning, Florisil column cleanup, petroleum ether and three mixed ether eluants	to separate PCBs from most pesticides
C4	(p. 304-19)	Acetonitrile-petroleum ether partitioning, Florisil column cleanup, petroleum ether and three methylene chloride eluants	to separate PCBs from most pesticides
C5	(p. 304-21)	Gel permeation chromatography (GPC)	for efficient analysis of many samples (can be automated)

C6	(p. 304-24)	GPC, Florisil column (4 g) cleanup, three methylene chloride eluants	when C5 provides insufficient cleanup
C7	(p. 304-27)	Florisil column (4 g) cleanup, two mixed ether eluants, optional alkaline hydrolysis	to decrease time, solvent use compared to C1
C8	(p. 304-29)	Dispersion on alumina, Florisil column cleanup, three mixed ether eluants	to reduce time compared to C1; screening test only
C9	(p. 304-31)	Dispersion on alumina, Florisil columncleanup, three methylene chloride eluants	to reduce time compared to C3; screening test only

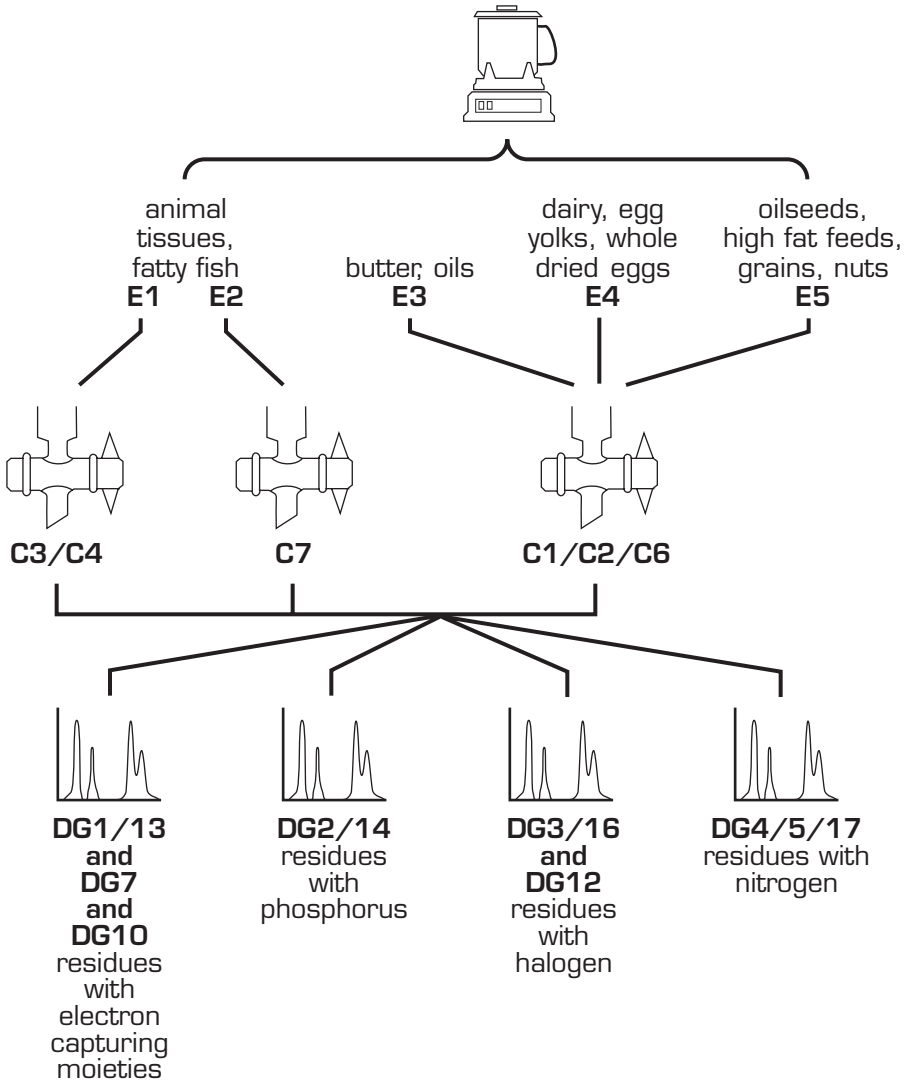


Determinations (D)

(See Section 302 for full details of GLC modules.)

DG 1	(p. 302-33)	GLC, 100% methyl siloxane column, 200°, EC detector	residues with halogen, sulfur, other moieties
DG 2	(p. 302-35)	GLC, 100% methyl siloxane column, 200°, FPD-P	residues with phosphorus
DG 3	(p. 302-37)	GLC, 100% methyl siloxane column, 200°, ELCD-X	residues with halogen
DG 4	(p. 302-39)	GLC, 100% methyl siloxane column, 200°, ELCD-N	residues with nitrogen
DG 5	(p. 302-41)	GLC, 100% methyl siloxane column, 200°, N/P	residues with nitrogen or phosphorus
DG 7	(p. 302-45)	GLC, 100% methyl siloxane column, 130°, EC detector	early eluting residues with halogen, sulfur, other moieties
DG10	(p. 302-51)	GLC, 100% methyl siloxane column, 230°, EC detector	late eluting residues with halogen, sulfur, other moieties
DG12	(p. 302-55)	GLC, 100% methyl siloxane column, 230°, ELCD-X	late eluting residues with halogen
DG13	(p. 302-57)	GLC, 50% phenyl, 50% methyl siloxane column, 200°, EC detector	residues with halogen, sulfur, other moieties
DG14	(p. 302-59)	GLC, 50% phenyl, 50% methyl siloxane column, 200°, FPD-P	residues with phosphorus
DG16	(p. 302-63)	GLC, 50% phenyl, 50% methyl siloxane column, 200°, ELCD-X	residues with halogen
DG17	(p. 302-65)	GLC, 50% phenyl, 50% methyl siloxane column, 200°, N/P detector	residues with nitrogen or phosphorus

Figure 304-a
Recommended Approach: Fatty Foods



VALIDATION

Many combinations of the method modules are possible. The following combinations have undergone interlaboratory validation and are recommended for use:

E1 + C1 + DG1

Validation reports:

Carr, R.L. (1971) *J. Assoc. Off. Anal. Chem.* **54**, 525-527. Collaborative study leading to AOAC official final action status for a-BHC, p,p¢-DDE, p,p¢-TDE, p,p'-DDT, dieldrin, and heptachlor epoxide in fish.

Sawyer, L.D. (1973) *J. Assoc. Off. Anal. Chem.* **56**, 1015-1023. AOAC collaborative study leading to AOAC official final action status for polychlorinated biphenyls in poultry fat and fish.

AOAC official method reference: *Official Methods of Analysis of the AOAC* (1990) 15th ed., 970.52, A, B, E, H, I, J, L, N, O, and R.

E2 + C7 + DG1

Validation report:

Erney, D. R. (1983) *J. Assoc. Off. Anal. Chem.* **66**, 969-973. Collaborative study leading to AOAC official final action status for p,p'-DDE, p,p'-DDT, p,p'-TDE, dieldrin, heptachlor epoxide, and polychlorinated biphenyls in fish.

AOAC official method reference: *Official Methods of Analysis of the AOAC* (1990) 15th ed., 983.21.

E4 + C1 + DG1

Validation reports:

Johnson, L. (1965) *J. Assoc. Off. Agric. Chem.* **48**, 668-675. Collaborative study leading to AOAC official final action status for dieldrin and heptachlor epoxide in dairy products.

Carr, R.L. (1970) *J. Assoc. Off. Anal. Chem.* **53**, 152-154. Collaborative study leading to AOAC official final action status for BHC, p,p'-DDE, p,p'-TDE, o,p'-DDT, p,p'-DDT, lindane, and methoxychlor in dairy products.

Krause, R.T. (1973) *J. Assoc. Off. Anal. Chem.* 5**6**, 721-727. Collaborative study leading to AOAC official final action status for Perthane in dairy products.

Sawyer, L.D. (1978) *J. Assoc. Off. Anal. Chem.* **61**, 282-291. Collaborative study leading to AOAC official final action status for polychlorinated biphenyls in dairy products.

AOAC official method reference: *Official Methods of Analysis of the AOAC* (1990) 15th ed., 970.52, A, B, E, H, I, J, L, N, O, and R.

E3 + C1 + DG1

Validation report:

Wells, C. (1967) *J. Assoc. Off. Anal. Chem.* **50**, 1205-1215. Validation study leading to AOAC official final action status for dieldrin and heptachlor epoxide in vegetable oils.

AOAC official method reference: *Official Methods of Analysis of the AOAC* (1990) 15th ed., 970.52 A, B, E, H, I, J, L, N, O, and R.

E1 + C6 + DG1 and E3 + C6 + DG1

Validation report:

Griffitt, K.R., *et al.* (July 1983) "Miniaturized Florisil Column Cleanup of Chlorinated and Organophosphate Eluates in Total Diet Samples," LIB 2722, FDA, Rockville, MD.

E1 EXTRACTION OF FAT WITH SODIUM SULFATE, PETROLEUM ETHER

Reference

Porter, M.L., *et al.* (1970) *J. Assoc. Off. Anal. Chem.* **53**, 1300-1303

Principles

Fat and residues are removed from fish and animal tissue by dissolving them in petroleum ether. Anhydrous sodium sulfate removes water from the tissue and helps to disintegrate the sample.

Apparatus

blender, high-speed; explosion-proof Waring Blendor, 1 qt jar

Büchner funnel (Büchner), porcelain, 12 cm diameter

chromatographic column, 25 mm id × 50 mm, plain

filter paper, Shark Skin®, to fit Büchner funnel

Kuderna-Danish concentrator (K-D), 500 mL or 1 L, with Snyder column, plain receiving flask

vacuum filtration flask, 500 mL

Reagents

boiling chips, 20-30 mesh carborundum

petroleum ether, distilled from all-glass apparatus

sodium sulfate, anhydrous, granular, reagent grade; see Section 204 for handling directions

Directions

- (Sample size may be adjusted to provide weight of fat appropriate to cleanup step.) Weigh 25-50 g thoroughly ground and mixed fish or animal tissue into blender jar. Add 100 g sodium sulfate.
- Alternately blend and mix with spatula until sample and sodium sulfate are well mixed. Scrape down sides of blender jar and break up caked material with spatula.
- Add 150 mL petroleum ether and blend at high speed 2 min.
- Decant petroleum ether supernate through Büchner fitted with two Shark Skin® papers and filter with suction into vacuum filtration flask.
- Scrape down sides of blender jar and break up caked material with spatula.
- Re-extract residue in blender jar with two 100 mL portions petroleum ether, blending 2 min each time. (After 1 min blending, stop blender, scrape material from sides of blender jar and break up caked material with spatula; continue blending 1 min.) Scrape down sides of blender jar and break up caked material between extractions. Decant petroleum ether supernates through Büchner and combine with first extract.
- After last blending, transfer residue from blender jar to Büchner, rinsing blender jar and material in Büchner with three 25-50 mL portions petroleum ether. Immediately after last rinse, press residue in Büchner with bottom of clean beaker to force out remaining petroleum ether.

- Pour combined extracts and rinses through 25 mm × 50 mm column of sodium sulfate and collect eluate in K-D with plain tube. Wash flask and then column with small portions petroleum ether.
- Add boiling chip to K-D and evaporate most of petroleum ether from combined extracts and rinses.
- Clean up extracted fat using one of the cleanup steps.

E2 SMALL SCALE EXTRACTION OF FAT WITH SODIUM SULFATE, PETROLEUM ETHER

Reference

Erney, D.R. (1974) *J. Assoc. Off. Anal. Chem.* **57**, 576-579

Principles

Fat and residues are removed from fish and animal tissue by dissolving them in petroleum ether. Anhydrous sodium sulfate removes water from the tissue and helps to disintegrate the sample. Sample size and amounts of reagents are somewhat reduced from amounts used in E1, for subsequent small scale cleanup of C7.

Apparatus

funnel, glass

homogenizer, Sorvall/Omni type, with 400 mL cup

volumetric flask (volumetric), 250 mL

Reagents

glass wool, Pyrex, see Section 204 for handling directions

petroleum ether, distilled from all-glass apparatus

sodium sulfate, anhydrous, granular, reagent grade; see Section 204 for handling directions

Directions

- Weigh 20 g thoroughly ground and mixed fish or animal tissue into homogenizer cup. Moisten 40 g sodium sulfate with petroleum ether and add to sample.
- Mix sample, using stirring rod, let stand 20 min, and mix again.
- Add 100 mL petroleum ether to sample and blend 1-2 min.
- Centrifuge balanced homogenizer cup at 2000 rpm 1-2 min to obtain clear petroleum ether extract.
- Place glass wool plug in funnel, overlay with 20 g sodium sulfate, and place funnel in 250 mL volumetric. Decant petroleum ether extract through layer of sodium sulfate into volumetric.
- Mix sample again with stirring rod, add 100 mL petroleum ether, and extract as before.
- Repeat extraction with 70 mL petroleum ether, combining all three extractions in same 250 mL volumetric.
- Dilute to volume with petroleum ether.
- Clean up with C7, Florisil chromatography on 4 g column. No intermediate separation step is required if ≤0.2 g fat is used, as specified in C7.
- Note that this extraction yields a dilute solution of fat, rather than fat with most solvent removed as in other extraction modules. To clean up this extract with a module other than C7, solvent must first be removed.

E3 EXTRACTION OF FAT BY FILTERING

Reference

Mills, P.A. (1959) *J. Assoc. Off. Agric. Chem.* **42**, 734-740

Principle

Fat and residues are removed from butter by melting and filtering to remove solids. No extraction is needed for edible oil.

Apparatus

filter paper

funnel, glass

Directions

- Warm butter at about 50° C until fat separates; decant through dry filter paper placed in glass funnel. Collect oil.
- Clean up extracted fat using one of the cleanup steps.

E4 EXTRACTION OF FAT WITH SOLVENTS FROM DENATURED PRODUCT

Reference

Mills, P.A. (1959) *J. Assoc. Off. Agric. Chem.* **42**, 734-740

Principles

Fat and residues from cheese, milk, egg yolks, or dried whole eggs are dissolved in ethyl ether and petroleum ether after the product has been denatured with oxalate and alcohol. The ether extract is washed with large quantities of water to remove co-extractives.

(Methodology for high fat egg products has not been studied extensively; this method is recommended as the most applicable.)

Apparatus

blender, high-speed; explosion-proof Waring Blendor, 1 qt jar

centrifuge, explosion-proof, to hold 500 mL bottles

centrifuge bottle, glass, 500 mL. Use glass stopper or cover rubber stopper with aluminum foil to avoid contamination.

chromatographic column, 25 mm id × 50 mm, plain

delivery tube apparatus (Figure 304-b), fabricated in laboratory

Kuderna-Danish concentrator (K-D), 500 mL, with Snyder column, plain receiving flask

separatory funnel (separator), 1 L

Reagents

alcohol, ethyl or methyl

ethyl ether, distilled from all-glass apparatus, with 2% ethanol as preservative; see Section 204 for peroxide test

1+1 (v/v) ethyl ether/petroleum ether

petroleum ether, distilled from all-glass apparatus

sodium chloride, reagent grade

sodium chloride aqueous solution, saturated

sodium (or potassium) oxalate, reagent grade

sodium sulfate, anhydrous, granular, reagent grade; see Section 204 for handling directions

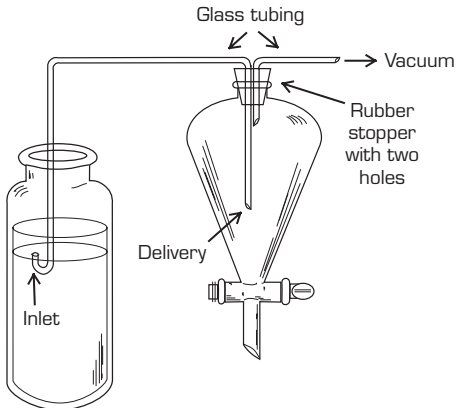
Directions

- *Cheese and Eggs*—If experience with product indicates emulsions will not be broken by centrifuging, add 1 mL water/2 g sample before blending. Place 25-100 g diced cheese or 25-50 g eggs (to provide about 3 g fat), about 2 g sodium or potassium oxalate, and 100 mL ethyl or methyl alcohol in blender jar and blend 2-3 min. Pour blender contents into centrifuge bottle.

  Milk — Dilute evaporated milk with equal amount of water. Place 100 g fluid milk or diluted evaporated milk into centrifuge bottle, add 100 mL ethyl or methyl alcohol and about 1 g sodium or potassium oxalate, and mix.

- To mixture in centrifuge bottle, add 50 mL ethyl ether and shake vigorously 1 min; then add 50 mL petroleum ether and shake vigorously 1 min.
- Centrifuge at about 1500 rpm about 5 min. Never leave stoppers in bottles during centrifuging.
- Transfer solvent layer, with delivery tube apparatus, into separator containing 500-600 mL water and 30 mL saturated sodium chloride solution. Re-extract aqueous residue twice, shaking vigorously with 50 mL portions (1+1) ethyl ether/petroleum ether; centrifuge and transfer solvent layer into separator after each extraction.
- Cautiously mix combined extracts and water. Drain and discard water.
- Gently rewash solvent layer with two 100 mL portions water, discarding water each time. If emulsions form, add about 5 mL saturated sodium chloride solution to solvent layer or include sodium chloride with water wash.
- Pour ether solution through 25 mm × 50 mm column of sodium sulfate and collect eluate in K-D with plain tube. Wash separator and then column with small portions petroleum ether.
- Add boiling chip to K-D and evaporate most of petroleum ether from combined extracts and rinses.
- Clean up extracted fat using one of the cleanup steps.

Figure 304-b
Delivery Tube Apparatus



Glass tube, inserted in one hole of two-hole rubber stopper, is used to draw upper solvent layer from centrifuge bottle into separatory funnel. Siphon tube is straight or bent in U-shape and inlet end placed at interface of two phases in centrifuge bottle. Second hole in stopper is fitted with another glass tube. Vacuum drawn through second tube causes upper phase from centrifuge bottle to transfer into separator.

(Corrigan, E. (Nov. 1963) (FDA) *Bureau By-Lines* **5**, 20; Sawyer, L.D., and Baca, J.R. (May 1978) LIB 2188, FDA, Rockville, MD.)

E5 EXTRACTION OF FAT WITH SOLVENTS

Reference

Sawyer, L.D. (1982) *J. Assoc. Off. Anal. Chem.* **65**, 1122-1128

Principles

Fat and residues are removed from ground oilseeds, high fat feeds or feed materials, grains, or nuts by dissolving in petroleum ether and ethyl ether, followed by ethyl alcohol. The organic extract is washed with large quantities of water to remove co-extractives.

Apparatus

centrifuge, explosion-proof, to hold 500 mL bottles

chromatographic column, 25 mm id, plain

homogenizer, Polytron Model PT 10-35, with PT 35K generator containing knives, head equipped with metal (not Teflon) bushing or Sorvall/Omni type

homogenizer jar, Sorvall/Omni stainless steel cup, 400 mL, or centrifuge bottle, glass, 500 mL

Kuderna-Danish concentrator (K-D), 500 mL, with Snyder column, plain receiving flask

separatory funnels (separators), 1 L

Reagents

alcohol, ethyl

ethyl ether, distilled from all-glass apparatus, with 2% ethanol as preservative; see Section 204 for peroxide test

1+1 (v/v) ethyl ether/petroleum ether

petroleum ether, distilled from all-glass apparatus

sodium chloride, reagent grade

sodium chloride aqueous solution, saturated

sodium sulfate, anhydrous, granular, reagent grade; see Section 204 for handling directions

Directions

- Weigh 50 g ground sample of oilseeds, high fat feeds or feed materials, grains, or nuts in Sorvall/Omni cup or in centrifuge bottle. Add 200 mL petroleum ether and blend 1.5 min at high speed with Polytron PT 10-35 homogenizer or Omni-Mixer.
- Centrifuge extraction vessel and decant solvent into 500 mL beaker. Never leave stoppers in bottles during centrifuging.
- Add 150 mL (1+1) ethyl ether/petroleum ether to vessel and blend; centrifuge and decant into same beaker as before.
- Set beaker containing combined ethers under gentle air stream to concentrate to about 100 mL.
- Re-extract residue in extraction vessel with 150 mL ethyl alcohol for 1.5 min. Centrifuge and decant solvent into 1 L separator.

- Add 50 mL ethyl alcohol to extraction vessel, wash residue in vessel by gently blending, then centrifuge and decant into same separator as before.
- Add concentrated ethers from first two extractions to separator. Rinse beaker using small (about 5 mL) petroleum ether washes and add washes to separator.
- Mix separator contents well and add 600 mL water and about 40 mL saturated sodium chloride solution.
- Hold separator in horizontal position and mix thoroughly 45 sec. Let layers separate and drain aqueous portion into second 1 L separator containing 100 mL petroleum ether.
- Mix contents of second separator thoroughly about 15 sec and let layers separate. Drain and discard aqueous portion and drain petroleum ether into original separator. Wash combined ethers with two 100 mL portions water.
- Pour ether solution through 25 mm × 50 mm column of sodium sulfate and collect eluate in K-D with plain tube. Wash separator and then column with small portions petroleum ether.
- Add boiling chip to K-D and evaporate most of petroleum ether from combined extracts and rinses in K-D.
- Clean up extracted fat using one of the cleanup steps.

C1 ACETONITRILE-PETROLEUM ETHER PARTITIONING, FLORISIL COLUMN CLEANUP, THREE MIXED ETHER ELUANTS

Reference

Mills, P.A. (1959) *J. Assoc. Off. Agric. Chem.* **42**, 734-740

Principles

Extracted fat is carefully weighed to avoid overloading the capacity of the cleanup step. Pesticide residues are isolated from fat by partition between petroleum ether and acetonitrile. Most of the fat is retained in petroleum ether while residues partition into acetonitrile in proportion to their partitioning coefficient in that system. In the subsequent step, residues in acetonitrile are partitioned back into petroleum ether when added water reduces their solubility in acetonitrile.

Residues in solution are separated from sample co-extractives on a column of Florisil adsorbent; eluants of increasing polarity sequentially remove residues from the column.

Cleanup steps C2, C3, and C4 offer alternative Florisil elution systems. The eluants used in C2 produce different elution patterns than C1, which can sometimes be valuable for confirmation. C2 can also be used as an additional cleanup step.

C3 or C4 are used for analyses directed at the determination of polychlorinated biphenyls. The Florisil column is eluted with petroleum ether prior to elution with the other mixtures, in order to separate the polychlorinated biphenyls from most pesticide residues and to provide a cleaner extract for their determination.

Apparatus

chromatographic column, 25 mm id × 50 mm, plain

chromatographic column, 22 mm id × 300 mm, Teflon stopcock, coarse porosity fritted disc

Kuderna-Danish concentrator (K-D), 500 mL, with Snyder column, two-ball micro-Snyder column, volumetric or graduated receiving flask

separatory funnels (separators), 125 mL and 1 L

Reagents

acetonitrile, distilled from all-glass apparatus; see Section 204 for distillation directions

acetonitrile saturated with petroleum ether

boiling chips, 20-30 mesh carborundum

ethyl ether, distilled from all-glass apparatus, with 2% ethanol as preservative; see Section 204 for peroxide test

Florisil, PR grade; see Section 204 for handling and testing directions and calculation of lauric acid value

hexane, distilled from all-glass apparatus

methylene chloride, distilled from all-glass apparatus

petroleum ether, distilled from all-glass apparatus

sodium chloride, reagent grade

sodium chloride aqueous solution, saturated

sodium sulfate, anhydrous, granular, reagent grade; see Section 204 for handling directions

eluants: 6% (v/v) ethyl ether/petroleum ether
15% (v/v) ethyl ether/petroleum ether
50% (v/v) ethyl ether/petroleum ether

Directions

- Cleanup is applicable to ≤3 g fat. Evaporate solvent from solution of extracted fat and determine amount of fat extracted by one of the following operations:
  - When total amount of fat in sample is expected to be >3 g and analysis is not for volatile residues, transfer concentrated solution to tared beaker, using small amounts of petroleum ether, and evaporate to dryness at steam bath temperature under current of dry air. Weigh and record weight of fat extracted. Take ≤3 g fat for cleanup. Calculate weight of sample analyzed as:

$$\frac{\text{weight fat taken for cleanup}}{\text{weight fat extracted}} \times \text{weight original sample}$$

  - When it is known that amount of fat in sample is <3 g, do not evaporate solvent further but clean up total amount of fat solution. Calculate weight of sample analyzed as: weight original sample.
  - When it is known that fat content is >3 g, or that residue level is high, do not evaporate solvent further. Adjust to known, appropriate volume, transfer aliquot to tared beaker, evaporate solvent from aliquot, and weigh to determine fat content. Clean up aliquot of volume containing ≤3 g fat. Calculate weight of sample analyzed as:

$$\frac{\text{volume fat solution taken for cleanup}}{\text{total volume of solution}} \times \text{weight original sample}$$

  - When analysis for volatile chemicals is desirable, do not evaporate petroleum ether at steam bath temperature. Adjust to known, appropriate volume, transfer aliquot to tared beaker, evaporate solvent from aliquot, and weigh to determine fat content. Clean up remaining solution or appropriate aliquot. Calculate weight of sample analyzed as:

$$\frac{\text{weight fat solution taken for cleanup}}{\text{total volume of solution}} \times \text{weight original sample}$$

Acetonitrile/Petroleum Ether Partitioning

- Weigh ≤3 g fat into 125 mL separator, and add petroleum ether so that total volume of fat and petroleum ether in separator is 15 mL. Take smaller weight of fish oil if experience indicates tendency to emulsion formation during partitioning.
- Add 30 mL acetonitrile saturated with petroleum ether, shake vigorously 1 min, let layers separate, and drain acetonitrile into 1 L separator containing 650 mL water, 40 mL saturated sodium chloride solution, and 100 mL petroleum ether.

- Extract petroleum ether solution in 125 mL separator with three additional 30 mL portions acetonitrile saturated with petroleum ether, shaking vigorously 1 min each time, and combine all extracts in the 1 L separator.
- Hold 1 L separator in horizontal position and mix thoroughly 30-45 sec. Let layers separate and drain aqueous layer into second 1 L separator.
- Add 100 mL petroleum ether to second 1 L separator, shake vigorously 15 sec, and let layers separate. Discard aqueous layer, combine petroleum ether with petroleum ether in original 1 L separator, and wash with two 100 mL portions water.
- Discard washings and drain petroleum ether layer through 25 mm × 50 mm column of sodium sulfate into K-D. Rinse separator and then column with three 10 mL portions petroleum ether.
- Add boiling chip to K-D and evaporate combined extract and rinses to 5-10 mL for transfer to Florisil column.

Florisil Column Cleanup

- Place activated Florisil (4" or weight determined by lauric acid value) in 22 mm id column; add about 0.5" sodium sulfate. Prewet column with 40-50 mL petroleum ether. Place K-D with volumetric or graduated receiving flask under column to receive eluate.
- Transfer sample extract solution to column, letting it pass through at about 5 mL/min. Rinse container (and sodium sulfate if present) with two 5 mL portions petroleum ether, transfer rinsings to column, and rinse walls of chromatographic tube with additional small portions petroleum ether.
- Elute column at about 5 mL/min with 200 mL 6% ethyl ether/petroleum ether eluant.
- Change K-Ds and elute at about 5 mL/min with 200 mL 15% ethyl ether/petroleum ether eluant.
- Change K-Ds and elute at about 5 mL/min with 200 mL 50% ethyl ether/petroleum ether eluant.
- Add boiling chips to K-Ds and concentrate each eluate to suitable definite volume. When volume <5 mL is needed, use two-ball micro-Snyder or micro-Vigreaux column during final evaporation in receiving flask.
- Use appropriate determinative steps, such as DG1 or DG13, DG7, and DG10, to identify and measure residues. First eluate (6%) is usually suitable for GLC determination without further cleanup.

ALTERNATIVES:

C2 ACETONITRILE-PETROLEUM ETHER PARTITIONING, FLORISIL COLUMN CLEANUP, THREE METHYLENE CHLORIDE ELUANTS

Reference

Mills, P.A., *et al.* (1972) *J. Assoc. Off. Anal. Chem.* **55**, 39-43

Principles

Florisil is eluted with mixtures of methylene chloride, hexane, and acetonitrile. The resulting second eluate is cleaner than the second eluate of C1, although 90% of the fat placed on the column is eluted by the third eluant. The eluants can elute pesticide chemicals of a greater polarity than can be eluted by C1. C2 is preferred for analysis of fats and oils, for residues of endosulfan, and for separation of heptachlor epoxide and octachlor epoxide.

Additional Reagents

eluants: 1—20% methylene chloride/hexane (v/v). Dilute 200 mL methylene chloride with hexane. Allow mixture to reach room temperature, and adjust volume to 1 L with hexane.

2—50% methylene chloride/0.35% acetonitrile/49.65% hexane (v/v/v). Pipet 3.5 mL acetonitrile into 500 mL methylene chloride and dilute with hexane. Allow mixture to reach room temperature and adjust to 1 L with hexane.

3—50% methylene chloride/1.5% acetonitrile/48.5% hexane (v/v/v). Pipet 15 mL acetonitrile into 500 mL methylene chloride and dilute with hexane. Allow mixture to reach room temperature and adjust to 1 L with hexane.

Directions

- Follow C1 above, except replace directions for Florisil cleanup with the following:
- Place activated Florisil (4" or weight determined by lauric acid value) in 22 mm id column; add about 0.5" sodium sulfate. Prewet column with 40-50 mL hexane. Place K-D with volumetric or graduated receiving flask under column to receive eluate.
- Transfer sample extract solution to column, letting it pass through at about 5 mL/min. Rinse container (and sodium sulfate if present) with two 5 mL portions hexane, transfer rinsings to column, and rinse walls of chromatographic tube with additional small portions hexane.
- Elute column at about 5 mL/min with 200 mL eluant 1.
- Change K-Ds and elute with 200 mL eluant 2.
- Change K-Ds and elute with 200 mL eluant 3.
- Add boiling chips to K-Ds and concentrate each eluate to suitable definite volume. When volume <5 mL is needed, use two-ball micro-Snyder or micro-Vigreaux column during evaporation.

C3 ACETONITRILE-PETROLEUM ETHER PARTITIONING, FLORISIL COLUMN CLEANUP, PETROLEUM ETHER AND THREE MIXED ETHER ELUANTS

Reference

Reynolds, L.M. (1969) *Bull. Environ. Contam. Toxicol.* **4**, 128-143

Principles

Polychlorinated biphenyls are separated from most pesticide residues by elution of Florisil column with petroleum ether prior to elution with ethyl ether/petroleum ether mixtures.

Directions

- Follow C1 above, except insert the following before elution of Florisil column with 6% ethyl ether/petroleum ether:
- Elute column at about 5 mL/min with 250 mL petroleum ether. Change K-Ds.

C4 ACETONITRILE-PETROLEUM ETHER PARTITIONING, FLORISIL COLUMN CLEANUP, PETROLEUM ETHER AND THREE METHYLENE CHLORIDE ELUANTS

Reference

Reynolds, L.M. (1969) *Bull. Environ. Contam. Toxicol.* **4**, 128-143

Principles

Polychlorinated biphenyls are separated from most pesticide residues by elution of Florisil column with petroleum ether prior to elution with methylene chloride mixtures.

Directions

- Follow C3 above, except insert the following before elution of Florisil column with eluant 1:
- Elute column at about 5 mL/min with 250 mL petroleum ether. Change K-Ds.

C5 GEL PERMEATION CHROMATOGRAPHY (GPC)

References

Griffitt, K.R., and Craun, J.C. (1974) *J. Assoc. Off. Anal. Chem.* **57**, 168-172

Hopper, M.L (1982) *J. Agric. Food Chem.* **30**, 1038-1041

Principles

Fat is separated from residues by gel permeation (size exclusion) chromatography. The solution of fat extracted from fatty food is placed on a column and eluted with solvents. The fat is eluted first and discarded, leaving residues in the next portion of eluant.

Cleanup C6 offers optional Florisil column cleanup subsequent to GPC.

Apparatus

filtration device for solutions, 10 mL syringe with Luer-Lok tip, fitted with either (a) 13 mm diameter Swinny stainless steel filter holder and 13 mm diameter filters, 5.0 µm LS-type, or (b) disposable membrane filters, 25 mm diameter, 5 µm Teflon membrane, encased in polypropylene. (Pre-assembled devices that do not require a syringe are also available.)

GPC apparatus; automated equipment optional but recommended. GPC apparatus must include:

1) sample introduction valve
2) pump, low pressure, suitable for use with organic solvents, capable of 5 mL/min flow
3) sample loading loop, 1/16" Teflon tubing coiled in cylindrical form, about 13 mL capacity
4) pulse dampener, about 6' of 1/8" copper tubing coiled and closed at one end, installed between pump and sample introduction valve with a connecting tee. Pulse dampener is needed only when pump is not pulseless.

GPC column, glass, 25 mm id × 300 or 500 mm with organic solvent plunger kit

GPC syringe, 10 mL syringe with Luer-Lok tip, with Millipore Swinny stainless steel adapter, Millipore 5.0 µm LS-type filter

graduated cylinder (graduate), glass-stoppered (g-s), calibrated

Kuderna-Danish concentrator (K-D), 500 mL, with Snyder column, two-ball micro-Snyder column, graduated receiving flask

Reagents

acetone, distilled from all-glass apparatus

Bio-Beads SX-3 resin, 200-400 mesh (Bio-Rad Laboratories, Richmond, CA; pretested resin is available from ABC Laboratories)

hexane, distilled from all-glass apparatus

methylene chloride, distilled from all-glass apparatus

eluant: 50% (v/v) methylene chloride/hexane

Preparation of GPC Column

- Weigh 35 g Bio-Beads SX-3 into 400 mL beaker.
- Add 150 mL 50% methylene chloride/hexane.
- Stir beads with glass or steel rod until all beads have swelled and no clumps are present.
- Pour slurry into GPC column with aid of stirring rod.
- Hold column in upright position with plunger tightened about 25 mm from bottom of usable length of column, ignoring threaded ends.
- Continuously add slurry to column so beads never become completely settled until all beads have been added.
- Place other plunger in column after beads have settled and liquid has drained off.
- Compress each plunger equal distance from its respective end until bed length is about 200 mm.
- Connect column to GPC solvent delivery system, and pump solvent from bottom to top of column until all air is expelled.
- Adjust flow rate of system to 5 mL/min and check column pressure. Adjust operating pressure for column to 8-11 psig by moving plunger(s).
- Allow GPC system to equilibrate by pumping solvent through it.
- Re-adjust flow rate to 5 mL/min if it has changed.

Calibration of GPC Column

Elution of Fat

- Melt and filter butter through fluted filter paper into suitable container.
- Weigh 5 g warm filtered butter (do not include water layer) into 25 mL g-s graduate; dilute to 25 mL with 50% methylene chloride/hexane; mix until fat is dissolved (0.2 g fat/mL).
- Filter fat solution through filtration device and load 5 mL fat solution onto GPC column.
- Elute with 50% methylene chloride/hexane.
- Collect column effluent in tared beakers in 10 mL increments from 0 to 100 mL.
- Evaporate solvent, cool and weigh beakers to calculate amount fat eluted in each 10 mL increment. (For manual GPC, collect 10 mL fractions in separate graduates and transfer to tared beakers for evaporation and calculation of fat.)
- Most (98%) fat should elute in first 60 mL. If >5% of fat appears in 60-70 mL fraction or later, reject column and prepare new one by repacking with original batch of beads. Visual evaluation of yellow fat band as it passes through column usually shows tailing or streaking when column is inadequate. Use new batch of beads if second column is still inadequate.

Elution of Pesticides

- Prepare mixed standard solution containing 0.2 µg diazinon/mL, 0.6 µg ethion/mL, 0.1 µg lindane/mL, 0.4 µg parathion/mL, and 0.05 µg pentachloroaniline/mL in 50% methylene chloride/hexane.
- Filter mixed standard solution through filtration device and load 5 mL onto GPC column
- Elute with 50% methylene chloride/hexane.
- Collect 10 mL fractions from 0 through 160 mL.
- Transfer each fraction to K-D fitted with graduated receiving flask and add 50 mL hexane and 2-3 boiling chips; concentrate each to 10 mL.
- Use determinative steps DG2 and DG3 (Section 302) to calculate recoveries; column is normal if diazinon and ethion start to elute in either 50-60 mL or 60-70 mL fraction, and lindane starts to elute in 90-100 mL fraction.
- Determine what volume should be discarded (usually first 60 mL) and what should be collected (usually 60-160 mL fraction) by examining fat and mixed standard elution profiles developed above. Use these calibrated fraction volumes in subsequent calibration steps and in sample cleanup.

Directions

- Use GPC column prepared and calibrated as described above. Column can be used repeatedly.
- Method is applicable to ≤1 g fat sample in 5 mL; better cleanup is provided if weight of fat in 5 mL is restricted to 0.75 g.
- Concentrate solution of extracted fat to small volume. Add 100 mL methylene chloride and reconcentrate.
- Based on estimate of weight of fat in extract, use calibrated graduate large enough to create final solution containing ≤0.2 g/mL. Transfer concentrated solution to graduate. Add enough methylene chloride to provide half the final volume, then fill to final volume with hexane.
- Pipet 1 mL into tared beaker; evaporate solvent and weigh. If weight is >0.2 g, adjust concentration of remaining solution to ≤0.2 g/mL with 50% methylene chloride/hexane.
- Centrifuge cloudy solutions before loading them onto GPC. Filter all solutions through filtration device before GPC.
- Fill GPC sample loading loops with extract using GPC syringe. Load 5 mL fat solution (equivalent to ≤1 g fat) onto GPC column. Load more than one loop with same solution if needed to increase final total sample equivalent.
- Elute column with 160 mL 50% methylene chloride/hexane. Collect and discard volume previously calibrated to contain fat. Separately collect final portion, previously calibrated to contain residues.
- Transfer GPC eluate to K-D with 5 mL graduated receiving flask and concentrate to <3 mL. Add hexane and reconcentrate to <1 mL.

- Use appropriate determinative steps to identify and measure residues. Dilute final concentrated eluate with acetone for determination of organophosphorus residues by DG2 or DG14 (Section 302). Dilute with hexane for determination of organohalogen residues by DG3 or DG16. Clean up on Florisil (C6) before determination of residues with electron capture detector (DG1, 7, 10, and 13).

ALTERNATIVE:

C6 GPC, FLORISIL COLUMN (4 G) CLEANUP, THREE METHYLENE CHLORIDE ELUANTS

Reference

Griffitt, K.R., *et al.* (July 1983) "Miniaturized Florisil Column Cleanup of Chlorinated and Organophosphate Eluates in Total Diet Samples," LIB 2722, FDA, Rockville, MD

Principles

For additional cleanup of samples, residues are further separated from sample coextractives on a small column of Florisil adsorbent; three eluants of increasing polarity sequentially remove residues from the column.

Additional Apparatus

chromatographic column, 10 mm id × 300 mm, Teflon stopcock, coarse porosity fritted disc

Additional Reagents

acetonitrile, distilled from all-glass apparatus; see Section 204 for distillation directions

Florisil, PR grade; see Section 204 for handling and testing directions and calculation of lauric acid value

sodium sulfate, anhydrous, granular, reagent grade; see Section 204 for handling directions

eluants: 1—20% methylene chloride/hexane (v/v). Dilute 200 mL methylene chloride with hexane. Allow mixture to reach room temperature, and adjust volume to 1 L with hexane.

2—50% methylene chloride/0.35% acetonitrile/49.65% hexane (v/v/v). Pipet 3.5 mL acetonitrile into 500 mL methylene chloride and dilute with hexane. Allow mixture to reach room temperature and adjust to 1 L with hexane.

3—50% methylene chloride/1.5% acetonitrile/48.5% hexane (v/v/v). Pipet 15 mL acetonitrile into 500 mL methylene chloride and dilute with hexane. Allow mixture to reach room temperature and adjust to 1 L with hexane.

Directions

- Follow directions of C5 up through elution of GPC column. Concentrate GPC eluate to <5 mL.
- Place activated Florisil (weight = 110/lauric acid value × 4 g) in 10 mm chromatographic column; add about 2 cm sodium sulfate. Completely open stopcock and tap column to settle adsorbent. Prewet column with 5 mL hexane. Do not allow column to go dry. Place K-D with volumetric or graduated receiving flask under column to receive eluate.

- Transfer concentrated GPC eluate to column, letting it pass through at about 5 mL/min. Rinse container with two 3 mL portions hexane, transfer rinsings to column, and rinse walls of chromatographic tube with additional small portions hexane.
- Elute column at about 5 mL/min with 35 mL eluant 1.
- Change K-Ds and elute with 35 mL eluant 2.
- Change K-Ds and elute with 40 mL eluant 3.
- Concentrate each eluate to suitable definite volume in K-D. When volume <5 mL is needed, use two-ball micro-Snyder or micro-Vigreaux column during evaporation.

C7 FLORISIL COLUMN (4 G) CLEANUP, TWO MIXED ETHER ELUANTS, OPTIONAL ALKALINE HYDROLYSIS

References

Erney, D.R. (1974) *J. Assoc. Off. Anal. Chem.* **57**, 576-579

Erney, D.R. (1983) *J. Assoc. Off. Anal. Chem.* **66**, 969-973

Principles

Residues are separated from fat on a small column of Florisil adsorbent. Because no prior isolation step is included to minimize co-extractives, only 0.2 g extracted fat is placed on the Florisil column. Two eluants of increasing polarity sequentially remove residues from the column. The second eluate is cleaned up further with alkaline hydrolysis if needed, a step applicable only to chemicals stable to hot alkali.

Apparatus

chromatographic column, 10 mm id × 300 mm, Teflon stopcock, coarse porosity fritted disc

Kuderna-Danish concentrator (K-D), 125 mL, with Snyder column, two-ball micro-Snyder column, graduated and 10 mL volumetric receiving flask

Reagents

boiling chips, 20-30 mesh carborundum

ethyl ether, distilled from all-glass apparatus, with 2% ethanol as preservative; see Section 204 for peroxide test

Florisil, PR grade; see Section 204 for handling and testing directions and calculation of lauric acid value

hexane, distilled from all-glass apparatus

petroleum ether, distilled from all-glass apparatus

sodium sulfate, anhydrous, granular, reagent grade; see Section 204 for handling directions

eluants: 6% (v/v) ethyl ether/petroleum ether

15% (v/v) ethyl ether/petroleum ether

2% (w/v) potassium hydroxide in methanol or ethanol

1+1 (v/v) water/alcohol

Directions

Florisil Column Cleanup

- Cleanup is applicable to ≤0.2 g fat.
- Concentration of fat in extract prepared by E2 is about ≤0.2 g in 25 mL. To use this cleanup for extracts prepared by E1, E3, E4, or E5, first dilute all or part of extracted fat to ≤0.2 g in 25 mL.
- Transfer 25 mL aliquot to tared beaker and place on steam bath until solvent is evaporated. Leave on steam bath additional 30 min, remove, and cool. Weigh fat.
- If extract contains ≤0.2 g/25 mL, transfer 25 mL aliquot to 125 mL K-D with graduated receiving flask. If extract contains >0.2 g/25 mL, transfer volume containing ≤0.2 g fat to K-D.

- Add several boiling chips and concentrate solution to about 3 mL on steam bath. Let cool, remove Snyder column, rinse K-D with two 1 mL portions petroleum ether. Use current of air to concentrate sample to 3 mL.
- Place activated Florisil (weight = 110/lauric acid value× 4 g) in 10 mm id glass column; add about 2 cm sodium sulfate. Completely open stopcock, tap tube to settle adsorbent, and mark tube 1 cm above sodium sulfate layer.
- Add about 25 mL petroleum ether to Florisil column. As solvent level reaches mark, place 125 mL K-D with 10 mL volumetric receiving flask under column.
- Using disposable pipet, transfer 3 mL concentrated sample extract to column; wash flask with 1 mL petroleum ether and add wash to column. Solvent level must not go below mark; temporarily close stopcock if necessary.
- Elute column with 35 mL 6% ethyl ether/petroleum ether.
- When solvent level reaches mark, change K-Ds. Elute column with 35 mL 15% ethyl ether/petroleum ether.
- Concentrate each eluate to suitable definite volume in K-D. When volume <5 mL is needed, use two-ball micro-Snyder or micro-Vigreaux column during evaporation.
- Use appropriate determinative steps, such as DG1 or DG13, DG7, and DG10, to identify and measure residues. Second eluate may need further cleanup prior to GLC; use optional alkaline hydrolysis if residues are stable to hot alkali.

Optional Alkaline Hydrolysis

- Concentrate 15% ethyl ether/petroleum ether eluate to 2 mL with current of air.
- Add 1 mL 2% potassium hydroxide/alcohol, attach micro-Snyder column to flask, and carefully reduce volume to ≤1 mL on steam bath. Reflux 15 min, then let cool.
- Add 2 mL (1+1) water/alcohol and 5 mL hexane to flask, and shake 1 min. Centrifuge to separate layers.
- Transfer as much hexane layer as possible to second flask, using disposable pipet.
- Add another 5 mL hexane to flask, and repeat extraction.
- Concentrate combined hexane extracts to appropriate volume for determination.

C8 DISPERSION ON ALUMINA, FLORISIL COLUMN CLEANUP, THREE MIXED ETHER ELUANTS

References

Luke, M.A., and Doose, G.M. (Jan. 1978) "A Rapid Analysis for Pesticide Residues in Milk and Other Fatty Foods," LIB 2120A, FDA, Rockville, MD

Gillespie, A.M., and Walters, S.M. (May 1983) "An Alumina Blending Technique for the Separation of Pesticides from Lipids (Based on LIB 2120A)," LIB 2716, FDA, Rockville, MD

Principles

Extracted fat is dispersed on deactivated alumina and pesticide residues are removed with a mixture of water and acetonitrile; most of the fat is retained by the alumina. In the subsequent step, residues in acetonitrile are partitioned back into petroleum ether when added water reduces their solubility in acetonitrile. Residues in solution are separated from remaining sample co-extractives on a column of Florisil adsorbent; three eluants of increasing polarity sequentially remove residues from the column.

Cleanup step C9 offers an alternative Florisil elution system, which produces different elution patterns than C8.

Apparatus

Büchner funnel (Büchner), porcelain, 12 cm diameter

chromatographic column, 25 mm id × 50 mm, plain

chromatographic column, 22 mm id × 300 mm, Teflon stopcock, coarse porosity fritted disc

filter paper, Whatman No. 40, 15 cm diameter. Wash paper by soaking in 20% water/acetonitrile to remove substances that interfere in GLC determination. Dry and store in closed container.

homogenizer, Sorvall/Omni type, with 400 mL cup

Kuderna-Danish concentrator (K-D), 500 mL, with Snyder column, two-ball micro-Snyder column, graduated and volumetric receiving flasks

separatory funnel (separator), 1 L

shaker, mechanical, Burrell wrist action

Reagents

acetonitrile, distilled from all-glass apparatus; see Section 204 for distillation directions

alumina, Fisher Adsorption Alumina, No. A-540, 80-200 mesh

boiling chips, 20-30 mesh carborundum

ethyl ether, distilled from all-glass apparatus, with 2% ethanol as preservative; see Section 204 for peroxide test

Florisil, PR grade; see Section 204 for handling and testing directions and calculation of lauric acid value

glass wool, Pyrex; see Section 204 for handling directions

hexane, distilled from all-glass apparatus

methylene chloride, distilled from all-glass apparatus

petroleum ether, distilled from all-glass apparatus

sodium chloride, reagent grade

sodium chloride aqueous solution, saturated

sodium sulfate, anhydrous, granular, reagent grade; see Section 204 for handling directions

20% (v/v) water/acetonitrile

eluants: 6% (v/v) ethyl ether/petroleum ether

15% (v/v) ethyl ether/petroleum ether

50% (v/v) ethyl ether/petroleum ether

Preparation of Alumina

- Wash alumina to remove substances that interfere in GLC determination. Soak in methylene chloride ≥1 hr; decant and discard methylene chloride. Soak in hexane ≥1 hr; decant and discard hexane. Dry alumina on steam bath.
- Place washed and dried alumina in 260° C muffle furnace 4 hr. Transfer to tightly closed container and withdraw convenient amounts (*e.g.*, 500 g) for deactivation. Add water incrementally to final concentration of ≥16% (w/w) but ≤19% (w/w), shaking briefly after each addition. Caution: heat will be produced when water is added. Shake ≥4 hr on mechanical shaker. Store in tightly closed container. Alumina thus prepared has been found to be stable for at least 4 mon.

Directions

Dispersion on Alumina

- Cleanup is applicable to ≤2 g fat.
- Transfer concentrated solution of extracted fat to tared beaker, using small amounts of petroleum ether, and evaporate to dryness at steam bath temperature under current of dry air. Weigh and record weight of fat extracted. Calculate weight of sample analyzed as:

$$\frac{\text{weight fat taken for cleanup}}{\text{weight fat extracted}} \times \text{weight original sample}$$

- Weigh ≤2 g fat into 150 mL beaker containing 50 g deactivated alumina; mix well.
- Transfer mixture to homogenizer cup and add 350 mL 20% water/acetonitrile. Blend 2-4 min at high speed.
- Transfer most of contents, without rinsing, to Buchner fitted with filter paper; filter without suction. Alternatively, centrifuge blended contents 5 min at 1500 rpm and decant supernate through glass funnel with glass wool.
- Record mL solvent recovered; calculate g sample as:

$$\text{g sample} = \text{g fat} \times \frac{\text{mL solvent recovered}}{350}$$

Partitioning

- Transfer filtrate to 1 L separator containing 100 mL petroleum ether and shake vigorously 30 sec.
- Add 10 mL saturated sodium chloride solution and 500 mL water.
- Hold 1 L separator in horizontal position and thoroughly mix 30-45 sec. Let layers separate and drain aqueous layer into second 1 L separator.
- Add 100 mL petroleum ether to second 1 L separator, shake vigorously 15 sec, and let layers separate. Discard aqueous layer, combine petroleum ether with petroleum ether in original separator, and wash with two 100 mL portions water.
- Discard washings and drain petroleum ether layer through 25 mm × 50 mm column sodium sulfate into 500 mL K-D. Rinse separator and then column with three 10 mL portions petroleum ether.
- Add boiling chip to K-D and concentrate combined extract and rinses to 5-10 mL for transfer to Florisil column.

Florisil Column Cleanup

- Place activated Florisil (4" or weight determined by lauric acid value) in 22 mm id glass column; add about 0.5" anhydrous sodium sulfate. Prewet column with 40-50 mL petroleum ether. Place K-D with volumetric or graduated receiving flask under column to receive eluate.
- Transfer sample extract solution to column, letting it pass through at about 5 mL/min. Rinse container (and sodium sulfate if present) with two 5 mL portions petroleum ether, transfer rinsings to column, and rinse walls of chromatographic tube with additional small portions petroleum ether.
- Elute column at about 5 mL/min with 200 mL 6% ethyl ether/petroleum ether eluant.
- Change K-Ds and elute at about 5 mL/min with 200 mL 15% ethyl ether/petroleum ether eluant.
- Change K-Ds and elute at about 5 mL/min with 200 mL 50% ethyl ether/petroleum ether eluant.
- Add boiling chips to K-Ds and concentrate each eluate to suitable definite volume. When volume <5 mL is needed, use two-ball micro-Snyder or micro-Vigreaux column during final evaporation in receiving flask.
- Use appropriate determinative steps, such as DG1 or DG13, DG7, and DG10, to identify and measure residues.

ALTERNATIVE:

C9 DISPERSION ON ALUMINA, FLORISIL COLUMN CLEANUP, THREE METHYLENE CHLORIDE ELUANTS

Principles

Florisil is eluted with mixtures of methylene chloride, hexane, and acetonitrile. The resulting second eluate is cleaner than the second eluate of C8, although 90% of the fat placed on the column is eluted by the third eluant. The eluants can elute pesticide chemicals of a greater polarity than can be eluted by C8. C9 is preferred for analysis of fats and oils, for residues of endosulfan, and for separation of heptachlor epoxide and octachlor epoxide.

Additional Reagents

eluants: 1—20% methylene chloride in hexane (v/v). Dilute 200 mL methylene chloride with hexane. Allow mixture to reach room temperature, and adjust volume to 1 L with hexane.

2—50% methylene chloride/0.35% acetonitrile/49.65% hexane (v/v/v). Pipet 3.5 mL acetonitrile into 500 mL methylene chloride and dilute with hexane. Allow mixture to reach room temperature and adjust to 1 L with hexane.

3—50% methylene chloride/1.5% acetonitrile/48.5% hexane (v/v/v). Pipet 15 mL acetonitrile into 500 mL methylene chloride and dilute with hexane. Allow mixture to reach room temperature and adjust to 1 L with hexane.

Directions

- Follow C8 above, except replace the directions for Florisil column cleanup with the following:
- Place activated Florisil (4" or weight determined by lauric acid value) in 22 mm id glass column; add about 0.5" anhydrous sodium sulfate. Prewet column with 40-50 mL hexane. Place K-D with volumetric or graduated receiving flask under column to receive eluate.
- Transfer sample extract solution to column letting it pass through at about 5 mL/min. Rinse container (and sodium sulfate if present) with two 5 mL portions hexane, transfer rinsings to column, and rinse walls of chromatographic tube with additional small portions hexane.
- Elute column at about 5 mL/min with 200 mL eluant 1.
- Change K-Ds and elute with 200 mL eluant 2.
- Change K-Ds and elute with 200 mL eluant 3.
- Add boiling chips to K-Ds and concentrate each eluate to suitable definite volume. When volume <5 mL is needed, use two-ball micro-Snyder or micro-Vigreaux column during final evaporation in receiving flask.
- Use appropriate determinative steps, such as DG1 or DG13, DG7, and DG10, to identify and measure residues.

DETERMINATION

Inject concentrated extract equivalent to ≤3 mg fat (or 20 mg whole product) into the following GLC systems (Section 302) for determination of residues.

Minimum recommended determinations:

DG1 or **DG13**	residues with halogen, sulfur, or other moieties
or	
DG3 or **DG16**	organohalogen residues
DG10	late eluting residues, especially pyrethroids
or	
DG12	late eluting organohalogen residues, especially pyrethroids
DG7	early eluting residues with halogen, sulfur, or other moieties
DG2 or **DG14**	organophosphorus residues; large amounts of sulfur may interfere
DG4	organonitrogen residues; selective to nitrogen, but co-extractives may contain nitrogen
DG5 or **DG17**	organonitrogen and organophosphorus residues

For accurate quantitation, reference standards should be dissolved in same solvent as concentrated extract, only peaks >10% FSD should be measured, and peak sizes of residue and reference standard should match within ±25%.

See Chapter 5 for additional information about operation of GLC systems; Section 504 provides information about quantitation of residues.

See Section 205 for additional information about reference standards.

See Section 104 for additional information about reporting residues and determining compliance with regulations.

See Section 105 for additional information about analytical limits of quantitation.

CONFIRMATION

After residues have been tentatively identified and quantitated by comparison to appropriate reference standards, confirm identity according to principles discussed in Section 103. Use appropriate tables of data (PESTDATA, tables accompanying each method, Index to Methods) to choose most appropriate determinative steps.

Table 302-a: Recovery of Chemicals Through Method 302 (E1-E3 + DG1-DG19)
(acetone extraction, partitioning or Hydromatrix removal of water, GLC determination with various columns and detectors)

Chemical	Recovery[1]	Notes[2]
(4-chlorophenyl)-urea	NR	
1,2,4,5-tetrachloro-3-(methylthio)benzene	R	
1,2,4-triazole	V	N detector required.
2,3,5-trimethacarb	C	N detector required.
2,4,5-trichloro-alpha-methylbenzenemethanol	R	
2,4-dichloro-6-nitrobenzenamine	C (110%)	n=1
2,6-dichlorobenzamide	C	
2-chloroethyl myristate	C	
2-methoxy-3,5,6-trichloropyridine	C	Low temperature column recommended.
3,4,5-trimethacarb	C	N detector required.
3,4-dichloroaniline	V (44-84%)	
3,5-dichloroaniline	S (15-62%)	
3-(3,4-dichlorophenyl)-1-methoxyurea	R	GLC not reliable for quantitation.
3-carboxy-5-ethoxy-1,2,4-thiadiazole	NR	
3-chloro-5-methyl-4-nitro-1H-pyrazole	C	OV-101 peak tails severely.
3-ketocarbofuran	S (0-150%)	
3-methyl-4-nitrophenol	V (65-153%)	Interferences from sample extract may have caused variable results.
4'-hydroxy bifenthrin	C	High temperature GLC column required.
4-(dichloroacetyl)-1-oxa-4-azapiro[4.5]decane	C	Low level (0.05 ppm) fortification in corn grain obscured by matrix.
4-(phenylamino)phenol	C	
4-chlorobenzenamine	S (23-43%)	
4-chlorophenoxyaniline	S (10-29%)	Poor EC detector sensitivity; halogen-selective detector required.
6-benzyladenine	C	N detector required.
acephate	C	Wide bore or DEGS column required.
acetochlor	C	
acrinathrin	V (80-136%)	

[1] Codes: C: complete (>80%); P: partial (50-80%); S: small (<50%); V: variable (approximate percentage when known); R: recovered but no quantitative information available; NR: not recovered.

[2] Notes assume that extract is examined by GLC with columns at 200° C and, at a minimum, halogen-selective detector (DG3 or 16) and phosphorus-selective detectors (DG2 or 14 or 19). Notes indicate those chemicals that can be determined only by use of columns, temperatures, and/or detectors other than the minimal ones.

Table 302-a: Recovery Through 302 (E1-E3 + DG1-DG19)

Chemical	Recovery[1]	Notes[2]
alachlor	C	
aldrin	C	
allidochlor	C	Low temperature DEGS column used.
alpha-cypermethrin	C	
ametryn	C	N or S detector required.
aminocarb	C	N detector required.
amitraz	S (0-70%)	N detector, high temperature column required.
anilazine	V	GLC response variable.
aramite	C	
atrazine	C	
azinphos-ethyl	C	
azinphos-methyl	C	DEGS column unsuitable.
azinphos-methyl oxygen analog	C	
bendiocarb	C	N detector required.
benfluralin	C	
benodanil	C	
benoxacor	C	
bensulide	C	Results may be variable with certain GLC systems.
benzoylprop-ethyl	P (79%)	
BF 490-1	C	
BF 490-2	C	
BF 490-9	C	
BHC, alpha-	C	
BHC, beta-	C	
BHC, delta-	C	
bifenox	C	
bifenthrin	V (66-133%)	
binapacryl	C	N detector required.
biphenyl	C	FID required.
bitertanol	C	GLC with high temperature column, N/P detector required.
bromacil	C	
bromophos	C	
bromophos-ethyl	C	

Table 302-a: Recovery Through 302 (E1-E3 + DG1-DG19)

Chemical	Recovery[1]	Notes[2]
bromopropylate	C	
bromuconazole	C	
BTS 27919	C	N detector required.
Bulan	C	
bupirimate	C	N or S detector required.
butachlor	C	
butralin	V (77-90%)	N detector required.
butylate	V (73-99%)	N detector required.
cadusafos	C	
captafol	C	
captan	C	
carbaryl	C	N detector required for GLC determination; GLC not the method of choice.
carbetamide	C	N detector required.
carbofuran	C	N detector required for GLC determination; GLC not the method of choice.
carbophenothion	C	
carbophenothion oxygen analog	C	
carbophenothion sulfone	C	
carbosulfan	P (47-75%)	N or S detector required.
carboxin	C	N or S detector required.
CGA 100255	S (37-146%)	N detector required, but response is poor.
CGA 118244	V	
CGA 14128	C	
CGA 150829	V (40-111%)	N detector required; wide bore or DEGS column recommended.
CGA 171683	C	N detector, wide bore or DEGS column required.
CGA 37734	C	N detector required but response variable.
CGA 91305	V	
CGA 94689A	V (44-108%)	N detector required.
CGA 94689B	S (39-94%)	N detector required, but response varies widely with different columns.
CGA-232449	C	Needs N detector.
chlorbenside	C	
chlorbromuron	V (73-100%)	

Table 302-a: Recovery Through 302 (E1-E3 + DG1-DG19)

Chemical	Recovery[1]	Notes[2]
chlorbufam	C	
chlordane	C	
chlordane, cis-	C	
chlordane, trans-	C	
chlordimeform hydrochloride	P (80%)	
chlorethoxyfos	V (64-119%)	Recoveries performed with electron capture detector.
chlorfenapyr	P (73-82%)	
chlorfenvinphos, alpha-	C	
chlorfenvinphos, beta-	C	
chlorflurecol methyl ester	C	
chlorimuron ethyl ester	P (69-70%)	
chlormephos	C	Low temperature column required.
chlornitrofen	C (80%)	
chlorobenzilate	C	
chloroneb	C	Low temperature column required.
chloropropylate	P (64%)	
chlorothalonil	S	Recovery may be 0%.
chlorothalonil trichloro impurity	R	
chloroxuron	C	
chlorpropham	C	
chlorpyrifos	C	
chlorpyrifos oxygen analog	C	Wide bore or DEGS column recommended.
chlorpyrifos-methyl	C	
chlorthiophos	C	
chlorthiophos oxygen analog	C	
chlorthiophos sulfone	C	
chlorthiophos sulfoxide	C	
clodinafop-propargyl	V	Recovery test yielded very high recoveries (>200%) from wheat.
clofentezine	R	Degrades on GLC in presence of extract.
clomazone	C	
cloquintocet-mexyl	V (57-137%)	
coumaphos	C	

Table 302-a: Recovery Through 302 (E1-E3 + DG1-DG19)

Chemical	Recovery[1]	Notes[2]
coumaphos oxygen analog	C	
CP 51214	C	
crotoxyphos	C	
crufomate	C	
cyanazine	C	
cyanofenphos	C	
cyanophos	C	
cycloate	C	N or S detector required.
cycluron	C	N detector required.
cyfluthrin	C	High temperature column required.
cymoxanil	V (70-107%)	N detector required; GLC rrts and responses variable.
cypermethrin	C	
cyprazine	C	
cyproconazole	C	
cyprodinil	C	Needs N detector
cyromazine	S (16-20%)	
dazomet	S (<10%)	
DCPA	C	
DDE, o,p'-	C	
DDE, p,p'-	C	
DDT, o,p'-	C	
DDT, p,p'-	C	
deltamethrin	C	
demeton-O	C	
demeton-O sulfone	C	
demeton-O sulfoxide	C	
demeton-S	C	
demeton-S sulfone	C	Wide bore or DEGS column recommended.
demeton-S sulfoxide	C	Wide bore or DEGS column required.
des N-isopropyl isofenphos	C	
desisopropyl iprodione	P (67-84%)	
desmethyl norflurazon	V (63-200%)	
di-allate	C	

Table 302-a: Recovery Through 302 (E1-E3 + DG1-DG19)

Chemical	Recovery[1]	Notes[2]
dialifor	C	
diazinon	C	
diazinon oxygen analog	C (80%)	
dichlobenil	C	Low temperature column required.
dichlofenthion	C	
dichlofluanid	C	
dichlone	P (55%)	May break down.
dichlorvos	C	Low temperature column required; wide bore or DEGS recommended.
diclobutrazol	C	Wide bore column recommended.
diclofop-methyl	C	
dicloran	C	
dicofol, o,p'-	C	
dicofol, p,p'-	C	
dicrotophos	C	Wide bore or DEGS column required.
dieldrin	C	
diethatyl-ethyl	C	
difenoxuron	R	79-95% recovered at 1 and 5.5 ppm, but subject to interferences.
dimethachlor	C	
dimethametryn	C	N or S detector required.
dimethipin	C	
dimethoate	C	Wide bore or DEGS column recommended.
dimethomorph (prop)	V (87-133%)	High temperature column required.
dinitramine	C	N detector required.
dinobuton	C	
dinocap	C	N detector required.
dioxabenzofos	C	
dioxacarb	C	N detector required; used Megabore Carbowax column.
dioxathion	V (72-94%)	
diphenamid	V (57-155%)	N detector required.
diphenyl 2-ethylhexyl phosphate	C	mean recovery 104.2%, n=15
diphenylamine	C	N detector required.
disulfoton	C	

Table 302-a: Recovery Through 302 (E1-E3 + DG1-DG19)

Chemical	Recovery[1]	Notes[2]
disulfoton sulfone	C	
disulfoton sulfoxide	C	Wide bore or DEGS column required.
dithianon	NR	Breaks down in presence of extract.
diuron	C	Low temperature column required.
edifenphos	C	High recovery (113-121%) reported.
endosulfan I	C	
endosulfan II	C	
endosulfan sulfate	C	
endrin	C	
endrin aldehyde	C	
EPN	C	
esfenvalerate	C	High temperature column required.
etaconazole	C	Wide bore column recommended.
ethalfluralin	C	
ethephon	NR	
ethiofencarb	C	N or S detector required; responses variable.
ethiolate	C	Low temperature column, N or S detector required.
ethion	C	
ethion oxygen analog	C	
ethirimol	P (73%)	
ethofumesate	C	S selective detector required.
ethoprop	C	
ethoxyquin	C	N detector required.
ethyl p-toluene sulfonamide	C	N or S detector required.
ethylenethiourea	S (0-48%)	Short, low temperature DEGS or wide bore column, N or S detector required.
etridiazole	C	Low temperature column recommended.
etrimfos	C	
etrimfos oxygen analog	C	
famphur	C	
famphur oxygen analog	C	Quantitation affected by poor GLC.
fenamiphos	C	
fenamiphos sulfone	C	

Table 302-a: Recovery Through 302 (E1-E3 + DG1-DG19)

Chemical	Recovery[1]	Notes[2]
fenamiphos sulfoxide	C	
fenarimol	C	
fenarimol metabolite B	NR	
fenarimol metabolite C	S (6%)	
fenbuconazole	C	
fenfuram	C	
fenitrothion	C	
fenitrothion oxygen analog	C	
fenoxaprop ethyl ester	S (0-40%)	
fenoxycarb	C	N detector required.
fenpropimorph	C	N detector required.
fensulfothion	C	
fensulfothion oxygen analog	C	
fensulfothion sulfone	C	
fenthion	C	
fenthion oxygen analog	C	
fenthion oxygen analog sulfoxide	C	
fenthion sulfone	C	
fenvalerate	C	High temperature column required.
fipronil	S (0-72%)	Corn forage sample interfered with determination.
flamprop-M-isopropyl	C	
flamprop-methyl	C	
fluazifop butyl ester	C (78-112%)	
fluchloralin	C	
flucythrinate	C	High temperature column required.
fludioxonil	V (49-121%)	Requires N detector.
flusilazole	C	Wide bore column recommended.
fluvalinate	C	High temperature column required.
FOE 5043	C	
folpet	C	
fonofos	C	
fonofos oxygen analog	V (57-108%)	
formothion	C	

Table 302-a: Recovery Through 302 (E1-E3 + DG1-DG19)

Chemical	Recovery[1]	Notes[2]
fosthiazate	C	
fuberidazole	C	May break down in solution. Temp program separated from interference in tomato.
furilazole	C	
G-27550	C	N detector required.
Gardona	C	
heptachlor	C	
heptachlor epoxide	C	
heptenophos	C	
hexachlorobenzene	C	
hexaconazole	C	
hexazinone	P (57-76%)	N detector required; high temperature column may be needed.
imazalil	C	Wide bore column recommended.
imazamethabenz methyl ester	C	N detector required, though halogen-selective detector may respond.
IN-A3928	S (23-39%)	
IN-B2838	P (75-84%)	
IN-T3935	S (20%)	
IN-T3936	S (29-34%)	
IN-T3937	S (25%)	N detector required.
iprobenfos	C	
iprodione	C	
iprodione metabolite isomer	C	
isazofos	C	
isocarbamid	C	
isofenphos	C	
isofenphos oxygen analog	C	
isopropalin	C	N detector required.
isoprothiolane	C	
isoproturon	S (44-67%)	GLC poor; requires wide bore column; compound may degrade.
isoxaben	C	N detector required.
isoxaflutole	NR	Crop interference may have prevented measurement of recovery.

Table 302-a: Recovery Through 302 (E1-E3 + DG1-DG19)

Chemical	Recovery[1]	Notes[2]
jodfenphos	C	
kresoxim-methyl	P (73-89%)	
KWG 1323	C	
lambda-cyhalothrin	C	
lenacil	C	N detector required.
leptophos	C	
leptophos oxygen analog	C	
leptophos photoproduct	C	
lindane	C	
linuron	V (57-101%)	
malathion	C	
malathion oxygen analog	C	
MB 46513	C	
MB45950	S (0-35%)	
MB46136	S (0-19%)	
mecarbam	C	
mefluidide	R	123% recovered of 3 ppm added; subject to interference, poor GLC.
melamine	NR	
mephosfolan	C	
metalaxyl	C	N detector required but response variable.
metasystox thiol	C	
metazachlor	C	
methabenzthiazuron	C (85-86%)	
methamidophos	V	For complete recovery, use variation from PAM I 302 E5/E6
methidathion	C	
methiocarb	C	N or S detector required for GLC determination; GLC not the method of choice.
methiocarb sulfone	S	Some reports of no recovery; N or S detector required.
methiocarb sulfoxide	P (60-80%)	GLC not preferred, requires N or S detector, wide bore or DEGS column.
methoprotryne	C	Wide bore column recommended.
methoxychlor olefin	C	

Table 302-a: Recovery Through 302 (E1-E3 + DG1-DG19)

Chemical	Recovery[1]	Notes[2]
methoxychlor, p, p'-	C	
methyl 4-chloro-1H-indole-3-acetate	R	
metobromuron	C	
metolachlor	C	
metolcarb	C	N detector, wide bore, DEGS, or low temperature OV-17 column required.
metoxuron	V (73-110%)	Requires low temperature column.
metribuzin	V	N or S detector required.
metribuzin, deaminated diketo metabolite	NR	
metribuzin, deaminated metabolite	C	N or S detector required.
metribuzin, diketo metabolite	NR	
mevinphos, (E)-	C	Wide bore or DEGS column required for separation from (Z)-.
mevinphos, (Z)-	C	Wide bore or DEGS column required for separation from (E)-.
MGK 264	C	
mirex	P (71-83%)	
molinate	C	Recovery tested at 0.053 and 0.264 ppm.
monocrotophos	C	Response enhanced by co-extractives. Wide bore or DEGS column required.
monolinuron	C	
myclobutanil	C	Wide bore column recommended.
myclobutanil alcohol metabolite	S (30-55%)	Poor N/P detector sensitivity.
myclobutanil dihydroxy metabolite	NR	
N, N-diallyl dichloroacetamide	C	
naled	C	May break down to dichlorvos on GLC column. Wide bore or DEGS column required.
napropamide	C	N detector, wide bore or DEGS column required.
neburon	C	
nitralin	C	N or S detector required.
nitrapyrin	C	
nitrofen	C	
nitrofluorfen	C	
nitrothal-isopropyl	C	N detector required.
nonachlor, cis-	C	

Table 302-a: Recovery Through 302 (E1-E3 + DG1-DG19)

Chemical	Recovery[1]	Notes[2]
nonachlor, trans-	C	
norea	C	N detector required.
norflurazon	V (60-200%)	
nuarimol	C	
octachlor epoxide	C	
octhilinone	C	N or S detector required.
ofurace	C	
omethoate	C	Wide bore or DEGS column required.
ovex	C	
oxadiazon	C	
oxadixyl	C	N detector required.
oxamyl oxime metabolite	C	Lower temperature column needed to separate from coextractives.
oxycarboxin	R	Matrix enhancement of response causes high results.
oxydemeton-methyl	C	Wide bore or DEGS column required.
oxydemeton-methyl sulfone	C	Wide bore or DEGS column required; poor GLC makes quantitation questionable.
oxyfluorfen	C	Poor N/P detector sensitivity.
oxythioquinox	C	N or S detector required; wide bore or short DEGS column recommended.
paclobutrazol	C	Wide bore column recommended.
parathion	C	
parathion oxygen analog	C	
parathion-methyl	C	
PB-9	V (106-215%)	
pebulate	C	
penconazole	C	Wide bore column recommended.
pendimethalin	C	N detector required.
pentachloroaniline	C	
pentachlorobenzene	C	
pentachlorobenzonitrile	C	
pentachlorophenyl methyl ether	C	
pentachlorophenyl methyl sulfide	C	
permethrin, cis-	C	High temperature column recommended.

Table 302-a: Recovery Through 302 (E1-E3 + DG1-DG19)

Chemical	Recovery[1]	Notes[2]
permethrin, trans-	C	High temperature column recommended.
Perthane	C	
phenthoate	C	
phenylphenol, o-	C	FID required.
phorate	C	
phorate oxygen analog	C	
phorate oxygen analog sulfone	C	
phorate oxygen analog sulfoxide	C	GLC retention times and responses variable.
phorate sulfone	C	GLC variable.
phorate sulfoxide	C	GLC retention times and responses variable.
phosalone	C	
phosalone oxygen analog	C	Poor GLC detector sensitivity.
phosfolan	C	
phosmet	C	
phosphamidon	C	
phoxim	C	Low temperature column required; degrades at 200°.
phoxim oxygen analog	C	
piperophos	C	
pirimicarb	C	N detector required.
pirimiphos-ethyl	C	
pirimiphos-ethyl oxygen analog	C	
pirimiphos-methyl	C	
pretilachlor	C	
probenazole	C	N or S detector required; FPD-S more sensitive than N/P.
prochloraz	C	High temperature column required.
procyazine	C	
procymidone	C	
prodiamine	C	Recoveries of 0.5 and 1 ppm from apples: 110, 125%, respectively.
profenofos	C	
profluralin	V (40-90%)	
Prolan	P (58%)	

Table 302-a: Recovery Through 302 (E1-E3 + DG1-DG19)

Chemical	Recovery[1]	Notes[2]
promecarb	V	N detector required; GLC not determinative step of choice.
prometryn	C	N or S detector required.
pronamide	C	
propachlor	C	
propanil	C	
propargite	C	S detector required.
propazine	C	
propetamphos	C	
propham	C	N detector required; low temperature column recommended.
propiconazole	C	Wide bore column recommended.
propoxur	C	N detector required for GLC.
prothiofos	C	
prothoate	C	
PYPAC	V (144-162%)	Low temperature column, N detector required.
pyracarbolid	C	N detector required.
pyrazon	C	Wide bore column recommended.
pyrazophos	C	
pyridaphenthion	C	S detector is less sensitive than FPD or N/P.
pyrimethanil	C	
pyriproxyfen	C	N detector required.
quinalphos	C	
quintozene	C	
quizalofop ethyl ester	C	Wide bore column recommended.
RH-6467	S (0-17%)	
RH-9129	V (68-92%)	
RH-9130	P (48-71%)	
ronnel	C	
ronnel oxygen analog	C	
RPA202248	NR	
schradan	C	
SDS-67131	C	
simazine	C	

Table 302-a: Recovery Through 302 (E1-E3 + DG1-DG19)

Chemical	Recovery[1]	Notes[2]
simetryn	C	N or S detector required.
sulfallate	C	
sulfanilamide	NR	
sulfotep	C	Wide bore or DEGS column recommended.
Sulphenone	C	
sulprofos	C	
sulprofos oxygen analog sulfone	C	
sulprofos sulfone	C	
sulprofos sulfoxide	C	
TCMTB	C	
TDE, p,p'-	C	
TDE, p,p'-, olefin	C	
tebuconazole	C	
tecnazene	C	
tefluthrin	C	Recovery tested at 0.275 and 1.374 ppm.
TEPP	C	
terbacil	C	
terbufos	C	
terbufos oxygen analog	C	
terbufos oxygen analog sulfone	C	
terbufos sulfone	C	
terbumeton	C	Recoveries of 0.5 and 1 ppm from apples: about 120%.
terbuthylazine	C	
terbutryn	C	N or S detector required.
tetradifon	C	
tetramethrin	C	
tetrasul	C	
thiabendazole	C	N or S detector required for GC determination.
thiazopyr	C	Recovery at 0.5 ppm; interferences prevented measurement at 0.1 ppm.
thiobencarb	C	
thiometon	C	Degrades while standing in extract.
thionazin	C	

Table 302-a: Recovery Through 302 (E1-E3 + DG1-DG19)

Chemical	Recovery[1]	Notes[2]
THPI	C	N detector, wide bore or DEGS column required.
tolylfluanid	C	
toxaphene	C	
tralkoxydim	V (38-106%)	Recoveries of two OV-101 peaks are different from one another.
tralomethrin	C	
tri-allate	C	
triadimefon	C	Wide bore column recommended.
triadimenol	C	Wide bore column recommended.
triazamate	C	
triazophos	C	
tribufos	C	
trichlorfon	C	Often converts to dichlorvos on GLC column. Wide bore or DEGS column required.
tricyclazole	C	N or S detector required; wide bore column recommended.
tridiphane	C	
trietazine	C	Recovery tested at 0.11 and 0.55 ppm.
triflumizole	C	Wide bore column recommended.
trifluralin	C	
triflusulfuron methyl ester	V (67-106%)	
triphenyl phosphate	C	
tris(2-ethylhexyl) phosphate	C (68-112%)	mean recovery 97.6%, n=11
tris(beta-chloroethyl) phosphate	C	
tris(chloropropyl) phosphate	C	
Tycor	C	May break down in solution. Temp program separated from interference in tomato.
vamidothion sulfone	C	
vinclozolin	C	
vinclozolin metabolite B	C	Severely subject to influence of matrix; levels <1.0 ppm had very high recovery.
vinclozolin metabolite E	C	Severely subject to influence of matrix; levels <1.0 ppm had very high recovery.
vinclozolin metabolite F	R	Poor chromatography, influence of matrix prevent quantitation of recovery.
vinclozolin metabolite S	V (59-137%)	

Table 302-b: Recovery of Chemicals Through Method 302 (E1-E3 + C5 + DG1-DG19)
(acetone extraction, partitioning or Hydromatrix removal of water, Florisil column cleanup, GLC determination with various columns and detectors)

Chemical	Recovery[1,2]	Eluant, C5[3]	Notes[4]
2,3-dihydro-3,3-methyl-2-oxo= -5-benzofuranyl methyl sulfonate	NR	15	No elution from Florisil only in 15% EE/PE; 50% not tested.
2,6-dichlorobenzamide	NR	50	83% elution from Florisil only in 200 mL ethyl ether.
2-hydroxy-2,3-dihydro-3,3-methyl= -5-benzofuranyl methyl sulfonate	NR	15	No elution from Florisil only in 15% EE/PE; 50% not tested.
2-methoxy-3,5,6-trichloropyridine	P (65%)	15	Low temperature column recommended.
acetochlor	C	15+50	80% eluted in 15% EE/PE.
alachlor	P (68%)	15	
aldrin	C	15	
allethrin	C (80%)	15	
atrazine	C	15+50	Eluted in 50% EE/PE.
azinphos-ethyl	C	15	
BHC, alpha-	C	15	
BHC, beta-	C	15	
BHC, delta-	C	15	
bifenthrin	C	15	
binapacryl	C (83%)	15	
bioresmethrin	NR	15	Some elution from Florisil in 200 mL 50% EE/PE, more in 200 mL 75% EE/PE.
biphenyl	C	15	FID required.
bromophos	C	15	
bromopropylate	C (80%)	50	
bupirimate	S (10-30%)	15	
captafol	NR	15	Some elution from Florisil in 50% EE/PE after 15%.

[1] Codes: C: complete (>80%); P: partial (50-80%); S: small (<50%); V: variable (approximate percentage when known); R: recovered but no quantitative information available; NR: not recovered.

[2] Recovery results refer to complete method; blank entry in this column indicates Florisil elution was tested but not complete method.

[3] Eluants(s) in which chemical is eluted from Florisil, according to directions in 302 C5, *i.e.*, 15 and 50% ethyl ether/petroleum ether (EE/PE). Entries for chemicals not recovered indicate which eluants were used in tests.

[4] "Florisil only" refers to tests in which elution patterns were tested by added reference standard solutions directly to Florisil column.

Table 302-b: Recovery Through 302 (E1-E3 + C5 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C5[3]	Notes[4]
captan	S (25%)	15	Additional elution from Florisil in 50% EE/PE after 15%.
carfentrazone ethyl ester	C	15	Some additional elution in 50% EE/PE possible.
chlorbenside	C	15	
chlordane	C	15	
chlordane, cis-	C	15	
chlordane, trans-	C	15	
chlordimeform hydrochloride	NR	15-50	
chlorfenapyr	C	15+50	
chlorflurecol methyl ester	NR	15	No elution from Florisil only in 15% EE/PE; 50% not tested.
chlornitrofen	C	15	
chlorothalonil	C (81%)	15	Additional elution from Florisil in 200 mL 50% EE/PE.
chlorpyrifos	C	15	
chlorpyrifos oxygen analog	NR	15	Elution from Florisil with 50% EE/PE not tested.
chlorpyrifos-methyl	C	15	
chlorthiophos oxygen analog	NR	15	No elution from Florisil only in 15% EE/PE; 50% not tested.
chlorthiophos sulfone	S (8%)	15	
chlorthiophos sulfoxide	NR	15	No elution from Florisil only in 15% EE/PE; 50% not tested.
clofentezine	S (27-50%)	15	Complete elution from Florisil only; degrades on GLC in presence of extract.
cymiazole	NR	6-15-50	No elution from Florisil only in 6, 15, or 50% EE/PE or 100% EE.
cypermethrin	C	15	
DCPA	C	15	
DDE, o,p'-	C	15	
DDE, p,p'-	C	15	
DDT, o,p'-	C	15	
DDT, p,p'-	C	15	
deltamethrin	C	15	Very poor EC detector sensitivity.
diazinon	C	15	
dichlofluanid	C	15+50	60% eluted in 15% EE/PE

Table 302-b: Recovery Through 302 (E1-E3 + C5 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C5[3]	Notes[4]
diclobutrazol	P	15	56-70% elution from Florisil only; 50% not tested; wide bore column recommended.
dicloran	C	15	
dicofol, o,p'-	C	15	
dicofol, p,p'-	C	15	May be variable.
dieldrin	C	15	
difenoxuron	NR	15	No elution from Florisil only in 15% EE/PE; 50% not tested.
dimethachlor	NR	15	No elution from Florisil only in 15% EE/PE; 50% not tested.
dinobuton	C (83%)	15	
endosulfan I	C	15	
endosulfan II	C	15	
endosulfan sulfate	C	15	Additional elution possible in 50% EE/PE.
endrin	C	15	
esfenvalerate	C	15	
etaconazole	S	15	30% elution from Florisil only; 50% not tested; wide bore column recommended.
fenfuram	P (45%)	15	Partial recovery also when Florisil eluted with MeCl2 eluant #3.
fenpropathrin	C	15	
fenson	C	15	
fensulfothion sulfone	NR	50	No elution from Florisil only in 50% EE/PE.
fenvalerate	C	15	
flamprop-M-isopropyl	NR	15	Complete elution from Florisil only in 50% EE/PE plus additional EE.
flamprop-methyl	NR	6-15-50	No elution from Florisil only in 6, 15, or 50% EE/PE; complete elution with EE.
fluchloralin	C	15	
flucythrinate	C	15+50	About 80% eluted in 15% EE/PE
flusilazole	S	15	35-44% elution from Florisil only; wide bore column recommended.
fluvalinate	C	15	
FOE 5043 thioglycolate sulfoxide	NR		not eluted from Florisil
folpet	C	15+50	78% eluted in 15% EE/PE
fonofos	C	15	

Table 302-b: Recovery Through 302 (E1-E3 + C5 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C5[3]	Notes[4]
fuberidazole	NR	15	No elution from Florisil only in 15% EE/PE; 50% not tested.
heptachlor	C	15	Elution from Florisil not always complete.
heptachlor epoxide	C	15	
hexachlorobenzene	C	15	Recovery test run with 6,15,50% EE/PE, elution in 6%.
hexaconazole	NR	15	
imazalil	NR	15	No elution from Florisil only in 15% EE/PE; 50% not tested.
iprodione	S (24-97%)	15	Complete elution from Florisil requires more polar eluants.
isocarbamid	NR	15	No elution from Florisil only in 15% EE/PE; 50% not tested.
lindane	C	15	
metalaxyl	NR	15	
methabenzthiazuron	NR	15	No elution from Florisil only in 15% EE/PE; 50% not tested.
methoprotryne	NR	15	No elution from Florisil only in 15% EE/PE; 50% not tested.
methyl 3,5-dichlorobenzoate	C	15	
mirex	C	15	
myclobutanil	NR	15	No elution from Florisil only in 15% EE/PE; 50% not tested.
nitrapyrin	C	15	
nitrofen	C	15	
nitrothal-isopropyl	C	15	
nonachlor, trans-	C	15	
norflurazon	NR	15	Elution from Florisil with 50% EE/PE not tested.
ovex	C	15	
oxadiazon	C	15	
oxythioquinox	C (79-96%)	15	
paclobutrazol	P	15	44-55% elution from Florisil only; 50% not tested; wide bore column recommended.
parathion	C	15	
parathion-methyl	C	15	

Table 302-b: Recovery Through 302 (E1-E3 + C5 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C5[3]	Notes[4]
penconazole	S	15	42-54% elution from Florisil only; 50% not tested; wide bore column recommended.
pentachloroaniline	C	15	Recovery test run with 6,15,50% EE/PE, elution in 6%.
pentachlorobenzene	C	15	Recovery test run with 6,15,50% EE/PE, elution in 6%.
pentachlorophenyl methyl sulfide	C	15	Recovery test run with 6,15,50% EE/PE, elution in 6%.
permethrin, cis-	C	15	High temperature column recommended.
permethrin, trans-	C	15	High temperature column recommended.
phenmedipham	NR	15	No elution from Florisil only in 15% EE/PE; 50% not tested.
phenothrin	P (60%)	15	No additional elution in 200 mL 50% EE/PE; wide bore column recommended.
phenylphenol, o-	C	15	FID required.
phorate sulfoxide	NR	15-50	Recovery test included elution from Florisil with 50% EE/PE only.
pirimicarb	S (25%)	15	
pretilachlor		15	Elution from Florisil only complete in 15% EE/PE.
procymidone	C	15	
prodiamine		15	Elution from Florisil only complete in 15% EE/PE.
propargite	C	15	
propham	C	15	N detector required; low temperature column recommended.
propiconazole	P	15	46-50% elution from Florisil only; 50% not tested; wide bore column recommended.
pyrethrins	C	15+50	Most eluted in 15% EE/PE.
quintozene	C	15	
quizalofop ethyl ester	C	15	
simazine	P (69%)	15	
TDE, p,p'-	C	15	
tebufenozide	NR	15	
TEPP	NR	15	
terbumeton	NR	15-50	No elution from Florisil only in 15 or 50% EE/PE.
terbuthylazine	C	15	

Table 302-b: Recovery Through 302 (E1-E3 + C5 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C5[3]	Notes[4]
tetradifon	C	15	
thiometon	C	15	
THPI	NR	15-50	Only small amount recovered (5%) in subsequent elution with 200 mL EE .
toxaphene	C	15	
tralomethrin	C	15	
triadimefon	S (7%)	15	72-84% elution from Florisil only; 50% not tested; wide bore column recommended.
triadimenol	S	15	40-45% elution from Florisil only; 50% not tested; wide bore column recommended.
tricyclazole	NR	15	No elution from Florisil only in 15% EE/PE; 50% not tested.
triflumizole	P	15	46-52% elution from Florisil only; 50% not tested; wide bore column recommended.
vinclozolin	C	15	

Table 302-c: Recovery of Chemicals Through Method 302 (E1-E3 + C3 + DL1)

(acetone extraction, partitioning or Hydromatrix removal of water, charcoal/silanized Celite column cleanup, HPLC with post-column hydrolysis and derivatization, fluorescence detection)

Chemical	Recovery[1]	Rrt[2]	ng[3]	Notes
2,3,5-trimethacarb	C			
3,4,5-trimethacarb	C			
3-hydroxycarbofuran	C	0.6	10	mean recovery 97.6%, n=45
aldicarb	C	0.83	14	mean recovery 89.2%, n=210
aldicarb sulfoxide	C	0.33	9	mean recovery 98.6%, n=108
aldoxycarb	C	0.4	9	mean recovery 102%, n=111
aminocarb	C			
bufencarb	C	1.44	19	Major peak is listed. mean recovery 97.4%, n=27
butocarboxim	S (0-108%)	0.75	15	mean recovery 56.1%, n=22
carbaryl	C	1.06	7	mean recovery 98.1%, n=147
carbofuran	C	1	10	mean recovery 97.4%, n=121
dioxacarb	P (72%)	0.67	15	
isoprocarb	C	1.13	8	
methiocarb	C	1.26	10	mean recovery 99.9%, n=67
methomyl	C	0.46	10	mean recovery 94.1%, n=128
metolcarb	C	0.85	10	mean recovery 90.7%, n=12
oxamyl	C	0.44	10	mean recovery 94.3%, n=41
promecarb	C	1.31	10	mean recovery 99.9%, n=29
propoxur	C	0.98	8	mean recovery 92.2%, n=48
XMC	C	1.06	10	mean recovery 95.6%, n=28

[1] Codes: C: complete (>80%); P: partial (50-80%); S: small (<50%); V: variable (approximate percentage when known); R: recovered but no quantitative information available; NR: not recovered.

[2] Retention time, relative to carbofuran, on C-8 column described in DL1.

[3] ng that cause 50% full scale deflection detector response in DL1.

Table 302-d: Recovery of Chemicals Through Method 302 (E2/E3 + C1 + DG1-DG19)
(acetone extraction, Hydromatrix removal of water, Florisil cleanup with one methylene chloride eluant, GLC determination with various columns and detectors)

Chemical	Recovery[1]	Notes[2]
1,2,3,5-tetrachlorobenzene	V (63-141%)	mean recovery 91.6%, n=16
2,3,5,6-tetrachloroaniline	C (67-110%%)	mean recovery 85.5%, n=10
2,4-dichloro-6-nitrobenzenamine	V (65-123%)	mean 91%, n=7
aldrin	C	mean recovery 86.9%, n=16
allethrin	C	mean recovery 91.8%, n=4
alpha-cypermethrin	C	mean recovery 98.0%, n=15
azafenidin	V (45-160%)	High Temperature column required.
BHC, alpha-	V (68-89.5%)	mean recovery 79.0%, n=2
bifenthrin	C (59-110%)	mean recovery 91.4%, n=15
bromopropylate	NR	
butachlor	C	mean recovery 91.7%, n=2
captafol	C	mean recovery 101.7%, n=4
captan	V (0-139%)	mean recovery 65.2%, n=20
carbaryl	C	recovery 107%, n=1
chlordane	P (64%)	mean recovery 64.4%, n=2
chlordane, cis-	C	
chlordane, trans-	C	
chlorobenzilate	NR	mean recovery 5.5%, n=11
chlorothalonil	S (0-93%)	mean recovery 36.9%, n=17
chlorpropham	V (76-95%)	mean recovery 86.1%, n=2
chlorpyrifos	C	mean recovery 88.9%, n=27
chlorpyrifos-methyl	V (54-116%)	mean recovery 80.6%, n=16
clodinafop-propargyl	V (56-104%)	
cloquintocet-mexyl	NR	Not eluted from Florisil only.
cyfluthrin	V (60-117%)	mean recovery 86.5%, n=15
cypermethrin	C	
DCPA	P	mean recovery 77.0%, n=4
DDE, o,p'-	C	

[1] Codes: C: complete (>80%); P: partial (50-80%); S: small (<50%); V: variable (approximate percentage when known); R: recovered but no quantitative information available; NR: not recovered.

[2] Notes assume that extract is examined by GLC with columns at 200° C and, at a minimum, halogen-selective detector (DG3 or 16) and phosphorus-selective detectors (DG2 or 14 or 19). Notes indicate those chemicals that can be determined only by use of columns, temperatures, and/or detectors other than the minimal ones.

Table 302-d: Recovery Through 302 (E2/E3 + C1 + DG1-DG19)

Chemical	Recovery[1]	Notes[2]
DDE, p,p'-	C	mean recovery 83.7%, n=6
DDT, o,p'-	V (58-111%)	mean recovery 86.3%, n=15
DDT, p,p'-	C	mean recovery 91.4%, n=2
deltamethrin	C	mean recovery 88.5%, n=5
diazinon	C	
dichlone	P	recovery 58.6%, n=1
diclofop-methyl	V (56-135%)	mean recovery 88.2%, n=29
dicloran	V (57-118%)	mean recovery 82.1%, n=14
dicofol, o,p'-	C	
dicofol, p,p'-	C	mean recovery 107%, n=4
dieldrin	C	mean recovery 88.4%, n=146
endosulfan I	V (64-89%)	mean recovery 76.2%, n=4
endosulfan II	C	mean recovery 93.6%, n=4
endosulfan sulfate	C	mean recovery 91.9%, n=28
endrin	C	mean recovery 99.1%, n=2
esfenvalerate	V (70-138%)	mean recovery 95.9%, n=16
fenarimol	S (0-33%)	mean recovery 13%, n=21
fenhexamid	NR	Not recovered from Florisil only.
fenoxaprop ethyl ester	C	mean recovery 97.8%, n=2
fenvalerate	V (65-162%)	mean recovery 93.8%, n=21
fluchloralin	C	mean recovery 91.8%, n=16
fluvalinate	C (64-113%)	mean recovery 93.4%, n=10
folpet	C	mean recovery 120%, n=4
haloxyfop methyl ester	C	recovery 126%, n=1
heptachlor	C	mean recovery 81.7%, n=2
heptachlor epoxide	V (58-118%)	mean recovery 87.3%, n=49
hexachlorobenzene	C	
hexythiazox	V (36-89%)	
iprodione	S (0-95%)	mean recovery 32.4%, n=26. Complete recovery requires 50% EE/PE eluant.
iprodione metabolite isomer	V (32-149%)	mean recovery 85.0%, n=24
isopropalin	C	mean recovery 85.8%, n=4
lambda-cyhalothrin	C	mean recovery 106%, n=16, range 87-133%
lindane	C	mean recovery 84.7%, n=54

Table 302-d: Recovery Through 302 (E2/E3 + C1 + DG1-DG19)

Chemical	Recovery[1]	Notes[2]
linuron	C	mean recovery 88.3%, n=18
methamidophos	C	mean recovery 91.2%, n=1
methoxychlor olefin	C (63-104%)	mean recovery 88.2%, n=10
methoxychlor, o, p'-	C	72-123% recoveries, TDS
methoxychlor, p, p'-	V (76-130%)	mean recovery 100.9%, n=19
metolachlor	NR	
mirex	V (37-110%)	mean recovery 79.3%, n=15
nitrapyrin	V (69-123%)	mean recovery 96.1%, n=2
nonachlor, cis-	C	
nonachlor, trans-	C	
nuarimol	NR	
octachlor epoxide	C	mean recovery 91.5%, n=23
oxadiazon	C	mean recovery 89.4%, n=2
oxyfluorfen	C	
parathion	C	mean recovery 117%, n=1
pentachloroaniline	C	
pentachlorobenzene	C	
pentachlorophenyl methyl ether	C	mean recovery 89.5%, n=1
pentachlorophenyl methyl sulfide	V (49-112%)	mean recovery 80.7%, n=15
permethrin, cis-	C	mean recovery 91.6%, n=4
permethrin, trans-	C	mean recovery 93.5%, n=4
phenylphenol, o-	V (76-129%)	FID required; mean recovery 97.6%, n=16
phosalone	V (27-116%)	mean recovery 75.4%, n=16
procymidone	C	mean recovery 90.7%, n=3
propanil	C	mean recovery 100.2%, n=2
propargite	V (71-125%)	mean recovery 93%, n=23
prothiofos	V (36-127%)	mean recovery 75.8%, n=21
pyrethrins	C	mean recovery 83.5%, n=6
quintozene	P	mean recovery 79.6%, n=14
sulfallate	V (39-87%)	mean recovery 58.9%, n=5
TDE, o,p'-	V (70-145%)	mean recovery 97.7%, n=17
TDE, p,p'-	C	
TDE, p,p'-, olefin	V (41-128%)	mean recovery 78.4%, n=21
tecnazene	C	mean recovery 83.2%, n=1

Table 302-d: Recovery Through 302 (E2/E3 + C1 + DG1-DG19)

Chemical	**Recovery[1]**	**Notes[2]**
tetradifon	C	mean recovery 111%, n=4
thiobencarb	C	mean recovery 90.4%, n=2
toxaphene	C	mean recovery 94.1%, n=5
tralomethrin	C (67-103%)	mean recovery 87.5%, n=11
tridiphane	V (54-110%)	mean recovery 85.1%, n=16
trifluralin	P	mean recovery 57.0%, n=3
vinclozolin	V (61-109%)	mean recovery 86.8%, n=14

Table 302-e: Recovery of Chemicals Through Method 302 (E1/E4 + C4 + DL1)
(acetone extraction, partitioning to remove water, C-18 cartridge cleanup, HPLC with post-column derivatization and fluorescence detection)

Chemical	Recovery[1]	Rrt[2]	ng[3]	Notes
3-hydroxycarbofuran	C	0.6	10	mean recovery 94.7%, n=2
aldicarb	C	0.83	14	mean recovery 87.4%, n=8
aldicarb sulfoxide	C	0.33	9	mean recovery 89.6%, n=9
aldoxycarb	V (70-104%)	0.4	9	mean recovery 88.7%, n=8
bitertanol	C			GLC with high temperature column, N/P detector required.
bufencarb	C	1.44	19	Major peak is listed. recovery 107%, n=1
carbaryl	C	1.06	7	mean recovery 88.9%, n=45
carbofuran	C	1	10	mean recovery 96.2%, n=3
dioxacarb	C	0.67	15	recovery 91.1%, n=1
methiocarb	C	1.26	10	mean recovery 97.9%, n=5
methiocarb sulfoxide	S	0.64	12	recovery 42.0%, n=1
methomyl	C	0.46	10	mean recovery 96.4%, n=36
oxamyl	C	0.44	10	mean recovery 95.8%, n=33
phenylphenol, o-	C			DL2 required; mean recovery 86.9%, n=8.
piperonyl butoxide	C			mean recovery 91.8%, n=5
pronamide	C			
propoxur	C	0.98	8	mean recovery 85.2%, n=6

[1] Codes: C: complete (>80%); P: partial (50-80%); S: small (<50%); V: variable (approximate percentage when known); R: recovered but no quantitative information available; NR: not recovered.

[2] Retention time, relative to carbofuran, on C-8 column described in DL1.

[3] ng that cause 50% full scale deflection detector response in DL1.

Table 302-f: Recovery of Chemicals Through Method 302 (E7 + C6 + DG1-DG3, DG6-DG7, DG10, DG13-DG14, or DG16)
(acetone extraction and solid phase extraction cartridges, liquid-liquid partitioning, SAX cartridge cleanup, GLC determination)

Chemical	Recovery[1]	Notes
3-hydroxycarbofuran	C	Determination by DL1.
acephate	C	
aldicarb	C	Determination by DL1.
aldicarb sulfoxide	C	Determination by DL1.
alpha-cypermethrin	C	
atrazine	C	
azinphos-methyl	C	Recoveries tend to be >100%.
BHC, alpha-	C	
BHC, beta-	C	Recoveries tend to be >100%.
BHC, delta-	C	
bitertanol	C	GLC with high temperature column, N/P detector required.
carbaryl	C	Determination by DL1.
carbendazim	C	Determined by UV detector at 280 nm.
carbofuran	C	Determination by DL1.
carbophenothion	C	
chlorothalonil	C	based on two recoveries
chloroxuron	C	Determination by DL3.
chlorpropham	C	
chlorpyrifos	C	
chlorpyrifos-methyl	C	
chlorthiophos	C	
cyanazine	C	
cyfluthrin	C	
DCPA	C	
DDE, o,p'-	C	
DDE, p,p'-	C	
DDT, o,p'-	C	
DDT, p,p'-	C	

[1] Codes: C: complete (>80%); P: partial (50-80%); S: small (<50%); V: variable (approximate percentage when known); R: recovered but no quantitative information available; NR: not recovered.

Table 302-f: Recovery Through 302 (E7 + C6 + DG1-DG3, DG6-DG7, DG10, DG13-DG14, or DG16)

Chemical	Recovery[1]	Notes
deltamethrin	C	
demeton-O	C	
demeton-S sulfone	C	Recoveries tend to be >100%.
diazinon	C	
dichlofluanid	C	Recoveries tend to be >100%.
dichlorvos	P (75%)	
dicloran	C	
dicofol, p,p'-	C	
dicrotophos	C	
dieldrin	C	
dimethoate	C	
dioxathion	C	
diphenylamine	C	N detector required.
disulfoton sulfone	C	
diuron	C	Determination by DL3.
endosulfan I	C	
endosulfan II	C	
endosulfan sulfate	C	
endrin	C	
EPN	C	
esfenvalerate	C	
etaconazole	C	
ethion	C	
fenamiphos	P (79%)	
fenarimol	V (79, 99%)	
fenpropimorph	C	N detector required
fenthion	C	
fenuron	C	Determination by DL3.
fluridone	P (65%)	
folpet	C	
hexachlorobenzene	P (76%)	
imazalil	C	
iprodione	C	
lindane	C	

Table 302-f: Recovery Through 302 (E7 + C6 + DG1-DG3, DG6-DG7, DG10, DG13-DG14, or DG16)

Chemical	Recovery[1]	Notes
linuron	C	Determination by DL3.
malathion	C	
methamidophos	C	
methidathion	C	
methiocarb	C	Determination by DL1.
methomyl	C	Determination by DL1.
methoxychlor, o, p'-	C	
methoxychlor, p, p'-	C	
metobromuron	C	Determination by DL3.
metoxuron	C	Determination by DL3.
mevinphos, (E)-	C	
mevinphos, (Z)-	C	
monocrotophos	C	
monolinuron	C	Determination by DL3.
monuron	C	Determination by DL3.
myclobutanil	C	
neburon	C	Determination by DL3.
omethoate	C	Wide bore or DEGS column required.
oryzalin	C	N or S detector required.
oxamyl	C	Determination by DL1.
parathion	C	
parathion oxygen analog	C	
parathion-methyl	C	
parathion-methyl oxygen analog		C
penconazole	C	
pentachloroaniline	C	
permethrin, cis-	C	
permethrin, trans-	C	
phenylphenol, o-	C	
phorate sulfone	C	
phosalone	C	
phosmet	C	
pirimiphos-ethyl	C	
pirimiphos-methyl	C	

Table 302-f: Recovery Through 302 (E7 + C6 + DG1-DG3, DG6-DG7, DG10, DG13-DG14, or DG16)

Chemical	Recovery[1]	Notes
procymidone	C	
propanil	C	
propiconazole	C	
prothiofos	C	
pyrazophos	C	
pyridaphenthion	C	
quintozene	C	
ronnel	C (122%)	n=1
simazine	C	
sulfotep	C	
TDE, p,p'-	C	
tetradifon	C	
thiabendazole	C	Determined by UV detector at 280 nm; confirm, increase sensitivity with DL7.
THPI	C	
triadimefon	C	
triadimenol	C	
triazophos	C	
vinclozolin	C	

Table 303-a: Recovery of Chemicals Through Method 303 (E1-E5 + C1 or C2 + DG1-DG19)

(acetonitrile or water/acetonitrile extraction, partitioning into petroleum ether, Florisil column cleanup, GLC determination with various columns and detectors)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
1,1'-(2,2-dichloroethylidene)= bis(2-methoxybenzene)	R			
1,2,3,5-tetrachlorobenzene	P (75%) C	6	 1	
1,2,3-trichlorobenzene	C	6	1	Elutes in PE forerun.
1,2,4,5-tetrachloro-3-(methyl= thio)benzene	C	6	1	
1,2,4-triazole	NR	6-15-50	1-2-3	
1-hydroxychlordene	R	15		
10,10-dihydromirex	C	6		
10-monohydromirex	C	6		
2,3,5,6-tetrachloroaniline	R			
2,3,5,6-tetrachloroanisidine	C	6	2	
2,3,5,6-tetrachloroanisole	C	6	1	
2,3,5,6-tetrachloronitroanisole	C	6	1+2	
2,3,5-trimethacarb	S (18%) NR	50	 1-2-3	
2,4,5-trichloro-alpha-methyl= benzenemethanol	R	15		

[1] Codes: C: complete (>80%); P: partial (50-80%); S: small (<50%); V: variable (approximate percentage when known); R: recovered but no quantitative information available; NR: not recovered.

[2] Recovery results refer to complete method; blank entry in this column indicates Florisil elution was tested but not complete method. Separate results are listed for C1 and C2 only if recovery is affected by Florisil elution system.

[3] Eluants(s) in which chemical is eluted from Florisil, according to directions in 303 C1, *i.e.*, 6, 15, and 50% ethyl ether/ petroleum ether (EE/PE). Entries for chemicals not recovered indicate which eluants were used in tests.

[4] Eluants(s) in which chemical is eluted from Florisil, according to directions in 303 C2, *i.e.*, methylene chloride (CH_2Cl_2) eluants #1, 2, and 3. Entries for chemicals not recovered indicate which eluants were used in tests.

[5] "Florisil only" refers to tests in which elution patterns were tested by added reference standard solutions directly to Florisil column.

[6] Reference to petroleum ether (PE) forerun refers to Florisil elution performed as in 304 C3 or C4; not usually used in analysis of nonfatty foods.

Table 303-a: Recovery Through 303 (E1-E5 + C1 or C2 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
2,4-dichloro-6-nitrobenzenamine	R	15	2	Complete elution from Florisil only in 15% EE/PE or CH_2Cl_2 eluant #2.
2,6-dichlorobenzamide	NR	6-15-50	1-2-3	
2,8-dihydromirex	C	6		
2-chloroethyl caprate	C	15	2	
2-chloroethyl laurate	C	15	2	
2-chloroethyl linoleate	V (36-114%)	15	2	
2-chloroethyl myristate	V (48-112%)	15	2	
2-chloroethyl palmitate	V (38-107%)	15	2	
2-methoxy-3,5,6-trichloropyridine	P(60-78%) C	6+15	 1+2	
3,4,5-trimethacarb		50		Partial (20-35%) elution from Florisil only in 50% EE/PE.
	NR		1-2-3	
3,4-dichloroaniline	S (8%)	15		35% elution from Florisil only in 15% EE/PE.
3,4-dichlorophenylurea	NR	6-15-50		
3,5-dichloroaniline	S (12-48%)	6+15	1+2	Partial (73%) elution from Florisil only in 15% EE/PE.
3-(3,4-dichlorophenyl)-1-methoxyurea	NR	6-15-50		
3-desmethyl sulfentrazone	NR	6-15-50	1-2-3	
3-hydroxymethyl-2,5-dimethyl=phenyl methylcarbamate	NR	6-15-50	1-2-3	
3-ketocarbofuran	NR	6	1	60% recovered from Florisil only in 6% EE/PE or CH_2Cl_2 #1; also elutes with PE.
3-methyl-4-nitrophenol	NR	6-15-50	1-2-3	
3-tert-butyl-5-chloro-6-hydroxy=methyluracil	NR	6-15-50	1-2-3	

Table 303-a: Recovery Through 303 (E1-E5 + C1 or C2 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
4-(dichloroacetyl)-1-oxa-4-azapiro[4.5]decane	P (50-62%)	50	3	Complete elution from Florisil only in 50% and in CH_2Cl_2 eluant #3.
4-chloro-6-methoxyindole	R	15		
4-chlorobenzylmethyl sulfone	NR	6-15-50	1-2-3	
4-chlorobenzylmethyl sulfoxide	NR	6-15-50	1-2-3	
4-hydroxymethyl-3,5-dimethyl=phenyl methylcarbamate	NR	15-50	1-2-3	<20% elution from Florisil only in 15+50% EE/PE; <10% in CH_2Cl_2 eluants 1,2,3.
6-chloro-2,3-dihydro-3,3,7-methyl-5H-oxazolo(3,2-a)pyrimidin-5-one	NR	6-15-50	1-2-3	
6-chloro-2,3-dihydro-7-hydroxy methyl-3,3-methyl-5H-oxazolo=(3,2-a)pyrimidin-5-one	NR	6-15-50	1-2-3	
6-chloronicotinic acid	NR	6-15-50	1-2-3	
8-monohydromirex	C	6		
acetochlor	C (80-86%) P (55-68%)	50	3	Complete elution from Florisil only in CH_2Cl_2 eluant #3.
acifluorfen	NR	6-15-50	1-2-3	
acrinathrin	V(67-100%) V(66-96%)	15	2	
alachlor	C	50	3	16% elution from Florisil only in CH_2Cl_2 eluant #3.
aldrin	C	6	1	
allethrin	C	50	3	
allidochlor	NR	6-15	1-2-3	
alpha-cypermethrin	C		2	
anilazine	S (4-88%)	15+50	2+3	
aramite	P	15		Poor GLCsensitivity.

Table 303-a: Recovery Through 303 (E1-E5 + C1 or C2 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
Aroclor 1016	C	6	1	Elutes in PE forerun.
Aroclor 1221	C	6	1	Elutes in PE forerun.
Aroclor 1242	C	6	1	Elutes in PE forerun.
Aroclor 1248	C	6	1	Elutes in PE forerun.
Aroclor 1254	C	6	1	Elutes in PE forerun.
Aroclor 1260	C	6	1	Elutes in PE forerun.
Aroclor 1262	C	6	1	Elutes in PE forerun.
Aroclor 1268	C	6		Elutes in PE forerun.
Aroclor 4465	C	6	1	Elutes in PE forerun.
atrazine	S (25%) NR	50	 1-2-3	
azinphos-ethyl	P (50%)	50	3	49-79% elution from Florisil only in CH_2Cl_2 eluant #3.
azinphos-methyl	NR	6-15-50	1-2-3	
benfluralin	C	6	2	
benoxacor	P	15+50	2+3	60-75% elution from Florisil only in EE/PE; 40-80% in CH_2Cl_2 eluants.
bensulide	P (70%)	50	3	14% elution from Florisil only in CH_2Cl_2 eluant #3.
benzoylprop-ethyl	NR	6-15-50	1-2-3	
BHC, alpha-	C	6	1	Partially elutes in PE forerun.
BHC, beta-	C	6	1	
BHC, delta-	C	6+15	1	EE/PE elution variable.
bifenox	C	15+50	2+3	
bifenthrin	C	6+15	2	
binapacryl	P	15		

Table 303-a: Recovery Through 303 (E1-E5 + C1 or C2 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
bis(2-ethylhexyl) phthalate	C	15+50		Poor EC detector sensitivity.
bis(trichloromethyl)disulfide	R	6		
bromacil	NR	6-15-50	1-2-3	
bromophos	C	6		
bromophos-ethyl	C	6		
bromopropylate	C NR	15+50	 1-2-3	
bromoxynil butyrate	V (20-143%)	15+50	2	
bromoxynil octanoate	V (70-127%) S (15-42%)	15+50	 2	
Bulan	P (60%)	15	 2	 Complete elution from Florisil only in CH_2Cl_2 eluant #2.
butachlor	C	50		Also complete (83%) recovery from Florisil only in CH_2Cl_2 eluant 3.
butralin	C	6+15+50		Elution from Florisil variable.
butyl benzyl phthalate	C	15+50		
cadusafos	NR	6-15-50	1-2-3	
captafol	P (75-80%)	50	3	
captan	P (75%) P (50%)	50	 3	
captan epoxide	NR	6-15		
carbophenothion	C	6	 2	Elution from Florisil may be variable. <60% elution from Florisil only in CH_2Cl_2 eluant #2.
carbophenothion oxygen analog	NR	6-15-50	1-2-3	
carbophenothion sulfone	C (80%)	6	1	Elutes in PE forerun.
carboxin	NR	6-15-50		

Table 303-a: Recovery Through 303 (E1-E5 + C1 or C2 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
carboxin sulfoxide	NR	6-15-50	1-2-3	
CGA 118244	NR	6-15-50	1-2-3	
CGA 120844	NR	6-15-50	1-2-3	
CGA 14128		50	1-2-3	9-22% elution from Florisil only in EE/PE; no elution in CH_2Cl_2 eluants.
CGA 171683		15+50	3	Complete elution from Florisil only in 15+50% EE/PE, 70% in CH_2Cl_2 eluant #3.
CGA 205374	NR	6-15-50	1-2-3	
CGA 37734	NR	6-15-50	1-2-3	
CGA 91305	NR	6-15-50	1-2-3	
CGA 94689A	NR	6-15-50	1-2-3	
CGA 94689B	NR	6-15-50	1-2-3	
chlorbenside	S	6	1	Recovery 25-85% using EE/PE eluants; may be better with CH_2Cl_2.
chlorbromuron	V (45-67%)	50	3	Complete elution from Florisil only.
chlorbufam		15	2+3	Complete elution from Florisil only in 15% EE/PE, 77% in CH_2Cl_2 eluants 2+3.
chlordane	C	6	1	
chlordane, cis-	C	6	1	May elute in PE forerun.
chlordane, trans-	C	6	1	
chlordecone	S (45%) NR	15+50	 1-2-3	Elution from Florisil variable.
chlordene	C	6	1	Elutes in PE forerun.
chlordene epoxide	C	15		
chlorethoxyfos	C	6	1	

Table 303-a: Recovery Through 303 (E1-E5 + C1 or C2 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
chlorfenvinphos, beta-	S (0-49%)	50		
	NR		1-2-3	
chlorimuron ethyl ester	NR			Variable (75-92%) elution from Florisil only in 50% EE/PE.
chlornitrofen	C	6+15	2	Variable elution from Florisil in EE/PE.
chlorobenzilate	C	15+50		
	NR		3	Some variable elution from Florisil only in eluant #3.
chloroneb	C	6	2	82% elution from Florisil only in CH_2Cl_2 eluant #2.
chloropropylate	C	15+50		
			3	Some variable elution from Florisil only in CH_2Cl_2 eluant #3.
chlorothalonil	NR	6-15-50		
	C		2+3	
chlorothalonil trichloro impurity	NR	6-15-50		
	R		2+3	
chloroxuron	NR	6-15-50	1-2-3	
chlorpropham	C	15	2	
chlorpyrifos	C	6	2	
chlorpyrifos oxygen analog	NR	6-15-50		
chlorpyrifos-methyl	C	6	2	
chlorsulfuron	NR	6-15-50		
chlorthiophos	C	6		
			2	11% elution from Florisil only in CH_2Cl_2 eluant #2.
chlorthiophos oxygen analog	NR	6-15-50	1-2-3	
chlorthiophos sulfone		50		55% elution from Florisil only in 50% EE/PE.
	C		3	

Table 303-a: Recovery Through 303 (E1-E5 + C1 or C2 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
chlorthiophos sulfoxide	NR	6-15-50	1-2-3	
clofentezine	S (15-24%)	15	2	Complete elution from Florisil only; degrades on GLC in extract.
clomazone		50	3	88% elution from Florisil only in 50% EE/PE, 54-74% in CH_2Cl_2 eluant #3.
clopyralid methyl ester		50		17% elution from Florisil only in 50% EE/PE.
Compound K	C		1	
coumaphos	NR	6-15-50	3	Complete elution from Florisil only in CH_2Cl_2 eluant #3.
coumaphos oxygen analog	NR	6-15-50	1-2-3	
CP 51214	NR	6-15-50	1-2-3	
crotoxyphos	NR	6-15-50	1-2-3	
crufomate	NR	6-15-50		
cyanazine	NR	6-15-50		
cycloate	V (43-65%) C	15+50	3	
cyfluthrin	P (60%)	15		
cymoxanil	NR	6-15-50	1-2-3	Not eluted from Florisil.
cypermethrin	C	15	2	
cyproconazole	NR	6-15-50	1-2-3	
cyprodinil	NR	6-15-50	1-2-3	Not eluted from Florisil.
dazomet	NR	6-15-50	1-2-3	
DCPA	C	15	2	
DDE, o,p'-	C	6	1	Partially elutes in PE forerun.
DDE, p,p'-	C	6	1	Elutes in PE forerun.

Table 303-a: Recovery Through 303 (E1-E5 + C1 or C2 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
DDMS	R	6		
DDT, o,p'-	C	6	1	
DDT, p,p'-	C	6	1	
deltamethrin	S (32-65%)	15		Very poor EC detector sensitivity.
	C		2	
deltamethrin, trans-	P (50-67%) V (47-142%)	15	2	
demeton-O	NR	6-15		
demeton-S	NR	6-15-50		
des N-isopropyl isofenphos	S (30%)	50		
desdiethyl simazine	NR	6-15-50	1-2-3	
desethyl simazine		50		43% elution from Florisil only in 50% EE/PE.
	NR		1-2-3	
desisopropyl iprodione		50	1-2-3	17% eluted from Florisil only with 50% EE/PE; not eluted with CH_2Cl_2 eluants.
desmethyl norflurazon	NR	6-15-50	1-2-3	
di-allate	C	6		
di-n-octyl phthalate	C	15+50		Poor and variable EC detector sensitivity.
dialifor	C	15		
			2	Complete elution from Florisil only in CH_2Cl_2 eluant #2.
diazinon	C	15	3	
diazinon oxygen analog	NR	6-15-50	1-2-3	
dibutyl phthalate	C	15+50		
dichlobenil	P	15	2	
dichlofenthion	C	6	2	

Table 303-a: Recovery Through 303 (E1-E5 + C1 or C2 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
dichlofluanid	C V (51-91%)	15+50	2+3	Elution from Florisil variable in CH_2Cl_2 eluants.
dichlone	NR S (30%)	6-15-50	2+3	Elution from Florisil variable.
dichlorobenzene, p-	C	6	1	
dichlorobenzophenone, o,p'-	C	15	2	
dichlorobenzophenone, p,p'-	C	15	2	
dichlorvos	NR	6-15-50	1-2-3	
diclobutrazol	NR	6-15-50	1-2-3	
diclofop-methyl	C	15	2	
dicloran	S (35%)	15+50	2+3	
dicofol, o,p'-	V (50-100%)	6+15	2	Elution from Florisil may be variable.
dicofol, p,p'-	V (68-99%) V (78-90%)	6+15	1+2	Elution from Florisil variable.
dicrotophos	NR	6-15-50		
dieldrin	C	15	2	
diethatyl-ethyl	NR	6-15-50	1-2-3	
diethyl phthalate	P	15+50		Poor EC detector sensitivity.
diisobutyl phthalate	P (75%)	15+50		About 80% elution from Florisil only in 15+50% EE/PE.
diisohexyl phthalate	C	15+50		Poor EC detector sensitivity.
diisooctyl phthalate	C	15+50		Poor EC detector sensitivity.
Dilan	P (65%)	15		
dimethenamid	NR	6-15-50	1-2-3	
dimethipin	NR	6-15-50	1-2-3	
dimethoate	NR	6-15-50	1-2-3	

Table 303-a: Recovery Through 303 (E1-E5 + C1 or C2 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
dimethomorph (prop)	NR	6-15-50	1-2-3	Recovery tested using high temperature column.
dimethyl phthalate	P	6+15+50		Partial elution from Florisil only in all EE/PE; poor EC detector sensitivity.
dinocap	P	15		75% elution from Florisil only in 15% EE/PE.
			2	Complete elution from Florisil only in CH_2Cl_2 eluant #2.
dioxabenzofos	P (72%)	15		
dioxathion	NR	6-15-50		
			2	45% elution from Florisil only in CH_2Cl_2 eluant #2.
diphenamid	NR	6-15		
diphenylamine	S (<20%)	6+15		<10% elution from Florisil only in each 6 and 15% EE/PE.
disulfoton	P (50-74%)	6		25-40% elution from Florisil only in 6% EE/PE.
	NR		1-2-3	
disulfoton sulfone	NR	6-15-50		
diuron	NR	6-15-50	1-2-3	
endosulfan I	C	15	2	
endosulfan II	C	15+50	2	
endosulfan sulfate	C	50	2	
endrin	C	15		
	V		2	60-90% elution from Florisil only in CH_2Cl_2 eluant #2.
endrin alcohol	P (50%)	15+50		
			2+3	Partial (48%) elution from Florisil only in CH_2Cl_2 eluant #2, 28% in #3.
endrin aldehyde	P (50%)	15+50		
endrin ketone	C	50	2	

Table 303-a: Recovery Through 303 (E1-E5 + C1 or C2 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
EPN	C	15		
			2	71% elution from Florisil only in CH_2Cl_2 eluant #2.
EPTC	P (63%)	15		
esfenvalerate	C	15	2	
ethalfluralin	C	6	2	Elution from Florisil in CH_2Cl_2 eluants may be variable.
ethametsulfuron methyl ester	NR	6-15-50	1-2-3	
ethephon		6+15+50	1+2+3	5-25% eluted from Florisil only in each eluate.
ethiofencarb	NR	6-15-50		
ethion	C	6		
			2	60-90% elution from Florisil only in CH_2Cl_2 eluant #2.
ethoprop	P (55%)	50		
	NR		1-2-3	
ethoxyquin	NR	6-15-50		
ethylenethiourea	NR	6-15-50	1-2-3	
etridiazole	C	6		Other data show poor recovery through C1.
			2	Percent elution from Florisil only varies in different reports.
etrimfos	C	15	2+3	
famphur	NR	6-15-50		
fenac	NR	6-15-50		
fenamiphos	NR	6-15-50	1-2-3	
fenamiphos sulfone	NR	6-15-50	1-2-3	
fenamiphos sulfoxide	NR	6-15-50	1-2-3	
fenarimol	P (60%)	50		Quantitation may be influenced by presence of sample extract.
	S (40%)		3	

Table 303-a: Recovery Through 303 (E1-E5 + C1 or C2 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
fenarimol metabolite B	NR	6-15-50		
fenarimol metabolite C		6		17% elution from Florisil only in 6% EE/PE; no elution in 15 or 50% EE/PE.
fenbuconazole	NR	6-15-50	1-2-3	
fenitrothion	C	15	2	
fenoxaprop ethyl ester	V (58-125%)	50	3	Partial (70%) elution from Florisil only in either elution system.
fenpropathrin	V (43-71%)	15		Complete (111-116%) elution from Florisil only in 15% EE/PE.
	P (55-65%)		2	Partial (56-62%) elution from Florisil only in CH_2Cl_2 eluant #2.
fenpropimorph		50		Partial (49-63%) elution from Florisil only in 50% EE/PE.
			1-2-3	Not recovered from Florisil only in CH_2Cl_2 eluates.
fensulfothion	NR	6-15-50	1-2-3	
fensulfothion oxygen analog	NR	6-15-50		
fensulfothion sulfone	NR	6-15-50		
fenthion	S (45%)	6+15		
	NR		1-2-3	
fenthion oxygen analog	NR	6-15-50	1-2-3	
fenthion oxygen analog sulfoxide	NR	6-15-50	1-2-3	
fenthion sulfone	NR	6-15-50	1-2-3	
fenvalerate	C	15	2	
fipronil	S (21-41%)	50	3	
fluazifop butyl ester	C	15	3	Poor EC detector sensitivity with OV-225.
fluchloralin	C	6	2	

Table 303-a: Recovery Through 303 (E1-E5 + C1 or C2 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
flucythrinate	C	15		
			2+3	Elution from Florisil only 95% in CH_2Cl_2 eluant 2, 6% in eluant 3.
fluridone	NR	6-15-50		
fluvalinate	C	15		
			2	Complete elution in Florisil only in CH_2Cl_2 eluant 2.
folpet	C	15+50		
	C (80%)		2+3	Complete elution from Florisil only in CH_2Cl_2 eluants #2 & 3.
fonofos	C	6	2+3	
fonofos oxygen analog	NR	6-15-50	1-2-3	
formothion	NR	6-15-50	1-2-3	
fosthiazate	NR	6-15-50	1-2-3	
furilazole	S (28-50%)	50	3	Complete elution from Florisil only in 50% EE/PE, CH_2Cl_2 #3.
Gardona	NR	6-15-50	1-2-3	
GS-31144	NR	6-15-50	1-2-3	
heptachlor	C	6	1	
heptachlor epoxide	C	6	2	
hexachlorobenzene	C	6	1	Elutes in PE forerun.
hexachlorobutadiene	V (62-88%)	6		Elutes in PE forerun.
	P (78%)		1	
hexachlorophene	NR	6-15-50		
hexachlorophene dimethyl ether	NR	6-15		
hexazinone	NR	6-15-50	1-2-3	
hexythiazox	S (2-20%)	50		Florisil pattern and recoveries vary; may elute in 15%, may be complete.
	C		2+3	

Table 303-a: Recovery Through 303 (E1-E5 + C1 or C2 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
hydroxy chloroneb	NR	6-15		
imazalil	NR	6-15-50		
imidacloprid	NR	6-15-50	1-2-3	
IN-A3928	NR	6-15-50	1-2-3	
IN-B2838	NR	6-15-50	1-2-3	
IN-T3936	NR	6-15-50	1-2-3	
iprodione	S (5-56%) NR	50	 1-2-3	
iprodione metabolite isomer	S (21-100%)	50		
isazofos	C	50		Recovery test performed on corn grain and beef liver.
	P		2+3	
isofenphos	C	15+50		
isopropalin	C	6		
isoxaflutole (prop)	V (60-120%)	50		Complete elution from Florisil only in 50% EE/PE
	NR		3	32-56% elution from Florisil only in CH_2Cl_2 eluant 3.
Korax	NR	6-15		
KWG 1323	NR	6-15-50	1-2-3	
leptophos	C	6	2	
lindane	C	6	1	
linuron	V (42-64%) S (19-33%)	50	 3	
malathion	C	15+50	3	Elution from Florisil variable in EE/PE eluants.
malathion oxygen analog	NR	6-15-50	1-2-3	
MB45950	P (50-73%)	15+50	2+3	
MB46136	S (28-55%)	50	2+3	

Table 303-a: Recovery Through 303 (E1-E5 + C1 or C2 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
mecarbam		50		Partial (43%) elution from Florisil only in 50% EE/PE.
merphos	C	6+15+50	3	Elution from Florisil variable. Partial (48%) elution from Florisil only in CH_2Cl_2 eluant #3.
metalaxyl	NR	6-15-50	1-2-3	
methabenzthiazuron	NR	6-15-50	1-2-3	
methidathion	S (35%)	50	3	Complete elution from Florisil only in CH_2Cl_2 eluant #3.
methidathion oxygen analog	NR	6-15-50	1-2-3	
methidathion sulfone	NR	6-15-50	1-2-3	
methidathion sulfoxide	NR	6-15-50	1-2-3	
methiocarb sulfone	NR	6-15-50	1-2-3	
methomyl	NR	6-15-50	1-2-3	
methoxychlor olefin	C	6	2	
methoxychlor, o, p'-	C	6		
methoxychlor, p, p'-	C	6	2	
methyl 4-chloro-1H-indole-3-acetate		R	50	
	NR		1-2-3	Not eluted from Florisil only in CH_2Cl_2 eluants.
metobromuron	NR	6-15-50	1-2-3	
metolachlor	S (28-70%)	50		
	NR		1-2-3	
metoxuron	NR	6-15-50	1-2-3	
metribuzin	NR	50	1-2-3	Complete elution from Florisil only in 50% EE/PE; may be S recovery thru method.
metribuzin, deaminated diketo metabolite		NR	6-15-50	1-2-3
metribuzin, deaminated metabolite	NR	6-15-50	1-2-3	

Table 303-a: Recovery Through 303 (E1-E5 + C1 or C2 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
metribuzin, diketo metabolite	NR	6-15-50	1-2-3	
mevinphos, (E)-	NR	6-15-50		
mevinphos, (Z)-	NR	6-15-50		
mirex	C	6	1	Elutes in PE forerun.
monocrotophos	NR	6-15-50	1-2-3	
monuron	NR	6-15-50	1-2-3	
myclobutanil	NR	6-15-50	1-2-3	
myclobutanil alcohol metabolite	NR	6-15-50	1-2-3	
myclobutanil dihydroxy metabolite	NR	6-15-50	1-2-3	
N, N-diallyl dichloroacetamide	S (41-51%)	15+50	2+3	Complete elution from Florisil only in 15+50% EE/PE, CH_2Cl_2 eluants 2+3.
N-(3,4-dichlorophenyl)-N'-methylurea		NR	6-15-50	
naled	NR	6-15-50	1-2-3	
neburon	NR	6-15-50	1-2-3	
nitralin	P (60%)	50		50-80% elution from Florisil only in 50% EE/PE.
			3	75% elution from Florisil only in CH_2Cl_2 eluant #3.
nitrapyrin	C	6	2	Complete elution from Florisil only in 6% EE/PE or CH_2Cl_2 eluant #2.
nitrofen	C	15	2	
nitrofluorfen	C	15	2	
nonachlor, cis-	C	6	1	
nonachlor, trans-	C	6	1	Elutes in PE forerun.
norflurazon	NR	6-15-50		
NTN33823	NR	6-15-50	1-2-3	
NTN35884	NR	6-15-50	1-2-3	

Table 303-a: Recovery Through 303 (E1-E5 + C1 or C2 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
nuarimol		50		35% elution from Florisil only in 50% EE/PE.
	NR		1-2-3	
octachlor epoxide	C	6	1	
omethoate	NR	6-15-50	1-2-3	
oryzalin	NR	6-15-50		
ovex	C	15	2	
oxadiazon	C	15		
oxadixyl	NR	6-15-50	1-2-3	
oxamyl oxime metabolite	NR	6-15-50	1-2-3	
oxyfluorfen	C	15	2	Poor N/P detector sensitivity.
parathion	C	15	2	
parathion oxygen analog	NR	6-15-50	1-2-3	
parathion-methyl	C	15	2	
parathion-methyl oxygen analog	NR	6-15-50	1-2-3	
PB-9	NR	6-15-50	1-2-3	
pebulate	P (70%)	15		68% elution from Florisil only in 15% EE/PE; none eluted in 50%.
pendimethalin	C	15	2	
pentachloroaniline	C	6	1	
pentachlorobenzene	C	6	1	Elutes in PE forerun.
pentachlorobenzonitrile	C	15	2	
pentachlorophenyl methyl ether	C	6	1	
pentachlorophenyl methyl sulfide	C	6	1	

Table 303-a: Recovery Through 303 (E1-E5 + C1 or C2 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
permethrin, cis-	V (60-115%)	6+15		Complete elution from Florisil only in 6+15% EE/PE; high temp column recommended.
	C		2	High temperature column recommended.
permethrin, trans-	V (60-115%)	6+15		Complete elution from Florisil only in 6+15% EE/PE; high temp column recommended.
	C		2	High temperature column recommended.
Perthane	C	6	1	
Perthane olefin	C	6	1	
phenthoate	C	15+50		
phorate	V (40-75%)	6		Elution from Florisil quite variable.
	C		1	
phorate oxygen analog	NR	6-15-50	1-2-3	
phorate oxygen analog sulfone	NR	6-15-50	1-2-3	
phorate oxygen analog sulfoxide	NR	6-15-50	1-2-3	
phorate sulfone	NR	6-15-50		
	S (34-38%)		3	38% elution from Florisil only in CH_2Cl_2 eluant #3.
phorate sulfoxide	NR	6-15-50	1-2-3	
phosalone	C	50	2+3	
phosmet	NR	6-15-50		
			3	Partial (60%) elution from Florisil only in CH_2Cl_2 eluant #3.
phosmet oxygen analog	NR	6-15-50		
phosphamidon	NR	6-15-50	1-2-3	
photodieldrin	C	15+50	2	
pirimiphos-ethyl	C	15+50	3	
pirimiphos-methyl	C	15	3	

Table 303-a: Recovery Through 303 (E1-E5 + C1 or C2 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
PPG-2597	NR	6-15-50	1-2-3	
PPG-947	NR	6-15-50	1-2-3	
procymidone	C (84%)	15		
profenofos	P (65%)	50		Partial (56%) elution from Florisil only in 50% EE/PE.
			3	Partial (38%) elution from Florisil only in CH_2Cl_2 eluant #3.
profluralin	V (70-100%)	6		Complete elution from Florisil only in 6% EE/PE.
Prolan	S (40%)	15		
			2	Complete elution from Florisil only in CH_2Cl_2 eluant #2.
prometryn	P (50%)	50		Variable (22-67%) elution from Florisil only in 50% EE/PE.
	NR		1-2-3	
pronamide	P (63-71%)	15+50		39% elution from Florisil only in 6% EE/PE, 24% in 50% EE/PE.
propachlor	NR	6-15-50	1-2-3	Trace amount may be eluted in CH_2Cl_2 eluant #3.
propanil	NR	6-15		
			3	Partial (41%) elution from Florisil only in CH_2Cl_2 eluant #3.
propargite	C	15	2	
propazine	S (41%)	15+50		Complete but variable elution from Florisil only in 15%+50% EE/PE.
			3	Also elution of trace amount from Florisil only in CH_2Cl_2 eluant #2.
propetamphos	C (80%) P (50%)	15+50	2+3	

Table 303-a: Recovery Through 303 (E1-E5 + C1 or C2 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
propham	P (53%)	15		Addn 8-16% elution from Florisil only in 50% EE/PE; none in 50% thru method.
propiconazole	NR	6-15-50	1-2-3	
prosulfuron	NR	6-15-50	1-2-3	
prothiofos	C C	6	 2	 79% elution from Florisil only in CH_2Cl_2 eluant #2.
pyrazon	NR	6-15-50	1-2-3	
pyrazon metabolite B	NR	6-15-50	1-2-3	
pyrethrins	C	50		
pyrimethanil	S (11-51%)	50	3	Complete elution from Florisil only in 50% EE/PE or CH_2Cl_2 eluant #3.
quinalphos	C	15		
quintozene	C	6	1	
RH-6467	NR	6-15-50	1-2-3	
RH-9129	NR	6-15-50	1-2-3	
RH-9130	NR	6-15-50	1-2-3	
ronnel	C	6	2	
ronnel oxygen analog	NR	6-15-50		
RPA202248	NR	6-15-50	1-2-3	
S-bioallethrin	C	50		
schradan	NR	6-15-50		
sethoxydim	NR	6-15-50	3	CH_2Cl_2 elution from Florisil only tested with eluant #3 only, not #1 or #2.
sethoxydim sulfoxide	NR	6-15-50	3	CH_2Cl_2 elution from Florisil only tested with eluant #3 only, not #1 or #2.

Table 303-a: Recovery Through 303 (E1-E5 + C1 or C2 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
simazine	NR	50		Complete elution from Florisil only in 50% EE/PE.
	NR		1-2-3	
Strobane	C	6	1	
sulfallate	C	6+15	2	Elution with EE/PE may be variable.
sulfanilamide	NR	6-15-50	1-2-3	
sulfotep	C	6+15		Wide bore column recommended.
			2	50% elution from Florisil only in CH_2Cl_2 eluant #2.
Sulphenone		50	3	Complete elution from Florisil only in 20+25% EE/PE or in CH_2Cl_2 eluant #3.
TCMTB	P (50-67%)	15		P (61-62%) elution from Florisil only in 15% EE/PE; no elution in 50% EE/PE.
TDE, o,p'-	C	6	1	
TDE, p,p'-	C	6	1	
TDE, p,p'-, olefin	C	6	1	Partially elutes in PE forerun.
tebufenozide	NR	6-15-50	1-2-3	
tebupirimfos	V (50-115%)	6+15	2+3	Elution from Florisil only also variable.
tebupirimfos oxygen analog	NR	6-15-50	1-2-3	
tecnazene	C	6	1	
teflubenzuron	NR	6-15-50	1-2-3	
terbacil	NR	6-15		
			2+3	30% elution from Florisil only in CH_2Cl_2 eluant #2, 13% in eluant #3.
terbufos	P (62%)	6		
terbufos oxygen analog sulfone	NR	6-15-50	1-2-3	

Table 303-a: Recovery Through 303 (E1-E5 + C1 or C2 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
terbufos sulfone	NR	6-15-50		
	C (77-86%)		2+3	Elution from Florisil only in CH_2Cl_2 eluant #2 (46%) and #3 (37%).
terbuthylazine	P (57%)	15+50		
tetradifon	C	15	2	
tetraiodoethylene	P (65%)	6		
tetramethrin	NR	6-15-50	1-2-3	Trace amount may elute from Florisil in CH_2Cl_2 eluant #3.
tetrasul	C	6	1	
thiabendazole	NR	6-15-50		
thiobencarb		15	2+3	40% elution from Florisil only in 15% EE/PE; 42% inCH_2Cl_2 #2, 11% in CH_2Cl_2 #3.
thiometon	NR	6-15-50		
thionazin	P (59%)	15+50		Complete (80%) elution from Florisil only in 15% and/or 50% EE/PE.
THPI	NR	6-15-50		
toxaphene	C	6	1	
tralkoxydim		50		20% elution from Florisil only in 50% EE/PE.
	NR		1-2-3	
tralomethrin	V (50-100%)	15	2	
tri-allate	C	6	2	
triadimefon	S (27-40%)	50		35% elution from Florisil only in 50% EE/PE.
	NR		1-2-3	No elution from Florisil in CH_2Cl_2 eluants.
triadimenol	NR	6-15-50		
triazamate	NR	6-15-50	1-2-3	

Table 303-a: Recovery Through 303 (E1-E5 + C1 or C2 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
tribufos	C	15+50	3	Partial, variable elution from Florisil only in CH_2Cl_2 eluant #3.
tributyl phosphate	R	50		
trichlorfon	NR	6-15-50	1-2-3	
trichloronat	C	6		
tridiphane	C	6	1+2	20% elution from Florisil only in CH_2Cl_2 eluant #1, 80% in #2.
trifluralin	C	6	2	
triflusulfuron methyl ester	NR	6-15-50	1-2-3	
tris(chloropropyl) phosphate	NR	6-15-50	1-2-3	
Tycor	S (1-19%)	50	3	Complete elution from Florisil only in 50% EE/PE, 50-60% in CH_2Cl_2 #3.
vernolate	P (65%)	15		
vinclozolin	C	15	2	
vinclozolin metabolite B	P (55-66%) V (60-105%)	6+15	2	
vinclozolin metabolite E	S (9-39%)	15+50		
vinclozolin metabolite F	NR	6-15-50	1-2-3	
vinclozolin metabolite S	P (55-70%)	15	2	
WAK4103	NR	6-15-50	1-2-3	

Table 304-a: Recovery of Chemicals Through Method 304 (E1-E5 + C1-C4 + DG1-DG19)
(extraction of fat from fatty products, acetonitrile/petroleum ether partitioning, Florisil column cleanup, GLC determination with various columns and detectors)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
1,2,3-trichlorobenzene	P (60%)	6		Elutes in PE forerun.
			1	Complete elution from Florisil only in CH_2Cl_2 eluant #1; elutes in PE forerun.
1,2,4-triazole	NR	6-15-50	1-2-3	
2,3,5-trimethacarb		50		50% elution from Florisil only in 50% EE/PE eluant.
	NR		1-2-3	
2,4-dichloro-6-nitrobenzenamine		15	2	
2,6-dichlorobenzamide	NR	6-15-50	1-2-3	
2-chloroethyl caprate	C	15	2	
2-chloroethyl laurate	C	15	2	
2-chloroethyl linoleate	P (73-80%)	15	2	
2-chloroethyl myristate	V (42-80%)	15	2	
2-chloroethyl palmitate	P (50-59%)	15	2	
2-methoxy-3,5,6-trichloropyridine	C	6+15	1+2	
3,4,5-trimethacarb		50		Partial (20-35%) elution from Florisil only in 50% EE/PE.
	NR		1-2-3	
3,4-dichlorophenylurea	NR	6-15-50		
3,5-dichloroaniline	S (22-43%)	15	2	
3-(3,4-dichlorophenyl)-1-methoxyurea		NR	6-15-50	

[1] Codes: C: complete (>80%); P: partial (50-80%); S: small (<50%); V: variable (approximate percentage when known); R: recovered but no quantitative information available; NR: not recovered.

[2] Recovery results refer to complete method; blank entry in this column indicates Florisil elution was tested but not complete method. Separate results are listed for C1 and C2 only if recovery is affected by Florisil elution system used.

[3] Eluants(s) in which chemical is eluted from Florisil, according to directions in 304 C1, *i.e.*, 6, 15, and 50% ethyl ether/petroleum ether (EE/PE). Entries for chemicals not recovered indicate which eluants were used in tests.

[4] Eluants(s) in which chemical is eluted from Florisil, according to directions in 304 C2, *i.e.*, methylene chloride (CH_2Cl_2) eluants #1, 2, and 3. Entries for chemicals not recovered indicate which eluants were used in tests.

[5] "Florisil only" refers to tests in which elution patterns were tested by added reference standard solutions directly to Florisil column.

[6] Reference to petroleum ether (PE) forerun refers to Florisil elution performed as in 304 C3 or C4.

Table 304-a: Recovery Through 304 (E1-E5 + C1-C4 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
3-desmethyl sulfentrazone	NR	6-15-50	1-2-3	
3-hydroxymethyl-2,5-dimethyl= phenyl methylcarbamate	NR	6-15-50	1-2-3	
3-ketocarbofuran	NR	6	1	60% recovered from Florisil only in 6% EE/PE or CH_2Cl_2 #1; also elutes with PE.
3-methyl-4-nitrophenol	NR	6-15-50	1-2-3	
3-tert-butyl-5-chloro-6-hydroxy= methyluracil	NR	6-15-50	1-2-3	
4-chlorobenzylmethyl sulfone	NR	6-15-50	1-2-3	
4-chlorobenzylmethyl sulfoxide	NR	6-15-50	1-2-3	
4-hydroxymethyl-3,5-dimethyl= phenyl methylcarbamate	NR	15-50	1-2-3	<20% elution from Florisil only in 15+50% EE/PE; <10% in CH_2Cl_2 eluants 1,2,3.
6-chloro-2,3-dihydro-3,3,7-methyl-5H-oxazolo(3,2-a)pyrimidin-5-one	NR	6-15-50	1-2-3	
6-chloro-2,3-dihydro-7-hydroxy= methyl-3,3-methyl-5H-oxazolo= (3,2-a)pyrimidin-5-one	NR	6-15-50	1-2-3	
6-chloronicotinic acid	NR	6-15-50	1-2-3	
acetochlor	P (52-70%)	15+50	2+3	Complete elution from Florisil only in 50% EE/PE or CH_2Cl_2 eluant #3.
acifluorfen	NR	6-15-50	1-2-3	
acrinathrin	NR			Complete elution from Florisil only in 15% EE/PE.
	V(27-80%)		2	
alachlor	C	50		
	S (23%)		3	16% elution from Florisil only in CH_2Cl_2 eluant #3.
aldrin	C	6	1	
allethrin	C	50		Elution from Florisil in EE/PE may be variable.
	P (66-75%)		3	

Table 304-a: Recovery Through 304 (E1-E5 + C1-C4 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
anilazine	P	15+50	2+3	
aramite	NR			
Aroclor 1016	C	6	1	Elutes in PE forerun.
Aroclor 1221	C	6	1	Elutes in PE forerun.
Aroclor 1242	C	6	1	Elutes in PE forerun.
Aroclor 1248	C	6	1	Elutes in PE forerun.
Aroclor 1254	C	6	1	Elutes in PE forerun.
Aroclor 1260	C	6	1	Elutes in PE forerun.
Aroclor 1262	C	6	1	Elutes in PE forerun.
Aroclor 4465	C	6	1	Elutes in PE forerun.
atrazine	NR		1-2-3	
azinphos-ethyl	S (14%)	50	3	49-79% elution from Florisil only in CH_2Cl_2 eluant #3.
azinphos-methyl	NR	6-15	1-2-3	
benfluralin	C	6	2	
benoxacor	C	15+50	2+3	
bensulide	C	50	3	14% elution from Florisil only in CH_2Cl_2 eluant #3.
benzoylprop-ethyl	NR	6-15-50	1-2-3	
BHC, alpha-	C	6	1	Partially elutes in PE forerun.
BHC, beta-	C	6	1	
BHC, delta-	C	6+15	1	EE/PE elution variable.
bifenox	P (51-78%)	15+50	2+3	51-58% elution from Florisil with EE/PE; 56-78% with CH_2Cl_2.
bifenthrin		6+15	2	

Table 304-a: Recovery Through 304 (E1-E5 + C1-C4 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
binapacryl	P (65%)	15		
bis(2-ethylhexyl) phthalate	C	15+50		Poor EC detector sensitivity.
bromacil	NR	6-15-50	1-2-3	
bromophos	C	6		
bromophos-ethyl	P (59-78%)	6		
bromopropylate	C NR	15+50	 1-2-3	
Bulan	P (75%)	15	 2	 Complete elution from Florisil only in CH_2Cl_2 eluant #2.
butyl benzyl phthalate	P (70%)	15+50		Complete elution from Florisil only in15+50% EE/PE.
cadusafos	NR	6-15-50	1-2-3	
captan	C (80%)	50		
carbophenothion	P (60%)	6	 2	Elution from Florisil may be variable. <60% elution from Florisil only in CH_2Cl_2 eluant #2.
carbophenothion oxygen analog	NR	6-15-50	1-2-3	
carbophenothion sulfone	P (66%)	6	1	Elutes in PE forerun.
carboxin	NR	6-15-50		
carboxin sulfoxide	NR	6-15-50	1-2-3	
CGA 118244	NR	6-15-50	1-2-3	
CGA 120844	NR	6-15-50	1-2-3	
CGA 14128		50	1-2-3	9-22% elution from Florisil only in EE/PE; not recovered in CH_2Cl_2 eluants.
CGA 171683		15+50	3	Complete elution from Florisil only in 15+50% EE/PE, 70% in CH_2Cl_2 eluant #3.
CGA 205374	NR	6-15-50	1-2-3	

Table 304-a: Recovery Through 304 (E1-E5 + C1-C4 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
CGA 37734	NR	6-15-50	1-2-3	
CGA 91305	NR	6-15-50	1-2-3	
CGA 94689A	NR	6-15-50	1-2-3	
CGA 94689B	NR	6-15-50	1-2-3	
chlorbenside	P	6	1	Recovery 50% using EE/PE eluants; may be better with CH_2Cl_2.
chlorbromuron	V (44-100%)	50	3	Complete elution from Florisil only.
chlorbufam		15	2+3	Complete elution from Florisil only in 15% EE/PE, 77% in CH_2Cl_2 eluants 2+3.
chlordane	C	6	1	
chlordane, cis-	C	6	1	May elute in PE forerun.
chlordane, trans-	C	6	1	
chlordecone	P NR	15+50	 1-2-3	Elution from Florisil variable.
chlordene	C	6	1	Elutes in PE forerun.
chlorfenapyr (prop)	S (30-50%)	50	2	Complete elution from Florisil only in 50% EE/PE and CH_2Cl_2 eluant 2.
chlorfenvinphos, alpha-	NR	6-15-50		
chlornitrofen	C	6+15	2	Variable elution from Florisil in EE/PE.
chlorobenzilate	P (75%) NR	15+50	 3	Some variable elution from Florisil in CH_2Cl_2 eluant #3.
chloroneb			2	82% elution from Florisil only in CH_2Cl_2 eluant #2.
chloropropylate	C	15+50	 3	Some variable elution from Florisil in CH_2Cl_2 eluant #3.

Table 304-a: Recovery Through 304 (E1-E5 + C1-C4 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
chlorothalonil	NR C (80-90%)	6-15-50	 2+3	
chlorothalonil trichloro impurity	NR	6-15-50		
chloroxuron	NR	6-15-50	1-2-3	
chlorpropham	C	15	2	
chlorpyrifos	P (74-83%)	6	2	
chlorsulfuron	NR	6-15-50		
chlorthiophos	C	6	2	11% elution from Florisil only in CH_2Cl_2 eluant #2.
chlorthiophos oxygen analog	NR	6-15-50	1-2-3	
chlorthiophos sulfone		50		55% elution from Florisil only in 50% EE/PE.
chlorthiophos sulfoxide	NR	6-15-50	1-2-3	
clomazone		50	3	88% elution from Florisil only in 50% EE/PE, 54-74% in CH_2Cl_2 eluant #3.
clopyralid methyl ester		50		17% elution from Florisil only in 50% EE/PE.
coumaphos	NR C (76-93%)	6-15-50	 3	High temperature or short column GLC needed.
coumaphos oxygen analog	NR	6-15-50	1-2-3	
CP 51214	NR	6-15-50	1-2-3	
crotoxyphos	NR	6-15-50	1-2-3	
crufomate	NR	6-15-50		
cycloate	S (39-61%) S (24-37%)	15+50	 3	
cymoxanil	NR	6-15-50	1-2-3	Not eluted from Florisil.
cypermethrin	C (81%)	15	2	

Table 304-a: Recovery Through 304 (E1-E5 + C1-C4 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
cyproconazole	NR	6-15-50	1-2-3	
cyprodinil	NR	6-15-50	1-2-3	Not eluted from Florisil.
DCPA	C	15	2	
DDE, o,p'-	C	6	1	Partially elutes in PE forerun.
DDE, p,p'-	C	6	1	Elutes in PE forerun.
DDT, o,p'-	C	6	1	
DDT, p,p'-	C	6	1	
deltamethrin	P (77-80%)	15	2	Very poor EC detector sensitivity.
deltamethrin, trans-	NR			Partial (33%) elution from Florisil only in 15% EE/PE, complete in CH_2Cl_2 eluant #2.
desdiethyl simazine	NR	6-15-50	1-2-3	
desethyl simazine		50		43% elution from Florisil only in 50% EE/PE.
	NR		1-2-3	
desmethyl norflurazon	NR	6-15-50	1-2-3	
di-n-octyl phthalate	C	15+50		Poor and variable EC detector sensitivity.
dialifor	P (50%)	15		
			2	Complete elution from Florisil only in CH_2Cl_2 eluant #2.
diazinon	C	15	3	
diazinon oxygen analog	NR	6-15-50	1-2-3	
dibutyl phthalate	C	15+50		
dichlobenil	C (80%)	15	2	
dichlofenthion	V (69-89%)	6		
			2	
dichlone	NR	6-15-50		
	S (25%)		2+3	Elution from Florisil variable.

Table 304-a: Recovery Through 304 (E1-E5 + C1-C4 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
dichlorobenzene, p-	C	6	1	
dichlorobenzophenone, o,p'-	C	15	2	
dichlorobenzophenone, p,p'-	C	15	2	
dichlorvos	NR	6-15-50	1-2-3	
diclobutrazol	NR	6-15-50	1-2-3	
diclofop-methyl	C	15	2	
dicloran	P (50%)	15+50	2+3	
dicofol, o,p'-	S (25-50%)	6+15	2	Elution from Florisil may be variable.
dicofol, p,p'-	P (61-85%) S (36-58%)	6+15	 1+2	Elution from Florisil variable.
dieldrin	C	15	2	
diethatyl-ethyl	NR	6-15-50	1-2-3	
diethyl phthalate	P	15+50		Poor EC detector sensitivity.
diisobutyl phthalate		15+50		About 80% elution from Florisil only in 15+50% EE/PE.
diisohexyl phthalate		15+50		Complete elution from Florisil only in 15+50% EE/PE; poor EC sensitivity.
diisooctyl phthalate	C	15+50		Poor EC detector sensitivity.
Dilan	P (65%)	15		
dimethenamid	NR	6-15-50	1-2-3	
dimethipin	NR	6-15-50	1-2-3	
dimethoate	NR	6-15-50	1-2-3	
dimethomorph (prop)	NR	6-15-50	1-2-3	
dimethyl phthalate		6+15+50		Partial elution from Florisil only in all EE/PE; poor EC detector sensitivity.

Table 304-a: Recovery Through 304 (E1-E5 + C1-C4 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
dinitramine	P (78-80%)	15		Some elution from Florisil in 6% EE/PE.
dinocap	P (60%)	15		75% elution from Florisil only in 15% EE/PE.
			2	Elution from Florisil only, complete in CH_2Cl_2 eluant #2.
dioxathion			2	45% elution from Florisil only in CH_2Cl_2 eluant #2.
disulfoton		6		25-40% elution from Florisil only in 6% EE/PE.
	NR		1-2-3	
diuron	NR	6-15-50	1-2-3	
endosulfan I	C	15	2	
endosulfan II	C	15+50	2	
endosulfan sulfate	C	50	2	
endrin	C	15		
	V		2	60-90% elution from Florisil only in CH_2Cl_2 eluant #2.
endrin alcohol	C	15+50		
			2+3	Partial (48%) elution from Florisil only in CH_2Cl_2 eluant #2, 28% in #3.
endrin aldehyde	C	15+50		
endrin ketone	C	50	2	
EPN	C	15		
			2	71% elution from Florisil only in CH_2Cl_2 eluant #2.
esfenvalerate	C	15	2	
ethalfluralin	C	6	2	Elution from Florisil in CH_2Cl_2 eluants may be variable.
ethametsulfuron methyl ester	NR	6-15-50	1-2-3	
ethephon		6+15+50	1+2+3	5-25% eluted from Florisil only in each eluant.

Table 304-a: Recovery Through 304 (E1-E5 + C1-C4 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
ethiofencarb	NR	6-15-50		
ethion	C	6		
			2	60-90% elution from Florisil only in CH_2Cl_2 eluant #2.
ethoprop	S (45%)	50		
	NR		1-2-3	
ethoxyquin	NR	6-15-50		
ethylenethiourea	NR	6-15-50	1-2-3	
etridiazole	P (68-73%)	6		Other data shows poor recovery through C1.
			2	Percent elution from Florisil only varies in different reports.
etrimfos	C	15	2+3	
fenac	NR	6-15-50		
fenamiphos	NR	6-15-50	1-2-3	
fenamiphos sulfone	NR	6-15-50	1-2-3	
fenamiphos sulfoxide	NR	6-15-50	1-2-3	
fenarimol	C	50		Quantitation may be influenced by presence of sample extract.
	V (72-110%)		3	
fenarimol metabolite B	NR	6-15-50		
fenarimol metabolite C		6		17% elution from Florisil only in 6% EE/PE; no elution in 15 or 50% EE/PE.
fenbuconazole	NR	6-15-50	1-2-3	
fenitrothion	C	15	2	
fenoxaprop ethyl ester	V (65-110%)	50	3	Partial (70%) elution from Florisil only in either elution system.

Table 304-a: Recovery Through 304 (E1-E5 + C1-C4 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
fenpropathrin	V (59-114%)	15		Elution from Florisil only, complete (111-116%) in 15% EE/PE.
	V (58-91%)		2	Elution from Florisil only, partial (56-62%) in CH_2Cl_2 eluant #2.
fenpropimorph		50		Partial (49-63%) elution from Florisil only in 50% EE/PE.
			1-2-3	Not recovered from Florisil only in CH_2Cl_2 eluates.
fensulfothion	NR	6-15-50	1-2-3	
fenthion	NR	6-15	1-2-3	
fenthion oxygen analog	NR	6-15-50	1-2-3	
fenthion oxygen analog sulfoxide	NR	6-15-50	1-2-3	
fenthion sulfone	NR	6-15-50	1-2-3	
fenvalerate		15		Complete elution from Florisil only in 15% EE/PE.
	C (81%)		2	
fipronil	V (55-97%)	50	3	
fluazifop butyl ester	V (50-110%)	15	3	Poor EC detector sensitivity with OV-225.
fluridone	NR	6-15-50		
folpet	P (50%)	15+50		
			2+3	Complete elution from Florisil only in CH_2Cl_2 eluants #2 & 3.
fonofos	C	6	2+3	
fonofos oxygen analog	NR	6-15-50	1-2-3	
formothion	NR	6-15-50	1-2-3	
fosthiazate	NR	6-15-50	1-2-3	
Gardona	NR	6-15-50	1-2-3	
GS-31144	NR	6-15-50	1-2-3	
heptachlor	C	6	1	

Table 304-a: Recovery Through 304 (E1-E5 + C1-C4 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
heptachlor epoxide	C	6	2	
hexachlorobenzene	P (60%)	6		Loss in partitioning from PE to acetonitrile/water; elutes in PE forerun.
			1	Complete elution from Florisil only in CH_2Cl_2 eluant #1; elutes in PE forerun.
hexachlorobutadiene	P (63%)		1	Elutes in PE forerun.
hexachlorophene	NR	6-15-50		
hexachlorophene dimethyl ether	NR	6-15		
hexazinone	NR	6-15-50	1-2-3	
hexythiazox	NR	6-15-50	1-2-3	Complete elution from Florisil only in 15+50% EE/PE, CH_2Cl_2 eluants 2+3.
imazalil	NR	6-15-50		
imidacloprid	NR	6-15-50	1-2-3	
IN-A3928	NR	6-15-50	1-2-3	
IN-B2838	NR	6-15-50	1-2-3	
IN-T3936	NR	6-15-50	1-2-3	
iprodione		50		Partial (4-19%) elution from Florisil only in 50% EE/PE.
	NR		1-2-3	
isoxaflutole (prop)	S (37-126%)	50		Complete elution from Florisil only in 50% EE/PE.
	NR			32-56% elution from Florisil only in CH_2Cl_2 eluant 3.
KWG 1323	NR	6-15-50	1-2-3	
lactofen	C	50	2+3	
leptophos	C	6	2	
lindane	C	6	1	
linuron	V (42-62%)	50		
			3	

Table 304-a: Recovery Through 304 (E1-E5 + C1-C4 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
malathion	C	15+50	3	Variable elution from Florisil in EE/PE eluants.
malathion oxygen analog	NR	6-15-50	1-2-3	
MB45950	V (60-190%)	15+50	2+3	
MB46136	V (54-140%)	50	2+3	
mecarbam		50		Partial (43%) elution from Florisil only in 50% EE/PE.
merphos	C	6+15+50		Variable elution from Florisil in EE/PE eluants.
			3	Partial (48%) elution from Florisil only in CH_2Cl_2 eluant #3.
metalaxyl	NR	6-15-50	1-2-3	
methabenzthiazuron	NR	6-15-50	1-2-3	
methidathion	P (50%) C (80%)	50	 3	
methidathion oxygen analog	NR	6-15-50	1-2-3	
methidathion sulfone	NR	6-15-50	1-2-3	
methidathion sulfoxide	NR	6-15-50	1-2-3	
methiocarb sulfone	NR	6-15-50	1-2-3	
methomyl	NR	6-15-50	1-2-3	
methoxychlor olefin	C	6	2	
methoxychlor, p, p'-	C	6	2	
methyl 4-chloro-1H-indole-3-acetate	NR		1-2-3	Not eluted from Florisil only in CH_2Cl_2 eluants.
metobromuron	NR	6-15-50	1-2-3	
metolachlor	NR		1-2-3	
metoxuron	NR	6-15-50	1-2-3	
metribuzin	NR	50	1-2-3	Complete elution from Florisil only in 50% EE/PE; may be S recovery thru method.

Table 304-a: Recovery Through 304 (E1-E5 + C1-C4 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
metribuzin, deaminated diketo metabolite	NR	6-15-50	1-2-3	
metribuzin, deaminated metabolite	NR	6-15-50	1-2-3	
metribuzin, diketo metabolite	NR	6-15-50	1-2-3	
mevinphos, (E)-	NR	6-15-50		
mirex	P (75%)	6	1	Loss in partitioning from PE to acetonitrile/water; elutes in PE forerun.
monocrotophos	NR	6-15-50	1-2-3	
monuron	NR	6-15-50	1-2-3	
myclobutanil	NR	6-15-50	1-2-3	
myclobutanil alcohol metabolite	NR	6-15-50	1-2-3	
myclobutanil dihydroxy metabolite	NR	6-15-50	1-2-3	
N, N-diallyl dichloroacetamide	S (32-47%)	15+50	2+3	Complete elution from Florisil only in 15+50% EE/PE, CH_2Cl_2 eluants 2+3.
N-(3,4-dichlorophenyl)-N'-methylurea	NR	6-15-50		
naled	NR	6-15-50	1-2-3	
neburon	NR	6-15-50	1-2-3	
nitralin	P (70%)	50	3	50-80% elution from Florisil only in 50% EE/PE. 75% elution from Florisil only in CH_2Cl_2 eluant #3.
nitrapyrin	V (32-111%)	6	2	Complete elution from Florisil only in 6% EE/PE or CH_2Cl_2 eluant #2.
nitrofen	C	15	2	
nitrofluorfen	C	15	2	
nonachlor, cis-	C	6	1	

Table 304-a: Recovery Through 304 (E1-E5 + C1-C4 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
nonachlor, trans-	C	6	1	Elutes in PE forerun.
norflurazon	NR	6-15-50		
NTN33823	NR	6-15-50	1-2-3	
NTN35884	NR	6-15-50	1-2-3	
nuarimol	C	50		35% elution from Florisil only in 50% EE/PE.
	NR		1-2-3	
octachlor epoxide	C	6	1	
omethoate	NR	6-15-50	1-2-3	
oryzalin	NR	6-15-50		
ovex	C	15	2	
oxadiazon	P (75%)	15		
oxadixyl	NR	6-15-50	1-2-3	
oxamyl oxime metabolite	NR	6-15-50	1-2-3	
oxyfluorfen	C	15	2	
parathion	C	15	2	
parathion oxygen analog	NR	6-15-50	1-2-3	
parathion-methyl	C	15	2	
parathion-methyl oxygen analog	NR	6-15-50	1-2-3	
PB-9	NR	6-15-50	1-2-3	
pendimethalin	P (33-56%)	15		
	P (66-82%)		2	
pentachloroaniline	C	6	1	
pentachlorobenzene	C	6	1	Elutes in PE forerun.
pentachlorobenzonitrile	P (60%)	15		
			2	Complete elution from Florisil only in CH_2Cl_2 eluant #2.
pentachlorophenyl methyl ether	C	6	1	

Table 304-a: Recovery Through 304 (E1-E5 + C1-C4 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
pentachlorophenyl methyl sulfide	C	6	1	
permethrin, cis-		6+15		Complete elution from Florisil only in 6+15% EE/PE; high temp column recommended.
	C (82%)		2	High temperature column recommended.
permethrin, trans-		6+15		Complete elution from Florisil only in 6+15% EE/PE; high temp col recommended.
	C (82%)		2	High temperature column recommended.
Perthane	C	6	1	
Perthane olefin	C	6	1	
phorate	V (80%)	6		Elution from Florisil quite variable, may be 0%.
	C		1	
phorate oxygen analog	NR	6-15-50	1-2-3	
phorate oxygen analog sulfone	NR	6-15-50	1-2-3	
phorate oxygen analog sulfoxide	NR	6-15-50	1-2-3	
phorate sulfone	NR	6-15-50		
	S (12-20%)		3	38% elution from Florisil only in eluant 3.
phorate sulfoxide	NR	6-15-50	1-2-3	
phosalone	C	50	2+3	
phosmet			3	Partial (60%) elution from Florisil only in CH_2Cl_2 eluant #3.
phosmet oxygen analog	NR	6-15-50		
phosphamidon	NR	6-15-50	1-2-3	
photodieldrin	C	15+50	2	
pirimiphos-ethyl	C	15+50	3	
pirimiphos-methyl	C	15	3	

Table 304-a: Recovery Through 304 (E1-E5 + C1-C4 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
PPG-1576	P	50	2+3	72-85% elution from Florisil only in EE/PE; 54-75% in CH_2Cl_2 eluants.
PPG-2597	NR	6-15-50	1-2-3	
PPG-947	NR	6-15-50	1-2-3	
procymidone	P (76%)	15		
profenofos	P (50%)	50		Partial (56%) elution from Florisil only in 50% EE/PE.
			3	Partial (38%) elution from Florisil only in CH_2Cl_2 eluant #3.
Prolan	S (25%)	15		
			2	Complete elution from Florisil only in CH_2Cl_2 eluant #2.
prometryn	P (70%)	50		Variable (22-67%) elution from Florisil only in 50% EE/PE.
	NR		1-2-3	
propachlor	NR	6-15-50	1-2-3	Trace amount may be eluted in CH_2Cl_2 eluant #3.
propanil	NR	6-15		
			3	Partial (41%) elution from Florisil only in CH_2Cl_2 eluant #3.
propargite		15	2	Complete elution from Florisil only in 15% EE/PE or CH_2Cl_2 eluant #2.
propazine	NR	15+50		Complete but variable elution from Florisil only in 15%+50% EE/PE.
			3	Also elution of trace amount from Florisil only in CH_2Cl_2 eluant #2.
propham	P (80%)	15		Addition 8-16% elution from Florisil only in 50% EE/PE; none in 50% thru method.
propiconazole	NR	6-15-50	1-2-3	

Table 304-a: Recovery Through 304 (E1-E5 + C1-C4 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
prosulfuron	NR	6-15-50	1-2-3	
prothiofos	C	6		
			2	79% elution from Florisil only in CH_2Cl_2 eluant #2.
pyrazon	NR	6-15-50	1-2-3	
pyrazon metabolite B	NR	6-15-50	1-2-3	
pyrethrins	C	50		
pyrimethanil	S (0-40%)	50	3	Complete elution from Florisil only in 50% EE/PE or CH_2Cl_2 eluant 3.
	P (75-82%	50	3	
quintozene	C	6	1	
RH-6467	NR	6-15-50	1-2-3	
RH-9129	NR	6-15-50	1-2-3	
RH-9130	NR	6-15-50	1-2-3	
ronnel	C	6	2	
RPA202248	NR	6-15-50	1-2-3	
sethoxydim	NR	6-15-50	3	CH_2Cl_2 elution from Florisil tested with eluant #3 only, not #1 or #2.
sethoxydim sulfoxide	NR	6-15-50	3	CH_2Cl_2 elution from Florisil tested with eluant #3 only, not #1 or #2.
simazine		50		Complete elution from Florisil only in 50%EE/PE.
	NR		1-2-3	
Strobane	C	6	1	
sulfallate	C	6+15	2	Elution with EE/PE may be variable.
sulfanilamide	NR	6-15-50	1-2-3	

Table 304-a: Recovery Through 304 (E1-E5 + C1-C4 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
sulfotep	P (65-70%)	6+15	2	65% elution from Florisil with EE/PE; 70% with CH_2Cl_2; need wide bore column.
Sulphenone		50	3	Complete elution from Florisil only in 20+25% EE/PE or in CH_2Cl_2 eluant #3.
TCMTB	P (61-62%)	15		P (61-62%) elution from Florisil only in 15% EE/PE; no elution in 50% EE/PE.
TDE, o,p'-	C	6	1	
TDE, p,p'-	C	6	1	
TDE, p,p'-, olefin	C	6	1	Partially elutes in PE forerun.
tebufenozide	NR	6-15-50	1-2-3	
tebupirimfos	V (57-171%)	6+15	2+3	Elution from Florisil only also variable.
tebupirimfos oxygen analog	NR	6-15-50	1-2-3	
tecnazene	C	6	1	
teflubenzuron	NR	6-15-50	1-2-3	
terbacil	NR	6-15	2+3	30% elution from Florisil only in CH_2Cl_2 eluant #2, 13% in eluant #3.
terbufos	S (16%)	6		
terbufos oxygen analog	NR	6-15-50	1-2-3	
terbufos oxygen analog sulfone	NR	6-15-50	1-2-3	No elution from Florisil in either elution system.
terbufos sulfone	NR C	6-15-50	2+3	Elution from Florisil only in CH_2Cl_2 eluant #2 (46%) and #3 (37%).
tetradifon	C	15	2	
tetraiodoethylene	P (65%)	6		

Table 304-a: Recovery Through 304 (E1-E5 + C1-C4 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
tetramethrin	NR	6-15-50	1-2-3	May be elution of trace amount from Florisil in CH_2Cl_2 eluant #3.
tetrasul	C	6	1	
thiobencarb	V (<50-86%)	15		40% elution from Florisil only in 15% EE/PE.
			2+3	42% elution from Florisil only in CH_2Cl_2 #2, 11% in CH_2Cl_2 #3.
thiometon	NR	6-15-50		
thionazin	NR	15+50		Complete (80%) elution from Florisil only in 15% and/or 50% EE/PE.
THPI	NR	6-15-50		
toxaphene	C	6	1	
tralkoxydim		50		20% elution from Florisil only in 50% EE/PE.
	NR		1-2-3	
tralomethrin	S (0-50%)	15	2	
tri-allate	C	6	2	
triadimefon	S (13-62%)	50		35% elution from Florisil only in 50% EE/PE.
	NR		1-2-3	No elution from Florisil in CH_2Cl_2 eluants.
triadimenol	NR	6-15-50		
triazamate	NR	6-15-50	1-2-3	
tribufos	P (60%)	15+50		
			3	Partial, variable elution from Florisil only in eluant #3.
trichlorfon	NR	6-15-50	1-2-3	
trichloronat		6		Complete elution from Florisil only in 6% EE/PE.
tridiphane		6	1+2	20% elution from Florisil only in CH_2Cl_2 eluant #1, 80% in #2.

Table 304-a: Recovery Through 304 (E1-E5 + C1-C4 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
trifluralin	C	6	2	
triflusulfuron methyl ester	NR	6-15-50	1-2-3	
tris(chloropropyl) phosphate	NR	6-15-50	1-2-3	
Tycor	S (12-162%)	50	3	Complete elution from Florisil only in 50% EE/PE, 50-60% in CH_2Cl_2 #3.
vinclozolin	C	15	2	
vinclozolin metabolite B	C	6+15	2	Recovery in 6% EE/PE; other studies showed split into 15%.
vinclozolin metabolite E	NR	6-15-50		
vinclozolin metabolite F	NR	6-15-50	1-2-3	
vinclozolin metabolite S	V (47-81%) C	15	 2	
WAK4103	NR	6-15-50	1-2-3	

Table 304-b: Recovery of Chemicals Through Method 304 (E1-E5 + C6 + DG1-DG19)
(extraction of fat from fatty products, cleanup with gel permeation and Florisil column chromatography, GLC determination with various columns and detectors)

Chemical	Recovery[1]	Eluant[2]	Notes
1,2,3,5-tetrachlorobenzene	V (41-138%)	1	mean recovery 85.9%, n=15
1,2,4,5-tetrachloro-3-(methylthio)= benzene	C	1	mean recovery 86%, n=11
2,3,5,6-tetrachloroanisidine	V (47-108%)	2	mean recovery 82.8%, n=10
2,3,5,6-tetrachloroanisole	C	1	mean recovery 89.6%, n=10
2,3,5,6-tetrachloronitroanisole	V (47-135%)	1+2	mean recovery 76.4%, n=10
2-chloroethyl linoleate	V (0-102%)	2	mean recovery 66.86%, n=7
2-chloroethyl palmitate	V (0-105%)	2	mean recovery 68.0%, n=7
alachlor	S (0-121%)	3	mean recovery 47.6%, n=13
aldrin	C	1	mean recovery 85.2%, n=21
alpha-cypermethrin	C	2	mean recovery 89.3%, n=12
anilazine	S (4-87%)	2+3	mean recovery 32%, n=11
bromopropylate	NR	1-2-3	
captan	S (0-88%)	3	mean recovery 33.4%, n=13
carbophenothion	NR	1-2-3	mean recovery 3%, n=12
chlorfenvinphos, beta-	NR	1-2-3	mean recovery 2%, n=13
chlornitrofen	C	2	69-114% recovered, TDS
chlorobenzilate	NR	1-2-3	
chlorothalonil	S (0-86%)	2+3	mean recovery 37%, n=26
chlorpropham	C	2	mean recovery 82%, n=14
chlorpyrifos	C	2	mean recovery 83%, n=39
chlorpyrifos-methyl	C	2	mean recovery 83.4%, n=14
cyfluthrin	P	2	mean recovery 77.8%, n=14
DDE, p,p'-	P	1	recovery 75.3%, n=1
DDT, o,p'-	C	1	mean recovery 88.2%, n=13
diazinon	C	3	69-114% recovery, TDS
dichlofenthion	C	2	mean recovery 87.5%, n=12
diclofop-methyl	C	2	mean recovery 88.1%, n=25
dicloran	V (49-111%)	2+3	mean recovery 76.8%, n=12
dieldrin	C	2	mean recovery 87.7%, n=130

[1] Codes: C: complete (>80%); P: partial (50-80%); S: small (<50%); V: variable (approximate percentage when known); R: recovered but no quantitative information available; NR: not recovered.

[2] Eluants(s) in which chemical is eluted from Florisil, according to directions in 304 C6, *i.e.*, methylene chloride (CH_2Cl_2) eluants #1, 2, and 3. Entries for chemicals not recovered indicate which eluants were used in tests.

Table 304-b: Recovery Through 304 (E1-E5 + C6 + DG1-DG19)

Chemical	Recovery[1]	Eluant[2]	Notes
endosulfan I	C	2	mean recovery 89.0%, n=12
endosulfan sulfate	C	2	mean recovery 88.9%, n=27
endrin	C	2	recovery 82.7%, n=1
esfenvalerate	C	2	mean recovery 88.8%, n=12
ethion oxygen analog	NR	1-2-3	
fenarimol	S (0-58%)	3	mean recovery 13.5%, n=13
fenthion	NR	1-2-3	mean recovery 0.5%, n=13
fenvalerate	V (69-130%)	2	mean recovery 91%, n=14
fluchloralin	C	2	mean recovery 81%, n=11
haloxyfop methyl ester	C	2+3	mean recovery 91.3%, n=4 , 80% eluant 2 remainder in 3.
heptachlor	C	1	recovery 96.7%, n=1
heptachlor epoxide	C	2	mean recovery 84.4%, n=28
iprodione	S (0-52%)	3	mean recovery 16.2%, n=16 ; trace amount eluated by elauant 3.
iprodione metabolite isomer	V (12-120%)	3	mean recovery 73.2%, n=28 .
lindane	C	1	mean recovery 87.2%, n=40
linuron	V (43-114%)	3	mean recovery 73.5%, n=24
mecarbam	V (13-92%)	3	mean recovery 71.6%, n=15 .
methidathion	C	3	
methoxychlor, o, p'-	C	2	83-124% recoveries, TDS
methoxychlor, p, p'-	C	2	mean recovery 88.2%, n=12
metolachlor	NR	1-2-3	
mirex	C	1	mean recovery 89.4%, n=17
nonachlor, cis-	C	1	
nuarimol	NR	1-2-3	
octachlor epoxide	C	1	mean recovery 90.5%, n=32
parathion	C	2	mean recovery 81.7%, n=82
pentachlorophenyl methyl sulfide	C	1	mean recovery 84.7%, n=17
Perthane	C	1	mean recovery 87.5%, n=15
phenthoate	P		mean recovery 76.0%, n=12; eluant data to be tested.
phosalone	S (17-78%)	2+3	mean recovery 39.8%, n=16
phosmet	S	3	37%, 67% recoveries, TDS
pirimiphos-ethyl	V (29-109%)	3	mean recovery 67.2%, n=13

Table 304-b: Recovery Through 304 (E1-E5 + C6 + DG1-DG19)

Chemical	Recovery[1]	Eluant[2]	Notes
propargite	P	2	mean recovery 79.5%, n=12
prothiofos	P	2	mean recovery 71.9%, n=13
pyrazophos	C		recovery 107%, n=1 ; eluant data to be tested.
sulprofos	NR	1-2-3	
TDE, o,p'-	C	1	mean recovery 95.9%, n=15
TDE, p,p'-	C	1	mean recovery102.4%, n=14
TDE, p,p'-, olefin	C	1	mean recovery 86.0%, n=13
tecnazene	C	1	58-108% recoveries, TDS
tetradifon	C	2	
tridiphane	C	1+2	mean recovery 84.5%, n=13
vinclozolin	C	2	mean recovery 83.3%, n=12

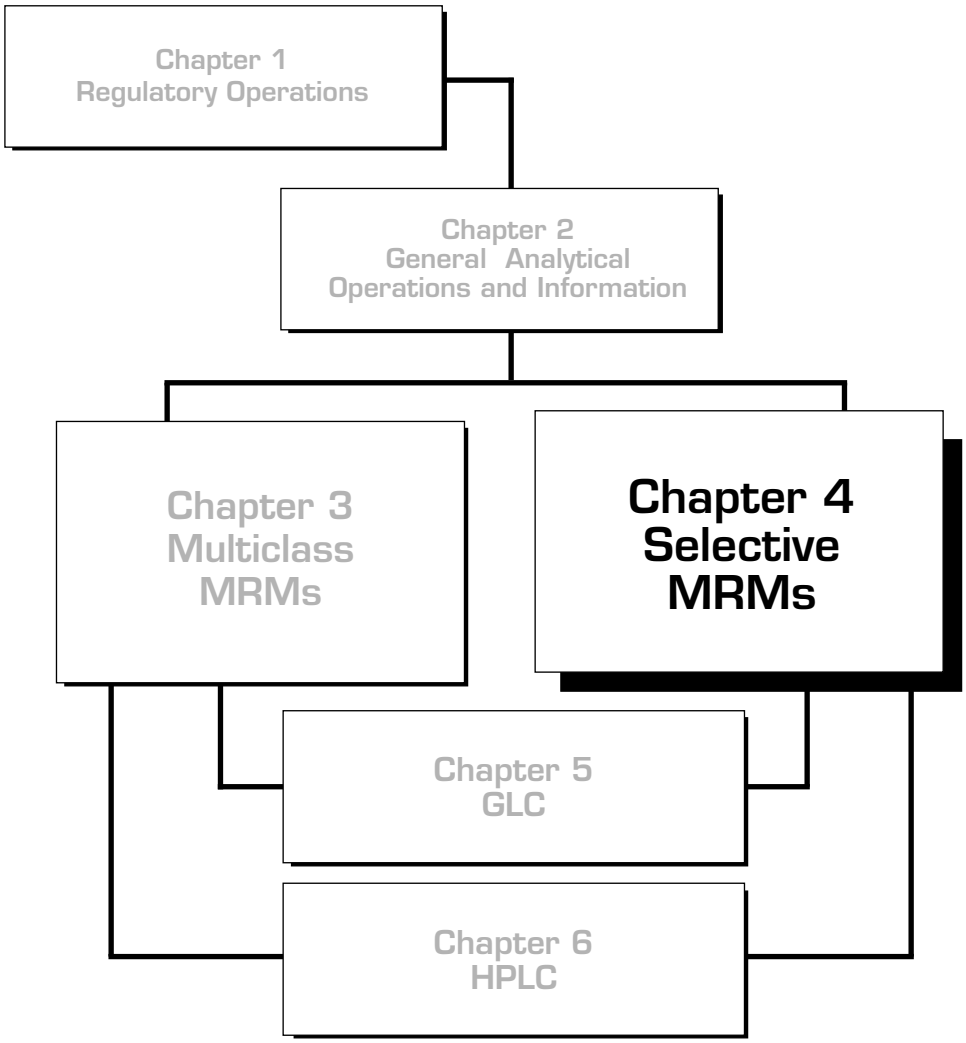
Table 304-c: Recovery of Chemicals Through Method 304 (E2 + C7 + DG1-DG19)
(extraction of fat from fatty products, Florisil column cleanup, GLC determination with various columns and detectors)

Chemical	Recovery[1, 2]	Eluant[3]	Notes
aldrin	C	6	mean recovery 94.8%, n=19
Aroclor 1254	C	6	mean recovery 80.0%, n=18
chlordane	C	6	recovery 83.0%, n=1
chlordane, cis-	C	6	recovery 93.0%, n=1
chlordane, trans-	C	6	recovery 124%, n=1
chlorpyrifos	C	6	mean recovery 88.0%, n=12
DDE, p,p'-	C	6	recovery 107%, n=1
DDT, p,p'-	C	6	mean recovery 87.2%, n=8
diazinon	C	15	mean recovery 107%, n=1
dieldrin	C	15	mean recovery 97.1%, n=31
heptachlor epoxide	C	6	
nonachlor, cis-	C	6	recovery 103%, n=1
nonachlor, trans-	S(0.8-32%)	6	mean recovery 16.4%, n=2
octachlor epoxide	C	6	recovery 83.0%, n=1
pentachlorobenzene	C	6	recovery 100%, n=1
TDE, p,p'-	V (59-93%)	6	mean recovery 75.5%, n=4

[1] Codes: C: complete (>80%); P: partial (50-80%); S: small (<50%); V: variable (approximate percentage when known); R: recovered but no quantitative information available; NR: not recovered.

[2] Recovery results do not include the optional alkaline hydrolysis step.

[3] Eluants(s) in which chemical is eluted from Florisil, according to directions in 304 C7, *i.e.*, 6 and 15% ethyl ether/petroleum ether (EE/PE). Entries for chemicals not recovered indicate which eluants were used in tests.



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401: METHOD FOR N-METHYLCARBAMATES

BASIC REFERENCES

Krause, R.T. (1979) *J. Chromatogr.* **185**, 615-624

Krause, R.T. (1980) *J. Assoc. Off. Anal. Chem.* **63**, 1114-1124

Krause, R.T. (1985) *J. Assoc. Off. Anal. Chem.* **68**, 734-741

GENERAL PRINCIPLES

N-methylcarbamate insecticide residues, including carbamate metabolites, are extracted with methanol. The extract is cleaned up by partitioning and column chromatography on a charcoal/Celite column. Residues are selectively determined with an HPLC system consisting of a reverse phase (RP) column, post-column hydrolysis and derivatization, and detection of the resultant derivative with a fluorescence detector.

Variations in the determinative step may be used for additional residues not of the N-methylcarbamate structure.

APPLICABILITY

Consult Guide to PAM I for additional information pertinent to the appropriate application of multiresidue methodology.

Method is applicable to residues with an N-methylcarbamate structure in either nonfatty or fatty foods when determinative step includes post-column hydrolysis and derivatization. See Table 401-a, following method description, for chemicals tested through the method.

Method is applicable to naturally fluorescent residues when post-column hydrolysis and derivatization are not performed. See Table 401-b, following Table 401-a.

Certain commodities, *e.g.*, oranges, contain naturally fluorescent co-extractives that interfere with analysis.

Limit of quantitation is 0.01 ppm carbofuran in high moisture products (fresh fruits and vegetables) and about 0.02 ppm in dry products.

REFERENCE STANDARDS

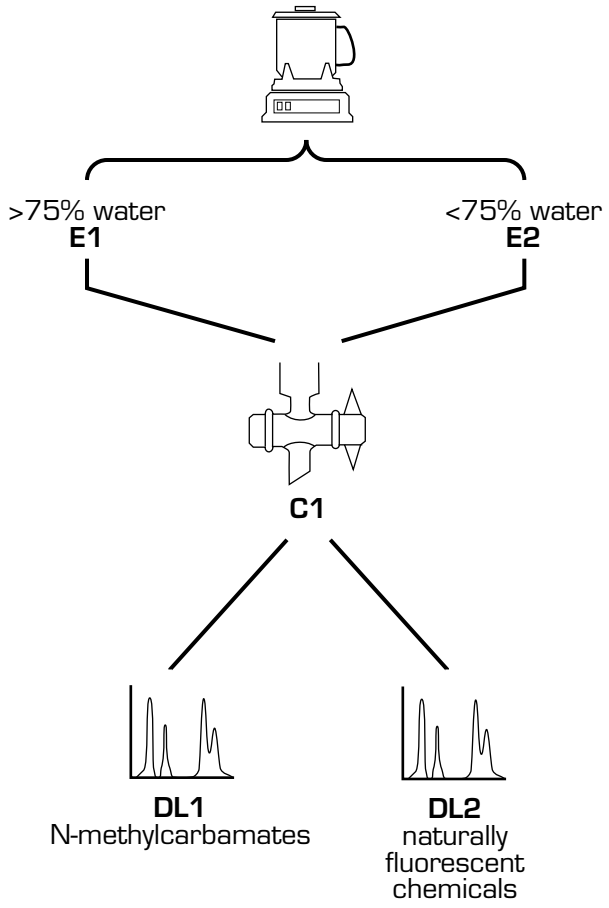
Dissolve reference standards of N-methylcarbamates in methanol to produce concentrations of 1 µg/mL. Store solutions in actinic glassware, and keep in refrigerator when not in use. Most carbamate standards stored in this manner are stable for several months. However, methiocarb sulfone and sulfoxide degrade within hours and days, respectively, even with stated storage precautions.

STEPS OF THE METHOD

Extraction (E)			**Recommended Use**
E1	(p. 401-3)	Extraction with methanol	high moisture products
E2	(p. 401-4)	Extraction with methanol, reduced sample size	low moisture products
Cleanup (C)			
C1	(p. 401-5)	Two stage liquid-liquid partitioning and charcoal/Celite column cleanup	all products

Determination (D)			Recommended Use
DL1	(p. 401-9)	HPLC, post-column hydrolysis and derivatization, fluorescence detection	N-methylcarbamate residues
DL2	(p. 401-13)	HPLC, fluorescence detection	naturally fluorescent residues

Figure 401-a
Method for N-Methylcarbamates



VALIDATION

The following combination has undergone interlaboratory validation and is recommended for use:

E1 + C1 + DL1

Validation report:

Krause, R.T. (1985) *J. Assoc. Off. Anal. Chem.* **68**, 726-733. Collaborative study leading to AOAC official final action status for aldicarb, aldicarb sulfone, bufencarb, carbaryl, carbofuran, 3-hydroxycarbofuran, methiocarb, methomyl, and oxamyl in grapes and potatoes.

AOAC official method reference: *Official Methods of Analysis of the AOAC* (1990) 15th ed., 985.23.

E1 EXTRACTION WITH METHANOL

Reference

Krause, R.T. (1980) *J. Assoc. Off. Anal. Chem.* **63**, 1114-1124

Principles

Residues are extracted from high moisture products (>75% water) with methanol, found to be the most effective extractant for N-methylcarbamates in tests using radiolabeled materials. The filtered extract is concentrated with a system that permits evaporation of the relatively high boiling point methanol without destroying heat-labile residues.

Apparatus

Buchner funnel (Buchner), porcelain, 12 cm diameter

evaporator, vacuum rotary with circulating chilled liquid (see Figure 401-b). Maintain 1+1 water/ethylene glycol solution in condensing coils and around receiving flask at –15° C with 1/2 horsepower cooling unit. Insulate condenser with Styrofoam or other material. Control evaporator vacuum with vacuum pump and gauge; manometer may be used but is not preferred.

filter paper, Sharkskin, or 597 S&S, to fit Buchner

flask, round-bottom (r-b), 2 L, T 24/40

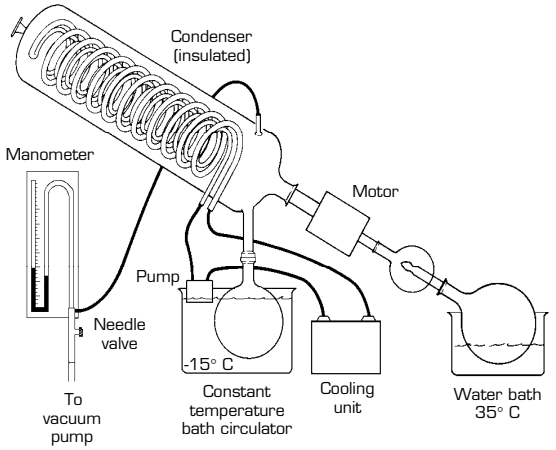
homogenizer, Polytron Model PT 10-35, with PT 35K generator containing knives, head equipped with metal (not Teflon) bushing

homogenizer jar, four-side, glass, 1 qt

magnetic stirrer, star, 10 mm diameter × 8 mm height

vacuum filtration flask, 500 mL

Figure 401-b
Vacuum Rotary Evaporator



To remove higher boiling solvents from solutions containing heat-labile residues. Water/ethylene glycol at –15° C cools receiving flask of rotary evaporator and (insulated) evaporator condenser.

Reagents

methanol, distilled from all-glass apparatus

Directions

- Add 150 g chopped high moisture product and 300 mL methanol to homogenizer jar.
- Homogenize mixture 30 sec at about half speed (setting of 7) and then 60 sec at full speed.
- Vacuum filter homogenate through Buchner fitted with filter paper and collect filtrate in 500 mL vacuum filtration flask. Reduce vacuum during filtration if filtrate begins to boil.
- Transfer portion of filtrate equivalent to 100 g sample to 2 L r-b flask.

$$\text{volume 100 g sample} = \frac{\text{mL water}}{\text{100 g sample}} + 200 \text{ mL methanol} - 10 \text{ mL contraction factor}$$

- Add enough water to r-b flask to total 100 mL water.
- Add star magnetic stirrer to r-b flask. Place 250 mL trap on 2 L r-b flask and attach to vacuum rotary evaporator.
- Circulate refrigerated (–15° C) (1+1) water/ethylene glycol through evaporator condensing coils; maintain receiving flask at –15° C by immersion in refrigerated bath.
- Apply vacuum slowly to minimize frothing by regulating with needle valve. After full vacuum is applied, slowly place flask in 35° C water bath. Concentrate extract to 75 mL.

ALTERNATIVE:

E2 EXTRACTION WITH METHANOL, REDUCED SAMPLE SIZE

Principle

Reduced sample size permits same amount of solvent to extract residues effectively from low moisture products (<75% water).

Directions

- Proceed as in E1, except extract 75 g ground low moisture product with 300 mL methanol.
- Transfer portion of filtrate equivalent to 50 g sample to 2 L r-b flask.

$$\text{volume 50 g sample} = \frac{\text{mL water}}{\text{50 g sample}} + 200 \text{ mL methanol}$$

- Continue as in E1, "Add enough water to r-b flask to total 100 mL water."

C1 LIQUID-LIQUID PARTITIONING AND CHARCOAL/CELITE COLUMN CLEANUP

Reference

Krause, R.T. (1980) *J. Assoc. Off. Anal. Chem.* **63**, 1114-1124

Principles

Residues in aqueous extract are transferred to acetonitrile by liquid-liquid partitioning in the presence of sodium chloride. Co-extractives are removed from the acetonitrile solution by partitioning them into petroleum ether, which is discarded. Residues are partitioned from acetonitrile into methylene chloride. Methylene chloride solution is cleaned up on a charcoal/Celite column, and residues are eluted with toluene/acetonitrile.

Apparatus

chromatographic column, 22 mm id × 300 mm, Teflon stopcock, coarse porosity fritted disc

chromatographic column, 25 mm id, plain

evaporator, vacuum rotary, as described in E-1

flasks, round-bottom (r-b), 250 and 500 mL, 1 L, T 24/40

magnetic stirrer, star, 10 mm diameter × 8 mm height

separatory funnel (separator), 250 and 500 mL

vacuum adapter, side arm, with T 24/40 joints

Reagents

acetonitrile, distilled from all-glass apparatus; see Section 204 for distillation directions

Celite 545

charcoal (Nuchar S-N), produced by Westvaco Corp. and available from Eastman Kodak, Cat. No. 118 0454

1+4 (w/w) charcoal/Celite, combined after each is prepared as directed below; mix thoroughly and store in sealed container

dichlorodimethylsilane

glass wool, Pyrex

hydrochloric acid, concentrated, reagent grade

isopropanol, distilled from all-glass apparatus

methanol, distilled from all-glass apparatus

methyl red

methylene chloride, distilled from all-glass apparatus

petroleum ether, distilled from all-glass apparatus

sodium chloride, reagent grade

2% (w/v) sodium chloride/water

20% (w/v) sodium chloride/water

sodium sulfate, anhydrous, granular, reagent grade; see Section 204 for handling directions

toluene, distilled from all-glass apparatus

eluant: 25% (v/v) toluene/acetonitrile

Preparation of Silanized Celite 545

- Slurry 150 g Celite 545 with 1 L (1+1) hydrochloric acid/water in 2 L beaker, cover with watch glass, and stir magnetically while boiling 10 min.
- Cool slurry, filter, and wash with distilled or HPLC grade water until filtrate is neutral.
- Wash Celite with 500 mL methanol followed by 500 mL methylene chloride, and then air dry Celite in hood on watch glass to remove solvent.
- Transfer Celite to 1 L glass-stoppered (g-s) Erlenmeyer flask. Heat unstoppered flask in 120° C oven overnight and then cool flask in desiccator.
- Place flask in hood and carefully pipet 3 mL dichlorodimethylsilane onto Celite. Stopper flask, mix well, and let flask remain at room temperature 4 hr.
- Add 500 mL methanol to flask, mix, and let stand 15 min.
- Filter silanized Celite and wash with isopropanol until neutral.
- Air dry silanized Celite in hood to remove isopropanol.
- Dry silanized Celite in 105° C oven 2 hr and cool in desiccator. Store silanized Celite in g-s container.
- Test Celite for total silanization with two tests. Place about 1 g Celite in 50 mL water; silanized Celite will float. Place second 1 g Celite in 20 mL toluene saturated with methyl red; silanized Celite will appear yellow. If particles of Celite are dispersed in water and/or appear pink with methyl red/toluene solution, active sites still exist on Celite; repeat silanization.

Purification of Charcoal

- Slurry 100 g Nuchar S-N with 700 mL hydrochloric acid, cover with watch glass, and stir magnetically while boiling 1 hr.
- Add 700 mL water, stir, and boil additional 30 min.
- Cool slurry, filter, and wash with water until neutral.
- Wash Nuchar S-N with 500 mL methanol followed by 500 mL methylene chloride, and air dry Nuchar S-N in hood to remove solvent.
- Dry Nuchar S-N in 120° C oven 4 hr. Cool in desiccator. Store Nuchar S-N in g-s container.

Testing of Charcoal/Celite

- Prepare cleanup column of (1+4) (w/w) charcoal/Celite as described below.
- Prepare methanol solution of 5 µg/mL each carbaryl, methiocarb, methiocarb sulfoxide, and methomyl. Use freshly prepared mixed standard solution; methiocarb sulfoxide degrades in solution.

- Pipet 5 mL solution into 250 mL r-b flask and 5 mL into 25 mL actinic volumetric flask.
- Dilute solution in volumetric flask to 25 mL with methanol; use as HPLC reference standard.
- Evaporate standard solution in r-b flask just to dryness with vacuum rotary evaporator as described below. After last trace of methanol has evaporated, remove r-b flask from evaporator and dissolve carbamate residue in 10 mL methylene chloride.
- Transfer methylene chloride solution in r-b flask to prepared adsorbent column and elute as described below.
- After evaporation of eluate in r-b flask, dissolve residue in 25 mL methanol, filter aliquot through filtration device, and quantitate recovery of carbamates as in DL1. Nuchar S-N is considered satisfactory if recovery is ≥95%.

Directions

Partitioning

- Transfer concentrated extract from E1 or E2 to 500 mL separator containing 15 g sodium chloride. Shake separator until sodium chloride is dissolved.
- Wash r-b flask with three 25 mL portions acetonitrile, transferring each to 500 mL separator; shake separator 30 sec, and let layers separate 5 min.
- Drain aqueous phase into 250 mL separator containing 50 mL acetonitrile, shake 20 sec, let layers separate, and discard aqueous layer.
- Add 25 mL 20% aqueous sodium chloride solution to acetonitrile in 500 mL separator, shake 20 sec, let layers separate, and transfer aqueous solution to 250 mL separator.
- Shake 250 mL separator 20 sec, let layers separate, and discard aqueous layer.
- Add 100 mL petroleum ether to 500 mL separator, shake 20 sec, let layers separate, and drain acetonitrile layer into second 500 mL separator.
- Transfer acetonitrile in 250 mL separator to first 500 mL separator which contains petroleum ether, shake 20 sec, let layers separate, and transfer acetonitrile to second 500 mL separator.
- Add 10 mL acetonitrile to first 500 mL separator, shake, let layers separate, and transfer acetonitrile to second 500 mL separator. Discard petroleum ether layer.
- Add 50 mL 2% aqueous sodium chloride solution to acetonitrile in second 500 mL separator. Extract mixture successively with 100, 25, and 25 mL methylene chloride, shaking each 20 sec (shake 25 mL portions gently).
- Drain lower methylene chloride/acetonitrile layers through 22 mm id column containing about 5 cm sodium sulfate. Collect eluate in 1 L r-b flask.
- Add star magnetic stirrer to r-b flask. Place 250 mL trap on 1 L r-b flask and attach to vacuum rotary evaporator.
- Circulate refrigerated (–15° C) (1+1) water/ethylene glycol through evaporator condensing coils; maintain receiving flask at –15° C by immersion in refrigerated bath.

- Apply vacuum slowly to minimize frothing by regulating with needle valve. After full vacuum is applied, slowly place flask in 35° C water bath.
- Remove r-b flask from evaporator immediately after last traces of solution have evaporated and add 10 mL methylene chloride to r-b flask.

Charcoal/Celite Cleanup

- Fit one-hole No. 5 rubber stopper onto tip of chromatographic column with stopcock, add side-arm vacuum adapter and 500 mL r-b flask, open stopcock, and connect apparatus to vacuum line.
- Place 0.5 g silanized Celite in chromatographic column, tamp, add 5 g charcoal/Celite mixture, and tamp again. Add 1-2 cm glass wool plug on top of adsorbent.
- Prewash column with 50 mL 25% toluene/acetonitrile eluant. Close stopcock when prewash solution is about 0.5 cm from top of glass wool.
- Disconnect vacuum, discard solution in r-b flask, and reconnect flask to apparatus.
- Transfer 10 mL methylene chloride extract from partitioning steps to column and elute column at 5 mL/min.
- Wash 1 L r-b flask with 10 mL methylene chloride and then with 25 mL eluant. Transfer each separately to column and elute each to top of glass wool before adding next solution.
- Add 100 mL eluant and elute column at 5 mL/min. Turn off stopcock when top of eluant reaches top of glass wool.
- Evaporate solution in 500 mL r-b flask just to dryness using vacuum evaporator as above. Remove flask from evaporator immediately after all solution has evaporated.
- Immediately pipet 5 mL methanol into 500 mL r-b flask to dissolve residue.
- Cleaned up extract contains 20 g sample equivalent/mL solution for high moisture products and 10 g sample equivalent/mL solution for low moisture products.

DL1 HPLC, POST-COLUMN DERIVATIZATION, FLUORESCENCE DETECTION

Reference

Krause, R.T. (1978) *J. Chromatogr. Sci.* **16**, 281-288

Principles

Residues in methanol solution are separated on a C-8 reverse phase HPLC column using acetonitrile/water gradient mobile phase. Residues eluting from the column are hydrolyzed in-line to methylamine under alkaline conditions. Methylamine is reacted, also in-line, with o-phthalaldehyde and 2-mercaptoethanol to form a fluorophore that is measured by a fluorescence detector. This post-column derivatization-fluorescence detection determinative step is very selective for residues containing the N-methylcarbamate structure.

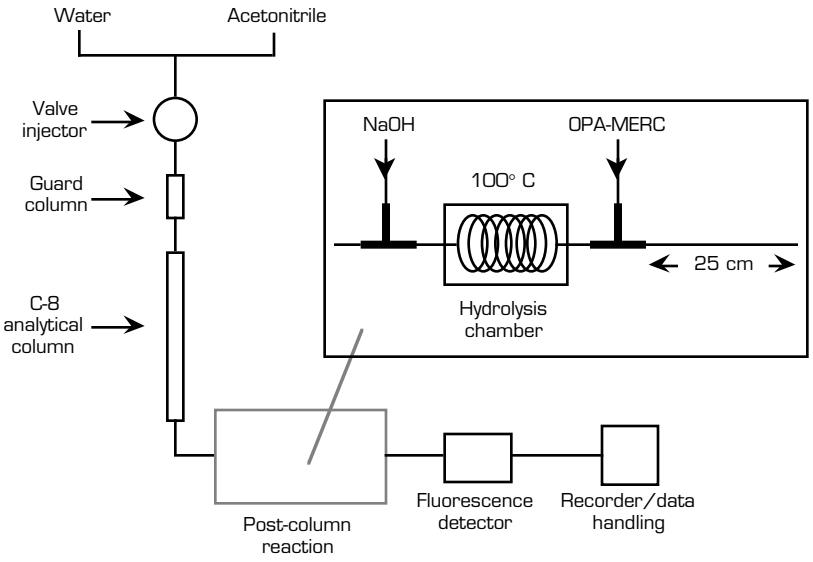
Apparatus

filtration device for solutions, 10 mL syringe with Luer-Lok tip, fitted with either (a) 13 mm diameter Swinny stainless steel filter holder and 13 mm diameter filters, 5.0 µm LS-type, or (b) disposable membrane filters, 13 mm diameter, 0.22 µm nylon membrane, encased in polypropylene. (Preassembled devices that do not require a syringe are also available.)

HPLC system (Figure 401-c) must meet system suitability test below. Complete system consists of:

1) mobile phase delivery system, programmable HPLC gradient system
2) injector, automatic sampler with 10 µL injection loop
3) guard column, stainless steel, containing 25-37 µm pellicular C-8 or C-18 packing
4) column oven or heater

Figure 401-c
HPLC System for Determination of N-Methylcarbamates



5) analytical column, 25 cm × 4.6 mm id, containing 6 µm Zorbax C-8 spherical particles. Column packing should consist of 5 or 6 µm spherical silica particles bonded with monofunctional octyl silane reagent to form monomolecular bond.

6) connecting tubing, No. 304 stainless steel (1.6 mm od × 0.18 mm id) to connect injector, column, and first tee

7) post-column derivatization unit, as shown in telescoped portion of Figure 401-b. Units used during collaborative study of method were assembled from the following parts:

   a) reservoirs for sodium hydroxide and OPA-MERC reaction solutions, 60 cm × 25 mm id glass columns with Teflon fittings; pressurize reservoirs with nitrogen gas, adjusted to create appropriate reagent flow. (Pumps can be substituted for nitrogen gas pressure.)

   b) 6 m × 0.5 mm id Teflon restriction coil, to connect each reservoir to 15 cm × 0.18 mm id stainless steel tubing, which in turn is connected to 0.74 mm id stainless steel mixing tee (Valco Instruments Co., Cat. No. ZVT-062) for connection to flow of mobile phase

   c) carbamate hydrolysis chamber, stainless steel tubing, 3 m × 0.48 mm id No. 321, coiled to fit in small oven capable of maintaining constant, uniform 100° C

   d) reaction tube, 25 cm stainless steel tubing between tee that delivers OPA-MERC solution and 1.5 cm × 0.3 mm id detector cell tubing

   Commercial post-column derivatization units that replace these components are now available from several manufacturers (ABI, Pickering Instruments, Waters). Systems with dual piston pumps are preferred.

8) fluorescence detector, dual monochromator, equipped with ≤20 µL cell

9) recorder, strip chart recorder or computing integrator compatible with detector

Reagents

acetonitrile, UV grade distilled from all-glass apparatus. Before use, degas acetonitrile in glass bottles by applying vacuum and slowly stirring with magnetic stirrer 5 min. Acetonitrile other than HPLC grade may cause broad, nonreproducible peaks in chromatograms.

2-mercaptoethanol (MERC), 98+%

methanol, distilled from all-glass apparatus

o-phthalaldehyde (OPA), chromatographic grade

sodium borate buffer solution, 0.05 M. Add 19.1 g ACS grade sodium tetraborate decahydrate ($Na_2B_4O_7 \bullet 10\ H_2O$) and about 500 mL degassed HPLC grade water to 1 L volumetric flask. Heat flask in steam bath to dissolve sodium tetraborate, cool to room temperature, and dilute to volume with degassed HPLC grade water. Mix well, but gently, to minimize re-incorporation of air into solution.

sodium hydroxide solution, 0.05 N. Prepare clear supernate sodium hydroxide as follows: to one part sodium hydroxide (reagent quality containing <5% sodium carbonate), add one part water and swirl until solution is complete. Stopper and set aside until sodium carbonate has settled, leaving clear liquid

(about 10 days). Pipet 27 mL clear supernate sodium hydroxide into 100 mL volumetric flask. Dilute to volume with water and mix (5 N sodium hydroxide).

Pipet 10 mL 5 N sodium hydroxide into 1 L volumetric flask. Dilute to volume with degassed HPLC grade water and mix well, but gently, to minimize reincorporation of air into solution.

water, HPLC grade, commercial product or prepared from water purification equipment that produces distilled, deionized water. For HPLC, degas water as described for acetonitrile. Water must be adequately purified to prevent plugging HPLC column and extraneous peaks in chromatograms. All water used in HPLC procedure must be HPLC grade. ("Water" that does not specify HPLC grade means distilled water.)

OPA-MERC reaction solution. Weigh 500 mg OPA, transfer to 1 L volumetric flask, add 10 mL methanol, and swirl to dissolve OPA. Add about 500 mL 0.05 M sodium borate buffer solution and 1 mL 2-mercaptoethanol. Dilute to volume with sodium borate buffer solution. Mix well, but gently, to minimize reincorporation of air into solution. (Borate solution purchased in plastic bottles or low purity grades of OPA may cause excessively high background fluorescence.) Solution is acceptable for about 2 days when stored at room temperature, about 1 week when stored in refrigerator or under helium.

System Operation

- Adjust mobile phase flow rate to 1.50±0.02 mL/min with 50% acetonitrile/HPLC grade water.
- Adjust flow rates of 0.05 N sodium hydroxide and OPA-MERC reaction solution to 0.50±0.02 mL/min each. Operate column oven at 35° C and hydrolysis chamber at 100° C.
- Set fluorescence detector excitation and emission wavelengths to 340 and 455 nm, respectively, and slit widths to 15 and 12 nm, respectively. Set detector photomultiplier tube gain to low and time constant to 1 sec.
- Equilibrate system 10 min with 12% acetonitrile/HPLC grade water, inject sample, and begin 30 min linear gradient to 70% acetonitrile/HPLC grade water.
- Adjust sensitivity so that 10 ng carbofuran produces 50±5% full scale deflection on printer-plotter. Baseline noise should be <2%, and carbamates should elute as shown in Figure 401-c.
- If system will not be used for several days, replace aqueous mobile phase with methanol and pump through system. Drain sodium hydroxide and OPA-MERC reaction solutions from their reservoirs, and wash reservoirs and associated tubing with water, then methanol. When starting system, change mobile phase to HPLC grade water, and wash reservoirs and associated tubing with water before adding reaction solutions.

System Suitability Test

See Chapter 6, HPLC, for further information about evaluating HPLC systems.

- Prepare mixed standard solution containing 1 µg/mL each aldicarb sulfoxide, aldicarb sulfone, carbofuran, and carbaryl.
- Chromatograph solution, using HPLC system operation described above. Retention times will be about 6.5, 8, 20, and 21 min, respectively.

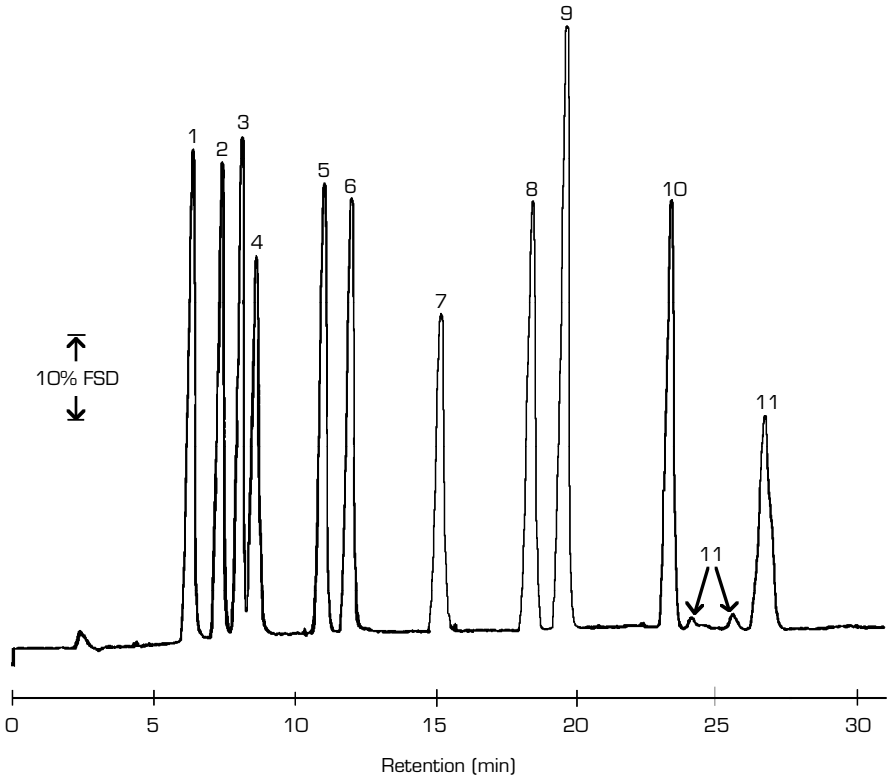
- Baseline resolution should be achieved between aldicarb sulfoxide and aldicarb sulfone and between carbaryl and carbofuran.

Directions

See Figure 401-d for typical chromatogram of carbamate pesticides and metabolites.

- Filter methanol extract from C1 r-b flask through filtration device.
- Collect filtrate in 10 mL centrifuge tube or other suitable container. About 4.5 mL filtrate will be collected. Exact volume of filtrate collected is not critical because sample concentration (g sample/mL methanol) is known.
- If solution requires dilution, pipet aliquot into another container and dilute to volume, as needed.
- Inject 10 µL methanol solution into HPLC system.
- Tentatively identify residue peaks on basis of retention times. Measure peak area or height and determine residue amount by comparison to peak area or height obtained from known amount of appropriate reference standard(s). To ensure valid measurement of residue amount, sizes of peaks from sample residue and reference standard should match within ±25%. Chromatograph reference standard(s) immediately after sample.

Figure 401-d
HPLC Chromatogram of Carbamates and Metabolites



Chromatographed at conditions described in DL1, with post-column derivatization. 1) aldicarb sulfoxide; 2) aldicarb sulfone; 3) oxamyl; 4) methomyl; 5) 3-hydroxycarbofuran; 6) methiocarb sulfoxide; 7) aldicarb; 8) carbofuran; 9) carbaryl; 10) methiocarb; 11) bufencarb.

ALTERNATIVE:

DL2 HPLC, FLUORESCENCE DETECTION

Reference

Krause, R.T. (1983) *J. Chromatogr.* **255**, 497-510

Principles

Residues in methanol solution are separated on a C-8 reverse phase HPLC column using an acetonitrile/water gradient mobile phase. Naturally fluorescent residues eluting from the column are detected and measured by a fluorescence detector.

Directions

- Set up and operate an HPLC system in the same manner as DL1, except:

  operate hydrolysis chamber at ambient temperature.

  set detector excitation and emission wavelengths at 288 and 330 nm, respectively.

  turn off pumps or nitrogen flow that add sodium hydroxide and OPA-MERC reaction solutions to mobile phase.

- Perform determination as in DL1.

CONFIRMATION

Confirm tentative identification of naturally fluorescent residues by using DL2. See Table 401-b for list of chemicals for which this confirmatory step is appropriate. Use excitation and emission wavelengths that are optimum for residue being confirmed.

Confirm N-methylcarbamates that include phenolic structure by injecting final extract into HPLC with post-column hydrolysis-electrochemical detection method described in Krause, R.T. (1988) *J. Chromatogr.* **442**, 333-343. This method is based on selective detection of phenolic group of insecticides, rather than carbamate moiety. Intact carbamates are separated by reverse phase HPLC using gradient acetonitrile/water mobile phase as described above. Eluted carbamates are hydrolyzed in-line with dilute sodium hydroxide at 100° C, and resulting phenols are detected with coulometric electrochemical detector. Technique has been tested with six carbamates (bufencarb, carbaryl, carbofuran, 3-hydroxycarbofuran, isoprocarb, and methiocarb) and four crops (apples, cabbage, grapes, and tomatoes).

402: METHOD FOR ACIDS AND PHENOLS

BASIC REFERENCE

Hopper, M.L. *et al.* (1992) *J. AOAC Int.* **75**, 707-713

GENERAL PRINCIPLES

Acidic and phenolic residues are extracted from commodity acidified with sulfuric acid by various techniques dictated by the type of commodity. The extract is cleaned up by gel permeation chromatography (GPC). Residues in the concentrated extract are methylated by ion pair alkylation and further cleaned up by Florisil column chromatography. The resulting methyl esters are determined by GLC. Certain residues can be determined only by element-selective GLC detectors.

APPLICABILITY

Consult Guide to PAM I for additional information pertinent to the appropriate application of multiresidue methodology.

Method is applicable to a wide variety of fatty and nonfatty foods; several different extraction steps are available for the different food types. Cleanup by GPC is most practical when a large number of analyses are being performed. Method was originally designed for chlorophenoxy acids, but has been found applicable to a variety of acidic and phenolic residues. See Table 402-a, following method description, for pesticides and metabolites tested through the steps of this method.

REFERENCE STANDARDS

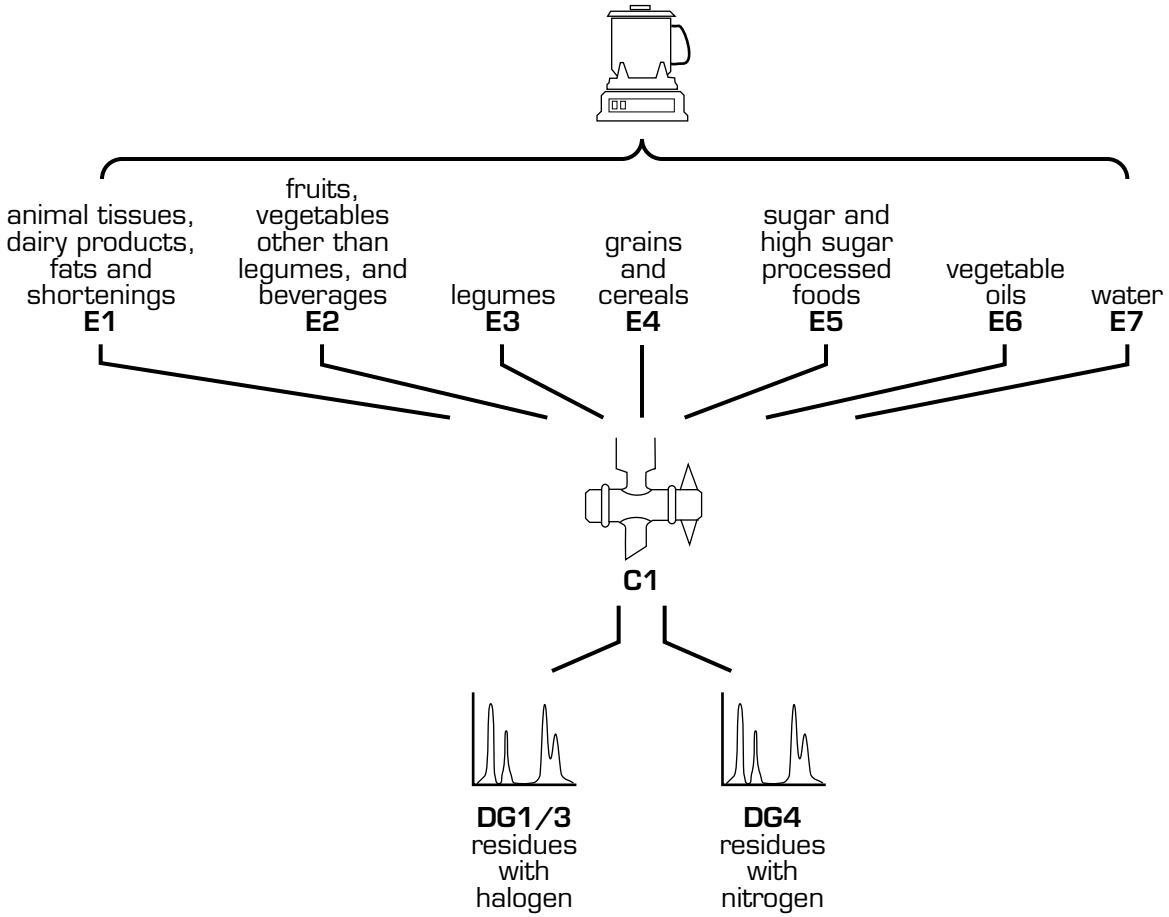
Use reference standards of methyl esters/ethers of acids or phenols, if available. Otherwise, use standards of the acids/phenols methylated through C1a and cleaned up through C1b. Prepare stock solutions in acetone.

STEPS OF THE METHOD

Extraction (E)			**Recommended Use**
E1	(p. 402-3)	Extraction with solvents from acidified, denatured products	animal tissues, dairy products, fats, and shortenings
E2	(p. 402-7)	Extraction with acidified methylene chloride	fruits, vegetables other than legumes, and beverages
E3	(p. 402-9)	Extraction with acidified methanol	legumes
E4	(p. 402-10)	Extraction with acidified water/ methanol	grains and cereals
E5	(p. 402-11)	Extraction with acidified methanol	sugar and high sugar processed foods
E6	(p. 402-13)	Dissolution in methylene chloride/ hexane	vegetable oils
E7	(p. 402-15)	Extraction with acidified methylene chloride	water
Cleanup and methylation (C)			
C1	(p. 402-17)	GPC cleanup, methylation, and Florisil column cleanup	all products

Determination (D)			Recommended Use
DG1	(p. 302-25)	GLC, 100% methyl siloxane column, 200°, EC detector	halogenated acids and phenols
DG3	(p. 302-29)	GLC, 100% methyl siloxane column, 200°, ElCD-X	halogenated acids and phenols
DG4	(p. 302-31)	GLC, 100% methyl siloxane column, 200°, ElCD-N	acids and phenols containing nitrogen

Figure 402-a
Method for Acids and Phenols



VALIDATION

The following combination has undergone validation in a single laboratory, over many years, with repeated recoveries performed in conjunction with FDA's Total Diet Study; these are recommended for use:

E1 + C1 + DG1

Validation report:

Hopper, M.L. *et al.* (1992) *J. AOAC Int.* **75**, 707-713. Where slight differences occur between the method description in Section 402 and in the Hopper *et al.* 1992 publication, this section reflects standard operating procedure in the Total Diet Study.

E1 EXTRACTION WITH SOLVENTS FROM ACIDIFIED, DENATURED PRODUCTS

Reference

Hopper, M.L. *et al.* (1992) *J. AOAC Int.* **75**, 707-713

Principles

Fat and residues are dissolved in ethyl ether and petroleum ether after the fatty product has been denatured with oxalate and alcohol and acidified with sulfuric acid. Ether extract is washed with large quantities of water to remove co-extractives.

Apparatus

blender, high speed; explosion-proof Waring Blendor, 1 qt jar

centrifuge, explosion-proof, to hold 500 mL bottles

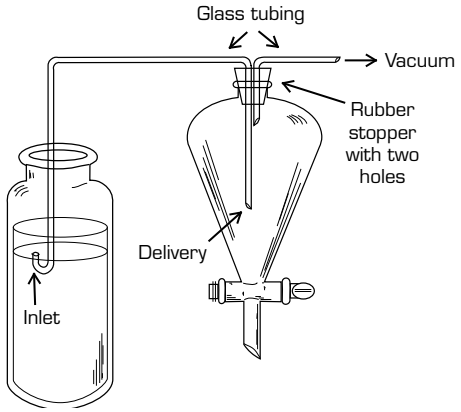
centrifuge bottle, glass, 500 mL. Use glass stopper or cover rubber stopper with aluminum foil to avoid contamination.

delivery tube apparatus (Figure 402-b), fabricated in laboratory

Kuderna-Danish concentrator (K-D), 500 mL, with Snyder column, plain receiving flask

separatory funnel (separator), 250 mL and 1 L

Figure 402-b
Delivery Tube Apparatus



Glass tube, inserted in one hole of two-hole rubber stopper, is used to draw upper solvent layer from centrifuge bottle into separatory funnel. Siphon tube is straight or bent in U-shape and inlet end placed at interface of two phases in centrifuge bottle. Second hole in stopper is fitted with another glass tube. Vacuum drawn through second tube causes upper phase from centrifuge bottle to transfer into separator.

(Corrigan, E. (Nov. 1963) (FDA) Bureau By-Lines 5, 20; Sawyer, L.D., and Baca, J.R. (May 1978) LIB 2188, FDA, Rockville, MD.)

Reagents

boiling chips, 20-30 mesh carborundum

ethyl ether, distilled from all-glass apparatus, with 2% ethanol as preservative, see Section 204 for peroxide test

methanol, distilled from all-glass apparatus

methylene chloride, distilled from all-glass apparatus

petroleum ether, distilled from all-glass apparatus

sodium (or potassium) oxalate, reagent grade

sodium chloride aqueous solution, saturated

10% sulfuric acid, reagent grade

1+1 (v/v) ethyl ether/petroleum ether

50% (v/v) methylene chloride/hexane

Directions

For each batch of samples analyzed, fortify commodity with chlorophenoxy acid herbicide(s) and pentachlorophenol and analyze it with others to verify adequacy of recoveries.

Animal Tissues

- Weigh 50 g sample into blender jar.
- Add 50 mL distilled water, 100 mL methanol, 10 mL 10% sulfuric acid, and about 2 g sodium or potassium oxalate.
- Blend at high speed 2 min and transfer to centrifuge bottle with aid of powder funnel. Rinse blender jar and funnel with 50 mL ethyl ether and add to bottle.

Dairy Products

- Grind cheese and other solid products before analysis. Weigh appropriate amount into centrifuge bottle: 100 g milk or other relatively low fat commodity, 25-50 g cheese or other high fat commodity.
- Add 100 mL methanol, 10 mL 10% sulfuric acid, and about 2 g sodium or potassium oxalate; mix. Add 50 mL ethyl ether.

Fats and Shortenings

- Weigh 25 g sample into centrifuge bottle.
- Add 50 mL distilled water, 100 mL methanol, 10 mL 10% sulfuric acid, and about 2 g sodium or potassium oxalate; mix. Add 50 mL ethyl ether.

All

- Shake centrifuge bottle vigorously 1 min; then add 50 mL petroleum ether and shake vigorously 1 min.
- Centrifuge about 5 min at 1500 rpm. Transfer top (solvent) layer, with delivery tube apparatus, into 1 L separator containing 500-600 mL water, 30 mL saturated sodium chloride solution, and 10 mL 10% sulfuric acid.
- Re-extract aqueous residue in centrifuge bottle twice, shaking vigorously 1 min with 50 mL (1+1) ethyl ether/petroleum ether. After each extraction, centrifuge and transfer solvent layer into same 1 L separator.
- Mix combined extracts and water in separator thoroughly but cautiously to prevent emulsion formation. Drain and discard water.
- Rewash (gently) solvent layer twice with 100 mL water, 10 mL 10% sulfuric acid and 30 mL saturated sodium chloride solution; discard wash solution each time. If emulsions form, add additional 5 mL saturated sodium chloride solution to wash.
- After final wash is discarded, transfer ether layer to 250 mL separator. Let stand ≥30 min. Drain and discard any water and emulsion from separator.
- Transfer ether to K-D with plain receiving flask, add boiling chips, and evaporate solvent.
- Cool, add 50 mL methylene chloride to extracted fat, and mix. Add boiling chips and evaporate on steam bath until level in receiving flask does not change and there is still methylene chloride in Snyder column traps. Cool.
- Use approximate fat content of commodity, as listed in Section 201, to determine what dilution is required to achieve concentration of ≤0.16 g fat/mL. Quantitatively transfer fat from receiving flask to glass-stoppered (g-s) graduate of appropriate volume. Use 50% methylene chloride/hexane for rinsing during transfer and then dilute solution to predetermined volume with that solvent mixture.

- Transfer aliquot of solution to tared vessel; evaporate to dryness at steam bath temperature under current of dry air. Weigh and record weight of fat extracted. If necessary, adjust remaining solution volume so that solution contains ≤0.16 g fat/mL.
- Clean up extract by C1; equivalent weight of whole product cleaned up is:

$$\frac{\text{original sample weight}}{\text{mL final extract}} \times \text{mL loaded onto GPC}$$

E2 EXTRACTION WITH ACIDIFIED METHYLENE CHLORIDE

Reference

Hopper, M.L. *et al.* (1992) *J. AOAC Int.* **75**, 707-713

Principle

Residues are extracted from fruits and vegetables with methylene chloride after acidification.

Apparatus

blender, high speed; explosion-proof Waring Blendor, 1 qt jar

centrifuge, explosion-proof, to hold 500 mL bottles

centrifuge bottle, glass, 500 mL. Use glass stopper or cover rubber stopper with aluminum foil to avoid contamination.

delivery tube apparatus, see Figure 402-a in E1; apparatus with straight tube inlet (rather than U-shape) is preferred

graduated cylinder (graduate), 250 mL

Kuderna-Danish concentrator (K-D), 500 mL, with Snyder column, 10 mL graduated receiving flask

separatory funnel (separator), 250 mL

Reagents

boiling chips, 20-30 mesh carborundum

glass wool, Pyrex

hexane, distilled from all-glass apparatus

methylene chloride, distilled from all-glass apparatus

10% sulfuric acid, reagent grade

Directions

For each batch of samples analyzed, fortify commodity with chlorophenoxy acid herbicide(s) and pentachlorophenol and analyze it with others to verify adequacy of recoveries.

- Weigh 100 g sample (chopped fruits or vegetables other than legumes or beverages) into blender jar. Add 10 mL 10% sulfuric acid and 250 mL methylene chloride.
- Blend 2 min at high speed and pour into centrifuge bottle with aid of powder funnel.
- Centrifuge 5 min at 1500 rpm. Transfer top (water) layer, with delivery tube apparatus, into 1 L separator, then discard.
- Carefully decant methylene chloride (leaving cake in centrifuge bottle) through funnel containing glass wool plug into separator. Let stand ≥30 min. Drain and discard any water and emulsion from separator.
- Transfer methylene chloride to graduate and record volume.

- Transfer measured volume of methylene chloride to K-D with 10 mL receiving flask. Add boiling chips and concentrate to 5 mL on steam bath. Dilute extract to 10 mL with hexane and mix.
- Clean up extract by C1; equivalent weight of product cleaned up is:

$$100\ \text{g} \times \frac{\text{mL methylene chloride recovered}}{250\ \text{mL}} \times \frac{\text{mL loaded onto GPC}}{10\ \text{mL}}$$

E3 EXTRACTION WITH ACIDIFIED METHANOL

Reference

Hopper, M.L. *et al.* (1992) *J. AOAC Int.* **75**, 707-713

Principle

Residues are extracted from legumes with methanol after acidification and partitioned into methylene chloride.

Apparatus

blender, high speed; explosion-proof Waring Blendor, 1 qt jar

centrifuge, explosion-proof, to hold 500 mL bottles

centrifuge bottle, glass, 500 mL. Use glass stopper or cover rubber stopper with aluminum foil to avoid contamination.

graduated cylinder (graduate), 100 and 250 mL

Kuderna-Danish concentrator (K-D), 500 mL, with Snyder column, 10 mL graduated receiving flask

separatory funnel (separator), 2 L

Reagents

boiling chips, 20-30 mesh carborundum

glass wool, Pyrex

hexane, distilled from all-glass apparatus

methanol, distilled from all-glass apparatus

methylene chloride, distilled from all-glass apparatus

sodium chloride aqueous solution, saturated

10% sulfuric acid, reagent grade

Directions

For each batch of samples analyzed, fortify commodity with chlorophenoxy acid herbicide(s) and pentachlorophenol and analyze it with others to verify adequacy of recoveries.

- Weigh 100 g chopped legume vegetable into blender jar. Add 10 mL 10% sulfuric acid and 200 mL methanol.
- Blend mixture in blender jar 2 min at high speed and pour into centrifuge bottle with aid of powder funnel.
- Centrifuge 5 min at 1500 rpm and carefully pour top layer through funnel containing glass wool plug into 250 mL graduate. Measure volume re-covered if <250 mL or take 250 mL.
- Pour measured extract into 2 L separator and add 100 mL methylene chloride. Shake separator 30 sec, then add 30 mL saturated sodium chloride solution, 10 mL 10% sulfuric acid, and 650 mL water.
- Shake 30 sec and allow emulsion to settle ≥30 min. Drain and discard any water and emulsion from separator.

- (If emulsion does not break, drain emulsion into centrifuge bottle and centrifuge at 1500 rpm 5 min. Siphon off and discard water layer. Pour methylene chloride through funnel containing glass wool plug into 250 mL separator. Let stand ≥30 min to ensure complete separation of any remaining water. Drain and discard any water and emulsion from separator.)
- Transfer methylene chloride layer to 100 mL graduate and record volume.
- Transfer measured volume of methylene chloride to K-D with 10 mL receiving flask. Add boiling chips and concentrate extract on steam bath to about 5 mL. Dilute to 10 mL with hexane and mix.
- Clean up extract by C1; equivalent weight of legumes cleaned up is:

$$100\text{ g} \times \frac{\text{mL extract recovered after centrifugation}}{200 + 10 + (100\text{ g} \times \%\text{ moisture})} \times \frac{\text{mL methylene chloride recovered}}{100\text{ mL}} \times \frac{\text{mL loaded onto GPC}}{10\text{ mL}}$$

ALTERNATIVE:

E4 EXTRACTION WITH ACIDIFIED WATER/METHANOL

Principle

Water/methanol replaces methanol for extraction from grains and cereal products to accommodate their low moisture.

Additional Reagents

30% (v/v) water/methanol

Directions

- Weigh 50 g ground grain or cereal product into blender jar. Add 10 mL 10% sulfuric acid and 340 mL 30% water/methanol.
- Continue as in E3, "Blend mixture in blender jar 2 min. . ."
- Equivalent weight of grains or cereal products cleaned up is:

$$50\text{ g} \times \frac{250\text{ mL}}{340\text{ mL} + 10\text{ mL}} \times \frac{\text{mL methylene chloride recovered}}{100\text{ mL}} \times \frac{\text{mL loaded onto GPC}}{10\text{ mL}}$$

E5 EXTRACTION WITH ACIDIFIED METHANOL

Reference

Hopper, M.L. *et al.* (1992) *J. AOAC Int.* **75**, 707-713

Principles

Residues are extracted from sugar or high sugar processed foods with aqueous methanol after acidification; residues are then transferred to methylene chloride by partitioning.

Apparatus

blender, high speed; explosion-proof Waring Blendor, 1 qt jar

centrifuge, explosion-proof, to hold 500 mL bottles

centrifuge bottle, glass, 500 mL. Use glass stopper or cover rubber stopper with aluminum foil to avoid contamination.

graduated cylinder (graduate), 250 mL

Kuderna-Danish concentrator (K-D), 500 mL, with Snyder column, 10 mL graduated receiving flask

separatory funnel (separator), 250 mL and 2 L

Reagents

boiling chips, 20-30 mesh carborundum

glass wool, Pyrex

methanol, distilled from all-glass apparatus

methylene chloride, distilled from all-glass apparatus

sodium chloride aqueous solution, saturated

10% sulfuric acid, reagent grade

Directions

For each batch of samples analyzed, fortify commodity with chlorophenoxy acid herbicide(s) and pentachlorophenol and analyze it with others to verify adequacy of recoveries.

- Weigh 50 g sugar or high sugar processed food into blender jar. Add 10 mL 10% sulfuric acid, 100 mL water, and 200 mL methanol.
- Blend 2 min at high speed. Pour mixture through powder funnel containing glass wool plug into 2 L separator.
- Add 250 mL methylene chloride and shake 30 sec. Add 700 mL water, 10 mL 10% sulfuric acid, and 35 mL saturated sodium chloride solution. Shake separator 1 min and let emulsion settle.
- Drain remaining emulsion and methylene chloride into 500 mL centrifuge bottle and centrifuge 5 min at 1500 rpm. Siphon off and discard water layer.
- Pour methylene chloride layer through funnel containing glass wool plug into 250 mL separator. Let stand ≥30 min to ensure complete separation of any remaining water. Drain and discard any water and emulsion from separator.

- Transfer methylene chloride layer to 250 mL graduate and record volume.
- Transfer measured volume of methylene chloride to K-D fitted with 10 mL receiving flask. Add boiling chips and concentrate extract on steam bath to about 5 mL. Dilute to 10 mL with hexane and mix.
- Clean up extract by C1; equivalent weight of product cleaned up is:

$$50\ \text{g} \times \frac{\text{mL methylene chloride recovered}}{250\ \text{mL}} \times \frac{\text{mL loaded onto GPC}}{10\ \text{mL}}$$

E6 DISSOLUTION IN METHYLENE CHLORIDE/HEXANE

Reference

Hopper, M.L. *et al.* (1992) *J. AOAC Int.* **75**, 707-713

Principle

No actual extraction of residues is done for vegetable oils; instead, they are dissolved in solvent for subsequent cleanup on GPC.

Reagents

hexane, distilled from all-glass apparatus

methylene chloride, distilled from all-glass apparatus

50% (v/v) methylene chloride/hexane

Directions

For each batch of samples analyzed, fortify commodity with chlorophenoxy acid herbicide(s) and pentachlorophenol and analyze it with others to verify adequacy of recoveries.

- Weigh 16 g pure vegetable oil into 100 mL volumetric flask. Dilute to volume with 50% methylene chloride/hexane (0.16 g/mL).
- Clean up oil solution by C1; equivalent weight of product cleaned up is:

$$\frac{16\text{ g}}{100\text{ mL}} \times \text{mL loaded onto GPC}$$

E7 EXTRACTION WITH ACIDIFIED METHYLENE CHLORIDE

Reference

Hopper, M.L. *et al.* (1992) *J. AOAC Int.* **75**, 707-713

Principle

Residues in water are extracted with methylene chloride after acidification.

Apparatus

Kuderna-Danish concentrator (K-D), 500 mL, with Snyder column, two-ball micro Snyder column, 10 mL graduated receiving flask

separatory funnel (separator), 1 L

Reagents

acetone, distilled from all-glass apparatus

methylene chloride, distilled from all-glass apparatus

10% sulfuric acid, reagent grade

Directions

For each batch of samples analyzed, fortify commodity with chlorophenoxy acid herbicide(s) and pentachlorophenol and analyze it with others to verify adequacy of recoveries.

- Weigh 500 g water, transfer to 1 L separator. Add 10 mL 10% sulfuric acid and 60 mL methylene chloride. Shake vigorously 1 min.
- Allow layers to separate and drain methylene chloride layer into K-D with 10 mL receiving flask. Repeat extraction with two 60 mL portions methylene chloride. Combine all extracts.
- Add boiling chips and evaporate to near dryness on steam bath. Add 50 mL acetone and evaporate to about 3 mL.
- No GPC cleanup is necessary. Proceed to methylation, C1b. Entire solution (therefore, entire weight of original product) is methylated.

C1 GPC CLEANUP, METHYLATION, AND FLORISIL COLUMN CLEANUP

C1a GPC CLEANUP

Reference

Hopper, M.L. (1982) *J. Agric. Food Chem.* **30**, 1038-1041

Principles

Co-extractives in the extract are separated from residues on GPC by molecular size exclusion; larger molecules (fats, *etc.*) elute first and are discarded. Advance calibration of the GPC column dictates the optimum amount of eluate to discard in order to remove most large molecule co-extractives and recover as much residue as possible. Once calibrated, the column can be used repeatedly.

Apparatus

filtration device for solutions, 10 mL syringe with Luer-Lok tip, fitted with either (a) 13 mm diameter Swinny stainless steel filter holder and 13 mm diameter filters, 5.0 µm LS-type, or (b) disposable membrane filters, 25 mm diameter, 5 µm Teflon membrane, encased in polypropylene. (Pre-assembled devices that do not require a syringe are also available.)

GPC apparatus; automated equipment optional but recommended. GPC apparatus must include:

1) sample introduction valve
2) pump, low pressure, suitable for use with organic solvents, capable of 5 mL/min flow
3) sample loading loop, 1/16" Teflon tubing coiled in cylindrical form, about 13 mL capacity
4) pulse dampener, about 6' of 1/8" copper tubing coiled and closed at one end, installed between pump and sample introduction valve with a connecting tee. Pulse dampener is needed only when pump is not pulseless.

GPC column, glass, 25 mm id × 300 or 500 mm with organic solvent plunger kit

GPC syringe, 10 mL syringe with Luer-Lok tip, with Millipore Swinny stainless steel adapter, Millipore 5.0 µm LS-type filter

Kuderna-Danish concentrator (K-D), 500 mL, with Snyder column, two-ball micro Snyder column, 5 and 10 mL volumetric or graduated receiving flasks

Reagents

Bio-Beads SX-3 resin, 200-400 mesh (Bio-Rad Laboratories, Richmond, CA; pretested resin is available from ABC Laboratories)

hexane, distilled from all-glass apparatus

methylene chloride, distilled from all-glass apparatus

eluant: 1+1 (v/v) methylene chloride/hexane

Preparation of GPC column

- Weigh 35 g Bio-Beads SX-3 into 400 mL beaker.
- Add 150 mL 50% methylene chloride/hexane.
- Stir beads with glass or steel rod until all beads have swelled and lumps are no longer present.
- Pour slurry into GPC column with aid of stirring rod.
- Hold column in upright position with plunger tightened about 25 mm from bottom end of usable length of column, ignoring threaded ends.
- Add slurry to column continuously so beads never become completely settled until all beads have been added.
- Place other plunger in column after beads have settled and liquid has drained completely.
- Compress each plunger an equal distance from its respective end until bed length is about 200 mm.
- Connect column to GPC solvent delivery system, and pump solvent from bottom to top of column until all air is expelled.
- Adjust flow rate of system to 5 mL/min and check column pressure. Adjust operating pressure for column to 8-11 psig by moving plunger(s).
- Allow GPC system to equilibrate by pumping solvent through it.
- Re-adjust flow rate to 5 mL/min if it has changed.

Calibration of GPC column

Elution of Fat

- Melt butter and decant butterfat and solids through fluted filter paper into suitable container; do not include water layer. Heat funnel if necessary to facilitate filtration.
- Weigh 4 g warm, filtered butterfat into 25 mL g-s graduate; dilute to 25 mL with 50% methylene chloride/hexane; mix until fat is dissolved (0.16 g fat/mL).
- Filter fat solution through filtration device and load 5 mL onto GPC column.
- Elute with 50% methylene chloride/hexane.
- Collect column effluent in tared beakers in 10 mL increments from 0 to 100 mL.
- Evaporate solvent from each beaker, cool, and weigh to calculate amount of fat eluted in each 10 mL increment. (For manual GPC, collect 10 mL fractions in separate graduates and transfer to tared beakers for evaporation and calculation of fat.)
- 95% of fat should elute in first 60 mL. If >5% of fat appears in 60-70 mL fraction or later, reject column and prepare new one by repacking with original batch of beads. Visual evaluation of yellow band of fat as it passes through column usually shows tailing or streaking when column is inadequate. Use new batch of beads if second column is still inadequate.

Elution of Pesticides

- Prepare mixed standard solution containing 1.2 µg ethion/mL, 0.4 µg diazinon/mL, 0.4 µg heptachlor epoxide/mL, 0.2 µg dicloran/mL, and 0.6 µg dieldrin/mL in 50% methylene chloride/hexane.
- Place 5 mL aliquot standard solution in K-D with 10 mL receiving flask; add 50 mL hexane and 2-3 boiling chips; concentrate to 10 mL. This removes methylene chloride prior to determinative step.
- Filter mixed standard solution through filtration device and load 5 mL onto GPC column.
- Elute with 50% methylene chloride/hexane.
- Collect 10 mL fractions from 0 through 160 mL.
- Transfer each fraction to K-D with 10 mL receiving flask and add 50 mL hexane and 2-3 boiling chips; concentrate to 10 mL.
- Calculate recoveries by comparison to mixed standard solution that has also undergone concentration to 10 mL. Use determinative steps DG1 and DG2.
- Column is normal if diazinon and ethion start to elute in either 50-60 mL or 60-70 mL fraction, and dicloran starts to elute in 90-100 mL fraction.
- Determine what volume should be discarded (usually first 60 mL) and what should be collected (usually 60-160 mL fraction) by examining fat and mixed standard elution profiles developed above. Use these calibrated fraction volumes in subsequent calibration steps and in sample cleanup.

Elution of Pesticides from Fat

- Weigh 1.6 g warm, filtered butterfat into tared 10 mL g-s graduate, add 5 mL mixed standard solution from pesticide elution test above, dilute to volume with 50% methylene chloride/hexane, and mix until fat is dissolved.
- Filter fortified fat solution through filtration device and load 5 mL onto GPC column.
- Elute with 160 mL 50% methylene chloride/hexane.
- Discard and collect respective volumes determined during calibration.
- Transfer collected fraction to K-D with 10 mL receiving flask and concentrate as in pesticide elution test.
- Calculate recoveries as in pesticide elution test.
- For normal column, ≥80% diazinon, parathion, and ethion, and ≥95% of organochlorine pesticides are recovered.

Elution of Herbicides

- Prepare mixed standard solution of 0.1 µg 2,4,5-T/mL and 0.05 µg pentachlorophenol/mL in 50% methylene chloride/hexane by diluting acetone stock solutions.
- Load 5 mL onto GPC column and elute with 160 mL 50% methylene chloride/hexane.
- Discard and collect respective volumes determined above.

- Transfer collected fraction to K-D with 5 mL receiving flask.
- Add 2-3 boiling chips and concentrate.
- Cool, add 50 mL acetone and fresh boiling chips, and reconcentrate to about 3 mL.
- Cool solution and methylate as in C1b.
- Clean up methylated solution on Florisil as in C1c and concentrate to 5 mL.
- Calculate amount of herbicide in concentrated extract using determinative step DG1 and reference standards of pentachlorophenol methyl ether and 2,4,5-T methyl ester. Convert amount of methylated chemicals to amount of corresponding acid/phenol by multiplying respective values by 0.95, which represents ratio of molecular weights of acid/phenol to methylated chemical for both 2,4,5-T and pentachlorophenol (255.49/269.52 and 266.35/280.37, respectively). Calculate percentage recovered by comparing converted values to amounts added (0.5 µg 2,4,5-T and 0.25 µg pentachlorophenol).
- Gel column is acceptable if ≥80% of added 2,4,5-T and pentachlorophenol are recovered.

Directions

- Use GPC column prepared and calibrated as described above. Column can be used repeatedly.
- Centrifuge cloudy solutions of extract from E1-E6 before loading them onto GPC. Filter all solutions through filtration device before GPC.
- Load filtered sample extract onto GPC column in 5 mL loops. About 0.8 g fat or 15-50 g equivalent weight of nonfatty product can be loaded in each loop. Use original sample weight, aliquots taken during extraction, volume of final extract, and loading loop size to calculate amount being loaded.
- If targeted limit of quantitation is lower than can be achieved with this size aliquot, load additional aliquots of extract in separate 5 mL loops and combine concentrated eluates from each after GPC cleanup.
- Elute column with 160 mL 50% methylene chloride/hexane. Discard volume previously calibrated and collect remainder in beaker or graduate. Transfer collected eluate to K-D fitted with 10 mL receiving flask. Rinse collection vessel with several mL acetone and add to concentrator.
- Add 2-3 boiling chips and concentrate to about 3 mL. Cool, add 50 mL acetone and fresh boiling chips, and reconcentrate to about 1 mL. Use micro-Snyder column to reach final volume if necessary. Methylate cleaned up extract by procedure in C1b.

C1b METHYLATION

Reference

Hopper, M.L. (1987) *J. Agric. Food Chem.* **35**, 265-269

Principles

Acids and phenols in cleaned up extract solution are ionized by an alkali (tetrabutylammonium hydroxide) and methylated with methyl iodide to their respective esters and ethers.

Apparatus

graduated cylinder (graduate), 10 mL, g-s

microliter syringes: 25, 50, or 100 µL, for adding reagents

water bath, capable of maintaining 40° C

Reagents

acetone, distilled from all-glass apparatus

hexane, distilled from all-glass apparatus

methyl iodide, certified grade

tetrabutylammonium hydroxide (TBAH) titrant, 1.0 M in methanol

Cautions

Note the following cautions that must be observed during methylation:

Use well ventilated hood and protective gloves when adding reagents for methylation.

With each batch of samples, also methylate aliquot of same mixed standard solution of chlorophenoxy acids and pentachlorophenol used for fortification in above recovery tests. Clean up methylated standard on Florisil column.

Determine completeness of methylation by calculations using primary standards of chlorophenoxy acid methyl esters and pentachlorophenyl methyl ether (*i.e.*, not esters/ether generated by this methylation step). If methylation reaction appears incomplete by test, prepare fresh mixed standard solution of acids and pentachlorophenol (these solutions have been found susceptible to degradation).

Methylate fresh mixed standard and old mixed standard, and clean up both by Florisil chromatography. Determine degree of methylation in both old and new standards as above.

The comparison between degrees of methylation will indicate whether problem is caused by standard or by methylation step. If low recoveries are not repeated with new standards, problem can be assumed to be with old standards. If recoveries of old and new standards are both poor (*i.e.*, consistently <40% or >120%), methylation step is at fault.

Presence of water may prevent complete methylation.

Directions

- Dilute concentrated extract from GPC, C1a, to 3 mL with acetone.
- Add 80 µL 1.0 M TBAH/methanol and 40 µL methyl iodide. Immediately stopper tube.
- Mix, then place stoppered tube in 40° C water bath for 1.5 hr, with water level of bath above fluid level in tube.
- Remove tube from water bath and attach to 250 mL K-D. Add 50 mL hexane and boiling chips. Evaporate to about 1 mL (avoiding dryness).
- Dilute to appropriate volume with hexane, add 2 mL distilled water, and shake stoppered tube. Discard water.
- Clean up methylated extract on Florisil, C1c.

C1c FLORISIL COLUMN CLEANUP

Reference

Griffitt, K.R. *et al.* (Feb. 1983) "Miniaturized Florisil Cleanup of Chlorophenoxy Acid Herbicides and Pentachlorophenol in Total Diet Samples," LIB 2695, FDA, Rockville, MD

Principle

Solution of methylated residues is cleaned up by adsorption chromatography on Florisil column.

Apparatus

chromatographic column, 10 mm id × 300 mm, Teflon stopcock, coarse porosity fritted disc

Kuderna-Danish concentrator (K-D), 125 or 250 mL, with Snyder column, graduated or volumetric receiving flask

Reagents

acetonitrile, distilled from all-glass apparatus; see Section 204 for distillation directions

boiling chips, carborundum, 20 mesh, or other suitable boiling chips

ethyl ether, distilled from all-glass apparatus, with 2% ethanol as preservative; see Section 204 for peroxide test

Florisil, PR grade; see Section 204 for handling and testing directions and calculation of lauric acid (LA) value

hexane, distilled from all-glass apparatus

methylene chloride, distilled from all-glass apparatus

sodium sulfate, anhydrous, granular, reagent grade; see Section 204 for handling directions

eluants: 1—20% methylene chloride/hexane (v/v). Dilute 200 mL methylene chloride with hexane. Allow mixture to reach room temperature, and adjust volume to 1 L with hexane.

2—50% methylene chloride/0.35% acetonitrile/49.65% hexane (v/v/v). Pipet 3.5 mL acetonitrile into 500 mL methylene chloride and dilute with hexane. Allow mixture to reach room temperature and adjust to 1 L with hexane.

Florisil Check

For each batch of Florisil, verify that weight calculated from LA value is suitable by this test.

- Place activated Florisil (weight = 110/LA value × 4 g) in chromatographic column; add about 2 cm sodium sulfate.
- Add 5 mL mixed standard solution of 0.1 µg 2,4,5-T methyl ester/mL, 0.05 µg pentachlorophenyl methyl ether/mL, and 0.2 µg picloram methyl ester/mL.
- Elute as directed below, concentrate eluates to 5 mL, and examine by DG1.
- Calculate recoveries by comparison to original mixed standard solution.
- Elute column further with about 100 mL ethyl ether and collect in 10 mL fractions.
- Concentrate each fraction to 5 mL, and examine each by DG1.
- Determine volume of ethyl ether needed to elute picloram methyl ester from column and record for each batch of Florisil.
- Pentachlorophenyl methyl ether should elute in eluate 1; 2,4,5-T methyl ester (and methyl esters of other chlorophenoxy acids) should elute in eluate 2. Picloram methyl ester should elute in about 100 mL ethyl ether.
- If test chemicals do not elute as expected, test adjusted weights of Florisil (similar to tests described in Section 204) or use different batch of Florisil.

Directions

- Add appropriate weight of Florisil (determined above) to 10 mm id × 300 mm column; add about 2 cm sodium sulfate.
- Prewash column with 15 mL hexane. Do not allow column to go to dryness.
- Place K-D fitted with appropriate receiving flask under column.
- Quantitatively transfer methylated extract from C1b to column. Rinse flask with hexane and add to column. Extract and rinse volume together should be ≤15 mL.
- With stopcock completely open, elute column with 35 mL eluant 1.
- Change K-Ds and elute column with 60 mL eluant 2.
- To analyze for picloram, change K-Ds and elute column with appropriate volume of ethyl ether determined above.
- Add boiling chips to K-Ds and concentrate each eluate to suitable definite volume. When volume <5 mL is needed, use two-ball micro-Snyder or micro-Vigreaux column during evaporation.

- Add 50 mL hexane to K-D containing Eluate 2 and reconcentrate to remove final traces of acetonitrile. Add 50 mL hexane to K-D containing ethyl ether eluate and reconcentrate.

DETERMINATION

Determine methylated residues in concentrated solution from C1 with determinative steps DG1, DG3, and DG4 (see Section 302).

Chlorinated residues are determined using DG1 or DG3; residues containing nitrogen may be determined using DG4.

Inject volume of concentrated extract equivalent to the following weights, based on whole product:

	Products ≥20% fat	*Products <20% fat*
Eluate 1	≤5 mg	≤10 mg
Eluate 2	≤10 mg	≤20 mg
Ethyl ether	≤10 mg	≤20 mg

CONFIRMATION

Confirm tentatively identified residues according to the principles discussed in Section 103. Review PESTDATA (Appendix I) to find GLC systems applicable to residue; rechromatograph on other systems if available.

403: METHOD FOR PHENYLUREA HERBICIDES

BASIC REFERENCE

Luchtefeld, R.G. (1987) *J. Assoc. Off. Anal. Chem.* **70**, 740-745

GENERAL PRINCIPLES

Phenylurea herbicide residues are extracted with methanol. The extract is cleaned up by partitioning and column chromatography on a Florisil column. Residues are selectively determined with an HPLC system consisting of a reverse phase (RP) column, post-column photodegradation and derivatization, and detection of the resultant derivative by fluorescence detection.

APPLICABILITY

Consult Guide to PAM I for additional information pertinent to the appropriate application of multiresidue methodology.

Method is applicable to residues of phenylurea herbicides in nonfatty foods. See Table 403-a, following method description, for pesticides tested through the method.

Limits of detection and quantitation for selected compounds are also included in Table 403-a; method is applicable to determination of at least 14 phenylurea herbicides at concentrations of 0.05, 0.5, and 1.0 ppm in 14 nonfatty food products.

REFERENCE STANDARDS

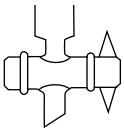
Prepare stock solutions (1 mg/mL) of each standard in HPLC grade isopropyl alcohol. Combine appropriate amounts of stock standard solutions and further dilute with (1+1) acetonitrile/HPLC grade water for working standards. Use standards at concentration of 1.25 µg/mL for HPLC determination. Keep stock solutions refrigerated prior to use and prepare working standards weekly.

STEPS OF THE METHOD

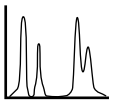
Extraction (E)			**Recommended Use**
E1	(p. 403-3)	Extraction with methanol	nonfatty foods
Cleanup (C)			
C1	(p. 403-5)	Liquid-liquid partitioning and Florisil column cleanup	nonfatty foods
Determination (D)			
DL3	(p. 403-7)	HPLC, post-column photolysis and derivatization, fluorescence detection	phenylureas
DL4	(p. 403-10)	HPLC, different mobile phase	alternative to DL3, confirmation of identity

Figure 403-a
Method for Phenylureas

nonfatty foods
E1



C1



DL3 and DL4
phenylurea
residues

VALIDATION

The following combination has undergone interlaboratory validation and is recommended for use:

E1 + C1 + DL3

Validation report:

Luchtefeld, R.G. (May 1989) "Validation of a Multiresidue Procedure for Detemining Phenylurea Herbicides," LIB 3309, FDA, Rockville, MD

E1 EXTRACTION WITH METHANOL

Reference

Luchtefeld, R.G. (1987) *J. Assoc. Off. Anal. Chem.* **70**, 740-745

Principles

Residues of phenylurea herbicides are extracted from nonfatty foods with methanol, and the methanol solution is separated from food solids by centrifugation.

Apparatus

centrifuge, explosion-proof, to hold 500 mL bottles

centrifuge bottle, glass, 500 mL. Use glass stopper or cover rubber stopper with aluminum foil to avoid contamination.

funnel, glass

graduated cylinder (graduate), 250 mL

homogenizer, Polytron Model PT 10-35, with PT 35K generator containing knives, head equipped with metal (not Teflon) bushing

Reagents

glass wool, Pyrex

methanol, distilled from all-glass apparatus

Directions

- Weigh 50 g sample into centrifuge bottle. Add 100 mL methanol and homogenize mixture 1.5 min using Polytron with speed set at 8.
- Centrifuge homogenate 5 min at 1500 rpm and decant 80-100 mL through funnel fitted with glass wool plug into 250 mL graduate.
- Add 100 mL methanol to material in centrifuge bottle and homogenize 1.0 min with speed set at 8.
- Centrifuge homogenate and decant as much liquid as possible through funnel into same 250 mL graduate.
- Record volume of combined methanol extract.

C1 LIQUID-LIQUID PARTITIONING AND FLORISIL COLUMN CLEANUP

Reference

Luchtefeld, R.G. (1987) *J. Assoc. Off. Anal. Chem.* **70**, 740-745

Principles

Co-extractives are removed by adding sodium chloride to the methanol extract and partitioning with hexane, which is discarded. Residues are partitioned from methanol into methylene chloride. Concentrated methylene chloride extract is cleaned up on a Florisil column, and residues are eluted with acetone/methylene chloride.

Apparatus

chromatographic column, 10 mm id × 300 mm, Teflon stopcock, coarse porosity fritted disc

chromatographic column, 25 mm id, plain

Kuderna-Danish concentrators (K-D), 250 and 500 mL, with Snyder column and 5 mL graduated receiving flasks

separatory funnel (separator), 500 mL and 1 L

Reagents

acetone, distilled from all-glass apparatus

acetonitrile, HPLC grade

boiling chips, 20-30 mesh carborundum

Florisil, PR grade; see Section 204 for handling and testing directions

hexane, distilled from all-glass apparatus

methylene chloride, distilled from all-glass apparatus

sodium chloride, reagent grade

sodium chloride aqueous solution, saturated

sodium sulfate, anhydrous, granular, reagent grade; see Section 204 for handling directions

eluant: 20% (v/v) acetone/methylene chloride

1+1 (v/v) acetonitrile/HPLC grade water

Directions

Partitioning

- Transfer entire methanol extract (E1) to 500 mL separator.
- Add 30 mL saturated sodium chloride solution and 50 mL hexane, and shake mixture 30 sec. Let layers separate.
- Transfer lower aqueous layer to 1 L separator containing 500 mL water and 30 mL saturated sodium chloride solution. Discard hexane.
- Add 200 mL methylene chloride to the 1 L separator and shake 1 min.
- Drain methylene chloride through 25 mm × 50 mm column of sodium sulfate into 500 mL K-D fitted with 5 mL receiving flask.

- Re-extract aqueous phase with two 100 mL portions methylene chloride. Drain each extract through sodium sulfate into K-D.
- Rinse sodium sulfate with 50 mL methylene chloride; add rinse to K-D.
- Add boiling chip to K-D and evaporate combined extracts and rinse to about 3 mL for transfer to Florisil column.

Florisil Cleanup

- Place 4 g activated Florisil in 10 mm id chromatographic column; add 1 cm sodium sulfate. Wash column with 30 mL methylene chloride and discard wash.
- When methylene chloride reaches top of sodium sulfate, place 250 mL K-D with 5 mL graduated receiving flask under column. Transfer concentrated extract to column. Rinse container with about 3 mL methylene chloride and add rinse to column.
- When extract reaches top of sodium sulfate, rinse column with two 3 mL portions methylene chloride; allow each rinse to reach top of sodium sulfate.
- When final rinse reaches top of sodium sulfate, elute column with 50 mL 20% acetone/methylene chloride.
- Concentrate solution on steam bath to about 3 mL. Remove receiving flask and evaporate to dryness, using stream of nitrogen and water bath at 40° C.
- Pipet 2 mL (1+1) acetonitrile/HPLC grade water into receiving flask.
- Calculate sample equivalent in final cleaned up extract according to following formula:

$$\frac{\text{g sample}}{\text{mL extract}} = \frac{\text{mL methanol filtrate collected}}{200\ \text{mL} + (50\ \text{g} \times \%\ \text{water in sample}) - 5} \times \frac{50\ \text{g}}{2\ \text{mL final extract}}$$

DL3 HPLC, POST-COLUMN PHOTOLYSIS AND DERIVATIZATION, FLUORESCENCE DETECTION

Reference

Luchtefeld, R.G. (1985) *J. Chromatogr. Sci.* **23**, 516-520

Principles

Residues in acetonitrile/water solution are separated on a C-18 RP HPLC column using methanol/water gradient mobile phase. Residues eluting from the column are photolyzed in-line to primary amines by exposure to UV light; the flow's passage through a sleeve of Teflon tubing increases exposure to UV light. The amines are reacted in-line with o-phthalaldehyde and 2-mercaptoethanol to form fluorophores that are measured by a fluorescence detector. The determinative step is very selective for phenylureas.

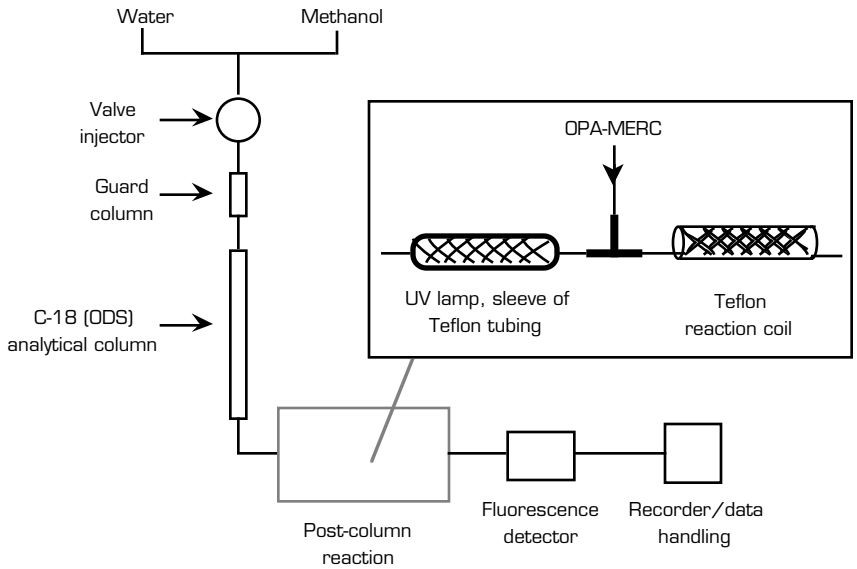
Apparatus

filtration device for solutions, 10 mL syringe with Luer-Lok tip, fitted with either (a) 13 mm diameter Swinny stainless steel filter holder and 13 mm diameter filters, 5.0 µm LS-type, or (b) disposable membrane filters, 13 mm diameter, 0.22 µm nylon membrane, encased in polypropylene. (Preassembled devices that do not require a syringe are also available.)

HPLC apparatus (Figure 403-b) must meet system suitability test below. Complete system consists of:

1) mobile phase delivery system: dual pump gradient system
2) injector with 40 µL valve loop injector
3) guard column, direct connect cartridge system containing prepacked C-18 cartridge (Alltech Associates, Deerfield, IL 60015, Cat. No. 28013)
4) column oven or heater, to maintain analytical column at constant temperature

Figure 403-b
HPLC System for Determination of Phenylurea Herbicides



5) analytical column, 25 cm × 4.6 mm id, containing spherical particles with monomeric bonded layer of octadecylsilane (ODS, C-18); *e.g.*, Econosphere C-18, Alltech Associates

6) post-column photolysis and derivatization unit as shown in telescoped portion of Figure 403-a. Unit is assembled from:

   a) UV lamp for photodegradation, 17 cm × 9 mm od (Model 80-1178-01) with power supply (Model 90-0001-01), BHK Inc., Pomona, CA

   b) Teflon sleeve for photodegradation lamp, 10′ × 0.5 mm id delay coil (Cat. No. 5-9206, Supelco, Inc., Supelco Park, Bellefonte, PA). Place coiled Teflon tubing over lamp and connect one end to mixing tee and other end to column.

   c) low flow-rate pump for OPA-MERC solution (Model 396-31, LDC/Milton Roy, Riviera Beach, FL). Connect cone-shaped coil of 13′ × 1/8" od stainless steel tubing as pulse dampener between pump and 0.5 mm id Teflon tubing, which is connected to tee.

   d) mixing tee, stainless steel, 0.25 mm bore, 1/16" standard fittings (No. ZT1C, Valco Instruments, Inc., Houston, TX)

   e) reaction coil, 10′ × 0.5 mm id delay coil (Supelco; Cat. No. 5-9206), with one end connected to mixing tee and other end to detector

7) fluorescence detector

Reagents

acetic acid, glacial, reagent grade

2-mercaptoethanol (MERC), reagent grade. Prepare stock solution by diluting 10 mL MERC to 100 mL with methanol.

methanol, HPLC grade. Before use, degas in glass bottles by helium sparging or other suitable method.

monobasic potassium phosphate, certified ACS grade. Prepare 0.002 M solution by dissolving 0.27 g in 1 L HPLC grade water.

o-phthalaldehyde (OPA), reagent grade. Prepare stock solution by dissolving 300 mg OPA in 100 mL methanol.

sodium borate buffer solution. Dissolve 19.1 g ACS grade sodium tetraborate decahydrate ($Na_2B_4O_7 \bullet 10\ H_2O$) in approximately 500 mL HPLC grade water. Dilute to 1 L with HPLC grade water and adjust pH to 10.5 with sodium hydroxide, using pH meter. Filter through filtration device.

water, HPLC grade, commercial product or prepared from water purification equipment that produces distilled, de-ionized water. For HPLC, degas water as described for acetonitrile. Water must be adequately purified to prevent plugging HPLC column and extraneous peaks in chromatograms. All water used in HPLC procedure must be HPLC grade. ("Water" that does not specify HPLC grade means distilled water.)

OPA-MERC solution. Transfer about 250 mL degassed sodium borate buffer solution to 500 mL volumetric flask. Add 25 mL OPA stock solution and 5 mL MERC stock solution. Dilute to volume with borate buffer solution with mixing.

System Operation

- Operate column overnight with 0.3 mL/min (90+10) 0.002 M monobasic potassium phosphate/methanol mobile phase, using single or dual pump system. Change mobile phase to 10% methanol/HPLC grade water and operate 30 min at 1.0 mL/min. If chromatogram baselines begin to drift after several days of use, repeat this procedure.
- Maintain analytical column at 35° C. Equilibrate system with 40% methanol/HPLC grade water at 1.0 mL/min, with 0.2 mL/min OPA-MERC solution added through mixing tee. Allow detector to stabilize after starting OPA-MERC solution flow and turning on UV photodegradation lamp.
- After injecting sample or standard, begin 30 min linear gradient from 40% methanol/HPLC grade water to 80% methanol/HPLC grade water. Operate detector at excitation wavelength of 340 nm and emission wavelength of 455 nm. Adjust detector sensitivity to obtain 50% recorder or integrator deflection when 40 µL 1.0 µg/mL diuron solution is injected. Diuron elutes in approximately 24 min under these conditions.
- Flush pump and tubing used for addition of OPA-MERC solution daily after use by pumping 3% glacial acetic acid in water through system at 1.0 mL/min for about 20 min.
- After each day's use, rinse HPLC column 20 min with 80% methanol/HPLC grade water. Column may be stored in this mobile phase.

System Suitability Test

See Chapter 6, HPLC, for further information about evaluating HPLC systems.

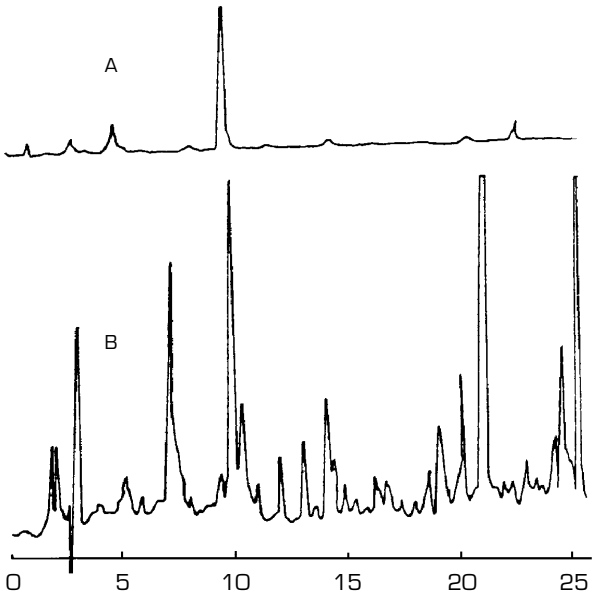
- Prepare solution containing 1 µg/mL each of metobromuron and diuron.
- Chromatograph solution three times. Retention times are approximately 22 and 24 min for metobromuron and diuron, respectively.
- Determine relative standard deviation (RSD) (standard deviation/mean) of peak heights measured in three chromatograms; also determine resolution between the two peaks, according to formulas in Figure 602-a.
- RSD is <3% and resolution will be ≥1.5 on adequate system.

Directions

Figure 403-c compares chromatogram from this system to one from system using UV detection at 245 nm.

- Filter both acetonitrile/water sample extract, C1, and reference standard solution(s) through filtration device; inject 40 µL filtrate into HPLC system.
- Compare detector responses (peak height is preferred) to sample extract and to reference standard using same chromatographic conditions.
- Use (1+1) acetonitrile/HPLC grade water to make further dilutions of sample extract, as necessary to make peak heights of sample and standard match closely.

Figure 403-c
HPLC Chromatograms of Carrot Extract



(A) HPLC determinative step, Section 403 DL3; (B) Detection by UV detector at 245 nm. Large peak in chromatogram A is caused by interference from carrot sample.

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ALTERNATIVE:

DL4 HPLC, DIFFERENT MOBILE PHASE

Reference

Luchtefeld, R.G. (1985) *J. Assoc. Off. Anal. Chem.* **70**, 740-745

Principles

HPLC chromatographic pattern for phenylureas is changed by using a gradient mobile phase of acetonitrile/water instead of methanol/water. Differences provide useful confirmatory evidence of residue identity.

Additional Reagent

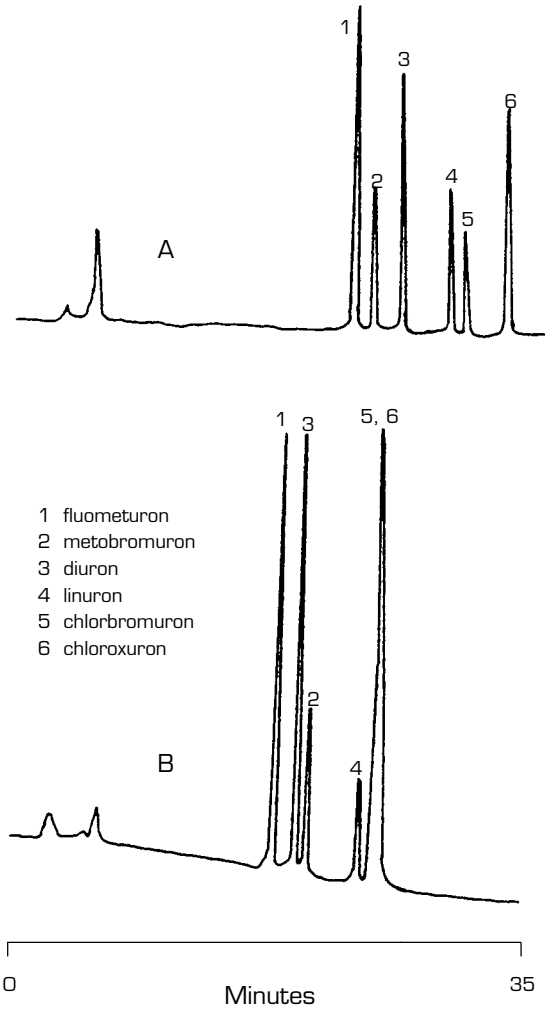
acetonitrile, HPLC grade. Before use, degas in glass bottles by helium sparging or other suitable method.

Directions

Figure 403-d demonstrates different chromatographic patterns produced by DL3 and DL4. Table 403-a lists retention times (relative to diuron) for 14 phenylurea herbicides on the two systems.

- Set up and operate HPLC system as in DL3, except use 30 min linear gradient from 30% acetonitrile/HPLC grade water to 80% acetonitrile/HPLC grade water as mobile phase. All other operations remain the same.

Figure 403-d
Chromatographic Pattern Comparison



Chromatography of six phenylurea herbicides on Econosphere ODS column, 25 cm × 4.6 mm id with two different mobile phases. [A] linear gradient from 40-80% methanol/water in 30 min; 1 mL/min flow rate. [B] linear gradient from 30-80% acetonitrile/water in 30 min; 1 mL/min flow rate.

[Reprinted with permission of Association of Official Analytical Chemists, from *J. Assoc. Off. Anal. Chem.* (1987) **70**, 740-745, Figure 5, page 743.]

CONFIRMATION

Confirm tentatively identified residues according to the principles discussed in Section 103. Rechromatograph on whichever HPLC system, DL3 or DL4, was not used in original determination.

404: METHOD FOR BENZIMIDAZOLES

BASIC REFERENCE

Gilvydis, D.M., and Walters, S.M. (1990) *J. Assoc. Off. Anal. Chem.* **73**, 753-761

GENERAL PRINCIPLES

Residues are extracted with methanol and partitioned into methylene chloride after initial acidification and again after subsequent alkalinization of the extract. Residues are separated and quantitated by reverse phase (RP) ion pair HPLC with both UV and fluorescence detectors.

APPLICABILITY

Consult Guide to PAM I to find additional information pertinent to appropriate application of multiresidue methodology.

Method is applicable to fruits and vegetables and determines residues of allophanate, MBC (resulting from use of benomyl, carbendazim, or thiophanate-methyl), thiabendazole, and thiophanate-methyl. UV detector responds to all residues to which the method is applicable, but fluorescence detector responds only to MBC and thiabendazole. Alternative determinative step permits examination for residues in commodities from which interfering materials are co-extracted. Method variations applicable to coffee beans are designed for determination of MBC only.

Method limit of quantitation is 0.1 ppm for each residue. UV detection is nonselective and prone to crop interferences, so limit of quantitation may be affected by the particular commodity. Limit of quantitation for thiabendazole can be increased to 0.01 ppm by use of a fluorescence detector at conditions of maximum absorbance for the compound.

See Table 404-a, following method description, for results of recovery tests.

REFERENCE STANDARDS

Obtain reference standards from repository or commercial sources.

Prepare stock solutions (25 µg/mL) in acetone of each of following: allophanate, MBC, thiabendazole, and thiophanate-methyl. (Do not use benomyl as reference standard, because overnight standing is required to ensure complete decomposition to MBC.) Solutions are stable in acetone and in HPLC mobile phase.

Prepare mixed standard solution by combining 1 mL of each stock standard solution, evaporate to dryness with gentle heat (30-40° C) under stream of nitrogen, dissolve residue in 4.0 mL methanol, add 6.0 mL HPLC ion pairing solution, and mix to give final concentration of 2.5 µg/mL of each standard.

STEPS OF THE METHOD

Choose from these method options:

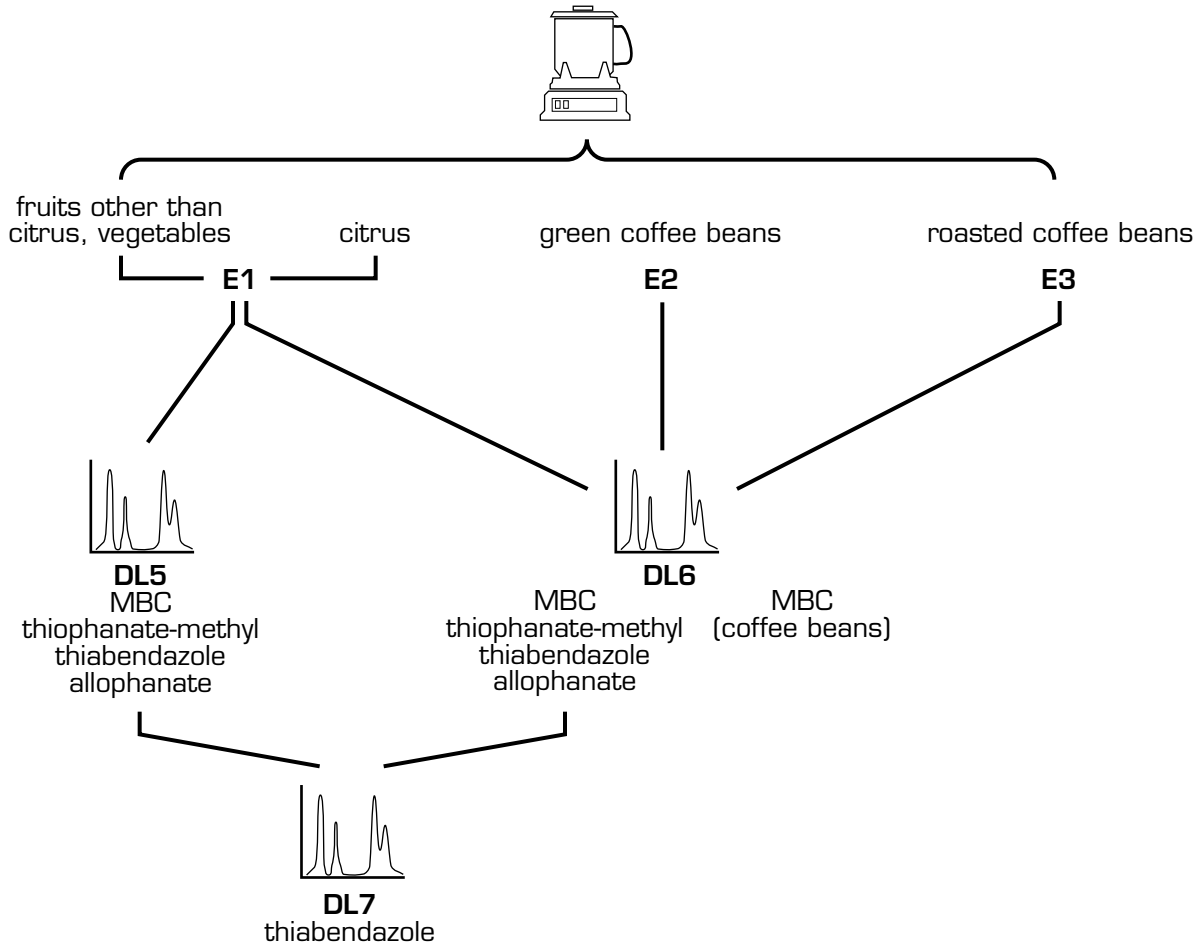
Extraction (E)		**Recommended Use**
E1 (p. 404-5)	extraction with methanol, transfer to methylene chloride	fruits and vegetables
E2 (p. 404-7)	extraction with methanol, removal of oil with hexane, transfer to methylene chloride	green coffee beans

E3	(p. 404-8)	extraction with methanol, removal of oil with methylene chloride, transfer to methylene chloride	roasted coffee beans

Cleanup: There are no traditional cleanup steps in this method.

Determination (D)			**Recommended Use**
DL5	(p. 404-9)	HPLC, 4.1 mM ion pairing reagent in mobile phase, UV and fluorescence detection	fruits and vegetables
DL6	(p. 404-12)	HPLC, 32.7 mM ion pairing reagent in mobile phase, UV and fluorescence detection	citrus, coffee beans (green and roasted)
DL7	(p. 404-14)	HPLC, 4.1 mM ion pairing reagent in mobile phase, changes in detector settings	thiabendazole

Figure 404-a
Method for Benzimidazoles



VALIDATION

Several combinations of method options are possible. The following combinations have undergone interlaboratory validation and are recommended for use:

E1 + DL5

Validation report:

Gilvydis, D.M. (July 1990) Quarterly Report on methods research, FDA internal communication

E2 + DL6

Validation report:

Jacobs, R.M., and Yess, N.J. (1993) *Food Addit. and Contam.* **10**, 575-577

E3 + DL5

Validation report:

Roy, R.R. (1993) FDA private communication of results (applied to many different commodities) from Interagency Agreement No. FDA 224-90-2479, work performed by USDA National Monitoring and Residue Analysis Laboratory, Gulfport, MS

E1 EXTRACTION WITH METHANOL, TRANSFER TO METHYLENE CHLORIDE

Reference

Gilvydis, D.M., and Walters, S.M. (1990) *J. Assoc. Off. Anal. Chem.* **73**, 753-761

Principle

Residues are extracted with methanol and partitioned into methylene chloride after initial acidification and again after subsequent alkalinization of the extract.

Apparatus

Buchner funnel (Buchner), porcelain, 12 cm diameter

evaporator, vacuum rotary

filter paper, sharkskin, to fit Buchner funnel

flask, round-bottom (r-b), 1 L

separatory funnel (separator), 500 mL

shaker, mechanical, Burrell wrist action

vacuum filtration flask, 1 L

Reagents

All solvents must be suitable for liquid chromatography and spectrophotometry and must be filtered through 0.2 µm filter.

hydrochloric acid, reagent grade, 1 M

methanol, distilled from all-glass apparatus

methylene chloride, distilled from all-glass apparatus

sodium chloride, 1% aqueous solution

sodium hydroxide, 5 M and 1 M solutions

sodium sulfate, anhydrous, granular, reagent grade; see Section 204 for handling directions

Directions

- Weigh 50 g finely chopped sample into 500 mL glass-stoppered (g-s) Erlenmeyer flask. Add 100 mL methanol and shake 10 min on mechanical shaker. To analyze bananas (whole or pulp only), shake vigorously by hand prior to mechanical shaking to disperse mass.
- Filter contents with suction through Buchner fitted with filter paper into vacuum filtration flask. Rinse Erlenmeyer and filter cake with additional 50 mL methanol.
- Transfer filtrate to 500 mL separator, and add 10 mL 1 M hydrochloric acid and 100 mL 1% sodium chloride solution; for low moisture crops, *e.g.*, wheat grain, use 150 mL 1% sodium chloride solution. Mix and allow to cool.
- Extract aqueous filtrate with two 100 mL portions methylene chloride, shaking vigorously 1 min each time.

- Dry methylene chloride layers through column of about 70 g sodium sulfate, collecting in r-b flask. Save aqueous methanol layer in separator for later extractions.
- Rinse sodium sulfate with about 50 mL methylene chloride and collect in r-b flask.
- Evaporate methylene chloride in r-b flask just to dryness using vacuum rotary evaporator with ≤30° C water bath.
- Dissolve material in r-b flask in 4 mL methanol. This extract contains residues of allophanate and thiophanate-methyl and some MBC and thiabendazole.
- Determine residues in this extract with DL5, except use DL6 for citrus. Avoid delay in determination to minimize loss of thiophanate-methyl by degradation.
- Drain aqueous methanol phase from separator into beaker. Rinse separator with two 10 mL portions water, and add rinsings to beaker.
- Adjust pH of solution in beaker to 7.5-8 with 5 M and 1 M sodium hydroxide solutions and 1 M hydrochloric acid, as necessary. Do not allow solution to become strongly alkaline during adjustment.
- Return solution to separator and extract with two 100 mL portions methylene chloride, shaking vigorously 1 min each time.
- Dry methylene chloride layers through column of about 70 g sodium sulfate, collecting in r-b flask. (Sodium sulfate column used to dry previous methylene chloride layer, above, may be reused.) Rinse sodium sulfate with about 50 mL methylene chloride and collect in r-b flask.
- Evaporate combined methylene chloride extracts to dryness in vacuum rotary evaporator. Dissolve material in r-b flask with 4 mL methanol. Most MBC and thiabendazole are recovered in this extract.
- Determine residues with DL5, except use DL6 for citrus.

E2 EXTRACTION WITH METHANOL, REMOVAL OF OIL WITH HEXANE, TRANSFER TO METHYLENE CHLORIDE

Reference

Gilvydis, D.M., and Walters, S.M. (Aug. 1989) "Modification of LIB 3217 for Carbendazim (MBC) in Green and Roasted Coffee Beans," LIB 3353, FDA, Rockville, MD

Principles

Residues are extracted from green coffee beans with methanol and the filtrate is acidified. Oils are removed from acidified extract by partitioning into hexane, which is discarded. Aqueous methanol extract is then made alkaline, and residues are extracted into methylene chloride by partitioning. Only MBC is targeted by this analysis, so extraction from acidified filtrate (done in E1) is not performed.

Apparatus

Buchner funnel (Buchner), porcelain, 12 cm diameter

evaporator, vacuum rotary

filter paper, sharkskin, to fit Buchner funnel

flask, round-bottom (r-b), 1 L

separatory funnel (separator), 500 mL

shaker, mechanical, Burrell wrist action

vacuum filtration flask, 1 L

Reagents

All solvents must be suitable for liquid chromatography and spectrophotometry and must be filtered through 0.2 µm filter.

hexane, distilled from all-glass apparatus

hexane, methanol-saturated

hydrochloric acid, reagent grade, 1 M

methanol, distilled from all-glass apparatus

methylene chloride, distilled from all-glass apparatus

sodium chloride, 1% aqueous solution

sodium hydroxide, 5 M and 1 M solutions

sodium sulfate, anhydrous, granular, reagent grade; see Section 204 for handling directions

Directions

- Grind coffee beans, using centrifugal mill, to pass 20-mesh screen.
- Weigh 50 g prepared sample into 500 mL g-s Erlenmeyer flask. Add 100 mL methanol and shake 10 min on mechanical shaker.
- Filter contents with suction through filter paper in Buchner and rinse with additional 50 mL methanol. Transfer filtrate to 500 mL separator.

- Add to filtered extract in separator 10 mL 1 M hydrochloric acid and 150 mL 1% sodium chloride solution. Mix and allow to cool.
- Extract acidified methanol once with 100 mL methanol-saturated hexane. Drain lower aqueous layer into beaker and discard hexane.
- Adjust pH of solution in beaker to 7.5-8 with 5 M and 1 M sodium hydroxide solutions and 1 M hydrochloric acid, as necessary. Do not allow solution to become strongly alkaline during adjustment.
- Return solution to separator and extract with two 100 mL portions methylene chloride, shaking vigorously 1 min each time.
- Dry methylene chloride layers through column of about 70 g sodium sulfate, collecting in r-b flask. Rinse sodium sulfate with about 50 mL methylene chloride and collect in r-b flask.
- Evaporate combined methylene chloride extracts to dryness in vacuum rotary evaporator. Dissolve material in r-b flask with 4 mL methanol. Residues of MBC will be in solution. Determine with DL6.

ALTERNATIVE:

E3 EXTRACTION WITH METHANOL, REMOVAL OF OIL WITH METHYLENE CHLORIDE, TRANSFER TO METHYLENE CHLORIDE

Reference

Gilvydis, D.M., and Walters, S.M. (Aug. 1989) "Modification of LIB 3217 for Carbendazim (MBC) in Green and Roasted Coffee Beans," LIB 3353, FDA, Rockville, MD

Principle

Roasted coffee beans contain more oil than green coffee beans, so methylene chloride, rather than hexane, is used to remove oil.

Directions

- Extract as in E2, except after acidification of filtered extract and cooling of mixture:
- Extract acidified methanol once with 100 mL methylene chloride, shaking vigorously 1 min. Drain lower aqueous layer into beaker and discard methylene chloride.
- Continue, as in E2, by making solution alkaline.

DL5 HPLC, ION PAIR MOBILE PHASE, UV AND FLUORESCENCE DETECTOR

Reference

Gilvydis, D.M., and Walters, S.M. (1990) *J. Assoc. Off. Anal. Chem.* **73**, 753-761

Principles

Benzimidazole residues are chromatographed on RP HPLC system. MBC and thiabendazole are ionized in acidic mobile phase and paired with negatively charged counter-ions from sodium decanesulfonate to control system selectivity and separate analytes from interfering sample co-extractives. Thiophanate-methyl and allophonate, neutral at the conditions in the column, are unaffected by mobile phase modifications and chromatograph according to RP principles.

UV and fluorescence detectors each respond to MBC and thiabendazole; UV detector also responds to thiophanate-methyl and allophanate.

Apparatus

Filtration device for ion pairing solution: stainless steel glass filter holder, 300 mL capacity, fitted with hydrophilic membrane of pH range 2-10, 0.45 µm pore size, 47 mm diameter. Use with vacuum filtration flask, 1 L.

Filtration device for sample solutions, 10 mL syringe with Luer-Lok tip, fitted with nylon 13 mm diameter disposable filter unit, 0.45 µm pore size.

HPLC system must meet system suitability test below. Complete system consists of:

1) mobile phase delivery system, constant volume isocratic pump
2) injector, automatic sample injection module, preferably with loop volume ≥25 µL
3) analytical column, 25 cm × 4.6 mm id, containing highly end-capped 5 µm C-18 bonded silica suitable for chromatography of basic compounds
4) guard column, compatible with analytical column, packed with same or comparable C-18 bonded silica
5) column oven, with configuration that accommodates guard and analytical columns
6) variable wavelength UV detector or photodiode array detector, equipped with flow cell of about 8 µL
7) fluorescence detector, dual monochromator, equipped with 5 µL flow cell
8) recorder, strip chart recorder or computing integrator compatible with each detector

Reagents

1-decanesulfonate, sodium salt, 98% pure

ion pairing solution, 4.1 mM 1-decanesulfonate, sodium salt. Pipet 7.0 mL phosphoric acid into 200 mL HPLC grade water; dissolve 1.0 g 1-decanesulfonate, sodium salt in this mixture. Pipet 10.0 mL triethylamine into solution and dilute to 1 L with HPLC grade water. Filter through <1 µm porosity membrane. (pH of solution should be about 2.4.)

methanol, distilled from all-glass apparatus

mobile phase, prepared by mixing *manually* 65 parts ion pairing solution with 35 parts methanol. (Do not mix mobile phase using pump, because exothermic reaction caused by mixing generates bubbles that prevent stable pump operation.) Before use, degas by sparging with helium while stirring solvent with magnetic stirrer.

phosphoric acid, 85%

triethylamine, 99% pure

water, HPLC grade, commercial product or prepared from water purification system that produces distilled, de-ionized water. Resistivity of water must be >12 megohms-cm.

System Operation

- Connect fluorescence detector in tandem with (following) UV detector.
- Set column oven temperature at 40° C and equilibrate system with mobile phase at flow rate of 1.5 mL/min for ≥30 min or until constant retention times are achieved for all four analytes.
- Rinse system with 50% methanol/water when not using for extended period (*e.g.*, ≥24 hr). When not in use for shorter periods, maintain slow flow (0.1-0.2 mL/min) of mobile phase to prevent salt deposition.
- To rid column of highly retained sample components as necessary, rinse column with methanol after rinsing with 50% methanol/water; then rinse with 50% methanol/water again before introducing mobile phase. Mobile phase must be rinsed from column with aqueous solvent before adding methanol and *vice versa* to avoid salt precipitation and possible clogging of system.
- Operate UV detector initially at absorbance wavelength of 250 nm; immediately following elution of allophanate, reset wavelength to 280 nm and rezero baseline for determination of thiophanate-methyl, MBC, and thiabendazole. Adjust detector and/or recorder sensitivity so that 40-70% full scale deflection (FSD) is obtained for 62.5 ng of each standard (25 µL mixed standard solution).
- Operate fluorescence detector at excitation wavelength of 280 nm (20 nm slit width) and emission wavelength of 310 nm (10 nm slit width). Adjust detector, attenuator, and/or recorder sensitivity so that 60-80% FSD is obtained for 62.5 ng MBC (25 µL mixed standard solution); at these conditions, 62.5 ng thiabendazole will cause 40-50% FSD.
- Alternatively, use wavelength-programmable fluorescence detector and wavelength-programmable or diode array UV detector for concurrent determinations of residues at optimum wavelength settings for each.

System Suitability Test

See Chapter 6, HPLC, for further information about evaluating HPLC systems.

- Prepare mixed standard solution (2.5 ng/µL for each compound) as directed above. Prepare additional dilutions to produce mixed standard solutions of 4 ng/µL and 0.4 ng/µL.
- Allow HPLC system to equilibrate at conditions described in System Operation.

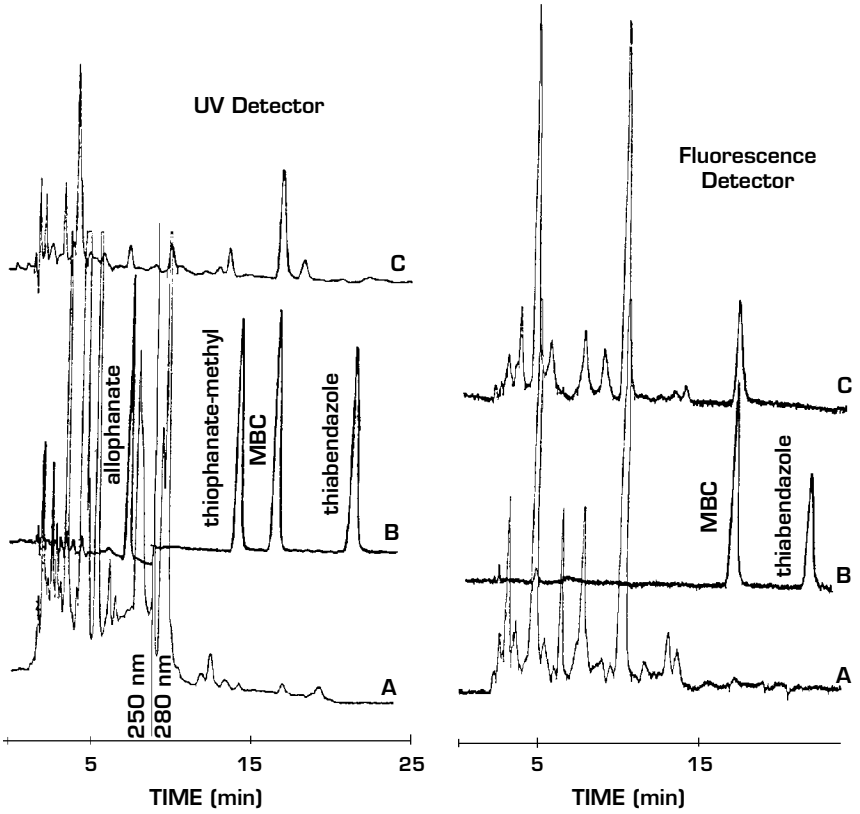
- Inject at least three 25 µL portions 2.5 ng/µL mixed standard solution. Determine following parameters:
  1) retention time and peak height for each peak; relative standard deviations (RSD) for repetitive retention times and peak height measurements
  2) column efficiency (N) for thiabendazole peak
  3) asymmetry factor (As) for thiabendazole peak
- Inject each of three different concentrations of mixed standard solutions (10-100 ng/25 µL injection). Plot peak height vs. amount injected.
- HPLC systems adequate for analysis for benzimidazoles will meet following minimum criteria: retention times of about 8, 15, 18, and 23 min for allophanate, thiophanate-methyl, MBC, and thiabendazole, respectively (retention times may vary among columns but should remain constant for particular column); RSD <0.5% for retention times and <3% for peak heights of individual peaks in three consecutive chromatograms; N>12,000 and As <1.3 for thiabendazole peak.
- Examine systems not meeting these criteria for problems, using various troubleshooting sections of Chapter 6. Correct problems uncovered by troubleshooting until system meets criteria defined above.
- System will typically respond linearly to 10-100 ng of each compound, but linear range may vary among systems. Perform quantitative analyses only within calculated linear range of system as determined above. Dilute sample extracts as needed to permit injection of analyte level within linear range. Adjust amounts injected so that peak heights of analyte and reference standard do not differ >25% from one another.

Directions

See Figure 404-b for typical chromatograms produced by HPLC system.

- To extract from E1-E3 (dissolved in 4.0 mL methanol), add 6.0 mL ion pairing solution; mix. Residue *must* be dissolved in methanol prior to adding ion pairing solution.
- Filter through 0.45 µm porosity membrane; filter will plug as solution is applied, so filter only volume needed for HPLC determination, about 1 mL.
- Inject 25 µL sample solution and chromatograph as described in System Operation.
- Compare chromatographic response (peak retention times, heights, and/or areas) with that of standard solution and calculate residue amount.
- If further dilutions are necessary, use mixture of 4:6 methanol:ion pairing solution as diluent.
- To convert calculated MBC (MW 191.2) to equivalent benomyl (MW 290.4), multiply by 1.52.
- To convert calculated MBC to equivalent thiophanate-methyl (MW 342.4), multiply by 1.79.
- Peaks of 50% FSD at conditions established for screening analysis are equivalent to about 0.5 ppm each of thiophanate-methyl, allophanate, and thiabendazole; MBC peak of 50% FSD at these conditions represents about 0.3 ppm.

Figure 404-b
Chromatograms of Benzimidazole Compounds



Chromatograms of: (A) peach extract partitioned from the acidic phase of 404 E1, (B) standard solution, (C) peach extract partitioned from basic phase of 404 E1. HPLC operation as directed in DL5. Sample contains 0.14 ppm field-incurred MBC.

ALTERNATIVES:

DL6 HPLC, CONCENTRATED ION PAIR MOBILE PHASE, UV AND FLUORESCENCE DETECTOR

Reference

Gilvydis, D.M., and Walters, S.M. (Aug. 1989) "Modification of LIB 3217 for Carbendazim (MBC) in Green and Roasted Coffee Beans," LIB 3353, FDA, Rockville, MD

Principles

Concentration of ion pairing reagent is increased eight times to increase k′ values of analytes and improve separation from early eluting co-extractives.

Additional Reagents

ion pairing solution, 32.7 mM 1-decanesulfonate, sodium salt. Pipet 7.0 mL phosphoric acid into 200 mL HPLC grade water; dissolve 8.0 g 1-decanesulfonate, sodium salt in this mixture. Pipet 10.0 mL triethylamine into solution and dilute to 1 L with HPLC grade water. Filter through <1 µm porosity membrane. (pH of solution should be about 2.4.)

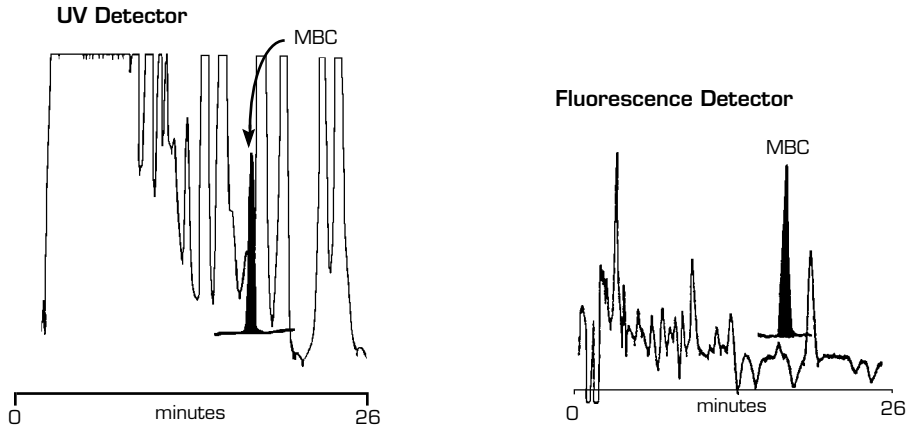
mobile phase, prepared by mixing *manually* 63 parts ion pairing solution with 37 parts methanol. Do not mix using pump. Before use, degas by sparging with helium while stirring solvent with magnetic stirrer.

Directions

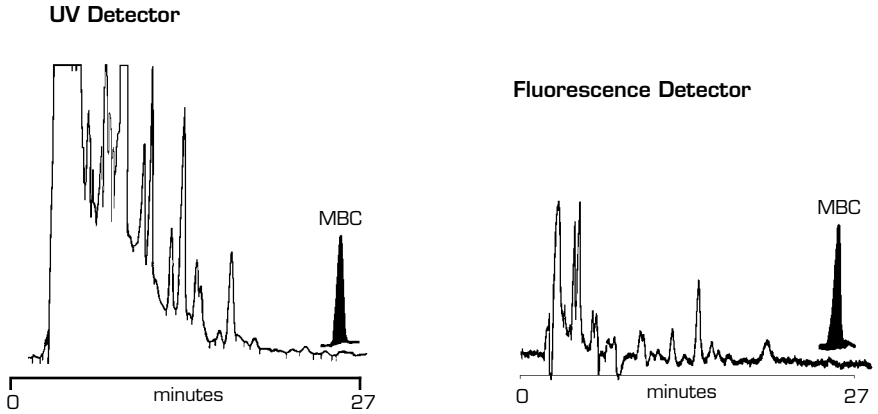
See Figure 404-c for depiction of improvements provided by higher concentration of ion pairing solution in mobile phase.

- Perform HPLC determination as in DL5, except use ion pairing solution and mobile phase described here. At these conditions, MBC will elute in about 26 min.

Figure 404-c
Effect of Ion Pairing Solution Concentration on Chromatography of MBC



HPLC operated as directed in DL5, 4.1 mM 1-decanesulfonate, sodium salt



HPLC operated as directed in DL6, 32.7 mM 1-decanesulfonate, sodium salt

Chromatograms of extract from green coffee beans, extracted according to 404 E2, and chromatographed at conditions directed in DL5 and DL6. Superimposed chromatograms of MBC represent 0.3 ppm.

DL7 HPLC, ION PAIR MOBILE PHASE, CHANGES IN DETECTOR SETTINGS

Reference

Gilvydis, D.M., and Walters, S.M. (1990) *J. Assoc. Off. Anal. Chem.* **73**, 753-761

Principle

Changes in detector settings provide increased sensitivity to thiabendazole.

Directions

- Perform HPLC determination as in DL5, except adjust wavelength of UV detector absorbance to 305 nm and adjust excitation and emission wavelengths of fluorescence detector to 305 and 345 nm, respectively.
- Dilute sample extract containing thiabendazole and rechromatograph. UV detector response to thiabendazole at this setting will be about twice that obtained at 280 nm, and fluorescence detector response to thiabendazole will be about 10 times that obtained at 280 and 310 nm.

CONFIRMATION

See Section 103 for additional information about confirmation.

Confirm initial findings of residues by rechromatographing with alternative systems described here, as appropriate to residue(s) found.

Table 401-a: Recovery of Chemicals Through Method 401 (E1-E2 + C1 + DL1)
(methanol extraction, cleanup with partitioning and charcoal/Celite column, HPLC with post-column derivatization and fluorescence detection)

Chemical	Recovery[1]	Rrt[2]	ng[3]	Notes
2,3,5-trimethacarb	C			
3,4,5-trimethacarb	C			
3-hydroxycarbofuran	C	0.6	10	
3-hydroxymethyl-2,5-dimethyl= phenyl methylcarbamate	P (70%)			
3-hydroxymethyl-4,5-dimethyl= phenyl methylcarbamate	C			
3-ketocarbofuran	V (67-110%)	0.85	11	
4-hydroxymethyl-3,5-dimethyl= phenyl methylcarbamate	C			
aldicarb	C	0.83	14	
aldicarb sulfoxide	P (50-60%)	0.33	9	
aldoxycarb	C	0.4	9	
bendiocarb	C	1	10	
bufencarb	C	1.44	19	Major peak is listed.
butocarboxim	C	0.75	15	
carbaryl	C	1.06	7	
carbofuran	C	1	10	
dioxacarb	C	0.67	15	
ethiofencarb	P (70-82%)	1.1	15	Breaks down to 2 peaks; other rrt 0.5.
fenobucarb	C	1.47	10	
isoprocarb	C	1.13	8	
methiocarb	C	1.26	10	
methiocarb sulfone	C	0.79	11	
methiocarb sulfoxide	C	0.64	12	
methomyl	C	0.46	10	32% recovery from peanuts.
metolcarb	C	0.85	10	
oxamyl	C	0.44	10	
promecarb	C	1.31	10	

[1] Codes: C: complete (>80%); P: partial (50-80%); S: small (<50%); V: variable (approximate percentage when known); R: recovered but no quantitative information available; NR: not recovered.

[2] Retention time, relative to carbofuran, on C-8 column described in DL1.

[3] ng that cause 50% full scale deflection detector response in DL1.

Table 401-a: Recovery Through Method 401 (E1-E2 + C1 + DL1)

Chemical	Recovery[1]	Rrt[2]	ng[3]	Notes
propoxur	C	0.98	8	
thiodicarb	P (40-60%)	0.99	11	Recovery C if analytical breakdown product (methomyl) also measured.
XMC	C	1.06	10	

Table 401-b: Recovery of Chemicals Through Method 401 (E1-E2 + C1 + DL2)

(methanol extraction, cleanup with partitioning and charcoal/Celite column, HPLC with fluorescence detection)

Chemical	Recovery[1]	Rrt[2]	ng[3]	Notes[4]
carbaryl	C	1.06	3	Ex L 288, Em L 330.
carbofuran	C	1	90	Ex L 288, Em L 330.
CGA 161149	V (43-99%)	0.73	10	Ex L 288, Em L 330.
CGA 195654	S (15-132%)	0.57	300	Ex L 288, Em L 330.
dioxacarb	C	0.67	180	Ex L 265, Em L 294.
fluometuron	V (60-100%)	1.09	50	Ex L 288, Em L 330. Low level residues may be obscured by matrix interferences.
isoprocarb	C	1.13	370	Ex L 264, Em L 292.
naphthaleneacetamide	P (77%)	0.75	3	Ex L 288, Em L 320. For C rec., elute charcoal with additional 100 mL petr ether
napropamide	C	1.36	4	Ex L 288, Em L 330.
phosalone	C	1.7	90	Ex L 288, Em L 330.
phosalone oxygen analog	C	1.3	90	Ex L 288, Em L 330.
piperonyl butoxide	C	1.74	5	Ex 288, Em L 330.
propoxur	C	0.98	40	Ex L 276, Em L 300.

[1] Codes: C: complete (>80%); P: partial (50-80%); S: small (<50%); V: variable (approximate percentage when known); R: recovered but no quantitative information available; NR: not recovered.

[2] Retention time, relative to carbofuran, on C-8 column described in DL1/DL2.

[3] ng that cause 50% full scale deflection detector response in DL2.

[4] Excitation (Ex) and emission (Em) wavelengths found optimum for the chemical.

Table 402-a: Recovery of Chemicals Through Method 402 (E1-E7 + C1 + DG1 or DG3 or DG4)
(extraction from acidified mixture, GPC, methylation, and Florisil cleanup, determination by GLC)

Chemical	Recovery[1-3]	Notes[4,5]
2,3,5,6-tetrachlorotere= phthalic acid	E1: NR E2: NR	Methylated completely, but did not elute from GPC.
2,3,5-triiodobenzoic acid	E1: V (66-86%) E2: V (79-138%)	No ester reference standard.
2,3,6-TBA	E1: C E2: C	
2,3-dihydro-3,3-methyl-2-oxo-5-benzofuranyl methyl sulfonate	E1: NR E2: NR	Chemical did not methylate.
2,4,5-T	E1: P E2: P	79% mean recovery, 31% CV, n=270, nonfat and fat.
2,4-D	E1: P E2: P	72% mean recovery, 34% CV, n=186, nonfat and fat.
2,4-DB	E1: C E2: C	
2-hydroxy-2,3-dihydro-3,3-methyl-5-benzofuranyl methyl sulfonate	E1: NR E2: NR	Chemical did not methylate.
3,5,6-trichloro-2-pyridinol	NR	Some (<20%) recovered in 100mL ethyl ether.
3,5-dibromo-4-hydroxy= benzoic acid	S (0-42%)	
3-carboxy-5-ethoxy-1,2,4-thiadiazole	NR	Methyl ester not eluted from Florisil column.
3-chlorosulfonamide acid	NR	Complete recovery from Florisil only, 14% from GPC.
3-methyl-4-nitrophenol		Methyl ether completely eluted from Florisil, but only 30% from GPC.

[1] Codes: C: complete (>80%); P: partial (50-80%); S: small (<50%); V: variable (approximate percentage when known); R: recovered but no quantitative information available; NR: not recovered.

[2] Extraction module used during testing (*e.g.*, E1) is indicated with each result.

[3] Florisil eluted with Eluant 1 and Eluant 2 only; chemicals eluted in ethyl ether (EE) are considered NR through basic method as normally performed.

[4] Ester/ether elutes from Florisil with Eluant 2 unless otherwise noted.

[5] When no reference material available for ester/ether, recoveries calculated against acid/phenol methylated per method.

Table 402-a: Recovery Through 402 (E1-E7 + C1 + DG1 or DG3 or DG4)

Chemical	Recovery[1-3]	Notes[4,5]
4-chlorobenzoic acid	E1: S (27-66%) E2: S (2-76%)	Low temperature column needed to detect methyl ester.
4-CPA	E1: S (32-69%) E2: C	Chromatographs only on wide bore GLC. No ester reference standard.
6-chloropicolinic acid	NR	Methylates, but methyl ester does not elute from Florisil.
AC 263,222 ammonium salt	NR	Methyl ester not eluted from Florisil.
acifluorfen	E1: P (54-69%)	
alloxydim-sodium	E1: NR E2: NR	Does not methylate.
arsanilic acid		Compound did not methylate under method conditions.
benazolin	E1: NR E2: NR	28-32% recovered if Florisil eluted with additional 100 mL EE. Complete recovery if Florisil eluted with additional 100 mL EE.
bifenox	E1: C E2: C	Parent is methyl ether.
bromacil	E2: NR	Complete recovery if Florisil eluted with additional 100 mL EE.
bromofenoxim	E1: P (57-86%) E2: C	No ether reference standard.
bromoxynil	E1: P (50-68%) E2: C	No ether reference standard.
chloramben	E1: S (40-43%) E2: P (49-59%)	
chloroxuron	E1: NR E2: NR	Does not methylate.
clofencet potassium salt	NR	Does not methylate.
cloprop	E1: P (50-66%) E2: C	Chromatographs only on wide bore GLC. No ester reference standard.

Table 402-a: Recovery Through 402 (E1-E7 + C1 + DG1 or DG3 or DG4)

Chemical	Recovery[1-3]	Notes[4,5]
CP 106070	NR	Does not methylate.
CP 106077	NR	Some methylation, but does not elute from GPC.
CP 108064	E1: NR	NR through method even in 100 mL EE; recovered from GPC only, Florisil only.
	E2: NR	Complete recovery if Florisil eluted with additional 100 mL EE.
CP 108669	NR	Some methylation, but does not elute from GPC.
CP 92429	NR	Does not methylate.
CP 95200	NR	Some methylation, but does not elute from GPC.
CP 97290	NR	Does not methylate.
cyclanilide	E1: C E2: V (45-67%)	
dicamba	E1: P (71-76%) E2: C	
dichlorprop	E1: C (80%) E2: C (72-104%)	No ester reference standard.
diclofop	E1: S (43-51%) E2: V (81-200%)	
dinoseb	E1: NR E2: NR	Does not methylate.
disul-Na	E1: NR	Does not methylate; parent 50% recovered if Florisil eluted with 100 mL EE.
	E2: NR	Does not methylate; complete recovery of parent with 100 mL EE.
DNOC	E1: S (45-50%)	Nitrogen detector required. No ether reference standard.
	E2: C	
dodine	E1: NR E2: NR	Does not methylate.
fenac	E1: C (74-92%) E2: C	No ester reference standard.
flumetsulam	E1: NR E2: NR	Methylated product not soluble in hexane.

Table 402-a: Recovery Through 402 (E1-E7 + C1 + DG1 or DG3 or DG4)

Chemical	Recovery[1-3]	Notes[4,5]
fluroxypyr	E1: S (23-30%)	Two peaks result; 3-7% more eluted with100 mL EE. No ester ref std.
	E2: P (64-77%)	Two peaks result; complete recovery with 100 mL EE. No ester ref std.
haloxyfop	E2: P (54%)	Florisil elution with 100 mL EE not tested.
HOE-038182	E1: NR E2: S (30-41%)	Methylation was complete, but ester not recovered. Elution from Florisil only with eluant #2 + 100 mL EE.
HOE-099730	NR	Does not methylate.
imazamox	NR	Methyl ester not eluted fromFlorisil.
ioxynil	E1: C (80-87%) E2: C	No ether reference standard.
iprodione urea	NR	Methyl ether not eluted from Florisil.
MCPA	E1: C (78-89%) E2: C	
MCPB	E1: C (70-106%)	Chromatographs only on wide bore GLC. No ether reference standard.
	E2: C	
mecoprop	E1: C (73-84%)	Wide bore GLC recommended. No ester reference standard.
	E2: C	
PB-7	E1: NR	Complete recovery if Florisil eluted with additional 100 mL EE.
	E2: NR	
pentachlorophenol	E1: P	70% mean recovery, 31% CV, n=275, nonfat and fat.
	E2: P	
picloram	E1: NR	6-10% recovered if Florisil eluted with additional 100 mL EE.
	E2: NR	Complete recovery if Florisil eluted with additional 100 mL EE.
PPG-947	E1: P (49-78%)	Two peaks from methylation; only one seen by halogen detector.
pyrithiobac-sodium	E1: S (7-13%)	Additional 31-34% recovered if Florisil eluted with 100 ml EE.

Table 402-a: Recovery Through 402 (E1-E7 + C1 + DG1 or DG3 or DG4)

Chemical	Recovery[1-3]	Notes[4,5]
RPA203328	NR	Small (0-34%) recovery in 100 mL ethyl ether.
silvex	E1: C E2: C	
triadimenol	E1: NR E2: NR	Methyl ether not eluted from Florisil.
triclopyr	E1: C E2: C	Recovery from fatty foods may be <50%.
vinclozolin metabolite B	E1: S (26-43%) E2: S (27-43%)	Methylated product is parent vinclozolin; 62% recovery through Florisil only.

Table 403-a: Recovery of Chemicals Through Method 403 (E1 + C1 + DL3 and DL4)
(methanol extraction, cleanup by partitioning and Florisil chromatography, HPLC with post-column photolysis and derivatization, fluorescence detection)

		DL3: Methanol/ Water Mobile Phase		DL4: Acetonitrile/ Water Mobile Phase	
Chemical	**Recovery[1]**	**Rrt[2]**	**Notes[3]**	**Rrt[2]**	**Notes[3]**
chlorbromuron	C	1.16	LD 0.006, LQ 0.022	1.28	LD 0.003, LQ 0.011
chlorotoluron	C	0.87		0.91	
chloroxuron	C	1.25	LD 0.002, LQ 0.008	1.28	LD 0.001, LQ 0.003
diuron	C	1	LD 0.002, LQ 0.007	1	LD 0.001, LQ 0.003
fenuron	C	0.42		0.49	
fluometuron	C	0.87	LD 0.002, LQ 0.006	0.93	LD 0.001, LQ 0.003
isoproturon	C	0.96		1	
linuron	C	1.12	LD 0.004, LQ 0.014	1.23	LD 0.005, LQ 0.017
metobromuron	C	0.91	LD 0.004, LQ 0.015	1.04	LD 0.004, LQ 0.014
metoxuron	C	0.62		0.67	
monolinuron	C	0.91		0.99	
monuron	C	0.72		0.75	
neburon	C	1.34		1.43	
siduron	C	1.08		1.16	

[1] Codes: C: complete (>80%); P: partial (50-80%); S: small (<50%); V: variable (approximate percentage when known); R: recovered but no quantitative information available; NR: not recovered.

[2] Retention time, relative to diuron, on the HPLC system described.

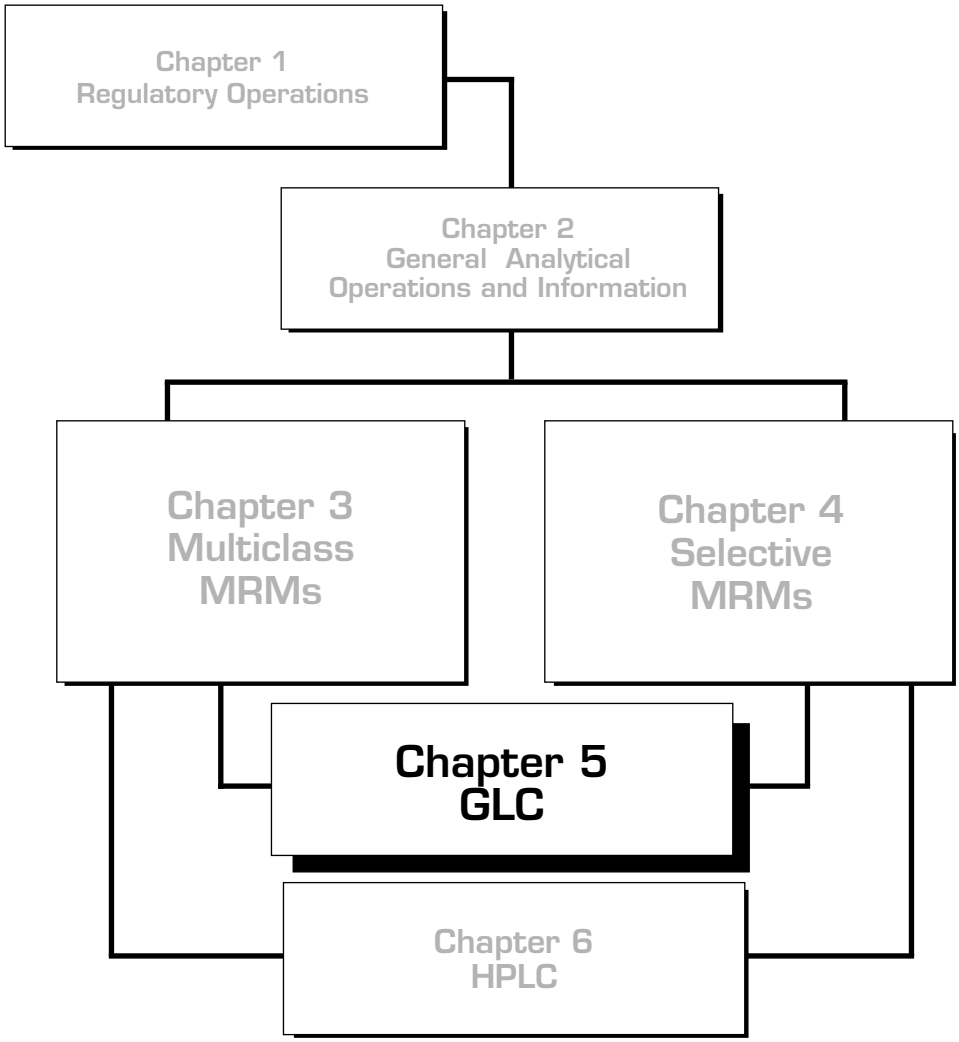
[3] LD, limit of detection: concentration (ppm) of phenylurea found to cause response three times baseline noise; LQ, limit of quantitation: concentration (ppm) found to cause response 10 times baseline noise.

Table 404-a: Recovery of Chemicals Through Method 404 (E1-E3 + DL5)
(methanol extraction, partitioning into methylene chloride, HPLC with ion pairing mobile phase and UV and fluorescence detection)

Chemical	Recovery[1]	Notes
allophanate	C	Determined by UV detector at 250 nm.
benomyl	C	Determined as MBC (carbendazim) by UV detector at 280 nm.
MBC[2]	C	Determined by UV detector at 280 nm.
thiabendazole	C	Determined by UV detector at 280 nm; confirm, increase sensitivity with DL7.
thiophanate-methyl	C	Determined by UV at 280 nm; degrades in extract, must be determined quickly.

[1] Codes: C: complete (>80%); P: partial (50-80%); S: small (<50%); V: variable (approximate percentage when known); R: recovered but no quantitative information available; NR: not recovered.

[2] Residue may result from use of: (1) benomyl, never found as a residue itself, because it is rapidly converted to MBC; (2) thiophanate-methyl, which degrades slowly to MBC; or (3) carbendazim, as MBC is called when used as a fungicide itself (not registered in the U.S.).



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501: GENERAL INFORMATION

Multiresidue methodology by definition requires determinative steps capable of separating analytes from one another so each can be detected and measured individually. Both gas-liquid chromatography (GLC) and high performance liquid chromatography (HPLC) provide these capabilities, and both are used in modern laboratories.

GLC has been the predominant determinative step in pesticide multiresidue methodology for over 30 years. Because GLC involves interaction between a vapor phase and liquid phase, its application is restricted to analytes that can be vaporized without degradation. For heat-labile chemicals, HPLC offers a variety of alternative schemes for separating analytes according to chemical or physical characteristics, but GLC's relative simplicity and ruggedness cause it to remain the determinative step of choice for residues to which it is applicable.

501 A: PRINCIPLES

Separation in GLC is achieved by differences in distribution of analytes between mobile and stationary phases, causing them to move through the column at different rates and from it at different times [1]. A measured aliquot of solution is injected into a gas chromatographic column through an inlet heated to a sufficiently high temperature that analytes are vaporized. In this state, the flow of inert gas that forms the mobile phase sweeps analytes through the column; retarding this movement is the analyte's solubilization in the liquid phase. During passage through the column, analytes that were injected in the same solution separate from one another because of their different vapor pressures and selective interactions with the liquid phase [2]. When analytes elute from the column and enter a detector, the detector responds to the presence of a specific element or functional group within the molecule. The detector's response causes a change in electronic signal, which is proportional to the amount of residue; the signal is amplified and recorded as a chromatogram.

Analytes are identified by the time it takes them to pass through a column of specific liquid phase (retention time), at a specified temperature and gas flow. Quantities are calculated from the detector response. Both retention time and response are compared to values obtained for a reference standard solution injected into the same system.

501 B: EQUIPMENT FOR GLC

Gas Chromatographic Components

The basic gas chromatograph consists of an inlet system, column, detector, electronic equipment to amplify the detector signal, and a recorder or other data-handling device. Carrier gas(es), with appropriate pneumatic system(s), are also integral to the GLC system. The inlet system, column, and detector are maintained in temperature-controlled environments.

The following are desirable features in GLC hardware:

1) Inlet, column oven, and detector should be individually heated and temperature-controlled. Temperature should be maintained to ±0.1° C.

Control of detector temperature usually is not as critical but should be well controlled, constant, and not affected by such things as line voltage fluctuations.

2) Temperature readout should be available for column, detector, and inlet. (Check accuracy of instrument temperature indicators with accurate pyrometer.)

3) Instrument design should be simple enough to facilitate troubleshooting and repairs. Design should permit easy removal or inspection of either column or detector without affecting the temperature of the other.

4) System should be designed to prevent or minimize contact between sample injection and any metal parts; system should be all-glass (or as near as possible).

   Several sizes of packed and open tubular capillary columns are used in residue analysis, and hardware for inlet and column must accommodate configurations that will be needed. Section 502, Columns, includes directions for adapting equipment.

5) Certain detectors may require multiple heated zones, including combustion furnaces. For flexibility, designs that permit ready access for servicing and maintenance are preferred. Section 503 provides details on various detectors used in pesticide residue determination.

6) Electrical signal monitoring equipment is usually one of two designs: (1) amplifier with 1 or 10 mV output, compatible with strip chart recorder, and (2) amplifier with 1 or 10 V output, compatible with data processing by either electronic integrator or computer. Other remote devices such as autosamplers can be easily adapted to any of these systems.

Other Apparatus

Gas Regulators. Two-stage gas pressure regulators with stainless steel diaphragms are required for all GLC determinations of trace residues. Regulators with a secondary stage maximum pressure of 80 psi are acceptable, but those with 200 psi offer more flexibility. If a hydrogen purifier is used (below), the latter type of regulator is required, because higher pressure is needed.

Gas lines that connect gas tanks to the chromatograph must be clean and free of components that contain oil or gas-purgeable elastomers; "refrigeration grade" copper (*i.e.*, cleaned of all oil) is preferred. Tubing (even refrigeration grade) should be sequentially rinsed with methylene chloride and acetone before use. Plastic and nylon lines must be avoided to reduce the likelihood of air contaminating the gas.

Syringes. The most common syringes for injection of food extracts into a chromatograph are 5 and 10 µL fixed needle syringes with 22° bevel points; some other sizes may be needed for special purposes. Hamilton syringes or equivalent are available from all chromatography suppliers. Plunger "guides" are available as options to minimize bending the plunger during injection.

Some specialty products exist to facilitate injection and minimize aggravation, and each has found favor with some analysts. For example, syringes with removable needles permit replacement of needles on which "burrs" have formed that destroy septa; removable needles with a "side port point" do not shred the septa as do standard bevel point needles; and syringes with plungers and needles made of a titanium alloy cannot be bent.

Reagents and Gases

Reagents associated with GLC include column liquid phases and solid supports, gases used for mobile phase and for detector reactions, and certain other reagents relevant to detector operation. Most of these reagents are discussed further in pertinent sections of this chapter; only gases, including filters used to remove contaminants from gas flow, are included in this introductory section.

Helium, hydrogen, and nitrogen are most commonly used as column carrier gases. Purity is always critical to avoid damage to the column, and more stringent purity requirements may be imposed by the detector. Purity specifications of the instrument manufacturer should always be followed.

Helium and hydrogen requirements range from 99.999-99.9999% purity, depending on the detector. Even with the highest purity, oxygen traps, available from chromatography suppliers, are recommended; traps that change color when permeated with oxygen are ideal for alerting the analyst to potential problems.

Purchase of ultra high purity helium and hydrogen may not be necessary if specially designed purifiers are used. Purifiers are available that permit use of commercial grade gases (99.995%) at a much lower price, justifying the cost of the purifier. Different purifiers are needed for helium and hydrogen; they are not interchangeable. FDA has had successful experiences with:

hydrogen purifiers: Model 560, AADCO Instruments, Inc., Clearwater, FL; Model 8372V, Consolidated Technologies, Inc., West Chester, PA

helium purifiers: Product # HP, Valco Instrument Co., Houston, TX; Model 2-3800, Supelco, Bellefonte, PA

Nitrogen is used as a carrier gas only for packed columns (Section 502 B). Either nitrogen or argon/methane (95+5 or 90+10) is also required as a carrier and/or makeup gas for the electron capture detector (Section 503 B). Commercial grades of these gases are acceptable if oxygen and moisture traps are used between the gas tank and the chromatograph.

501 C: RESIDUE METHODOLOGY FOR GLC DETERMINATION

Applications of analytical methodology require consideration of many factors to assure compatibility of method steps. The following factors related to extraction and cleanup of food samples profoundly influence accuracy and reliability of GLC determinative steps.

Cleanup

Solvent extraction of pesticide residues also extracts food constituents ("co-extractives") from the sample. Cleanup steps are included in residue analytical methods to remove co-extractives that can interfere in the determinative step of the analysis or cause damage to the column and/or detector.

For many years, predominant use of the nonselective electron capture (EC) detector caused justifiable concern about potential detector response to nonpesticidal co-extractives. In addition, documented cases in which sample co-extractives damaged GLC columns and caused subsequent breakdown of injected residues supported the need for extensive cleanup prior to GLC determination [3].

More recently, several factors have reduced emphasis on cleanup. The more selective GLC detectors now in use have decreased the likelihood that sample or reagent artifacts might be mistaken for pesticide residues. In addition, use of capillary columns, which are more efficient than equivalent packed columns, result in increased peak height response for the same amount of analyte. The amount of extract injected can thus be reduced without changing the level of quantitation, and this in turn reduces the likelihood of damage to the GLC system. Inlet liners and adapters used with capillary columns (Section 502 C) also provide the column with some degree of protection from damage caused by co-extractives. Finally, there are many incentives to perform more analyses with the same or fewer resources and to minimize the volume of solvents that must be purchased and disposed of. These factors contribute to a trend toward performing only minimal cleanup of sample extracts during routine surveillance analyses, with the intention of cleanup with applicable step(s) if an extract is found to contain interfering materials.

Despite these compelling reasons to reduce cleanup, GLC systems that are not protected from co-extractives deteriorate faster than those into which only cleaned up extracts are injected. The column and/or detector may be damaged by injection of insufficiently cleaned up samples, especially when the method and the chromatograph are used repeatedly. Such detrimental effects can occur even when the chromatogram appears to be clean enough for residue identification and measurement. Experience with a variety of sample types should make the analyst aware of these occurrences.

Detector response to sample co-extractives (artifacts) is still possible even with element-selective detectors. Although a selective detector is less likely to respond to chemically unrelated artifacts than the nonselective EC detector, artifacts containing an element to which the detector responds can still interfere with residue analysis. This occurs most often with nitrogen-selective detectors because of the number of nitrogenous chemicals in foods, but it can occur with any detector. Likelihood of interferences and potential for mistaken identity increase with decreasing cleanup.

Insufficiently clean extracts may also affect quantitative accuracy when determining residues that are polar or otherwise subject to adsorption by active sites in a GLC column. Such chemicals usually exhibit poor chromatography when standard solutions are injected, because adsorption delays or inhibits the chemical during its passage through the column. Peak tailing and/or changes in retention times are caused by adsorption. The net effect is an apparently diminished detector

response, which is especially evident if peak height measurements are used rather than peak area.

In contrast, when an uncleaned extract containing the same analyte is injected into the GLC system, co-extractives compete for the column's active sites, and the analyte moves through the column in a tighter chromatographic band. Analyte concentration (per unit time) entering the detector thus increases, and detector response (peak height) is greater. Quantitation by the usual practice (*i.e.*, comparison of detector responses to residue and reference standard) results in calculation of an inaccurately high residue level, especially if peak heights are compared. Quantitative accuracy can be improved for such chemicals by employing more rigorous cleanup of the extract or by using a GLC column with fewer active sites.

An appropriate balance is needed between efficiency in processing samples and accuracy in determining residues. Every injected extract should be sufficiently clean that it (1) does not jeopardize the column beyond the point that it can be easily repaired; (2) does not introduce substances that will degrade co-injected or subsequently injected residues; (3) does not foul any part of the detector, including combustion tube, flame, radioactive source, *etc.*; (4) minimizes introduction of artifacts to which the detector will respond; and (5) does not cause a disproportionate response enhancement of the residue in the extract.

Reagent Blanks

The analyst must ascertain that no interference from reagents and/or glassware occurs during residue analysis. Scrupulous attention is required to eliminate all such contaminants, and routine analysis of reagent blanks should be specified in the laboratory quality assurance plan (Section 206).

Contaminants can be introduced from a variety of sources. Studies with the EC detector have identified interferences from impure solvents, adsorbents, sodium sulfate, glass wool, Celite, blender gaskets, laboratory air filters, and polyethylene containers. The more nonselective the detector, the more likely it is to respond to interferences introduced by reagents or the environment. A thorough examination of the reagent blank is also necessary for methods that use a relatively selective detector. One example demonstrated that chemicals extracted by petroleum ether from a polyethylene squeeze bottle caused response by both an EC and a halogen-selective detector [3]. Contaminants can even be pesticides themselves, present on glassware or microliter syringes used in prior analyses, or present in the laboratory environment because of pest control treatment.

When interferences are discovered and the source(s) identified, every effort must be made to reduce or eliminate the problem. Solvents can be purchased to meet requirements or may be redistilled. Solids frequently can be washed and/or heated prior to use. Section 204 provides purity tests and procedures for purifying certain commonly used reagents; other reagent purification procedures are included in pertinent method descriptions in Chapters 3 and 4. Sometimes the method cleanup step removes interferences added to the sample during previous steps, but whether this is accomplished must be determined by a complete investigation of the method reagent blank.

Equipment should be washed thoroughly and rinsed with solvent as soon as possible after use. Syringe plungers and needles should be wiped with lint-free wipers

dipped in an appropriate solvent (*e.g.*, acetone), and the barrel should be cleaned by drawing solvent through the needle and out the top by a vacuum applied to the top. Particular care should be taken to assure elimination of residues from glassware or syringes previously in contact with high concentrations of pesticides.

Choice of Solvent

The solvent in which the final extract is dissolved must be compatible with the detector(s) in the GLC determinative step(s). The most basic requirement is that the solvent not contain elements to which the detector responds. Specifically, no amount of chlorinated solvent, such as methylene chloride, can remain in extracts being examined by an EC or halogen-selective detector, and no trace of acetonitrile can be present in extracts examined with nitrogen-selective detectors.

Other effects besides element selectivity cause incompatibility between detectors and solvents. For example, acetonitrile has an unexplained adverse effect on response of the EC detector, and aromatic and halogenated solvents may increase detector response of the N/P detector and eventually render it useless.

Solvent volatility must also be considered when using a detector that requires a solvent venting time. For these detectors, the most volatile practical solvent in which residues are soluble should be chosen to minimize length of venting time and avoid potential loss of early eluting analytes.

Solvent volatility has another practical effect related to the ease with which the extract can be concentrated. Final volume of concentrated extract must be sufficiently small that the volume injected into the GLC system contains sufficient equivalent sample weight necessary to reach the targeted level of quantitation (Section 105). Sensitivity of a particular detector to residues of interest governs how much sample equivalent must be injected, and column type and arrangement limit the volume that can be injected. In cases where a very small final extract volume is needed, or where the concentration step begins with a very large solvent volume, practicality dictates the choice of a volatile solvent to minimize time needed for concentration.

501 D: INJECTION TECHNIQUES

The technique used to inject extracts and reference standards into the chromatograph is critical to system performance. Improper syringe handling can lead to myriad problems, including asymmetrical peak shapes and nonreproducible retention times or responses. Autoinjectors are increasingly used for residue determination, but manual injection is still practiced.

Manual Injection

If extracts and standards are injected manually, it is imperative that each analyst develop and follow good technique in syringe handling and sample introduction. This can be achieved through practice and care. Several methods presently in use for filling syringes and injecting include:

1) A volume of solvent greater than or equal to needle volume is drawn into the syringe, followed by a small amount of air. The extract (or reference standard solution) is then drawn completely into the syringe barrel, where

its volume can be measured by reading both ends of the liquid. Injection is then made. The initial solvent flushes the extract or standard into the chromatograph. This technique is referred to as the "solvent flush" or "sandwich" technique.

2) The syringe is filled by drawing extract (or standard solution) completely into the barrel (*i.e.*, none is left in the needle). Total volume is measured by reading both ends of the liquid. Injection is made, with the syringe removed quickly from the inlet. The syringe plunger is withdrawn until whatever volume of liquid remains is completely in the barrel of the syringe, where it is measured as before. The difference in liquid volume before and after injection is the amount actually injected. It is important when using this technique to remove the syringe from the heated injection port as quickly as possible after injection to avoid any evaporation of liquid remaining in the syringe.

3) The syringe is filled to the desired volume, the volume noted, and the injection made. The volume measured is considered to be the volume injected. This technique introduces error, because it ignores the volume in the needle and the volume that remains after injection. The effective error can be minimized by use of the same solvent for both sample extract and standard solution and by injection of the same volume of each.

Whichever injection technique is chosen, it must be performed reproducibly. Each analyst should choose the injection technique he/she finds most reproducible and use it routinely. Poor precision among chromatograms from repetitive injections may be caused by faulty syringes or poor analyst technique, as well as by inappropriate solvents or inadequate sample cleanup. Volume of liquid in the syringe should be measured by holding the syringe in the same manner each time while looking toward a light background. The same injection technique must be used for both the sample extract and the reference standard to which it will be compared.

Choice of injection technique is not solely based on personal preference; type of column being used (packed *vs* capillary) must also be considered. Any technique described above can be applied when using packed columns. However, too much solvent can overwhelm the small diameter capillary column, so injection volume must be limited. Several inlet systems and injection options are used with capillary columns to accommodate both column restrictions and volume requirements of residue determination (Section 502 C). Consistently good capillary column results have been achieved with manual injection and the solvent flush technique. The syringe needle should remain in the inlet 1 sec for each μL injected to allow the pressure surge from vaporization of solvents to dissipate.

The syringe manufacturer's recommendations for use and care of the syringe should be followed. Syringes must be kept free of traces of analyte. This should be checked occasionally by injecting a volume of pure solvent; if the syringe is clean, no peaks other than the solvent peak will appear.

Autoinjectors

The best injection performance is achieved using an autoinjector (also called autosampler). Various commercially available autoinjectors can be interfaced with

GLC systems. For normal use of autoinjectors, extracts and standard solutions are placed in disposable glass vials with vapor-tight septum caps. The autoinjector wets the syringe completely and removes air bubbles by pumping extract (or standard solution) into the barrel. It then draws a precisely measured volume of solution into the barrel and injects it into the chromatograph. Between injections, the autoinjector flushes the needle with appropriate solvent to clean it. Beyond the improved reproducibility achieved with autoinjectors, their use permits unattended operation of the chromatograph and frees the chromatographer to perform other tasks.

501 E: REFERENCE STANDARDS

Section 205 provides information on pesticide standards. The importance of reliable standard solutions to accurate pesticide analyses cannot be overemphasized. Solvents used for GLC standard solutions are subject to the same requirements and limitations listed above for extracts.

The quality assurance plan for analyses involving GLC determination should include routine injection of a mixed standard solution. The mixture should include compounds normally used as markers for retention time and response and should also include compounds prone to adsorption or degradation. Vulnerable compounds serve as indicators of problems developing in the system; *e.g.*, the presence of p,p′-DDT in such a solution serves to alert the analyst when degradation to p,p′-TDE occurs. GLC systems used for determination of organophosphorus or other polar residues should be checked with a solution that includes, at a minimum, methamidophos, acephate, and monocrotophos. Response to acephate may disappear in systems that contain too much glass wool, and response to methamidophos may not be seen if it elutes with the solvent front or if column packing is of poor quality; both these situations can be avoided by monitoring the system with routine injection of an appropriate mixed standard. Frequency of injection of mixed standard, at least twice during an 8-hr period, should be specified in the quality assurance plan.

For best quantitative results, reference standards should be dissolved in the same solvent that is used for the final sample extract. In addition, reference standards should be injected within minutes of the sample containing the residue(s) to be quantitated, and responses to residue and standard should match within ±25% for accurate quantitation.

References

[1] *Standard Practice for Gas Chromatography Terms and Relationships*, ASTM E 355-77, reapproved 1983, ASTM, Philadelphia, PA

[2] Jennings, W. (1987) *Analytical Gas Chromatography*, Academic Press, Orlando, FL

[3] Burke, J.A., and Giuffrida, L.A. (1964) *J. Assoc. Off. Agric. Chem.* **47**, 326-342

502: COLUMNS

502 A: INTRODUCTION

Separations among analytes in GLC are achieved within the column. Although choice of detector dictates which class of analytes can be determined, individual detection and measurement of multiple analytes would not be possible without the separations provided by the column.

Columns are available in several different physical configurations, each of which offers advantages and disadvantages to pesticide residue determination. The two basic types of GLC columns currently used in pesticide residue determination are (1) packed columns, in which liquid phase is immobilized as a film on particles of fine mesh solid support and packed into 2-4 mm id columns, and (2) open tubular capillary columns, in which liquid phase is immobilized as a film on the interior walls of a capillary tube. Capillary columns are further distinguished by internal diameter: wide bore (0.53 mm id), traditional (0.25-0.32 mm id), and narrow bore (≤0.25 mm id). Each type of column requires unique hardware and operating parameters.

In all GLC columns, identity of the liquid (stationary) phase is the primary factor dictating what separations are achievable. Carrier gas (mobile phase) is also integral to GLC operation and must be included in any discussion of columns. However, only inert gases are used as carrier gases, so few options exist. Operating parameters that affect column efficiency, including column temperature and carrier gas identity and flow rate, provide additional variables that can be adjusted to achieve separations required for the analysis.

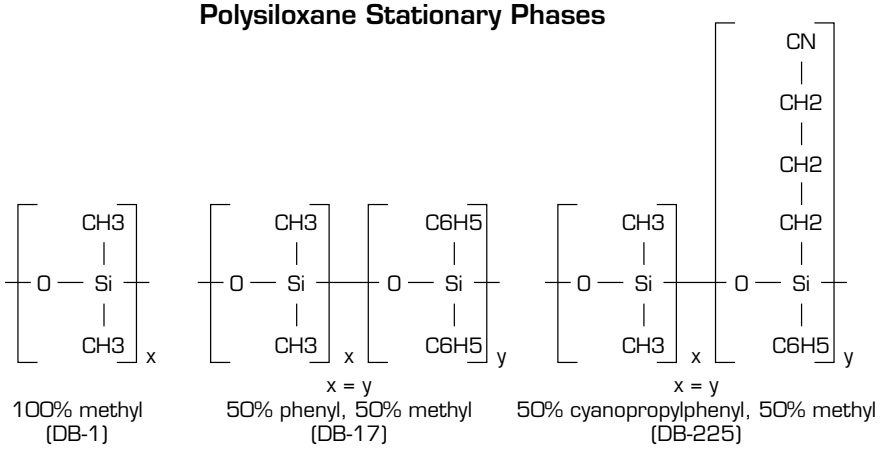
Column Specifications

Descriptions of GLC columns and operating conditions must specify the following: type of column (packed or capillary); its length, in meters (or feet), and internal diameter (id), in mm; identity and amount of liquid phase; identity of solid support, including pretreatments and mesh size (packed columns only); operating temperature; and carrier gas identity and flow rate.

Liquid phases used in GLC are viscous materials able to be thinly dispersed on solid support or on an internal column wall. Many different liquid phases are available, but relatively few are in routine use for pesticide residue determination, because pesticide residues usually either chromatograph on one of these phases or are not amenable to GLC. The chemical structure of the most common phases consists of a polysiloxane backbone with various substituent groups; Figure 502-a illustrates several of these.

Liquid phase polarity, important to its separation capabilities, varies with polarity and concentration of substituent group(s) on the polysiloxane. Thus, in terms of polarity, methyl<5% phenyl<cyanopropylphenyl<50% phenyl<cyanopropyl. The 100% methyl-substituted phase, least polar of those in Figure 502-a, is best suited to separation of nonpolar analytes; it has been used for many years as a general purpose phase for a wide variety of pesticide residues. The phase with 50% cyanopropylphenyl-substitution is the most polar of those shown and is a better choice for more polar analytes.

Figure 502-a
Polysiloxane Stationary Phases



Equivalent products suitable for different column configurations are commercially available for most common liquid phases; Table 502-a lists some of these products.

Although the table refers to liquid phases themselves, most pesticide residue laboratories no longer purchase liquid phases as materials for preparing columns in-house. Instead, laboratories that use packed columns usually purchase them prepacked or at least purchase packing material precoated with liquid phase. Residue laboratories always purchase commercially prepared capillary columns.

The liquid phase for a particular analysis is selected to take advantage of differences in chemical and physical properties of the analytes involved. No one liquid phase is universally applicable to the wide range of chemical and physical properties found in pesticide residues, so a variety of liquid phases of different polarities should be available in a residue laboratory.

For packed columns, the amount of liquid phase, often called "liquid load," is described as a percentage, *i.e.*, weight liquid phase × 100/(weight liquid phase + weight solid support). For open tubular columns, the amount of liquid phase is described as film thickness (μm) of the layer of liquid phase bonded to the internal wall of the column.

GLC columns are always heated to a temperature at which analytes remain in the vapor phase. Both isothermal and temperature-programmed operation are possible. Use of capillary columns with temperature programming is becoming increasingly common, but this operation will not be described further in this chapter because FDA has not yet validated its use on an interlaboratory basis. Maximum operating temperatures vary with specific stationary phases; information on each is provided by the manufacturer. Increasingly polar stationary phases (*e.g.*, cyanopropylphenyl) have significantly lower maximum operating temperatures than nonpolar phases (*e.g.*, 100% methyl). In use, maximum operating temperature is usually 20° C higher for temperature programming than for isothermal work.

Column Parameters

The following column characteristics or parameters are commonly used to describe chromatographic behavior or to measure column performance; terminology of these parameters is illustrated in Figure 502-b. Evaluation and comparison

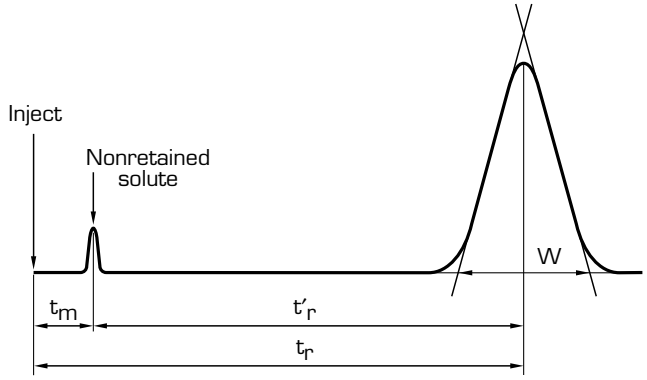
Table 502-a: Common GLC Liquid Phases Used in Pesticide Residue Determination

Equivalent Commercially Available Products[1]

Basic Structure, Substitutions	Capillary Open Tubular	Packed
Polysiloxane, 100% methyl	DB-1(ht), HP-1, HP-101, 007-1(MS), SP-2100 SPB-1, BP-1, CP-Sil 5CB, Ultra 1, RSL-150, RSL-160, Rtx-1, SP-2100, CB-1, OV-1, PE-1, SE-30, AT-1	OV-101, OV-1, SP-2100, DC 200, CP-Sil 5, SE-30
Polysiloxane, 50% phenyl, 50% methyl	DB-17 (ht), HP-17, PE-17, 007-17(MPS-50), AT-50, SP-2250, Rtx-50, RSL-300	OV-17, OV-11, SP-2250, OV-22, DC-710
Polysiloxane, 50% cyanopropyl-phenyl, 50% methyl	DB-225, HP-225, OV-225, SP-2330, CP-Sil 43CB, RSL-500, Rtx-225, BP-225, CB-225, PE-225, 007-225, AT-225	OV-225
Polysiloxane, 14% cyanopropyl-phenyl, 86% methyl	DB-1701, SPB-7, CP-Sil 19CB, Rtx-1701, BP-10, CB-1701, OV-1701, PE-1701, 007-1701	OV-1701
Polysiloxane, 5% phenyl, 95% methyl	DB-5 (ht), HP-5, Ultra-2, OV-5, SPB-5, Rtx-5, CP-Sil 8CB, RSL-2000, BP-5, CB-5, PE-5, SE-52, 007-2(MPS-5), SE-54	OV-3, OV-73, CP-Sil 8
Polysiloxane, 50% trifluoro-propyl, 50% methyl	DB-210, RSL-400, SP-2401	OV-210, SP-2401, OV-202, OV-215
Polyethylene glycol	DB-WAX, HP-20M, Carbowax, Supelcowax 10, CP-WAX 52CB, SUPEROX II, Stabilwax, BP-20, CB-WAX, PE-CW	Carbowax 20M, Supelcowax 10
Diethylene glycol succinate	No equivalent	DEGS (no longer produced)

[1] Commercial codes for each material are related to their manufacturer:
007: Quadrex, New Haven, CT
AT, RSL, SUPEROX: Alltech Associates, Inc., Deerfield, IL
BP: SGE, Inc., Austin, TX
Carbowax: Union Carbide Corp.
CB, CP-Sil, CP-WAX: Chrompak International BV, Middleburg, The Netherlands
DC: Dow Corning Corp., Midland, MI
DB: J&W Scientific, Folsom, CA
DEGS: Analabs, Inc., New Haven, CT
HP and Ultra: Hewlett-Packard, Co., Wilmington, DE
OV: Ohio Valley Specialty Chemical Co., Marietta, OH
PE: Perkin Elmer Corp., Norwalk, CT
Rtx, Stabilwax: Restek Corp., Bellefonte, PA
SE: General Electric
SP, SPB, and Supelcowax: Supelco, Inc., Bellefonte, PA

Figure 502-b
GLC Column Parameters



Column parameters are calculated from measurements on a chromatogram produced by the column.

of columns can be based on such parameters. More detailed discussion of parameters and conditions affecting each are found in any basic chromatography text, such as those listed in Section 505, Bibliography.

Retention Time. The most basic measurement in chromatography is retention time, the time between sample introduction and elution of the analyte, measured at the peak maximum (t_r in Figure 502-b). Retention time is corrected for the time required for a nonretained solute to reach the detector (tm), often called dead time or holdup time. Corrected retention time (t'_r) is the difference between t_r and t_m. For practical convenience, the peak caused by the solvent is used as the nonretained solute in pesticide residue determinations.

Analyte retention time depends on the extent to which the analyte is retained by the particular stationary phase under a given set of conditions. Retention time is constant when column temperature and carrier gas flow are constant, so this characteristic is the GLC measurement that serves to identify the analyte; it can be measured electronically in seconds or manually in mm from the resulting chromatogram. Retention time measured from injection to peak maximum is often called "absolute retention time."

Absolute retention time is affected by many column conditions that can vary, including amount of liquid phase, temperature, carrier gas flow rate, column length, and system volume. Thus, absolute retention times are insufficiently reproducible to list in tables of data intended to assist an analyst in identifying analytes. Instead, "relative retention times" are calculated and listed, because they are far more reproducible from day to day and among different instruments or laboratories.

Relative retention time (rrt) of an analyte is the ratio of its corrected retention time (t'_r) to the corrected retention time of a "marker" (reference) compound. The pesticide chlorpyrifos, molecular formula $C_9H_{11}C_{13}NO_3PS$, is used in this manual as the marker compound for most systems, because it chromatographs well and contains all the heteroatoms to which selective GLC detectors respond; retention times relative to chlorpyrifos (rrt_c) for many pesticides and related compounds are listed in Appendix I, PESTDATA.

For the same (or equivalent) liquid phase, rrt of an analyte is independent of column type (packed *vs* capillary), liquid load, column length, or carrier gas flow rate change. The rrts for a particular liquid phase vary significantly only with column temperature; rrt_cs in Appendix I are valid only at the temperature specified for each column.

Capacity Factor. Capacity factor describes the retentive behavior of a sample component relative to the "retentive behavior" of a nonretained component. The

capacity factor of an analyte depends only on the time the analyte spends in the stationary phase, which is, chromatographically speaking, far more important than the time spent in the mobile phase. The capacity factor (k) of an analyte is calculated from analyte retention time as $k = (t_r - t_m)/t_m$.

(Capacity factor should not be confused with "sample capacity," which describes the maximum amount (*e.g.*, 50 ng) of an analyte that can be injected onto a chromatograph before column overload occurs. Column sample capacity depends on percent liquid load in packed columns and on column id and film thickness in capillary columns.)

Selectivity. Stationary phase selectivity is simply defined as the ability of a phase to differentiate between analytes in the same injection. The selectivity term is technically not interchangeable with polarity [1]. A polar column may exhibit very poor selectivity for a particular chemical species. In general, nonpolar stationary phases exhibit greatest selectivity for nonpolar analytes, and polar stationary phases exhibit greatest selectivity for polar analytes. Selectivity of a GLC system is defined by both the stationary phase and the analytes. In the literature, selectivity (α) is synonymous with separation factor, relative retention, and selectivity factor and is calculated as k_B/k_A, where k_B and k_A are capacity factors of two adjacent peaks. In this calculation, a is always ≥ 1.0, but a separation factor of 1.0 indicates that no separation is possible in that system [2].

Resolution. Resolution is the degree of separation between two chromatographic peaks and is related to time (capacity factor), selectivity, and efficiency. Considerable information about resolution and its related parameters is available in general textbooks on chromatography. For practical purposes, however, it is enough to know that optimizing selectivity by choice of stationary phase will optimize resolution. Despite the importance of column efficiency in analyzing complex samples, especially at low levels, increasing efficiency will not solve all separation problems and often will only increase analysis time. A different choice of stationary phase may solve a resolution problem more easily than a longer column will. Resolution is considered optimized when calculated k values range between 2-10.

Efficiency. In qualitative terms, column efficiency refers to the degree to which injected analyte is able to travel through the column in a narrow band. Visually, a more efficient column produces narrower, sharper peaks on the chromatogram. The more efficient the column, the better able it is to resolve analytes that elute close to one another. Greater efficiency results in greater signal-to-noise ratio and hence increases sensitivity. Efficiency is measured quantitatively by calculating theoretical plates according to the formula

$$N = 16\ (RT/w)^2$$

where N = total theoretical plates, RT = absolute retention time in mm, and w = width of peak base in mm, measured as the distance at the baseline between lines drawn tangent to the two sides of the peak. The analyte on which theoretical plates are calculated must be specified, because comparisons are only valid for analytes eluting at the same absolute retention time. Column efficiency can also be expressed as height equivalent of one theoretical plate (HETP), *i.e.*, column length (cm)/N; using this expression, smaller numbers represent more efficient columns. Calculation of theoretical plates/column length permits comparisons of different length columns.

Basic GLC texts, such as those listed in Section 505, provide additional explanations about theoretical plate measurements, qualitative effect of column efficiency on peak shape, and practical means of improving peak shape. Column efficiency is referred to in this chapter when discussing relative advantages of different types of columns.

502 B: PACKED COLUMNS

During most of the over 30 years of GLC use in pesticide residue determination, packed column GLC prevailed as the only practical option. During early development of open tubular capillary columns, when only traditional capillaries were available, packed columns offered distinct advantages in ease of use and capacity for injection of larger volumes of extract. Current availability of wide bore capillary columns has reversed the trend, however, and use of packed columns is diminishing.

Packed columns still offer advantages in ease of installation; no additional inlet adapters or other specialized hardware are needed to install packed columns into chromatographs designed for packed column operation. Packed columns can also still withstand repeated injections of extract better than capillary columns. However, recent improvements in inlet systems and operating parameters for wide bore columns have increased their capacity for injected extract. Combined with the innately greater efficiency and inertness of wide bore columns, these improvements are encouraging the shift from packed to wide bore columns for routine use.

Components of Packed Columns

Packed columns consist of packing material made by coating inert solid support with a thin film of stationary liquid phase, glass or metal tubing to contain the packing material, and silanized glass wool plugs used to hold the packing material in place within the tubing.

Solid Support. The solid support in packed GLC columns provides a large inert surface onto which the stationary liquid phase is deposited as a relatively uniform thin film. Solid support should provide as large a surface area as possible and should interact as little as possible with analytes. Desirable properties of solid supports are large surface area per unit volume, chemical inertness at high temperatures, mechanical strength, thermal stability, ability to be wetted uniformly by a stationary liquid phase, and ability to hold a liquid phase strongly.

The most frequently used solid supports for GLC column packings are derived from diatomaceous earth. The structure of the diatomaceous earth consists essentially of three-dimensional lattices containing silicon with active hydroxyl and oxide groups on the surface. Untreated diatomaceous earth has considerable surface activity that must be reduced before it becomes a suitable support material. Several techniques have been used to deactivate the surface activity of diatomaceous earth. Most frequently, the diatomaceous earth is acid-washed and then silanized with an agent such as dimethyldichlorosilane.

Different commercially available solid supports and even different lots of the same support may have different surface areas or variations in inertness toward particular analytes. Unpredictable behavior among solid supports provided the impetus

for most laboratories to purchase precoated packing. Variations in solid support activity are of greatest concern when determining pesticide residues that are difficult to chromatograph, because such analytes are easily adsorbed or degraded during chromatography. Adsorption or degradation of an analyte on a poor quality solid support can affect the relative retention time of the analyte and the size and shape of the resulting peak. The most inert solid support material available should always be used to prepare column packings.

Chromatographic solid supports are available in a variety of mesh sizes. A support material of 80/100 mesh contains particles that will pass through an 80-mesh screen but not through a 100-mesh screen. Experiments have shown that column efficiency improves as solid support mesh number increases (particle size decreases) [3]. However, to maintain the same gas flow through a column, carrier gas pressure must be increased as solid support particle size decreases. Mesh size of 100/120 was shown to produce optimum efficiency for a 6' column of 4 mm id. Columns 4-6' long and 2-4 mm id, filled with column packings prepared from 80/100 or 100/120 mesh solid supports, are routinely used for residue determination.

Liquid Phase Load. No matter what liquid stationary phase is used, liquid load influences column efficiency and capacity (amount of sample extract that can be injected onto the column). Packing materials with loads ranging from <1 to 5% are routinely used for pesticide residue determination.

Liquid phase load can be varied without changing relative retention times of compounds if the same column temperature is used. At the same column temperature and gas flow, a column with less liquid phase will allow compounds to elute more quickly than a higher load column. Carrier gas flow can be lowered when using columns with less liquid phase to permit compounds to elute at approximately the same time as from higher load columns operated at higher gas flows.

Laboratory observations indicate that compounds with a tendency to degrade on or be adsorbed by a column are more likely to do so when a lower liquid phase load is used, probably because the lower load is incapable of covering all solid support active sites. In these cases, analyte retention time and peak size will be affected, as described above. Residue analysts should be aware of the pitfalls of low load columns when dealing with compounds that are easily degraded or adsorbed.

Column Tubing. Almost all columns used in pesticide residue determinations are made from glass tubing. Although some gas chromatographs require metal columns, so many problems occur with metal that they should be avoided. In the past, new glass columns had to be cleaned and silanized in the laboratory to remove any residual caustic materials and to deactivate the column. Today, most glass columns are silanized by the manufacturer and are purchased ready to use. Inadequate deactivation of glass columns can cause peak tailing due to adsorption or degradation of the sample or standard on the active sites of the column itself.

Glass Wool. Glass wool for use in GLC columns must be silanized to prevent compound adsorption; presilanized glass wool is available commercially or silanization can be performed by the laboratory. A plug of silanized glass wool is always used at the outlet (detector) end of a packed column to hold the packing material in place. Glass wool can also be used in the inlet end of a packed column, but opinions vary about the advisability of this practice.

Used in the inlet end of a packed column, glass wool can cause adsorption or degradation of certain sensitive compounds. Problems with normally stable compounds can also occur when deposits of sample co-extractives collect on the glass wool.

In particular, when deposits of fatty extracts accumulate at the top of the column, analytes in subsequent injections can be partially trapped; errors in residue quantitation result. Elimination of glass wool at the inlet end of the column appears to minimize this problem by allowing injected co-extractives to spread over a portion of the column where subsequent analytes cannot be trapped so readily.

In other cases, glass wool in the inlet end of the column may prevent the rapid deterioration of columns caused by injecting co-extractives from fatty foods or other commodities that are difficult to clean up. Co-extractives trapped on the glass wool plug can be eliminated by replacing the plug, an easier, quicker, and less expensive process than replacing the packing material.

Choosing whether to use glass wool in the inlet end of the column appears to depend on several factors, including type of packing material used, commodity being analyzed, analytes of interest, type of detector, and method of analysis. Experience will dictate when the advantages of glass wool in the column inlet outweigh the disadvantages; a laboratory attempting to locate the source of problems in a GLC determination should definitely investigate the effects of glass wool in the column inlet.

Preparation of Packed Columns

Acceptable techniques for packing empty GLC columns are designed to fill the column with as much packing material as possible (*i.e.*, to pack the material as tightly as possible) while breaking the fewest particles. Column efficiency increases with the amount of properly coated support in the column, and adsorption and degradation problems are minimized when careful handling of the packing material creates the fewest broken (active) sites.

Poor packing technique causes visible differences in column performance (efficiency) and peak symmetry. Loosely packed columns or columns containing too little column packing are inefficient and a cause of inadequate separations. On the other hand, a column packed too tightly requires excessive carrier gas pressure, which can result in the column becoming plugged with broken particles.

To pack a glass column:

- Insert about 1-2" silanized glass wool into detector end of column, far enough from end to prevent packing material from extending into detector base where temperatures are usually much higher than column operating temperature.

- Use rubber tubing to connect detector end of column to vacuum source (aspirator or vacuum pump); attach funnel with short piece of rubber tubing to inlet end of column.

- Apply partial vacuum at detector end of column, and slowly add prepared packing material through funnel.

- Tap column gently while adding packing material, to settle it as tightly as possible; do not use a vibrator to help settle packing.
- Continue to tap gently along entire length of column while adding more packing, until column is full or within 1" of being filled at inlet end.

To reuse a glass column:

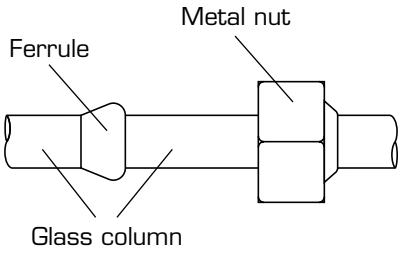
- Remove old packing.
- Rinse empty column successively with 5% potassium hydroxide/methanol and dilute hydrochloric acid.
- Rinse empty column thoroughly with successive portions water, alcohol, and ethyl acetate to eliminate accumulations of liquid phase and/or sample co-extractives from column walls.
- Dry empty column before repacking.

Installation of Packed Columns

In modern GLC equipment, glass columns, filled with packing material, are connected directly to the detector (metal) and injector (metal), an arrangement that eliminates dead space in the system. The availability of ferrules that are thermally stable at high temperatures makes these glass-to-metal connections possible and eliminates problems once associated with such connections. Ferrules with these capabilities include those made from Vespel, graphite, or Vespel/graphite mixture.

To install a glass column in the chromatograph:

Figure 502-c
Ferrules for Connecting Glass and Metal



[Reprinted with permission of Supelco, Inc., adapted from Supeltex M-1 Packed Column Ferrules data sheet (1987).]

- Slide stainless steel or brass (usually 1/4" Swagelok) nuts onto detector and inlet ends of column followed by ferrule, as shown in Figure 502-c.
- Connect nuts on column to corresponding hardware on injector and detector.
- Tighten each nut finger tight.
- To seal ferrules, alternately tighten detector nut and injector nut using standard wrench, following instructions provided by ferrule manufacturer. Unevenly exerted pressure on either end of column may twist and break it.
- Turn on carrier gas (30-60 mL/min) and flush with gas for about 20 min.
- After all oxygen has been flushed from column by flow of carrier gas (and not before), turn on column oven to heat column.

- Check connections for leaks after establishing carrier gas flow.

During installation, always hold the bottom of the column for support. Do not overtighten the nuts, which can force the column against the bottom of the injector/detector and break the column. Refer to the instruction manuals provided by the instrument manufacturer for more specific instructions on column installation.

Conditioning of Packed Columns

Column "bleed" is degradation of stationary liquid phase that causes a background signal as the detector responds to its presence. Column bleed occurs in all columns and is not in itself a symptom of damage. However, excessive or increasing bleed, seen as a rise in baseline, may be caused by damage to the column. Bleed increases when the column is operated at higher temperatures, and damage may be caused by operation at temperatures higher than allowed for a particular stationary phase.

To minimize column bleed, newly packed columns must be conditioned before they are connected to the detector; conditioning purges volatile components that could contaminate the detector and produce an unsteady baseline. Column conditioning involves heating the column above normal operating temperatures for an extended period prior to its use. The column must not be connected to the detector during conditioning. In most cases, a normal carrier gas flow is maintained during column conditioning. Excessively high conditioning temperatures will shorten column life.

Minimizing column bleed by conditioning is essential to good operation. If liquid phase is bleeding from the column, frequent detector cleaning will be necessary, sensitivity of the GLC system will change, quantitative results will probably be affected, baselines will drift, and good quality chromatograms will not be obtained.

"Stabilized" liquid phases are designed to be more thermally stable than their nonstabilized equivalents, because they bleed less at normal operating conditions. However, conditioning of stabilized packings is still required before use.

Conditioning procedures vary with the type of column packing and are provided by the manufacturer in the literature supplied with the packing.

Rejuvenation of Packed Columns

Column deterioration during use is most often caused by inadequate cleanup of samples injected onto the column (see Section 501 C). Extracts of materials containing large amounts of fats or oils (*e.g.*, dairy products, animal tissue, and fish oils) are difficult to clean up thoroughly. Injection of excessive amounts of oily extract can cause irreversible damage to a GLC column. Waxy or colored material co-extracted from nonfatty foods may also damage the GLC column, but this effect is not as readily apparent as that caused by oily co-extractives. Care should be taken to minimize the amount of any co-extractive material injected, including the use of additional or alternative cleanup techniques when original cleanup is inadequate.

No matter how rigorous the sample cleanup, some accumulation of co-extractives on the column will occur. To prevent column deterioration, the column must be periodically cleaned. Packed columns are most often cleaned by removing up to several inches of packing at the inlet end of the column and replacing it with new (preferably conditioned) packing. To perform this operation:

- Turn off column oven heat and permit oven to cool.
- When oven is cool, turn off carrier gas and remove column.
- Remove contaminated packing with disposable pipet or other device, and swab inside of glass column with acetone using pipe cleaner or other appropriate device to remove fatty deposits or other matrix contamination that have adhered to interior column wall.
- Add fresh packing to column, in same way described above for packing new columns.

Criteria for Acceptable Packed Columns

Column performance must meet the following criteria for successful pesticide residue determination. Exact performance will vary somewhat as the column ages, but minimum criteria should be met through its lifetime; when the column no longer meets these standards, it should be replaced.

Some of these criteria relate to careful column preparation and conditioning and are important to check when the column is new. Others relate to the potential for gradual column deterioration and contamination during use. Some other part of the GLC system may be responsible for the system's failure to meet criteria, so all parts should be examined when the system is malfunctioning.

1) Chromatography of selected compounds should result in a single symmetrical peak with no breakdown. Endrin frequently chromatographs as two or three peaks when columns are not satisfactory, and methoxychlor breaks down to its olefin. DDT deteriorates to TDE or DDE or may be lost entirely on a contaminated column. None of these conditions should be tolerated.

2) Peak resolution of selected compounds should be complete. For example, dieldrin and endrin can be separated from one another on most columns that are performing well; a mixture of the two should be chromatographed routinely to monitor changes in resolution as the column ages.

3) Peak heights for several compounds should be reproducible when repetitive injections are made. Poor reproducibility (≥5%) can have several causes external to the column: improper injection technique, a faulty syringe, a faulty septum, or detector malfunction. Poor reproducibility can also indicate breakdown or adsorption of the compound on the column. Compounds used to test the column for general acceptability are those that may break down or be adsorbed by columns but can be successfully chromatographed, such as endrin. When a column is used to analyze for compounds that are hard to chromatograph, it should first be checked with a compound such as endrin.

Sometimes, injection of large concentrations of compounds that are difficult to chromatograph may improve their chromatography. Some pesticide chemicals may not chromatograph well until a column is more thoroughly conditioned by prolonged use. If a compound shows tailing, or little or no response, or if multiple peaks are obtained from injection of a single standard of known purity, adsorption, degradation, or some other column effect may be the cause. Chromatography of some compounds may not be satisfactory until the column has been used extensively.

4) Instrument response to varying amounts of a compound should be linear. A nonlinear response can have many causes, but breakdown or adsorption of the compound on the column may be indicated when the system is linear for some compounds but not for others. It is especially important to ascertain linearity for each compound of interest when the compounds are difficult to chromatograph.

5) A 4 mm id packed column should have about 500 theoretical plates/foot of column length, as measured on a peak produced by p,p′-DDT. (Retention time of the peak used affects theoretical plate calculation, so measurement of the p,p′-DDT peak at whatever time it elutes from an individual column is an admitted oversimplification, but is adequate for the purpose defined here.)

   Theoretical plate counts <500 do not necessarily render a column unacceptable, but performance of columns with <400 plates/foot should be closely observed. Routine measurement of theoretical plates will alert the analyst to unsatisfactory new columns or to deterioration of columns already in use and is recommended as a part of the routine check on instrument performance.

Recommended Operating Procedures for Packed Columns

Each GLC determinative step in Chapters 3 and 4 is described in terms of its specifications and operating conditions. Most of these describe wide bore capillary columns, now recommended for routine use in pesticide residue determination; only Sections 302 DG20-DG23 describe systems with packed columns, because the DEGS column of those modules has no wide bore equivalent. However, most GLC data (rrts and responses) included in Appendix I, PESTDATA, were developed with packed columns during the many years in which they were in use. Table 502-b provides operating conditions for packed columns useful in pesticide residue determination.

Column liquid phase and temperature are dictated by the analytes being sought in a particular method. Choice of carrier gas depends on the requirements of the detector; in some cases, argon/methane is used to accommodate the ^{63}Ni electron capture detector. Carrier gas flow rate is typically 30-60 mL/min. Injection volume is typically 3-8 µL.

Columns must be protected from damage that can occur when the stationary phase is exposed to oxygen at high temperature. Increased bleed of degradation products from oxidation will occur, and the phase can be damaged permanently. After any exposure to air, *e.g.*, during septum change, the column should be

Table 502-b: Operating Conditions for Packed Columns[1]

	OV-101	OV-17	OV-225
Liquid load, %	5	3	3
Injector temperature, ° C	225	220	225
Target rrt_c[2] (marker compound)	3.1 ± 0.06 (p,p′-DDT) 2.56 ± 0.05 (ethion)	3.5 ± 0.07 (p,p′-DDT) 3.36 ± 0.07 (ethion)	3.6 ± 0.06 (p,p′-DDT) 3.9 ± 0.1 (ethion) 0.69 ± 0.02 (lindane)
Elution time, min[3]	4	4	5.5
Conditioning[4]	1° C/min to 250° 250° C, ≥16 hr	1° C/min to 250° 250° C, ≥72 hr	50° C; 2° C/min to 245° 245° C, ≥60 hr
Special requirements			Do not use with ElCD or N/P

[1] All columns are: 1.8 m × 2 or 4 mm id; liquid phase coated on 80/100 mesh Chromosorb W HP, or equivalent.
[2] Column temperature is 200° C, adjusted as needed to produce specified rrt_c for marker compound.
[3] Approximate elution time of chlorpyrifos with carrier gas (nitrogen, helium, or argon/methane, as required by detector) at about 60 mL/min.
[4] Conditioning performed with column disconnected from detector. Degas with 60 mL/min nitrogen for 1 hr; temperature program as specified; hold at specified temperature with nitrogen flowing for specified time period.

checked for leaks and then flushed with carrier gas for 15-20 min before restoring the column to operating temperature.

If it is necessary to change carrier gas tanks while the column remains at operating temperature, interruption of column carrier gas flow can be avoided by turning off secondary valve pressure, which is usually at 40-80 psi. While the gas flow continues bleeding into the column, the main tank valve can be turned off and the regulator moved to a new tank.

502 C: OPEN TUBULAR CAPILLARY COLUMNS

Capillary column GLC has existed almost as long as packed column GLC and is now preferred for determining pesticide residues in foods. Capillary columns provide greater inertness, chemical and thermal stability, column efficiency (and thus system sensitivity), resolution, operating temperature range, and column-to-column reproducibility than equivalent packed columns.

In addition to the nature of the liquid phase, many factors affect capillary column performance and applicability, including bore size, film thickness, operating temperature, column length, and carrier gas identity and flow rate. Most often, a change in one column parameter improves some features of column performance and diminishes others, so choice of column for a particular analysis is based on an assessment of the most important feature(s).

Capillary columns are available with internal diameters ranging from 0.050-0.53 mm. Efficiency increases as capillary column bore size decreases. Traditional (0.25-0.32 mm id) and narrow bore (<0.25 mm id) capillary columns are noted for extraordinary efficiency (≥5000 theoretical plates/m), which improves signal-to-noise ratio and thus sensitivity. High efficiency also provides the improved resolution necessary for analyses of complex samples. However, sample capacity decreases with decreasing bore size, and columns become less forgiving of improper handling. In addition, the low carrier gas flow rates used (≤0.9 mL/min for narrow bore and ≤3 mL/min for traditional) require specialized flow control hardware.

For certain determinations, advantages offered by narrow bore columns outweigh their disadvantages. Thin film, narrow bore capillary columns are ideal for specialized "ultra trace" determinations at levels of part per trillion and below, *e.g.*, for determination of dioxin residues. Once adjustments are made to accommodate requirements of narrow bore columns related to gas flow, sample capacity, and injection technique, they provide the ultimate efficiency, resolution, and sensitivity needed for these determinations.

The low gas flows required with narrow bore capillary columns also make them the best choice for use in certain instruments. For example, interfacing narrow bore columns directly to mass spectrometers has become an industry standard, because the low flow is compatible with the requirements imposed by vacuum conditions within the spectrometer (≤1 mL/min maximum flow). Use of narrow bore columns obviates the need to divert carrier gas before effluent reaches the spectrometer.

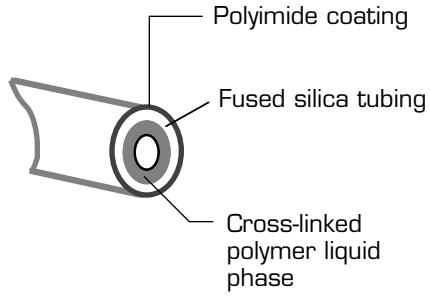
In contrast, either wide bore (0.53 mm id) or traditional capillary columns are preferred for routine pesticide residue determination, with wide bore the most popular. Although wide bore columns are less efficient than narrow bore, they offer greater sample capacity; depending on the film thickness, wide bore columns may have sample capacities comparable to packed columns. Carrier gas flow of ≤6 mL/min is recommended for optimum efficiency, but if this results in excessively long analysis time, the larger internal diameter of a wide bore column can accommodate 20-30 mL/min without generating excessive column head pressure. Wide bore columns can be operated at these higher gas flows ("packed column conditions") without the specialized pneumatics required for low flow rates. Performance of wide bore columns can be optimized by changes in carrier gas flow rates and other system parameters, such as injection technique.

Column Description

Early open tubular capillary columns were made from glass, with liquid (stationary) phase coating the interior wall. These columns were fragile and subject to significant liquid phase bleed. The columns assumed the shape of the "cage" on which they were mounted and thus required straightening before inserting the ends in inlets or detectors. A high degree of operator skill was necessary for their use.

The disadvantages of capillary columns were minimized or eliminated when several features were vastly improved. Columns are now made from fused silica, a synthetic quartz, coated on the outside with polyimide, which makes them rugged, flexible, and easy to handle. Stationary phases are now cross-linked polymers bonded to the interior column wall, effectively eliminating column bleed.

Figure 502-d
Capillary Column Cross-Section



Drawing (not to scale) of GLC capillary columns; id of fused silica tubing ranges from 0.05-0.53 mm.

Figure 502-d shows a cross-sectional view of a typical modern open tubular capillary column.

Tubing for capillary columns is produced by drawing fused silica through a furnace. The exterior of the drawn capillary tubing is then coated with a plastic polyimide coating and the interior cleaned and deactivated. Exact processes used by manufacturers are proprietary and beyond the scope of this chapter. The resultant tubing is very flexible, rugged, and reasonably inert and requires only minimal care in handling. It is easily cut and may be coiled around cages and flexed as necessary for instrument connections. When released, the tubing straightens, simplifying connections. Capillary columns are available in lengths from 10-60 m; 15 m or 30 m columns are usually used for determination of pesticide residues in foods.

Stationary phases are no longer simply coated on the interior walls. Individual stationary phase polymer "strands" are cross-linked, and the cross-linked stationary phase is covalently bonded to the deactivated interior wall of the tubing by proprietary processes. Columns prepared in this manner are more thermally stable than coated phases, so they can be operated at higher temperatures; they are also more efficient. Cross-linked phases exhibit minimal bleed and resist being stripped by solvent, to the degree that they can be rinsed with solvent to remove nonvolatile contaminants. The process of cross-linking also facilitates preparation of thicker films (*i.e.*, 1.0-8.0 µm) that are otherwise difficult to prepare. Chemically, the stationary phases are equivalent to those coated on solid support for packed column use, so relative retention times for analytes are essentially the same in equivalent packed and capillary columns, as long as column temperature is the same [4].

Capillary columns are available with films ranging from 0.10-5.0 µm thick. Columns with <0.32 mm id usually have film thickness of 0.10-1.0 µm, while those of ≥0.32 mm id have films 0.1-≥5.0 µm. Film thickness is proportional to sample (analyte) capacity, *i.e.*, thicker films accommodate more analyte without overload. Theoretically, a 0.53 mm id column has a sample capacity of 53, 130, 530, and 2600 ng for film thicknesses of 0.1, 0.2, 1.0, and 5.0 µm, respectively; sample capacity for a 0.25 mm id capillary column is about half as much for each film thickness [2].

Column efficiency, however, is inversely proportional to film thickness. Thick film columns are also more retentive than thin film columns, so retention times are longer and analyte peaks broader on the former. Thick film columns are also more susceptible to column bleed.

Because polar stationary phases (*e.g.*, cyanopropylphenyl) are difficult to coat onto column walls, they are usually only available in film thicknesses up to 1.0 µm. Polar stationary phases tend to bleed more than their nonpolar counterparts even under ideal conditions.

Film thickness for columns used in pesticide determination is normally 1.0 or 1.5 µm, which provides optimum balance between phase thermal stability, analyte

retention, and analyte column capacity. Relationships between film thickness and column efficiency, thermal stability, analyte retention, and capacity are discussed in detail in most modern GLC books (Section 505).

Each stationary phase has upper and lower temperature limits that define the operating range, but only the upper limit is of concern in pesticide determination. Operation at temperatures exceeding the upper limit accelerates phase degradation. Heating a column without carrier gas flow, or exposing it to any oxygen at temperatures ≥100° C, even for short periods, can damage phases rapidly and irreversibly.

Injection onto Capillary Columns

The small internal diameter of all capillary columns imposes specific requirements on how injection is performed; the narrower the diameter, the more rigid the requirements. (These injection options should not be confused with injection techniques discussed in Section 501 D. That section covers choices between manual and automatic injection and among various techniques for handling syringes. This section refers to ways to accommodate injection and vaporization of solvent into the restricted space available in capillary columns.)

Extensive research into means of introducing solutions onto capillary columns has produced four major injection techniques, called split, splitless, on-column, and direct. Each has advantages and disadvantages, and each has found uses in particular GLC applications. FDA studies, however, support recommendations that pesticide residue GLC determinations be performed with direct injection, using a retention gap, onto wide bore capillary columns [5]. This system eliminates or minimizes problems such as band broadening, peak splitting, and intolerance to variable injection volumes [6-9]. Direct injection involves introduction of the sample into a hot, vaporizing inlet with total transfer (no splitting) of injected materials onto the analytical column. GLC inlets designed for packed columns are easily converted to use with direct injection; kits for this purpose are commercially available. Injection volumes of 0.5-6.0 μL are used with direct injection.

Direct injection is not suitable for use with narrow bore columns or low gas flows, so references such as those in Section 505 should be studied for further information on the other injection techniques not covered here.

Capillary Column Systems

The practical necessities of residue determination require that a minimum weight of sample equivalent be examined by the determinative step. When capillary column GLC is used for determination, provision must be made to ensure that the volume of extract needed for injection of this weight does not overwhelm the capacity of the column. The following arrangements are required to accommodate physical limitations imposed by capillary columns. Because requirements become more stringent as internal diameter decreases, different recommendations may apply to wide bore and traditional capillary columns.

Retention Gaps (Guard Columns). Use of a "retention gap" [10] is recommended for capillary column GLC used in pesticide residue determination. A retention gap is a segment of deactivated fused silica tubing (without stationary phase) that is placed between the instrument inlet and the top of the capillary column; in effect,

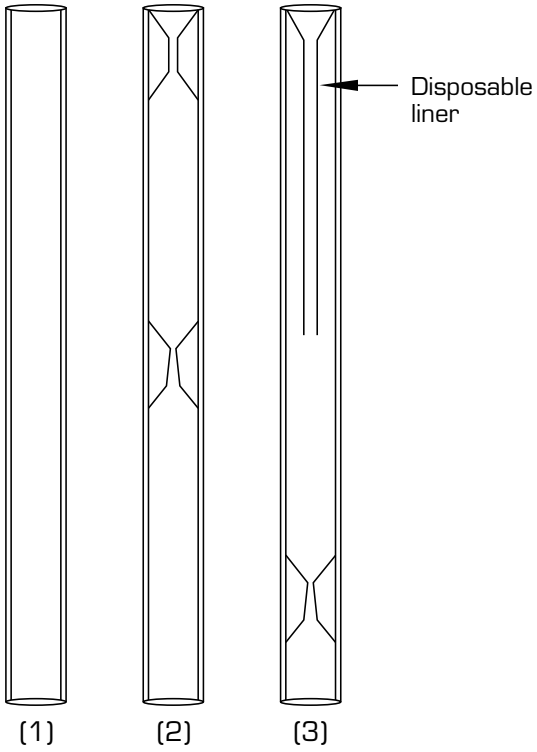
it serves as an extension of the column inlet. Tubing 0.53 mm id and 1-5 m long is commonly used; a length of 5 m is recommended for pesticide residue determination.

A retention gap serves at least two purposes: (1) It provides space for the injected solution to vaporize and expand, thus permitting injection of solvent volumes (>1 µL) that could not otherwise be injected into capillary tubing; and (2) it provides surface area for deposition of co-extractives, thereby protecting the analytical column from buildup of nonvolatiles that can cause loss of efficiency and analyte decomposition or adsorption; in this role, the retention gap is often called a "guard column." Properly installed, a retention gap will not noticeably reduce column efficiency.

Inlet Adapters. Direct injection of extracts and standard solutions onto capillary GLC columns requires a glass adapter to minimize analyte contact with hot metal surfaces. Adapter design has gradually evolved to meet the practical needs of trace level determinations.

Figure 502-e shows three styles of inlet adapters evaluated for use with direct injection. Adapter 1, the straight tube adapter, is simplest. A capillary column or retention gap is inserted into the bottom of this adapter with a stainless steel reducing union. This adapter, containing a small glass wool plug, was successfully used with various wide bore columns to determine pesticide residues in foods analyzed by the method described in Section 302 [11]. However, this style adapter is not recommended, because it allows exposure of analytes to the hot metal reducing union. In addition, injection of large volumes can result in flashback of solvent vapors and analytes into the instrument pneumatic systems.

Figure 502-e
Inlet Adapters for Capillary Columns



Inlet adapter used with capillary columns: (1) straight, (2) adapter with restrictors, and (3) adapter with disposable liner.

Adapters designed with tapered restrictors at the point where the column or retention gap connects are preferable to straight tube adapters; this design eliminates contact of analytes with the hot metal reducing union. Adapter 2 displays a commercially available direct flash injection liner [12] with a nontapered restrictor at the top and a tapered restrictor below for connection to a column. The top restrictor minimizes both flashback during injection and contact of analytes and solvent with the septum area of the inlet. This adapter was successfully used with extracts from Section 302 [13], and its performance was validated with an interlaboratory trial involving similar extracts cleaned up with Florisil [14]. The only drawback with this adapter is difficulty in cleaning.

A major improvement in inlet adapters is the addition of an easily replaced disposable liner. Originally, an adapter intended for use with on-column injection (not pictured in Figure 502-e) was inverted and modified to include a disposable Supelco PureCol™ inlet liner, a small amount of column packing, and a small glass wool plug for determination of organochlorine pesticides in fatty foods [15]. Subsequently, during application to analysis of nonfatty foods, the column packing material was found to be unnecessary. Successful application of this adapter-liner combination led to commercial production of Adapter 3, designed specifically for use with a replaceable liner, as shown.

Use of a liner protects the inlet adapter, because nonvolatile co-extractives deposit on the liner rather than the adapter. A contaminated liner is easily replaced without disturbing the connection between the adapter and the column; depending on instrument design, the liner is changed after removing the septum or after removing the adapter-liner combination from the GLC inlet.

Chromatographic efficiency using any of these adapters will deteriorate with repeated injections of food extracts. Efficiency can be restored by removing the adapter from the instrument, cleaning, and resilanizing. After resilanizing, the adapter (without a column attached) should be heated overnight to normal inlet temperature with 10 mL/min gas flow to remove excess silanizing reagent.

Septa. In GLC, injections are made by microliter syringes through septa made of materials that permit passage of a needle and then reseal after the needle is withdrawn. For troubleshooting purposes, chromatographers must be aware of the problems that can be caused by septa. Each septum has a limited useful life, after which it leaks and must be replaced. Leaking septa cause inaccuracies in quantitation, problems with chromatography, and exposure of the system to air. Materials from which septa are made can contribute to system bleed and/or can become brittle with use. Shards from damaged septa can also pass into wide bore columns and block gas flow.

Connections. Any adapter installed in the GLC inlet is sealed by means of a nut and high temperature ferrule. Ferrules are available in various sizes, shapes, and materials; GLC instrument manufacturers specify requirements for ferrules to be used in each instrument. Typically, ferrules of 100% graphite are used, though ferrules consisting of graphite and Vespel are also common and sometimes required (*e.g.*, graphite/Vespel is used in GC-MS because graphite ferrules out-gas).

Analytical columns are connected to retention gaps with "low dead volume" or "zero dead volume" butt connectors. Various styles are available from chromatographic supply companies, including ferrule, adhesive, and "press-in" types. The simplest and least expensive are the press-in types, in which each tube is pushed into opposite ends of a flared connector to form a seal. Press-in connectors are suitable for most applications and are ideal for connecting 0.53 mm retention gaps to smaller diameter analytical columns. The "universal" style of press-in connectors, *i.e.*, those that connect tubing of any sizes, have been found to work best.

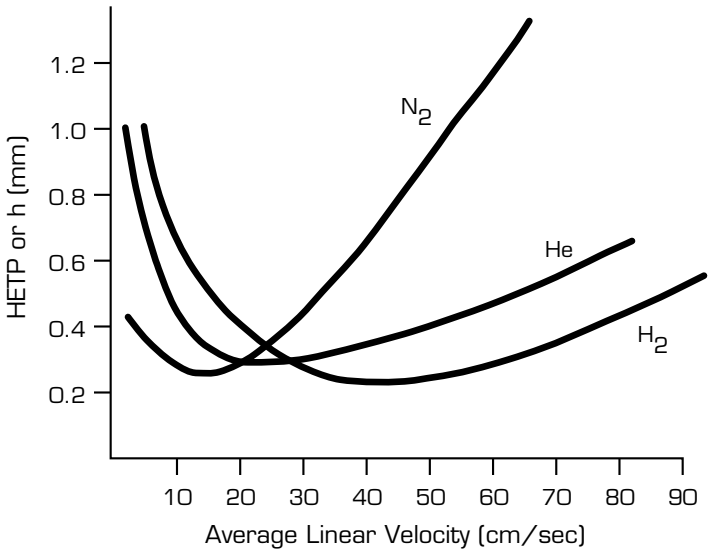
The two most critical connections in capillary GLC are those that connect the analytical column to the inlet and to the detector. Connections not only must be leak-free, but positioning is critical to optimum performance. Most manufacturers of gas chromatographs provide detailed instructions for positioning the column outlet at a specific location in detectors. These instructions must be followed exactly. If the manufacturer's instructions for proper positioning of capillary

column ends are not available, optimum positioning must be determined experimentally.

Inlet column positioning is also critical but is simplified with inlet adapters or liners that have tapered glass restrictors for column seating. The restriction is positioned at the proper location in the inlet, and the column is inserted firmly to form a seal between the polyimide coating and tapered fitting. Adapters or liners with no tapered glass fitting require careful, precise measurements for proper column positioning. Manufacturer's instructions for positioning tubing in inlet adapters or liners must be followed exactly.

Carrier Gases. Hydrogen and helium are the carrier gases of choice with capillary columns, because their flow rates can be increased with less loss of efficiency than is seen with nitrogen. The van Deemter curves for nitrogen, helium, and hydrogen (Figure 502-f) display the effect on column efficiency (HETP) of increasing average linear velocity (cm/sec, calculated as column length in cm/retention time in sec, of an unretained peak) in a typical capillary column.

Figure 502-f
Van Deemter Curves



Effect of carrier gas flow rate on column efficiency for several gases, measured using 30 m × 0.25 mm id column, 0.25 mm film thickness.

Minimum HETP (*i.e.*, maximum efficiency) for nitrogen carrier gas occurs at very low linear velocity (flow rate) and over a narrow range. Any increase of flow causes a substantial decrease in column efficiency. Chromatography at a flow rate required for usable column efficiency results in unacceptably long analysis time.

Compared to nitrogen, van Deemter curves for helium and hydrogen show greatest efficiency at higher flow rates. Use of these gases at their optimum flow reduces analyte elution time. The much shallower curves for these gases also demonstrate that increasing the flow above optimum to further reduce analysis time results in acceptable losses of efficiency. For these reasons, helium and hydrogen are commonly used as carrier gases for most capillary GLC applications.

Two different modes of operation, differentiated by carrier gas flow rate, are possible with wide bore capillary columns. Maximum column efficiency is achieved in "capillary column mode," *i.e.*, with carrier gas flows ≤6 mL/min. However, at these low flow rates, chromatographic time is considerably longer than the time to which pesticide analysts are accustomed with packed columns.

Operation of wide bore columns in "packed column mode," *i.e.*, 10-25 mL/min carrier gas flow, at 200° C (isothermal) combines the advantages of packed columns' faster elution with open tubular columns' greater deactivation (fewer active sites). In this mode, column efficiency equals or exceeds that of equivalent packed columns. At the same time, polar pesticides that require polar liquid phases (*e.g.*, DEGS) for packed column chromatography can be successfully chromatographed on capillary columns, even with nonpolar liquid phases [13, 14]. Because analyte relative retention times vary only with stationary phase and column temperature, extensive data (Appendix I, PESTDATA) compiled over the years for packed columns may be used for tentative identification of residues found with capillary columns operated at the same temperature [4]. Directions for operation of a wide bore capillary column in packed column mode, validated by interlaboratory study [5], are presented below.

Carrier gas should be of the highest possible purity, because use of highest purity gas will extend capillary column life. Moisture and oxygen traps should be used for all gases.

Makeup Gases. Because modern GLC detectors are designed for optimum performance at gas flows greater than those preferred for capillary columns, some systems require additional "makeup gas" to be added before effluent enters the detector. In addition to providing proper flow rate for optimum detector performance, makeup gas efficiently sweeps analytes from the end of the column into the detector. A gas different from the carrier gas may be used if the detector requires a specific moderating gas, such as argon/methane or nitrogen for an electron capture detector.

Makeup gas used for detector moderation should be of the purity level recommended by the manufacturer. As with carrier gases, moisture and oxygen traps should be used for all gases.

Installation and Conditioning of Capillary Columns

Regardless of the connection being made, proper cutting of the column and retention gap is critical. Square, clean cuts minimize flow disturbances and allow tubing of the same or different diameters to connect smoothly. Cleaving tools for cutting polyimide-coated fused silica capillary tubing are available from most chromatography supply companies. After scribing the polyimide coating with a cleaving tool, the tubing is cut by applying gentle pressure to bend the column opposite where it was scribed. If properly scribed, the column will break cleanly at that point. New cuts should be examined with a 10-20X magnifier to ensure that the cut is square and clean with no ragged edges or chips in the polyimide coating. Tubing should be recut if necessary to achieve a proper finish. All cuts should be made after installing any ferrules (especially graphite) onto the tubing to eliminate the possibility of small shaved ferrule particles being deposited in the end of the tubing.

Two techniques can be used to facilitate marking critical measurements for proper column positioning with either detectors or inlets. Water-soluble typewriter correction fluid can be used to mark columns at the desired length; the fluid does not react with the polyimide coating and is easily removed. Alternatively, a small slice of septum pushed onto the column can act as a marker and simultaneously hold the nut and ferrule in place for easier maneuvering in the oven.

After the inlet, column, retention gap, and detector have been connected, carrier gas flow should be established and all connections thoroughly checked for leaks. Every connection should be treated as the source of a potential leak, so connections should be minimized. Manufacturer's instructions must always be followed carefully to obtain leak-free connections. Soap solutions must never be used to detect leaks, because they can be aspirated into the system; the resulting contamination with nonvolatile materials can be removed only by rinsing the contaminated area. Electronic leak detectors are preferred. All leaks should be eliminated prior to heating the column.

Capillary columns with cross-linked, bonded stationary phases do not require the extensive conditioning of packed columns, because the stationary phases are more stable than those in packed columns and less susceptible to bleed. Usually, purging the column thoroughly with carrier gas, then heating it to 20-30° C above the maximum operating temperature for 1 hr is sufficient to condition the column. Conditioning is performed with carrier gas flowing; the detector may be connected during conditioning of capillary columns. It is critical that the upper temperature limit for the column not be exceeded.

Rejuvenation of Capillary Columns

With time and use, nonvolatile residues accumulate in all capillary columns, regardless of the use of retention gaps or other protective measures. Efforts to improve deteriorated chromatography should always begin with removal of portions of a contaminated retention gap or replacement of the retention gap. If the analytical column is also contaminated and replacement of the retention gap is insufficient to improve chromatography, a portion of the inlet end of the analytical column can be removed by cutting the column as previously described. If a capillary column ≥5 m is used, removal of a relatively short segment does not significantly affect its overall length or behavior, even if segments are removed repeatedly.

When removal of a contaminated segment of column is insufficient to restore appropriate chromatography, the column can be rinsed with solvents to help remove accumulated residues; rinsing may be performed with the retention gap attached. Column rinsing is possible because of the stability of the cross-linked, bonded phase.

Kits for rinsing columns are commercially available. Most kits consist of a vial that serves as a solvent reservoir; the vial has fittings for insertion of the column and for connection to a gas supply that pressurizes the solvent. The detector end of the column is inserted into the vial containing the rinse solvent, and gas pressure forces solvent backward through the column. The column should be rinsed with a sequence of solvents in order of decreasing polarity, starting with water and ending with hexane. Each solvent should be miscible with the preceding one. After the column is rinsed and excess liquid purged with gas flow, the column is re-installed and purged with carrier gas at ambient temperature for 30-60 min. The column should be conditioned by heating to 20° C above operating temperature for at least 15 min, but the upper temperature limit for the column must not be exceeded.

Recommended Operating Procedure for Wide Bore Columns (Isothermal)

The following procedure describes the setup and operation of a wide bore capillary column in packed column mode. Both direct injection adapters (below) were successfully validated [5, 14], and either may be used. Option 1 is recommended, because the disposable liner can be easily replaced once it has become contaminated with injected co-extractives.

Apparatus and Reagents.

column, fused silica capillary column, bonded with one of the substituted polysiloxane phases (Table 502-a), 30 m long × 0.53 mm id, 1.0 or 1.5 µm film thickness

retention gap, deactivated fused silica tubing, 5 m long × 0.53 mm id

capillary column connectors, low dead volume or zero dead volume, suitable for connecting analytical column to retention gap

direct injection adapter. Use new adapter as is without silanizing. Resilanize used and cleaned adapters with 10% dimethyldichlorosilane/toluene; after resilanization, heat to 240° C overnight with gas flow before use. Optional adapters are:

1) Silanized direct injection adapter (Figure 502-e, Adapter 3), 1/4" od, 4 mm id (Cat. No. 210-1071, J&W Scientific, Folsom, CA). This adapter is specially made to FDA specifications and is available with the special order part number. Direct injection adapter is 220 mm total length. Inlet end, 125-130 mm long measured from top of restrictor, has notches. Column oven end descends 75 mm below flared end of tapered restrictor. Column oven end may be shortened if desired; if cut, leave at least 20 mm tubing to attach adapter/column reducing union and lightly fire polish cut end. With Option 1 only: column inlet liner, PureCol™ for 4 mm id columns (Cat. No. 2-0540M, Supelco, Inc., Bellefonte, PA, or equivalent)

2) Double restrictor adapter, 1/4" od (Figure 502-e, Adapter 2). Adapter has two restrictors. Top restrictor allows passage of syringe needle into chamber formed by two restrictors. Lower restrictor is tapered for connection of column end into adapter. Silanize adapter prior to use if not silanized by manufacturer or if adapter has been cleaned. These adapters may be purchased cut to specified length or as longer version to be cut by user.

pesticide grade silanized glass wool; see Section 204 for silanization in laboratory; must be free of nitrogen, chlorine, phosphorus, or sulfur contaminants

capillary installation kit, required if chromatograph was designed for packed column. Kit should include necessary fittings to attach retention gap to inlet adapter and, if necessary, to detector, with provision for makeup gas at detector connection.

Gow-Mac Leak Detector, available from chromatography suppliers

Magnifier, 20X

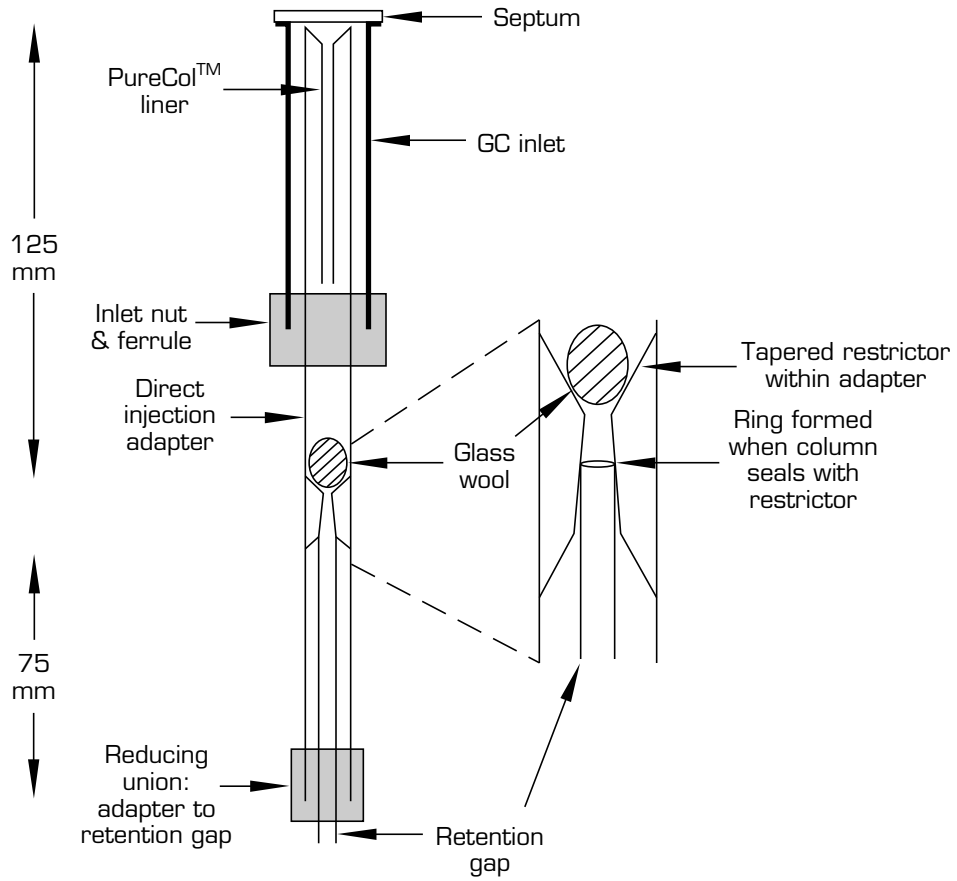
typewriter correction fluid, water based (if needed to mark correct positions on capillary tubing)

capillary column cleaving tool

Instrument Setup. Connect apparatus according to following directions; review instrument manufacturer's instructions and adjust directions as necessary to accommodate specific equipment. Figure 502-g shows the Option 1 inlet system arrangement:

- Place ferrules and nuts on end(s) of retention gap and column.
- Cut ends of column and retention gap with capillary cleaving tool. It is imperative that ends be cut *after* placement of ferrules to eliminate possibility of ferrule fragments becoming settled in tubing. Examine new cuts with 20X magnifier to assure that ends are square and smooth. Recut as necessary to obtain smooth, square ends.

Figure 502-g
Capillary Column Inlet System



- Clean ends of column and retention gap with wiper wetted with methanol.

- Attach retention gap to column, using appropriate capillary column connector.

- Secure retention gap to column cage.

- If using injection adapter (1), place small plug of glass wool in inlet end and push down as far as restrictor, then place disposable PureCol™ liner in inlet end of adapter. If using injection adapter (2), use as is without additional equipment.

- Gently insert adapter into instrument inlet or fitting until it touches top of injector. Move it back about 1-2 mm and tighten nut. Exercise care not to fracture end of adapter or liner when tightening nuts and ferrules during installation; overtightening can cause leaks and deform expensive hardware. If considerable effort is necessary to tighten fitting, it may already be deformed and should be replaced. (Follow manufacturer's instructions on how to tighten ferrules, check for leaks with leak detector, then tighten in small increments, *e.g.*, 1/4 turns, using correct size wrench to obtain leak-free connection.)

- Attach reducing union (for connecting adapter to retention gap) to column oven end of adapter.

- Insert end of retention gap through reducing union and into adapter. Push retention gap firmly into flared portion of restrictor until seal is formed between polyimide coating of retention gap and adapter restrictor wall. Formation of seal is evidenced by visible "ring" at contact point between tubing and restrictor wall (Figure 502-g, enlarged area). Tighten column nut on reducing union.

- If necessary, install makeup gas fitting to detector inlet using appropriate length and diameter of silanized narrow bore (1 mm id) glass tubing. Install column into detector as directed by detector manufacturer's instructions (if available) for positioning column. See section on connections, above, for additional cautions about effects of column positioning.

- Use helium carrier gas to get best column efficiency and compatibility with various detectors. For 30 m × 0.53 mm id columns, set initial flow to 20 mL/min.

- Use makeup gas as needed to accommodate optimum detector operation. Nitrogen, helium, argon/methane, or other gases may be used, as required for proper detector operation. Adjust flow of makeup gas so that total flow of carrier and makeup gases equals optimum gas flow recommended by detector manufacturer. Makeup gas flows of 5-40 mL/min are typical.

System Startup and Inspection.

- After installation and establishment of initial carrier and makeup gas flows, check all connections and fittings for leaks with electronic leak detector. Do *not* heat system until it is leak-free and has been thoroughly purged with carrier gas for 15-20 min to avoid damage from oxygen.

- Heat column to 230° C for 1 hr or until detector baseline has stabilized. Reduce temperature to 200° C and recheck both carrier and makeup gas flow rates. Adjust carrier gas flow and column temperature as necessary to meet retention time and rrt_c requirements for specific system being used. Re-adjust makeup flow as necessary to maintain optimum detector flow.

- Evaluate system for linearity, repeatability, and tolerance for varying injection volumes. System should be linear over at least one full scale deflection on integrator/recorder. For accurate quantitation, responses to repetitive injections of standard reference material should have percent relative standard deviation ≤5%. Limitations on injection volumes must be determined for each system. Experience has shown that injection volumes of 1.0-6.0 µL are normally tolerated without adverse affects on analyte response.

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503: DETECTORS

503 A: INTRODUCTION

GLC detectors are devices that indicate the presence of eluted components in the carrier gas emerging from the column. Depending on the way in which they measure the quantity of the analytes, detectors are classified as differential concentration, differential mass, or integral [1]. The electronic equipment associated with the detector amplifies the signal and causes the response to be recorded.

Definitions of Detector Characteristics

Characteristics of detector operation that are critical to qualitative and quantitative determination of residues include sensitivity, selectivity, and linearity. Certain terminology is common to the discussions of these characteristics in different detectors.

Sensitivity. Detector sensitivity refers to the relationship between amount of analyte injected and response of the detector. Detector response is the change in measured detector signal that results from a change in amount of analyte present within the detector volume; measured detector signal includes the amplification provided by associated electronics. Sensitivity is often described by referring to the smallest amount of a specific analyte that causes a measurable detector signal.

FDA methodology has traditionally specified detector sensitivity in terms of ng of a specified compound that causes 50% full scale deflection (FSD) on a recording or data system. That convention is continued in this chapter and in the determinative step descriptions in Chapter 3 methods.

Selectivity. Detectors must be selective to be suitable for use in determining any trace residue, including pesticides. Selectivity refers to the detector's preferential response to one or more elements ("heteroatoms") or functional groups that might be present in analytes of interest. Response of the detector to these moieties must far exceed its response to carbon, hydrogen, and oxygen if the resulting chromatogram is to distinguish between residues and food co-extractives present in the same extract.

Nonselective detectors, such as flame ionization (FID) and thermal conductivity (TC), respond to solutes in proportion to the mass of each that elutes from the column. Such detectors are impractical for most residue determinations.

Among detectors that are suitable for residue determination, selectivity to the moiety of interest varies considerably. Probably no detector is completely "specific" to one heteroatom or functional group; instead, degrees of selectivity can be described in terms of the relative response of the detector to the same weight of different compounds or moieties.

In practical terms, the greater the detector selectivity, the less sample cleanup is needed (within the boundaries discussed in Section 501 C) and the greater the inherent degree of confirmation that is provided by the determination. Conversely, the less selective the detector to the type of analyte being detected, the greater the precaution needed in preparing samples, avoiding reagent contamination, and confirming residue identity.

Some relatively selective detectors are subject to interferences from co-extractives that contain the heteroatom to which they respond, in particular those detectors that respond to the presence of nitrogen or sulfur. Sulfur-selective detectors are subject to considerable interference from organic disulfides present in foods such as onions, rutabagas, and brassicas. Use of cleanup steps to remove or react the interfering co-extractives may also cause the analyte(s) to be lost. Nitrogen-containing compounds are extractable from all foods; for this reason, no detector that selectively responds to elemental nitrogen can ever be totally successful in analysis of foods for trace-level contaminants. Several HPLC determinative steps have been developed for particular nitrogen-containing functional groups (Sections 401 and 403), and these have been more successful because interference from other forms of nitrogen is avoided.

Linearity. Use of a detector within its linear range is a prerequisite for the simplified way in which residues are routinely quantitated in pesticide residue determinations (Section 504). Terms associated with detector linearity are: dynamic (response) range, over which a change in the amount of chemical present within the detector volume produces a measurable change in detector response, and linear (response) range, the portion of the dynamic range over which a change in the amount of a chemical present within the detector volume produces a *proportional* change in detector response. A detector's linear range is the range of analyte weight over which the sensitivity of the detector is constant to ±5.0%, as determined from a linearity plot of response/weight *vs* log weight [2].

FDA laboratories evaluate the dynamic range of a detector and then operate in a segment of that range that exhibits appropriate linearity. For added assurance that quantitation is accurate, sufficient extract and reference standard solutions are injected to cause detector responses to residue and standard to agree within 25%.

503 B: ELECTRON CAPTURE DETECTOR

The electron capture (EC) detector has been used for many years to analyze foods for organohalogen pesticide residues. The earliest EC detector in common use had a ^{3}H (tritium) radioactive source; this was later replaced by detectors using a ^{63}Ni source. The continued popularity of the EC detector results from its high sensitivity to halogen and certain other moieties, as well as its ruggedness and low maintenance needs. Its sensitivity makes it applicable to determination of residues at the ppb and even ppt level, its wide dynamic response range facilitates its use with automatic data systems, and its high operating temperature (≤400° C) minimizes detector contamination by sample co-extractives and column bleed.

These advantages are offset by the EC detector's low selectivity compared to other detectors used in residue determination. When using the EC detector, appropriate methodology precautions are necessary to prevent introduction of interferences from food samples, reagents, or the environment (Section 501 C).

EC detectors used in FDA laboratories have ^{63}Ni sources and constant current, variable frequency design. Several manufacturers produce and market such detectors, all of which operate on the principles described below but vary in source activity, cell volume, and geometry. Most EC detectors currently in FDA laboratories are from Hewlett-Packard (HP), Wilmington, DE; Tremetrics, Inc. (formerly Tracor, Inc.), Austin, TX; or Varian Associates, Sunnyvale, CA. Discussions of detector characteristics in this section refer to detectors from these manufacturers.

Principles

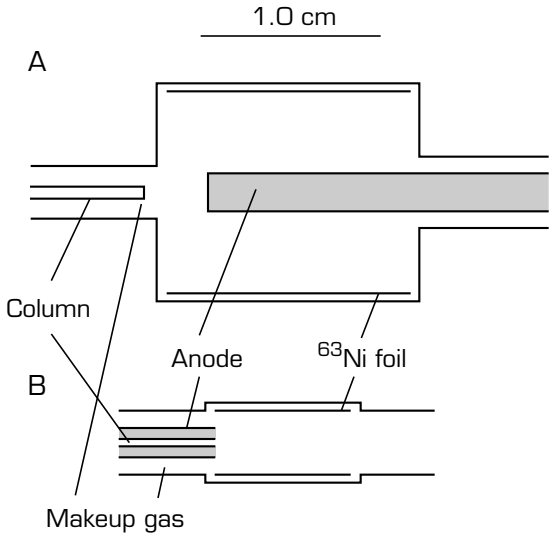
High energy beta particles, emitted by the ^{63}Ni source, collide with carrier gas molecules to produce low energy electrons. These electrons are continually collected at the cell anode by applying voltage pulses to the cell electrodes. Cell current thus produced is measured and the pulse interval (frequency) adjusted to maintain constant cell current. A standing pulse frequency describes the equilibrium condition that exists when only carrier gas is passing through the cell.

When molecules of an electrophilic substance enter the detector, electrons are "captured" to a degree dependent on the amount and electron affinity of the substance. As the electron supply is thus decreased, pulse frequency increases to generate the exact number of electrons necessary to maintain the established constant current. Change in frequency required to maintain constant cell current is converted to voltage, and this signal is sent to the recording device as the detector's response to the analyte.

Design

Two basic differences exist in EC cell design, one a pin-cup with ^{63}Ni plated on the cell wall and an anode suspended in the center of the cell cavity from the top, and the other with ^{63}Ni plated onto a cylinder aligned parallel to the column and cell flow. The electrode leads enter the cell cavity at right angles to the source and gas flow. Figure 503-a, diagrams A and B, display these respective designs.

Figure 503-a
Two EC Detector Designs



A, pin-cup EC, ^{63}Ni plated on cell wall; B, ^{63}Ni plated onto cylinder.

[Reprinted with permission of John Wiley & Sons, Inc., from Detectors for Capillary Chromatography (Copyright ©1992) Hill, H.H., and McMinn, D.G., ed., Chapter 5, by Grimsrud, E.P., Figure 5.2, p. 86.]

Apparatus and Reagents

Section 501 B provides general information on apparatus and reagents required for GLC. Further materials or specifications for this detector are described below.

Radioactive Source: Special Requirements. The presence of radioactive material in EC detectors brings them under the authority of the Nuclear Regulatory Commission (NRC). The following special procedures must be followed by a laboratory with an EC detector:

1) Labeling. According to NRC regulations, each chromatograph containing a ^{63}Ni detector must have a label signifying the isotope, activity, and date at which the activity was determined and the words "Caution: Radioactive Material." The NRC will accept the manufacturer's label on the detector if it contains the necessary information. If this information is not present, stick-on labels must be applied to the gas chromatograph. Appropriate labels have been provided to FDA laboratories by FDA's

Winchester Engineering and Analytical Center (WEAC), Winchester, MA. In addition, the Department of Health and Human Services (DHHS) requires that a sign with the following notice be posted on each gas chromatograph: "This equipment contains a radioactive source registered with the RSO (*i.e.*, the WEAC Radiation Safety Officer) as required by license from the NRC. Notify the RSO before removing the source from this location or upon any change in custodial responsibility" [3].

2) Venting. In addition to the labeling requirement, DHHS also requires that chromatographs with ^{63}Ni or other radioactive sources be vented through plastic tubing into a chemical hood or room exhaust [3].

3) Wipe tests. Any laboratory with a ^{63}Ni EC detector is required, as part of the licensing procedure, to perform a wipe test of all accessible exterior parts of the detector each January and July. WEAC supplies FDA laboratories with cotton-tipped swabs for performing the wipe tests. Each detector is wiped with an alcohol-moistened swab using moderate pressure, with particular emphasis on potential leak areas such as the outlet terminus and joints. Each swab is returned to WEAC in a mailing tube so that radiation removed from the detector exterior can be measured. A Certificate of Inspection for each detector is provided by WEAC and returned with new swabs.

4) Cleaning. NRC licenses in effect in FDA laboratories permit use of EC detectors containing ^{63}Ni but do not allow their dismantling and cleaning. All FDA ^{63}Ni EC detectors are shipped to the WEAC facility for cleaning [4].

Laboratories outside FDA must either make arrangements with a properly licensed laboratory for detector cleaning services or obtain the appropriate NRC license. Laboratories that have an existing license for use of ^{63}Ni EC detectors may be able to obtain from NRC an amendment that permits cleaning. Proof that the laboratory is capable of handling such materials safely is required before NRC will grant such an amendment.

Carrier and Makeup Gas. EC detector manufacturers recommend the use of argon/methane (95+5 or 90+10) for greatest detector linear range; however, nitrogen is used satisfactorily in some FDA laboratories. An external switch on the chromatograph permits selection of pulse width and cell current to accommodate whichever gas is predominant upon reaching the detector. Only reliable, high purity grade gas should be used, with oxygen and moisture traps on all gases going to the detector.

Often, the carrier gas and flow rate chosen for optimum column efficiency do not result in the best detector operation. Most EC detectors are configured to allow makeup gas to be added to the flow from the column so that detector operation is enhanced. In the most common example, argon/methane is essential to operation and is added to helium column carrier gas to produce optimum EC detector response and stability.

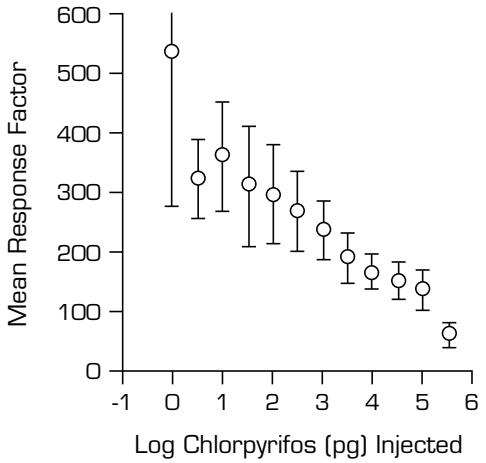
Detector Characteristics

The following basic characteristics of EC detectors must be understood for proper application to pesticide residue determination:

Sensitivity. Magnitude of response of a ^{63}Ni EC detector to a particular compound is dependent on the electron-capturing ability of the compound. Under conditions described below, the minimum detectable amount of an electron-capturing compound to which the detector will respond is typically in the 1-10 pg range.

Reproducibility of response at this low level is not as good as that for larger amounts of the same compound. A series of injections of 1-300,000 pg chlorpyrifos was made monthly over a 6-mon period [5]; variation in response factor over that time is shown in Figure 503-b. Standard deviations of response factors for each weight indicate that response stability was considerably better at levels ≥3 pg than at the 1 pg level, although response to 1 pg was reproducible over the course of any one day. Although this experiment was performed with an HP 5713A instrument, minimum response is expected to be equivalent with other ^{63}Ni constant current detectors.

Figure 503-b
Reproducibility



Mean and standard deviation of different weights of chlorpyrifos injected monthly (6 mon) into GLC with ^{63}Ni EC detector.

Selectivity. EC detectors respond to molecules containing an electrophoric group, *i.e.*, a highly polar moiety that provides an electron-deficient center in the molecule; examples include halogen, sulfur, phosphorus, and nitro- and α-dicarbonyl groups [2]. Because the response is not to a single element nor is it proportional to the amount of an element in a molecule, statements on detector selectivity can refer only to ranges or to relative responses to particular analytes.

Relative to its response to hydrocarbons, an EC detector may display 100-1000 fold greater response to mono- and disubstituted halogens and up to 10^6 times greater to polysubstituted halogens [6]. However, other molecules that contain only carbon, hydrogen, and oxygen may also be electrophoric, and EC detector response is far less selective to halogen relative to these molecules; examples include quinones, cyclooctatetracene, 3,17-diketosteroids, o-phthalates, and conjugated diketones [2].

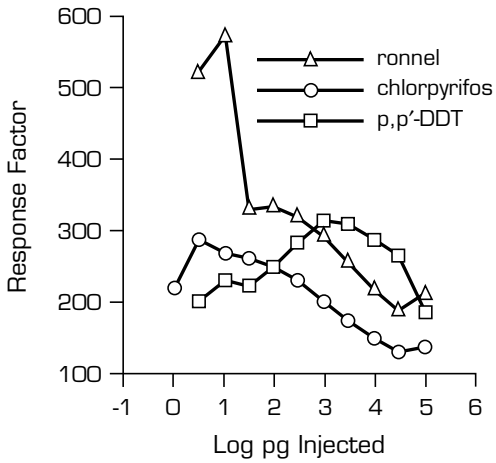
The relative lack of selectivity of the EC detector provides a bonus of applicability to a variety of analytes; *e.g.*, if an extract contains residues of pesticides containing halogen and also other residues containing sulfur, use of EC-GLC permits simultaneous determination of each. Lack of selectivity is more often a detriment to residue analysis, however; in practice, the EC detector's value is dependent on how free of interfering co-extractives the final extract is. Food co-extractives or environmental contaminants with electrophoric characteristics compromise the determination by causing responses that interfere with residues or that are mistakenly interpreted as residues.

Many examples of the interfering substances have been documented during long use of EC detectors. In addition to examples noted in Section 501 C, artifacts from plastics, rubber products, hand lotions, and cleaning solutions have been seen.

Certain fruits and vegetables also contain nonhalogenated substances that cause EC response, and these are not always removed by Florisil column cleanup (Section 303 C1, C2). Some of the sample types known to contain artifacts that cause an EC response include cabbage, radishes, and lettuce (6% Florisil eluate), carrots (15% Florisil eluate), and onions (both 6 and 15% eluates). Recommendations for avoiding interferences are included in the recommended operating procedures for EC, below.

Linearity. The mode of operation of the ^{63}Ni constant current detector produces a dynamic response range of greater than five orders of magnitude. Experiments in which an automatic data system was interfaced with the detector [5] showed that detector response to increasing quantities of injected material was still increasing when the data system became saturated. In order to plot the dynamic response over such a large range, the response (units in which response is measured/pg injected) is plotted *vs* log pg injected. A linear dynamic response range would produce a straight line parallel to the x-axis in such a plot. Plots of typical dynamic response ranges of the HP ^{63}Ni detector to three pesticides are shown in Figure 503-c.

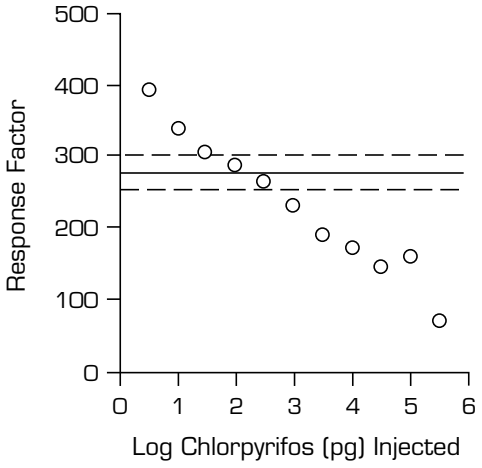
Figure 503-c
Dynamic Response Ranges



Response range of ^{63}Ni EC detector to three halogenated pesticides.

Instead of straight horizontal lines, the plots indicate a variation in response factor with amount of pesticide injected. These plots show that the detector is not linear over its entire dynamic response range. Within smaller segments of this range, however, the detector displays acceptable linear response.

Figure 503-d
Linear Response Range



Response range of ^{63}Ni EC detector is linear (±10%) to amounts of chlorpyrifos injected over a limited portion of the dynamic response range.

The range over which response is considered linear is dependent on the definition of linearity chosen. For example, in Figure 503-d the linear range is constant within ±10%; detector response is linear from approximately 30-500 pg chlorpyrifos. With this same definition, detector response can also be considered linear over other segments of the dynamic range. A change in definition (*i.e.*, different % variation permitted) would change the ranges for which detector response is considered linear.

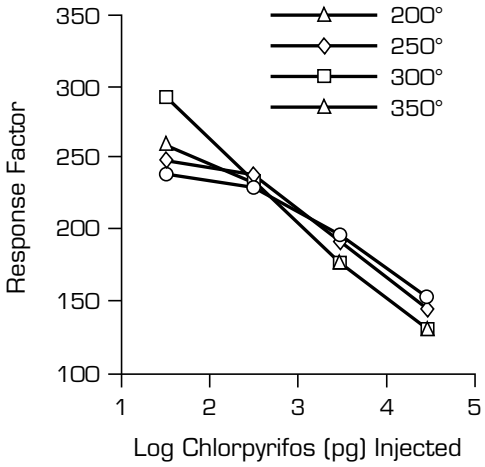
The general rule that detector response to residue and reference standard should match within 25% is especially important when using the EC detector.

Other Influences on Detector Performance

The following parameters were studied for HP ^{63}Ni constant current detectors [5] and, in a more limited study, for a Tracor ^{63}Ni constant current detector [7].

Detector Temperature. Experiments with the HP ^{63}Ni constant current detector [5] documented its dynamic response range for seven pesticides at four detector temperatures; Figure 503-e displays results for chlorpyrifos. Detector temperature caused only slight changes in response to any particular amount of pesticide and caused no consistent change over the whole dynamic range. Thus, there is no reason to choose detector temperature on the basis of enhanced response.

Figure 503-e
Effect of Detector Temperature

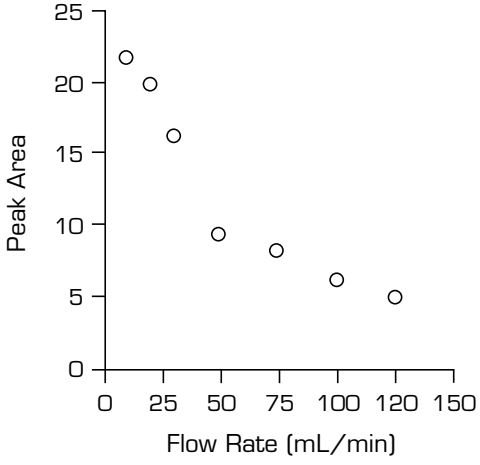


Dynamic response range of ^{63}Ni EC detector at four different temperatures.

Manufacturers provide recommendations for operating temperatures of detectors. Varian recommends operating the detector at 30° above column temperature, HP recommends 250-300° C, and Tracor recommends operation at 350° C. Detector contamination by materials eluting from the GLC column can be minimized with use of higher detector temperatures, but the ^{63}Ni radioactive foil must not be operated at >400° C.

Flow Rates. Column flow rate is usually chosen to optimize column efficiency and permit reasonable analysis time. The effect of flow rate on detector operation must also be considered, however, because response of the concentration-sensitive EC detector decreases with increased flow rate.

Figure 503-f
Effect of Carrier Gas Flow Rate



Response range of ^{63}Ni EC detector to 100 pg chlorpyrifos at different carrier gas flow rates.

Plots in Figure 503-f illustrate the effect of flow rate on response for the HP detector; a similar plot was obtained with the Tracor detector. In each case, the optimum flow rate range is that which provides for response stability, *i.e.*, flow at which a small change in rate does not cause a large change in response.

HP and Varian recommend a minimum flow rate of 30 mL/min through their detectors, and Tremetrics recommends 60-100 mL/min [6, 8, 9]. Each recommends use of makeup gas to bring the total flow through the detector to 60-100 mL/min. Makeup gas is usually needed for capillary columns (Section 502 C) and may be either the same as the carrier gas or different. For example, when hydrogen

or helium carrier gas is used to obtain maximum resolution and efficiency on the column, makeup gas of argon/methane or nitrogen must be used for proper detector operation.

Recommended Operating Procedures

The following steps should be taken for a new detector and after each time a clean detector is installed. (See above for special requirements for radioactive sources.)

- ^{63}Ni constant current detectors are usually delivered already installed in the chromatograph. To re-install after cleaning, follow manufacturer's directions for setting up the instrument and conditioning the column [6, 8, 9]. Ensure that all heaters, temperature sensors, and electrical connectors are properly positioned. Never connect a column to a cool detector.

- Heat detector until it reaches the maximum operating temperature recommended by the manufacturer, then attach column and equilibrate overnight at operating temperature and flow rate.

- Determine the instrument attenuation required to cause 40-80% FSD in response to 1.5 ng chlorpyrifos.

- Determine detector dynamic response range to chlorpyrifos and other standards of interest by plotting response factor (response/unit weight) *vs* weight injected on a semilogarithmic scale. Do not operate instrument outside the dynamic response range.

Earlier Tracor models allow for determining the pulse frequency profile and saturation current. To operate these models, refer to the instructions in the manufacturer's operation manual. Newer models have these parameters preset, so adjustments are not necessary or possible.

To minimize interferences that can occur during determination with EC detectors, follow these rules:

- Exercise extreme caution to avoid introduction of contaminants from reagents, apparatus, and environmental sources; routine inclusion of reagent blanks in laboratory quality assurance procedures will monitor success of these precautions.

- Employ suitable cleanup procedures for extracts that will be examined by EC detectors. Elution through Florisil is usually required before EC determination, though even this is not a guarantee that artifacts from foods will not cause response.

- Always confirm the identity of residues that have been tentatively identified by EC GLC; confirmation may include GLC with element-selective detectors or other techniques (Section 103).

503 C: FLAME PHOTOMETRIC DETECTOR

The flame photometric detector (FPD) is the detector of choice for determination of organophosphorus residues and the only practical detector for organosulfur compounds. In its phosphorus-selective mode (FPD-P), the detector is one of the most element-selective GLC detectors available, although large amounts of sulfur will cause a response. The sulfur-selective mode (FPD-S) offers less selectivity (phosphorus can interfere) and is linear only on a semilogarithmic scale, but it provides useful confirmation for sulfur compounds tentatively identified by EC determination. Neither nitrogen nor chlorine cause any practical interference in either mode. In addition, the ratio of FPD-P and FPD-S responses can be used to calculate an analyte's P:S ratio for confirmatory purposes.

Methods designed for use with FPD determination sometimes include only minimal cleanup. However, column contamination can be caused by repeated injections of extracts from such methods, and the cautions outlined in Section 501 C must be observed.

The majority of FPD detectors in use in FDA laboratories were produced by three manufacturers: HP, Tremetrics (formerly Tracor), and Varian Associates. Discussion of detector characteristics in this section is limited to these models.

Principles

GLC column effluent is burned in a flame fed by a mixture of hydrogen and air. Characteristic optical emissions are produced when compounds containing phosphorus or sulfur are decomposed in the flame, and these emissions are viewed by a conventional photomultiplier tube through a narrow bandpass (interference) filter of appropriate wavelength. Choice of filter determines whether emissions produced by phosphorus or sulfur reach the photomultiplier tube. A filter with maximum transmittance at 526 nm, corresponding to the emission wavelength of HPO, permits detection of phosphorus compounds, while one with maximum transmittance at 394 nm, the emission wavelength of S_2, detects sulfur compounds. A single optical filter and photomultiplier tube may be used, or two filters and photomultiplier tubes can be assembled to permit response to both phosphorus and sulfur simultaneously.

Design

Figures 503-g and 503-h are diagrams of single and dual flame FPDs, respectively. In each, column effluent enters the detector from the bottom, where it is mixed with hydrogen gas. Air is added before the effluent and hydrogen emerge from the jet or at the same time they emerge. The emission from the resulting flame is measured by a photomultiplier tube after passing through the proper filter.

Figure 503-g
Single-Flame FPD

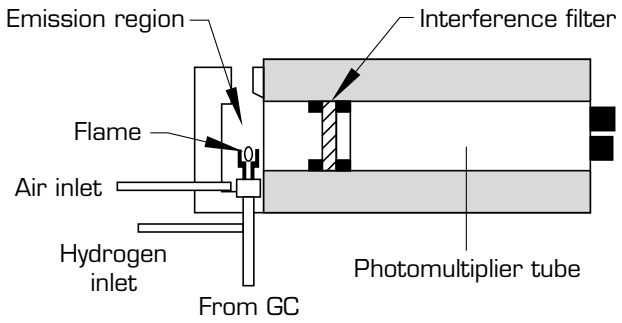


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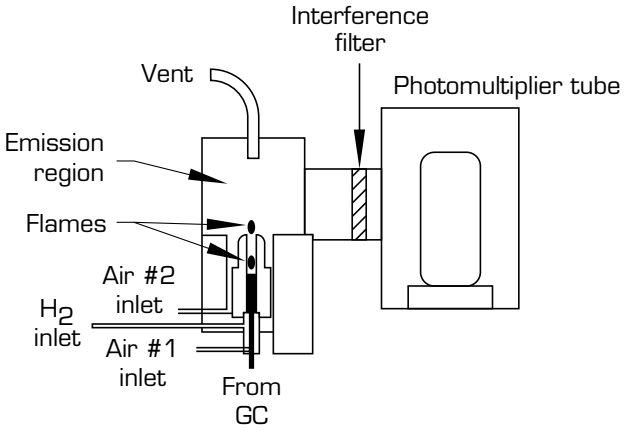
Figure 503-h
Dual-Flame FPD



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The Varian design differs from the others in that two air-hydrogen jets are used, "to separate the region of sample decomposition from the region of light emission" [10]. Sample decomposition occurs in the presence of air and hydrogen in the first combustion. Above that area, additional air is added to the combusted mixture; this supports a second flame, whose optical emission is filtered and measured as detector response.

Apparatus and Reagents

Section 501 B provides general information on apparatus and reagents required for GLC. Nitrogen or helium, of at least 99.998% purity, are usually used as carrier gases for columns connected to FPDs. Hydrogen and air are used as reactant gases in the detector flame. Manufacturers' recommendations for hydrogen purity vary from 99.995-99.999%. Air purity should be zero grade (maximum total hydrocarbon <2 ppm) or CGA Grade E [10-12].

Detector Characteristics

Sensitivity. The minimum amount of phosphorus detectable by the FPD-P is about 0.01 ng; for the FPD-S, about 0.04 ng sulfur. Detector sensitivity is greatly dependent on the condition of the photomultiplier tubes. Response varies among tubes, and the use of a variable voltage output with the power supply (or a variable voltage power supply) makes precise attainment of specific sensitivities easier to accomplish. Light leaking into a photomultiplier tube will increase the noise level and decrease the detector's effective sensitivity by making it less able to detect small amounts of analyte.

Response of the FPD-S (394 nm filter) to sulfur is proportional to the square of the concentration of sulfur. When the 526 nm filter is used (FPD-P mode), response to sulfur is also proportional to the square of the concentration. This relationship affects both selectivity ratios and linearity of the detector in both the P and S modes.

Selectivity. The response of the FPD-P detector is about 10^5 times greater to phosphorus than to carbon. The selectivity of phosphorus to sulfur in the FPD-P mode varies with the amount of sulfur present because of the square root relationship of response to sulfur concentration. For the Varian detector, P:S selectivity varies from $>10^4$ for very low concentrations of sulfur to about 50 for very high concentrations. Preliminary experience with the Varian detector indicates that it has a greater P:S selectivity than the other models. It is assumed that this increased selectivity is due to the stacked flame arrangement of this detector.

Selectivity of the FPD-S also varies with concentration of sulfur because of the square root relationship. The sulfur-to-carbon ratio varies from $>10^6$ at high sulfur concentration to 10^4 at low concentration. The S:P selectivity varies from $>10^4$ at high sulfur concentrations to 10 at low concentrations [10].

Selectivity of the FPD-S varies with the ability of the individual filter to screen out wavelengths associated with phosphorus. Experimentally, FPD-S detector response to a mixture of pesticides was compared using several different 394 nm filters. When conditions for each system were set to cause equal response to propargite (sulfur only), all filters permitted FPD-S response to methamidophos, chlorpyrifos, acephate, and omethoate (both phosphorus and sulfur), and most permitted response to monocrotophos (phosphorus only). Only one filter of those tested did not permit FPD-S response to monocrotophos; *i.e.*, the particular filter was far more selective to sulfur than the others. Further examination of the spectrum of light passed by the different 394 nm filters showed a distinct difference in the amount of absorbance at 526 nm; as expected, the filters that permitted FPD-S detection of monocrotophos passed much more 526 nm light than did the filter that did not detect it [13].

Linearity. Response of the FPD-P (526 nm filter) to phosphorus is linear over about four orders of magnitude.

Because of the square root relationship, response of the FPD-S to sulfur can be plotted as a straight line only if semilog paper is used. Most newer instruments provide a switch that automatically converts the detector output signal to its square root for an apparently linear response. However, quantitation using this converted signal is accurate only if the signal is carefully "zeroed," and the detector response is less sensitive at this setting. Many laboratories choose to measure the unconverted signal and plot response *vs* weight injected on semilog paper. Quantitation using the FPD-S is always less reliable than with other detectors.

Other Influences on Detector Performance

Detector Temperature. Each of the three manufacturers recommends a minimum detector operating temperature of 120° C. Recommended maxima range from 250° C (Tremetrics) to 350° C (Varian), with HP intermediate at 300° C.

Physical deterioration of parts of the detector can occur or be accelerated at higher temperatures. Both O rings and the casing for the photomultiplier tube have been seen to deteriorate at high temperatures. During routine operation, O rings should be changed periodically (about every 6-12 mon); all O rings in a particular detector should be changed at the same time. All manufacturers warn against continued operation at maximum temperature. Normal detector operating temperature should be about 20° C above that of the column, usually ≤250° C.

Gas Flow Rate. Optimum gas flow rate varies among detectors, and directions provided by the manufacturer of the specific detector should be followed. Additional experimentation may be required to optimize flow rates for any particular detector.

Detector Voltage. Satisfactory operation of the FPD requires use of a highly stabilized voltage power supply. Depending on the manufacturer, voltage may range from 350-850 V and may be obtained from either a variable or set voltage

supply source. Detectors are usually shipped with the recommended voltage preset at the factory. Manufacturers' operations manuals give specific instructions for varying voltage when this is an option.

Sensitivity of the detector can be increased by increasing the voltage, but an upper limit is imposed by the simultaneously increasing noise. Optimum voltage can be determined by comparing detector response to an amount of compound as the voltage is varied.

Adequate Chromatography. Nonlinear response of FPD-P to oxygen analogs of organophosphorus pesticides (P=O compounds) is often noted. Because the detector (526 nm filter) is linear over a wide range for P=S compounds (*i.e.*, most parent organophosphorus pesticides), the difficulty is assumed to be caused by degradation of P=O compounds. Once attributed to a defect in detector design, this problem is now considered to be caused by poor chromatography of these polar compounds, and thus a column problem. Use of wide bore capillary columns (Section 502 C) minimizes the effect.

Recommended Operating Procedures

FPD-P. The following steps should be taken for detector operation:

- Install detector if necessary, according to instructions provided in manufacturer's manual [10-12]. FPD usually comes installed in chromatograph.
- Set detector temperature as recommended by manufacturer, at least 20° C above column temperature.
- Establish flow rate of column carrier gas as suitable for proper column operation (Section 502). Set flows of hydrogen and air as recommended by detector manufacturer or as determined from experimentation to provide optimum operation. Connect column to detector.
- If voltage is set by user, follow manufacturer's directions.
- Ignite flame after all instrument temperatures are equilibrated and with carrier gas flowing into detector.
- Turn on air.
- Depress ignitor and hold.
- Slowly turn on hydrogen.
- Release ignitor after hydrogen has been turned completely on. Slight increase in signal should occur when flame is ignited. Alternatively, check for lighted flame by holding mirror or other shiny object at exhaust end of detector. Presence of condensed moisture indicates that flame is present.
- If flame does not light, turn off hydrogen and repeat previous steps. Increasing air flow and/or decreasing carrier flow may help in igniting flame.

- Turn on auxiliary gas if needed.

- Determine detector response by injecting 1.5 ng chlorpyrifos. Adjust electrometer sensitivity so that 1.5 ng gives about 50% FSD (40-80% is satisfactory). Adjust voltage to change response, if variable power supply is available. At given voltage, changes in flow rates may improve sensitivity and chromatography.

FPD-S.

- Follow procedures above for FPD-P, but use 394 nm filter.

- If more than one 394 nm filter is available, test to determine which is most selective to sulfur over phosphorus by injecting mixture of methamidophos, chlorpyrifos, acephate, omethoate (each containing S and P), and monocrotophos (P only). A filter that does not permit response to monocrotophos, or that permits least response to it, is the best choice for sulfur selectivity.

- For greatest sensitivity, do not use electrometer square root function; instead, plot response *vs* amount injected on semilog paper and quantitate from that calibration. FPD-S is sufficiently insensitive that it should be set up to provide the greatest sensitivity possible while still maintaining reasonable baseline noise; this will vary from instrument to instrument.

Troubleshooting

Consult the manufacturer's operation and service manual for recommendations specific to detector model being used. Note the following additional suggestions:

Symptom	Possible Solution
Noisy and/or wandering baseline	Install flow controllers to prevent gas flow fluctuation; normal baseline is very straight with <1% noise in P mode and <2% in S mode.
	Check by shining flashlight on detector. Recorder will show response if leak exists. Replace O rings. If this does not work, seal light leaks with black tape or other material. Photomultiplier tube should never be exposed to light when connected to power supply or it will burn out.
	Clean detector.
Low sensitivity	Check for photomultiplier tube light leaks as above.
Peak broadening or tailing, poor response reproducibility	Improve chromatography by changing to capillary column or other column suited to chemistry of analyte.
	Rejuvenate capillary column.

503 D: ELECTROLYTIC CONDUCTIVITY DETECTOR

The electrolytic conductivity detector (ElCD) is capable of operating in modes selective to halogen, nitrogen, or sulfur. ElCDs can also be configured for selective detection of nitrosamines or esters or for the nonspecific detection of carbon-containing compounds.

For pesticide residue determination, the ElCD is most often used in the halogen mode (ElCD-X), where it exhibits much greater selectivity to halogen than does the EC detector and yet responds to <1 ng of most organohalogen pesticide residues in foods. Although ElCD in the nitrogen mode (ElCD-N) has been shown suitably sensitive for use in residue determination, it is used routinely for that purpose in only a few laboratories, because adequate operation is more difficult to establish and maintain. In addition, problems associated with interferences from nitrogen-containing commodity co-extractives (Section 503 A) apply to ElCD-N.

Only ElCDs in the halogen and nitrogen modes are discussed in detail in this section.

Presently, two manufacturers market ElCDs, Tremetrics and OI Corp., College Station, TX. The Tremetrics "Hall 1000" and "Hall 2000" replaced the original "Hall 700A," which was marketed by Tracor Inc. (now Tremetrics); the latter model is no longer commercially available but continues to be used in many residue laboratories. OI markets the "4420" and a newer "5200." FDA experience is limited to the Hall 700A, Hall 1000, and OI 4420, so only these models will be discussed in this chapter.

Principles

GLC column effluent is pyrolyzed in a nickel reaction tube at >800° C in the presence of hydrogen reactant gas. Heat causes most of the compounds in the reaction tube to be pyrolyzed to their elemental form, but the presence of reactant gas results in other chemical reactions. Products formed during reaction of the analytes are either removed by appropriate scrubbers prior to entering a conductivity cell or are swept into the conductivity cell *via* carrier gas where they are dissolved in a circulating conductivity solvent (electrolyte).

In the conductivity cell, electrolyte conductivity is constantly monitored for changes caused by dissolution of the reaction products. Change in conductivity is converted to a voltage signal that produces an electrical peak at the detector output. ElCD hardware is configured into various operating modes by appropriate selection of reactant gas, electrolyte, ion exchange resin, and chemical scrubbers used to remove interferences. Detector sensitivity is affected by reaction conditions as well as by electrolyte flow rate and reactant gas flow rate.

Design

Figure 503-i displays a block diagram of a GLC system with ElCD; terminology is generalized to display the basic system arrangement that applies to all models of ElCD; some design differences exist between models. Figure 503-j displays a simplified diagram of the ElCD reactor and conductivity cell.

Figure 503-i
Block Diagram of the EICD

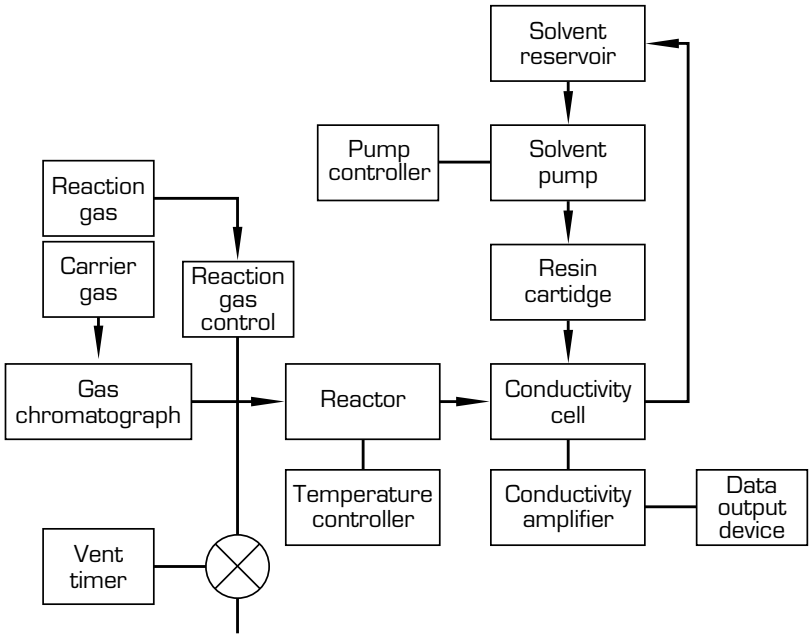

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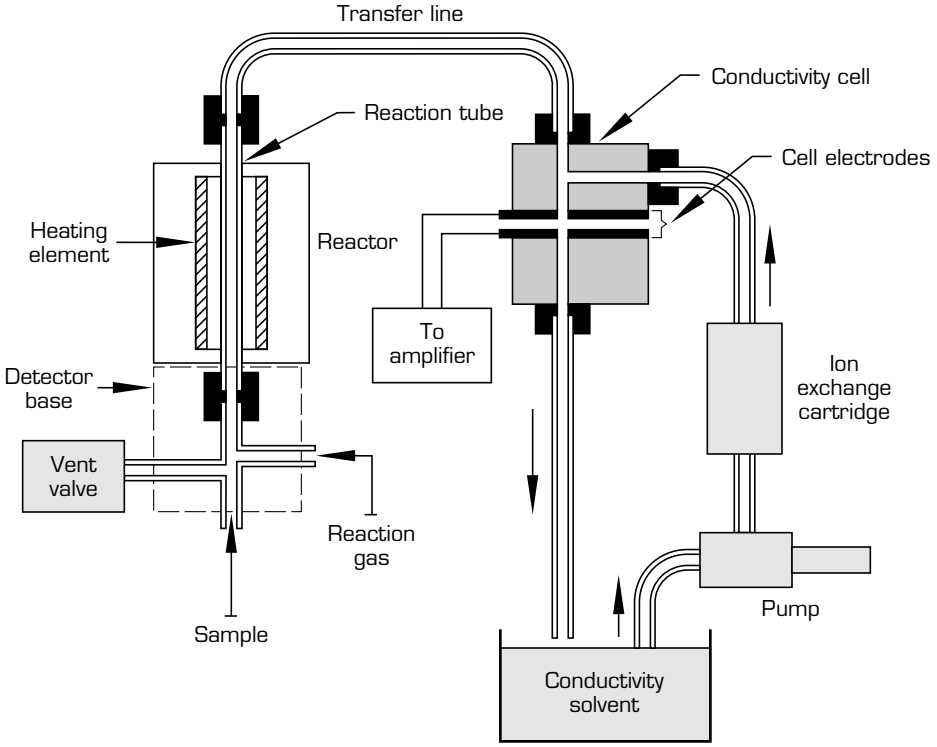
Figure 503-j
EICD Reactor and Conductivity Cell



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Each model ElCD includes four major units:

1) Reactor unit, including reactant gas inlet, nickel reaction tube, and solvent vent to prevent injection solvent from entering the reaction tube.

2) Electrolyte unit, including solvent reservoir, ion exchange tube, and solvent pump. The OI 4420 uses resin in two stages, a "roughing" resin within the electrolyte reservoir and a "finishing" resin through which the electrolyte passes before entering the conductivity cell.

3) Conductivity cell.

4) Signal processing unit, which converts conductivity cell signal for display and provides power for other detector components.

The Hall 700A differs from other ElCDs in that it has a reference cell measuring the conductivity of the electrolyte without any dissolved reaction products; electrolyte conductivity is subtracted from that of the analytical cell, so that background signal is removed from the final measurement. The Hall 1000 and OI 4420 measure absolute conductivity with no subtraction of signal related to electrolyte conductivity.

ELCD-X

Principles

ElCD response in the halogen mode results from formation of HF, HCl, HBr, or HI by catalytic reduction of analytes containing fluorine, chlorine, bromine, or iodine, respectively. The heated nickel reaction tube provides all the ingredients for reaction: a chamber for mixing analyte and hydrogen gas, heat, and the nickel surface for catalysis.

Carrier gas transports the acid formed in the reaction tube into a conductivity cell. The acid dissolves in deionized n-propanol electrolyte, increasing electrolyte conductivity and producing a measurable response (peak) at the detector output.

To prevent neutralization of the acid formed in the reaction tube, pH of the n-propanol electrolyte must also be slightly acidic. Electrolyte acidity is maintained by circulation through ion exchange resin.

Apparatus and Reagents

Section 501 B provides general information on apparatus and reagents required for GLC. Consult appropriate instrument manuals for purchasing information and proper procedure for replacing the following reaction tubes, resins, scrubbers, and electrolyte:

additional nickel reaction tubes. Tubes are purchased from the instrument manufacturer or from other suppliers of nickel tubing. Use of tubing not designed for analytical purposes will probably require additional cleaning and/or polishing to be suitable [14]; laboratory prepared tubing has not been successfully used in the OI 4420 models.

gases, helium, ultra high purity, 99.999%, used as column carrier gas; hydrogen, ultra high purity, 99.999%, used as reactant gas

gas filters, capable of removing oxygen and water from carrier gas

n-propanol, distilled from all-glass apparatus, for electrolyte

resin materials, to replace resin tube when selectivity deteriorates. Some extra resin material should be provided with the detector and more can be ordered from the manufacturer, or a system that replaces the whole unit can be purchased to avoid the inconvenience of replacing the resin [15-17]. Consult appropriate detector manual for instructions on replacing resin. (Resin and electrolyte are sold as a unit by Tremetrics, Inc., because the resin bed is contained within the conductivity reservoir.)

Resins are subject to deterioration and are dated; they may be unsuitable for use after storage, even if refrigerated as directed.

Detector Characteristics

Sensitivity. An FDA interlaboratory trial involving eight Hall 700A ElCDs in the halogen mode, each operated at the same basic parameters, showed that each detector was different in terms of the minimum amount of halogenated material to which it would respond [18]. In this study, the most responsive detector was 10-25 times more sensitive than the least responsive detector to the same amount of the same compound. However, each properly functioning detector was capable of detecting 0.05 ppm lindane in the presence of sample extract. The more sensitive detectors could readily measure 0.01 ppm lindane. No comparable study has been performed with the other model ElCDs.

The following parameters affect ElCD sensitivity:

1) Reactant gas purity. Impurities in the reactant gas can undergo chemical reaction in the reaction tube, and resulting products may be soluble in the electrolyte. If this occurs, conductivity may be raised sufficiently to obscure measurement of small amounts of analyte (*i.e.*, the signal-to-noise ratio will be reduced). Adherence to manufacturers' purity recommendations is critical. Gases meeting manufacturers' specifications occasionally contain traces of hydrochloric acid that can destroy the detectors; use of appropriate gas filters is required even on high purity gases.

2) Reactant gas flow rate. ElCD-X response was shown to increase with increasing reactant gas flow rate, up to about 60 mL/min, in a study of the Hall 700A ElCD [19]. Above that flow rate, response remained essentially constant up to 100 mL/min. Manufacturers suggest reactant gas flow rate of 50-75 mL/min for the Hall 700A [15], 25 mL/min for the Hall 1000 [16], and 100 ± 10 mL/min for the OI 4420 [17]. FDA laboratories routinely use 60-80 mL/min for the Hall 700A and the Hall 1000 and 100 mL/min for the OI 4420.

3) Reaction tube condition. The nickel reaction tube catalyzes the reaction and must be free of contamination. After a period of use, nickel tubes become contaminated because of fouling by bad gases, sample reaction products, septum or column bleed, or other causes; chromatograms at this point characteristically show a slow return to baseline after venting, peak tailing, and loss of response. Once contaminated to this degree, the tube must be replaced, because no successful reconditioning process has been developed.

4) Reaction temperature. In a study using the Hall 700A in the halogen mode, reaction temperature was not found to affect detector sensitivity significantly. No significant differences in detector response occurred when chlorinated compounds were injected at temperature control potentiometer settings of 850, 730, and 630° C [19]. However, a minimum potentiometer setting of 900° C is recommended for ElCD-X, because it is suspected that potentiometer setting is not an accurate reflection of the actual temperature of the reaction tube and because it is reasonable to assume that more efficient reduction of halogen occurs at higher temperatures. Reaction tubes whose operation is compromised by other problems (*e.g.*, contamination from samples, column bleed, or poor quality nickel tube) may show fluctuations in sensitivity with changes in temperature.

   Reaction furnaces ("reactors") of OI 4420 detectors have been subject to repeated burnout, requiring replacement. The manufacturer offers a smaller, cartridge-style heating element as a reactor replacement in an upgrade to the detector; this model is also expected to have a limited lifetime but will be easier and less expensive to replace than the original reactors.

5) Electrolyte flow rate. Electrolyte flow rate significantly affects detector sensitivity by affecting the length of time the dissolved reaction products spend in the conductivity cell. As the electrolyte flow decreases, response increases. Below a certain flow rate, however, baseline noise increases and further decrease in flow rate results in no additional improvement in the signal-to-noise ratio.

   Respective manufacturers recommend 0.5 mL/min electrolyte flow rate for the Hall 700A, 0.6 mL/min for the Hall 1000, and 0.02-0.05 mL/min for the OI 4420. FDA laboratories usually use 0.35 ± 10% mL/min flow for the Hall 700A and the Hall 1000 and 0.035-0.050 mL/min for the OI 4420.

Selectivity. The ElCD is made selective to halogens by using hydrogen reactant gas and n-propanol electrolyte. HX, formed by pyrolysis of halogenated compounds in the presence of hydrogen, is readily soluble in n-propanol. Other compounds formed in the reactor, such as H_2S and NH_3, do not usually cause detector response because they are not ionized in n-propanol and therefore cannot change solvent conductivity. No scrubber is needed to remove interfering reaction products from the gas flow in the halogen mode. Large quantities of nitrogen and possibly carbon dioxide may cause a response, however.

(An optional oxidative mode operation for halogen selectivity, using oxygen as reactant gas, is far less selective, produces greater noise, and requires use of scrubbers capable of removing SO_2/SO_3 from the reaction products. This operation has

never been implemented for residue determination because it is far less preferable than the reductive mode.)

Because detector selectivity to halogen is dependent on reaction conditions and on solubility of reaction products in the electrolyte, all the following factors affect selectivity:

1) Gas purity. Impurities in column carrier gas and/or hydrogen reactant gas can introduce other chemical species that may interfere with halogen detection; only ultrapure gases are acceptable. Gases must be free of any level of halogenated compounds.

2) Reaction tube condition. The nickel reaction tubes that contain and catalyze the reaction are known to vary from one another in their ability to convert halogen to HX. A tube that initially produces an acceptable response may deteriorate over a period of use because of contamination. As previously discussed, the tube must be replaced when this occurs.

3) Reaction temperature. Reaction temperature may affect selectivity by influencing the degree to which reduction of halogen to HX occurs. Reaction temperature setting of 900° C is recommended to achieve efficient reduction.

4) Ion exchange resin. The ion exchange resin affects selectivity by controlling pH of the electrolyte and by continuously removing ionized reaction products from the electrolyte. The resin used for the halogen mode maintains the n-propanol electrolyte at a slightly acidic pH. Presence of the ionized HX then produces a measurable change in solvent conductivity. When the resin fails to control pH of the electrolyte appropriately, certain species other than HX are also able to ionize, and detector selectivity deteriorates. Failure to maintain slightly acidic electrolyte results in negative or "V"-shaped peaks. Replacement of the resin with fresh material re-establishes the necessary selectivity.

For unknown reasons, addition of n-propanol to the reservoir of the OI 4420, to replace evaporated solvent, can cause severe damage to the resin. When this occurs, resin and electrolyte must be replaced [20].

Linearity. Linear dynamic range of the Hall 700A ElCD-X varies with the compound and with the individual detector [19]. Moreover, the typical degree of linearity and length of linear range are not sufficiently reliable to eliminate the need for matching peak heights of residue and standard when quantitating. Each system should be tested to measure its linear range. For accurate quantitation of residues, peak sizes must be within 25% of one another.

Other Influences on Detector Performance

Solvent Venting. ElCD reactor units include a vent line positioned just before the heated reaction tube. The relatively large volume of injection solvent, eluting through the column prior to the analytes, is diverted through this port to prevent its entry into the reaction tube. Venting prevents combustion of hydrocarbon solvent in the reaction tube and thus protects the tube from carbon deposition that decreases catalytic performance, nickel tube lifetime, and detector response.

Contamination of the reaction tube and transfer line is most severe when acetone is used and then not completely vented.

Efficiency of venting is affected by several factors, including reactant gas flow rate, combustion tube diameter, and the percentage of flow that is vented. Both increased flow of reactant gas and decreased combustion tube id improve vent efficiency by increasing back pressure.

The percentage of total effluent that is vented is critical. While efficient venting of most solvent is necessary, not all solvent can be vented, because lack of positive pressure permits electrolyte to flow from the reservoir and enter the nickel reaction tube, contaminating it. Vent rate is preset by the manufacturer, but the OI 4420 includes a vent system with a pressure-regulated "T" that permits the user to adjust the vent rate by turning a threaded rod. Manufacturer directions specify adjusting the rate so that about 50-60 mL/min total gas flow exits the vent; frequent monitoring of the flow is necessary, because the vent split ratio fluctuates.

Many of the recurring problems with the OI 4420 detector were traced to the vent system. It is now recommended that the original vent flow valve be replaced with a constant flow port, which is capable of maintaining a constant vent flow while the vent is open; the detector upgrade offered by the manufacturer includes this replacement. Even this change, however, does not vent most of the solvent, and its combustion in the reaction tube may cause subsequent problems [20].

Position of Capillary Column. Results observed during evaluation of a capillary column with OI 4420 ElCD-X indicate that the most critical element for successful operation is proper positioning of the column outlet in the reactor [21, 22]. When the capillary column is installed as directed in the detector manual (*i.e.*, column outlet placed about 0.5" into the nickel reaction tube) a noisy baseline with frequent "spiking" is observed.

These studies demonstrate that positioning the column outlet between the solvent vent and the reactant gas inlet produces optimal results. In this position, the column is outside the nickel tube and away from the extremely high temperatures of the reactor. This positioning also ensures efficient venting, because the reactant gas takes the path of least resistance and flushes the injection solvent through the vent rather than through the small id transfer line to the combustion tube; the possibility of tube contamination is thus reduced. A steady baseline is maintained when the column is installed between the vent and the reactant gas inlet. This same positioning is also optimal when wide bore capillary columns are used. A redesigned base for mounting the detector on the chromatograph may minimize the importance of user attention to positioning the column. Operation of ElCDs with packed columns is not as sensitive to column position.

Transfer Line Cleanliness. More often than not, broad, tailing peaks are caused by a dirty transfer line between the reactor and the conductivity cell. Contaminants (*i.e.*, unreacted hydrocarbons) can deposit in the transfer line and produce active (adsorptive) sites. When hydrochloric or other gaseous acids pass through the transfer line, they may be adsorbed; this phenomenon causes tailing or, in severe cases, total loss of detector response. Transfer lines can be rinsed with the injection solvent being used [23]; the ease with which this can be performed varies with the detector model.

Recommended Operating Procedures

Except for positioning the column in the reactor (above), follow directions from the appropriate detector manual to set up and operate the GLC system with ElCD. The following additional recommendations are based on experiences in FDA laboratories:

- Reaction tube temperature: Set potentiometer that controls reaction tube temperature to 900° C to ensure that temperature is high enough to completely reduce analytes. To protect reaction tube from possible deactivation by column bleed, do not allow reactor temperature to drop below that of column.

- Reactant gas flow: Maintain hydrogen flow of about 60-100 mL/min through reaction tube to ensure complete reduction of sample. Measure flow rate using bubble meter and stopwatch, either at point where gases enter conductivity cell (with column carrier gas turned off) or at return line to solvent reservoir (with both column carrier gas and solvent pump turned off). Column temperature should be reduced to room temperature if carrier gas is off for any length of time.

- Solvent flow rate: For optimum performance, pump n-propanol electrolyte through conductivity cell at 0.35 ± 10% mL/min for Hall 700A and Hall 1000 and 0.035-0.050 mL/min for OI 4420 ElCD-Xs. Measure flow rate by placing line that usually carries solvent to reservoir into a graduated cylinder and measuring accumulation over known time.

System Suitability Test

Monitor detector selectivity by regularly injecting aliquot of mixed standard solution containing the following: 100 ng diisobutyl phthalate, 100 ng ethion, 1 ng chlorpyrifos, 100 ng methyl palmitate, 100 ng caffeine, and 2 µg octadecane. Properly operating detector will respond only to chlorpyrifos and possibly to caffeine. If response to caffeine is seen, calculate selectivity ratio as:

$$\frac{\text{detector response to chlorpyrifos} \times 100}{\text{detector response to caffeine}}$$

If selectivity ratio for chlorpyrifos:caffeine is <500:1 or if any response to other compounds is seen, improve selectivity by following the suggestions for troubleshooting, below.

Routinely monitor detector response to halogen by injecting solutions containing at least lindane, chlorpyrifos, and p,p′-DDT. If response decreases, follow directions in troubleshooting section to determine cause. While monitoring halogen response, also note peak shapes on chromatograms. Deteriorating shape (*i.e.*, increased tailing) of all peaks may be caused by various factors covered in troubleshooting section. Breakdown of p,p′-DDT (evidenced by smaller peak plus appearance of another peak at retention time of p,p′-TDE) has been found to be caused by prior injection of extracts prepared by the method of Section 302. This condition disappears over time if no further extracts from that method are injected.

Troubleshooting

Each detector manual [15-17] contains a section on troubleshooting that should be consulted when problems occur. Another reference [24] also contains excellent information on operation of the ElCD and potential problem areas. Beyond the advice offered in those references, these additional suggestions for optimum detector performance are offered:

Symptom	Possible Solution
Selectivity of chlorpyrifos: caffeine is <500:1	Replace resin in cartridge and n-propanol electrolyte.
	Change nickel reaction tube. Several different tubes may have to be tried, because each converts halogen to HX to different extent.
	Elevate reaction tube temperature to determine whether sample is being completely reacted.
	Replace column if liquid phase contains halogen or nitrogen.
Loss of detector sensitivity	Verify purity of gases; use only ultra high purity gases.
	Remove transfer line and rinse with injection solvent or replace transfer line.
	Replace column if liquid phase contains halogen or nitrogen.
Breakdown of p,p′-DDT to p,p′-TDE	Remove first 1-2" of packing from GLC column and replace with fresh, conditioned packing, *i.e.*, clean front end of column (Section 502).
Broad, tailing peaks	Replace or clean transfer line.
	Clean front end of column.
	Replace nickel reaction tube.
	Replace column if liquid phase contains halogen or nitrogen.
Slow return to baseline after venting	Replace nickel reaction tube.
Breakdown of analytes (but normal return to baseline after venting, normal peak shape)	Clean front end of column.

ElCD-N

Principles

ElCD response in the nitrogen mode results when organic nitrogen is pyrolyzed to ammonia with hydrogen reactant gas at high temperature. Acidic or sulfur gases, resulting from pyrolytic reduction of halogen and sulfur compounds in the reactor, are selectively removed by quartz threads coated with potassium hydroxide. Carrier gas transports the ammonia into the conductivity cell, where it dissolves in an aqueous electrolyte to form ammonium hydroxide:

$$NH_3 + H_2O \longrightarrow NH_4^+ + OH^-$$

As a weak base, ammonium hydroxide readily ionizes in the aqueous electrolyte and becomes a conducting species. The change in electrolyte conductivity caused by dissolution/ionization of ammonia produces a measurable response (peak) at the detector output.

Apparatus and Reagents

Section 501 B provides general information on apparatus and reagents required for GLC. The section above on ElCD-X provides additional information about replacement nickel reaction tubes, gas purity, and gas filters. Consult appropriate instrument manuals for purchasing information and proper procedure for replacing the following reaction tubes, resins, scrubbers, and electrolyte:

stainless steel, nickel, or copper gas lines from gas cylinders to the instrument

water, HPLC grade, prepared from water purification equipment, or equivalent commercial product; 18 megaohm resistance required

fresh electrolyte, prepared from reagent grade t-butanol and HPLC grade water

additional scrubbers for use in nitrogen mode. Nitrogen scrubber generally lasts 3-6 mon. However, under certain circumstances scrubber may last <1 mon.

resin, may be ordered from detector manufacturer, as described above for ElCD-X. Nitrogen mode operation is particularly sensitive to failures caused by resin deterioration, so use of fresh resin is critical.

Detector Characteristics

Sensitivity. The ElCD-N detector is capable of producing as much as 50% FSD response to 1 ng carbaryl or 0.5-1 ng chlorpyrifos. As with the ElCD-X operation, sensitivity of the ElCD-N depends on reaction conditions, reactant gas flow rate, and electrolyte flow rate. In addition, the condition of the chemical scrubber used in the nitrogen mode will affect detector sensitivity.

The control module (signal processing unit) of the OI 4420 offers several sensitivity settings, labelled according to the different modes of operation. Despite the

label, the setting for the halogen mode should be used during nitrogen mode operation for greatest sensitivity.

Selectivity. Selectivity for N:P is much better than that of the N/P detector and is the chief virtue of the ElCD-N; selectivity of the OI 4420 in the nitrogen mode was found to be 2400:1 for chlorpyrifos (molecular formula $C_9H_{11}Cl_3NO_3PS$):bromophos ($C_8H_8BrCl_2O_3PS$) [25]. Selectivity for N:X is also very high as long as the scrubber is efficiently removing HX from the reaction products prior to dissolution in electrolyte; N:X selectivity is higher in this mode than is X:N selectivity in the halogen mode. Selectivity was found to be 4800:1 for chlorpyrifos: aldrin ($C_{12}H_8Cl_6$)[25]. As with the ElCD-X operation, selectivity of the ElCD-N is affected by reaction conditions and by parameters that affect ionization of the reaction products in the electrolyte.

Any parameter that affects the conversion of organic nitrogen to NH_3 influences selectivity. The most important of these parameters are reactant gas purity, condition of the reaction tube, and reaction temperature. In addition, the chemical scrubber (*i.e.*, quartz threads coated with potassium hydroxide), placed between the reactor and conductivity cell, prevents interferences from reaching the electrolyte, dissolving in it, and causing detector response.

Conditions that affect ionization of the reaction products, including nature of the ion exchange resin and electrolyte type and pH, also influence detector selectivity. Electrolyte pH must be slightly basic to prevent neutralization of the basic ammonium hydroxide; appropriate pH is maintained by passing the electrolyte through an ion exchange resin before it enters the conductivity cell. Aqueous electrolyte is used in the nitrogen mode because water is one of the few neutral solvents capable of ionizing a weak electrolyte like ammonia. Water purity is critical to detector selectivity; the high purity water specified above in Apparatus and Reagents is necessary.

Incorporation of carbon dioxide in the electrolyte affects its pH and thus detector selectivity. Carbon dioxide can enter the electrolyte from improper venting or from permeation through tubing during transfer to the conductivity cell. Early versions of the OI 4420 included a permeation chamber filled with ammonia in water, through which the Teflon tubing transferring the electrolyte passed. This arrangement was used only for nitrogen mode operation, with the intent of permitting ammonia to permeate the electrolyte and keep it sufficiently basic, but it was not satisfactory and the permeation chamber is no longer included with the system. The Hall 2000 uses a stainless steel transfer line to minimize permeation of gases into the electrolyte. Purging the electrolyte with hydrogen or helium for 1 hr, after t-butanol and water are mixed, may also be used to dispel carbon dioxide and air and improve detector performance [26].

Linearity. The linearity of ElCD-N detector response to any particular chemical is approximately three orders of magnitude, within the range of 10 pg-100 ng. Response to each chemical, depending on its chromatography and percentage nitrogen, has a lower threshold of linearity; below that level, response can be measured but is not linear. Response to an amount beyond the upper limit of linearity often appears as a double peak [20].

Other Influences on Detector Performance

Factors that influence operation of the ElCD-X detector, *i.e.*, venting efficiency and transfer line cleanliness, are also important parameters in ElCD-N operation,

although use of a scrubber in the nitrogen mode minimizes transfer line contamination. As with the ElCD-X, the optimal position for a capillary column in the OI 4420 detector was found to be between the vent line and the reactant gas inlet [25]. In addition, the detector operations described below are critical to acceptable detector operation.

Composition of Electrolyte. An important parameter for acceptable performance is electrolyte composition [25]. During early studies of the OI 4420, 50% n-propanol/water was recommended as a replacement for 0.1% hexanol/deionized water, originally recommended by the manufacturer. In the meantime, however, 10% t-butanol/water has become the recommended electrolyte for OI 4420 nitrogen mode operation; 50% n-propanol/water is recommended for Hall 700A and Hall 1000 detectors. As discussed above, water purity is critical in the nitrogen mode.

Condition of Scrubber. The scrubber can become exhausted and must be replaced when the detector begins responding to halogenated compounds. Solvents containing halogen or sulfur should not be used in the nitrogen mode because they will rapidly deplete the scrubber.

Recommended Operating Procedures

Follow the explicit directions from the appropriate detector manual to set up and operate the GLC system with ElCD, and incorporate special directions discussed above, including use of high purity (18 megaohm resistance) water and hydrogen purging of the mixed electrolyte prior to use, whenever electrolyte is changed.

System Suitability Test

Currently, there is no standardized system suitability check performed by FDA laboratories for ElCD-N detectors. Suggested system suitability tests may be found in detector operation manuals. It is recommended that a solution containing at least one compound containing nitrogen as the only heteroatom, one halogenated compound, and one hydrocarbon be injected into the system; a properly functioning system should show no response to the halogenated compound or to the hydrocarbon and should have no inverted (below baseline) peaks.

Troubleshooting

Detector operations manuals and Reference 24 each contain sections on troubleshooting. In addition, the following suggestions for optimum detector performance are based on the FDA evaluation of wide bore column and OI 4420 ElCD-N for determination of nitrogen-containing pesticide residues in food:

Symptom	Possible Solution
Peak tailing	Replace scrubber.
	Replace nickel reaction tube.
	Replace older OI 4420 detector base.
Poor linearity	Replace scrubber.

	Replace electrolyte.
	Replace older OI 4420 detector base.
Excessive noise	Replace electrolyte.
	Replace gas line filters.
	Replace gas.
	Check for temperature fluctuations and correct as necessary.
	Replace nickel reaction tube.
	Check for and correct gas flow instabilities.
	Remove bubbles in OI cell by turning pump switch off for 1-2 sec, then turning on, or increase pump speed to maximum for 1-2 min.

General Precautions for ElCDs

The following precautions should be followed to ensure optimum performance of the ElCD in both halogen and nitrogen modes:

- Avoid column liquid phases that contain halogen or nitrogen, because the phase may bleed and de-activate the reaction tube and/or raise the conductivity of the electrolyte.

- Avoid injecting standards or sample extracts in solvents containing halogen or nitrogen. Even though the solvent is vented, traces may remain and affect detector operation. This effect becomes critical in cases where detector selectivity is already poor.

- Maintain constant carrier gas and reactant gas flow at all times. Reducing gas flows overnight to conserve gas may result in diminished responses when detector conditions are re-established the next day.

- When carrier gas flow must be interrupted, *e.g.*, to change columns or septa, cool reactor furnace first. Exposure of nickel reaction tube to oxygen at high temperature invariably damages performance and usually requires subsequent replacement of tube. Before reheating furnace, thoroughly purge system with carrier gas; 15 min is sufficient when capillary columns are used.

- Do not allow solvent return line to dip below surface of solvent in reservoir. Violating this rule will lead to backup of solvent into reaction tube anytime gas flow is inadvertently stopped.

- Vent injection long enough to ensure removal of solvents or volatile sample co-extractives that can interfere with determination. Adequate venting also protects reaction tube and conductivity cell. Vent time of 0.5-0.75 min is adequate for wide bore column and Hall 700A detector; 0.75->1.3 min is required for OI 4420.

503 E: NITROGEN/PHOSPHORUS DETECTOR

The nitrogen/phosphorus (N/P) detector is selective to residues containing nitrogen and/or phosphorus atoms. Modern N/P detectors evolved from Kolb and Bischoff's 1974 design [27], itself an evolution of the potassium chloride thermionic detector (KClTD); the KClTD, introduced in the mid-1960s, was the first selective detector for phosphorus residues [28]. Most N/P detectors are more responsive to phosphorus than to nitrogen, but this section emphasizes use as a nitrogen-selective detector, because the FPD-P (Section 503 C) is preferred for phosphorus residues.

Although N/P detectors are selective and sensitive, problems associated with their reliability and performance have deterred their routine application for pesticide residue determination in FDA laboratories. In addition, the N/P's ability to distinguish residues from sample matrix unequivocally is hindered by the presence of nitrogen in many commodity co-extractives, a dilemma common to all nitrogen detectors. Despite these shortcomings, an N/P detector, optimized for nitrogen selectivity, can play a valuable role in examining extracts for residues; many pesticides contain no other heteroatom than nitrogen. Response of the N/P detector also provides complementary evidence about element(s) present in a residue, information often needed for confirmation of identity (Section 103, Table 103-a).

Several different manufacturers produce N/P detectors. Among these are: Chrompack, Inc., Raritan, NJ; DETector Engineering & Technology, Inc., Walnut Creek, CA; Hewlett-Packard Company, Wilmington, DE; Perkin Elmer Corporation, Instrument Division, Norwalk, CT; Shimadzu Scientific Instruments, Inc., Columbia, MD; Tremetrics, Inc., Austin, TX; and Varian Instrument Division, Walnut Creek, CA.

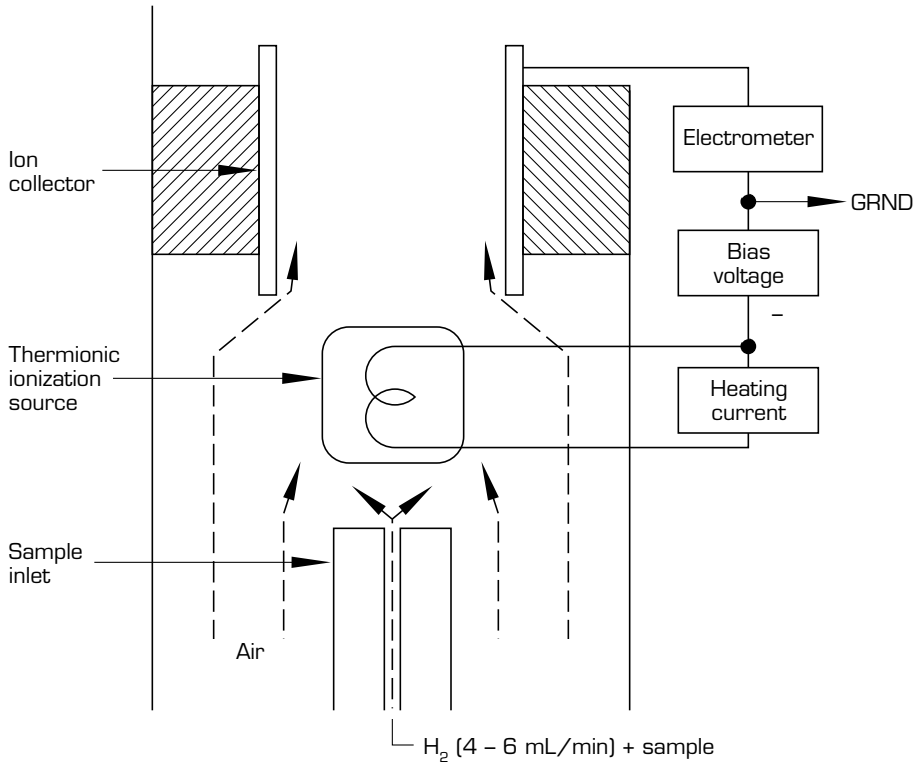
Principles

GLC column effluent impinges onto the surface of an electrically heated and polarized alkali source in the presence of an air/hydrogen plasma; ionization occurs and the flow of ions between plasma and an ion collector is amplified and recorded. Detector response to analytes results from the increased ionization that occurs when compounds containing nitrogen or phosphorus elute from the column. At gas flow rates used for N/P operation, the degree of ionization of compounds containing nitrogen or phosphorus is >10,000 times greater than for hydrocarbons. Mechanisms that explain the enhanced response to nitrogen and phosphorus are not yet fully understood and are beyond the scope of this manual; both gas phase ionization and surface ionization processes have been proposed [29].

Design

An N/P detector is similar to an FID to which an electrically heated source of alkali has been added between the jet and the ion collector; Figure 503-k provides a schematic diagram of typical components. Commercially available N/P designs vary considerably, with different collector electrodes, collector polarity, and optimum potential between jet and collector; Figure 503-l displays several of these variations. The most important component, the alkali source, is usually manufactured by impregnating a glass or ceramic matrix with an alkali metal salt. Variations among alkali source designs represent attempts to optimize selectivity to

Figure 503-k
N/P Detector Components



[Reprinted with permission of John Wiley & Sons, Inc., from *Detectors for Capillary Chromatography* (Copyright ©1992) Hill, H.H., and McMinn, D.G., ed., Chapter 7, by Patterson, P. L., Figure 7.1, p. 142.]

nitrogen (selectivity to phosphorus over hydrocarbons is adequate for most designs), detector operating stability, and source ruggedness for extended operating life. Some but not all detector models permit adjustment of the alkali source height above the jet for optimization of sensitivity and selectivity.

All N/P detectors provide electronic heating of the alkali source to 600-800° C. The plasma in the region of the salt is sustained by flows of hydrogen and air. The alkali source exhibits longer operating life and more stable and reproducible response under these conditions than in the presence of a flame.

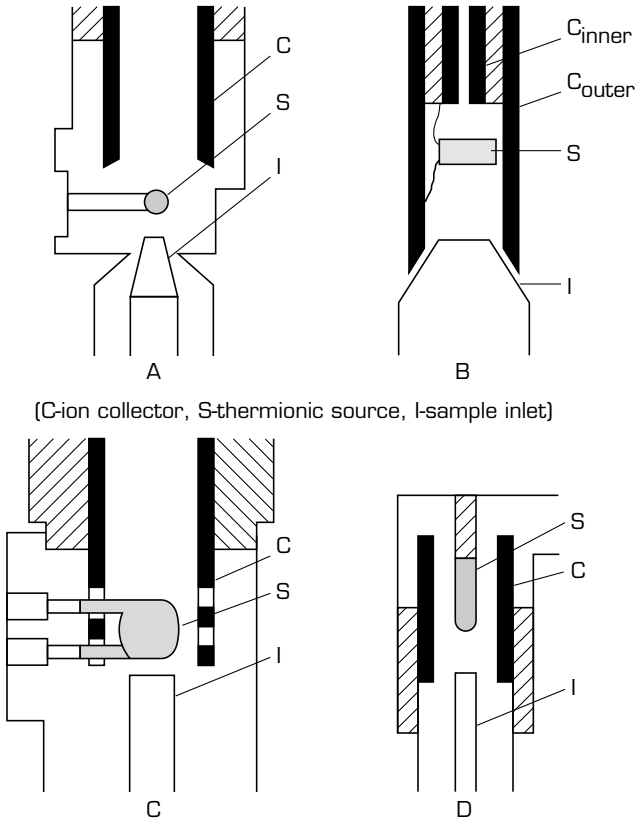
Apparatus and Reagents

Section 501 B provides general information on apparatus and reagents required for GLC.

Detector Characteristics

Sensitivity. N/P detectors are capable of producing detectable peaks in response to as little as 5-10 pg nitrogen-containing compounds or to 1-5 pg phosphorus-containing compounds [30]. FDA experience indicates that the pesticide for which the greatest N/P response occurs is diazinon, which contains two nitrogen atoms and one phosphorus atom; 25 pg diazinon should cause a response of approxi-

Figure 503-I
N/P Detector Configurations



(C-ion collector, S-thermionic source, I-sample inlet)

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mately 5% FSD; tebuthiuron, with four nitrogen atoms (no phosphorus), requires about 50 pg for 5% FSD response when chromatographed on a wide bore capillary column at standardized sensitivity (Appendix I, PESTDATA).

N/P sensitivity is most influenced by hydrogen flow and the magnitude of heating current supplied to the alkali source. Alkali source position may affect sensitivity, but not all designs permit adjustment of source height. Response diminishes over the lifetime of the alkali source. Variations in response are also seen among individual alkali sources.

Detector response to nitrogen is most affected by hydrogen flow rate, with response increasing as hydrogen flow decreases; optimum flow for the particular detector must be determined experimentally. Typical hydrogen flow rate for optimum nitrogen sensitivity is 1-5 mL/min. Response also increases with increasing alkali source current, but little improvement is realized, because detector background can also increase. Lifetime of the alkali source may also be shortened by operation at higher current.

Response to nitrogen compounds is not strictly proportional to the amount of elemental nitrogen in the molecule; variations based on molecular structure occur. Although the reactions that occur within the detector plasma are effective in decomposing analytes into common species, those compounds that easily decompose to the cyano radical usually cause higher response than do amides or nitro compounds [29].

Selectivity. Selectivity of the N/P detector is about 10^3–10^5 for N:C response, 10^4–5×10^5 for P:C response, and 0.1–0.5 for N:P response [29]. Factors that affect detector sensitivity do not always affect selectivity similarly. While both sensitivity and selectivity to nitrogen improve with decreasing hydrogen flow, only sensitivity (but not selectivity) improves with source heating current, because background noise and response to other elements increase simultaneously.

Linearity. Manufacturers of N/P claim linearity of response over four or five orders of magnitude. No FDA studies have been done on modern N/P detectors to measure detector linearity relative to amount of pesticides. Laboratories using N/P detectors must evaluate the linear range, work within that range, and match

peak sizes of residue and standard within 25% for accurate quantitative determination.

Other Influences on Detector Performance

Detector Temperature. Detector output is very sensitive to temperature changes within the active zone where ionization occurs; for stability of operation, conditions that permit variations in temperature should be avoided. Temperature of the alkali source is controlled by the electrical current at which it is operated but is also affected by hydrogen flow and, to a lesser extent, the rate of air and column carrier gas flowing past the source. The detector walls are heated separately. Stability is improved when the N/P detector itself is operated at a high temperature, because this minimizes the temperature gradient between the alkali source and the surrounding wall; reducing the gradient minimizes change in source temperature that occurs when high concentrations of analytes pass through the detector [29].

Age of Alkali Source. Each alkali source has a finite lifetime; eventually each must be replaced. Both sensitivity and selectivity decrease as the source ages, so regular calibration of detector performance is required. Source activity can be conserved by reducing hydrogen flow when the detector is not in use; however, manufacturer's instructions regarding source current and gas flow must be followed carefully to avoid destruction of the source. Operation of the detector at the lowest source current compatible with desired sensitivity is also recommended, as is maintenance of the detector at 100-150° C when not in use to prevent water condensation. Because degradation occurs more rapidly with higher source heating current, increasing the electrometer sensitivity to maintain constant detector sensitivity is preferable to increasing source heating current [29].

Replacement of the alkali source and re-establishment of optimum operating conditions can be troublesome and time-consuming with some detector designs. Design quality is at least partly judged by the stability of the source itself and even more so by the ease with which the source can be replaced and stable operation re-established.

Gas Flow Stability. Stable flow of hydrogen and air is critical for constant and linear response. High precision gas flow valves, standard equipment on some chromatographs, may be required for acceptable operation.

Position of Column Outlet. For maximum sensitivity and optimal peak shape, the GLC column should be positioned about 1-3 mm from the tip of the detector jet. The column should not protrude into the flame, because the polyimide coating on capillary columns will decompose and the resulting nitrogen products cause high background signal and noise. If the column outlet is too far below the tip, peaks may tail and/or be reduced in size because of the dead volume between the column and the alkali source [30].

Solvents and Reagents. Use of certain materials can have a detrimental effect on efficient operation of N/P detectors and should be avoided. For example, injection of extracts containing even trace amounts of acetonitrile can cause large detector response and preclude examination of the early eluting portion of the chromatogram; such extracts must be evaporated or azeotroped to remove all acetonitrile before injection. In addition, halogenated solvents may destroy the alkali source

and thus should not be injected. (Some N/P detectors are designed to permit use of halogenated solvents, but this must be ascertained prior to injection.)

Packed column stationary phases containing cyano groups (*e.g.*, OV-225) are unacceptable for use with N/P detectors. Equivalent bonded phase capillary columns have little bleed and may be acceptable, however. Other materials to avoid include those known to cause problems in many GLC systems, *e.g.*, septa not designated for high temperature use, impurities in gases, and leak-detecting solutions.

Certain common materials can appear as contaminants in determinations using N/P detectors. Nicotine is usually detected when cigarette smoking occurs in the vicinity; if phosphate detergents are used to wash glassware, or if the GLC column or glass wool is treated with phosphoric acid, trace amounts remain and are detectable during determination.

Recommended Operating Procedures

The following directions, adapted from the instrument manual for one N/P detector [31], have not yet been tested within FDA but are proposed as a way of optimizing detector operation:

- Follow manufacturer's directions for installation and operation. Pay particular attention to recommendations related to the alkali source, including situations that should be avoided to prevent its destruction. Use of wide bore capillary column with retention gap (Section 502 C) is recommended; makeup gas should not be necessary if column carrier flow rate of 10-20 mL/min is used.

- Follow manufacturer's directions to establish detector operation selective to nitrogen. Adjust detector parameters and instrument attenuation so that 1.0 ng chlorpyrifos causes 50% FSD.

- Prepare test solution containing 2.0 ng/µL azobenzene (containing 310 pg N), 2.0 ng/µL parathion-methyl (110 pg N and 230 pg P), 4.0 ng/µL malathion (380 pg P), and 4 µg/µL n-heptadecane (3.4 µg C) in isooctane.

- Inject 1 µL test solution, and adjust detector attenuation and range to keep peaks on scale. Examine relative responses of detector to four components; negative deflection of pen is normal in area of solvent peak.

- Experiment with effect of hydrogen flow on detector selectivity to nitrogen by re-injecting test solution after changing hydrogen flow rate in increments of 0.5 mL/min.

- Based on experimental results, use hydrogen flow rate that produces greatest ratio of response for parathion-methyl:malathion, as long as azobenzene peak is ≥4 times heptadecane peak at that flow; malathion peak can be expected to always be larger than parathion-methyl peak.

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504: QUANTITATION

504 A: INTRODUCTION

Accurate quantitation of pesticide residues identified by GLC is always of critical importance. Whether the analysis has been performed for purposes of monitoring or for enforcement of regulations, the consequences always have potential long term impact. All analyses that claim to produce quantitative results must be calculated in a consistent, reliable manner.

Accurate quantitation depends on use of accurate reference standards, use of a GLC system whose response is linearly proportional to the weight of chemical being detected (or for whose nonlinear response adjustment can be made), and use of proper technique for measuring detector responses. Given these conditions, quantitation is based on a simple proportion equation, *i.e.*:

$$\frac{\text{quantity of analyte}}{\text{detector response to analyte}} = \frac{\text{quantity of standard}}{\text{detector response to standard}}$$

Quantity of analyte, the unknown value, is readily calculated from the known quantity of standard and the measured detector responses.

This section assumes that the first two conditions for accurate quantitation, *i.e.*, accurate reference standards and a linear GLC system, are met. Only techniques for measuring detector response are discussed here.

Measurement of detector response for use in the above formula has traditionally involved manual measurement of the peak that represents detector response on a chromatogram drawn by a strip chart recorder. Section 504 B provides directions for the most practical ways of manually measuring peaks.

Modern automated data handling systems electronically integrate the detector output signal and produce a numerical representation of peak size. Step-by-step directions for such systems are not included in this manual, however, because each is unique; analysts using electronic integration must follow the directions provided by the manufacturer. Section 504 C provides general guidance to the appropriate application of electronic integration and advice about avoiding pitfalls that can occur.

Whether the detector response (peak) is measured manually or electronically, proper positioning of the baseline below the peak is critical. Accuracy of the measurement depends in part on how well the detector's response to the residue can be distinguished from its response to sample co-extractives and co-eluting residues. Typically, a residue peak in a sample chromatogram may occur on a sloping baseline, on top of another peak, or incompletely separated from another peak; in contrast, the reference standard solution usually causes a single symmetrical peak. Quantitative accuracy is sacrificed if the residue peak's baseline is not properly delineated. To measure peaks manually, the analyst must literally draw the baseline on the chromatogram before measuring; to use automated measurement, the analyst must configure the system to include only that part of the signal that can reasonably be assumed to represent the residue.

Appropriate setting of the baseline is integral to the directions below for measuring the peaks. In some cases, choice of appropriate baseline for particular residues will be shown by example.

504 B: MANUAL QUANTITATION

Many methods of quantitating gas chromatographic peaks have been presented in the literature, but through the years most laboratories that perform manual measurements of peaks have relied on the two simplest approaches: measurement of peak height and measurement of area of the triangle that best fits the peak ("triangulation"). Peak area is the more accurate representation of detector response, but peak height is a justifiable approximation of area when peak shape makes height proportional to area. Advances in column techniques (Section 502) have resulted in improved peak symmetry and resolution, thus encouraging use of peak height for quantitation.

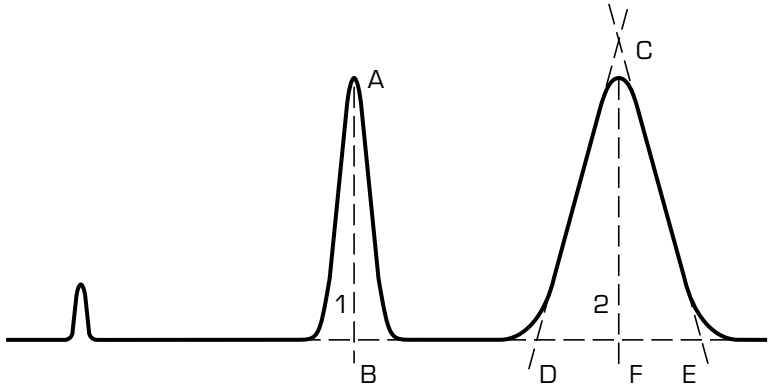
Other techniques for manual measurement of peaks have been described in various chromatographic texts; these include calculation of the product of peak height and width at half height, product of retention time and peak height, weight of peaks cut from chromatogram, peak area measured by a planimeter, and peak area measured by a mechanical integrator attached to the recorder. Comparison of results among some of these techniques indicated their validity [1], but none are described in this section because they are time-consuming and more difficult and offer no significant advantage over those presented here.

Measurement of Peak Height

Peak height measurements are recommended for early eluting peaks, peaks of width <10 mm, and very small peaks. If analyte and reference standard peaks are narrow and approximately the same size, comparison of peak heights is less subject to measurement error than is triangulation. Peak height measurements are very sensitive to changes in operating conditions, so operating parameters must be closely controlled for accurate quantitation.

To measure peak height, construct a baseline beneath the peak and measure the length of the perpendicular from peak apex to midpoint of the constructed baseline. In Figure 504-a, this is represented by line AB on Peak 1.

Figure 504-a
Manual Peak Measurement



Measurement by: (1) peak height and (2) triangulation.

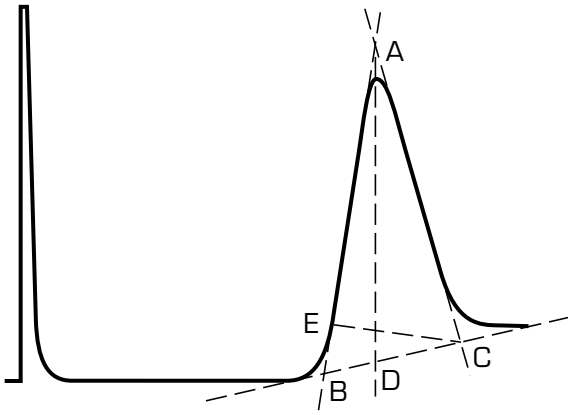
Measurement of Area by Triangulation

Measurement by triangulation involves drawing a triangle that approximates a peak's dimensions and calculating the area of the triangle. This method requires

extreme care in construction of the triangle and in measuring its dimensions. Special treatment is required for peaks on sloping baselines and for skewed (asymmetrical) peaks. The technique is subject to error when the peak is narrow but is preferred over measurement of peak height when the peak is >10 mm wide at the base.

To construct the triangle, draw a baseline below the peak and draw inflection tangents to the peak, as shown in Figure 504-a, Peak 2. Drop a perpendicular bisect from the constructed apex to the baseline. Measure triangle height (length of bisect from baseline to constructed apex, CF in the figure) and the base (length of baseline between its intersection with the tangents, DE in the figure). Calculate area as 1/2 (base × height), *i.e.*, 1/2 (DE × CF).

Figure 504-b
Triangulation of Peak on Sloping Baseline



When the chromatogram baseline slopes under a peak, the line dropped from the intersection of tangents does not serve as an accurate measure of height because it is not perpendicular to the baseline; *e.g.*, Figure 504-b, line AD in triangle ABC. To measure the area of such a triangle, draw a perpendicular to one of the tangents (CE in Figure 504-b). Then use its length as the triangle height and the length of the tangent (AB) as the base. Calculate area from these values using the standard formula, *i.e.*, 1/2 (AB × CE).

Skewed peaks present another challenge to the validity of area measurement by triangulation. As a peak becomes more skewed, less and less of its area is included within the triangle drawn to approximate it. Skewed peaks may be tailing or fronting, depending on what physical phenomena caused the poor chromatography. The preferred solution to quantitation of skewed peaks is to improve chromatography sufficiently to cause peaks to be symmetrical. Use of a more polar column, changing column or inlet temperature, or optimizing the injection system may effect the improvement.

If manual quantitation must be performed on a skewed peak, measurement of the area using the formula for calculating area of a trapezoid is preferred [2]. In this system, peak widths at 15 and 85% of height are measured and used in the formula: area = 1/2 × height × (width at 15% + width at 85%). Calculations performed in this way have been shown to accurately represent peak area even for increasingly skewed peaks [3].

504 C: ELECTRONIC INTEGRATION

Electronic integration devices provide laboratories with powerful tools to accomplish their work more efficiently. Over the years, technology has progressively improved from simple desktop integrators to software programs operated by computers at all capability levels. The more powerful "automated data handling systems" can automate the entire determinative step, including monitoring of instrument temperatures and flow rates, operation of autoinjectors, acquisition of

retention times and detector responses, and interpretation of those values for residue identification and quantitation. Unattended operation of instruments is common when automated systems are available; some systems are capable of simultaneous management of multiple instruments. Automated data handling is often incorporated into computerized laboratory information management systems capable of producing both the final laboratory report and the documentation necessary for quality assurance requirements (Section 206).

Discussion of entire systems is beyond the scope of this section. The focus is instead on measurement of peaks by electronic integration of the signal produced by a detector, *i.e.*, summation of the change in electronic signal per unit time. Beyond that generalized description of the integration process, each system operates under a unique "integration algorithm" that specifies how it will choose what part of the signal to integrate. The accuracy with which the system can measure detector response to a particular analyte depends on the algorithm itself, on the configuration options available to the user, and on the user's conscientiousness in choosing appropriate options. If an electronic integrator is properly configured, its measurement of peaks is the fastest, most accurate, and most reproducible available.

Major pitfalls exist, however, in the uncritical acceptance of results generated by electronic integration. Proper configuration of the algorithm, to the extent permitted by the system, is critical. After chromatograms have been run and results presented, review by a competent analyst is essential, because no integration algorithm can ever handle perfectly all the variations that occur in the chromatographic environment. The analyst must understand the concepts incorporated into the algorithm, be able to interpret the visual display of the chromatogram provided by the system, and evaluate whether integration was appropriately performed.

Data systems that perform electronic integration vary in the amount of "memory" available for storing data. Although simple integrators have only enough memory to process one chromatogram at a time, computer-based systems can usually store data associated with many chromatograms. In the latter case, when review of a chromatogram suggests that the original integration was performed improperly, the system can be reconfigured and a new calculation made from the stored data. If the system lacks the memory required to permit recalculation, the sample must be rechromatographed with the integrator reconfigured. Alternatively, the peak(s) can be measured manually from a printed chromatogram.

Optimum quantitation accuracy with any electronic integrator is dependent on the operator's making complete use of options available within the integrator. The following approach is recommended:

- Configure integrator for the GLC system. At the minimum, configure the integrator for the particular GLC system in use, rather than operating with default settings. Develop an integrator configuration for each GLC system routinely used. Store integrator settings as a "program," if the system permits, or keep a written record if necessary.

- Optimize peak and baseline recognition by considering the typical chromatograms the GLC system can be expected to produce. Chromatographic features and the conditions that determine them include: baseline noise, varying with type of detector; expected peak widths, dependent on column

and conditions; and inclusion of a solvent peak, dependent on whether or not solvent is vented. Most electronic integrators can be configured to specify the following options:

1) Size (in whatever value the integrator generates) below which a response is not recognized as a peak; sometimes called "area reject."

2) Range of peak base widths within which detector response is recognized as a peak.

3) Increase in baseline slope above which detector response is recognized as a peak; referred to as "threshold."

4) Appearance of multiple inflection points before the apex, used to identify the existence of two or more peaks when no "valley" exists between them. Some systems can classify such "shoulders" as front or rear.

5) Slope of peak above which response is recognized as the solvent peak; can be specified because solvent peak rises faster than most other peaks; value depends on detector, sensitivity, and column efficiency. May also permit recognition of peaks that appear on the tailing edge of the solvent.

- Use integrator features that demonstrate its operation. Most electronic integrators offer the option of displaying, on the chromatogram, an indication of exactly where the measurement started and ended. Some integrators can also be configured to show where the baseline was drawn. The analyst should take advantage of these features by choosing the option to print such indicators and should then use them in subsequent comparison of integrator measurements to the chromatogram.

- Configure integrator to accommodate particular chromatograms. An integrator configured by a pre-established program for a particular GLC system may not measure peaks accurately if the chromatogram includes responses to co-extractives or an unexpectedly complicated pattern of residues. Choose other options for configuration if experience with the commodity, method, or likely residues suggests in advance what type of chromatogram can be expected. For example, if the chromatogram is likely to contain isolated, symmetrical peaks on a flat, quiet baseline, configure the integrator to match peak width selection to measured width of the peak at half height, and set the threshold a few units below the highest value still capable of detecting the peak. In contrast, if the chromatogram is likely to contain peaks clustered together or with a noisy or sloping baseline, configure the integrator to accommodate those conditions. Table 504-a lists the effects produced when the two most important integrator settings, peak width and threshold, are varied.

- Review integrator measurements and reconfigure for accuracy. The analyst is ultimately responsible for accurate quantitation, so review and evaluation of chromatograms and integrator reports are essential. If examination reveals that the integrator inappropriately included or excluded portion(s) of the chromatogram, the following integrator options should be changed and the peak recalculated:

- Reposition baseline to appropriate base of the peak(s).

- Measure peak(s) appearing on top of much larger peak from baseline constructed to represent remainder of larger peak; sometimes called "tangent skim."

- Identify point at which to split incompletely resolved peaks, *i.e.*, where to end integration of one response and start integration of the next; sometimes called "split peak."

- Delete one or more peaks from integration; this does not remove peak from chromatogram.

- Integrate area within chromatogram as single number; useful when multicomponent residues, such as toxaphene, are being measured.

Table 504-a: Effects of Changing Electronic Integrator Settings

Setting		Result
Peak Width	Threshold	
High	High	Only major peaks detected; random noise eliminated
High	Low	Trace-level peaks detected; noise also recognized as peaks
Low	High	Peaks on sloping baselines detected; noise not detected
Low	Low	Narrow and broad peaks both detected (low peak width permits recognition of narrow peaks, while low threshold permits detection of broader peaks)

504 D: SPECIAL CONSIDERATIONS FOR COMPLEX CHROMATOGRAMS

Chromatograms that display residues of multicomponent chemicals or mixtures of two or more residues challenge the chemist to perform accurate measurement of peak size. Quantitative accuracy is further challenged when the residue has undergone degradation and the pattern of peaks does not match that of the most appropriate reference standard. The following procedures for quantitation of certain difficult residues have been developed during years of practical experience.

BHC (also known as HCH, hexachlorocyclohexane)

Technical grade BHC is a mixture of six chemically distinct isomers and one or more heptachlorocyclohexanes and octachlorocyclohexanes [4]; as a practical matter, the isomers α, β, γ, and δ are the only ones ever reported by FDA. The γ isomer is also known as lindane and is marketed as a separate pesticide. Currently, U.S. tolerances for BHC have been revoked, but residues are still found in imported commodities; U.S. tolerances for lindane remain on several commodities.

Residues of BHC can be expected to vary in relative amounts of the individual isomers for several reasons: (a) Separate use of both BHC and lindane is possible, (b) commercial formulations vary in the percentage of individual BHC isomers present, and (c) isomers undergo different rates of metabolism or environmental degradation; *e.g.*, the elimination rate of isomers fed to rats was 3 weeks for the α, γ, and δ isomers and 14 weeks for the β isomer [5]. This difference in animal metabolism rates explains the typical finding of β isomer as the predominant BHC residue in dairy products.

Detector response to the same amount of different isomers may also vary. When BHC isomers were chromatographed individually on a wide bore methyl silicone column, relative response of an electroconductivity (halogen mode) detector (ElCD-X) to each isomer ranged from 0.58-1.00, while ^{63}Ni electron capture (EC) detector responses at the same conditions varied from 0.43-1.30 (Table 504-b). Both detectors responded less to ß-BHC than to the other three isomers [6].

Hexachlorobenzene, an industrial chemical and impurity associated with the pesticide quintozene, elutes near the BHC residues on all commonly used GLC systems. Although hexachlorobenzene has only occasionally been found in the same sample as BHC, it is important to ascertain that it is not present before BHC residues are quantitated. Several packed columns were once cited as capable of separating hexachlorobenzene and the four important BHC residues from one another [7, 8]. Among the GLC systems described in Section 302, the best choice for separating these residues is DG18 (50% cyanopropylphenyl, 50% methyl siloxane column at 200° C, electron capture detector). The column of DG18 is not compatible with ElCD-X, so DG22 (DEGS column at 180° C, ElCD-X) is recommended for confirmation of BHC residues as long as β- and δ-BHC, which do not separate, are not both present.

To quantitate BHC most accurately:

- Choose GLC system that separates residues in the sample from one another; if possible, use a halogen-selective detector, such as ElCD-X.
- Quantitate each isomer separately against a standard of the respective pure isomer.

Table 504-b: Response of Two Detectors to Four BHC Isomers

BHC Isomer	Ng Required for 1/2 FSD		Response Relative to Lindane	
	^{63}Ni EC	ElCD-X	^{63}Ni EC	ElCD-X
Alpha	0.24	0.41	1.30	0.71
Beta	0.72	0.50	0.43	0.58
Gamma (lindane)	0.31	0.29	1.00	1.00
Delta	0.33	0.49	0.94	0.59

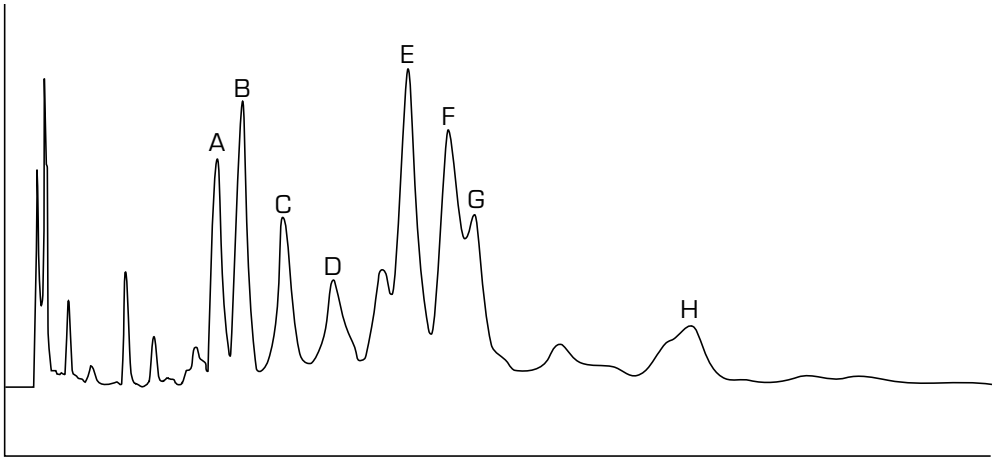
Chlordane

Chlordane is a technical mixture of at least 11 major components and 30 or more minor ones; Figure 504-c is a chromatogram of technical chlordane. Structures of the many chlordane constituents have been elucidated using GLC-mass spectrometry and nuclear magnetic resonance analytical techniques [9, 10]. The two major components of technical chlordane are trans- and cis-chlordane (Figure 504-c peaks E and F, respectively); the exact percentage of each in the technical material is not completely defined and is inconsistent from batch to batch.

At one time, heptachlor, a component of technical chlordane, was also marketed as a separate pesticide. When residues of heptachlor and its metabolite heptachlor epoxide were found in the same commodity as chlordane, the source of the former was in question. Currently, neither chlordane nor heptachlor is registered for use on foods in the United States, and tolerances for both have been revoked. Most residues that are now found occur in fish as a result of lingering environmental contamination.

The GLC pattern of a chlordane residue may differ considerably from that of the technical standard. Depending on the sample substrate and its history, residues of chlordane can consist of almost any combination of constituents from the technical chlordane, plant and/or animal metabolites, and products of degradation caused by exposure to environmental factors such as water and sunlight. Only limited information is available on which GLC residue patterns are likely to occur in which commodities (*e.g.*, References 11 and 12), and even this information may not be applicable to a situation where the route of exposure is unusual. For example, fish exposed to a recent spill of technical chlordane will contain a residue drastically different from a fish whose chlordane residue was accumulated through normal food chain processes.

Figure 504-c
Technical Chlordane



Chromatogram of 1.8 ng technical chlordane, chromatographed on system 302 DG 1. Labeled peaks are thought to represent, respectively: A, monochlorinated adduct of pentachlorocyclopentadiene with cyclopentadiene; B, co-elution of heptachlor and alpha chlordene; C, co-elution of beta chlordene and gamma chlordene; D, a chlordane analog; E, trans-chlordane; F, cis-chlordane; G, trans-nonachlor; H, co-elution of cis-nonachlor and "Compound K," a chlordane isomer.

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Form FDA 2905a (6/92)

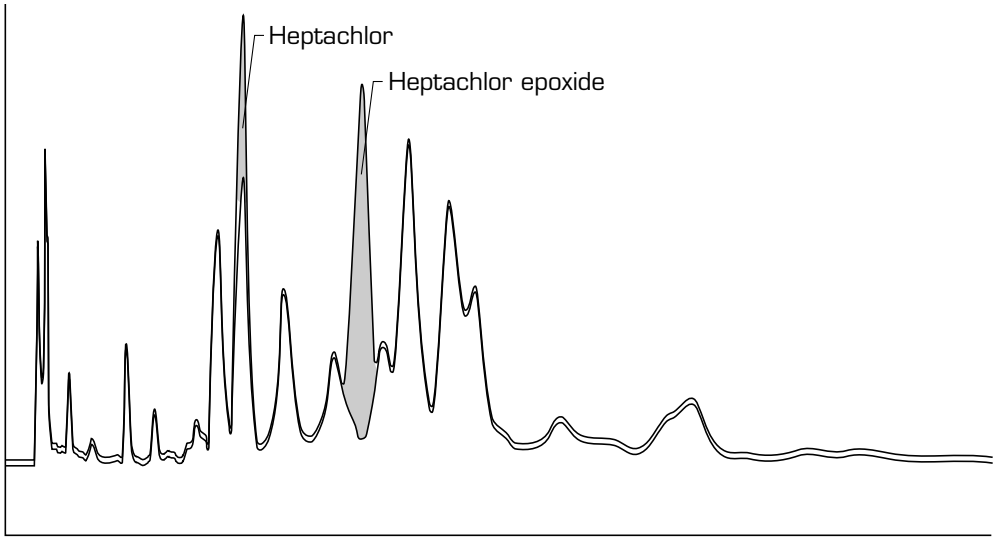
Because of this inability to predict a chlordane GLC residue pattern, no single method can be described for quantitating chlordane residues. The analyst must judge whether or not the residue's GLC pattern is sufficiently similar to that of a technical chlordane reference standard to use the latter as a reference standard for quantitation, then:

- When the chlordane residue does not resemble technical chlordane, but instead consists primarily of individual, identifiable peaks, quantitate each peak separately against the appropriate reference standard. Reference standards are available for at least 11 chlordane constituents, metabolites, or degradation products that may occur in the residue.

- When the GLC pattern of the residue resembles that of technical chlordane, quantitate chlordane residues by comparing the total area of the chlordane residue from peaks A through G (Figure 504-c) to the same part of the standard chromatogram. To define appropriate measurable area of chromatograms, adjust amount of extract injected so that the major residue peaks are about 50% full scale deflection (FSD), then inject an amount of reference standard that causes response within ±25% of that; peaks E and F in the two chromatograms should be about the same size. Construct the baseline beneath the standard from the beginning of peak A to the end of peak G. Use the distance from the trough between peaks E and F to the baseline in the chromatogram of the standard to construct the baseline in the chromatogram of the sample.

Peak H may be obscured in a sample by the presence of other pesticides. If H is not obscured, include it in the measurement for both standard and sample. If the heptachlor epoxide peak is relatively small, include it as part of the total chlordane area for calculation of the residue. If heptachlor and/or heptachlor epoxide are much out of proportion, as in Figure 504-d, calculate these separately and subtract

Figure 504-d
Chlordane, Heptachlor, Heptachlor Epoxide



Chromatogram of 1.8 ng technical chlordane, 0.1 ng heptachlor, and 0.3 ng heptachlor epoxide, superimposed on chromatogram of technical chlordane only; system 302 DG1.

their areas from total area to give a corrected chlordane area. (Note that octachlor epoxide, a metabolite of chlordane, can easily be mistaken for heptachlor epoxide on a nonpolar GLC column.)

(When measurement of total peak area by integration was compared to addition of peak heights for quantitation of chlordane residue in several samples, the results of the two techniques were reasonably close; results justify the use of the "peak height addition" technique for calculating total chlordane when no means of measuring total area is available. To quantitate by peak height addition, measure heights of peaks A, B, C, D, E, F, and G, in mm, from peak maximum of each to the baseline constructed under the total chlordane area, then add heights. The technique has inherent difficulties because not all the peaks are symmetrical and not all are present in the same ratio in the standard and in the sample.)

PCBs

Polychlorinated biphenyls (PCBs) were manufactured for many years in the United States by the Monsanto Co. and marketed under the trade name Aroclor. Each Aroclor product was a mixture of chlorobiphenyl congeners into which 1-10 chlorine atoms were substituted; 209 different congeners were possible. Common commercial products included Aroclor 1221, 1242, 1248, 1254, 1260, and 1262, with the last two digits in the name indicating the average percent chlorination in the particular mixture; Aroclor 1016, purportedly a purified version of Aroclor 1242, was also marketed. Aroclors are no longer used or marketed in the United States, but their residues remain in the environment, in foods like fish and shellfish, in animals, and in human tissue.

GLC chromatograms of PCB residues contain many peaks, and patterns vary considerably, because residues can result from any combination of Aroclor mixtures. Variations in residue patterns are also caused by degradation from weathering or metabolism. Different congeners vary in the degree to which they are excreted by or retained within an animal and by the degree to which they volatilize. This multiplicity of potential PCB residue patterns makes the task of identifying and quantitating residues extremely challenging. The presence in the extract of residues from chlorinated hydrocarbon pesticides further complicates the determination. Residues of p,p'-DDE are most likely to interfere in determination of PCBs, because both residues are often present in the same commodity and because their structural and behavioral similarities make them difficult to separate with normal analytical methodology.

Quantitation of PCB residues is best achieved by following these steps:

- Isolate PCB residues from sample co-extractives and from other residues to the degree possible before GLC determination. Certain cleanup step options in Chapter 3 methods are designed to separate PCBs from pesticide residues of similar structure; these options should be used to analyze any commodity in which PCB residues are likely to occur, especially fish and shellfish.

- Select the reference standard that most closely resembles the residue pattern. A single Aroclor or, more often, a mixture of Aroclors that produce the most similar pattern is used for quantitation. Judgment must be made about what proportion of different Aroclors should be combined to produce the appropriate reference standard.

- Use a GLC system that separates peaks efficiently. Packed or capillary columns may be used; wide bore capillary columns provide the best compromise of speed and efficiency.

- Choose from the following quantitation options the one that best suits the residue pattern. Both have been successfully collaborated in interlaboratory tests [13, 14]; choice depends on the degree to which the residue and reference standard match:

  1) When PCB residue pattern closely resembles that of a single Aroclor or mixture of Aroclors, quantitate by comparing total area or height of residue peaks to total area or height of peaks from appropriate reference standard(s). Measure total area or height response from common baseline under all peaks. Use only those peaks in the residue that can be attributed to chlorobiphenyls. These peaks must also be present in chromatogram of reference standards.

  2) When PCB residue pattern is significantly different from that of any Aroclor or mixture of Aroclors, quantitate by comparing area of each peak in residue to peak at same retention time in a specially calibrated lot of Aroclor reference standard (Table 504-c). To each peak thus measured, apply weight factor associated with that peak in particular reference standard, as listed in Table 504-c. Sum individual peak values to obtain total ppm PCB. This option can also be used when residue and reference standard chromatographic patterns match. The special Aroclor reference standards were calibrated using the separations effected by packed column chromatography, but the weight factors are also valid with chromatography on the equivalent wide bore capillary column operated in the packed column mode (Section 502 C).

Other quantitation techniques are sometimes used. One system makes use of capillary column chromatography, capable of separating most PCB congeners from one another, and a precalibrated reference standard mixture for which identity and weight percent of each congener have been established [15]. This "individual congener" capillary column method is significantly more time-consuming than measurement of individual peak areas or heights obtained by packed column GLC, and results from the two approaches are not significantly different when total PCBs are calculated [16, 17]. Several European countries use variations of the individual congener method by measuring, in sample and standard, only selected peaks [18]; in these countries, legal limits on PCB residues are defined in terms of results from the established quantitation method.

Accurate quantitation of both p,p'-DDE and PCBs in the same sample is possible only when chromatographed on a narrow bore capillary column. Quantitation of only the PCB residue, when p,p'-DDE is present, can be accomplished by first eliminating p,p'-DDE with derivatization and column chromatography [19].

Figure 504-e, a chromatogram of PCB residues isolated from chinook salmon, demonstrates the challenge of PCB determinations. Quantitation was performed by comparison to a mixture of Aroclors 1254 and 1260.

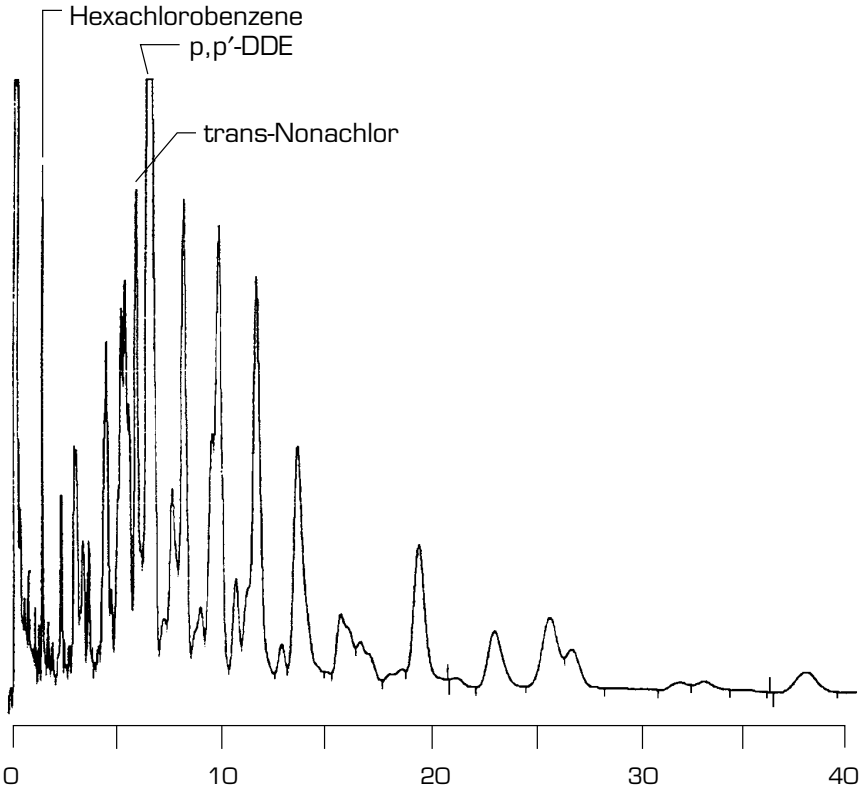
Table 504-c: Weight Percent Factors for Individual Gas Chromatographic Peaks in Aroclor Reference Standards

	Aroclor				
R_{DDE} (100x)[1]	1016 (77-029)[2]	1242 (71-696)[2]	1248 (71-697)[2]	1254 (71-698)[2]	1260 (71-699)[2]
11	0.2				
16	3.8	3.4	0.3		
21	8.1	10.3	1.1		
24	1.2	1.1	0.2		
28	16.8	15.8	6.0		
32	7.6	7.3	2.6		
37	18.5	17.0	8.7		
40	14.6	13.0	7.4		
47	11.6	9.9	15.7	7.1	
54	7.7	7.1	9.3	2.7	
58	6.4	4.4	8.3	1.2	
70	3.4	8.7	18.2	14.7	2.4
78		1.9	6.4		
84			4.6	18.6	3.6
98			3.4	8.3	2.8
104			3.3	14.1	
112			1.0		
117					4.4
125			2.3	15.6	11.0
146			1.2	9.0	13.3
160					5.5
174				7.4	10.0
203				1.3	10.9
232-244					11.2
280					12.5
332					4.2
360-372					5.4
448					0.8
528					2.0

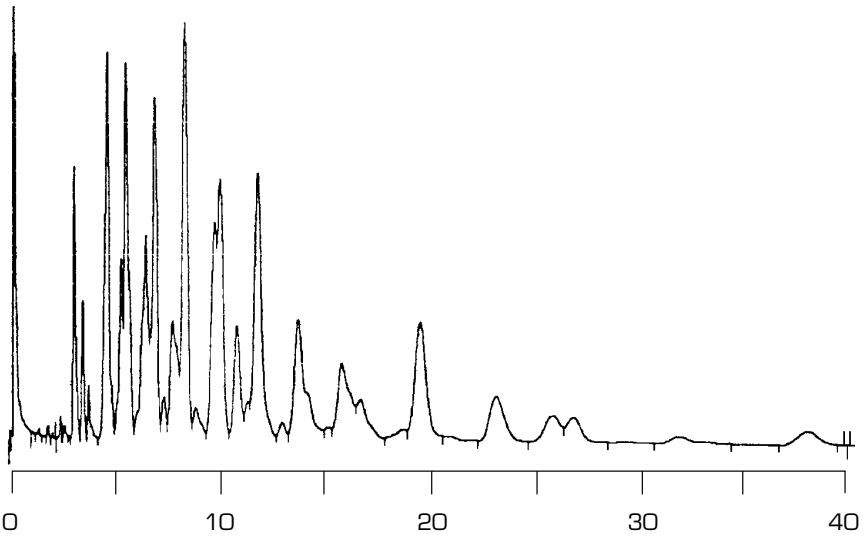
[1] Peaks are identified by their retention times relative to p,p′-DDE=100 at conditions described in Section 302 DG1.

[2] Food and Drug Administration Lot Nos. (Weight factors are valid only for these FDA Lot Nos.) Aroclor reference standards are available from Food and Drug Administration, Division of Pesticides and Industrial Chemicals, HFS-337, 200 C Street SW, Washington, DC 20204. Aroclor 1016 (77-029) was referred to as KB-06-256 in *J. Assoc. Off. Anal. Chem.* (1978) **61**, 272-281.

Figure 504-e
PCBs in Chinook Salmon



25 mg chinook salmon, extracted and cleaned up by method 304 E1+C3, chromatographed on system 302 DG1, with detector sensitivity greater than normal to permit measurement of low levels. Injection represents petroleum ether eluate from Florisil, which separates PCB residues from most but not all pesticides. Total PCB is 0.087 ppm, calculated using total area measurement, 0.090 using factors of Table 504-c; comparison is to mixed Aroclor standard below. Pesticides were identified but not quantitated.



1.23 ng Aroclor 1254 and 0.745 ng Aroclor 1260, chromatographed on same system as above.

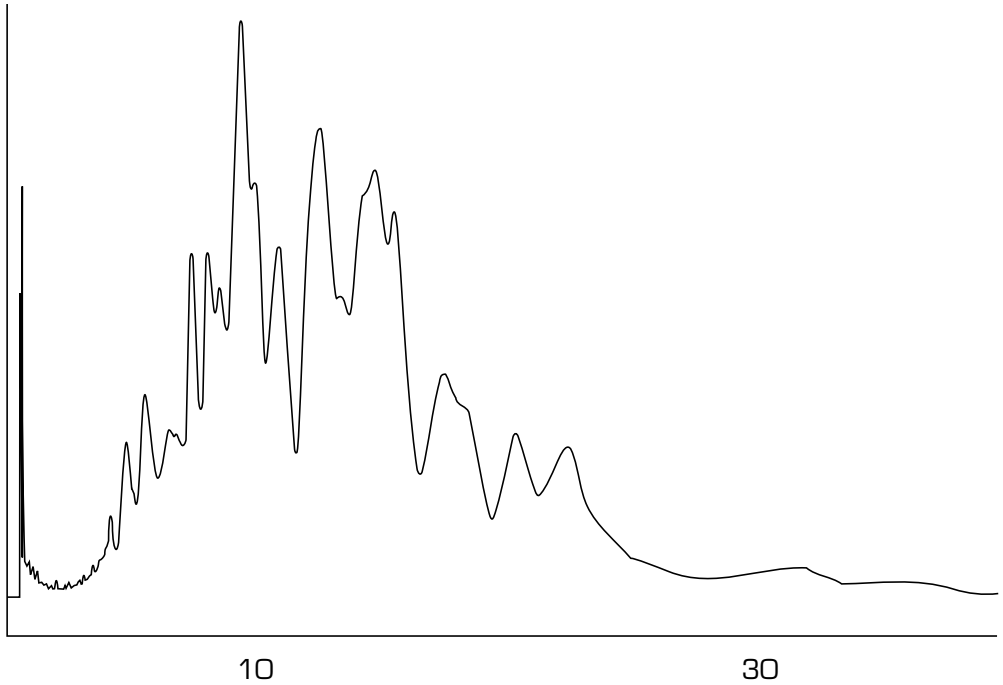
Toxaphene

Toxaphene is a complex mixture that results from the chlorination of camphene. The GLC chromatographic pattern for toxaphene does not display any individual peaks to simplify quantitation, but instead appears as a series of incompletely resolved peaks (Figure 504-f). Presence of other residues in the same extract as toxaphene requires estimation of baseline placement for quantitation. Reasonable accuracy is possible, but no truly quantitative technique has been developed.

To quantitate residues of toxaphene:

- Adjust amount of sample injected so that major residue peaks are 10-30% FSD.
- Inject amount of reference standard that causes response within ±25% of that of residue.
- Construct baseline for standard toxaphene between its extremities.
- Construct baseline under residue peaks, using distances of peak troughs to baseline on standard chromatogram as guide.
- Measure areas above baseline in sample and standard chromatograms for calculating level of residue. Relative heights and widths of matching peaks in the residue and reference standard will probably differ.

Figure 504-f
Toxaphene



Chromatogram of 11.4 ng toxaphene, chromatographed on system 302 DG1.

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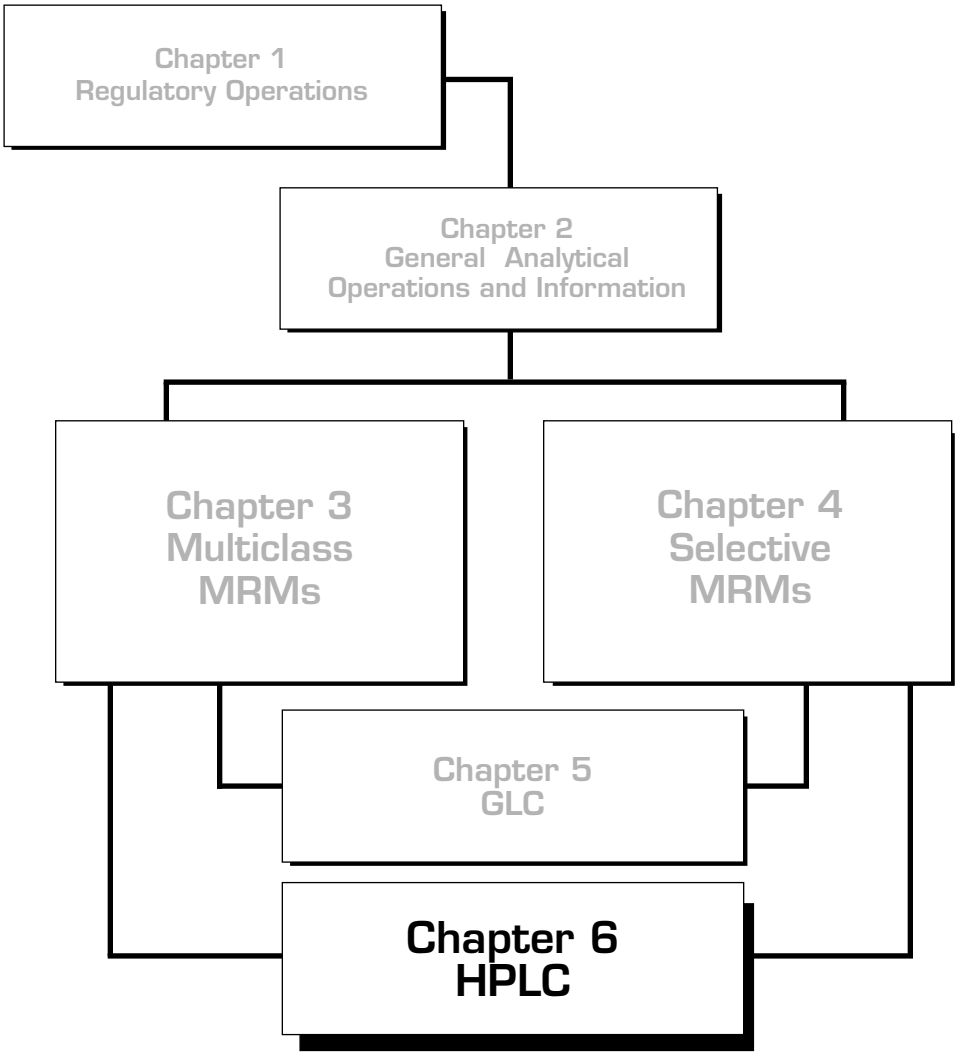

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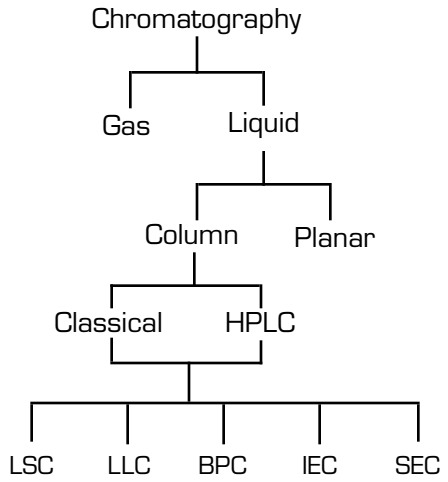
601: GENERAL INFORMATION

In recent years, high performance liquid chromatography (HPLC) has grown in popularity as a determinative step for residue analysis, until today it is accepted as complementary to the more traditional gas liquid chromatography (GLC). HPLC provides capabilities not possible with GLC, most importantly the ability to separate and quantitate residues of polar, nonvolatile, and heat-labile chemicals. These characteristics make HPLC the determinative step of choice for many residues previously beyond the applicability of multiresidue methodology.

601 A: PRINCIPLES

Figure 601-a
Chromatographic Separation Techniques



Chromatography comprises a family of separation techniques (Figure 601-a), all of which share common characteristics. A narrow initial zone of mixture is applied to a sorptive stationary phase having a large surface area. Development with mobile phase causes components of a mixture to move through the stationary phase at different rates and to separate from one another. Differential migration occurs because of differences in distribution between the two phases. The mobile phase can be a gas or a liquid. Liquid chromatography is divided into two main types, planar (thin layer and paper chromatography) and column. Column liquid chromatography, both the classical (low pressure) version and the high performance version discussed here, is further subdivided according to the mechanism of separation into five major types: liquid-solid (adsorption) chromatography, LSC; liquid-liquid (partition) chromatography, LLC; bonded phase chromatography, BPC; ion exchange chromatography, IEC; and size exclusion chromatography, SEC.

HPLC developed steadily during the late 1960s as high efficiency, small particle packings and improved instrumentation were produced. In contrast to classical column liquid chromatography, HPLC uses high pressure pumps; short, narrow columns packed with microparticulate phases; and a detector that continuously records the concentration of the sample.

HPLC systems use the principles of classical column chromatography in an analytical instrument. Development of HPLC has been directly related to availability of suitable hardware (columns, pumps, inlet systems, low dead volume fittings, *etc.*) that allows precise flow control under the elevated pressures needed, as well as the ability to manufacture a wide variety of column packing materials in particle sizes of exacting micron (μm) dimensions.

In contrast to GLC, where the gas mobile phase is inert and does not affect separation of analytes from one another, the HPLC mobile phase is critical to this

Chapter 6 is revised from a chapter on HPLC written for FDA in 1989-90 by Joseph Sherma, Ph.D., Lafayette College, Easton, PA.

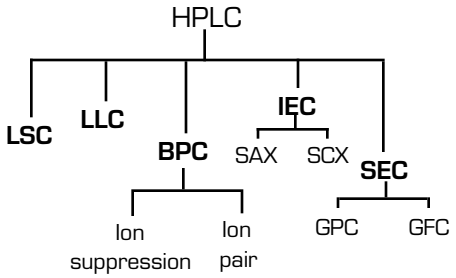
resolution. Choice of mobile phase is second only to the choice of operating mode in determining the suitability of the system to produce the desired separations.

HPLC had limited use for routine trace multiresidue analysis in the absence of sensitive element-selective detectors. Early development work relied primarily on refractive index (RI) or fixed wavelength UV absorbance detectors. Neither detector demonstrated sufficient sensitivity or selectivity for use in trace residue analysis. In the mid-1970s, the fluorescence detector was shown to provide the needed sensitivity and specificity for pesticides that are naturally fluorescent or can be chemically labeled with a fluorophore. This resulted in the first practical application of HPLC to multiresidue pesticide determination (see method for N-methylcarbamates, Section 401).

More recently, scientists have investigated photoconductivity and electrochemical detectors and certain applications of the newer multiwavelength UV detectors. This research indicates that these detectors can also fulfill the sensitivity and selectivity requirements for determination of certain pesticides at residue levels.

601 B: MODES OF OPERATION

Figure 601-b
HPLC Modes of Operation



Separations by HPLC are achieved using the five basic operational modes (Figure 601-b). The mode chosen for a particular application will depend on the properties of the analyte(s) to be separated and determined. For residue determination, as for HPLC analyses in general, BPC is the most widely used.

There are two variations within the five operational modes of HPLC operation; these distinctions are based on the relative polarities of stationary and mobile phases:

1) **normal phase (NP)** chromatography: stationary phase is more polar than the mobile phase; the least polar analytes elute first; analyte retention is increased by decreasing mobile phase polarity.

2) **reverse phase (RP)** chromatography: stationary phase is less polar than the mobile phase; the most polar analytes elute first; analyte retention is increased by increasing mobile phase polarity.

Liquid-Solid Chromatography

LSC, also called adsorption chromatography, uses an adsorbent, usually uncoated silica gel. The basis for separation is the selective adsorption of polar compounds, presumably by hydrogen bonding, to active silanol (SiOH) groups by orientation and on the surface of the silica gel. Analytes that are more polar will be attracted more strongly to the active silica gel sites. The solvent strength of the mobile phase determines the rate at which adsorbed analytes are desorbed and eluted.

LSC is useful for separation of isomers and classes of compounds differing in polarity and number of functional groups. It works best with compounds that have relatively low or intermediate polarity. Highly polar compounds may irreversibly adsorb on the column. Poor LSC separations are usually obtained for chemicals containing only nonpolar aliphatic substituents.

Liquid-Liquid Chromatography

LLC, also called partition chromatography, involves a solid support, usually silica gel or kieselguhr, mechanically coated with a film of an organic liquid. A typical system for NP LLC is a column coated with ß,ß'-oxy dipropionitrile and a nonpolar solvent like hexane as the mobile phase. Analytes are separated by partitioning between the two phases as in solvent extraction. Components more soluble in the stationary liquid move more slowly and elute later. LLC has now been replaced by BPC for most applications.

Bonded Phase Chromatography

BPC uses a stationary phase that is chemically bonded to silica gel by reaction of silanol groups with a substituted organosilane. Unlike LLC, the stationary phase is not altered by mobile phase development or temperature change. All solvents can be used, presaturation of the mobile phase with the stationary phase is not required, and gradient elution can be used to improve resolution.

Specialized applications of BPC have been developed for ionized compounds, which are highly water soluble and generally not well retained on RP BPC columns. Retention and separation can be increased by adding an appropriate pH buffer to suppress ionization (ion suppression chromatography) or by forming a lipophilic ion pair (ion pair chromatography) between the analyte and a counter ion of opposite charge. The resultant nonionic species are separated by the same column techniques used for naturally nonionic organic molecules.

Ion suppression is the preferred method for separation of weak acids and bases, for which the pH of the mobile phase can be adjusted to eliminate analyte ionization while remaining within the pH 2-8 stability range of bonded silica phases. The analyte is chromatographed by RP HPLC, usually on a C-18 column, using methanol or acetonitrile plus a buffer as the mobile phase. The technique is often preferred over IEC (see below) because C-18 columns have higher efficiency, equilibrate faster, and are generally easier to use reproducibly compared to ion exchange phases. Strong acids and bases are usually separated on an ion exchange column or by ion pair chromatography.

Ion pair chromatography is used to separate weak or strong acids or bases as well as other types of organic ionic compounds. The method involves use of a C-18 column and a mobile phase buffered to a pH value at which the analyte is completely ionized (acid pH for bases, basic pH for acids) and containing an appropriate ion pairing reagent of opposite charge. Trialkylammonium salts are commonly used for complex acidic analytes and alkylsulfonic acids for basic analytes. The ion pairs separate as if they are neutral polar molecules, but the exact mechanism of ion pair chromatography is unclear. Retention and selectivity are affected by the chain length and concentration of the pairing reagent, the concentration of organic solvent in the mobile phase, and its pH. Retention increases up to a point as the chain length of the pairing reagent or its concentration increases, then decreases or levels off [1].

Compounds not ionized at the operative pH will not pair with the reagent, but they may still be strongly retained by a C-18 column depending on their alkyl structure. In this case, however, retention will not increase with the addition of an ion pairing reagent, and some decrease in retention may occur, probably due to reagent competition for the stationary phase [1].

Ion Exchange Chromatography

IEC is used to separate ionic compounds. Microparticulate insoluble organic polymer resin or silica gel is used as the support. Negatively charged sulfonic acid groups chemically bound to the support produce strong acid cation exchange (SCX) phases. Positively charged quaternary ammonium ions bound to the support produce strong base anion exchange (SAX) phases. The most widely used resin support is cross-linked copolymer prepared from styrene and divinylbenzene. Mobile phases are aqueous buffers.

Separations in IEC result from competition between the analytes and mobile phase ions for sites of opposite charge on the stationary phase. Important factors controlling retention and selectivity include the size and charge of the analyte ions, the type and concentration of other ions in the buffer system, pH, temperature, and the presence of organic solvents.

Ion chromatography, a subcategory of IEC, has been used primarily for separations of inorganic cations or anions. Because a conductivity detector is usually employed, some means is required to reduce the ionic concentration and, hence, the background conductance of the mobile phase. A second ion exchange suppressor column to convert mobile phase ions to a nonconducting compound may be used. Alternatively, a stationary phase with very low exchange capacity may be used with a dilute, low conductance mobile phase containing ions that interact strongly with the column.

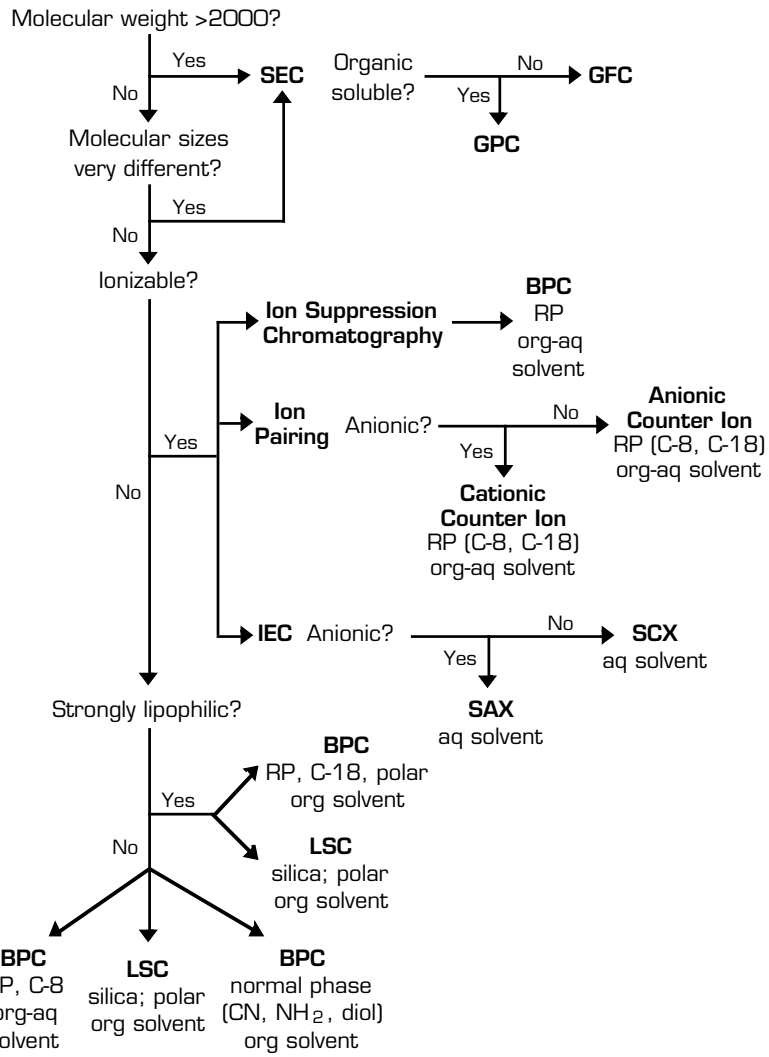
Size Exclusion Chromatography

SEC separates molecules based on differences in their size and shape in solution. SEC cannot separate isomers. SEC is carried out on silica gel or polymer packings having open structures with solvent-filled pores of limited size range. Small analyte molecules can enter the pores and spend a longer amount of time passing through the column than large molecules, which are excluded from the pores. Ideally, there should be no interaction between the analytes and the surface of the stationary phase.

Two important subdivisions of SEC are gel permeation chromatography (GPC) and gel filtration chromatography (GFC). GPC uses organic solvents for organic polymers and other analytes in organic solvents. GFC uses aqueous systems to separate and characterize biopolymers such as proteins and nucleic acids.

The chemist developing an HPLC method must first consider the properties of the analytes of interest and choose an HPLC separation method that best takes advantage of those properties. Many of the references in the bibliography (Section 608) offer guidance to making these choices. A general, simplified guide for selecting an HPLC mode according to the properties of the analyte(s) is illustrated in Figure 601-c; the guide is based on the principles of Snyder and Kirkland [2].

Figure 601-c
Guide to Selection of HPLC Mode
(based on analyte characteristics)



This scheme categorizes analytes as either ionic/ionizable (and therefore water soluble) or nonionic/nonionizable (not water soluble). Based on these distinctions, and on the polarity of the analytes, the diagram provides general rules for choosing an HPLC mode of operation likely to separate the analytes.

601 C: INSTRUMENTATION AND APPARATUS

Basic Components

The following basic components are typically included in an HPLC system (Figure 601-d): solvent reservoir(s); optional gradient-forming device; one or more precision solvent delivery pumps; injector; analytical column and optional precolumn and guard column; column oven; detector; recorder, integrator, or computerized digital signal processing device; and associated plumbing and wiring.

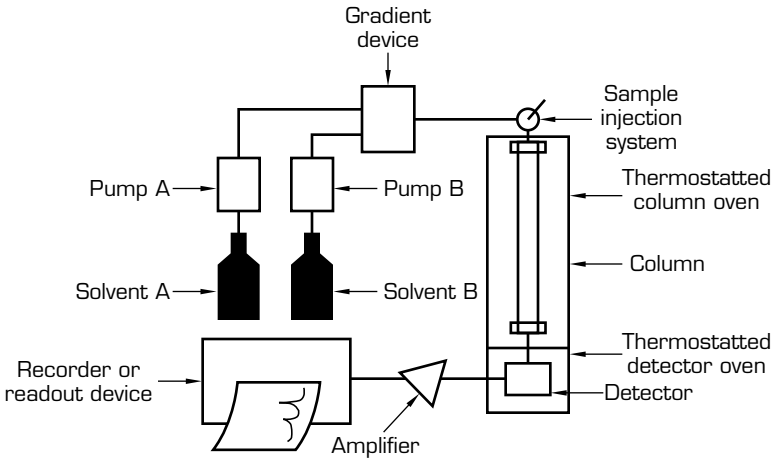


Figure 601-d
Block Diagram of HPLC System

[Reprinted with permission of McGraw-Hill Book Company, from West, C.D. (1987) Essentials of Quantitative Analysis, Figure 14.1, page 346.]

For analytical HPLC, typical flow rates of 0.5-5 mL/min are produced by pumps operating at 300-6000 psi. Although pumps are capable of high pressure operation, state-of-the-art 25 cm × 4 mm id columns with 5 μm packings typically produce 1000-2000 psi at 1 mL/min. High pressures should be avoided because they contribute to limited column life expectancies.

Sample extract is applied to the column from an injector valve containing a loop that has been filled with sample solution from a syringe. After passing through the column, the separated analytes are sensed by visible/UV absorption, fluorescence, electrochemical, photoconductivity, or RI detectors. To minimize extra-column peak spreading, the instrument components must be connected using low dead volume (ldv) fittings and valves and tubing as short and narrow in bore as possible.

Analytical HPLC may use either isocratic or gradient elution methods. Isocratic elution uses a mobile phase of constant composition, whereas the strength of the mobile phase in gradient elution is made to increase continually in some predetermined manner during the separation. Gradient elution, which requires an automatic electronic programmer that pumps solvent from two or more reservoirs, reduces analysis time and increases resolution for complex mixtures in a manner similar to temperature programming in GLC. Gradient elution capability is highly recommended for systems to be used for residue determination. However, it is not always possible to employ gradient elution because some HPLC column/solvent systems and detectors are not amenable to the rapid solvent and pressure changes involved.

Stationary phases are uniform, spherical, or irregular porous particles having nominal diameters of 10, 5, or 3 μm. Bonded phases produced by chemically bonding different functional groups to the surface of silica gel are most widely used, along with unmodified silica gel and size exclusion gels. Columns are usually stainless steel, 3-25 cm long and 4.6 mm id, prepacked by commercial manufacturers. There has been increasing use of microbore columns having diameters ≤2 mm. Although many HPLC separations can be carried out at ambient temperature, column operation in a thermostatted column oven is necessary for reproducible, quantitative results, because distribution coefficients and solubilities are temperature dependent.

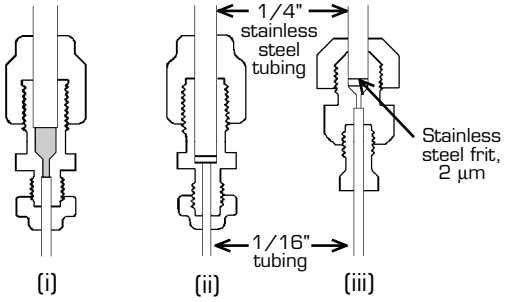
Depending on the nature of the analyte(s), certain additional equipment may be required. For example, apparatus and reagents for performing post-column

derivatization, as used in Section 401 for N-methylcarbamates, may be needed to convert analytes to compounds that can be detected with the required sensitivity and/or selectivity.

HPLC System Plumbing

Band broadening can occur not only in the analytical and guard columns, but also in dead volume in the injector, detector, or plumbing connecting the various components of the HPLC system. This effect, called extra-column dispersion, must be minimized for high efficiency. The proper choice and use of tubing and fittings are critical in this regard.

Figure 601-e
Column Outlet Fittings



(i) Conventional reducing union (dead volume is shaded); (ii) zdv union; (iii) ldv union.

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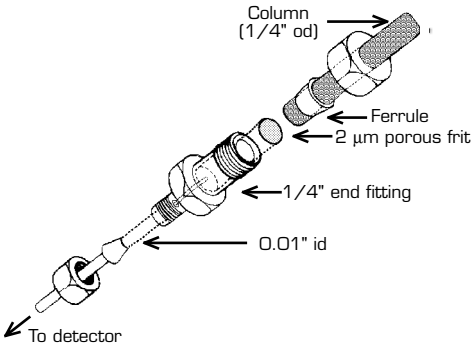
Fittings. Figure 601-e illustrates three types of column outlet fittings. The conventional fitting (i) used in GLC and general laboratory plumbing has excessive dead volume. It has been modified to produce a zero dead volume (zdv) fitting (ii) in which the metal column and the tubing are butted up directly against the stainless steel frit. There is evidence that the nature of the tubing connection in the zdv fitting may lead to some loss in efficiency, especially if the connection is not made carefully. The ldv fitting (iii) improves efficiency by use of a cone-shaped distributor connecting the gauze or frit at the end of the column with the tubing. A typical dead volume for the ldv fitting is 0.1 µL.

Figure 601-f
Low Dead Volume Fitting



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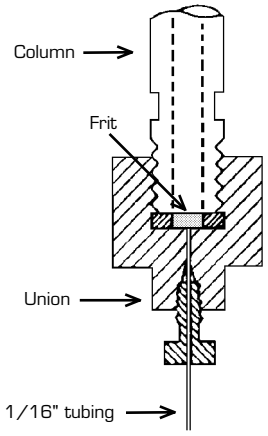
Columns are usually received from manufacturers with a 1/4–1/16" zdv or ldv outlet fitting and a 1/4" nut and cap or a reducing union at the inlet (*i.e.*, not 1/4" in size, but suitable for 1/4" tubing). Figure 601-f shows a complete ldv fitting connection between a column and a detector. The column fits snugly inside the stainless steel end fitting and is sealed by a high compression ferrule. A 2 µm porous frit is firmly seated between the column and end fitting. The column and detector are connected by a short length of stainless steel (or polymer) tubing. The column is also connected to the injection valve using a zdv or ldv fitting and a short length of stainless steel tubing.

Figure 601-g
Standard Internal Fitting



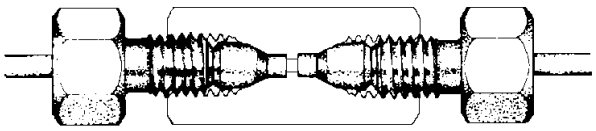
[Reprinted with permission of John Wiley and Sons, Inc., from Meyer, V.R. (1988) *Practical High Performance Liquid Chromatography,* Figure 6.18, page 80.]

External column end fittings (Figures 601-e and 601-f), which were formerly popular, are not durable during repeated attachments and removals. Thus, the internal fitting is practically standard today. This uses female threads in the fitting body and a male nut (Figure 601-g).

Unions. Unions are fittings that connect two pieces of tubing. The most commonly used type is the internal thread ldv type (Figure 601-h). The union is not drilled through completely, but a short (0.02") web of metal is left between the two pieces of tubing with a small diameter (approximately 0.02 or 0.01") hole drilled through. Even though the tubing ends do not butt against each other as in early zdv unions, there is essentially no dead volume added to the system through their use. For this reason, they are commonly classified as zdv unions. This type of union has fewer assembly, re-assembly, and tubing interchange problems than the early butt-together zdv type.

Assembly of Fittings. Fittings consist of four parts: the body, tubing, ferrule, and nut. The nut and ferrule are slid onto the tube end, the tube is pushed all the way into the fitting body and held there securely, the nut is finger-tightened, and then another three-quarter turn is made with a wrench. This procedure should assure that the ferrule is pressed ("swaged") onto the tubing. To replace the ferrule, the tubing must be cut and the fitting remade. When using fittings to connect system components, the nut should be finger-tightened and then tightened a one-half turn more with a wrench. If leaking is observed, slightly more tightening should be sufficient to complete the seal. Overtightening of nuts can lead to fitting distortion and leaks.

Figure 601-h
Internal Thread Low Dead Volume Fitting



[Reprinted with permission of Aster Publishing Corporation, from Dolan, J.W., and Upchurch, P. (1988) *LC-GC* **6**, Figure 3, page 788.]

Fitting components from different manufacturers have dissimilar designs, sizes, and thread types and are usually not interchangeable. Ferrules from different manufacturers have unique shapes, but they are usually interchangeable because the front edge is deformed when pressed onto the tubing. However, as a general rule, it is best to purchase all fittings and spare parts from one manufacturer. Even fittings from a given manufacturer differ slightly because of manufacturing tolerances. However, this is of concern only with microbore columns, for which dead volume is a greater consideration. For these columns, it is best to not even interchange fittings from the same manufacturer.

A variety of fittings are available that can be finger-tightened to the degree necessary to seal stainless steel tubing at 2000-6000 psi. All of these are based on the use of polymeric ferrules, but some have a steel nut, whereas others are all plastic.

They are used mostly on frequently attached and detached high pressure connections, such as between the injector and column or column and detector, and for polymer tubing waste lines from the injector or detector.

Fittings must be kept free of silica particles, which may scratch surfaces between the ferrule and union and cause leaks.

Tubing. Stainless steel tubing is available commercially that is supposedly ready for immediate use in HPLC systems. It is machine cut, polished, and deburred to provide perfectly square ends. It is also cleaned by sonication, passivated, washed, and rinsed with a solvent such as isopropanol to eliminate residual dirt or oils. Despite this careful preparation, it is a wise precaution to rinse new tubing with mobile phase under operating pressure before using it as part of the HPLC system.

The most commonly used tubing for connecting components of the chromatograph is 316 stainless steel, 1/16" od, with different inside diameters. Tubing with 0.01" (0.25 mm) id is commonly used in areas where dead volume must be minimized to maximize efficiency, *e.g.*, between the injector and column, precolumn and column, columns in series, and column and detector, and for preparing pulse damping spirals.

Typical lengths of tubing connections are 3-6 cm. Tubing with 0.005 or 0.007" id is used to connect microbore or short 3 µm particle size columns to detectors and injectors. Filtering of samples and solvents is especially critical to prevent clogging of this narrow bore tubing. Tubing with 0.02-0.05" id is available when ldv is not important and low resistance to flow and pressure drop is desirable. For example, 1 mm (0.04") tubing is often used between the pump and sample injector.

Tubing can be cut to any required length in the laboratory, but it is important not to distort the interior or exterior during the process. The simplest method is to score the tubing completely around the outside with a file and then bend it back and forth while holding it on either side of the score with two smooth-jawed pliers. The ends are filed smooth and deburred, and the tubing is thoroughly washed with solvent. If the bore should become closed by the bending and filing, the tube can be reamed out with an appropriate drill bit before final smoothing and washing. A number of types of manual and motorized tubing cutters are available from chromatography accessory suppliers. Proper cutting of tubing to make leak-free connections is an art that requires considerable practice.

Although stainless steel tubing and fittings are standard for systems using organic and salt-free aqueous solvents, corrosion becomes a problem with buffers containing salts, particularly halide salts at low pH. HPLC companies have available a variety of accessories that can solve this problem. These include titanium high pressure system components, for use in the flow stream at all points of mobile phase contact, and titanium or polymeric fluorocarbon tubing with id values similar to stainless steel. One such polymer is Tefzel (ethylene-tetrafluoroethylene copolymer), which can withstand pressures of 5000 psi or higher. (Teflon is limited to pressures <1000 psi.) Titanium and polymeric plumbing components are especially valuable for biochemical HPLC and ion chromatography.

Reference 3 is a valuable source of information to help avoid many tubing installation problems.

System Leaks. Leaks are relatively easy to detect in LC instruments because liquid will be visible around a loose fitting. A loss of system pressure when using a constant volume pump is a common sign that a leak may be present. If this occurs, all fittings, especially sample valve and column fittings, should be checked and tightened if necessary with two open-ended wrenches. Care must be taken not to overtighten. If leaking does not stop, the faulty fitting must be replaced.

601 D: SOLVENTS AND REAGENTS

The mobile phase in HPLC is chosen for its ability, in combination with a particular column, to provide the required separation of the analyte(s). The solvents used to prepare the mobile phase must be of high purity, most often HPLC grade, spectrophotometric grade, or distilled from all-glass apparatus. Other factors of importance include cost, viscosity, toxicity, boiling point, compressibility, UV transparency (if a UV detector is used), RI (if an RI detector is used), vapor pressure, flash point, odor, inertness with respect to sample compounds, and ability to cause corrosion. Choices of solvents and reagents cannot be made without careful consideration of the effect their presence can have on the entire HPLC system.

Solvents and reagents used in the HPLC determinative step and in sample preparation procedures preceding HPLC should not:

1) cause degradation or unintended reaction of the analyte(s);

2) cause the solvent delivery system to malfunction;

3) cause damage to the analytical column;

4) cause damage to the detector; or

5) contribute noise or increased or decreased detector response for the analyte.

Potential Problems

Many of the problems with mobile phases arise because of the presence of impurities, additives, dust or other particulate matter, or dissolved air. Examples of some specific potential problems with solvents and reagents and suggested solutions follow.

Degradation. Analytes can be degraded by solvents and reagents used in the extraction and cleanup steps of the analysis, or in the HPLC step itself. Analyte chemistry is usually known in advance, and reagents likely to cause degradation can be avoided. Unexpected reaction of the analyte(s) will usually be demonstrated by poor or no recovery of the compound(s) through the method, or by detection of additional reaction products in the determinative step.

The presence of impurities in solvents or reagents is often the cause of such unexpected reactions. For example, traces of oxidizing agents in solvents have been found to degrade N-methylcarbamates prior to their determination by HPLC. Purity of all reagents used in trace-level determinations should always be as high as possible.

Dissolved Gases. The presence of dissolved gases in solvents composing the mobile phase is a major cause of practical problems in HPLC. Gas bubbles can collect in pumps, the detector cell, or other locations in the HPLC system. This can affect the reproducibility of the volume delivered by the pump, or large bubbles may completely stop the pump from working. Detection can be affected in various ways. With the UV detector, air in the detector cell can cause seriously increased detector noise or high absorbance. Dissolved oxygen can interfere with detection at short wavelengths, as oxygen absorbs radiation at <200 nm. Solvents must be "degassed," a topic covered in Section 603 B, Mobile Phase Preparation.

Damage to Columns. HPLC columns are easily damaged and expensive to replace. Bases can remove the functional groups from bonded HPLC phases. Therefore, bases should not be used in analyses involving BPC unless their removal prior to chromatography can be assured. Bonded phases are usually stable in the pH range of approximately 2-8.

Microscopic particles and microorganisms can clog column frits or even the top of the column itself. If this happens, the pressure drop across the column for a given flow will gradually increase, and the column may eventually become completely blocked. Filtration of the sample solution and mobile phase to remove particles ≥5 μm, and the use of an appropriate precolumn and guard column, are recommended to protect the analytical column. Particles <5 μm may be of concern with some columns and detectors.

Any mobile phase, especially one containing water or methanol, can dissolve silica gel in unmodified and bonded silica gel columns. A precolumn containing silica gel can be positioned between pump and injector to saturate the mobile phase with silica gel so that the analytical column is not dissolved.

Both precolumns and guard columns are discussed in Section 602 E, Analytical Column Protection.

The potential for damage to the column by reagents used in post-column derivatization is unlikely but not impossible. If the flow of the mobile phase is stopped, post-column reagents can diffuse back through the column effluent onto the column. This can result in deterioration of the column packing.

Damage to Detectors. The potential for reagent damage varies with each detector. As stated above, the compressibility of dissolved gases in solvents can cause bubbles to appear in the detector cell and interfere with the analysis. Traces of oxygen are incompatible with electrochemical detectors operating in the reductive mode; oxygen can also cause quenching in fluorescence detectors, leading to reduced sensitivity. Degassing of solvents is required. Porous flow-through coulometric detectors can be clogged by the presence of particles ≥0.2 μm. Filtration of solvents through a 0.22 μm filter is essential when using this type of detector.

Solvent Impurities. Many reagent grade solvents contain levels of impurities that make them unsuitable for use in HPLC. Sometimes the impurities are added deliberately by manufacturers as antioxidants, stabilizers, or denaturing agents. For example, chloroform usually contains up to 1.0% methanol or ethanol, and tetrahydrofuran may contain butylated hydroxytoluene or hydroquinone. These impurities may cause increased or decreased detector response or change the mobile phase strength and/or selectivity.

In some cases, incompatibility of a solvent or reagent with the HPLC system can be determined in advance and avoided. In the case of unknown impurities, problems will be recognized only during use of the chemical; careful investigation will be needed to determine the cause of the problem. Even microorganisms in inadequately purified water can cause a high background signal in some detectors (see Water, below). Whenever possible, HPLC grade solvents should be used to prepare mobile phases. Spectral or pesticide grade solvents may not be adequately pure for HPLC use. Solvents should be adequately purified and tested before use.

Specific Solvents

Water. Water is probably the most commonly used solvent in HPLC because of its role as the strength-adjusting solvent in RP mobile phases. It is also one of the most difficult solvents to purify and maintain in the pure state. Purity of water is especially critical in the determination of trace residues, when detectors are operated at high sensitivity.

Purification of water by distillation, even triple distillation, is inadequate because volatile and codistilled organics will not be removed. Bonded RP columns will collect these impurities over long term use, which can alter the properties of the column or sometimes produce spurious peaks. Water can be purified by distillation from potassium permanganate, by passage through a coarse grained C-18 bonded phase column that is periodically regenerated with acetonitrile, or by means of a commercial water purification system.

One widely used water purification system (Millipore Milli-Q) pumps distilled water through a prefilter cartridge to eliminate particulates; then through sequential cartridges of charcoal, ion exchange resin, and Organex-Q; and finally through a 0.22 μm filter. The activated charcoal cartridge removes organic impurities that can interfere with spectroscopic detectors. The mixed bed ion exchange resin cartridge(s) removes inorganics and ionized organics, as well as impurities leached from the charcoal; this removal is essential for proper operation of electrochemical detectors. The Organex-Q cartridge eliminates any remaining organics, in addition to traces of material leached from the ion exchange cartridge. The final 0.22 μm filter removes microscopic particles and microorganisms not eliminated by the previous cartridges. This filtration step protects column frits, columns, and porous flow-through detectors from particles that could clog them. It also minimizes the possibility that microorganisms will grow sufficiently to cause a background detector signal. The quality of the feed water is improved and the life of the purification system is extended if a reverse osmosis system is included between the prefilter and carbon cartridges. This system lowers the base level of organics, inorganics, and microorganisms.

Microorganisms such as bacteria and algae multiply rapidly in water. Therefore, even when using water purified in the manner just described, it is wise to discard all remaining water at the end of each week. The HPLC system should be flushed with methanol to destroy any microorganisms that have entered it during the week. At the beginning of a new work week, the water reservoir should be washed with methanol prior to filling with newly purified water. Growth of microorganisms can also be prevented by adding 0.02% sodium azide or acetonitrile (which is present in many RP mobile phases) to the water.

Purified water is best stored in carefully cleaned glass containers. Plasticizers can leach into water stored in plastic containers, interfering with RP systems or

contaminating the column. Leaching of metals from glass containers is also a possibility, but this is usually less of a problem than introduction of organic impurities.

HPLC grade water can be purchased from a number of commercial sources. This water can be used successfully as received for most applications.

The following purity check can be used to test water for applicability in HPLC:

- Pump 100 mL water through C-18 column.
- With a UV detector in-line, run a linear gradient from 0 to 100% methanol at 1 mL/min for 10 min and hold for 15 min.
- If the UV baseline shift at 0.08 AUFS is <10% and very few peaks of <3-5% full scale deflection are observed, the water is pure enough for most applications.

Acetonitrile. Acetonitrile is commonly used in RP HPLC mobile phases. Manufacturers' specifications for HPLC solvent purity are usually based on acceptability for UV detectors. Specifications for fluorescence and electrochemical detectors are very difficult to define because of the complexity of instrumental parameters.

Methanol. Another of the more common solvents employed in RP HPLC is methanol, which suffers from the same inadequacy of specifications as acetonitrile. Methanol has the disadvantage of producing relatively viscous solutions when mixed with water, giving rise to much higher pressures than with other mobile phases.

Chlorinated Solvents. Some chlorinated solvents are stabilized against oxidative breakdown by addition of small amounts of methanol or ethanol. Alcohol will increase polarity of mobile phases and shorten elution times in NP HPLC. Also, reproducibility will be affected because the concentration of stabilizer will vary slightly from batch to batch.

Chlorinated solvents can be purchased without stabilizer, or the stabilizer can be removed by adsorption onto alumina, or by extraction with water followed by drying. Unstabilized chlorinated solvents may slowly decompose, producing hydrochloric acid, which degrades columns and corrodes stainless steel. The rate of decomposition may be accelerated by the presence of other solvents. Hydrochloric acid can be removed by passing the solvent through activated silica or calcium carbonate chips. Solvents can be stabilized with amylene to avoid these problems.

Gillespie *et al.* [4] noted problems such as increased detector response and discoloration of equipment when ethylene dichloride or methylene chloride was used in HPLC mobile phases. The problems described were attributed to a reaction between solvent impurities and stainless steel upon prolonged contact.

Ethers. Ethers contain additives to stabilize them against peroxide formation. For example, tetrahydrofuran is often stabilized by addition of small amounts of hydroquinone. This compound absorbs UV radiation and so interferes with UV absorption detection. It can be removed by distilling the solvent from potassium hydroxide pellets. Inhibitor-free tetrahydrofuran should be stored in a dark bottle and flushed with nitrogen after each use. Any peroxides that form should be periodically removed by adsorption onto alumina.

Reagent Blanks

Blank samples should be analyzed to ascertain that no interferences from reagents (or glassware) occur during analysis. Reagent blanks are especially important when using nonspecific optical detectors such as UV or RI detectors.

Safety Precautions

Beyond the concern over damage to HPLC systems that can be caused by reagents and solvents, it is important to protect the health of the analyst. An awareness of the toxicity of the chemicals in use is essential. Care must be taken to minimize exposure to toxic chemicals. See Reference 5 for more on laboratory safety for HPLC analysis.

601 E: SAMPLE PREPARATION

Sample Cleanup

Extracts to be analyzed by HPLC must be cleaned up (*i.e.*, interfering co-extractives removed) sufficiently to permit identification and quantitation of residues, and to prevent contamination or harm to any part of the HPLC system. The column and/or detector may be impaired by injection of dirty extracts, especially when many samples are analyzed.

Cleanup procedures for trace residue determination by HPLC must be developed to accommodate the selectivity of the detector. Dissolved interferences in the sample solution that appear in the chromatogram as extra peaks must be removed. Any materials that will be strongly adsorbed by the column must also be removed to prevent their affecting chromatographic characteristics of the column, causing baseline drift, or appearing as spurious peaks in later chromatograms.

A recent innovation combines cleanup of the sample extract in-line with the HPLC determinative step [6]. A short column of SCX resin replaces the sample loop in a six-port HPLC injection valve, where it effectively removes the analyte, formetanate hydrochloride, from the extract. Solvent flushing of the column while the short column is still off-line (disconnected from the analytical column) provides cleanup and substitutes for traditional separatory funnel partitionings. Subsequent switching of the valve places the cleanup column in-line with the analytical SCX column for elution and determination. This coupled column application and other multidimensional variations [7] provide simple, rapid analysis with minimum solvent use.

Sample Filtration

Removal of particulate matter in the sample solution is critical for HPLC stability. Both column frits and the top of the column packing can become clogged by particles, leading to increased back pressure and adverse effects on chromatographic results because of decreased column efficiency, production of split peaks, *etc.*

At a minimum, samples should be passed through a commercial clarification apparatus, such as a syringe and a 5 µm filter pad in a Swinny adapter, before injection. In residue determination, passing samples through filters with <1 µm

pores is preferred. If the detector in use is of the porous flow-through type, the sample should be filtered to remove particles >0.2 µm. In addition, in-line filters placed ahead of the column can be used to prevent clogging of column frits. It is important to ensure that the analyte is not lost on the filter medium, especially for quantitative determination. This should be determined by analysis of samples fortified with known concentrations.

Sample Solvent Degassing

Sample extracts should be prepared for injection using solvents that have been degassed in the same manner as mobile phase solvents (see Section 603 B, Mobile Phase Preparation). This will reduce the possibility of problems when the sample solvent enters the detector cell. The sample solution itself should not be degassed because evaporation will change its concentration.

Choice of Sample Solvent

Ideally, the sample should be dissolved in the mobile phase. This reduces the size of the solvent peak, thereby aiding identification of early eluting sample peaks. It also avoids sample precipitation on or before the column, which can result in the loss of peaks for the analyzed sample and appearance of unknown, randomly eluting peaks in chromatograms from subsequent injections. This could occur, for example, if the mobile phase is methanol/water and the sample is dissolved in neat methanol because of insolubility in the mobile phase. As a precaution after using a different sample solvent, the column should be flushed with a strong solvent that is compatible with the column, followed by equilibration with the mobile phase before injection of the next sample. Ultrasonic mixing may aid in dissolving the sample in the mobile phase or a similar solution.

If the sample must be prepared in a solvent different from the mobile phase, it should be compatible with the column, as close as possible to the mobile phase in composition, and of weaker elution strength if this is consistent with solubility requirements. In addition to possible sample precipitation as described above, injection in a stronger solvent can cause peak tailing. If a stronger solvent must be used, the smallest possible volume should be injected.

601 F: REFERENCE STANDARDS

General procedures for storage, handling, and preparation of solutions of analytical reference standards for pesticide residue analysis are covered in Section 205. Preparation, storage, and stability are described in greater detail in Reference 8. The nature of HPLC makes it the preferred determinative step for many unstable, reactive, or easily degraded pesticides. For this reason, the stability of the pesticide in the solvent used to prepare standard solutions requires particular attention.

Stock Solutions

Considerations for the choice of a solvent for preparing stock standard solutions are the same as for choosing a solvent in which to inject samples (see Section 601 E). If stability permits, standard solutions should be prepared in the mobile phase to be used in the HPLC analysis. However, many pesticides have limited stability in "reactive" solvents, such as methanol or water, often used for mobile phases. For example, the fungicides thiophanate-methyl, captan, folpet, and captafol can be

stored indefinitely in benzene, acetone, or isooctane, but they quickly degrade in methanol/water.

Alternatively, stock standard solutions can be prepared in a less reactive solvent with a fairly high volatility (*e.g.*, acetone). Working standard solutions can then be prepared by evaporation of the volatile solvent from an aliquot and subsequent dissolution in the HPLC mobile phase or other appropriate solvent.

Benzene is a good solvent for most pesticide standards, but its toxicity makes its use inadvisable. Isooctane and hexane dissolve most organochlorine pesticides; isooctane's low volatility minimizes evaporative loss during storage, but also precludes its use in cases where it is desirable to evaporate the original solvent prior to dissolution in the mobile phase. Chloroform is useful for triazines, methylene chloride or methanol for carbamates, acetone for benzimidazole-related fungicides, and methanol for phenylurea herbicides.

Because of possible deterioration due to evaporation and/or instability, it may be necessary to remake stock standard solutions frequently. Because standard reference materials are often supplied in limited quantities (<100 mg), use of a microbalance is preferred for accurate weighing of low mg quantities of standard for preparation of stock solutions. Direct preparation of dilute solutions in this way can also reduce the number of dilutions required to make the working standard solution.

Working Standard Solutions

These solutions are prepared at concentrations suitable to the detector in use and the expected levels of pesticides in sample extracts. Concentrations of working standard solutions should closely match those in sample extracts for the most reliable comparison of peak heights or areas. For general screening purposes or multiresidue analysis, working standard solutions can be made up as mixtures of pesticides resolvable by the method.

Stability of working standard solutions should be confirmed by periodic comparison against newly prepared solutions or fresh dilutions of stock solutions. Solvents used to prepare working standard solutions should be compatible with the sample solvent and the HPLC system (see Section 601 E) and should be checked for contaminants that could possibly interfere with the analysis.

Storage

Stock standard solutions should be stored in an explosion-proof refrigerator at ≤4° C. Benzene solutions can freeze at these temperatures and may crack containers. Organochlorine pesticide stock solutions can be stored for at least 6 months without deterioration. Organophosphorus and carbamate solutions are less stable and should be discarded 3-4 months after preparation. Some standard solutions degrade quickly and must be made fresh at least daily.

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602: COLUMNS

The nature and dimensions of the column packing, together with the nature of the mobile phase, largely determine the selectivity and efficiency of the separation that is achieved. HPLC columns are packed with small particles (usually 3-10 μm) having a narrow size distribution (approximately ± 20%). The use of microparticulate materials requires that the mobile phase be pumped through the column at high pressure. Columns can be prepared in the laboratory, but most analysts purchase commercial prepacked, pretested columns.

An HPLC column is a highly efficient filter, and any particulate matter or strongly retained impurity that is injected will remain on the top. To prevent deterioration of the analytical column, a guard column should be installed between it and the injection device. The guard column is discarded or repacked after a certain number of sample injections. A saturation precolumn situated between the pump and injector device may be used to ensure equilibrium between the two phases in a liquid-liquid chromatography system, or to prevent dissolution of silica from an unmodified or bonded silica analytical column. Although columns of different sizes have been used, 25 cm × 3-5 mm id columns packed with 5 or 10 μm stationary phase material have provided adequate separation in a reasonable time for many applications.

602 A: COLUMN SELECTION

Column selection is not a straightforward process. The best approach is to search the literature for work published on a separation that is the same as, or similar to, the one that needs to be accomplished. Many of the references in Section 608 discuss column selection techniques for different sample types, and most column manufacturers have published guides and technical data sheets that will aid in column selection.

A knowledge of the chemistry of the sample, often determined by some simple wet chemistry experiments, combined with a systematic trial and error approach, is probably the method used most often in column selection. If the molecular weight, range of solubility, and molecular or ionic structure of the analyte are known, a mode of separation can be selected as discussed previously (see Figure 601-c). The most appropriate column for that mode is then chosen, based on the experience of the analyst, column manufacturers' recommendations, or a search of the literature.

602 B: ANALYTICAL COLUMNS

Factors important in producing efficient columns include narrow particle size distribution in the packing and minimal dead volume in the tubing, fittings, cells, and other components of the HPLC instrument.

Most packed columns are made from stainless steel. In addition, glass cartridge columns are common and radial compression columns prepared from heavy wall polyethylene cartridges are available. The latter columns are radially compressed in a hydraulic press during use to minimize void volumes and wall effects and thereby increase column efficiency.

Recent advances in column technology include use of 3-10 cm columns packed with 3-5 μm particles. The major advantages of these shorter columns over conventional 25 cm columns are faster separations and improved sensitivity of detection. Another trend is the use of microbore columns, 0.2-1 mm id columns packed with conventional bonded phases. Microbore columns can be made very long, providing up to one million theoretical plates for difficult separations. They require only small volumes of mobile phases and allow novel detection possibilities, including flame ionization, chemical ionization mass spectrometry, and IR spectrometry.

Normal phase (NP) HPLC is carried out on adsorbent (silica gel, alumina) columns or polar bonded (cyano, amino, diol) columns. Liquid solid chromatography and polar bonded phase chromatography are suitable for separation of nonionic multifunctional compounds and isomers. Silica gel is by far the most used column for NP separations. However, because NP columns have not been used widely for analytical work, most discussion of columns in this section refers to various types of reverse phase (RP) chromatography used for pesticide determination.

Liquid-Solid Chromatography

Until recently, little use had been made of liquid-solid chromatography (LSC) for pesticide analysis. Now, however, a column of porous graphitic carbon, a nonpolar RP adsorbent, has been successfully applied to the determination of ethylenethiourea (ETU) using a strongly acid mobile phase [1]. Such columns offer stability for applications requiring pH extremes and are complementary to silica-based columns.

Bonded Phases

Most analytical HPLC systems use RP chromatography on silica-based C-18 or C-8 bonded phases. Other RP bonded packings include those having C-1, C-2, C-4, C-12, cyano, phenyl, diol, or cyclohexyl groups. In RP mode, the stationary phase is hydrophobic and nonpolar, and mobile phases are relatively polar (usually water with methanol or acetonitrile). Nonpolar sample components are strongly retained, and polar components are less retained. Bonded columns are stable and reproducible compared to nonbonded columns with physically adsorbed coatings, which they have almost completely replaced. The major limitation is the narrow pH range for column stability.

Most commercially available bonded phases are of the siloxane type, Si-O-Si-R. They are prepared by reacting surface silanol groups on silica with an organochlorosilane reagent, the organic portion of which is the moiety to be bonded (octyl, octadecyl, phenyl, aminopropyl, cyanopropyl, *etc.*). Packings can be prepared, for example, by using mono-, di-, or trichloroorganosilanes to produce products having different chromatographic properties. Monochloroorganosilanes react with silica to form a monomolecular layer of bonded organic groups. Di- or trichloroorganosilanes react with silica in the presence of a protic reagent to form a linear or cross-linked polymeric layer, the structures and properties of which are not as well defined as with monomeric phases. Polymer bonded phases have poorer mass transfer characteristics but higher loadability. Some of the accessible unreacted silanols on the silica surface after the primary bonding reaction may be removed by end-capping, which involves reaction with a less bulky reagent such as trimethylchlorosilane.

Most of the current bonded RP columns have 5 µm spherical silica as the base material. Pore size ranges from 60-300 nm, with 80-120 nm most common. To increase the range of pH stability, bonded columns having polystyrene-divinylbenzene (DVB) polymer as the base material have been developed. Another approach is a base material composed of alumina coated with a polybutadiene polymer layer to protect the bonded surface from attack by hydroxide. Stability up to pH 13 is possible for such columns because alumina is stable at this pH.

Short chain phases such as C-2 and C-4 are used to reduce hydrophobic interactions in separating high molecular weight analytes, such as proteins and peptides. Cyanopropyl phases can be used in NP work by selective interactions with the cyano functional group or as a short chain RP material for separation of polar analytes. Diol phases, whose structures involve two hydroxy groups on adjacent carbon atoms in an aliphatic chain, are less polar than silica and are used in both NP and RP chromatography. Phenyl phases are prepared by the reaction of dimethylphenylchlorosilane with silica gel. They are nonpolar and have special affinity for aromatic compounds. Cyclohexyl phases have selectivity for alicyclic compounds compared to straight chain compounds. Some RP columns are base-deactivated to optimize separation of basic compounds without tailing or need for mobile phase modifiers for ion pairing or ion suppression.

The determinative steps of Sections 401, 403, and 404, methods for N-methylcarbamates, substituted ureas, and benzimidazoles, respectively, provide examples of applications of bonded RP HPLC to pesticide residue analysis.

Ion Exchange

Four types of microparticulate packings are available for high performance ion exchange chromatography (IEC). Polystyrene-DVB polymeric gel resin particles of 5-10 µm diameter substituted with ionogenic groups were the earliest of these packings. The amount of DVB added for the polymerization reaction determines the degree of cross-linking and, hence, the pore structure. Resins with <6% DVB are not pressure stable and cannot be considered HPLC packings. Slow diffusion of analytes within the polymer matrix and the resulting poor efficiency led to development of pellicular ion exchange materials, consisting of a glass core, an intermediate coating of silica, and an outer ion exchanger polymer film. These materials suffer from low efficiency due to their relatively large particle size and low sample capacity.

Silica-based ion exchange packings are prepared in a manner similar to other bonded phases. Controlled porosity glass column packings with attached hydrophilic polymeric groups can be used for high speed separations of large ionic molecules such as proteins and nucleic acids.

Virtually all commercial ion exchange materials contain sulfonate (strong cation exchange), carboxylate (weak cation exchange), tetraalkylammonium ion (strong anion exchange), or an amine (weak anion exchange) functional group. The capacity of exchangers is a function of the pH of the mobile phase. Full exchange capacity is exhibited by different exchangers at the following pH values: strong cation, above 3; weak cation, above 8; strong anion, below 9; and weak anion, below 6. The wide exchange range of strong exchangers makes them most useful for general analytical work. The pH of the mobile phase controls retention by its effect on the ionic nature of both the sample and the exchange sites.

IEC has been applied to determination of residues of formetanate hydrochloride [2]. A strong cation exchange mechanism is used for the chromatography of this ionic residue.

Ion Pair

RP ion pair chromatography is an alternative to IEC. It is an extension of ion suppression chromatography, in which weak acids or bases are separated on an RP bonded column by addition of a pH modifier to the mobile phase to ensure that analytes are in their undissociated forms.

In ion pair chromatography, a charged organic compound is added to the mobile phase to form a neutral ion pair with an analyte of opposite charge. For example, an alkylsulfonate can be added to cationic samples and tetrabutylammonium phosphate to anionic substances. Ion pair chromatography is suitable for separating mixtures of anions, cations, and neutral substances; the pH of the mobile phase will suppress the ionic character of one of the types of ions, while the counter ion will react with the other type to form ion pairs. For example, tetrabutylammonium phosphate buffered to pH 7.5 can form ion pairs with strong and weak acids, and the buffering suppresses weak base ions. Amphoteric molecules can be chromatographed with either quaternary amine or sulfonate counter ions at an appropriate pH value.

Selectivity can be affected by the concentration and choice of the ion pair reagent. The k values (see Section 602 C) of analytes are proportional to the counter ion concentration. The longer the alkyl chain length, the greater are k values. Retention times can also be adjusted by changing the composition of the mobile phase, which is usually a mixture of water with either methanol or acetonitrile.

Quaternary ammonium salts in alkaline medium are damaging to silica gel. Columns should never be stored in such solutions. A precolumn placed in front of the injector, to saturate the mobile phase with silica gel, is highly recommended in these systems.

An example of pesticide determination using ion pair chromatography (on a bonded phase) is the determination of benzimidazole residues (Section 404). In addition, two methods for determining residues of paraquat and diquat use the ion pair mechanism, one with a polymeric (PRP-1) column [3], and the second with a silica column using NP mode chromatography [4].

Size Exclusion

Separations in the size exclusion (SEC) mode are based on molecular size and are controlled by the pore size of the packing material. Particle sizes in the 5-20 μm range are used to provide good column efficiency. Packings for SEC include semirigid organic gels, porous silica, and controlled pore glasses.

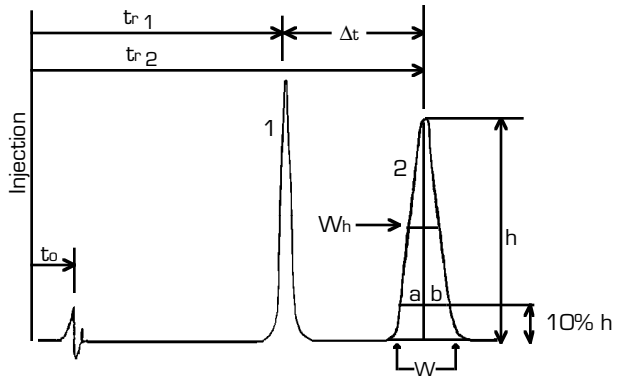
The major use of SEC in pesticide determination is for cleanup of residues from fatty samples by gel permeation chromatography, rather than as a determinative step. The most used packing for this purpose has been styrene-DVB copolymer such as Bio-Beads S-X3 (Section 304 C5, Section 402). The Bio-Beads S-X series offers exclusion limits from 400-14,000 molecular weight; S-X3 has a 2000 exclusion limit. The exclusion limit is determined by the amount of DVB cross-linking

of the gel, as well as by the degree of swelling that can occur in different solvents. Maximum expansion of the gel occurs in relatively nonpolar solvents. Typical solvents used include benzene, toluene, xylene, carbon tetrachloride, methylene chloride, and mixtures such as methylene chloride/hexane. The sample should not interact with the stationary phase in any way, *e.g.*, by adsorption. Stationary phase with a smaller particle size will provide greater peak capacity, and better and faster separations.

602 C: COLUMN EVALUATION

An HPLC column can be evaluated by measuring certain performance characteristics or parameters, many of which can be visualized or measured on the chromatograms produced by the column. Column efficiency and peak symmetry reflect the quality of the column, whereas the capacity factor and selectivity indicate its capability to retain and separate compounds of interest.

Figure 602-a
Calculation of Column Performance Parameters



Terms

t_r = retention time, mm
t_o = elution distance of unretained component, mm
t'_r = t_r-t_o (adjusted retention time)
a,b = peak half-width at 10% peak height, mm
Δt = time between peak maxima, mm
W = peak width at base, mm
h = peak height, mm
W_h = peak width at half height, mm

Capacity factor, k = t'_r/t_o
Selectivity, α = k_2/k_1 *or* t'_{r_2}/t'_{r_1}
Efficiency, n theoretical plates = $16(t_r/W)^2$ *or* $5.54(t_r/W_h)^2$
Efficiency, HETP = column length (cm)/n
Resolution, R_s = $2(\Delta t)/(W_1+W_2)$ or $(1/4)(\alpha-1)\sqrt{n}(k/1+k)$
Peak asymmetry, A_s = b/a

References: Walters, M.J., *et al.* (Nov.1980) "Recommendations for HPLC Columns," LIB 2447, FDA, Rockville, MD; *ASTM Standards on Chromatography* (1981) E682.

Several terms must be measured in order to calculate the parameters of a column. Figure 602-a provides a visual representation of these terms:

The time from injection to the peak maximum is known as the retention time, t_r. The retention time consists of two parts, t_o and t'_r. t_o is the time from injection to emergence of the solvent front, which may be noted as a small shift or disturbance in the baseline or a solvent peak if the sample solvent is different from the mobile phase and is sensed by the detector. t'_r, the adjusted retention time, equals t_r minus t_o. t'_r represents the time that the analyte is retained in the stationary phase.

Δt is the time between the maxima of two peaks, and W is the peak width determined between the intersections of tangents drawn on the sides of the peaks with the baseline. All of these time values can be measured in mm directly on the

recorder trace of the chromatogram. These terms are used to calculate the following parameters for evaluation of columns: capacity factor, selectivity, efficiency, resolution, and peak asymmetry.

The capacity factor, k, measures retention of an analyte by the column in terms of column volumes. It is affected by the strength (*e.g.*, polarity) of the mobile phase and the strength (retentivity) of the column packing. A k value of 2-10 for the most retained component is generally optimal for good resolutions but may be higher for difficult separations.

Selectivity is a thermodynamic factor that measures the ability of a particular column/mobile phase combination to provide different distribution constants for two substances, thereby causing a different degree of retention for the two substances, as indicated by the separation of their peak maxima. It is symbolized by α and calculated as the ratio of t'_r values or k values for two peaks, with the largest value placed in the numerator. Selectivity is affected by the chemistry of the entire system, including the functionality of the sample components.

Efficiency is a kinetic factor that indicates the ability of the column/mobile phase combination to produce narrow peaks. Efficiency is dependent on particle size, column dimensions, and packing technique. It is determined by the number of theoretical plates, n, and height equivalent to a theoretical plate, HETP.

Resolution is the ability of the column/mobile phase combination to separate the peaks representing two substances. It is a function of efficiency, selectivity, and retention and is improved by increasing the separation of the peaks (selectivity) and/or by decreasing their width (increasing efficiency). Resolution should be >1 to minimize error in quantitative analysis. A retention, k, of 2-10 is usually assumed.

Peak asymmetry describes the shape of a chromatographic peak. Theory assumes a symmetrical, Gaussian shape for peaks, but asymmetry can be caused by extra-column effects, poorly packed columns, deterioration of packing, incompatability between analyte and packing, *etc.* The peak asymmetry factor is the ratio, at 10% peak height, of the distance between the peak apex and the back side of the chromatographic curve to the distance between the peak apex and the front side of the chromatographic curve. A value of 1 indicates a symmetrical peak, a value >1 is a tailing peak, and a value <1 is a fronting peak.

Higher efficiency, which leads to sharper peaks, is achieved by using columns with small, uniform, tightly packed particles and optimized column flow rates. High selectivity, which is manifested by well separated peak maxima, is influenced mostly by the nature of the stationary and mobile phases.

602 D: COLUMN SPECIFICATIONS

The parameters described above can be used to evaluate column quality. Columns that produce the desired separation should be defined for future reference by the measured parameters. A "system suitability test" that specifies acceptable operation of the HPLC determinative step should be included with any method description; this may require the use of specific compounds involved in the procedure. System suitability test elements that relate to column specifications are listed in Table 602-a.

Table 602-a: HPLC Column Specification Elements

Physical Description

Packing material

- particle type: size, shape, pore size
- bonded surface type: functionality, mono or polymeric
- surface coverage: (% concentration or μmoles/m^2)
- additional silylation

Column dimensions

Performance Characteristics

[Requires that the test system be defined by specifying mobile phase solvent and flow rate, test solution compounds, and solvent. Characteristics must be related to peak(s) that were used to measure each.]

- Minimum theoretical plates, n
- Resolution, Rs
- Selectivity, α
- Capacity factor, k
- Asymmetry, As

At a minimum, a new analytical column should be checked for efficiency by calculating and recording the number of theoretical plates using an appropriate test solution. This value is compared with the manufacturer's specifications and used in later column quality control evaluations.

Expected minimum efficiency values are shown in Table 602-b. In general, efficiency (plates per meter) decreases with larger or less uniform size column packing, lower temperature, increased extra-column volume in the system, and larger samples. Efficiency improves when k = <2 unless extra-column effects are dominant.

Specifications and test systems for six satisfactory HPLC bonded phase silica columns were recommended at an early stage of HPLC application [5]. These recommendations are useful as a guideline for comparing and defining columns, but the specifications themselves are no longer applicable because of subsequent improvements in HPLC column technology. Other protocols for column testing and evaluation have been suggested. For example, Poole and Schuette [6] described test conditions and specifications for a 10 μm C-18 RP column using a mixture of resorcinol, naphthalene, and anthracene and a UV detector.

Commercial bonded silica RP columns from different manufacturers are not equivalent, and information on the degree of hydrocarbon coverage in a column is not usually provided. In addition, the free (unreacted) silanol sites vary among

Table 602-b: Minimum Efficiency Values
(in thousands of theoretical plates per meter)

	Particle Size		
Column Type	10 µm	5 µm	3 µm
porous RP bonded	12-20	35-40	80-100
porous silica gel adsorbent	24	40	
porous ion exchangers	10-15		
semirigid organic size exclusion gels	9-12		

columns and manufacturers, and these can have significant effects on the chromatography of polar analytes. A test scheme developed for classifying and selecting C-18 bonded columns was used to classify 12 brands of columns into three major groups based on a hydrophobicity index, free silanol index, and column efficiency [7].

602 E: ANALYTICAL COLUMN PROTECTION

HPLC analytical columns are expensive and subject to damage during use. The following items must be used to protect the column and prolong its useful life:

Filters

A major cause of column deterioration and damage is the buildup of particulate and chemical contamination at the head of the column. This can lead to increased back pressure and anomalous chromatographic results. Particle buildup is minimized by proper filtering of mobile phase solvents (see Section 603 B) and by choosing a sample solvent that will not cause precipitation (Section 601 E). In addition, in-line column filters help to eliminate particulate impurities.

Columns normally contain stainless steel inlet and outlet filters or frits to retain the column packing. The pore size of the frit must be smaller than the particle diameter of the packing, *e.g.*, a 2 µm frit for 5 µm packing. Frits are either incorporated into the ends of the column itself or made an integral part of the column end fittings.

Periodic cleaning of end fittings and frits in an ultrasonic bath in a solution such as 6 M nitric acid is recommended, especially when column back pressure increases. Before removing column end fittings, the manufacturer's literature should be read carefully for procedural instructions or notification of any loss of warranty if the column is taken apart. Some companies seal end fittings onto the column with epoxy and do not guarantee the column if the seal is broken.

Precolumns

The terms "precolumn" and "guard column" are often used interchangeably, but the two types of columns are different and serve separate primary functions. Precolumns are positioned in the HPLC system prior to the sample injector. Their purpose is to saturate the mobile phase with silica so that the silica or bonded silica analytical column packing is not dissolved during use. Precolumns packed with inexpensive, coarse silica are suitable for this mobile phase conditioning function.

Guard Columns

A guard column is inserted between the injector and analytical column to protect the latter from damage or loss of efficiency due to the presence of particulate matter or strongly adsorbed impurities from analytical samples. It can also serve as a saturator column to prevent dissolution of the stationary phase, in addition to, or instead of, a precolumn as described above. The use of a guard column is especially important when injecting relatively crude sample extracts or biological fluids.

Guard columns are short (2-6 cm) disposable columns containing the same packing as the analytical column. The guard column must be changed frequently, as dictated by the contamination level of the samples, to ensure that the lifetime of the analytical column, which should be several hundred hours running time, is not shortened.

The use of a commercial guard column having the same particle diameter packing as the analytical column, in combination with low dead volume fittings and short lengths of connection tubing, should cause essentially no loss in efficiency. Guard columns containing 5 or 10 μm particles can be purchased in the form of prepacked disposable cartridges (often 2 cm). They must be slurry packed if prepared in the laboratory. When a greater loss of efficiency is not critical, guard columns containing larger particle (20-40 μm) microporous or pellicular packings can be dry-packed in the laboratory using the tap and fill method, but the pellicular packings do not provide as much protection because of their lower surface area.

602 F: COLUMN MAINTENANCE AND TROUBLESHOOTING

Column Care

Chromatography companies usually supply a booklet describing recommended care and use of their columns. Topics covered typically include column description; directions for initial inspection, connection, equilibration, operation, regeneration, repair, and storage; mobile phase requirements; and information about solvent purification, protector columns, and replacement of frits. Any such literature should be read carefully and the suggestions followed as closely as possible. The following items describe routine handling and maintenance:

- Do not jar, drop, or vibrate columns.
- Pass solvent through the column in the direction specified by the manufacturer. If a flow direction is indicated, operation in the opposite direction may disturb the packing and reduce column efficiency.

- When starting up the HPLC system, gradually increase column flow rate and pressure to avoid pressure shock and formation of voids in the packing.

- Operate the column at a constant temperature using a column oven. If temperature significantly above ambient is used, raise the temperature slowly with solvent flowing. Elevated temperature improves column efficiency and reduces operating pressure by lowering solvent viscosity. However, thermal expansion of the column wall can lead to sinking or channeling of the column packing and loss of efficiency. Do not operate analytical columns at >60° C.

- Allow the column to equilibrate with the mobile phase, as indicated by a stable detector baseline, before injecting samples. Be sure that back pressure is acceptable for the required flow rate.

- Check fittings visually and by feel to be sure there are no mobile phase leaks. Leaks too small to see may be detected by the coolness of fittings to the touch.

- Particulate matter can become caught in the inlet frit, causing high back pressure. Replace the frit to return the column to the lowest possible operating pressure. Alternatively, clean the frit by washing with dilute nitric acid in an ultrasonic bath, dry, and replace. Never remove the bottom frit from the column. Use a precolumn filter to avoid the need to change the inlet frit and possibly disturb the column packing. Filter samples that may contain particulate matter to prevent contamination of the sample valve or column inlet frit. A commercial clarification kit that attaches to a syringe is a convenient way to filter samples.

- Do not overtighten column end fittings, or threads may be stripped, causing a leak.

- Flush the column with a solvent stronger than the mobile phase at the end of the day if dirty samples were injected.

- Handle columns gently to avoid shock and the formation of voids.

- Label columns with complete information on their source, identity, history and conditions of use, and regeneration and storage solvents. Keep a log notebook for each column from time of installation.

- Do not subject columns to operating conditions that may destroy their structure; be aware of the appropriate solvents and conditions that are compatible with the particular column.

Column Evaluation by Injection of Test Mixtures

Inject a test mixture to evaluate efficiency, selectivity, k, peak shape, *etc.*, according to the laboratory's instrument quality assurance requirements (see Section 602 C). Choose the test mixture according to the purpose of the test:

- To compare a chromatogram to the one supplied with a prepacked column by the manufacturer, use the same compounds and conditions specified by the manufacturer for comparable results.

- If an in-house test mixture for column assessment is needed, prepare it to contain the following types of components:

  1) an unretained (but not excluded) component for assessment of the volume between the particles and in the pores;

  2) a minimally retained component (k = about 0.2) to assess zone broadening caused mainly by the injector, column, and detector. Because the peak volume of this component will be small, it will be a critical test of the effect of these system components on performance;

  3) a moderately retained component (k = 1-3);

  4) a well retained component (k = 7-20). This component is optional because zone broadening will not be obvious because of the large peak volume; and

  5) a totally excluded component for determination of column void volume.

Column Storage

When no longer in use, columns should be equilibrated with an appropriate storage solvent, disconnected from the HPLC system, and the ends capped securely for storage.

Buffer solutions and halogen salts can easily damage column packings and stainless steel columns. Columns should be flushed with water after the use of buffers and should never be stored in such a solution. LSC columns are best stored in a dry organic solvent; RP columns in methanol, acetonitrile, or a water/acetonitrile or water/methanol mixture (use of water-free organic solvent reduces silica dissolution); IEC columns in a compatible solvent with the same ion as the form of the exchanger; and SEC columns in a solvent compatible with the swelling properties of the packing. Columns are not normally stored under pressure. The temperature and humidity of the storage area should be moderate and consistent.

Column Regeneration

Columns should not be operated with excessive pressure as this can create a void at the column head, resulting in a significant loss of efficiency. The cause of increased back pressure should be determined and steps taken to remedy the situation. Pressure buildup due to particulates can sometimes be relieved by back flushing the column or changing the frit at the head of the column. The simplest method of removing strongly retained material is washing with a solvent stronger than the mobile phase. If an appropriate guard column is in use, rejuvenation of the system should be possible in most cases by merely replacing the guard column.

A void at the head of the column can be observed after removing the inlet fitting. Voids can be filled with either glass beads or the same or similar packing as originally in the column.

Flushing with pure organic solvents such as methanol, tetrahydrofuran, chloroform, or acetonitrile is useful for regenerating bonded polar phase columns. When using any series of washes, the order of solvents should be weak to strong (nonpolar to polar for NP, and polar to nonpolar for RP), with consideration of mutual solubility at each stage. Basic impurities may be washed out with 1-5% aqueous phosphoric or acetic acid and acidic impurities with 1-5% aqueous pyridine. Biological materials and fats are removed from RP columns by washing with methylene chloride and making several 0.2-1 mL injections of dimethylsulfoxide during elution. Typically, 75 mL of each wash solvent is used at a flow rate of 0.5-3 mL/min. If washing does not remove adsorbed impurities from the top of the column bed, the upper, contaminated layers of packing must be removed with a spatula (exercising great care to avoid scratching the internal column wall), and the column repacked as described above for the case of a void.

In all cases, the last wash in the regeneration process should be with a solvent that is miscible with the mobile phase, and the column should be finally re-equilibrated with the mobile phase. After regeneration (or between washing stages to check progress), a test mixture should be chromatographed to evaluate plate number, k, and peak shape. Regeneration and return to equilibrium with the mobile phase can also be monitored by keeping the column connected to the detector and observing baseline drift. This should not be done if eluted impurities might contaminate the detector cell.

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603: MOBILE PHASE SELECTION, PREPARATION, AND DELIVERY

Mobile phases for different HPLC modes were described briefly under Modes of Operation (Section 601 B). Solvents used to prepare mobile phases were discussed under Solvents and Reagents (Section 601 D). This section presents additional considerations related to the preparation and delivery of mobile phases.

603 A: MOBILE PHASE SELECTION

Mobile phases in HPLC are usually mixtures of two or more individual solvents with or without additional additives or modifiers. The mobile phase is an active partner with the column in obtaining the required separation. The usual approach is to choose what appears to be the most appropriate column, and then to design a mobile phase that will optimize the retention and selectivity of the system.

The two most critical parameters for nonionic mobile phases are strength and selectivity. Mobile phase strength is related directly to polarity and ability to dissolve polar analytes in normal phase (NP) chromatography, while the opposite relationship exists for reverse phase (RP) chromatography. The general strategy for choosing a mobile phase is to find a solvent or solvent mixture with the correct strength to give k values in the optimum 2-10 range, and then to alter the phase to give the needed selectivity while maintaining the same strength. Solvents have been classified according to strength and selectivity to allow the selection process to be at least somewhat systematic.

Solvent strength for any solvent is dependent on the stationary phase adsorbent. An eluotropic series is a ranking of solvent strengths on a given adsorbent. Table 603-a lists solvent strengths for common solvents when used with different stationary phases.

Table 603-b shows solvents that are members of the different selectivity groups; the solvents most preferred for HPLC are underlined. Compounds in different groups interact in different ways with the analytes to be separated, *e.g.*, dispersion interactions, dipole forces, and hydrogen bonding. To optimize selectivity and improve separations, mobile phases are prepared from solvents in different selectivity groups.

Normal Phase Chromatography

The eight groups of solvents shown in Table 603-b emerged from Snyder's plot of solvents within a triangle of selectivity coordinates representing relative proton donor, proton acceptor, and dipole parameters [1, 2]. Maximum selectivity is obtained if one solvent is chosen from each group closest to the corners of the triangle. Based on other factors, such as viscosity and UV absorption properties, the three solvents chosen are usually diethyl ether or methyl tert-butyl ether (MTBE), chloroform, and methylene chloride. Hexane is used as the base solvent to adjust polarity (solvent strength). A binary mixture of hexane with one of these three solvents can be used to determine the appropriate solvent strength, and other binary, tertiary, and quaternary mixtures with the same strength can be tested in a systematic trial and error fashion for the required selectivity. The overall strength (P') of a mixture is the sum of the product of the individual P' values times the volume fraction for each component solvent. Other useful combinations of

Table 603-a: Properties of Common HPLC Solvents with Alumina Columns

Solvent	UV Cut-off, nm	Refractive Index	Boiling Point, ˚C	Viscosity cP, 25˚C	Solvent Polarity Parameter, P'	Solvent Strength Parameter, ε°	Group
isooctane	197	1.389	99	0.47	0.1	0.01	—
n-hexane	190	1.372	69	0.30	0.1	0.01	—
methyl t-butyl ether	210	1.369	56	0.27	2.5	0.35	—
benzene	278	1.501	81	0.65	2.7	0.32	VII
methylene chloride	233	1.421	40	0.41	3.1	0.42	V
n-propanol	240	1.385	97	1.9	4.0	0.82	II
tetrahydrofuran	212	1.405	66	0.46	4.0	0.82	II
ethyl acetate	256	1.370	77	0.43	4.4	0.58	VIa
chloroform	245	1.443	61	0.53	4.1	0.40	VIII
dioxane	215	1.420	101	1.2	4.8	0.56	VIa
acetone	330	1.356	56	0.3	5.1	0.56	VIa
ethanol	210	1.359	78	1.08	4.3	0.88	II
acetic acid		1.370	118	1.1	6.0	Large	IV
acetonitrile	190	1.341	82	0.34	5.8	0.65	VIb
methanol	205	1.326	65	0.54	5.1	0.95	II
water		1.333	100	0.89	10.2	Very Large	VIII

Table 603-b: Classification of Solvent Selectivity

Group		Solvents
I		aliphatic ethers, methyl t-butyl ether[1], tetramethylguanidine, hexamethylphosphoric acid amide, alkyl amines
II		aliphatic alcohols, methanol
III		pyridine derivatives, tetrahydrofuran, amides (except formamide), glycol ethers, sulfoxides
IV		glycols, benzyl alcohol, acetic acid, formamide
V		methylene chloride, ethylene chloride
VI	a)	tricresyl phosphate, aliphatic ketones and esters, polyesters, dioxane
	b)	sulfones, nitriles, acetonitrile, propylene carbonate
VII		aromatic hydrocarbons, toluene, halosubstituted aromatic hydrocarbons, nitro compounds, aromatic ethers
VIII		fluoroalcohols, m-cresol, water, chloroform

[1]Underlined solvents are those generally preferred.

[Both tables reprinted with permission of Elsevier Science Publishers, from Poole, C.F., and Schuette, S.A. (1984) *Contemporary Practice of Chromatography,* Table 4.16, page 260.]

solvents with different selectivity characteristics plus miscibility over the entire range of mixture composition include methylene chloride, MTBE, and acetonitrile in Freon FC-113 (1,1,2-trifluoro,1,2,2-trichloroethane), and methylene chloride, MTBE, and ethyl acetate in hexane.

Solvents for liquid-solid chromatography (LSC) HPLC should contain at least a small concentration (*e.g.*, 0.01-1%) of a polar modifier (water, alcohol, acetonitrile) to de-activate highly adsorptive sites that can cause tailing of chromatographic peaks. Water is the most important de-activator, and it can have a profound effect on chromatographic results. It is very difficult to control exactly the amount of water dissolved in nonpolar solvents such as pentane, hexane, heptane, and methylene chloride; this is one of the major causes of slow column equilibration with mobile phases and poor reproducibility in LSC. The following are useful precautions when using alumina and silica columns:

- Use 50% water-saturated solvents for silica gel and 25% water-saturated solvents for alumina, except for pentane, hexane, and heptane, which should contain 0.05% acetonitrile. (50% water-saturated means that the solvent has 50% of the water it would have if it were totally saturated. 50% water-saturated solvents are prepared by mixing equal volumes of dry solvent and saturated solvent or by passing dry solvents through a special moisture control column for specified time periods.)

- Change from one solvent to another in small steps along the eluotropic series. Do not attempt to follow a very polar solvent with a very nonpolar one directly, or *vice versa*.

- Chromatograph a test mixture repeatedly to test column equilibrium each time a column is used after being shut down or when changing mobile phases.

- If possible, use a separate column for each mobile phase to avoid problems associated with slow equilibrium. Avoid gradient elution with silica gel or alumina columns.

Reverse Phase Chromatography

This section will only consider mobile phases for bonded polar phase columns such as C-8 and C-18, which predominate in pesticide determinations. Classical liquid-liquid chromatography (LLC) will not be covered because it has been almost completely superseded by bonded phase chromatography.

In RP chromatography, the mobile phase is more polar than the stationary phase, and the most polar compounds elute first from the column. Mobile phases generally consist of mixtures of water, the weakest solvent for RP HPLC, or aqueous buffers with water-soluble organic solvents. Typically used solvents include, in order of decreasing polarity and increasing elution strength: methanol, acetonitrile, ethanol, isopropanol, 1-propanol, dioxane, and tetrahydrofuran (THF). Acid and basic buffers are used in the ion suppression mode to convert, respectively, weak acid and weak base analytes to their nonionic, hydrophobic forms, which are selectively retained on RP phases. Totally nonaqueous mobile phases are being increasingly used for the "nonaqueous RP" HPLC of polar substances.

The selectivity triangle approach described above for NP HPLC is applied equally well to RP HPLC. The solvents nearest to the corners of the triangle and having the requisite water solubility are acetonitrile (dipole interactions), methanol (proton acceptor), and THF (proton donor properties). It is most common to start with a water/methanol mixture to find the optimum solvent strength (P'), and then add one or both of the other solvents to maintain the strength but increase selectivity for the required separation. Each mobile phase modifier imparts a special selectivity by causing lower k values (faster elution) for compounds with a particular type of functional group.

A systematic four-solvent, seven-mixture mobile phase optimization strategy based on the approach described above is widely used for isocratic NP and RP HPLC [3]. In the case of acids or bases, an additional modifier to adjust pH may be necessary, *e.g.*, 1% acetic acid in water for the chromatography of phenols. One of the seven mixtures tested by this protocol should provide the required separation. If not, a different column, temperature, pH, or solvent modifier is required. An HPLC system with four solvent reservoirs and computerized solvent mixing capability makes this optimization routine a simple matter.

Ion Exchange Chromatography

Solvents for the separation of ionic compounds (*e.g.*, pesticides with acidic or basic groups) by ion exchange chromatography (IEC) include aqueous acids, bases, or buffers that allow the analytes to possess full or partial electronic charges and to be more or less attracted to the ionic groups of the stationary phase.

Ion exchange separations usually depend on several equilibria, the positions of which are a function of the following factors: the relative affinities of the analyte and the mobile phase counter ions, the ionic strengths of the analyte and counter ions, the acid or base strengths of the analyte and the stationary phase functional groups, and the mobile phase pH.

The general approach to designing a mobile phase is to first adjust the ionic strength to give analyte k values between 2 and 10, and then adjust the pH to control selectivity. Low ionic strength facilitates retention and high ionic strength elution. A change in pH affects both the character of the functional groups of the stationary phase and the ionization of the analyte. Retention is favored by a mobile phase/exchanger pH between the pKa values of the exchanger and the analyte (both must be charged). Elution is facilitated by mobile phase/exchanger pH above the pKa of a cation or below the pKa of an anion. Efficiency is improved by elevated temperatures and lower flow rates. An increase in counter ion concentration increases mobile phase strength. The proper choice of counter ion can improve selectivity. In general, exchangers prefer ions with higher charge, smaller hydrated diameter, and greater polarizability. Retention of analytes is favored if the exchanger is equilibrated with counter ions that are weakly held, and elution if the mobile phase/exchanger contain strongly held counter ions. Addition of an organic modifier generally increases solvent strength (especially if analytes are interacting with the mobile phase by a hydrophobic mechanism) and increases efficiency by lowering viscosity.

Ion Pair Chromatography

The mobile phase for ion pair chromatography is at a pH where the analyte is in its ionic form, and it also contains a pairing agent that conjugates with the analyte to form a hydrophobic, uncharged species that is selectively retained by a C-18 or C-8 bonded column. Typical pairing agents are a quaternary amine for weak acids and an alkyl sulfate or sulfonate for weak bases.

The choice of a mobile phase is aided by the following guidelines:

1) Methanol/water mixtures are preferred as the mobile phase to minimize counter ion solubility problems.

2) Short chain counter ions are recommended for analytes with little difference in molecular structure, and longer chain, hydrophobic counter ions for greater retention.

3) If silica-based bonded columns are used, the pH of the mobile phase must be maintained within the column's stability range. Use of porous polymer packings avoids this concern.

4) The mobile phase should be degassed before adding the counter ion to prevent possible foaming.

5) Typical concentrations for counter ions are 0.005-0.01 M, and 0.0005-0.001 M for buffer components.

6) Counter ions should not absorb UV light if a UV detector is in use.

7) To prevent salt precipitation, the pump should not be turned off until mobile phase is washed out of the system. Alternatively, a slow flow of mobile phase can be maintained overnight. It is best to have a dedicated column only for ion pair HPLC.

Size Exclusion Chromatography

Because of the nature of size exclusion chromatography, there are only two basic requirements for a mobile phase: it must readily dissolve the analyte and not damage the stationary phase. Solvents that are not compatible with polystyrene-divinylbenzene (DVB) phases include water, alcohols, acetone, methyl ethyl ketone, and dimethylsulfoxide. If the analyte does not dissolve well in the mobile phase, tailing and/or delayed elution due to interaction of the analyte with the stationary phase can occur. Adsorption effects are reduced by using a mobile phase chemically related to the stationary phase, *e.g.*, toluene for polystyrene-DVB columns.

Gradient Elution in HPLC

The preceding discussions relate principally to mobile phases for isocratic HPLC. Isocratic elution is widely used because of its convenience and reproducibility. It is not adequate, however, for separation of analytes containing components with greatly different retention times. Gradient elution improves resolution of early

eluting peaks while causing later eluting peaks to elute sooner and in a narrower band. Alternative approaches to the general elution problem include column coupling and flow and temperature gradient, but these will not be discussed here.

Solvent gradients are usually composed of a binary mixture of a weak solvent to which continuously increasing amounts of stronger solvent are added in a linear, convex, or concave relationship over time. Isocratic elution periods are often included at the beginning and/or end of the gradient sequence. Important considerations include the solvents chosen, the initial and final composition, and the gradient shape and steepness. Stepwise gradients are also possible. Methods are available for predicting and optimizing gradients for the different HPLC modes [4, 5], but the most suitable gradient for a particular separation is usually determined empirically.

Gradient elution is used widely in NP and RP bonded phase and IEC. It is not recommended for LSC and cannot be used with LLC or with refractive index (RI) or conductivity detectors. Regeneration at the end of the gradient must return the column to equilibrium with the initial solvent. Solubility considerations may require purging the system with an intermediate strength solvent, or it may be possible to simply pass 5-10 column volumes of the first solvent through the column.

pH and ionic strength gradients are common in IEC to control mobile phase strength and selectivity. Gradients for ion pair chromatography must be checked to be sure that the counter ion and buffer components are soluble in all solvent compositions used. Ion pair gradients may involve a solvent gradient with constant pH and counter ion concentration, or these may be changed along with, or instead of, the methanol/water (or other solvent) composition.

603 B: MOBILE PHASE PREPARATION

Mobile phases must be prepared from high purity solvents, including water that must be highly purified (see Section 601 D). Mobile phases must be filtered through ≤1 μm pore size filters and be degassed before use.

Filtering Solvents

Particulate matter in solvents can damage pumps, block flow in tubing, and degrade column performance. Filtering of all HPLC solvents should be a routine laboratory procedure. Filtering is especially important for removal of particles when solvents are stored over molecular sieves. Commercial units that attach to any vacuum line are available for simultaneous filtration and degassing of solvents, or similar apparatus can be assembled in the laboratory. Commercial nylon membrane filters with 0.22-1.2 μm pore size are compatible with all solvents commonly used in HPLC. Most commercial HPLC grade solvents are prefiltered through a 0.2 μm filter and should not require additional filtration.

Degassing Solvents

Degassing of solvents is necessary to avoid problems with columns, pumps, and detectors caused by gas bubbles in the system. The filtering step, if carried out with an aspirator or vacuum pump, can also provide degassing. Degassing of volatile

solvent mixtures with a vacuum can change the composition of the solvent; vacuum degassing should never be used for such mixtures.

Other degassing methods include boiling, use of an in-line degassing unit with a gas-permeable membrane, or by agitation in an ultrasonic bath. However, the most effective and convenient degassing method is helium sparging. A commercial unit can be used, or a setup can be made in the laboratory from Teflon tubing and an inlet line frit attached to a helium supply. The frit is immersed in the solvent reservoir, and helium is bubbled for a few minutes with about 3-4 psi pressure at the tank. The helium flow is reduced to a trickle during operation of the system. If solvent mixtures are made manually, individual solvents are degassed prior to preparation, and the mixture is kept under helium during use.

Preparation of Multisolvent Mobile Phases

Mobile phase mixtures can be prepared either by manual blending or by in-line mixing using the HPLC solvent blending and delivery apparatus. Laboratory glassware used for preparing mobile phases should be exceptionally clean so it does not introduce particles or impurities.

Two different approaches to manual preparation of solvents are possible. Either is valid, as long as the preparation method is clearly recorded so others can reproduce the results. In the first method, volumes of solvents A and B measured in graduated cylinders or pipets are mixed together in the mobile phase reservoir. In the second method, a measured volume of solvent A is placed in a volumetric flask, and the solution is diluted to the line with solvent B and transferred to the mobile phase reservoir. Solutions prepared by these methods will be slightly different, especially for water/alcohol mixtures, because of the nonexact additivity of volumes upon mixing. It is good practice to prepare the mobile phase fresh each day, especially if a volatile solvent is involved. If the mobile phase will be used for longer periods, it should be definitely proven, *e.g.*, by measuring RI or chromatographing a test mixture, that there is no change in composition with time.

If an error in composition is suspected for a mobile phase prepared in-line, a new batch of the mobile phase should be carefully prepared manually and the separation repeated.

Solvent Reservoirs

The solvent container should be made of a material from which the solvent cannot leach significant impurities, and should have a cover with a small opening through which the Teflon or stainless steel delivery tubing fits snugly but without constriction. The reservoir is placed away from sunlight or drafts to avoid temperature gradients, and above the solvent delivery system to provide siphon feed to the pump. The reservoir should be labeled with the composition and date of preparation of the mobile phase, and a solvent reservoir filter (sinker frit) should be attached to the end of the delivery tube.

603 C: MOBILE PHASE DELIVERY SYSTEMS

Pumps

The function of the pump in HPLC is to deliver the mobile phase through the column at high pressure with a controlled flow rate. Two major categories of pumps are constant flow or volume and constant pressure. Constant pressure pumps apply a constant pressure to the mobile phase; flow through the column is determined by the flow resistance of the column and any other restrictions in the system. Constant flow pumps generate a certain flow rate of mobile phase; the pressure depends on the flow resistance.

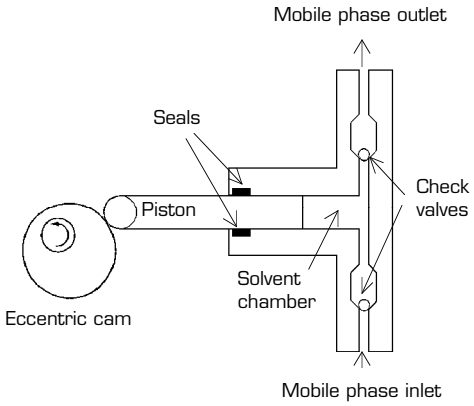
Constant flow pumps are recommended for HPLC because flow resistance may change with time due to swelling or settling of the column, small temperature variations, or buildup of particulate matter. These effects will cause flow rate changes with a constant pressure pump and result in nonreproducible retention data and erratic baselines.

A suitable pump should have the following characteristics:

1) The interior of the pump should be made of inert materials that resist corrosion by any solvents being used.

2) Pressures up to 6000 psi and a wide range of flow rates (0.1-≥10 mL/min) should be available, and the flow rate should be easy to change. High flow rate capability is especially important for preparative work.

Figure 603-a
Reciprocating Pump



[Reprinted with permission of John Wiley and Sons, Inc., from Lindsay, S. (1987) *High Performance Liquid Chromatography,* Figure 2.2d, page 21.]

3) The flow should be constant, reproducible within at least 1%, and pulseless or have a damping system to minimize detector noise generated by the pulses. It should be easy to set, measure, and change the flow rate.

4) It should be easy to change from one mobile phase to another.

5) The internal volume of the pump and all of the plumbing between the pump and the injector should be as small as possible.

6) The pump should be useful for isocratic or gradient operation.

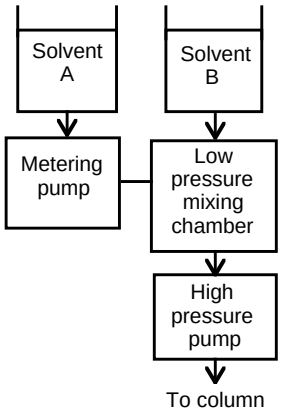
7) The pump should be adaptable to the use of small volumes of mobile phase, a high volume mobile phase reservoir, or a heated reservoir.

8) The pump should be easy to maintain and repair. Even with the best of care, seals, rings, and gaskets will require occasional replacement, and it will help if these are easy to access.

Neither constant pressure pumps nor screw-driven syringe constant flow pumps are described here because the reciprocating pump (Figure 603-a) is used in most HPLC instruments. In this pump, a small piston is driven in and out of a solvent chamber by a motor-driven eccentric cam and gear arrangement. On the forward stroke, the inlet check valve closes, the outlet valve opens, and the mobile phase is pumped to the column. On the return stroke, the outlet valve closes and the chamber is refilled.

Because the displaced volume is small, the pump must cycle frequently. Abrasion is minimized by using hard, smooth piston material such as borosilicate glass, sapphire, or chrome-plated steel. Solvent capacity of a reciprocating pump is unlimited if the external reservoir is filled as required. The internal chamber volume can be very small (*e.g.*, 10-100 µL), allowing rapid change of mobile phases. The flow rate, 0.01-50 mL/min, is changed by varying the length of the piston stroke or the speed of the motor. Piston seals and check valves must remain leak free. This requires regular maintenance and periodic replacement of parts. Access to valves and seals for maintenance is usually quite easy.

Figure 603-b
Gradient System I



[Reprinted with permission of John Wiley and Sons, Inc., adapted from Lindsay, S. (1987) *High Performance Liquid Chromatography,* Figure 2.2f, page 24.]

Figure 603-a shows a single-head reciprocating pump, in which solvent is delivered to the column for only one-half of the pumping cycle. Flow pulsations arise from the piston action, which may produce noise with some detectors during high sensitivity analyses. Flow noise is reduced if the pump is designed with a rapid stroke rate so the detector cannot respond rapidly enough to sense the flow changes. Other ways to obtain constant flow rate are the incorporation of dampeners or a feedback control system.

Twin- or dual-piston reciprocating pumps have two heads operated 180 degrees out of phase by the action of a single cam so that one pumps while the others refills, producing a constant, pulseless flow and reduced noise.

The diaphragm or membrane reciprocating pump is a variation of the piston pumps described above. A piston is driven back and forth by an eccentric disc. The movement is conveyed hydraulically to a flexible steel membrane, which flexes and displaces the solvent out to the column, and then pulls in mobile phase from the reservoir when the diaphragm returns. Check valves at the inlet and outlet ensure flow in the proper direction. The piston does not contact the solvent directly, so seals are not needed. Pulsations caused by discontinuous pumping and suction cycles are stabilized by incorporation of a damping system or two pistons synchronized to minimize pulse lag. Back pressure changes in the column and the elasticity of the diaphragm can cause flow rate

Figure 603-c
Gradient System II

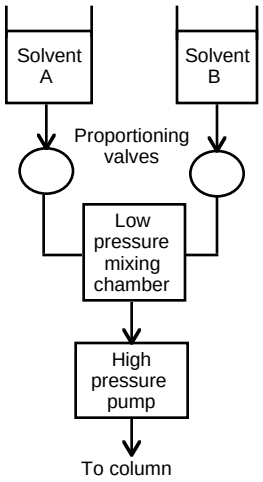


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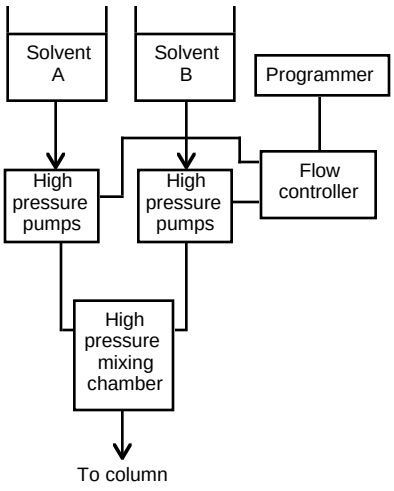
Figure 603-d
Gradient System III



[Reprinted with permission of John Wiley and Sons, Inc., adapted from Lindsay, S. (1987) *High Performance Liquid Chromatography,* Figure 2.2f, page 24.]

deviations. A feedback flow controller should be incorporated to ensure constant flow rate.

Gradient Programming Systems

Figures 603-b, -c, and -d show the arrangements for gradient-forming systems involving two solvents.

In Figure 603-b, the gradient is formed at low pressure by metering controlled amounts of solvents A and B from low pressure pumps into a mixing chamber (volume <1 mL) fitted with a magnetic stirrer, from which it is drawn into a high pressure pump for delivery to the column.

The arrangement in Figure 603-c is similar, but composition of solvent in the low pressure mixing chamber is regulated using microprocessor-controlled, solenoid-operated time-proportioning valves. Low pressure systems with no mixing chamber are also available. Such systems, which ensure that the gradient is not retarded, involve highly precise valve control and a great deal of mechanical and electronic equipment to mix extreme volume ratios.

In Figure 603-d, the separate solvents are pumped with two high pressure pumps into a high pressure mixing chamber. The type of gradient formed is controlled by programming the delivery rate of each pump. Electronic control must ensure that the total volume is always constant and that compensation is made for changes in viscosity. The post-pump chamber must provide rapid and complete mixing and have a small volume and no "dead" areas. This method is more expensive than low pressure mixing because a separate pump is required for each solvent and thus is decreasingly favored.

Technically sophisticated systems, usually involving low pressure, controlled mixing and delivery to the column with a high pressure pump, are now available for gradients involving three and four different solvents, which are becoming more widely used for separation of complex mixtures.

Errors in gradient formation can be caused by restricted lines and loose tubing connections, which can be corrected by the operator. Problems with valve controllers or software usually must be handled by a manufacturer's service technician.

603 D: MAINTENANCE AND TROUBLESHOOTING

Problems with Pumps

The following considerations are important in the maintenance and troubleshooting of all types of HPLC pumps:

- Have pumps set up by a manufacturer's service technician, who should explain proper operation, maintenance, troubleshooting procedures, and precautions to all users and in-house service personnel.

- Obtain all available operation manuals and require that they are read carefully by users. Many manufacturers provide extensive maintenance and troubleshooting information with their pumps.

- Stock an adequate supply of parts that require routine replacement or may be damaged or broken during routine maintenance, such as seals, plungers, fittings, cams, O-rings, heads, check valves, springs, clamps, *etc.*

- Maintain a log notebook for each pump. List maintenance and repair dates and procedures, and use and storage history.

- Do not store corrosive solvents or buffers in the pump overnight.

- Periodically lubricate pump motors with the proper grade of oil.

- Do not attempt to replace one solvent with another unless both are completely miscible.

- Degas solvents to avoid bubbles in the pump head(s) and filter all solvents. These are the two primary precautions for preventing pump problems.

- If possible, avoid highly volatile solvents (*e.g.*, pentane), which with the pumping action can cause volatilization and bubbles.

- Avoid pump overheating by working in a well-ventilated area.

- Confirm that the pressure limit switch, if available, is set properly.

- Inspect pump heads and fittings for leaks on a daily basis. Leaks can be caused by dirty pistons and worn piston seals. Gentle tightening of fittings usually eliminates leaks. Overtightening of fittings can cause leaks and permanently damage the part. This can be an expensive matter if the affected fitting threads into a pump check valve or head.

- Dirty, sticking, or malfunctioning check valves can cause irregular or inaccurate flow and drifting baselines, or stop flow altogether. Check valves can be replaced or cleaned. In either case, follow the manufacturer's instructions.

- Determine the useful lifetime of pump seals under the operating conditions in each laboratory. Replace the seals on a regular basis before the useful lifetime is over, or at least on a yearly basis. At the same time, inspect the piston for scratches.

- Verify the flow rate of pumps on a periodic basis, to an accuracy of 10%, by delivery into a graduated cylinder while timing with a stop watch. When an accuracy of ±1% is desired, use a stop watch and buret. Measure the time interval as the meniscus passes two marks on the buret a known volume apart. At least a 2 min period is desirable for this degree of accuracy. If the delivered flow rate is not within specifications, check for leaks and/or make adjustments or repairs as outlined in the operating manual.

- If the pump starts up but does not move the solvent, it is probably in need of priming. Pump-priming procedures vary from one instrument to another; check the correct procedure in the manufacturer's instrument manual.

Problems Caused by Air. Most problems with HPLC pumps are caused by air bubbles. These arise when air is drawn into the pump when the solvent reservoir runs dry or the solvent inlet line is lifted out of the reservoir; from leaks at the fittings that connect the inlet tubing to the pump; from bubbles generated when the mobile phase components are mixed; or from cavitation of the mobile phase in the inlet line or pump head. The symptom in each case is stoppage of flow or fluctuating pressure.

A sinker frit on the end of the inlet tubing or tight connection through a cap at the mouth of the reservoir will keep the tubing submerged in the bottom of the reservoir and prevent air in the reservoir from reaching the pump.

If an air leak on the inlet side of the pump is suspected, carefully tighten each of the fittings, including the check valve. Do not overtighten plastic fittings to the point of distortion. If the leak persists, disassemble the fittings and examine them for damage.

Recut suspect tube ends and re-assemble or replace suspect low pressure or compression fittings until the problem is solved. If buffers have been used, flush the fitting with nonbuffered solvent before re-assembly.

When RP solvents (*e.g.*, water and methanol) are mixed, the mixture has a lower capacity for dissolved gases than the pure component solvents. This is why bubbles often are seen evolving from freshly mixed mobile phase. With manual mixing, excess gas bubbles from the solution, but the mixture remains saturated with air. Therefore, when the pump begins to fill, pressure is reduced and gas bubbles form in the pump head. With low pressure mixing, solvents are combined just prior to the pump. Mobile phase entering the pump is supersaturated with air, which bubbles out in the pump. With high pressure mixing, solvents are mixed after the pump, so bubble problems should not occur in the pump. Proper degassing of solvents (Section 603 B) is essential.

Cavitation occurs when the pump draws solvent through a line with restricted flow and creates a partial vacuum in the line. This vacuum can cause dissolved air to expel, forming bubbles in the inlet line or pump head. Blockage of the inlet filter in the mobile phase reservoir is a common cause of cavitation that can be corrected by replacing the restricted filter. Another cause is a tightly fitting reservoir cap that is not properly vented. Drilling a very small (<1 mm) hole in the cap or loosening it can remedy this problem.

Problems Caused by Dirt. The most damaging pumping system problems are caused by dirt, a term that encompasses particulate matter introduced by the mobile phase, buffer evaporation, or wearing of seals. The main problems caused by the presence of dirt are malfunctioning check valves and premature pump seal wear.

Particulate matter can prevent proper sealing of check valves, resulting in pressure fluctuations and poor pump delivery. With high pressure mixing, a dirty check valve can cause proportioning problems. If simple flushing does not cure a

suspected check valve problem, the valve should be replaced. If the problem is eliminated, the dirty check valve should be cleaned, if possible, and later re-used. If it cannot be cleaned, it can be returned to the manufacturer for rebuilding. A dirty check valve is cleaned by rinsing with HPLC grade solvent or sonicating in 10% nitric acid followed by rinsing with HPLC grade water.

If a pump containing buffered mobile phase is shut off and allowed to sit overnight or longer without washing out the buffer, mobile phase behind the pump seal will evaporate and abrasive solid crystals will form. When the pump is restarted, these crystals will abrade the seal and cause accelerated wear. Abraded seal particles can also cause check valve problems and block the top column frit. Flushing with 10-20 column volumes of nonbuffered solvent at the end of each day is recommended. Some pumps are designed to allow direct flushing of buffer from behind the seal. The pump operation manual should be consulted.

Proportioning Problems. To prevent proportioning problems, solvents must flow freely with no restrictions. Change inlet (sinker) frits in solvent reservoirs before they become blocked. Make buffers fresh daily to extend frit lifetime by retarding microbial contamination. Make sure that low pressure fittings are sealed properly so that air cannot leak in and solvent out. Thoroughly degas solvents to prevent bubble problems. Elevate solvent reservoirs above the proportioning manifold to apply slight head pressure and improve the reliability of solvent delivery.

Run reference tests routinely to recognize mechanical problems with the proportioning valves and problems with the controlling software. For example, set the programmer so the solvent does not flow through the column. Use any convenient (miscible) solvents of HPLC quality in the pumps. Attach a recorder to the program monitor terminal jacks so the pen traces the gradient. Operate the programmer as outlined in the instrument operating manual. Compare the trace on the recorder to determine whether the correct programs are actually being produced. Test each program that is regularly used for actual analyses.

Another test may involve a series of 10% isocratic steps from 0 to 100% of solvent B (containing a UV absorber to allow detection), changed every 5 min. The resulting trace of absorption *vs* time should yield rising steps that have the same height and are fairly square. A third test is a 20 min blank gradient run at 4 mL/min using the same spiked B solvent. This trace should be essentially straight (especially between 5 and 95% B), with angular intersections at the 0% baseline and 100% plateau.

Bubble problems can be caused by air leaks, which can occur when a proportioning valve diaphragm becomes perforated or other damage to the solvent proportioning manifold occurs. The proportioning manifold can be tested by connecting the manifold inlet and outlet tubing with a union. If the bubble problem disappears, the manifold is bad and must be replaced.

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604: INJECTION SYSTEMS

The purpose of the injection system is to apply the sample extract onto the column in a narrow band. Three techniques are available: direct syringe injection, stop-flow syringe injection, and use of an injection valve.

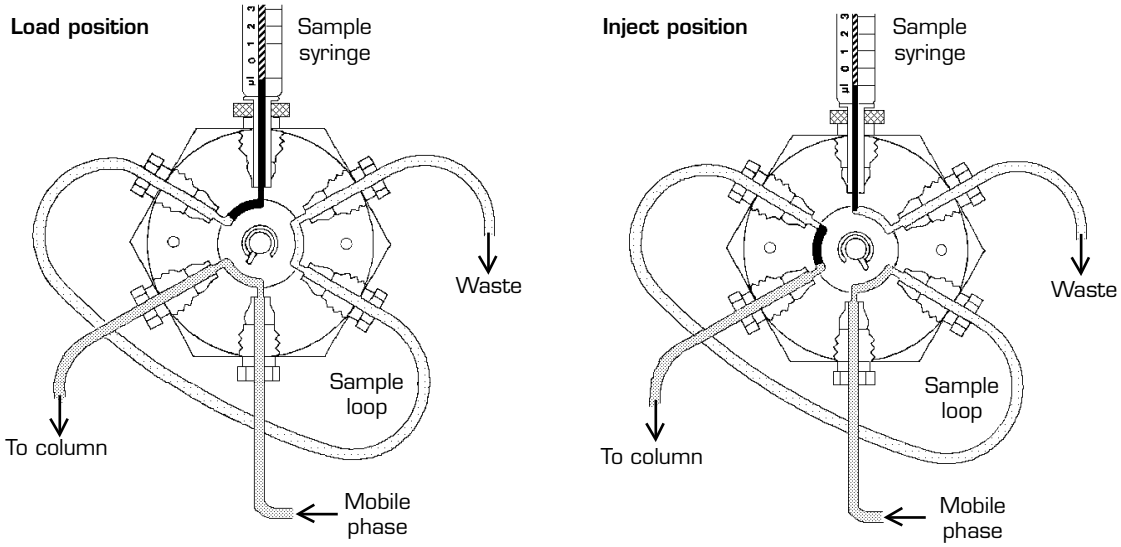
In direct syringe injection, the extract is injected into the flowing mobile phase through a septum using a high pressure syringe in a manner similar to GLC. A septumless injector is also available for direct syringe injection without interrupting mobile phase flow. In stop-flow injection, injection is made at ambient pressure after depressurizing the injection port by use of a sliding seal and shut-off valve, or by turning off the solvent delivery pump. The direct syringe injection and stop-flow injection techniques are now obsolete and rarely used.

604 A: INJECTION VALVES

The injection valve is at present the most widely used injection device for reproducibly introducing sample extracts into pressurized columns without flow interruption. Valves can be made with external or internal loops. Virtually all commercial external loop valves are variations of the six-port design shown in Figure 604-a. A fixed volume loop is connected across two of the ports. The extract is introduced through the injection port, and excess extract flows out through the waste port. The other two ports provide a path for the mobile phase as it is pumped into the column. External loops are available in sizes ranging from 5 µL to 2 mL. The 10 and 20 µL sizes are probably most widely used. Loops are usually made from standard 1/16" stainless steel, but other materials are used in biocompatible injectors.

When the valve is rotated to the load position on the left in Figure 604-a, mobile phase flows directly from the pump to the column and the loop connects the injection and waste ports. The loop is at ambient pressure and is filled with extract from a regular

Figure 604-a
External Loop Injector: Six-Port Injection Valve



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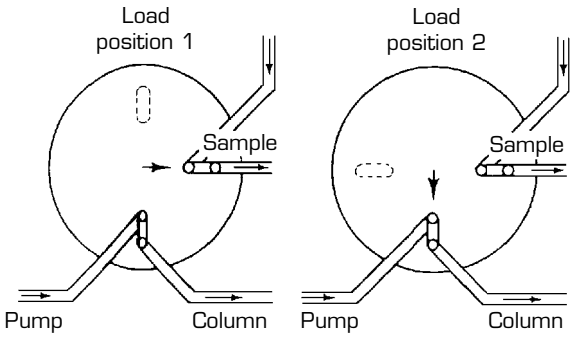
microliter syringe. When the valve is rotated to the inject position, shown on the right, mobile phase flows through the loop, sweeping the extract rapidly onto the column.

One common technique is to fill the loop completely with extract (the "filled loop" technique). In this case, the loop volume fixes the injection volume, and the loop must be changed to vary the extract volume. To achieve good reproducibility, an extract volume equal to twice the loop volume should be used to flush and fill the loop. If a Teflon waste tube is used, air and excess solvent will be seen going to waste. All air should be expelled when filling the loop. For applications in which limited extract is available, some valves have a special loop-filler port that permits loading of the loop with minimal waste.

Other methods for varying the volume of extract injected include filling a fixed volume loop completely with a combination of extract and solvent; partially filling the loop (the "partial loop" technique), following the manufacturer's procedural guidelines; or using special variable volume valves. For best results with the partial loop technique, the volume of extract injected should be <50% of the nominal loop volume, *e.g.*, 10 µL extract in a 25 µL loop. If a tiny air bubble is injected just before the extract to isolate it from previous solution in the loop, >50% of the nominal loop volume can be injected. The partial loop technique allows flexibility of injection volume, but precision is dependent on the analyst's skill in reproducibly injecting specific extract aliquots from a syringe.

Recently, the six-port valve has been adapted to place cleanup and analyte concentration steps in-line with the determinative step. A short cleanup column chosen for its ability to retain the analyte replaces the loop shown in Figure 604-a [1]. While the valve is in the load position, sample extract is injected onto the cleanup column; subsequent injection of solvent removes co-extractives to waste without removing the analyte. When the valve is rotated to the inject position, mobile phase flows through the cleanup column and elutes the analyte onto and through the analytical column to the detector. Variations of this technique can involve cleanup and analytical columns of the same or different HPLC modes [2]. When the injection valve is used this way, no loop is available, so the extract volume injected must be measured accurately in a syringe.

Figure 604-b
Internal Loop Injector



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Internal loop injectors are valves with four ports, with the loop in the form of an engraved groove or slot in the body of the valve. These are used for injecting extracts in the 0.05-5 µL range. Figure 604-b illustrates the internal loop injector. These injection valves are also available for microbore HPLC. This type of valve, which is designed for narrow bore (1-2 mm) columns, has a 0.2-1 µL interchangeable chamber and built-in needle port with 0.3 µL holdup volume to reduce extract loss.

Ten-port multifunctional valves are now available for HPLC. These can perform as a standard six-port injection valve and in addition allow extract injection followed by back flush, injection into two columns simultaneously, injection into either of two columns (random access), extract injection followed by precolumn back flush, trace enrichment, alternate extract injection from two streams, two-column selection with flow maintained in both, heart-cutting operations, and fast, sequential injections of a single extract.

Injection valves used with the filled loop technique are easy to use, provide the best precision for quantitative HPLC, typically <1% relative standard deviation, are easily adapted for automatic injection, and allow high pressure operation (up to 7000 psi). The partial loop injection technique is not as reproducible as the filled loop technique for manual injections. It is best used only for preliminary experiments when determining the optimum injection volume, or with automatic injectors that provide high precision automatic syringe delivery. To maximize column efficiency, the smallest convenient extract volume should be injected using an extract solvent that is weaker (*i.e.*, more polar for reverse phase HPLC) than the mobile phase.

The method used to insert the valve in the HPLC system is critical for minimizing loss of efficiency. A 5 cm length of 0.15 or 0.50 mm id tubing between the valve and column with connection via a low or zero dead volume fitting is recommended. When using the partial fill technique, the valve should be connected as shown in Figure 604-a. This plumbing arrangement delivers the extract to the column before the solution previously in the loop, preventing band spreading due to dilution of the extract.

604 B: AUTOMATIC INJECTORS

Automatic injectors are valve injectors whose rotation is controlled by pneumatic or electric actuators. Most use a mechanized syringe to dispense the extract into the loop. Systems with both fixed and adjustable volumes are available for unattended operation. A typical adjustable volume model allows injection of 1-100 µL, accurate measurement by a motor-driven microsyringe, a positive displacement mechanism to minimize extract waste, microprocessor-controlled injection sequencing, and a multivial sample turntable. The microprocessor controls movement of the turntable, sampling needle, microsyringe, and injection valve. All injection parameters, including sampling interval, sample number, and injections per sample, are entered from a keyboard. Injection precisions are typically quoted as 0.5-1%.

604 C: OPERATION, MAINTENANCE, TROUBLESHOOTING, AND REPAIR OF INJECTION VALVES

The following considerations are important for trouble-free operation, maintenance, and repair of injection valves:

- Read carefully the literature packed with the valve for information on installation, use, maintenance, and repair.

- The major cause of injector problems is particulate matter entering the valve. Particles can lodge in moving parts, scratch the rotor surface, and cause leakage. They can also block the connecting tubing or sample loop. To avoid formation of particles, dissolve the sample extract in the

mobile phase itself. If a solvent is used in which the analyte is more soluble, components of the extract may precipitate when contact is made with the mobile phase. To eliminate particulate matter, install an in-line 5 µm filter between the pump and the valve, and filter any extracts that have visible particulate material or are cloudy or opalescent.

- If blockage occurs, locate it and back flush the blocked passage; disassemble the valve and sonicate the blocked part in soapy water, rinse in clear water, and blow the passage clear with compressed air; or replace the blocked tubing. Return blocked valves that cannot be cleared in the laboratory for reconditioning by the manufacturer.

- To minimize rotor seal wear, prevent abrasive particles from entering the valve as described above, and do not allow buffered or corrosive mobile phases to remain in the valve for extended periods of time without flushing.

- Do not operate above the pressure limit of the valve (usually 1500-7000 psi) or leakage may occur. Operate at the lowest possible pressure to reduce rotor seal wear.

- Use valves that are constructed of materials compatible with extract and mobile phase components. In addition to the usual valves constructed from stainless steel with a polymeric rotor, specialty valves made from more inert materials are commercially available.

- Maintain a good supply of spare parts, *e.g.*, dead volume fittings, ferrules, and rotors. It is best to stock backup valves in case repair cannot be done in the laboratory and return of a malfunctioning valve to the manufacturer is necessary.

- Engrave an identification number on each valve, and keep a log notebook to monitor the history of use and repairs.

- Do not overtighten valve fittings. Overtightening can cause leaks or damage to the valve body.

- To minimize dead volume and peak broadening, use connecting tubing between the injection valve and the column, and the column and the detector, that is as short as possible and has a small id. See that valve tubing is straight and has a perpendicularly flat end that is sealed tightly inside the port in the valve body. Do not allow metal pieces formed in the tube-cutting process to enter the valve body.

- Identify crossport leaks by observing mobile phase emerging from a Teflon exit line when the valve is in the load or run position.

References

[1] Niemann, R.A. (1993) *J. AOAC Int,* **76**, 1362-1368

[2] Wojtowicz, E.J. (Feb. 1992) "HPLC Fluorometric Analysis of Benomyl and Thiabendazole in Various Agricultural Commodities," LIB 3650, FDA, Rockville, MD

605: DETECTORS

The function of an HPLC detector is to continuously and instantaneously monitor the mobile phase emerging from the column. The output of the detector is an electrical signal that results from measuring some property of the mobile phase and/or the analytes. Application of HPLC to trace residue determination is dependent on the availability of sufficiently specific and sensitive detectors. The common HPLC refractive index (RI) detectors are nonselective and require microgram quantities of analyte, so were never adequate.

The UV/VIS absorbance and fluorescence detectors are now commonly applied to pesticide residue determination, because methods research has resulted in schemes that utilize their capabilities while overcoming their limitations. Other detectors available for HPLC include photoconductivity, electrochemical, and mass spectrometric; published applications to residue determination increase each year. Use of combined detection systems is also increasing, *e.g.*, the combination of UV absorbance and electrochemical detection.

The following are important characteristics of HPLC detectors:

Sensitivity. Detector sensitivity is a gauge of the detector's response (signal) to the presence of an analyte. In HPLC applications, the usual quantitative measurement is called minimum detectability, defined as concentration of analyte that causes the detector to produce a response twice that of instrument noise.

Minimum detectability refers to detector response to an analyte in pure solution. "Limit of detection" or "limit of determination," however, takes into account the amount of sample extract that can be introduced to the detector and is partially dependent on the degree of cleanup provided by the analytical method (see Section 105). Injection volume is an important parameter in determining the limit of detection of an HPLC analysis, because aqueous extracts prepared for HPLC determination are not easily concentrated. However, HPLC can tolerate large volumes of solvent without loss of column performance, thus improving detection. Injection volumes up to 100 µL are not unusual for analytical HPLC.

Linearity. The linear range is the concentration range over which response is directly related to analyte concentration (*i.e.*, a plot of response *vs* concentration has a constant slope). Linearity is commonly expressed in units such as 1:10,000 or 10^4, which might indicate a range of 10^{-10} (the minimum detectable quantity) to 10^{-6} g/mL for the UV detector.

Selectivity of Response. Universal detectors respond to all components in the extract, while specific (selective) detectors respond to only certain compounds depending on their structure. Selective detectors are usually more sensitive and less affected by variations in the mobile phase.

Effect of Changing Conditions. Ideally, the detector should not be affected by changes in temperature or mobile phase composition.

Time Response. The detector-recorder combination should react rapidly so that quickly eluting, narrow peaks can be measured accurately. The time constant of the detector-recorder, which is defined as the time required to reach 63 or 98% of full

scale deflection, should be at least in the 0.1-0.3 sec range for high speed HPLC and narrow peaks. Modern instruments are capable of response in the millisecond range.

Cell Volume. Detector cell volume should be minimized to limit peak broadening and maximize efficiency. A cell volume of 8 μL is typical, with smaller values for micro-HPLC. The volume of associated tubing should also be minimized. However, as the volume of the detector cell decreases, sensitivity of detection is poorer. Detector design should eliminate dead corners in the cell from which the analyte is not quickly washed by the mobile phase.

Noise and Drift. Baseline noise, as indicated by variations in the recorder signal with no sample in the detector cell, is caused mostly by the electronics of the detector, recorder, or amplifier. Additional sources include the mobile phase (bubbles, changes in flow rate or pressure, leaks, impurities) and temperature fluctuations. To reduce the chance for bubble formation from depressurized mobile phase in the detector cell, modern detectors include a back pressure restrictor.

Baseline drift may occur when the HPLC system is first turned on. If drift persists after warmup, it is most likely due to changes in mobile phase composition, leaks, temperature variations, column bleed, or a gas bubble in the detector cell. Baseline drift can also be caused by slow elution of highly retained components left on the column from previous samples.

Nondestructiveness. An analyte can be collected for further characterization if its chemical form is not changed by the detector. UV and fluorescence detectors are examples of nondestructive detectors, while the electrochemical and photoconductivity detectors are destructive.

Ruggedness, reliability, and ease of use, maintenance, and repair are also important qualities to seek in HPLC detectors.

605 A: UV/VIS ABSORBANCE DETECTORS

The most popular HPLC detectors are the fixed and variable wavelength UV/VIS types. Fixed wavelength UV detectors most often operate at 254 nm, which is useful for molecules with aromatic rings, carbonyl, conjugated double bonds, and other suitable chromophores. Variable wavelength detectors usually operate in the 190-380 nm range with a deuterium source or 190-900 nm with a supplemental tungsten source. Other supplementary lamps that have been used are the 229 nm cadmium lamp and 214 nm zinc lamp.

Variable wavelength detectors provide several advantages. A wider applicability and significant increase in sensitivity often result from operating at wavelengths in the 190-230 nm range. This is due to the large molar absorptivities of many pesticides in this region. Typically, aromatic systems have a 10-50 times greater absorbance at 214 and 229 nm than at 254 nm. Different peaks in the chromatogram can be detected at different optimum wavelengths.

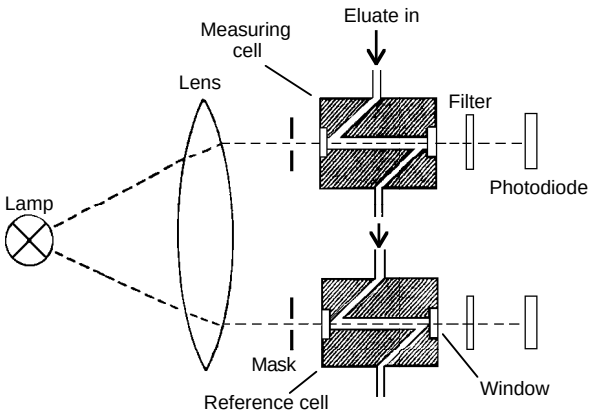
Very pure solvents, including water, must be used to avoid noise and unstable baselines at the lower wavelengths. Increased selectivity for pesticides with aromatic and certain other functional groups can be obtained by measuring at longer wavelengths such as 280 or 295 nm. This gives the analyst the ability to "edit out"

unwanted peaks. This approach, however, may lead to some loss in sensitivity if measurement of the analyte cannot be made at its wavelength of maximum absorbance. An additional advantage of a spectrophotometric-type detector is that unknown components can be identified by stopping the mobile phase flow and scanning the full UV/V IS spectrum of the component trapped in the sample cell (stop-flow scanning).

Fixed Wavelength UV Detectors

There are several types of fixed wavelength UV detectors that differ mainly in the output of the source. One type has a low pressure mercury lamp source that emits a sharp line spectrum. A filter passes the principal 254 nm line and removes other weaker lines. A second type has a modified mercury lamp with a phosphor and provides output at 254 or 280 nm. A third type employs a medium pressure mercury lamp and band pass filters to isolate emission lines at 220, 254, 280, 313, 334, or 365 nm.

Figure 605-a
UV/VIS Detector



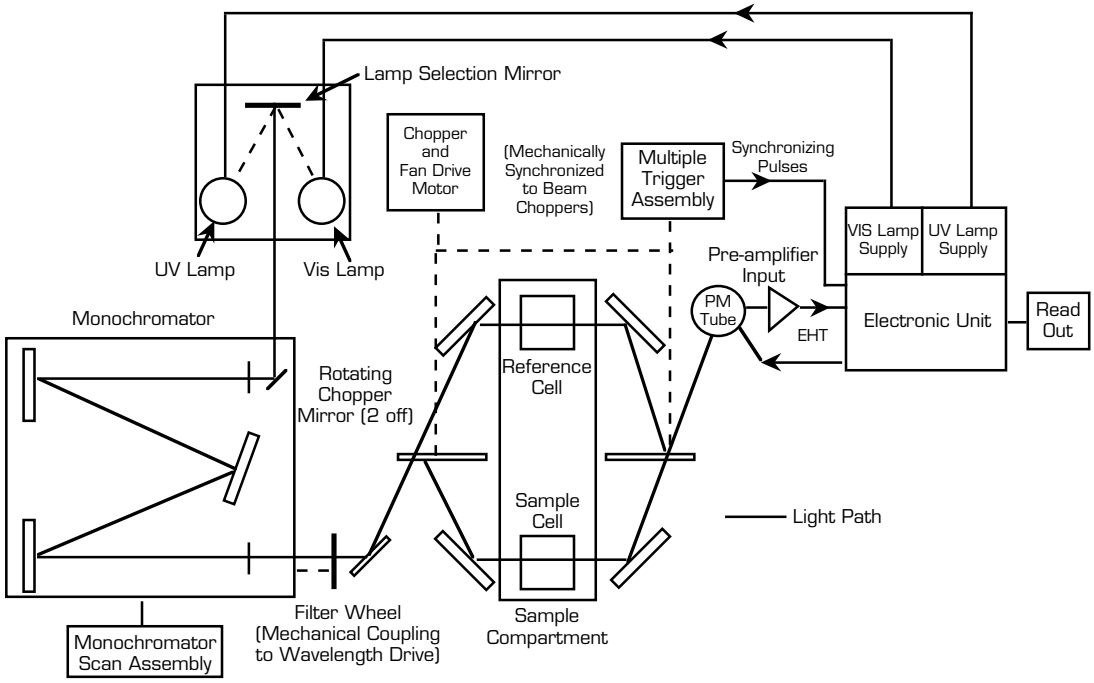
[Reprinted with permission of John Wiley and Sons, Inc., from Meyer, V.R. (1988) *Practical High Performance Liquid Chromatography*, Figure 5.5, page 69.]

Figure 605-a shows a diagram of the light path and liquid flow path for a double-beam fixed wavelength UV/VIS detector. Light from the source is focused by a quartz lens onto sample and reference cells. Column effluent flows continuously through the sample side (top), while the reference side (bottom) is filled with pure mobile phase or air. A wavelength of absorption is chosen by the filter. After passing through the filter, the radiation is chopped by a rotating sector so that alternating pulses fall on the detector (double beam in time). The presence of absorbing analyte in the sample cell decreases the intensity of the sample beam relative to the reference beam. The difference in signal between the two beams, which represents the absorbance of the analyte, is amplified and recorded.

In an alternative detector design, the sample and reference radiation fall on two photodetectors whose outputs pass through pre-amplifiers to a log comparator, which produces the absorbance signal. Double-beam design compensates for changes in source output and photomultiplier tube response.

Detector cells are typically 1 mm id with a 10 mm optical path, or approximately 8 µL volume. The most common designs are the H-cell, Z-cell, and the tapered cell. The latter design minimizes the effects of changing mobile phase RI, as can occur during gradient elution.

Figure 605-b
Variable Wavelength UV/VIS Detector



[Reprinted with permission of Elsevier Science Publishers, from Poole, C.F., and Schuette, S.A. (1984) *Contemporary Practice of Chromatography*, Figure 5.6A, page 377.]

Variable Wavelength UV Detectors

The second major UV/VIS detector is the continuously variable wavelength type (Figure 605-b). Light from a continuous deuterium source (or tungsten source for the visible region) is focused on the entrance slit of a grating monochromator, which disperses it into its component wavelengths. The monochromatic light emerging through the exit slit is divided into sample and reference beams by a beam splitter or chopper. The detector measures the difference in absorbance between the sample and reference.

Solvents

Many solvents absorb strongly in the UV and cannot be used in certain spectral regions. It is important to choose solvents that are transparent at the wavelength(s) being used. For example, carbon tetrachloride, benzene, and acetone cannot be used at 254 nm because they absorb too strongly. Hexane, chloroform, methanol, and water are transparent at 254 nm and provide a wide range of solvent strength for preparing mobile phases. The choice of solvents with UV cutoffs <220 nm is quite limited.

Performance Characteristics

The UV detector is sensitive to 10^{-6} to 10^{-10} g/mL of many compounds. It has a wide linear range and is relatively insensitive to small changes in flow or back pressure, although at the detection limit the detector is very sensitive to changes in temperature. Detection is limited to compounds that absorb at the chosen wavelength.

Gradient elution is possible provided the solvents do not absorb. At very sensitive settings, changes in RI, as caused by gradient elution or pressure and flow changes, can produce baseline shifts with some types of detector cells.

The fixed wavelength detector is less versatile but is much less expensive and often gives less noise than the continuously variable wavelength spectrophotometric detector. As mentioned above, the great advantage of the variable wavelength detector is the ability to optimize sensitivity and/or selectivity for each analyte by detection at the most favorable wavelength.

Multichannel or Photodiode Array Detectors

In a photodiode array detector, polychromatic radiation is passed through the detector flow cell, and emerging radiation is diffracted by a grating so that it falls on an array of photodiodes. Each diode receives a different narrow wavelength band. The complete array of diodes is scanned by a microprocessor many times a second. The resulting spectra may be displayed on a cathode ray tube monitor and/or stored in the instrument for transfer to a recorder or printer. The detector is best used in conjunction with a computerized data station, which allows various post-run manipulations, such as identity confirmation by comparison of spectra with a library of standard spectra recalled from disk storage. Detection can be made at a single wavelength or at a number of wavelengths simultaneously, or wavelength changes can be programmed to occur at specified points during the run. Absorbance ratios at selected wavelengths (*e.g.*, 254 and 280 nm) can be displayed for each peak, which aids in determining identity and the presence of unresolved components.

Applications

The UV detector has been the most widely used for pesticide residue determination. Section 404 uses UV and fluorescence detectors to determine benzimidazole residues, whereas other references describe combinations of UV and photoconductivity [1-3]; the photodiode array is applicable to determining paraquat and diquat [4].

Problems, Maintenance, and Troubleshooting

Air bubbles in UV flow cells can produce a series of very fast noise spikes on the chromatogram, or pronounced baseline drift. Falsely high absorbance readings can be caused by impure or improperly prepared mobile phase, large air bubbles in the flow cell, a misaligned flow cell, or dirty end windows. Gas bubbles develop in the detector cell because they are pumped through the system or the solvent is degassed in the detector. Prevent bubbles from being pumped through the system by eliminating system leaks, expelling air from the pumping system, avoiding very volatile solvents, and not stirring the mobile phase reservoir too vigorously. Prevent solvent degassing in the sample cell by degassing the mobile phase prior to use. If the cell has no back pressure valve, raise cell pressure above atmospheric by attaching ≥10' spiral steel or Teflon tubing to the detector outlet to act as a flow restrictor, and placing the tubing outlet above the detector. The tubing must not shut off flow completely, as too great a pressure increase could shatter the cell windows.

To dissolve gas bubbles lodged in the cell, briefly increase cell back pressure by holding a piece of rubber septum over the detector outlet or by connecting a syringe to the outlet. With aqueous systems, it may be necessary to fill the cell with methanol and repeat application of back pressure.

Protect the detector from temperature fluctuations by placing the system away from direct sunlight and drafts, and regularly monitor flow rate and pressure for change.

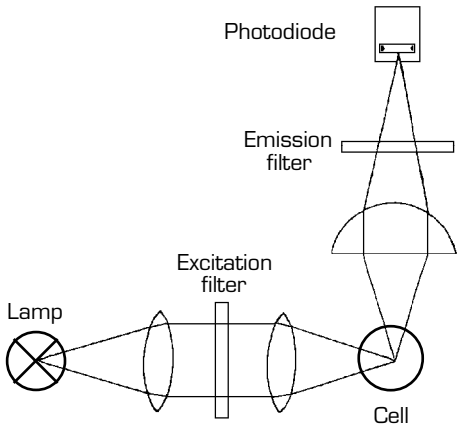
Detector response can drop because dirt in the cell or a bad source lamp reduces the level of radiation reaching the photocell. Some detectors have a meter that allows easy determination of light level. If it is low, clean the detector or change the source lamp. (Avoid eye damage by not viewing the light directly.) Consult the detector manual for the proper procedure for changing the lamp and cleaning the cell. The average life of a 254 nm lamp is approximately 5000 hr, but it should be replaced as soon as aging begins to cause significant intensity changes. Some cells can be taken apart, the optical components cleaned with a suitable solvent and dried, and the cell re-assembled. Others cannot be taken apart and are cleaned by flushing the cells with a series of solvents delivered from a 50 mL glass syringe, *e.g.*, acetone, 6 M nitric acid, distilled water, and acetone, then drying with a flow of clean, dry nitrogen before reconnection to the column. If necessary, allow 6 M nitric acid to stand in the cell overnight. To remove particles most effectively, draw nitric acid through the cell with a syringe in a direction opposite to the normal flow.

605 B: FLUORESCENCE DETECTORS

Fluorescence detectors provide two to three orders of magnitude more sensitivity than UV detection. Selectivity is also excellent because of the choice of excitation and emission wavelengths and the fact that only a small fraction of all compounds naturally fluoresce.

The simplest type of instrumentation is a fixed wavelength fluorometer with bandpass filters for both excitation and emission. More convenient and versatile fluorometric detectors can operate at variable wavelengths. These are equipped with monochromators to select excitation and emission wavelengths. Most compounds that fluoresce naturally have a rigid, planar conjugated cyclic structure. Nonfluorescent compounds can be detected if they are first converted to fluorescent compounds by pre- and post-column derivatization.

Figure 605-c
Fluorescence HPLC Detector



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Detector Design

Figure 605-c is a schematic diagram of a simple filter fluorometer detector. Light from a mercury lamp passes through a filter that selects the excitation wavelength. An interference filter providing a 10-20 nm

bandpass is commonly used. Lenses focus the radiation on the sample cell, which contains the flowing column effluent. If fluorescent compounds are present, they absorb the incident radiation and re-emit at a longer wavelength.

Although it is emitted in all directions, the re-emitted or fluorescent light is usually measured at a right angle to the direction of the incident light. A second filter isolates a suitable wavelength from the fluorescence spectrum and rejects any scattered exciting radiation from the source. A lens focuses the emitted light on a photomultiplier tube.

The second filter can be a bandpass filter for selectivity, or a cutoff filter for greater sensitivity.

The right angle measurement design allows monitoring of the incident beam as well as the emitted light, so that dual UV absorption and fluorescence detection is possible with some commercial detectors. A more common arrangement is to place a separate UV detector in tandem with a fluorescence detector to check the reliability of the fluorescence measurements, especially at low levels of quantitation.

Other more sophisticated fluorescence detectors use grating monochromators instead of filters and are termed continuous wavelength spectrofluorometric detectors. These usually have either a deuterium (190-400 nm) or xenon (200-850 nm) arc source. Because these sources are more unstable than a mercury discharge source, detector design is often modified to correct for fluctuations in source intensity by splitting off a portion of the exciting light to a reference detector. Variable wavelength detectors have the advantage of allowing low wavelength excitation, which is necessary for some naturally fluorescent compounds such as indoles and catecholamines.

Fluorescence detectors that use a laser as the excitation source are being studied. The intensity of lasers is about 10^4 higher than that of conventional sources, providing a 10-100 fold improvement in detection sensitivity. The use of lasers can reduce stray light, because their radiation is entirely monochromatic and coherent and the beam has a small cross section and is nondiverging. At present, the use of lasers as detector sources is limited by their high cost and relatively narrow range of available excitation wavelengths.

Solvents

The intensity of fluorescence is affected by the composition of the mobile phase and the presence of impurities. Quenching can occur with halide ions, water and other strong hydrogen-bonding solvents, and buffers. High temperature and oxygen in the mobile phase can also induce quenching. Compounds that fluoresce in organic solvents may show a shift in intensity and fluorescence maximum wavelength with change in solvent polarity.

Performance Characteristics

Sensitivity is greater for fluorescence because the signal is measured directly against a dark background, and signal intensity can be increased by an increase in source intensity. The sensitivity of fluorescence detectors is in the range of 1-100 pg/mL for favorable compounds. Because both the excitation and detected wavelengths can be varied, selectivity is high for fluorescence detection. The linear range is

generally two or three orders of magnitude at low concentrations where absorbance is <0.05. At higher concentration levels, the linear range can be very small.

Parameter Adjustments

Optimum wavelengths for fluorescence detection are chosen by scanning analyte excitation and emission spectra with a fluorescence spectrofluorometer. For most compounds, excitation and emission spectra are mirror images that more or less overlap at longer excitation and shorter emission wavelengths. When excitation and emission maxima are close together, optimization of fluorescence detector monochromator settings is critical for achieving maximal emission output while avoiding light-scattering effects due to the overlap. Careful choice of emission wavelength and slit width based on spectral characteristics is necessary for maximum sensitivity, as well as for controlling background noise if fluorescent impurities are present in the injected extract [5].

Applications

Section 401, method for N-methylcarbamates, uses post-column hydrolysis and derivatization to produce a chemical detectable by a fluorescence detector; a variation of that determinative step detects naturally fluorescent residues without the post-column reactions. Section 403 uses photolysis to degrade substituted ureas for subsequent fluorometric labeling and determination. Section 404 uses UV and fluorescence detectors for benzimidazole residues.

Detector Maintenance

Clean the cell compartment with chromic acid (or equivalent) cleaning solution, followed by thorough rinsing with dilute nitric acid and water. An overnight soaking in chromic acid cleaning solution may be necessary to remove stubborn impurities.

605 C: ELECTROCHEMICAL DETECTORS

Electrochemical (ECh) detectors include the conductivity detector for the determination of ionic analytes and amperometric, coulometric, and polarographic detectors for analytes with oxidizable or reducible functional groups. The first ECh detector for HPLC used polarography, but because use of this mode today is infrequent, it will not be discussed in this chapter. The most widely used type is the thin layer amperometric detector using a glassy carbon electrode, or, less frequently, a gold amalgam or carbon paste electrode, which can provide sensitive and selective determination of compounds with appropriate structures.

ECh detection can be used with reverse phase (RP) columns because of the high polarity of RP mobile phases. For the detector to function properly, the mobile phase must possess good electrical conductivity. Salt or buffer at a concentration of 0.05 M is often used to provide the required ionic strength. The detector is impractical for normal phase (NP) HPLC, and RP systems with high modifier concentrations may also cause problems.

ECh detection has been applied to the HPLC determination of phenols, amines, mercaptans, halogen compounds, ketones, aldehydes, and nitroaromatics. It is suitable for quantitation of various pesticide classes, such as dinitroaniline,

bipyridinium, triazine, and phenylurea herbicides; nitrophenyl, dinitrophenol, carbamate, and organophosphorus insecticides; and azomethine insecticides and fungicides. Any pesticide that produces an electroactive compound (phenol, aromatic amine, aromatic nitro compounds) on metabolism or decomposition, such as carbamates, ureas, anilides, *etc.*, can potentially be determined using the ECh detector.

Conductivity Detectors

Conductivity detectors measure the conductance (reciprocal of resistance) of the effluent, which is proportional to ionic analyte concentration if the cell is suitably designed. They are usually used to detect inorganic or organic ions after separation by ion exchange or ion chromatography. Since these modes of HPLC employ mobile phases with high conductances, it is necessary to incorporate a chemical or electronic means of eliminating the conductance of the mobile phase before the analyte can be measured sensitively and accurately with a conductivity detector. Typical detectors have a cell with a small (2 μL) active volume, composed of insulating material, into which graphite or noble metal electrodes are implanted.

A constant alternating voltage is applied to the electrodes, and conductance is measured with an appropriate circuit, such as a Wheatstone bridge. Since conductivity is highly temperature dependent, a means for automatic temperature compensation is usually included.

Amperometric and Coulometric Detectors

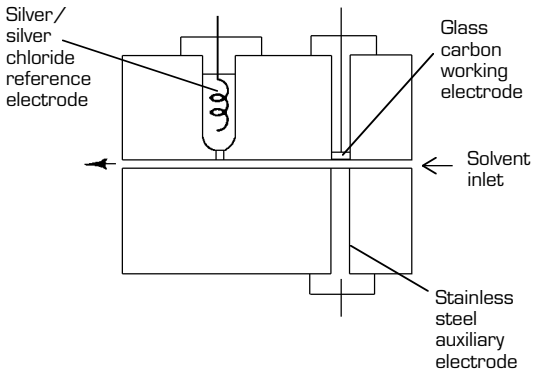
ECh detectors are most often amperometric or coulometric detectors that measure current associated with the oxidation or reduction of analytes. Oxidation is carried out at an anode bearing a positive potential, and reduction at a cathode having a negative potential. Different compounds require unique potentials for these electrochemical reactions to occur. The current produced by the electrode reaction is measured in a flow cell at the column outlet. Detector selectivity and sensitivity are changed by varying the potential of the electrode.

The use of electrochemical reduction as an HPLC detection method is impractical because oxygen is easily reduced, and it is difficult to remove oxygen completely from the mobile phase. Most ECh detector applications are, therefore, based on oxidation. Mobile phases that work best are aqueous/organic mixtures with added salts or buffers.

The coulometric type of ECh detector reacts all of the electroactive analyte passing through it, yielding a higher current for the electroactive species than the amperometric detector. However, background noise is also greater, so it is not more sensitive. The coulometric detector is insensitive to flow rate and temperature changes, and, like the GLC microcoulometric detector, it responds in an absolute manner, eliminating the need for calibration. However, it is more prone to electrode contamination and must be designed to provide strict potential control over the entire electrode area. The coulometric type of ECh detector is much less popular than the amperometric type.

The amperometric ECh detector uses a smaller electrode surface and reacts only about 1-10% of the electroactive analyte, so that most of the analyte leaves the detector cell unchanged. Small currents in the nanoampere range are produced.

Figure 605-d
Three Electrode Electrochemical Detector



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These currents can be amplified and measured accurately, leading to sensitivities as low as 0.1 pmol in favorable cases. Amperometric ECh detectors are also simple in design and relatively inexpensive and can be made with a very small internal volume (0.1-5 µL), thereby minimizing band broadening, but they are difficult to use.

Figure 605-d is a simplified schematic diagram of an amperometric ECh detector. Three electrodes are used: the working electrode at which the current due to the analyte is measured, a silver/silver chloride reference electrode against which the potential at the working electrode is selected, and a stainless steel auxiliary electrode to carry the current arising from the electrochemical reaction. The working electrode is most commonly glassy carbon, which is a highly polished, inert, and electrically conducting form of carbon. To maintain reproducibility, the solid carbon electrode requires regular maintenance in the form of polishing and cleaning, so detectors must be relatively simple to take apart and re-assemble.

The chromatogram is obtained by measuring the current at the working electrode, which is maintained at a fixed potential relative to the reference electrode, as the electroactive analyte elutes from the column. The working electrode potential is usually at or near the limiting current plateau of the analyte. The background current, which is constant for a given mobile phase flow rate and composition, is subtracted from the analytical signal to give a detector current that is proportional to the concentration of the analyte according to Faraday's law. Cyclic voltammetry is often used to obtain preliminary electrochemical data that determine the optimum applied potential and the effect of variables such as solution pH, mobile phase composition and concentration, and analyte structure.

Performance Characteristics

In general, ECh detection offers better sensitivity and selectivity than the UV detector for pesticide residue determination. Detection limits are generally at picogram levels, whereas for the UV detector, detection limits are, at best, low nanogram levels. On the other hand, the UV detector has greater long term stability and is easier to use on an everyday basis.

Detector operation and sensitivity are critically dependent on flow rate constancy, solution pH, ionic strength, temperature, cell geometry, condition of the electrode surface, and the presence of electroactive impurities (*e.g.*, dissolved oxygen, halides, trace metals). The detector cannot be used with flow or solvent programming if these changes affect the baseline, and waiting periods of ≥10 min are required for variations in conditions such as flow rate, applied voltage, or mobile phase, or for initial startup each day. Both increased flow rate and an increase in the volume of injected extract decrease detection sensitivity.

Applications

An ECh detector was used for oxidative detection of coulometrically reduced organonitro pesticides separated by RP HPLC [6]. Pesticides were separated on a C-8 bonded column and monitored indirectly by means of a porous graphite coulometric detector. The organonitro functional groups were reduced in the guard cell of the detector, and the reduction products were then detected by electrochemical oxidation. A 20-90% acetonitrile/water gradient with constant electrolyte concentration could be used without occurrence of a significant baseline change. The cell required periodic cleaning with dilute nitric acid and sodium hydroxide solutions to eliminate negative peaks.

HPLC with ECh detection was also used to determine 0.01-0.02 ppm ethylenethiourea (ETU) in foods by a revised official AOAC method [7, 8]. The prepared extract was chromatographed on a graphitized carbon column with acetonitrile/aqueous 0.1 M phosphoric acid/water (5:25:70) mobile phase, and the eluted ETU was detected by using an amperometric ECh detector having a gold/mercury working electrode.

605 D: PHOTOCONDUCTIVITY DETECTORS

The photoconductivity detector (PCD) is sensitive and selective for organic halogen, sulfur, and nitrogen compounds that form strong, stable ions upon photolysis. The effluent is split as it leaves the column; one-half is passed through the reference cell of a conductivity detector and the other half is irradiated with 214 or 254 nm UV light. Suitable analytes become ionized, and the resulting conductance is measured by the detector. Operation of the PCD requires an ion exchange resin to purify the mobile phase and lower background conductivity. Both RP and NP (nonaqueous) systems have been used with the PCD, but the former are more commonly used for pesticide determination and will be emphasized in this section.

Apparatus

Publications [9, 10] describing applications of a PCD to pesticide residue determination employed a system with a reciprocating pump, loop injector or autosampler, forced draft column oven, variable wavelength UV detector in tandem ahead of the PCD, dual recorders, a data system for peak integration, and C-18 and cyano bonded columns. A flow splitter was adjusted to give equal flow rate of column effluent through the analytical and reference cells of the detector. Balance of flows through the reference and analytical loops is facilitated by a metering valve in the solvent line exiting from the reference compartment of the conductivity cell. This apparatus, or an equivalent system, will be assumed in this section.

Performance Characteristics

The following performance characteristics have been determined by studies of a PCD-UV detector system for residue determination.

Mobile Phase Preparation. Optimum sensitivity and stable baselines are achieved when the mobile phase has a minimal ionic background concentration. De-ionization of the mobile phase solvents, either individually or as a mixture, is carried out

by circulation through a mixed bed cartridge (1:1 mixture of anion and cation exchange resins). Ion exchange treatment of aqueous mobile phases shortly before use with the PCD is recommended. A 24 hr period of resin circulation at 2.5 mL/min was chosen arbitrarily for "complete purification" of the solvent. Resin-treated acetonitrile was found to be incompatible with the PCD-UV detector system, and the use of resin-treated methanol rather than treated or untreated acetonitrile is recommended.

Temperature Control. More sensitive and consistent detection was obtained when the column, photolysis reaction chamber, conductivity cells, and associated plumbing were all maintained at a constant, elevated temperature (35-40° C) within a column oven.

Mobile Phase Flow Rate. The use of low flow rates improves detector response and reduces the expenditure of purified mobile phase. However, lower flow rates lead to longer analysis time unless the strength of the mobile phase can be increased without losing the required resolution. A compromise among speed, resolution, and detection sensitivity is necessary, depending on the requirements of a particular analysis.

Pressure. The PCD is very sensitive to pressure fluctuations. Thorough mobile phase degassing and subsequent gentle sparging with helium help maintain stable pumping pressure. Use of gradient elution is limited by pressure variations that occur as the mobile phase composition changes, leading to excessive baseline shift, especially in high sensitivity applications.

Reproducibility of Response. Improved reproducibility was shown to result from complete purification of the mobile phase by ion exchange resin treatment and, to a lesser degree, temperature control.

NP (Nonaqueous) Solvent Operation. The practical application of the PCD to NP HPLC is limited by the low polarity of the mobile phases used. This results in poor ion mobility and poor charge transfer in the conductivity cell and thus adversely affects peak shape and response. Some workers have attained adequate polarity for good response by adding acetic acid or other polar or ionic modifiers to nonaqueous mobile phases, but this approach is limited by the increase in background noise the added compounds can cause. Reduced background conductivity and diminished need for purification by de-ionization are advantages of the use of certain nonaqueous solvents.

Choice of Irradiation Wavelength. In general, the 254 nm mercury lamp provides greater detection sensitivity than the 214 nm zinc lamp. However, response can be improved for certain compounds if the zinc lamp is substituted for the mercury lamp. Greater stability is ensured if the detector, including the lamp, is left on at all times.

Sensitivity, Selectivity, and Linearity. The PCD can detect low ng levels of many pesticides and was found to be linear from 1-100 ng injected. Injection aliquots are typically 5 µL containing 1-20 ng pesticide. UV detection typically shows more background interferences from crop extracts than the PCD, indicating superior selectivity for the PCD. Because the PCD is more complex and sensitive to variations in system conditions (*e.g.*, de-ionization and degassing of mobile phases, temperature, pumping fluctuations), it should be operated with a tandem UV

detector to monitor the chromatographic system and aid in the diagnosis of anomalies.

Applications

The PCD has been included in several methods for pesticide residues [1-3, 10].

605 E: MASS SPECTROMETRIC DETECTORS

Mass spectrometric (MS) determination can be definitive, providing information on analyte retention and concentration while simultaneously confirming its identity. Interpretation of mass spectra permits determination of molecular mass, empirical formula, arrangement of molecular constituents, and, ultimately, molecular identity.

Successful use of MS as a chromatographic detector requires introduction of column effluent to the MS without breaking the vacuum in which the detector operates. This task is now easily accomplished for GLC-MS, but systems for vaporizing or otherwise eliminating the HPLC mobile phase before introduction to the MS are still being developed. Numerous interfaces and ionization techniques for HPLC-MS have been made available, but so far no one system serves all needs.

An HPLC-MS interface involves two stages: effluent introduction and analyte ionization. Available effluent introduction techniques include spray techniques, in which the analyte is introduced as an aerosol; direct liquid introduction, and mechanical transfer, such as the moving belt interface. Spray techniques are most common; the aerosol may be produced through nebulization of the effluent using thermal (*e.g.*, Thermospray), pneumatic (heated nebulizer, particle beam, Thermabeam), or electrostatic (electrospray, Ion Spray) processes.

The particle beam interface actually combines several processes to remove solvent from the effluent. After nebulization, the resulting aerosol loses its more volatile components in a desolvation chamber. Subsequently, the remaining aerosol is pumped through a momentum separator, a series of skimmers and pumps that divert most of the solvent vapors. The heavier analyte molecules then pass into the MS.

Several approaches to analyte ionization exist; the combination of effluent introduction and ionization must be compatible. Direct ionization of the effluent occurs in Thermospray or electrospray interfaces; vaporization and ionization occur simultaneously. Another direct ionization technique, called fast atom bombardment, ionizes the analyte by bombarding the effluent with fast moving argon atoms. Electron impact ionization can be achieved if the solvent is removed first, such as with the particle beam interface. Chemical ionization (CI) can be used without removal of solvent, using "filament-on" Thermospray, direct liquid introduction, or one of several atmospheric pressure chemical ionization (APCI) systems that now exist.

APCI is a recent and significant improvement in HPLC-MS interfacing technology. APCI is a process of ion formation that occurs at atmospheric pressure outside the MS. APCI ion sources are unique and versatile because they can be utilized with most of the interfaces described above. One unique advantage of APCI systems is that interfaces (*e.g.*, electrospray, heated nebulizer) can be readily interchanged

without venting the MS. APCI most commonly provides CI spectra, but with tandem MS instruments (MS/MS), more complex spectral information is obtainable.

Selection of an interface for a particular HPLC-MS application requires consideration of many factors. Among these, HPLC mobile phase flow rate may limit the choice of interface to those capable of handling the volume; *e.g.*, Ion Spray interface is limited to 200 µL/min, while Thermospray can accept 1 mL/min. Thermal stability of the analyte(s) may also restrict choice of interface. The interface must also be compatible with mobile phase composition; *e.g.*, nonvolatile salts or buffers may clog some interfaces, flammable solvents are generally unsuitable, and high aqueous content may inhibit volatilization.

Numerous applications of HPLC-MS have been published, and reviews of the applications are available [11, 12]. References to the use of particle beam interface [13-15] and to APCI [16, 17] provide information about application to pesticide residue determination.

605 F: DERIVATIZATION FOR DETECTION ENHANCEMENT

The detection properties of an analyte can in many cases be enhanced by pre- or post-column derivatization. Pre-column derivatization is usually carried out independent of the instrument and post-column derivatization is usually performed in-line. Derivatization reactions have been carried out mostly in conjunction with fluorescence detectors, but visible absorption and ECh detectors are also widely applied.

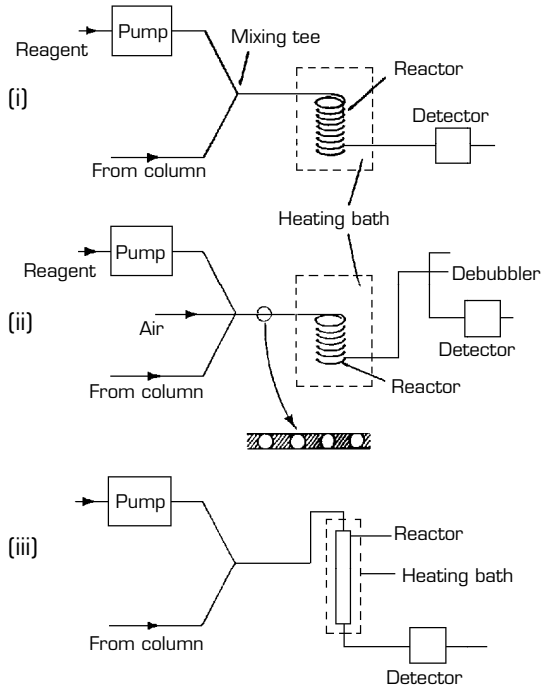
Comparison of Pre- and Post-Column Derivatization

Pre-column derivatization procedures have the advantages that long reaction times and extreme reaction conditions can be used, and reagents can be employed that have the same detection properties as the derivatives. This is not possible with post-column reactions, because excess reagent is fed with the effluent to the detector. Pre-column derivatization may serve as a purification step, and the derivatives may chromatograph more favorably than the parent compounds. However, the derivatives are usually more similar structurally than the parent compounds, reducing the chromatographic selectivity. Pre-column derivatization requires a quantitative reaction resulting in a stable and well defined product; by-products formed in the reaction may interfere with the analyte in the chromatogram, necessitating extensive cleanup after the derivatization reaction. No instrumental modifications are required for pre-column derivatization, unless it is carried out in-line. In general, post-column procedures allow for a higher degree of automation. Reactions for post-column derivatization should be rapid to avoid extra-column band broadening. An upper practical limit for high efficiency HPLC is about 20 min.

Post-Column Reactor Design

Post-column in-line derivatization is carried out in a reactor located between the column and detector. The mobile phase flow is not interrupted, although it may be augmented by addition of a secondary solvent to aid the reaction or meet detector requirements. This is especially important for ECh detectors, for which the mobile phase and derivatization reagent are seldom fully compatible. Since the reactor is located after the column, products of derivatization will not interfere

Figure 605-e
Post-Column Reactors



(i) Open tubular reactor; (ii) Segmented reactor; (iii) Packed bed reactor

[Reprinted with permission of John Wiley and Sons, Inc., from Lindsay, S. (1987) *High Performance Liquid Chromatography,* Figure 2.4p, page 79.]

with chromatography. The derivatization reaction does not have to go to completion or be well defined if it is reproducible. The reaction should take place in a reasonable time, and the reagent should not be detectable under the same conditions as the derivative. A high concentration of reagent is usually used to minimize dilution effects, and a moderately elevated temperature to speed the reaction.

Figure 605-e shows schematic representations of three types of post-column detectors, the designs of which are strongly influenced by the time required for the reaction. In all of these designs, controlled volumes of one or more reagents are added to the column effluent, followed by mixing and incubation for a certain time period with controlled temperature. Reagents are added at low pressure using pulse-free peristaltic pumps.

In the open tubular or open capillary reactor (i), reagent is pumped via a mixing tee into the column effluent containing the separated analytes. The reactor, which is a coil of stainless steel or Teflon capillary tubing (typically 0.3 mm id, 150-600 µL volume), provides the necessary time for the reaction without significantly contributing to band broadening. The combined streams are finally passed on to the detector. This type of reactor is suitable for fast (≤ about 1 min) derivatization reactions, *e.g.*, for determination of amines with o-phthalaldehyde reagent.

The segmented stream tubular reactor (ii) is used for slower reactions (5-30 min). Bubbles of air or a nonmiscible liquid are introduced into the stream at fixed time intervals. This segments the column effluent into a series of reaction volumes whose size is governed by the dimensions of the reaction tube and the frequency of the bubble introduction. Optimal conditions include small liquid segments introduced at high frequency; short, small id reaction tubes; and a high flow rate. This type of reactor reduces analyte diffusion and band broadening. The segmentation agent is generally removed by a phase separator prior to the detector, but noise can also be suppressed electronically.

For intermediate speed reactions (0.3-5 min), packed bed reactors (iii) consisting of a column containing a nonporous material such as glass beads have been used. Reagent is pumped into the flowing effluent stream, and the mixture enters the reactor column. Improper packing of the reaction column can lead to band broadening in the same way as for the analytical column.

A second detector can be placed ahead of the reaction detector to gather additional information about the analyte. For example, a UV detector can be utilized prior to a derivatization/fluorescence detector.

Post-column derivatization techniques can involve simple modification of solution pH. For example, post-column conversion from slightly acid to a pH above 8 increases the fluorescence of coumarin anticoagulant rodenticides and allows their sensitive determination with a fluorescence detector. More commonly, reagents are used that produce fluorescent, UV-absorbing, colored, or electroactive derivatives. Formation of a UV-absorbing derivative is difficult because most suitable reagents are also strongly absorbing. Reagents can be directly reacted with the analyte, or an initial hydrolysis or oxidation reaction is sometimes carried out, followed by derivatization of the product. Fluorescamine, dansyl chloride, and o-phthalaldehyde are examples of fluorogenic reagents, and ninhydrin is a common chromogenic reagent for amino acids. Derivatives containing nitroaromatic chromophores can be used for UV or ECh detection based on reduction. p-Aminophenol derivatives of carboxylic acids and p-dimethylaminophenyl isocyanate derivatives of arylhydroxyamines are also suitable for ECh detection.

The most important applications of post-column derivatization to pesticide determination have involved detection of amines. One example is the detection of N-methylcarbamate insecticides and metabolites as described in Section 401.

Photochemical reactors have been employed to convert compounds to a more readily detectable fluorescent species, or to a fragment that can be coupled with a detection- enhancing reagent. The reactor often consists of a Teflon or quartz coil wrapped around a high power UV lamp in a reflective housing. The length of the coil is optimized in relation to the desired irradiation time. An example of this technique applied to residue determination is the method for substituted urea herbicides, Section 403.

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606: RESIDUE IDENTIFICATION AND QUANTITATION

606 A: RESIDUE IDENTIFICATION

The first step in determining residues in a cleaned up, concentrated extract is to run a preliminary chromatogram. Tentative peak identification is made by comparing retention data with data for standards measured under identical conditions. If these data indicate the presence of one or more probable pesticide peaks, proper standard solutions are prepared for qualitative confirmation and quantitation.

Identical retention characteristics of an analyte and reference substance in a single determination do not assure accurate identification, because several compounds may have the same retention time under any given set of conditions. Confirmation of peak identity must be obtained by use of additional determinations (Section 103). Co-chromatography and HPLC using alternative (dissimilar) columns and/or selective detectors are most appropriate for residues determined with HPLC. Other chromatographic methods such as GLC or thin layer chromatography (TLC), or UV, IR, nuclear magnetic resonance (NMR), or mass spectrometry (MS) may also be useful for confirming residue identity.

Relative retention time, the ratio of the absolute retention of the compound of interest to that of a selected reference standard ("marker compound"), is more reproducible than the absolute retention time, so it is used to compare residue and standard peaks. Only the composition of the mobile phase influences the relative retention time, whereas absolute retentions can vary slightly from day to day or even from hour to hour if instrumental parameters, such as mobile phase flow rate, recorder chart speed, or injection technique vary. The marker compound may be chromatographed just before or after the sample, but it is best to include a portion of the marker compound in the injection of the sample extract; in this way, both residue and marker peaks are chromatographed at the same conditions and appear in the same chromatogram.

In addition to relative retention time, peak shape is often another useful aid in comparing sample and standard chromatograms. Residue identity can be confirmed by observing changes in absolute or relative retention time upon derivatization of both the analyte and the appropriate reference standard.

Co-chromatography

Co-chromatography provides an alternative means of qualitative analysis based on retention times. An amount of pure standard compound, thought to be the analyte, is added to a portion of the sample extract at approximately double the amount present, and an aliquot is re-injected. If the tentative identification of the residue was correct, only the peak due to the analyte will be intensified, and peak shape will not be distorted (*i.e.*, no shoulders or broadening will be produced). This method has the same limitation as comparison of retention times, in that it is possible that another compound with chromatographic characteristics corresponding to the added compound might be present. Compound identification using this technique is enhanced by high column efficiency and resolution and optimized operating parameters; when pesticides are well resolved from one another and from nonpesticide artifacts co-extracted from the sample substrate, there will be the greatest chance for the analyte and the co-injected standard to separate if they

are not the same compound. If the HPLC system has recycling capability, the co-injected mixture can be recycled several times to try to separate the analyte from the added standard.

Use of Alternative Columns

Concurrence of retention times between analyte and standard peaks, or absence of separation of peaks in a co-injected sample plus standard, on two or more different HPLC columns (each with a suitable mobile phase) gives greater assurance that the two peaks represent the same compound. However, the columns must be judiciously chosen so that their separations are governed by distinctly different mechanisms that produce different elution patterns. Reverse phase (RP) and normal phase (NP) partition columns and silica gel adsorption columns have been shown to be independent, complementary columns for confirmation of peak identity for many compound types.

Spectrometric Confirmation

It is possible to confirm identification spectrometrically by collecting the analyte as it elutes from the HPLC instrument, either manually or with an automatic fraction collector, and analyzing it with UV, MS, IR, or NMR. However, the practical application of this approach for trace pesticide determination is limited. Because the sensitivity of such instruments is limited, eluates from HPLC are often difficult to concentrate, and buffers and salts from RP mobile phases can interfere.

Spectrometric confirmation can be performed in-line. The absorbance (peak height) ratio at two different UV wavelengths, *e.g.*, 254 and 280 nm, can be characteristic for a particular compound, and comparison of the ratio for the analyte and a standard can be helpful for peak confirmation. The presence or absence of peaks or the signal ratio when using different selective detectors provides additional confirmational information. Combinations that have been employed for pesticide determination include the UV detector followed by a photoconductivity, fluorescence, or electrochemical detector. Specificity is obtained by the position of the absorption wavelength with the UV detector, the excitation and emission wavelengths with the fluorescence detector, and the reduction or oxidation potential with the electrochemical detector.

Identification can be made by use of a scanning UV/VIS detector. The spectrometer is initially set to a wavelength that produces a strong signal for the chromatographic peak to be identified. When the absorbance signal is at its maximum, the flow of mobile phase is stopped by means of a stop-flow valve and the full spectrum of the trapped component is scanned. The flow of mobile phase is then restarted and the analysis continued. A limitation of this approach is that UV and visible absorption spectra are not as characteristic as IR, NMR, and MS for compound identification.

Scanning of fluorescence spectra provides somewhat better characterization. If full spectrum detection is used for identification confirmation, the following criteria have been suggested [1]: the maximum absorption wavelength in the spectrum of the analyte should be the same as that of the standard material to which it is compared within a margin determined by the resolution of the detection system. For diode array detectors, this is typically ±2 nm. The spectrum of the analyte should not be visually different from the spectrum of the standard material

for the parts of the two spectra with a relative absorbance >10%. This criterion is met when the same maxima are present and when the difference between the two spectra is never >10% of the absorbance of the standard material at any point.

606 B: QUANTITATION

Techniques for quantitating detector response by measurement of chromatographic peaks are the same for HPLC as for GLC. Section 504 provides directions for both manual peak measurement and use of electronic integrators; these directions should be followed in HPLC quantitation.

Pesticide residues are quantitated by comparing the size (height or area) of the peak for each analyte and the size of a peak from a similar, known amount of each reference standard injected under the same HPLC conditions just before and/or after the sample injection. Only one standard concentration is required for each analyte if injections are made at concentration levels providing linear detector response. This procedure, which is the most widely used, is known as the external standardization method. Other quantitation methods, such as internal standardization and standard additions, have not been widely used for pesticide residue determination.

The exploratory chromatogram of the sample extract used to obtain qualitative analysis will indicate to the analyst the proper standard solution to be used. The solution should contain the pesticides to be quantitated at proper concentration levels to fall within the linearity range of the detector and also to produce peaks comparable in size (usually ±25%) to those obtained from the chromatogram of the sample extract. Injection of the standard mixture may show that additional dilution of the sample extract is required to produce peaks of the higher concentration pesticides that are within the linear detector range and similar in size to those from the standard mixture. If several standard mixtures are available at different concentration levels, selection of one closely approximating the unknown will facilitate the analysis. It cannot be emphasized too strongly that accurate quantitation is not possible unless standards are prepared and maintained properly and replaced on schedule.

Peak height linearity in HPLC can be lost due to band spreading when the sample solvent is significantly stronger than the mobile phase, *e.g.*, the sample is dissolved in methanol and injected into methanol/water (1:1) mobile phase in an RP column. If possible, the sample should be dissolved in the mobile phase to minimize this problem. Otherwise, the injection volumes must be carefully considered; the amounts of sample and standard injected should be equal, or the sample volume must be kept small and the volume causing the onset of band spreading determined and not exceeded.

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607: QUALITY ASSURANCE AND TROUBLESHOOTING

Previous sections of this chapter included troubleshooting advice related to the specific system components (Sections 601 D, 601 E, 602 F, 603 B, 603 D, 604 C, 605 A, and 605 B). This section provides testing, maintenance, and troubleshooting procedures for the HPLC system as a whole. These procedures are recommended to minimize poor performance, damage, and downtime of the HPLC system and to help ensure that results of HPLC analyses are accurate and precise.

607 A: LIQUID CHROMATOGRAPH MONITORING AND PERFORMANCE TESTING

Consult the instruction manual supplied with each instrument component for specific installation, operation, maintenance, and performance check procedures. The following tests are general in nature and should be appropriate for most HPLC systems and analyses.

Time of use:

- Check visually for solvent leaks at all fittings.
- Operate pump(s) at 1 mL/min, at the detector sensitivity anticipated, and note baseline noise. If noise problems exist, consult the instrument manual.
- Check detector sensitivity by chromatographing a reference standard (or mixture) appropriate for the particular detector being used; note detector response.
- Pretested columns, with which a test chromatogram is supplied by the manufacturer, are preferred. Verify the adequacy of new columns by repeating the manufacturer's test. At time of use, test the column by repeating the performance test specified when the column was purchased, or use an alternative in-house test. Verify the performance of columns packed in the laboratory in the same way and with the same reference material as for commercially packed columns.
- With the column in place and pumps, detector, and recorder or integrator in operation, inject several identical amounts of standard to check that reproducibility is within laboratory specifications (typically ±3%).
- Before using the system for a new application, determine that the detector response is linear and reproducible by construction of a standard curve. Using standards of different concentrations, occasionally spot-check that linearity and response factors are within laboratory specifications.
- Check that the mobile phase components (solvents, salts) are of adequate purity grade and are properly filtered and degassed. If there is indication of contamination or significant concentration change, prepare new mobile phase.

- Do not leave water or aqueous solutions of salts, acids, or bases in the pump(s). Flush the system with an appropriate pure organic or aqueous/organic solvent. Methanol is preferred for long term shutdown.

607 B: TROUBLESHOOTING FROM CHROMATOGRAMS

Efficiency is a measure of the ability of the column to produce narrow peaks (Section 602 C). It is expressed by the plate number (number of theoretical plates) of the column. The ability of the column to separate two components is termed resolution. Resolution is a function of the peak widths (efficiency), the separation between peak centers (selectivity), and the degree of retention for the components by the column (capacity). This section reviews some problems that can be detected by inspection of chromatograms and offers possible causes and solutions for them. Specific procedures for solving most of the problems will be found in the individual sections covering various instrument components.

- Peaks that elute too quickly with poor resolution are usually caused by a flow rate that is too high or a capacity factor that is too low. Increase the capacity factor (and affinity for the column) by using a mobile phase that is weaker, *i.e.*, less polar for adsorption chromatography and more polar for reverse phase (RP) chromatography.

- If peaks of a mixture are not well separated but capacity and efficiency are adequate (*i.e.*, k' = 2-10 and narrow peaks), selectivity is too low. Vary selectivity by changing to a second column operating with a completely different mechanism (*e.g.*, adsorption rather than RP), or try a different mobile phase with both of the columns. If the column is old, its resolution may have deteriorated; try a new column of the same type with the original mobile phase.

- Poor resolution can also be due to low column efficiency. Improve efficiency by lowering the flow rate to increase the number of theoretical plates and improve resolution. Alternatively, improve efficiency by increasing the column length (the analysis will take longer), by using a column with smaller diameter packing, or by increasing the operating temperature (mass transfer is improved). A void at the top of the column can also cause poor efficiency and resolution.

- Loss of retention from one chromatogram to the next can be caused by incomplete column equilibration after gradient elution, adsorption of sample impurities, or loss of column activity. Solve the first problem by proper column regeneration after each gradient elution. Remove adsorbed impurities by washing the column or replacing the top 2-3 mm column packing. Maintain constant column activity by using properly dried solvents (for adsorption chromatography). Use a guard column to help prevent adsorption of high molecular weight and polar impurities by the analytical column. Replace the guard column as required.

- An increase in retention times can be caused by too low flow rate, incorrectly prepared mobile phase mixture, the wrong solvent in one of the pump reservoirs, or too slow rate of change of gradient. If silica gel is being used, the activity of the column may have increased because of the use of more completely dried solvents.

- Additional causes of drifting retention times include differences among solvent batches, changes in the composition of a batch of mobile phase upon standing, changes in temperature, a nonconstant recorder drive or slipping chart paper, or changing mobile phase flow rate caused by nonreproducible pump delivery or a leak in the system.

- Tailing peaks in adsorption chromatography can result from column sites with too much activity. To solve this problem, add an optimum amount of a deactivator (*e.g.*, water) to the mobile phase.

- In RP HPLC, tailing can result if the sample is nearly insoluble in the mobile phase or if the sample is partly or completely ionic. Bonded RP columns have a high proportion of unreacted silanol groups (SiOH) available for secondary reaction with analytes. Some of these silanol groups cause bases to tail, and others affect acidic compounds. Different commercial columns are better or worse in terms of their ability to produce peaks with good peak shapes, but none will be completely free from tailing problems.

- Add appropriate mobile phase additives to ensure neutrality of analytes, thereby minimizing unwanted silanol interactions and the resultant peak tailing. The best additives are usually 10-50 mM triethylamine or dimethylhexylamine for suppressing base tailing, and approximately 1% acetic acid for eliminating the tailing of acids. If both acidic and basic sample components are present, combine the additives to give a cumulative effect.

- In ion exchange chromatography, the cause of tailing peaks can be mobile phase with too low an ionic strength or the wrong pH, or adsorption on the resin. Optimum buffer concentrations are sample dependent, generally ranging from 10-100 mM. Ideally, the pH of the mobile phase should ensure that the solute is completely ionized. Adsorption to the resin can often be eliminated by an increase in temperature or addition of a small percentage of organic modifier to the mobile phase.

- For all types of packings, tailing can be caused by a void at the top of the column or excessively long or wide connection tubing between the injection valve and column or between the column and detector. The latter type of cause is indicated if the tailing of early peaks is greater than that of later peaks, and if tailing is greater for faster flow rates.

- A peak exhibiting fronting (a slowly rising leading edge) is usually caused by overloading. Remedy this by injecting less sample.

- A peak exhibiting a doublet or a shoulder (or tailing) results from a dirty, channeled, or defective column. Regenerate dirty columns by washing or repacking the top of the bed. If the inlet frit rather than the packing is dirty, clean or replace the frit.

- Peaks with a staircase shape that never reach true maximum height result from an incorrect recorder damping control setting.

- A noisy recorder baseline can be caused by incorrect recorder damping control setting, or by incorrect grounding of the recorder or the HPLC

instrument, a defective source lamp or dirty cell windows (UV detector), or contaminated solvent. Baseline noise in the form of successive sharp spikes is most likely due to formation of bubbles in the detector cell. Baseline drift is caused by contamination of the detector cell or column, elution of adsorbed impurities, or a change in detector temperature.

- A negative recorder trace is usually caused by a leak between the sample and reference cell compartments; locate and repair.

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Index to PAM I Methods, by Chemicals Tested for Recovery

Codes: C: complete (recovery); P: partial; S: small; V: variable; R: recovered; NR: not recovered

Index to Names Used for Chemicals in PAM I

demeton-S sulfone: **Use:** demeton-S sulfone
demeton-S sulfoxide: **Use:** demeton-S sulfoxide
demeton-S-methyl: **Use:** metasystox thiol
demeton-S-methyl sulfoxide: **Use:** oxydemeton-methyl
demeton-S-methylsulphon: **Use:** oxydemeton-methyl sulfone
Demosan: **Use:** chloroneb
Derosal: **Use:** carbendazim
des N-isopropyl isofenphos: **Use:** des N-isopropyl isofenphos
des N-isopropyl isofenphos oxygen analog: **Use:** des N-isopropyl isofenphos oxygen analog
desdiethyl simazine: **Use:** desdiethyl simazine
desdiisopropyl propazine: **Use:** desdiethyl simazine
desethyl simazine: **Use:** desethyl simazine
desethyldesisopropyl atrazine: **Use:** desdiethyl simazine
desisopropyl atrazine: **Use:** desethyl simazine
desmedipham: **Use:** desmedipham
desmethyl diphenamid: **Use:** desmethyl diphenamid
desmethyl norflurazon: **Use:** desmethyl norflurazon
Dessin: **Use:** dinobuton
Devrinol: **Use:** napropamide
di(2-ethylhexyl) phthalate: **Use:** bis(2-ethylhexyl) phthalate
di(n-butyl) phthalate: **Use:** dibutyl phthalate
di-allate: **Use:** di-allate
di-n-octyl phthalate: **Use:** di-n-octyl phthalate
dialifor: **Use:** dialifor
dialifos: **Use:** dialifor
diazinon: **Use:** diazinon
diazinon metabolite: **Use:** CGA 14128
diazinon metabolite: **Use:** GS-31144
diazinon metabolite (hydrolysis product): **Use:** G-27550
diazinon oxon: **Use:** diazinon oxygen analog
diazinon oxygen analog: **Use:** diazinon oxygen analog
diazoxon: **Use:** diazinon oxygen analog
Dibrom: **Use:** naled
dibromochloropropane: **Use:** dibromochloropropane
dibutalin: **Use:** butralin
dibutyl 1,2-benzenedicarboxylate: **Use:** dibutyl phthalate
dibutyl phthalate: **Use:** dibutyl phthalate
dicamba: **Use:** dicamba
dicamba methyl ester: **Use:** dicamba methyl ester
Dicarbam: **Use:** carbaryl
dichlobenil: **Use:** dichlobenil
dichlobenil metabolite: **Use:** 2,6-dichlorobenzamide
dichlofenthion: **Use:** dichlofenthion
dichlofluanid: **Use:** dichlofluanid
dichlone: **Use:** dichlone
dichlormid: **Use:** N, N-diallyl dichloroacetamide
dichlorobenzene, p-: **Use:** dichlorobenzene, p-
dichlorobenzophenone, o,p'-: **Use:** dichlorobenzophenone, o,p'-
dichlorobenzophenone, p,p'-: **Use:** dichlorobenzophenone, p,p'-
dichlorodiphenyldichloroethane: **Use:** TDE, p,p'-
dichlorofenthion: **Use:** dichlofenthion
dichlorprop: **Use:** dichlorprop
dichlorprop methyl ester: **Use:** dichlorprop methyl ester
dichlorvos: **Use:** dichlorvos
diclobutrazol: **Use:** diclobutrazol
diclofop: **Use:** diclofop
diclofop-methyl: **Use:** diclofop-methyl
diclofop-methyl metabolite: **Use:** diclofop
diclofop-methyl metabolite: **Use:** HOE-038182
diclofop-methyl metabolite: **Use:** HOE-030291
dicloran: **Use:** dicloran
Dicloran impurity: **Use:** 2,4-dichloro-6-nitrobenzenamine
dicofol breakdown product: **Use:** dichlorobenzophenone, p,p'-
dicofol breakdown product: **Use:** dichlorobenzophenone, o,p'-
dicofol, o,p'-: **Use:** dicofol, o,p'-
dicofol, p,p'-: **Use:** dicofol, p,p'-
dicophane: **Use:** DDT, p,p'-
dicrotophos: **Use:** dicrotophos
Dicuran: **Use:** chlorotoluron
dieldrin: **Use:** dieldrin
dieldrin photoproduct: **Use:** photodieldrin
diethamine: **Use:** dinitramine
diethatyl-ethyl: **Use:** diethatyl-ethyl
diethyl ((dimethoxyphosphinothioyl)thio)butanedioate: **Use:** malathion
diethyl ((dimethoxyphosphinyl)thio)butanedioate: **Use:** malathion oxygen analog
diethyl (4-methyl-1,3-dithiolan-2-ylidene)phosphoramidate: **Use:** mephosfolan
diethyl 1,3-dithiolan-2-ylidenephosphoramidate: **Use:** phosfolan
diethyl 2-(ethylthio)ethyl phosphate: **Use:** demeton-O oxygen analog
diethyl 4-nitrophenyl phosphate: **Use:** parathion oxygen analog
diethyl 6-methyl-2-(1-methylethyl)-4-pyrimidinyl phosphate: **Use:** diazinon oxygen analog
diethyl phthalate: **Use:** diethyl phthalate
diethyl pyrazinyl phosphate: **Use:** thionazin oxygen analog
diethyl(3-chloro-4-methyl-2-oxo-2H-1-benzopyran-7-yl) phosphate: **Use:** coumaphos oxygen analog
diethylhexyl phthalate: **Use:** bis(2-ethylhexyl) phthalate
difenoconazole metabolite: **Use:** CGA 189138
difenoconazole metabolite: **Use:** CGA 205374
difenoconazole metabolite: **Use:** CGA 205375
difenoxuron: **Use:** difenoxuron
Difolatan: **Use:** captafol
diisobutyl phthalate: **Use:** diisobutyl phthalate
diisohexyl phthalate: **Use:** diisohexyl phthalate
diisooctyl 1,2-benzenedicarboxylate: **Use:** diisooctyl phthalate
diisooctyl phthalate: **Use:** diisooctyl phthalate
Dilan: **Use:** Dilan
Dimecron: **Use:** phosphamidon
dimephenthoate: **Use:** phenthoate
dimethachlor: **Use:** dimethachlor
dimethametryn: **Use:** dimethametryn
dimethazone: **Use:** clomazone
dimethenamid: **Use:** dimethenamid
dimethipin: **Use:** dimethipin
dimethoate: **Use:** dimethoate
dimethoate oxygen analog: **Use:** omethoate
dimethomorph (prop): **Use:** dimethomorph (prop)
dimethoxon: **Use:** omethoate
dimethyl (1,2-phenylenebis(iminocarbonothioyl))bis(carbamate): **Use:** thiophanate-methyl
dimethyl (1,2-phenylenebis(iminocarbonyl))bis(carbamate): **Use:** allophanate
dimethyl (2,2,2-trichloro-1-hydroxyethyl)phosphonate: **Use:** trichlorfon
dimethyl 1-methyl-3-(methylamino)-3-oxo-1-propenyl phosphate: **Use:** monocrotophos
dimethyl 1-methyl-N,N-(dimethylamino)-3-oxo-1-propenyl phosphate, (E)-: **Use:** dicrotophos
dimethyl 2,3,5,6-tetrachloro-1,4-benzenedicarboxylate: **Use:** DCPA

S-ethyl bis(2-methylpropyl)carbamothioate: **Use:** butylate
S-ethyl cyclohexylethylcarbamothioate: **Use:** cycloate
S-ethyl cyclohexylethylthiocarbamate: **Use:** cycloate
S-ethyl diethylcarbamothioate: **Use:** ethiolate
S-ethyl dipropylcarbamothioate: **Use:** EPTC
S-ethyl hexahydro-1H-azepine-1-carbothioate: **Use:** molinate
S-propyl butylethylcarbamothioate: **Use:** pebulate
S-propyl dipropylcarbamothioate: **Use:** vernolate
Safrotin: **Use:** propetamphos
Salithion: **Use:** dioxabenzofos
SAN 371F: **Use:** oxadixyl
SAN 619 F: **Use:** cyproconazole
SAN-582H: **Use:** dimethenamid
Sandofan: **Use:** oxadixyl
Santoquin: **Use:** ethoxyquin
Satisfar: **Use:** etrimfos
Saturn: **Use:** thiobencarb
Savey: **Use:** hexythiazox
schradan: **Use:** schradan
Scout: **Use:** tralomethrin
SD 11831: **Use:** nitralin
SD 15418: **Use:** cyanazine
SD 43775: **Use:** fenvalerate
SD 8447: **Use:** Gardona
Sector: **Use:** butralin
Selecron: **Use:** profenofos
Sencor: **Use:** metribuzin
Sencor, deaminated diketo metabolite: **Use:** metribuzin, deaminated diketo metabolite
Sencor, deaminated metabolite: **Use:** metribuzin, deaminated metabolite
Sencor, diketo metabolite: **Use:** metribuzin, diketo metabolite
SES: **Use:** disul-Na
sesone: **Use:** disul-Na
sethoxydim: **Use:** sethoxydim
sethoxydim sulfoxide: **Use:** sethoxydim sulfoxide
Sevin: **Use:** carbaryl
Sicarol: **Use:** pyracarbolid
siduron: **Use:** siduron
Silosan: **Use:** pirimiphos-methyl
silvex: **Use:** silvex
silvex methyl ester: **Use:** silvex methyl ester
simazine: **Use:** simazine
simazine metabolite: **Use:** desdiethyl simazine
simazine metabolite: **Use:** desethyl simazine
simetryn: **Use:** simetryn
Sinbar: **Use:** terbacil
Sinox: **Use:** DNOC
Sipcam: **Use:** hexythiazox
SN 100309: **Use:** pyrimethanil
sodium 2,2-dimethyl-4,6-dioxo-5-(1-((2-propenyloxy)imino)=butyl)cyclohexanecarboxylate ion(1-): **Use:** alloxydim-sodium
sodium 2-(2,4-dichlorophenoxy)ethanyl hydrogen sulfate: **Use:** disul-Na
sodium 2-chloro-6-((4,6-dimethoxy-2-pyrimidinyl)thio)benzoate: **Use:** pyrithiobac-sodium
sodium salt of ((2-(ethoxymethyl) (2-ethyl-6-methylphenyl)=amino)-2-oxoethyl)sulfinyl)acetic acid: **Use:** CP 97290
sodium salt of ((ethoxymethyl)(2-(1-hydroxyethyl)-6-methylphenyl)amino)oxoacetic acid: **Use:** CP 108669
sodium salt of ((ethoxymethyl)(2-ethyl-6-(hydroxymethyl)=phenyl)amino)oxoacetic acid: **Use:** CP 106077
sodium salt of (ethoxymethyl) (2-ethyl-6-methylphenyl)=amino)oxoacetic acid: **Use:** CP 95200
sodium salt of 2-((2-ethyl-6-methylphenyl) (ethoxymethyl)amino)-2-oxoethanesulfonic acid: **Use:** CP 92429
sodium salt of 2-((ethoxymethyl)(2-(1-hydroxyethyl)-6-methyl=phenyl)amino)-2-oxoethanesulfonic acid: **Use:** CP 106070
Sofac: **Use:** cyfluthrin
Solgard: **Use:** pirimiphos-ethyl
Solicam: **Use:** norflurazon
Sonalan: **Use:** ethalfluralin
Sonar X: **Use:** fluridone
Sonax: **Use:** etaconazole
Spectracide: **Use:** diazinon
Spike: **Use:** tebuthiuron
Splendor: **Use:** tralkoxydim
Sportak: **Use:** prochloraz
Spot Kleen: **Use:** thiophanate-methyl
Stalker: **Use:** chlorfenapyr (prop)
Stam F-34: **Use:** propanil
Standak: **Use:** aldoxycarb
Staple: **Use:** pyrithiobac-sodium
stirofos: **Use:** Gardona
Stomp: **Use:** pendimethalin
Stop-scald: **Use:** ethoxyquin
Strobane: **Use:** Strobane
Subdue: **Use:** metalaxyl
Suffix: **Use:** benzoylprop-ethyl
sulfallate: **Use:** sulfallate
sulfanilamide: **Use:** sulfanilamide
sulfentrazone metabolite: **Use:** 3-desmethyl sulfentrazone
sulfocarb: **Use:** aldoxycarb
sulfotep: **Use:** sulfotep
Sulphenone: **Use:** Sulphenone
sulprofos: **Use:** sulprofos
sulprofos oxygen analog sulfone: **Use:** sulprofos oxygen analog sulfone
sulprofos sulfone: **Use:** sulprofos sulfone
sulprofos sulfoxide: **Use:** sulprofos sulfoxide
Sumi-alpha: **Use:** esfenvalerate
Sumicidin: **Use:** fenvalerate
Sumilex: **Use:** procymidone
Sumisclex: **Use:** procymidone
Sumithion: **Use:** fenitrothion
Sumithrin: **Use:** phenothrin
Summit: **Use:** triadimenol
Suncide: **Use:** propoxur
Super-Suffix: **Use:** flamprop-methyl
Supermethrin: **Use:** cypermethrin
Supracide: **Use:** methidathion
Surecide: **Use:** cyanofenphos
Surflan: **Use:** oryzalin
Sutan: **Use:** butylate
systam: **Use:** schradan
Systhane: **Use:** myclobutanil
Systox thiol: **Use:** demeton-S
Systox thiol sulfone: **Use:** demeton-S sulfone
Systox thiono: **Use:** demeton-O
Systox thiono oxygen analog: **Use:** demeton-O oxygen analog
Systox thiono sulfone: **Use:** demeton-O sulfone
Talstar: **Use:** bifenthrin

Tomahawk: **Use:** pirimiphos-methyl
Top Hand: **Use:** acetochlor
Top Notch: **Use:** acetochlor
Topas: **Use:** penconazole
Topaz: **Use:** penconazole
Topaze: **Use:** penconazole
Topsin M: **Use:** thiophanate-methyl
Torak: **Use:** dialifor
Tordon: **Use:** picloram
Toxakil: **Use:** toxaphene
toxaphene: **Use:** toxaphene
tralkoxydim: **Use:** tralkoxydim
tralomethrin: **Use:** tralomethrin
tralomethrin metabolite: **Use:** deltamethrin
tralomethrin metabolite: **Use:** deltamethrin, trans-
Tramat: **Use:** ethofumesate
Treflan: **Use:** trifluralin
tri(beta-chloroethyl) phosphate: **Use:** tris(beta-chloroethyl) phosphate
tri(N-butyl) phosphate: **Use:** tributyl phosphate
tri-allate: **Use:** tri-allate
triadimefon: **Use:** triadimefon
triadimefon metabolite: **Use:** KWG 1342
triadimefon metabolite: **Use:** triadimenol
triadimefon metabolite: **Use:** KWG 1323
triadimenol: **Use:** triadimenol
triadimenol metabolite: **Use:** KWG 1342
triasulfuron metabolite: **Use:** CGA 150829
triasulfuron metabolite: **Use:** CGA 195654
triasulfuron metabolite: **Use:** CGA 161149
triazamate: **Use:** triazamate
triazamate (prop): **Use:** triazamate
Triazid: **Use:** amitraz
triazophos: **Use:** triazophos
tribufos: **Use:** tribufos
Tribunil: **Use:** methabenzthiazuron
tributyl phosphate: **Use:** tributyl phosphate
tributyl phosphorotrithioate: **Use:** merphos
trichlorfon: **Use:** trichlorfon
trichlorobenzyl chloride metabolite: **Use:** 2,3,6-TBA
trichloronat: **Use:** trichloronat
trichlorophenyl ethanol: **Use:** 2,4,5-trichloro-alpha-methylbenzenemethanol
triclopyr: **Use:** triclopyr
triclopyr metabolite: **Use:** 3,5,6-trichloro-2-pyridinol
triclopyr metabolite: **Use:** 2-methoxy-3,5,6-trichloropyridine
triclopyr methyl ester: **Use:** triclopyr methyl ester
tricyclazole: **Use:** tricyclazole
tricyclazone: **Use:** tricyclazole
tridiphane: **Use:** tridiphane
triflumizole: **Use:** triflumizole
trifluralin: **Use:** trifluralin
triflusulfuron methyl ester: **Use:** triflusulfuron methyl ester
triflusulfuron-methyl: **Use:** triflusulfuron methyl ester
Trifmine: **Use:** triflumizole
trimethacarb metabolite: **Use:** 4-hydroxymethyl-3,5-dimethylphenyl methylcarbamate
trimethacarb metabolite : **Use:** 3-hydroxymethyl-4,5-dimethylphenyl methylcarbamate
trimethacarb metabolite : **Use:** 3-hydroxymethyl-2,5-dimethylphenyl methylcarbamate
Trimidal: **Use:** nuarimol
Triminol: **Use:** nuarimol
triphenyl phosphate: **Use:** triphenyl phosphate
tris(2-chloroethyl) phosphate: **Use:** tris(beta-chloroethyl) phosphate
tris(beta-chloroethyl) phosphate: **Use:** tris(beta-chloroethyl) phosphate
tris(chloropropyl) phosphate: **Use:** tris(chloropropyl) phosphate
Tritex: **Use:** alloxydim-sodium
Trithion: **Use:** carbophenothion
Trithion oxygen analog: **Use:** carbophenothion oxygen analog
Trithion oxygen analog sulfone: **Use:** carbophenothion oxygen analog sulfone
Trithion oxygen analog sulfoxide: **Use:** carbophenothion oxygen analog sulfoxide
Trithion sulfone: **Use:** carbophenothion sulfone
Trithion sulfoxide: **Use:** carbophenothion sulfoxide
Tritisan: **Use:** quintozene
Triumph: **Use:** isazofos
Tropotox: **Use:** MCPB
Truban: **Use:** etridiazole
Trysben: **Use:** 2,3,6-TBA
Tsumacide: **Use:** metolcarb
Tunic: **Use:** methazole
Tupersan: **Use:** siduron
Tycor: **Use:** Tycor
UC-21865: **Use:** aldoxycarb
UC21149: **Use:** aldicarb
Ultracide: **Use:** methidathion
Unden: **Use:** propoxur
Uniroyal D-014: **Use:** propargite
Usb 3584: **Use:** dinitramine
Valexon: **Use:** phoxim
vamidothion metabolite: **Use:** vamidothion sulfone
Van Dyk 264: **Use:** MGK 264
Vangard: **Use:** etaconazole
Vapona: **Use:** dichlorvos
Vapotone: **Use:** TEPP
VC-13: **Use:** dichlofenthion
Vegadex: **Use:** sulfallate
Velpar: **Use:** hexazinone
Verdict: **Use:** haloxyfop methyl ester
Vernam: **Use:** vernolate
vernolate: **Use:** vernolate
Vigil: **Use:** diclobutrazol
vinclozolin: **Use:** vinclozolin
vinclozolin metabolite B: **Use:** vinclozolin metabolite B
vinclozolin metabolite B, methylated: **Use:** vinclozolin
vinclozolin metabolite D: **Use:** 3,5-dichloroaniline
vinclozolin metabolite E: **Use:** vinclozolin metabolite E
vinclozolin metabolite S: **Use:** vinclozolin metabolite S
vinclozoline metabolite F: **Use:** vinclozolin metabolite F
Viran: **Use:** parathion
Vitavax: **Use:** carboxin
Volaton: **Use:** phoxim
Vondcaptan: **Use:** captan
Voronit: **Use:** fuberidazole
Vydate: **Use:** oxamyl
WAK3745: **Use:** NTN35884
Wakil: **Use:** oxadixyl
Warbex: **Use:** famphur
Waylay: **Use:** napropamide
Weed B Gon: **Use:** 2,4-D

Weedone: **Use:** 2,4,5-T
Whip: **Use:** fenoxaprop ethyl ester
Wipeout: **Use:** hydramethylnon
WL 41706: **Use:** fenpropathrin
WL 85871: **Use:** alpha-cypermethrin
XMC: **Use:** XMC
XRD 498: **Use:** flumetsulam
Xymiazole: **Use:** cymiazole
Zeldox: **Use:** hexythiazox
zeta-cypermethrin: **Use:** cypermethrin
Zinophos: **Use:** thionazin
Zobar: **Use:** 2,3,6-TBA
Zolone: **Use:** phosalone
Zorial: **Use:** norflurazon

Index to CAS Registry Numbers for Chemicals in PAM I

n/a = number not available

Index to Subjects Covered in PAM I

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APPENDIX I: PESTDATA

Caution: Use this table only as a quick reference for tentative identification of residues found in samples analyzed by the most commonly used PAM I multiresidue methods. Always compare the residue to a standard chromatographed in your own gas chromatograph. Apply appropriate confirmatory tests to verify tentative identification. Note that PESTDATA cannot and does not contain all details; consult PAM I tables that accompany each method for more definitive information about the behavior of the compound through the steps of the methods.

NOTATIONS AND ABBREVIATIONS USED IN PESTDATA

(In all categories a hyphen indicates absence of data)

Name

Preferred name for each chemical. "*" indicates chemicals with multiple GLC peaks. Chlordane, Strobane, toxaphene and all Aroclors are listed only in the table ordered by name and do not appear in the tables ordered by relative retention times.

Molecular Formula

Numbers are not subscripted. Averages are used for multicomponent chemicals.

RRT/c

Columns list retention times (relative to chlorpyrifos) on GLC column indicated. Conditions under which these data were gathered are described in these Section 302 DG modules:

GLC Column	Section 302 DG modules
OV-101	DG1-DG5
OV-17	DG13-DG17
OV-225	DG18, DG19

Note that headers in these tables refer to GLC columns by the names used for packed columns, despite DG1-DG19's descriptions of wide bore capillary column systems, because so many of these rrts were developed using packed columns. Rrt data for equivalent packed and capillary columns are expected to be essentially identical and are combined in PESTDATA.

Responses

Data specify column and detector used. Numbers refer to weight (ng) that causes detector response of approximately 50% full scale deflection (FSD) on the recording device. Response values collected when the detector was combined with a wide bore capillary column include the notation "(WB)." Codes refer to detectors and operating conditions described in these Section 302 DG modules, except that all response values are based on detector sensitivity of 50% FSD to 1.5 ng chlorpyrifos:

Code	Detector	Section 302 DG modules
TR	tritium electron capture	none - obsolete detector
TI	thermionic (KCl)	none - obsolete detector
FP	flame photometric, phosphorus	DG2, DG14, DG19
FS	flame photometric, sulfur	DG15
NI	^{63}Ni electron capture	DG1, DG13, DG18
NP	nitrogen/phosphorus	DG5, DG17
HX	electroconductivity (halogen mode)	DG3, DG16
HN	electroconductivity (nitrogen mode)	DG4
MC	microcoulometric	none - obsolete detector

NOTES: Response values are approximate and can vary dramatically on different chromatographs. Most response values represent rounded-off or averaged values; some were collected under conditions different from those suggested in references.

Recoveries

Data on the recovery of the compound through several PAM I methods are listed in columns with the following headings. See the appropriate PAM I table(s) for more details, such as partial recoveries through Florisil.

Heading	Common Name	PAM I Section	PAM I Table
302	Luke (Los Angeles)	302 E1-E3, no cleanup	302-a
303	Mills, Onley, Gaither	303 E1-E5 + C1 or C2	303-a
304	Mills fatty food	304 E1-E5 + C1-C4	304-a
Ethers	Florisil elution system	303 C1, 304 C1 and C3	303-a, 304-a
CH_2Cl_2	alternative Florisil elution	303 C2, 304 C2 and C4	303-a, 304-a

Recovery codes have the following meanings: C: complete (>80%); P: partial (50-80%); S: small (<50%); V: variable; R: recovered but no quantitative information available; NR: not recovered.

Transmittal No. 2000-1 (10/1999)
Form FDA 2905a (6/92)

Appendix I: PESTDATA Chemicals in Order by Chemical Name

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
1,1'-(2,2-dichloroethylidene)bis(2-methoxybenzene)	C16H16Cl2O2	2.9	-	3.7	-	R	-	-	-
	Responses:								
1,2,3,4-tetrachlorobenzene	C6H2Cl4	-	-	0.09	-	-	-	-	-
	Responses: OV-17: NI0.2								
1,2,3,5-tetrachlorobenzene	C6H2Cl4	-	-	0.07	-	P #	-	6	1
	Responses: OV-17: NI0.1								
1,2,3-trichlorobenzene	C6H3Cl3	0.08	-	-	-	C	P	6	1
	Responses: OV-101: TR2								
1,2,4,5-tetrachloro-3-(methylthio)=benzene	C7H4Cl4S	0.49	0.35	0.48	R	C	-	6	1
	Responses: OV-101: NI0.3 OV-17: HX(WB)0.3 OV-225: NI0.3								
1,2,4,5-tetrachlorobenzene	C6H2Cl4	-	-	0.07	-	-	-	-	-
	Responses: OV-17: NI0.2								
1,2,4-triazole	C2H3N3	0.2	-	0.27	V	NR	NR	6-15-50	1-2-3
	Responses: DEGS: NP3								
1-hydroxychlordene	C10H6Cl6O	0.99	1.63	1.07	-	R	-	15	-
	Responses: OV-101: NI(WB)7 OV-17: NI1 OV-225: NI1								
1-methyl cyromazine	C7H13N6	-	-	0.72	-	-	-	-	-
	Responses: OV-17: NP1000								
10,10-dihydromirex	C10H2Cl10	2.67	-	-	-	C	-	6	-
	Responses: OV-101: NI7								
10-monohydromirex	C10HCl11	4.26	-	-	-	C	-	6	-
	Responses: OV-101: NI7								
2,3,5,6-tetrachloroanisidine	C7H5Cl4NO	0.59	0.73	0.66	-	C	-	6	2
	Responses: OV-101: NI0.5 OV-17: HX(WB)0.6 OV-225: NI0.5								
2,3,5,6-tetrachloroanisole	C7H4Cl4O	0.24	0.15	0.22	-	C	-	6	1
	Responses: OV-101: NI0.2 OV-17: HX(WB)0.3 OV-225: NI0.2								
2,3,5,6-tetrachloronitroanisole	C7H3Cl4NO3	0.56	0.63	0.56	-	C	-	6	1+2
	Responses: OV-101: NI0.4 OV-17: HX(WB)0.5 OV-225: NI0.4								
2,3,5,6-tetrafluoro-4-hydroxymethyl=benzoic acid	C8H4OF4	-	-	-	-	-	-	-	-
	Responses:								

* Multipeak chemical.

\# Recovery may vary with choice of Florisil elution system; see Tables 303-a, 304-a.

Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
2,3,5-trimethacarb	C11H15NO2	0.35	0.6	0.38	C	S #	NR	50	1-2-3
	Responses: OV-101: NP8 OV-17: NP4 OV-225: NP10								
2,3-dihydro-3,3-methyl-2-oxo-5-benzofuranyl methyl sulfonate	C11H12O5S	0.68	2.89	0.93	-	-	-	-	-
	Responses: OV-101: FS25/NI4 OV-17: FS54/NI4.5 OV-225: FS63/NI10								
2,4,5-T BEP ester*	C17H23Cl3O3	0.16	0.14	-	-	-	-	-	-
		0.68	0.66	-					
		1.08	0.91	-					
		2.85	1.19	-					
		3.3	2.78	-					
		5.3	3.28	-					
		7	7.7	-					
	Responses: OV-101: TR35								
2,4,5-T butoxyethyl ester*	C14H17Cl3O4	-	2.66	-	-	-	-	-	-
		2.91	3.3	-					
	Responses: OV-101: TR4								
2,4,5-T butyl esters*	C12H13Cl3O3	-	-	1.05	-	-	-	-	-
		-	-	0.86					
	Responses:								
2,4,5-T ethylhexyl ester	C16H21Cl3O3	3.38	-	2.62	-	-	-	-	-
	Responses: OV-101: NI5								
2,4,5-T isobutyl ester	C12H13Cl3O3	0.94	-	-	-	-	-	-	-
	Responses: OV-101: TR1								
2,4,5-T isooctyl ester*	C16H21Cl3O3	-	2.69	-	-	-	-	-	-
		2.56	3.1	-					
		2.96	3.4	-					
		3.25	3.8	-					
	Responses: OV-101: TR20								
2,4,5-T isopropyl ester	C11H11Cl3O3	0.67	0.65	-	-	-	-	-	-
	Responses: OV-101: TR2								
2,4,5-T methyl ester	C9H7Cl3O3	0.49	0.63	0.47	-	-	-	-	-
	Responses: OV-101: TR1								
2,4,5-T n-butyl ester	C12H13Cl3O3	1.1	-	-	-	-	-	-	-
	Responses: OV-101: TR1								
2,4,5-T propylene glycol butyl ether esters	C15H19Cl3O4	2.37	-	-	-	-	-	-	-
	Responses:								

Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
2,4,5-trichloro-alpha-methylbenzene=methanol	C8H7OCl3	0.34	-	0.25	R	R	-	15	-
	Responses:								
2,4-D BEP ester*	C17H24Cl2O4	-	0.08	-	-	-	-	-	-
		0.69	0.74	-					
		1.66	1.18	-					
		2	1.79	-					
		3.22	2.09	-					
		4.1	5.1	-					
		10.2	13	-					
	Responses: OV-101: TR60								
2,4-D butoxyethyl ester*	C14H18Cl2O4	-	1.67	1.44	-	-	-	-	-
		1.82	2.08	1.79					
	Responses: OV-101: TR12 OV-17: NI5								
2,4-D ethyl hexyl ester*	C16H22Cl2O3	-	1.51	-	-	-	-	-	-
		2.1	1.78	1.68					
	Responses: OV-101: NI5								
2,4-D isobutyl ester	C12H14Cl2O3	0.62	0.62	0.49	-	-	-	-	-
	Responses: OV-101: TR5								
2,4-D isooctyl ester*	C16H22Cl2O3	-	-	1.48	-	-	-	-	-
		2.04	1.78	1.78					
	Responses: OV-101: TR50 OV-17: NI5								
2,4-D isopropyl ester*	C11H12Cl2O3	-	0.62	-	-	-	-	-	-
		0.42	0.74	0.33					
	Responses: OV-101: TR10								
2,4-D methyl ester	C9H8Cl2O3	0.3	0.38	0.25	-	-	-	-	-
	Responses: OV-101: TR6								
2,4-D n-butyl ester	C12H14Cl2O3	0.72	-	-	-	-	-	-	-
	Responses: OV-101: TR40								
2,4-D propylene glycol butyl ether ester*	C15H2OCl2O4	-	1.42	-	-	-	-	-	-
		1.54	3.6	-					
	Responses: OV-101: TR20								
2,4-DB methyl ester	C11H12Cl2O3	0.62	0.72	-	-	-	-	-	-
	Responses: OV-101: TR28								
2,4-dichloro-6-nitrobenzenamine	C6H4Cl2N2O2	0.3	-	-	-	R	-	15	2
	Responses: OV-101: HX2/NI0.4/NP8								

Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
2,6-dichlorobenzamide	C7H5NOCl2	0.39	1.3	0.52	C	NR	NR	6-15-50	1-2-3
Responses:									
2,8-dihydromirex	C10H2Cl10	2.41	-	-	-	C	-	6	-
Responses: OV-101: NI4									
2-chloroethyl caprate	C8H15ClO2	0.32	-	-	-	C	C	15	2
Responses: OV-101: HX2									
2-chloroethyl laurate	C14H27ClO2	0.59	-	-	-	C	C	15	2
Responses: OV-101: HX2									
2-chloroethyl linoleate	C20H35ClO2	4.1	-	-	-	V	P	15	2
Responses: OV-101: HX15									
2-chloroethyl myristate	C16H31ClO2	1.17	-	-	C	V	V	15	2
Responses: OV-101: HX4									
2-chloroethyl palmitate	C18H35ClO2	2.35	-	-	-	V	P	15	2
Responses: OV-101: HX10									
2-hydroxy-2,3-dihydro-3,3-methyl-5-benzofuranyl methyl sulfonate	C11H14O5S	1	6.6	1.46	-	-	-	-	-
Responses: OV-101: FS48/NI135 OV-17: FS88/NI96 OV-225: FS175/NI400									
2-methoxy-3,5,6-trichloropyridine	C6H4Cl3NO	0.19	0.08	0.1	C	P #	C	6+15	1+2
Responses: OV-101: NI0.5/NI(WB)0.4/NP(WB)9 OV-17: HX1.5									
3, 5, 6-trichloro-2-pyridinol methyl ester	C6H4Cl3NO	0.32	0.44	0.36	-	-	-	-	-
Responses: OV-101: NI(WB)0.6/NP(WB)5									
3,4,5-trimethacarb	C11H15NO2	0.45	0.78	0.5	C	NR	NR	50	1-2-3
Responses: OV-101: NP25 OV-17: NP10 OV-225: NP200									
3,4-dichloroaniline	C6H5Cl2N	0.2	0.32	0.16	V	S	-	15	-
Responses: OV-101: HX0.6/NI16/NP1 OV-17: NI13/NP8 OV-225: NI30									
3,4-dichlorophenylurea	C7H6Cl2N2O	0.22	0.14	0.1	-	NR	NR	6-15-50	-
Responses: OV-101: HX9/NI18/NP60 OV-17: NI4 OV-225: NI6									
3,5-dichloroaniline	C6H5Cl2N	0.18	0.27	0.14	S	S	S	6+15	1+2
Responses: OV-101: HN(WB)1/HX0.5/NI9/NI(WB)16/NP0.9/NP(WB)1 OV-17: HN(WB)0.3/HX(WB)2/NI8/NI(WB)14/NP8/NP(WB)0.4 OV-225: NI20/NI(WB)25									
3-(3,4-dichlorophenyl)-1-methoxyurea	C8H8Cl2N2O2	0.21	-	1.36	R	NR	NR	6-15-50	-
Responses: OV-101: HX9/NI25 OV-17: NP250									
3-aminophenol	C6H7NO	-	-	-	-	-	-	-	-
Responses:									

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Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
3-carboxy-5-ethoxy-1,2,4-thiadiazole	C3H2N2O3S	0.22	0.25	0.2	NR	-	-	-	-
Responses: OV-101: NI170/NP40 OV-17: NI160/NP30 OV-225: NI100 DEGS: NI200/NP40									
3-chloro-5-methyl-4-nitro-1H-pyrazole	C4H4ClN3O2	1.07	-	-	C	-	-	-	-
Responses:									
3-desmethyl sulfentrazone	C10H8Cl2F2N4O3S	3.3	-	7.5	-	NR	NR	6-15-50	1-2-3
Responses: OV-101: NI(WB)0.1/NP(WB)37 OV-17: FS(WB)1500/NI(WB)0.1/NP(WB)73									
3-hydroxycarbofuran	C12H15NO4	-	-	-	-	-	-	-	-
Responses:									
3-hydroxymethyl-2,5-dimethylphenyl methylcarbamate	C11H15NO3	0.8	-	1.03	-	NR	NR	6-15-50	1-2-3
Responses: OV-101: NP200 OV-17: NP600									
3-ketocarbofuran	C12H12NO4	0.55	1.41	0.9	S	NR	NR	6	1
Responses: OV-101: HN(WB)17/NI(WB)15/NP(WB)9 OV-17: HN(WB)15/NI(WB)80/NP(WB)30 OV-225: NI(WB)80									
3-methyl-4-nitrophenol	C7H7O3N	0.38	0.63	0.26	V	NR	NR	6-15-50	1-2-3
Responses: OV-101: NI13/NP50 OV-17: NI5/NP20 OV-225: NI250									
3-methyl-4-nitrophenol methyl ether	C8H9O3N	0.17	0.22	0.13	-	-	-	-	-
Responses: OV-101: NI3/NP7 OV-17: NI1/NP2 OV-225: NI2									
3-phenoxybenzenemethanol	C13H12O2	1.28	-	1.6	-	-	-	-	-
Responses: OV-101: NI1000 OV-17: NI1000									
3-tert-butyl-5-chloro-6-hydroxy=methyluracil	C9H13ClN2O3	1.35	2.27	2.55	-	NR	NR	6-15-50	1-2-3
Responses: OV-101: HN(WB)4/HX(WB)40/NI(WB)67/NP(WB)39 OV-17: HN(WB)13/HX(WB)99/NI(WB)35/NP(WB)290 OV-225: NI(WB)630									
4,4'-dichlorobiphenyl	C12H8Cl2	-	-	0.51	-	-	-	-	-
Responses:									
4-(2,4-dichlorophenoxy)=benzenamine	C12H9Cl2NO	1.44	-	-	-	-	-	-	-
Responses: OV-101: TR60									
4-(dichloroacetyl)-1-oxa-4-azapiro=[4.5]decane	C10H15Cl2NO2	0.5	0.69	0.48	C	P	-	50	3
Responses: OV-101: NI1.4/NP34 OV-17: NI1.1/NP1.2 OV-225: NI1.6									
4-chloro-6-methoxyindole	C9H8NOCl	0.54	-	0.66	-	R	-	15	-
Responses: OV-101: HX2.5/NI1000 OV-17: HX0									
4-chlorobenzeneamine	C6H6ClN	-	-	0.07	S	NR	NR	6-15-50	1-2-3
Responses:OV-17:NP(WB)1.5									

Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
4-chlorobenzylmethyl sulfone	C8H9ClO2S	0.41	1.91	0.66	-	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI(WB)0.4 OV-17: NI(WB)0.8 OV-225: NI(WB)2								
4-chlorobenzylmethyl sulfoxide	C8H9ClOS	0.39	1.16	0.54	-	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI(WB)20 OV-17: NI(WB)16 OV-225: NI(WB)55								
4-chlorobiphenyl	C12H9Cl	-	-	0.2	-	-	-	-	-
	Responses:								
4-chlorophenoxyaniline*	C12H10ClNO	0.87	-	1.07	S	-	-	-	-
		1.28	-	1.31					
	Responses: OV-101: HX7/NI1100								
4-chlorophenylurea	C7H7ClN2O	0.54	-	1.07	NR	NR	NR	6-15-50	1-2-3
	Responses:OV-101:NI(WB)15 OV-17:NI(WB)40								
4-hydroxymethyl-3,5-dimethylphenyl methylcarbamate*	C11H15NO3	0.18	-	0.22	-	NR	NR	15-50	1-2-3
		0.27	-	0.31					
	Responses: OV-101: NI200 OV-17: NI150								
6-chloro-2,3-dihydro-3,3,7-methyl-5H-oxazolo(3,2-a)pyrimidin-5-one	C9H13ClN2O2	0.43	1.34	0.6	-	NR	NR	6-15-50	1-2-3
	Responses: OV-101: HN(WB)0.4/NI(WB)26/NP(WB)3 OV-17: HN(WB)0.4/HX(WB)4/NI(WB)36/NP(WB)2 OV-225: NI(WB)51								
6-chloro-2,3-dihydro-7-hydroxy=methyl-3,3-methyl-5H-oxazolo=(3,2-a)pyrimidin-5-one	C9H13ClN2O3	0.86	-	1.55	-	NR	NR	6-15-50	1-2-3
	Responses: OV-101: HN(WB)4/HX(WB)11/NI(WB)28/NP(WB)17 OV-17: HN(WB)3/HX(WB)17/NI(WB)19/NP(WB)12								
6-chloronicotinic acid*	C6H4NO2Cl	-	-	-	-	NR	NR	6-15-50	1-2-3
		-	-	-					
	Responses: DEGS: HX40/NI11/NP66								
8-monohydromirex	C10HCl11	3.74	-	-	-	C	-	6	-
	Responses: OV-101: NI5								
acephate	C4H10NO3PS	0.15	0.64	0.19	C	-	-	-	-
	Responses: OV-101: FP(WB)0.9/NP3 OV-17: FP(WB)0.6 OV-225: FP5								
acetochlor	C14H20NO2Cl	0.75	0.88	0.67	C	C #	P	50	3
	Responses: OV-101: HX5/NI9/NP5 OV-17: NI5 OV-225: NI5								
acifluorfen	C14H7ClF3NO3	1.05	1.47	0.88	-	NR	NR	6-15-50	1-2-3
	Responses: OV-101: HN(WB)170/HX(WB)980/NI(WB)40/NP(WB)270 OV-17: HN(WB)48/HX(WB)390/NI(WB)27/NP(WB)1000 OV-225: NI(WB)300								
acrinathrin	C26H21F6NO5	10.4	12.8	8.9	V	V	V #	15	2
	Responses: OV-101: NI15/NP125 OV-17: NI25/NP100 OV-225: NI40								

Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
alachlor	C14H2OClNO2	0.8	1	0.72	C	C	C #	50	3
	Responses: OV-101: NI(WB)7 OV-17: NI6 OV-225: NI6								
aldrin	C12H8Cl6	1.05	0.58	0.76	C	C	C	6	1
	Responses: OV-101: TR0.8 OV-17: NI1								
allethrin	C19H26O3	1.36	1.22	-	-	C	C #	50	3
	Responses: OV-101: NI8								
allidochlor	C8H12ClNO	0.09	-	-	C	NR	-	6-15	1-2-3
	Responses: OV-101: TR5								
alpha-cypermethrin	C22H19Cl2O3N	14	-	-	C	C	-	-	2
	Responses: OV-101: HX9/NI22								
ametryn	C9H17N5S	0.77	1.1	-	C	-	-	-	-
	Responses:								
aminocarb	C11H16N2O2	0.56	-	-	C	-	-	-	-
	Responses: OV-101: NP10								
amitraz	C19H23N3	-	-	-	S	-	-	-	-
	Responses:								
anilazine	C9H5Cl3N4	1.24	1.88	1.47	V	S	P	15+50	2+3
	Responses: OV-101: HX(WB)8/NI4 OV-17: NP20								
aramite*	C15H23ClO4S	2	2.77	-	C	P	NR	15	-
		2.14	3.05	-					
	Responses: OV-101: FP600/TR10000								
Aroclor 1016*	CHCl (mix)	0.2	0.24	-	-	C	C	6	1
		0.3	0.3	-					
		0.39	0.4	-					
		0.44	0.46	-					
		0.52	0.51	-					
		0.59	0.54	-					
		0.68	0.61	-					
		0.73	0.68	-					
		0.87	0.85	-					
		1	0.9	-					
		1.07	0.98	-					
		1.3	1.09	-					
	Responses:								

Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
Aroclor 1221*	C12H8.8Cl1.2	0.21	-	-	-	C	C	6	1
		0.27	-	-					
		0.32	-	-					
		0.37	-	-					
		0.4	-	-					
		0.53	-	-					
		0.6	0.15	-					
		0.65	0.21	-					
		0.7	0.24	-					
		0.77	0.3	-					
		0.9	0.4	-					
		1.01	0.43	-					
		1.3	0.46	-					
		1.45	0.5	-					
		1.55	0.54	-					
		1.8	0.61	-					
		1.9	0.68	-					
		2.12	0.92	-					
		2.26	1.04	-					
		2.7	1.16	-					
		3.16	1.24	-					
	Responses: OV-101: TR40								
Aroclor 1242*	C12H7Cl3	0.4	0.24	-	-	C	C	6	1
		0.52	0.3	-					
		0.58	0.4	-					
		0.68	0.46	-					
		0.73	0.54	-					
		0.88	0.61	-					
		0.98	0.68	-					
		1.05	0.85	-					
		1.24	0.9	-					
		1.42	0.98	-					
		1.52	1.1	-					
		1.77	1.36	-					
		1.87	1.59	-					
		2.24	1.75	-					
		2.61	2.01	-					
	Responses: OV-101: TR50								

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Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
Aroclor 1248*	C12H6.1Cl3.9	0.52	0.4	-	-	C	C	6	1
		0.58	0.47	-					
		0.68	0.54	-					
		0.82	0.61	-					
		0.87	0.68	-					
		0.98	0.85	-					
		1.05	0.9	-					
		1.25	0.98	-					
		1.42	1.1	-					
		1.52	1.18	-					
		1.77	1.37	-					
		1.88	1.6	-					
		2.24	1.75	-					
		2.59	2.01	-					
		3.1	2.72	-					
	Responses: OV-101: TR50								
Aroclor 1254*	C12H5Cl5	-	-	0.35	-	C	C	6	1
		0.89	0.68	0.48					
		1	0.85	0.63					
		1.07	0.9	0.81					
		1.3	0.99	0.97					
		1.55	1.1	1.3					
		1.82	1.17	1.43					
		1.92	1.39	1.84					
		2.24	1.48	1.98					
		2.68	1.6	2.26					
		3.14	1.75	2.55					
		3.7	2.03	2.91					
		4.2	2.46	3.3					
		4.4	2.79	4.3					
		5	3.8	4.8					
		5.9	4.3	5.2					
	Responses: OV-101: TR30								
Aroclor 1260*	C12H3.7Cl6.3	-	1.09	-	-	C	C	6	1
		1.31	1.17	-					
		1.53	1.61	-					
		1.9	1.81	-					
		2.11	2.06	-					
		2.25	2.18	-					
		2.68	2.45	-					
		2.9	2.76	-					

Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
Aroclor 1260* (cont'd)		3.2	3.12	-					
		3.6	3.48	-					
		4.2	3.7	-					
		5	4.3	-					
		5.9	5.2	-					
		6.6	5.8	-					
		8	7.2	-					
		9.3	8.8	-					
	Responses: OV-101: TR20								
Aroclor 1262*	C12H3.3Cl6.7	1.29	-	-	-	C	C	6	1
		1.53	-	-					
		1.89	-	-					
		2.11	-	-					
		2.26	-	-					
		2.66	-	-					
		2.88	-	-					
		3.12	-	-					
		3.6	-	-					
		4.2	-	-					
		5	-	-					
		5.9	-	-					
		6.5	-	-					
		6.7	-	-					
		8	-	-					
		9.3	-	-					
	Responses: OV-101: TR20								
Aroclor 1268*	C12H1Cl9	3.8	-	-	-	C	-	6	-
		4.7	-	-					
		5.4	-	-					
		7.3	-	-					
		8.7	-	-					
		10	-	-					
		13	-	-					
		16.2	-	-					
	Responses: OV-101: NI40								
Aroclor 4465*	CHCl (MIX)	2.08	-	-	-	C	C	6	1
		2.22	-	-					
		2.67	-	-					
		2.88	-	-					
		3.11	-	-					

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Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
Aroclor 4465* (cont'd)		3.6	-	-					
		4.2	-	-					
		4.5	-	-					
		5	-	-					
		5.4	-	-					
		5.9	-	-					
		6.5	-	-					
		6.6	-	-					
		8	-	-					
		9.3	-	-					
		12.1	-	-					
	Responses: OV-101: TR40								
atrazine	C8H14ClN5	0.43	0.74	0.44	C	S #	NR	50	1-2-3
	Responses: OV-101: TI58/TR200 OV-17: NI20								
azafenidin	C15H13Cl2N3O2	14	-	-	V	-	-	-	-
	Responses: OV-101:NI(WB)90								
azinphos-ethyl	C12H16N3O3PS2	6.9	-	14.8	C	P	S	50	3
	Responses: OV-101: TI58/TR200 OV-17: FP(WB)26/NI20								
azinphos-methyl	C10H12N3O3PS2	5.2	-	11.8	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: TI30/TR50								
azinphos-methyl oxygen analog	C10H12N3O4PS	3.7	-	10.1	C	-	-	-	-
	Responses: OV-101: FP20 OV-17: FP(WB)42								
benazolin methyl ester	C9H6O3SNCl	0.99	-	-	-	-	-	-	-
	Responses: OV-101: NI1								
bendiocarb	C11H13NO4	0.32	-	-	C	-	-	-	-
	Responses: DEGS: NP13								
benfluralin	C13H16F3N3O4	0.37	0.28	0.18	C	C	C	6	2
	Responses: OV-101: HX(WB)1.5/NI(WB)2 OV-17: HX(WB)1 OV-225: NI2								
benodanil	C13H10INO	2.43	-	4.5	C	-	-	-	-
	Responses: OV-101: NP60								
benoxacor	C11H11Cl2NO2	0.64	1.06	0.7	C	P	C	15+50	2+3
	Responses: OV-101: NI1/NP6 OV-17: NI1/NP7 OV-225: NI2								
bensulide	C14H24NO4PS3	9.5	-	20.2	C	P	C	50	3
	Responses: OV-101: FP100/NI(WB)9/TI190 OV-17: FP100								

Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
benzoylprop-ethyl	C18H17Cl2NO3	4.3	8.4	6	P	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI(WB)3 OV-17: NI8 OV-225: NI6								
BHC, alpha-	C6H6Cl6	0.4	0.48	0.35	C	C	C	6	1
	Responses: OV-101: TR0.4 OV-17: NI0.3								
BHC, beta-	C6H6Cl6	0.43	1.62	0.56	C	C	C	6	1
	Responses: OV-101: TR2 OV-17: NI1								
BHC, delta-	C6H6Cl6	0.5	1.71	0.67	C	C	C	6+15	1
	Responses: OV-101: HX0.5/TR0.4 OV-17: NI0.5								
bifenox	C12H9Cl2NO5	5	14.9	8.8	C	C	P	15+50	2+3
	Responses: OV-101: HX16/NI4								
bifenthrin	C23H22ClF3O2	4.9	3.8	4.5	V	C	-	6+15	2
	Responses: OV-101: NI8 OV-17: HX5/HX(WB)20								
binapacryl	C15H18N2O6	2.19	4.2	2.38	C	P	P	15	-
	Responses: OV-101: NI(WB)1 OV-17: NI22/NP(WB)100								
bis(2-ethylhexyl) phthalate	C24H38O4	6.4	4.5	6.1	-	C	C	15+50	-
	Responses: OV-101: NI(V)200								
bis(trichloromethyl)disulfide	C2Cl6S2	0.19	-	-	-	R	-	6	-
	Responses:								
bitertanol*	C20H23N3O2	9.4	-	11.8	C	-	-	-	-
		9.7	-	12.5					
	Responses: OV-17: NP(WB)200								
bromacil	C9H13BrN2O2	0.8	4.8	1.36	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: HN(WB)2/NI(WB)2/NP(WB)17 OV-17: HN(WB)1/HX(WB)8/NI(WB)6/NP(WB)5 OV-225: NI(WB)12								
bromacil methyl ether	C10H16BrN2O2	0.8	2.1	-	-	-	-	-	-
	Responses: OV-101: HN(WB)1.2/HX(WB)90/NI(WB)1.5/NP(WB)10 OV-225: NI(WB)3.8								
bromofenoxim methyl ether	C14H9Br2O6N3	0.3	-	-	-	-	-	-	-
	Responses: OV-101: HN(WB)6/NI1								
bromophos	C8H8BrCl2O3PS	1.11	1.29	1.16	C	C	C	6	-
	Responses: OV-101: FP3/NI(WB)1/TI3 OV-17: FP3/NI2 OV-225: NI6								
bromophos-ethyl	C10H12BrCl2O3PS	1.51	1.42	1.45	C	C	P	6	-
	Responses: OV-101: FP3/NI3/TI4 OV-17: FP(WB)0.3								
bromopropylate	C17H16Br2O3	4.4	6.5	-	C	C #	C #	15+50	1-2-3
	Responses: OV-101: TR12								

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Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
bromoxynil butyrate	C11H9Br2NO2	0.78	-	-	-	V	-	15+50	2
	Responses: OV-101: NI0.5/NP7.5								
bromoxynil methyl ether	C8H5BR2ON	0.3	-	-	-	-	-	-	-
	Responses: OV-101: NI0.4								
bromoxynil octanoate	C15H17Br2NO2	3.14	-	-	-	V #	-	15+50	2
	Responses: OV-101: NI4/NP40								
BTS 27271-HCl	C10H14N2•HCl	-	-	-	-	-	-	-	-
	Responses:								
BTS 27919	C9H11NO	-	-	-	C	-	-	-	-
	Responses:								
bufencarb*	C13H19NO2	-	-	-	-	-	-	-	-
		-	-	-					
		-	-	-					
		-	-	-					
		-	-	-					
	Responses:								
Bulan	C16H15Cl2NO2	3.06	7.5	4.4	C	P	P	15	2
	Responses: OV-101: NI(WB)1 OV-17: NI5 OV-225: NI6								
bupirimate	C13H24N4SO3	2	3.7	2.6	C	-	-	-	-
	Responses: OV-101: FS(WB)20/NI(WB)8 OV-17: NP(WB)300								
butachlor	C17H26ClNO2	1.73	1.83	1.46	C	C	-	50	-
	Responses: OV-101: HX9 OV-17: HX9 OV-225: NI14								
butralin	C14H21N3O4	1.15	1.22	0.93	V	C	-	6+15+50	-
	Responses: OV-101: NI7/NP3 OV-17: NI6/NP15 OV-225: NI8								
butyl benzyl phthalate	C19H20O4	3.06	5.1	4.5	-	C	P	15+50	-
	Responses: OV-101: NI35								
butylate	C11H23NOS	0.22	-	-	-	-	-	-	-
	Responses:								
butylisodecyl phthalate	C22H34O4	-	-	0.82	-	-	-	-	-
	Responses:								
cadusafos	C10H23O2PS2	0.37	0.27	0.29	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: FP(WB)0.5 OV-17: FP(WB)0.4/NI(WB)12/NP(WB)0.5 OV-225: FP(WB)1								
captafol	C10H9Cl4NO2S	3.11	-	5.4	C	P	-	50	3
	Responses: OV-101: NI3 OV-17: NI5								

Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
captan	C9H8Cl3NO2S	1.2	3.49	1.85	C	P	C	50	3
	Responses: OV-101: TR2 OV-17: NI2								
carbaryl	C12H11NO2	0.75	-	1.05	C	-	-	-	-
	Responses: OV-101: NP60								
carbetamide	C12H16N2O3	0.96	-	1.32	-	-	-	-	-
	Responses:								
carbofuran	C12H15NO3	0.39	-	-	C	-	-	-	-
	Responses:								
carbofuran-3-keto-7-phenol	C10H10O3	-	0.24	-	-	-	-	-	-
	Responses:								
carbofuran-7-phenol-DNP ether	C16H14N2O6	-	18.1	-	-	-	-	-	-
	Responses:								
carbophenothion	C11H16ClO2PS3	2.94	4.2	3.7	C	C	P	6	2
	Responses: OV-101: TI15/TR4 OV-17: FP8								
carbophenothion oxygen analog	C11H16ClO3PS2	2.17	4.2	3.06	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI6/TI15 OV-17: FP15								
carbophenothion oxygen analog sulfone	C11H16ClO5PS2	3.8	-	7.1	-	-	-	-	-
	Responses: OV-101: NI36/TI35 OV-17: FP(WB)24								
carbophenothion oxygen analog sulfoxide	C11H16ClO4PS2	4.2	-	2.87	-	-	-	-	-
	Responses: OV-101: TI250 OV-17: FP15								
carbophenothion sulfone	C11H16ClO4PS3	5.1	-	9.2	C	C	P	6	1
	Responses: OV-101: FP3/TI20 OV-17: FP30								
carbophenothion sulfoxide	C11H16ClO3PS3	5.4	-	4	-	-	-	-	-
	Responses: OV-101: FP3/TI35 OV-17: FP20								
carbosulfan	C20H32N2O3S	5.4	-	5.3	P	-	-	-	-
	Responses: OV-101: NP20								
carboxin	C12H13NO2S	1.87	-	-	C	NR	NR	6-15-50	-
	Responses: OV-101: FS50								
carboxin sulfoxide	C12H13NO3S	0.13	0.23	0.11	-	NR	NR	6-15-50	1-2-3
	Responses: OV-101: FS10 OV-17: FS30 OV-225: FS25								
CGA 100255	C15H12NO5	1.8	-	2.96	S	-	-	-	-
	Responses: OV-101: NP100 OV-17: NI1000/NP150								

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Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
CGA 118244	C15H13Cl2N3O3	7	-	11.4	V	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI40								
CGA 120844	C8H9NSO3	0.6	2.65	0.9	-	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI200/NP200 OV-17: NI100/NP300 OV-225: NI300								
CGA 14128	C12H21N2O4PS	0.75	0.8	0.68	C		-	50	1-2-3
	Responses: OV-101: NI2/NP2 OV-17: NI0.6/NP2 OV-225: NI2 D								
CGA 150829	C5H14N4O	0.22	-	0.14	V	-	-	-	-
	Responses: OV-101: NP0.5 OV-17: NP1								
CGA 171683	C6H5F4N3O2	0.06	0.08	0.04	C		-	15+50	3
	Responses: OV-101: NI30 OV-17: NI10 OV-225: NI40								
CGA 189138	C13H8O3Cl2	1.39	1.89	1.54	-	-	-	-	-
	Responses: OV-101: NI1000 OV-17: NI1000 OV-225: NI1000								
CGA 205374	C16H11N3O2Cl2	12	8.9	6.1	-	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI50 OV-17: NI200 OV-225: NI500								
CGA 205375	C16H13N3O2Cl2	6.7	-	1.59	-	-	-	-	-
	Responses: OV-101: NI1000 OV-17: NI1000								
CGA 236431	C8H7F3N2O2	0.17	-	0.11	-	-	-	-	-
	Responses: OV-101: NP200 OV-17: NP20								
CGA 236432	C9H9F3N2O2	0.26	-	0.13	-	-	-	-	-
	Responses: OV-101: NP20 OV-17: NP8								
CGA 27092	C8H7F3N2O	-	-	0.62	-	-	-	-	-
	Responses: OV-17: NP50								
CGA 37734	C10H13NO2	0.4	-	0.47	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NP(V)20 OV-17: NP100								
CGA 51702	C9H9F3N2O	0.46	-	0.49	-	-	-	-	-
	Responses: OV-101: NP2 OV-17: NP3								
CGA 72903	C7H6F3N	0.22	-	0.14	-	-	-	-	-
	Responses: OV-101: NP100 OV-17: NP50								
CGA 91305	C10H8Cl2N3O	1.15	4.3	1.54	V	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI3								
CGA 94689A	C15H21NO5	1.53	6.5	2.41	V	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI5/NP150 OV-17: NI5/NP38 OV-225: NI5								

Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
CGA 94689B	C15H21NO5	1.54	6.6	2.45	S	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI12/NP150 OV-17: NI10/NP75 OV-225: NI8								
chloramben methyl ester	C8H7Cl2NO2	0.44	1.03	-	-	-	-	-	-
	Responses: OV-101: HX0.8/NI0.8								
chlorbenside	C13H10Cl2S	1.39	1.62	1.54	C	S	P	6	1
	Responses: OV-101: NI6 OV-17: HX3/NI1								
chlorbromuron	C9H10BrClN2O2	1.27	3.39	1.42	V	V	V	50	3
	Responses: OV-101: HX12/NI19								
chlorbufam	C11H10ClNO2	0.42	0.75	0.45	C		-	15	2+3
	Responses: OV-101: HX4 OV-17: HN(WB)0.4								
chlordane*	C10H6Cl8	0.45	0.16	-	C	C	C	6	1
		0.63	0.5	-					
		0.73	0.52	-					
		0.81	0.85	0.23					
		0.97	0.9	0.53					
		1.16	1.45	0.61					
		1.45	1.54	0.88					
		1.62	2.69	1.33					
		2.61	3.33	1.47					
	Responses: OV-101: NI(WB)5 OV-17: NI11 OV-225: NI4								
chlordane, cis-	C10H6Cl8	1.66	1.54	1.48	C	C	C	6	1
	Responses: OV-101: TR1 OV-17: NI0.8								
chlordane, trans-	C10H6Cl8	1.49	1.46	1.34	C	C	C	6	1
	Responses: OV-101: TR1 OV-17: NI0.6								
chlordecone	C10H8Cl10O5	2.75	1.67	2.38	-	S #	P #	15+50	1-2-3
	Responses: OV-101: NI(WB)2 OV-17: HX2/NI5 OV-225: NI6								
chlordene	C10H6Cl6	0.56	0.4	0.32	-	C	C	6	1
	Responses: OV-101: NI(WB)1 OV-17: NI0.4 OV-225: NI0.3								
chlordene epoxide	C10H6Cl6O	0.84	0.65	-	-	C	-	15	-
	Responses: OV-101: NI0.6								
chlordene, alpha-	C10H6Cl6	0.82	0.64	0.67	-	-	-	-	-
	Responses: OV-101: TR2 OV-17: NI0.6								
chlordene, beta-	C10H6Cl6	0.98	0.84	0.89	-	-	-	-	-
	Responses: OV-101: TR1 OV-17: NI1								

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Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
chlordene, gamma-	C10H6Cl6	0.98	0.89	0.88	-	-	-	-	-
	Responses: OV-101: TR2 OV-17: NI1 OV-225: NI1								
chlorethoxyfos	C6H11Cl4O3PS	0.33	0.23	0.24	V	C	-	6	1
	Responses: OV-101: FP0.5/HX0.3/NI0.5 OV-17: FP0.5/HX0.3/NI0.3 OV-225: FP0.5/NI0.3								
chlorfenapyr (prop)	C15H11BrClF3N2O	2.21	-	2.34	P	-	S	50	2
	Responses: OV-101: NI2/NP50 OV-17: NI2/NP50 DEGS: NI4/NP190								
chlorfenvinphos, alpha-	C12H14Cl3O4P	1.21	1.58	1.29	C	-	NR	6-15-50	-
	Responses: OV-101: FP10/FP(WB)1.7/NI(WB)2/TI4 OV-17: FP(WB)2/NI3 OV-225: NI5								
chlorfenvinphos, beta-	C12H14Cl3O4P	1.29	2	1.52	C	S #	-	50	1-2-3
	Responses: OV-101: FP2/FP(WB)1.8/HX3/NI(WB)2/TI4 OV-17: FP4/FP(WB)2/NI3 OV-225: FP4/NI5								
chlorflurecol methyl ester	C15H11ClO3	1.73	-	1.88	C	-	-	-	-
	Responses: OV-101: HX8/NI3								
chlorimuron ethyl ester	C15H15ClN4O6S	0.13	0.15	0.1	P	NR	-	-	-
	Responses: OV-101: NI14/NI(WB)24/NP35 OV-17: NI(WB)1.4/NP23								
chlormephos	C5H12ClO2PS2	-	-	0.11	C	-	-	-	-
	Responses: OV-17: FP0.4								
chlornitrofen	C12H6Cl3NO3	2.85	4.7	-	C	C	C	6+15	2
	Responses: OV-101: TR5								
chlorobenzilate	C16H14Cl2O3	2.31	3.26	2.61	C	C #	P #	15+50	3
	Responses: OV-101: TR70 OV-17: NI15								
chloroneb	C8H8Cl2O2	0.19	0.19	-	C	C	-	6	2
	Responses: OV-101: NI3.5								
chloropropylate	C17H16Cl2O3	2.33	2.9	2.41	P	C	C	15+50	3
	Responses: OV-101: TR80 OV-17: NI15								
chlorothalonil	C8Cl4N2	0.55	1.44	0.74	S	C #	C #	6-15-50	2+3
	Responses: OV-101: HX1/NI0.6 OV-17: HX1/NI2								
chlorothalonil trichloro impurity	C8HCl3N2	0.32	-	-	R	R #	NR	6-15-50	2+3
	Responses:								
chloroxuron	C15H15ClN2O2	0.81	0.85	-	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: HX16/NI300								
chlorpropham	C10H12ClNO2	0.32	0.43	0.25	C	C	C	15	2
	Responses: OV-101: HX2 OV-17: NI80								

Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
chlorpyrifos	C9H11Cl3NO3PS	1	1	1	C	C	P	6	2
	Responses: OV-101: NI1.5/TI3 OV-17: FP2/NI2								
chlorpyrifos oxygen analog	C9H11Cl3NO4P	0.95	1.51	1.08	C	NR	-	6-15-50	-
	Responses: OV-101: FP27/NI6/TI8								
chlorpyrifos-methyl	C7H7Cl3NO3PS	0.72	0.86	0.79	C	C	-	6	2
	Responses: OV-101: FP1/FP(WB)1.3/HX1.5/NI1/NP1 OV-17: FP(WB)1.5								
chlorsulfuron	C12H12ClN5O4S	1.3	8.9	-	-	NR	NR	6-15-50	-
	Responses: OV-101: NI50								
chlorthiamid	C7H5Cl2NS	0.69	-	-	-	-	-	-	-
	Responses:								
chlorthiophos oxygen analog	C11H15Cl2O4PS	2.22	4.1	2.99	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: HX9/NI6 OV-17: FP10/HX11 OV-225: FP6								
chlorthiophos sulfone	C11H15Cl2O5PS2	5.3	18.8	9.1	C	C	-	50	3
	Responses: OV-101: HX20/NI9 OV-17: FP100/HX22 OV-225: FP39								
chlorthiophos sulfoxide	C11H15Cl2O4PS2	4.7	10.3	6.9	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: HX20/NI6 OV-17: FP25/HX17 OV-225: FP15								
chlorthiophos*	C11H15Cl2O3PS2	2.24	-	2.58	C	C	C	6	2
		2.36	-	2.77					
		2.56	-	3.16					
	Responses: OV-101: FP8 OV-17: FP5								
CL 202,347	C13H19N3O5	2.96	11.5	4.1	-	-	-	-	-
	Responses: OV-101: NI15/NP50 OV-17: NI20/NP100 OV-225: NI60								
clodinafop-propargyl	C17H13ClFNO4	3.26	5.8	4.67	V	V	-	50	3
	Responses: OV-101:NP(WB)30 OV-17:NI(WB)20 OV-225:NI(WB)5								
clofentezine	C14H8Cl2N4	5.9	-	9.8	R	S	-	15	2
	Responses: OV-101: HN(WB)10.5/HX20/NI100 OV-17: NP165								
clomazone	C12H14ClNO2	0.45	0.59	0.46	C		-	50	3
	Responses: OV-101: HX1.5/HX2/NI110 OV-17: HX2/NP11 OV-225: NI150								
clopyralid methyl ester	C7H4Cl2NO2	0.18	-	-	-		-	50	-
	Responses: OV-101: NI0.25								
cloquintocet-mexyl	C18H22ClNO3	4.8	6.6	6.3	V	NR	-	6-15-50	1-2-3
	Responses: OV-101:NP(WB)40 OV-17:NI(WB)20 OV-225:NI(WB)5								

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Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
Compound K*	C10H6Cl8	0.83	-	-	-	C	-	-	1
		2.53	2.66	-					
	Responses: OV-101: TR5								
coumaphos	C14H16ClO5PS	9	40	18	C	NR	C #	6-15-50	3
	Responses: OV-101: NI39/NP38 OV-17: FP50/FP(WB)26/NI38/NP34 OV-225: NI100								
coumaphos oxygen analog	C14H16ClO6P	8	45	16	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI200/NP130 OV-17: FP75/NI50/NP40 OV-225: NI150								
CP 108064, methylated	C15H21NO4	0.73	-	0.67	-	-	-	-	-
	Responses: OV-101: NP6 OV-17: NP6								
CP 51214	C14H21NO3	0.7	-	0.58	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NP13 OV-17: NP24								
crotoxyphos	C14H19O6P	1.37	2.85	1.9	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI60/TI10 OV-17: FP10/FP(WB)3								
crufomate	C12H19ClNO3P	1.08	2.33	1.3	C	NR	NR	6-15-50	-
	Responses: OV-101: TI6 OV-17: FP2/NI3								
cyanazine	C9H13ClN6	0.89	4.9	1.48	C	NR	-	6-15-50	-
	Responses: OV-101: NI4/TI26 OV-17: HX6								
cyanofenphos	C15H14NO2PS	3.1	8.2	4.6	C	-	-	-	-
	Responses: OV-101: FP3.5/NI3/NP(WB)3								
cyanophos	C9H10O3NSP	0.47	-	0.59	C	-	-	-	-
	Responses: OV-101: FP(WB)0.7/NI(WB)2/NP1 OV-17: FP(WB)0.7/NP(WB)1								
cyclanilide methyl ester	C12H11Cl2NO3	1.57	1.84	1.64	-	-	-	-	-
	Responses: OV-101: NI5/NP30 OV-17: NI6/NP30 OV-225: NI6								
cycloate	C11H21NOS	0.3	-	-	C	V #	S	15+50	3
	Responses: OV-101: FS2/NP15								
cyfluthrin*	C22H18Cl2FNO3	11.7	-	-	C	P	-	15	-
		12.5	-	-					
		12.8	-	-					
	Responses: OV-101: HX30/NI30								
cymiazole	C12H14N2S	0.73	-	0.89	-	-	-	-	-
	Responses: OV-101: NP2 OV-17: NP(WB)2								
cymoxanil	C7H10N4O3	0.25	0.5	0.16	V	NR	NR	6-15-50	1-2-3
	Responses: OV-101: HN(WB)1/NI(WB)23/NP(WB)10 OV-17: HN(WB)3/NI(WB)120/NP(WB)7 OV-225: NI(WB)70								

Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
cypermethrin*	C22H19Cl2NO3	-	29	-	C	C	C	15	2
		14.1	33	23					
		15.1	36	25					
	Responses: OV-101: NI90								
cyprazine	C9H14ClN5	0.64	1.22	0.74	C	-	-	-	-
	Responses: OV-101: HX1.5/NI13								
cyproconazole	C15H18ClN3O	2.04	1.61	2.69	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: HN(WB)182/HX(WB)73/NI(WB)72/NP(WB)12								
cyprodinil	C14H15N3	1.18	-	1.39	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NP(WB)2 OV-17: NP(WB)10								
cyromazine	C6H10N6	0.58	-	0.68	S	-	-	-	-
	Responses: OV-101: NP10 OV-17: NP2								
dazomet	C5H10N2S2	0.4	-	0.71	S	NR	-	6-15-50	1-2-3
	Responses: OV-101: HX500/NI300 OV-17: FS(WB)80/HN(WB)0.4/HX500/NI300								
DCPA	C10H6Cl4O4	1.06	1.13	1	C	C	C	15	2
	Responses: OV-101: NI1 OV-17: NI1 OV-225: NI1								
DDE, o,p'-	C14H8Cl4	1.55	1.28	1.51	C	C	C	6	1
	Responses: OV-101: TR2 OV-17: NI1								
DDE, p,p'-	C14H8Cl4	1.92	1.59	1.86	C	C	C	6	1
	Responses: OV-101: NI1.5 OV-17: NI1								
DDM	C13H10Cl2	0.72	-	-	-	-	-	-	-
	Responses:								
DDMS	C14H11Cl3	1.65	-	1.65	-	R	-	6	-
	Responses:								
DDMU	C14H9Cl3	1.47	-	-	-	-	-	-	-
	Responses:								
DDNS	C14H12Cl2	0.83	-	-	-	-	-	-	-
	Responses:								
DDNU	C14H10Cl2	0.83	-	-	-	-	-	-	-
	Responses:								
DDT, o,p'-	C14H9Cl5	2.55	2.27	2.7	C	C	C	6	1
	Responses: OV-101: TR4 OV-17: NI2								

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Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
DDT, p,p'-	C14H9Cl5	3.13	3.6	3.5	C	C	C	6	1
Responses: OV-101: TR4 OV-17: NI2									
deltamethrin*	C22H19Br2NO3	17.1	-	21	C	S #	P	15	2
		27	-	35					
		29	19.9	38					
Responses: OV-101: NI1300									
deltamethrin, trans-*	C22H19Br2NO3	17	-	6.2	-	P #	NR	15	2
		29	-	20					
		31	19.7	38					
Responses: OV-101: NI200									
demeton-O oxygen analog	C8H19O4PS	0.22	0.32	0.21	-	-	-	-	-
Responses: OV-101: TI6 OV-17: FP25									
demeton-O sulfone*	C8H19O5PS2	-	-	0.28	C	-	-	-	-
		0.71	2.95	0.96					
Responses: OV-101: FP5/TI12 OV-17: FP3									
demeton-O sulfoxide	C8H15O4PS2	0.87	-	1.05	C	-	-	-	-
Responses: OV-101: FP4									
demeton-O*	C8H19O3PS2	-	-	0.2	C	NR	-	6-15	-
		0.28	-	0.36					
Responses: OV-101: FP(WB)2 OV-17: FP2									
demeton-S	C8H19O3PS2	0.41	0.56	0.41	C	NR	-	6-15-50	-
Responses: OV-101: FP(WB)0.8/TI2 OV-17: FP0.8/FP(WB)0.8									
demeton-S sulfone	C8H19O5PS2	1.15	5.8	1.75	C	-	-	-	-
Responses: OV-101: FP40/TI20 OV-17: FP5 OV-225: FP60									
demeton-S sulfoxide	C8H19O4PS2	-	-	-	C	-	-	-	-
Responses: DEGS: FP30									
des N-isopropyl isofenphos	C12H18NO4PS	1.21	2.73	1.5	C	S	-	50	-
Responses: OV-101: FP2 OV-17: FP3									
des N-isopropyl isofenphos oxygen analog	C12H18NO5P	0.93	-	1.43	-	-	-	-	-
Responses: OV-101: FP(WB)5 OV-17: FP(WB)12									
desdiethyl simazine	C3H4ClN5	0.2	0.86	0.61	-	NR	NR	6-15-50	1-2-3
Responses: OV-101: HX25/NI20 OV-17: HN(WB)0.1/HX25 OV-225: NI20									
desethyl simazine	C5H8ClN5	0.3	0.8	0.53	-	NR	NR	50	1-2-3
Responses: OV-101: HX12/NI20 OV-17: HN(WB)0.1/HX12 OV-225: NI80									

Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
desisopropyl iprodione	C10H6Cl2N3O3	2.31	15	3.93	P		-	50	1-2-3
	Responses: OV-101: NI20/NP50								
desmedipham	C16H16N2O4	0.44	0.29	0.45	-	-	-	-	-
	Responses: OV-101: NI1000/NP300 OV-17: NI1000/NP218 OV-225: NI1000								
desmethyl diphenamid	C15H15NO	0.98	-	-	-	-	-	-	-
	Responses: OV-101: TI340								
desmethyl norflurazon	C11H7ClF3N3O	3.38	1.41	4.9	V	NR	NR	6-15-50	1-2-3
	Responses: OV-101: HX(WB)3/NI27 OV-17: HX(WB)3 OV-225: NI200								
di-allate	C10H17ClNOS	0.42	0.26	0.33	C	C	-	6	-
	Responses:								
di-n-octyl phthalate	C24H38O4	12	-	-	-	C	C	15+50	-
	Responses: OV-101: NI(V)330								
dialifor	C14H17ClNO4PS2	6.5	-	14.3	C	C	P	15	2
	Responses: OV-101: TI30/TR28 OV-17: FP25/FP(WB)31								
diazinon	C12H21N2O3PS	0.51	0.4	0.44	C	C	C	15	3
	Responses: OV-101: FP(WB)1/NI3/NP0.4 OV-17: FP0.7/FP(WB)0.9/NI4/NP0.25 OV-225: FP6/NI4.5								
diazinon oxygen analog	C12H21N2O4P	0.5	0.53	0.47	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI18/NP0.6 OV-17: NI30/NP0.6 OV-225: NI60								
dibromochloropropane	C3H5Br2Cl	0.04	-	0.03	-	-	-	-	-
	Responses: OV-101: TR0.6 OV-17: NI0.2								
dibutyl phthalate	C16H22O4	0.88	0.92	0.84	-	C	C	15+50	-
	Responses: OV-101: NI30								
dicamba methyl ester	C8H6Cl2O3	0.19	0.18	-	-	-	-	-	-
	Responses: OV-101: HX(WB)1.6/NI0.6								
dichlobenil	C7H3Cl2N	0.11	-	0.1	C	P	C	15	2
	Responses: OV-101: TR0.5 OV-17: NI0.6								
dichlofenthion	C10H13Cl2O3PS	0.67	0.64	0.56	C	C	V	6	2
	Responses: OV-101: FP1/FP(WB)3.5/NI1.9/TI2 OV-17: FP0.8/HX(WB)2								
dichlofluanid	C9H11Cl2FN2O2S2	0.9	1.71	1.01	C	C #	-	15+50	2+3
	Responses: OV-101: NI1/NP44								
dichlone	C10H4Cl2O2	0.55	0.92	-	P	S #	S #	6-15-50	2+3
	Responses: OV-101: NI2								

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Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
dichlorobenzene, p-	C6H4Cl2	0.03	-	0.02	-	C	C	6	1
	Responses: OV-101: TR37 OV-17: NI4								
dichlorobenzophenone, o,p'-	C13H8Cl2O	0.82	1.07	0.92	-	C	C	15	2
	Responses: OV-101: TR4 OV-17: NI2								
dichlorobenzophenone, p,p'-	C13H8Cl2O	0.99	1.25	1.08	-	C	C	15	2
	Responses: OV-101: TR3 OV-17: NI2								
dichlorprop methyl ester	C10H10Cl2O3	0.28	-	-	-	-	-	-	-
	Responses: OV-101: HX(WB)1.6/NI2								
dichlorvos	C4H7Cl2O4P	0.07	0.08	0.08	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: FP9/NI1/TI0.5 OV-17: FP2.5 OV-225: FP(WB)0.7								
diclobutrazol	C15H19Cl2N3O	2.02	3.4	2.03	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: HX7/NI7/NP(WB)8 OV-17: HN(WB)1.3/HX7/HX4/HX(WB)2/NI(WB)4/NP(WB)8 OV-225: NI7								
diclofop-methyl	C16H14Cl2O4	3.57	4.9	4.7	C	C	C	15	2
	Responses: OV-101: HX8/NI10 OV-17: HX10 OV-225: NI12								
dicloran	C6H4Cl2N2O2	0.42	0.96	0.45	C	S	P	15+50	2+3
	Responses: OV-101: TR0.5 OV-17: NI0.4								
dicofol, o,p'-*	C14H9Cl5O	0.86	-	-	C	V	S	6+15	2
		4.1	1.08	0.91					
	Responses: OV-101: NI5 OV-17: HX2								
dicofol, p,p'-*	C14H9Cl5O	1.04	-	-	C	V	P #	6+15	1+2
		4.4	1.28	1.08					
	Responses: OV-101: NI5 OV-17: HX3								
dicrotophos	C8H16NO5P	0.31	0.96	0.43	C	NR	-	6-15-50	-
	Responses: OV-101: FP(WB)0.6/TI10 OV-17: FP1/FP(WB)0.8								
dieldrin	C12H8Cl6O	1.91	1.87	1.84	C	C	C	15	2
	Responses: OV-101: HX1/NI1.5 OV-17: HX1.5/NI1								
diethatyl-ethyl	C16H22ClNO3	1.78	3.14	2	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: HX11/NI10/NP180 OV-17: NI11/NP200 OV-225: NI14								
diethyl phthalate	C12H14O4	0.26	-	-	-	P	P	15+50	-
	Responses: OV-101: NI3500								
difenoxuron	C16H18N2O3	0.97	-	0.96	-	-	-	-	-
	Responses: OV-101: HN(WB)5 OV-17: NP16								

Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
diisobutyl phthalate	C16H2204	0.65	0.61	0.56	-	P	-	15+50	-
	Responses: OV-101: NI20								
diisohexyl phthalate*	C20H30O4	2.45	-	-	-	C	-	15+50	-
		2.66	-	-					
		2.9	-	-					
		3.27	-	-					
	Responses: OV-101: TR340								
diisooctyl phthalate*	C24H38O4	0.91	-	-	-	C	C	15+50	-
		5.5	-	-					
		6.2	-	-					
		6.7	-	-					
		7.5	-	-					
		9	-	-					
		10.5	-	-					
	Responses: OV-101: TR850								
Dilan*	C15.5H14Cl2NO2	-	5.3	-	-	P	P	15	-
		-	4.8	-					
		2.33	5.8	-					
		2.81	7.5	-					
		3.39	8.2	-					
	Responses: OV-101: TR8								
dimethachlor	C13H18ClNO2	0.71	1.11	0.71	C	-	-	-	-
	Responses: OV-101: NI30 OV-17: NI10 OV-225: NI20								
dimethametryn	C11H21N5S	-	-	-	C	-	-	-	-
	Responses:								
dimethenamid	C12H18ClNO2S	0.72	0.98	-	-	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI(WB)19/NP(WB)14								
dimethipin	C6H10O4S2	0.41	2.71	0.81	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: FS0.5 OV-17: FS1.5 OV-225: FS2								
dimethoate	C5H12NO3PS2	0.4	1.6	0.62	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: FP(WB)0.7/NI(WB)6/NP1 OV-17: FP0.8/FP(WB)0.8/NI5								
dimethyl phthalate	C10H10O4	0.15	0.15	0.14	-	P	-	6+15+50	-
	Responses: OV-101: NI300								
dinitramine	C11H13F3N4O4	0.52	0.93	0.44	C	-	P	15	-
	Responses: OV-101: NI(WB)1/TI166 OV-17: NI1 OV-225: NI1								

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Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
dinobuton	C14H18N2O7	1.4	-	1.32	C	-	-	-	-
	Responses: OV-101: HN(WB)1.5 OV-17: HN(WB)1/NP(WB)50								
dinocap*	C18H24N2O6	-	-	3.5	C	P	P	15	2
		4	-	3.9					
		4.3	6.9	4.4					
		4.8	7.7	4.8					
		5.1	9.5	5.6					
	Responses: OV-101: NI(WB)6 OV-17: NI150								
dinoseb methyl ether	C11H14N2O5	0.63	-	-	-	-	-	-	-
	Responses: OV-101: HN1/NI1.2								
dioxabenzofos	C8H9O3PS	0.34	-	0.36	C	P	-	15	-
	Responses: OV-17: FP0.7								
dioxacarb	C11H13NO4	-	-	-	C	-	-	-	-
	Responses:								
dioxathion	C12H26O6P2S4	0.47	-	0.5	V	NR	-	6-15-50	2
	Responses: OV-101: NI100/TI10 OV-17: FP7/FP(WB)13								
diphenamid	C16H17NO	1.1	-	1.55	V	NR	-	6-15	-
	Responses: OV-17: NP(WB)25								
diphenylamine	C12H11N	0.29	-	0.25	C	S	-	6+15	-
	Responses:								
disul-Na	C8H7Cl2O5S•Na	0.23	-	-	-	-	-	-	-
	Responses: OV-101: NI3								
disulfoton	C8H19O2PS3	0.54	0.6	0.46	C	P #	NR	6	1-2-3
	Responses: OV-101: TI2 OV-17: FP1								
disulfoton sulfone	C8H19O4PS3	1.5	6.7	2.39	C	NR	-	6-15-50	-
	Responses: OV-101: TI7 OV-17: FP7								
disulfoton sulfoxide	C8H1903PS3	-	-	-	C	-	-	-	-
	Responses:								
dithianon	C14H4O2N2S2	4.7	53	11.3	NR	-	-	-	-
	Responses: OV-101: NI(WB)12 OV-17: NP(WB)120								
diuron	C9H10Cl2N2O	0.11	0.09	0.11	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI(WB)9 OV-17: NI12 OV-225: NI27								
DNOC methyl ether	C8H8N2O5	0.35	-	-	-	-	-	-	-
	Responses: OV-101: HN(WB)30								

Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
edifenphos	C14H15O2PS2	2.87	6.3	5.3	C	-	-	-	-
	Responses: OV-101: FP(WB)4/NI(WB)4 OV-17: FP(WB)8								
endosulfan I	C9H6Cl6O3S	1.64	1.38	1.47	C	C	C	15	2
	Responses: OV-101: HX1/NI1.3 OV-17: HX1/NI2								
endosulfan II	C9H6Cl6O3S	2.21	3.9	2.77	C	C	C	15+50	2
	Responses: OV-101: HX2/NI2 OV-17: HX3/NI2								
endosulfan sulfate	C9H6Cl6O4S	2.83	8.3	4	C	C	C	50	2
	Responses: OV-101: HX4/TR5 OV-17: HX6/NI6								
endrin	C12H8Cl6O	2.13	2.22	2.29	C	C #	C #	15	2
	Responses: OV-101: TR2 OV-17: NI2								
endrin alcohol	C12H8Cl6O	2.55	-	-	-	P	C	15+50	2+3
	Responses: OV-101: TR4								
endrin aldehyde	C12H8Cl6O	2.35	-	-	C	P	C	15+50	-
	Responses: OV-101: TR4								
endrin ketone	C12H8Cl6O	3.6	10.3	-	-	C	C	50	2
	Responses: OV-101: TR5								
EPN	C14H14NO4PS	4.5	10.6	6.9	C	C	C	15	2
	Responses: OV-101: NI0.5/TI16 OV-17: FP50/NI9								
epoxyhexachloronorbornene	C7H2Cl6O	-	-	0.2	-	-	-	-	-
	Responses:								
EPTC	C9H19NOS	0.12	-	-	-	P	-	15	-
	Responses: OV-101: TI30								
esfenvalerate	C25H22ClNO3	22.5	-	-	C	C	C	15	2
	Responses: OV-101: NI90								
etaconazole*	C14H15Cl2N3O2	2.36	-	-	C	-	-	-	-
		2.43	-	3.17					
	Responses: OV-101: NP(WB)12 OV-17: HX7								
ethalfluralin	C13H14F3N3O4	0.34	0.27	0.19	C	C	C	6	2
	Responses: OV-101: HX4/HX(WB)7/NI1 OV-17: HX6 OV-225: NI0.4								
ethametsulfuron methyl ester*	C15H18N6O6S	0.35	2.85	0.4	-	NR	NR	6-15-50	1-2-3
		0.55	3.6	0.95					
	Responses: OV-101: HN(WB)17/NI(WB)34/NP(WB)600 OV-17: HN(WB)30 OV-225: NI(WB)780								

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Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
ethephon	C2H6ClO3P	3.03	2.74	2.88	NR		-	6+15+50	1+2+3
	Responses: OV-101: NI170								
ethiofencarb	C10H15NO2S	0.6	1.4	0.78	C	NR	NR	6-15-50	-
	Responses: OV-101: FP20/FP400/FS1.5/HN(WB)3.5/NI950/NP(WB)4 OV-17: NP133								
ethiolate	C7H15NOS	0.06	-	-	C	-	-	-	-
	Responses: OV-101: FS9								
ethion	C9H22O4P2S4	2.56	3.93	3.36	C	C	C	6	2
	Responses: OV-101: FP(WB)1.9/NI(WB)3/NP2 OV-17: FP4/FP(WB)2.3/NI8 OV-225: FP2/NI8								
ethion oxygen analog	C9H22O5P2S3	1.88	4.1	-	C	-	-	-	-
	Responses: OV-101: NP1								
ethofumesate	C13H18O5S	0.86	1.93	1.02	C	-	-	-	-
	Responses: OV-101: FS32/NI315 OV-17: FS65/NI333 OV-225: FS56/NI638								
ethoprop	C8H19O2PS2	0.33	0.31	0.25	C	P #	S #	50	1-2-3
	Responses: OV-101: FP0.7/TI0.8 OV-17: FP1								
ethoxyquin	C14H19N0	0.6	1.64	0.7	C	NR	NR	6-15-50	-
	Responses: OV-101: NI20/NP15 OV-17: NI12/NP15 OV-225: NI30								
ethyl p-toluene sulfonamide	C9H13NO2S	-	-	-	C	-	-	-	-
	Responses:								
ethylenethiourea	C3H6N2S	0.5	2.33	0.66	S	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI2200/NP23 OV-17: NI4500/NP64 OV-225: NI6250								
etridiazole	C5H5Cl3N2OS	0.18	0.12	0.21	C	C	P	6	2
	Responses: OV-101: NI0.3/NP0.6 OV-17: HX(WB)0.8/NI0.4/NP0.5 OV-225: NI0.2								
etrimfos	C10H17N2O4PS	0.58	0.59	0.51	C	C	C	15	2+3
	Responses: OV-101: FP2/NI50 OV-17: NI50 OV-225: NI30								
etrimfos oxygen analog	C10H17N2O5P	0.51	0.8	0.63	C	-	-	-	-
	Responses: OV-101: FP7/NI1000/NP(WB)3.5								
famphur	C10H16NO5PS2	2.65	14	5	C	NR	-	6-15-50	-
	Responses: OV-101: FP8/TI40 OV-17: FP50/FP(WB)7								
famphur oxygen analog	C10H16NO6PS	2.26	-	4.4	C	-	-	-	-
	Responses: OV-101: FP54 OV-17: FP(WB)12								
fenac	C8H5Cl3O2	1.42	3.7	-	-	NR	NR	6-15-50	-
	Responses: OV-101: NI2800								

Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
fenac methyl ester	C9H7Cl3O2	0.32	-	-	-	-	-	-	-
	Responses: OV-101: NI0.4								
fenamiphos	C13H22NO3PS	1.66	3.7	2.41	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: FP8/NP8 OV-17: FP10/NP3 OV-225: NP6								
fenamiphos sulfone	C13H22NO5PS	4.5	-	8.4	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: FP50/NP20 OV-17: FP(WB)20/NP60								
fenamiphos sulfoxide	C13H22N04PS	5.2	-	8.1	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: FP50/NP55 OV-17: FP(WB)28/NP45								
fenarimol	C17H12Cl2N2O	6.6	-	10.1	C	P #	C #	50	3
	Responses: OV-101: HX10/NI5								
fenarimol metabolite B	C17H14N2OCl2	4.6	-	-	NR	NR	NR	6-15-50	-
	Responses: OV-101: HX19								
fenarimol metabolite C	C17H14N2OCl2	4.6	-	-	S		-	6	-
	Responses: OV-101: HX8								
fenbuconazole	C19H17ClN4	9.8	-	-	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI1000/NP70								
fenfuram	C12H11NO2	0.54	1.47	0.62	C	-	-	-	-
	Responses: OV-101: NP5/NP(WB)6								
fenhexamid	C14H17Cl2NO2	3.1	-	3.7	NR	NR	NR	6-15-50	1-2-3
	Responses:OV-101:NI(WB)2/NP(WB)220 OV-17:NI(WB)5								
fenitrothion	C9H12NO5PS	0.84	1.82	1.05	C	C	C	15	2
	Responses: OV-101: FP(WB)1/NP1 OV-17: FP3/FP(WB)1.1								
fenitrothion oxygen analog	C9H12NO6P	0.72	-	0.83	C	-	-	-	-
	Responses: OV-101: FP3 OV-17: FP10								
fenoxaprop ethyl ester	C18H16NO5Cl	8.1	11.3	10.5	S	V	V	50	3
	Responses: OV-101: NI250								
fenoxycarb	C17H19NO4	5	-	7.3	C	-	-	-	-
	Responses: OV-101: NP50 OV-17: NP50								
fenpropathrin	C22H23NO3	4.8	7	5.7	-	V #	V	15	2
	Responses: OV-101: NI7/NI(WB)0.2/NP30 OV-17: NI13 OV-225: NI10								
fenpropimorph	C20H33NO	1.09	-	0.62	C		-	50	1-2-3
	Responses: OV-101: NP33 OV-17: NP14								

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Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
fenson	C12H10O3ClS	-	2.03	-	-	-	-	-	-
	Responses: OV-225: NI1.5								
fensulfothion	C11H17O4PS2	2.4	-	3.8	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: TI12 OV-17: FP6/FP(WB)7								
fensulfothion oxygen analog	C11H17O5PS	2.49	-	5	C	NR	-	6-15-50	-
	Responses: OV-101: TI10 OV-17: FP5								
fensulfothion oxygen analog sulfone	C11H17O7PS2	1.99	-	3.8	-	-	-	-	-
	Responses: OV-101: TI45 OV-17: FP6								
fensulfothion sulfone	C11H17O5PS2	2.8	-	3.6	C	NR	-	6-15-50	-
	Responses: OV-101: NI9/NP7 OV-17: FP100								
fenthion	C10H15O3PS2	0.96	1.46	1.18	C	S #	NR	6+15	1-2-3
	Responses: OV-101: FP2/TI4 OV-17: FP3								
fenthion oxygen analog	C10H15O4PS	0.78	-	1.12	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: FP7 OV-17: FP9								
fenthion oxygen analog sulfone*	C10H15O6PS2	1.77	-	-	-	-	-	-	-
		2.29	-	4.1					
	Responses: OV-101: FP(WB)4 OV-17: FP(WB)8								
fenthion oxygen analog sulfoxide	C10H15O5PS	0.43	-	0.62	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: FP100 OV-17: FP135								
fenthion sulfone	C10H15O5PS2	2.39	-	4.7	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: FP22 OV-17: FP20								
fenvalerate*	C25H22ClNO3	20.3	44	35	C	C	C	15	2
		22.5	51	40					
	Responses: OV-101: NI90								
fipronil	C12H4Cl2F6N4OS	1.35	8.7	1.16	S	S	V	50	3
	Responses: OV-101: NI2/NP10 OV-17: NI1/NP5 OV-225: NI10								
flamprop-M-isopropyl	C19H19ClFNO3	2.46	-	2.81	C	-	-	-	-
	Responses: OV-101: HX9/NI9								
flamprop-methyl	C17H15ClFNO3	1.94	-	2.45	C	-	-	-	-
	Responses: OV-101: HX8/NI7								
fluazifop butyl ester	C19H20F3NO4	2.3	2.36	2.31	C	C	V	15	3
	Responses: OV-101: HX19/HX(WB)18/NI125 OV-17: HX40 OV-225: NI3000								
fluchloralin	C12H13ClF3N3O4	0.53	0.76	0.37	C	C	-	6	2
	Responses: OV-101: HX3/NI1.5 OV-17: HX3/NI0.5								

Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
flucythrinate*	C26H23F2NO4	14.7 16.1	36.9 42	21.4 24	C	C	-	15	2+3
	Responses: OV-101: NI40/NI(WB)15								
flumetsulam, methylated	C13H11F2N5O2S	12.4	-	-	-	-	-	-	-
	Responses: OV-101: NP(WB)14								
fluometuron	C10H11F3N2O	-	-	0.14	-	-	-	-	-
	Responses: OV-17: NP2								
fluridone	C19H14F3NO	16.3	24	-	-	NR	NR	6-15-50	-
	Responses: OV-101: HX400/NI1500								
fluroxypyr, methylated*	C8H7O3N2Cl2F	0.61 0.79	- -	- -	-	-	-	-	-
	Responses: OV-101: NI2								
flusilazole	C16H15F2N3Si	1.97	-	2.33	C	-	-	-	-
	Responses: OV-101: NP(WB)5 OV-17: HX24/HX(WB)5/NP(WB)6								
fluvalinate*	C26H22ClF3N2O3	- 25	56 59	35 38	C	C	-	15	2
	Responses:								
FMTU	C10H9F3N2O2	-	-	-	-	-	-	-	-
	Responses:								
folpet	C9H4Cl3O2NS	1.23	3.01	1.94	C	C	P	15+50	2+3
	Responses: OV-101: NI(WB)1 OV-17: NI9								
fonofos	C10H15OPS2	0.52	0.56	0.44	C	C	C	6	2+3
	Responses: OV-101: TI2 OV-17: FP0.7								
fonofos oxygen analog	C10H15O2PS	0.39	0.53	0.38	V	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NP1 OV-17: FP4/FP(WB)1								
formetanate hydrochloride	C11H16ClN3O2	0.9	-	0.45	-	-	-	-	-
	Responses: OV-101: NP400 OV-17: NP430								
formothion	C6H12NO4PS2	-	-	0.91	C	NR	NR	6-15-50	1-2-3
	Responses: OV-17: FP(WB)10								
fosthiazate	C9H18NO3PS2	1.08	3	1.66	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: FP100/NI1000/NP40 OV-17: FP10/NI120/NP7 OV-225: FP15/NI300								
fuberidazole	C11H8N2O	0.71	-	0.95	C	-	-	-	-
	Responses: OV-101: HN(WB)0.5/NP(WB)3 OV-17: NP5.5								

Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
furilazole	C11H13Cl2NO3	0.46	0.6	0.39	C	S	-	50	3
	Responses: OV-101: NI1.5/NP50 OV-17: NI1/NP1.8 OV-225: NI1.6								
G-27550	C8H12N2O	0.28	-	0.29	C	-	-	-	-
	Responses: OV-101: NP3 OV-17: NP3								
Gardona	C10H9Cl4O4P	1.58	2.72	1.97	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: FP9/TI8 OV-17: FP10/FP(WB)3								
GS-31144	C8H12N2O2	-	-	-	-	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI220/NP100 OV-17: NI750/NP100								
haloxyfop methyl ester	C16H13ClF3NO4	1.55	-	1.4	-	-	-	-	-
	Responses: OV-101: HX(WB)9/NI5 OV-17: HX(WB)6/NI4.5								
heptachlor	C10H5Cl7	0.83	0.52	0.6	C	C	C	6	1
	Responses: OV-101: NI0.6 OV-17: NI0.4								
heptachlor epoxide	C10H5Cl7O	1.29	1.22	1.15	C	C	C	6	2
	Responses: OV-101: HX0.7/TR1 OV-17: HX0.9/NI2								
heptachloronorbornene	C7H3Cl7	-	-	0.23	-	-	-	-	-
	Responses: OV-17: NI0.2								
heptenophos	C9H12ClO4P	-	-	-	C	-	-	-	-
	Responses:								
hexachlorobenzene	C6Cl6	0.45	0.25	0.33	C	C	P	6	1
	Responses: OV-101: HX0.5/NI0.25 OV-17: HX0.3/NI0.3 OV-225: NI0.3								
hexachlorobutadiene	C4Cl6	-	-	0.04	-	V #	P	6	1
	Responses: OV-17: NI0.1								
hexachlorocyclopentadiene	C5Cl6	0.12	-	0.06	-	-	-	-	-
	Responses: OV-101: TR0.4 OV-17: NI0.8								
hexachloroethane	C2Cl6	-	-	0.02	-	-	-	-	-
	Responses: OV-17: NI0.1								
hexachloronorbornadiene	C7H2Cl6	-	-	0.12	-	-	-	-	-
	Responses: OV-17: NI0.2								
hexachlorophene	C13H6Cl6O2	13	13	16	-	NR	NR	6-15-50	-
	Responses: OV-101: NI(WB)2 OV-17: NI400 OV-225: NI1200								
hexachlorophene dimethyl ether	C15H10Cl6O2	9.7	-	-	-	NR	NR	6-15	-
	Responses: OV-101: TR7								

Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
hexaconazole	C14H17Cl2N3O	1.86	2.91	1.79	C	-	-	-	-
	Responses: OV-101: HX6/NI3 OV-17: NP(WB)23								
hexazinone	C12H20N4O2	2.91	-	-	P	NR	NR	6-15-50	1-2-3
	Responses: OV-101: HN(WB)3/NP(WB)15								
hexythiazox	C17H21ClN2O2S	1.28	-	2.41	-	S #	NR	50	2+3
	Responses: OV-101: NI(WB)70/NP(WB)90 OV-17: FS(WB)90/NI(WB)80/NP(WB)80								
HOE-030291	C17H16Cl2O5	7.9	10.8	12.6	-	-	-	-	-
	Responses: OV-101: NI20 OV-17: NI70 OV-225: NI20								
hydramethylnon*	C25H24F6N4	2.55	-	-	-	-	-	-	-
		32	4.5	-					
		44	53	-					
	Responses: OV-101: NI(V)250								
hydroxy chloroneb	C7H6Cl2O2	0.15	0.24	-	-	NR	-	6-15	-
	Responses: OV-101: TR7								
imazalil	C14H14Cl2N2O	1.76	4	2.08	C	NR	NR	6-15-50	-
	Responses: OV-101: NI30/NP(WB)12 OV-17: HX19/HX(WB)2/NI(WB)5/NP(WB)10								
imazamethabenz methyl ester*	C16H20N2O3	-	-	2.44	C	-	-	-	-
		1.79	3.5	2.76					
	Responses: OV-101: HX540/HX(WB)50/NI45/NP(WB)40 OV-17: HX60								
imazethapyr ammonium salt methyl ester	C16H21N3O3	2.4	4.3	3	-	-	-	-	-
	Responses: OV-101: NI120/NP50 OV-17: NI120/NP70 OV-225: NI160								
imidacloprid	C9H10ClN5O2	1.84	-	-	-	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI200/NP50								
IN-A3928	C11H18N4O2	3.06	-	-	S	NR	NR	6-15-50	1-2-3
	Responses: OV-101: HN(WB)1.4/NP(WB)29								
IN-B2838	C10H15N3O3	0.7	-	0.8	P	NR	NR	6-15-50	1-2-3
	Responses: OV-101: HN(WB)0.6/NP(WB)23 OV-17: HN(WB)0.2/NP(WB)13								
IN-T3935	C11H18N4O3	4.7	-	-	S	-	-	-	-
	Responses: OV-101: HN(WB)73/NI(WB)230								
IN-T3936	C10H15N3O4	1.41	-	2.55	S	NR	NR	6-15-50	1-2-3
	Responses: OV-101: HN(WB)1.6/NP(WB)51 OV-17: HN(WB)1.2/NP(WB)53								
IN-T3937	C12H20N4O3	4.7	-	-	S	-	-	-	-
	Responses: OV-101: HN(WB)390/NI(WB)400								

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Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
ioxynil methyl ether	C8H5I2NO	0.71	-	-	-	-	-	-	-
	Responses: OV-101: HN(WB)5/NI1								
iprobenfos	C13H21O3PS	0.6	-	0.54	C	-	-	-	-
	Responses: OV-101: FP(WB)1 OV-17: FP2								
iprodione metabolite isomer	C13H13Cl2N3O3	5.3	-	7.5	C	S	-	50	-
	Responses: OV-101: HX(WB)20/NI1000 OV-17: HX80								
iprodione*	C13H13Cl2N3O3	-	-	5.2	C	S #	NR	50	1-2-3
		4.2	18	6.3					
	Responses: OV-101: HX13/NI15/NP15 OV-17: HX20/NI35/NP200 OV-225: NI75								
isazofos	C9H17ClN3O3PS	0.55	0.8	0.63	C	C #	-	50	2+3
	Responses: OV-101: NI30/NP0.4 OV-17: FP(WB)1/NI10/NP0.2 OV-225: NI20								
isocarbamid	C8H15N3O2	0.44	-	0.82	C	-	-	-	-
	Responses: OV-101: HN(WB)0.2/NP(WB)0.5 OV-17: NP7								
isofenphos	C15H24NO4PS	1.36	1.73	1.38	C	C	-	15+50	-
	Responses: OV-101: FP2/NI20 OV-17: FP2								
isofenphos oxygen analog	C15H24NO5P	1.17	1.74	1.24	C	-	-	-	-
	Responses: OV-101: FP10 OV-17: FP7 OV-225: FP5								
isopropalin	C15H23N3O4	1.14	1.24	1.01	C	C	-	6	-
	Responses: OV-101: NI2/NP3 OV-17: NI3/NP15 OV-225: NI2								
isoprothiolane	C12H18O4S2	1.6	4.1	3.17	C	-	-	-	-
	Responses: OV-101: FS(WB)10/NI(WB)3								
isoproturon	C12H18N2O	-	-	0.89	S	-	-	-	-
	Responses: OV-17: NP(WB)10								
isoxaflutole (prop)	C15H12SNO4F3	1.11	4.7	1.33	NR	V #	S #	50	3
	Responses: OV-101: NI40/NP100 OV-17: NI40/NP200 OV-225: NI40								
jodfenphos	C8H8Cl2IO3PS	-	-	2.11	C	-	-	-	-
	Responses: OV-17: FP7								
kresoxim-methyl	C18H19NO4	2	3.02	3.38	P	C	C	15+50	3
	Responses: OV-101: NI(WB)10/NP(WB)50 OV-17: NI(WB)15/NP(WB)40 OV-225: NI(WB)10								
KWG 1323	C14H16ClN3O3	0.99	1.91	0.96	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI3/NP35 OV-17: NI1/NP5 OV-225: NI3/NP65								
KWG 1342	C14H18ClN3O3	4	15	4.2	-	-	-	-	-
	Responses: OV-101: NI1000/NP1000 OV-17: NI200/NP50 OV-225: NP1000								

Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
lactofen	C19H15ClF3NO7	7.3	-	-	-	-	C	50	2+3
	Responses: OV-101: NI10								
lambda-cyhalothrin	C23H19ClF3NO3	7.4	-	8	C	-	-	-	-
	Responses: OV-101: NI10 OV-17: HX30								
leptophos	C13H10BrCl2O2PS	5.8	7.7	8.5	C	C	C	6	2
	Responses: OV-101: FP14/TI20/TR11 OV-17: FP(WB)15/NI12								
leptophos oxygen analog	C13H10BrCl2O3P	4.2	7.6	6.5	C	-	-	-	-
	Responses: OV-101: FP60/TI65								
leptophos photoproduct	C13H11Cl2O2PS	2.38	3.24	3.14	C	-	-	-	-
	Responses: OV-101: NI(WB)2/TI5 OV-17: NI5 OV-225: NI5								
lindane	C6H6Cl6	0.48	0.69	0.47	C	C	C	6	1
	Responses: OV-101: HX0.6/TR0.5 OV-17: NI0.4								
linuron	C9H10Cl2N2O2	0.85	2.13	0.95	V	V #	V	50	3
	Responses: OV-101: TR28								
malathion	C10H19O6PS2	0.91	1.49	1.05	C	C	C	15+50	3
	Responses: OV-101: NI(WB)7/NP1 OV-17: FP3/NI7 OV-225: NI44								
malathion oxygen analog	C10H19O7PS	0.68	1.55	0.87	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: TI7 OV-17: FP5								
MB45950	C12H4SN4F6Cl2	1.25	8	1.09	S	P	V	15+50	2+3
	Responses: OV-101: NI2/NP8 OV-17: NI1/NP6 OV-225: NI6								
MB46136	C12H4SO2N4F6Cl2	2.06	31	1.98	S	S	V	50	2+3
	Responses: OV-101: NI2/NP30 OV-17: NI2/NP10 OV-225: NI30								
MCPA methyl ester	C10H11ClO3	0.19	-	-	-	-	-	-	-
	Responses: OV-101: HX(WB)2/NI600								
mecarbam	C10H20NO5PS2	1.28	2.67	1.58	C		-	50	-
	Responses: OV-101: FP(WB)1.7/NI5 OV-17: FP3/FP(WB)1.9 OV-225: HN4								
mecoprop methyl ester	C11H13ClO3	0.19	-	-	-	-	-	-	-
	Responses: OV-101: HX(WB)2/NI30								
melamine	C3H6N6	0.42	-	0.42	NR	-	-	-	-
	Responses: OV-101: NP100 OV-17: NP9								
mephosfolan	C8H16NO3PS2	-	-	2.51	C	-	-	-	-
	Responses: OV-17: FP3								

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Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
merphos*	C12H27PS3	1.34 1.95	0.65 1.64	1.43 1.88	-	C	C	6+15+50	3
	Responses: OV-101: FP3/TI5 OV-17: FP25								
metalaxyl	C15H21NO4	0.81	-	0.9	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: HX1000/NI1000/NP7 OV-17: NI1000/NP50								
metamitron	C10H10N4O	2.24	-	-	-	-	-	-	-
	Responses: OV-101: NI20								
metasystox thiol	C6H15O3PS2	0.28	0.49	0.32	C	-	-	-	-
	Responses: OV-101: FP(WB)8 OV-17: FP(WB)0.8								
metasystox thiono*	C6H15O3PS2	- 0.31	- 0.49	0.18 0.32	-	-	-	-	-
	Responses: OV-101: FP6 OV-17: FP(WB)2								
metazachlor	C14H16ClN3O	1.5	-	1.46	C	-	-	-	-
	Responses: OV-101: NI34 OV-17: HX5/NP4								
methabenzthiazuron	C10H11N3OS	0.35	0.7	0.41	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: HN(WB)0.5/NI320/NP15 OV-17: NP24 OV-225: NI2550								
methamidophos	C2H8NO2PS	0.07	0.25	0.09	V	-	-	-	-
	Responses: OV-101: FP(WB)0.7 OV-17: FP1/FP(WB)0.6 OV-225: FP4								
methazole	C9H6Cl2N2O3	0.97	1.46	-	-	-	-	-	-
	Responses: OV-101: HX13/NI40								
methidathion	C6H11N2O4PS3	1.4	3.33	2.28	C	S	P #	50	3
	Responses: OV-101: FP5/FP(WB)1.6/NP3 OV-17: FP(WB)2.4/NI10 OV-225: NI50								
methidathion oxygen analog	C6H11N2O5PS2	1.07	-	1.8	-	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI100/NP40 OV-17: FP125/NI200/NP40								
methidathion sulfone	C5H8N2O3S2	0.56	2.29	0.82	-	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI150/NP100 OV-17: NI200/NP35 OV-225: NI400								
methidathion sulfoxide	C5H8N2O4S2	0.45	2.25	0.71	-	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI8/NP35 OV-17: NI8/NP10 OV-225: NI30								
methiocarb	C11H15NO2S	0.88	-	-	C	-	-	-	-
	Responses:								
methiocarb sulfone	C11H15NO4S	0.8	-	1.17	S	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI200/NP200								
methomyl	C5H10N2O2S	0.1	-	-	-	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI220								

Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
methoprotryne	C11H21N5OS	2.07	-	2.92	C	-	-	-	-
	Responses: OV-101: NP189 OV-17: NP(WB)8								
methoxychlor olefin	C16H14Cl2O2	2.97	3.7	4.2	C	C	C	6	2
	Responses: OV-101: NI(WB)8 OV-17: NI10 OV-225: NI9								
methoxychlor, o, p'-	C16H15Cl3O2	3.3	4.5	5	-	C	-	6	-
	Responses: OV-101: NI(WB)2 OV-17: NI9 OV-225: NI23								
methoxychlor, p, p'-	C16H15Cl3O2	4.7	7.2	7.2	C	C	C	6	2
	Responses: OV-101: TR9 OV-17: NI7								
methyl 2,3,5-triiodobenzoate	C8H5I3O2	2.28	-	-	-	-	-	-	-
	Responses: OV-101: NI1.5								
methyl 2,3,6-trichlorobenzoate	C8H5Cl3O2	0.23	0.21	0.18	-	-	-	-	-
	Responses: OV-101: TR0.6 OV-17: NI0.3								
methyl 3,5-dibromo-4-methoxy= benzoate	C9H8Br2O3	0.56	0.49	0.47	-	-	-	-	-
	Responses: OV-101: NI(WB)2 OV-17: NI(WB)1 OV-225: NI(WB)1.5								
methyl 4-chloro-1H-indole-3-acetate	C11H10ClNO2	1.17	-	-	R	R #	NR	50	1-2-3
	Responses: OV-101: MC25/NI200								
metobromuron	C9H11BrN2O2	0.67	1.44	0.69	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: HX8/NI5 OV-17: HX10								
metolachlor	C15H22ClNO2	1.03	1.21	0.93	C	S #	NR	50	1-2-3
	Responses: OV-101: HX5/NI12 OV-17: HX7 OV-225: NI9								
metolcarb	C9H11NO2	0.17	-	-	C	-	-	-	-
	Responses:								
metoxuron	C10H13ClN2O2	-	-	0.18	V	NR	NR	6-15-50	1-2-3
	Responses: OV-17: HX25								
metribuzin	C8H14N4OS	0.57	1.47	0.91	V	NR	NR	50	1-2-3
	Responses: OV-101: NI0.5/NP2 OV-17: NI0.4/NP11 OV-225: NI0.7								
metribuzin, deaminated diketo metabolite*	C7H11N3O2	0.5 0.73	0.79 1.29	0.44 0.52	NR	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI75 OV-17: NI150 OV-225: NI160								
metribuzin, deaminated metabolite	C8H13N3OS	0.83	3.77	1.06	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI60/NP45 OV-17: NI10/NP125 OV-225: NI60								
metribuzin, diketo metabolite	C7H12N4O2	0.56	1.41	0.55	NR	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI35/NP40 OV-17: NI6/NP20 OV-225: NI15								

Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
mevinphos, (E)-	C7H13O6P	0.16	-	0.13	C	NR	NR	6-15-50	-
	Responses: OV-101: FP2 OV-17: FP2								
mevinphos, (Z)-	C7H13O6P	0.13	-	0.15	C	NR	-	6-15-50	-
	Responses: OV-101: FP2/FP(WB)1 OV-17: FP2/FP(WB)0.6								
MGK 264	C17H25NO2	1.6	-	-	-	-	-	-	-
	Responses:								
mirex	C10Cl12	5.8	2.95	5.6	P	C	P	6	1
	Responses: OV-101: NI7 OV-17: NI4								
mirex, 5,10-dihydro-*	C10H2Cl10	2.14	-	-	-	-	-	-	-
		2.47	-	-					
		3.21	-	-					
		4.3	-	-					
	Responses: OV-101: NI100								
molinate	C9H17NOS	-	0.19	-	-	-	-	-	-
	Responses:								
monocrotophos	C7H14NO5P	0.31	1.6	0.5	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: FP(WB)0.7/TI15 OV-17: FP2/FP(WB)0.8 OV-225: FP3								
monolinuron	C9H11ClN2O2	0.48	0.91	0.48	C	-	-	-	-
	Responses: OV-101: HX4 OV-17: HX18 OV-225: NI180								
monuron	C9H11ClN2O	0.1	-	-	-	NR	NR	6-15-50	1-2-3
	Responses: OV-101: TR175								
myclobutanil	C15H17ClN4	1.9	7.2	2.6	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: HN(WB)0.7/NI25/NP(WB)7 OV-17: HX10/HX(WB)4/NI(WB)21/NP75/NP(WB)6								
myclobutanil alcohol metabolite	C15H17ClN4O	3.6	37.1	7.5	S	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI60 OV-17: NP375								
myclobutanil dihydroxy metabolite	C15H17N4O2Cl	6.5	-	11.5	NR	NR	NR	6-15-50	1-2-3
	Responses: OV-17: NI1000/NP1000								
N, N-diallyl dichloroacetamide	C8H11Cl2NO	0.19	0.22	0.14	C	S	S	15+50	2+3
	Responses: OV-101: HX1/NI1/NP1 OV-17: NI1 OV-225: NI1								
N-(3,4-dichlorophenyl)-N'-methyl= urea	C8H8Cl2N2O	0.17	0.14	0.1	-	NR	NR	6-15-50	-
	Responses: OV-101: HX20/NI9 OV-17: NI5 OV-225: NI12								
n-acetyl nitrofen	C14H11Cl2NO2	6.6	-	-	-	-	-	-	-
	Responses: OV-101: TR500								

Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
naled	C4H7Br2Cl2O4P	0.34	-	0.32	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NP11 OV-17: FP20/NI8								
napropamide	C17H20NO2	1.7	-	2.12	C	-	-	-	-
	Responses: OV-101: NP40								
neburon	C12H16Cl2N2O	0.11	-	-	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: TR25								
nitralin	C13H19N3O6S	3.8	24	6.3	C	P	P	50	3
	Responses: OV-101: NI(WB)1 OV-17: NI7 OV-225: NI21								
nitrapyrin	C6H3Cl4N	0.2	0.18	0.2	C	C	V	6	2
	Responses: OV-101: HX2/NI(WB)0.8/NP(WB)4 OV-17: HX(WB)0.6 OV-225: NI0.9								
nitrofen	C12H7Cl2NO3	2.03	3.8	2.71	C	C	C	15	2
	Responses: OV-101: NI(WB)1 OV-17: NI3 OV-225: NI3								
nitrofluorfen	C13H7ClF3NO3	0.96	1.45	0.86	C	C	C	15	2
	Responses: OV-101: HX5/NI1 OV-17: HX3 OV-225: NI1.5								
nitrothal-isopropyl	C14H17O6N	1.1	-	0.68	C	-	-	-	-
	Responses: OV-101: NI(WB)2 OV-17: NP(WB)65								
nonachlor, cis-	C10H5Cl9	2.52	3.33	2.61	C	C	C	6	1
	Responses: OV-101: HX1/NI2 OV-17: NI2 OV-225: NI3								
nonachlor, trans-	C10H5Cl9	1.75	1.45	1.42	C	C	C	6	1
	Responses: OV-101: TR2 OV-17: HX0.4/NI0.8								
norea	C13H15N2O	-	-	1.05	C	-	-	-	-
	Responses: OV-17: NP(WB)8								
norflurazon	C12H9ClF3N3O	4.5	-	5.01	V	NR	NR	6-15-50	-
	Responses: OV-101: HX50 OV-17: NP(WB)10								
NTN33823	C9H11N4Cl	3	-	1.18	-	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NP300 OV-17: NP1000								
NTN35884*	C9H9N5O2Cl	1.59	-	-	-	NR	NR	6-15-50	1-2-3
		5	-	-					
	Responses: OV-101: NI550/NP210								
nuarimol	C17H12ClFN2O	3.36	7.3	4.8	C	NR	C #	50	1-2-3
	Responses: OV-101: NI5 OV-17: HX4								
octachlor epoxide	C10H4Cl8O	1.33	0.94	1.05	C	C	C	6	1
	Responses: OV-101: TR1 OV-17: NI0.6								

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Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
octachlorocyclopentane	C5Cl8	-	-	0.22	-	-	-	-	-
	Responses: OV-17: NI0.2								
octhilinone	C11H19NOS	0.66	-	0.64	C	-	-	-	-
	Responses: OV-101: FS4								
ofurace	C14H16NO3Cl	2.62	18.6	5.4	C	-	-	-	-
	Responses: OV-101: HX17 OV-17: HX51 OV-225: NI44								
omethoate	C5H12NO4PS	0.25	1.11	0.39	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: FP(WB)0.9/TI25 OV-17: FP5/FP(WB)1.1 OV-225: FP5								
oryzalin	C12H8N4O6S	4.7	-	-	-	NR	NR	6-15-50	-
	Responses: OV-101: NI8800								
ovex	C12H8Cl2O3S	1.61	3.04	2.2	C	C	C	15	2
	Responses: OV-101: HX4/TR3 OV-17: HX5 OV-225: NI3								
oxadiazon	C15H18Cl2N2O3	1.97	2.48	1.96	C	C	P	15	-
	Responses: OV-101: HX4/NI4 OV-17: NI2								
oxadixyl	C14H18N2O4	2.5	14	5	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI1700/NP8 OV-17: NP14 OV-225: NI4500								
oxamyl	C7H13N3O3S	-	-	-	-	-	-	-	-
	Responses:								
oxamyl oxime metabolite	C5H10N2O2S	0.25	0.92	0.28	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: HN(WB)0.5/NI(WB)4/NP(WB)4 OV-17: HN(WB)0.4/NI(WB)3/NP(WB)3 OV-225: NI(WB)13								
oxycarboxin	C12H13NO4S	3.28	-	9.4	R	-	-	-	-
	Responses: OV-101: FS(WB)17/HN(WB)3/NP(WB)130 OV-17: HN(WB)4								
oxydemeton-methyl	C6H15O4PS2	0.46	0.49	0.31	C	-	-	-	-
	Responses: OV-17: FP(WB)4								
oxydemeton-methyl sulfone	C6H15O5PS2	0.72	-	1.48	C	-	-	-	-
	Responses: OV-101: FP(WB)2 OV-17: FP(WB)3								
oxydeprofos	C7H17O4SP	-	-	-	-	-	-	-	-
	Responses:								
oxyfluorfen	C15H11ClF3NO4	2	4	2.16	C	C	C	15	2
	Responses: OV-101: NI5 OV-17: HX9/NI2/NP350								
oxythioquinox	C10H6N2OS2	1.57	-	1.85	C	-	-	-	-
	Responses: OV-101: NI(WB)1								

Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
paclobutrazol	C15H20ClN3O	1.52	-	1.59	C	-	-	-	-
	Responses: OV-101: NP(WB)6 OV-17: HX7/HX(WB)3/NI(WB)85/NP(WB)5								
parathion	C10H14NO5PS	0.98	1.91	1.07	C	C	C	15	2
	Responses: OV-101: NI(WB)4/NP2 OV-17: FP2/NI4 OV-225: FP2/NI6								
parathion oxygen analog	C10H14NO6P	0.8	-	0.86	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI(WB)5/NP3 OV-17: FP4/NI15								
parathion-methyl	C8H10NO5PS	0.71	1.64	0.87	C	C	C	15	2
	Responses: OV-101: FP(WB)0.9/NI(WB)3/NP1.5 OV-17: FP2/FP(WB)1/NI3 OV-225: FP1/NI11								
parathion-methyl oxygen analog	C8H10NO6P	0.55	1.71	0.66	-	NR	NR	6-15-50	1-2-3
	Responses: OV-101: TI5/TR11 OV-17: FP10								
PB-7, methylated	C20H25ClN2O3S	23	57	43	-	-	-	-	-
	Responses: OV-101: NI150/NP300 OV-17: NI200/NP500 OV-225: NI300								
PB-9	C19H25ClN2O2S	25	87	46	V	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI300/NP500 OV-17: NI250/NP750 OV-225: NI500								
pebulate	C10H21NOS	0.17	-	0.1	C	P	-	15	-
	Responses: OV-101: HN(WB)0.4/NP7 OV-17: NP6								
penconazole	C13H15Cl2N3	1.24	-	1.32	C	-	-	-	-
	Responses: OV-101: NP(WB)3 OV-17: HX3/HX(WB)2/NI(WB)2/NP(WB)3								
pendimethalin	C13H19N3O4	1.22	1.48	1.21	C	C	P	15	2
	Responses: OV-101: NI3/NP5 OV-17: NI1.5/NP6 OV-225: NI3								
pentachloroaniline	C6H2Cl5N	0.67	0.79	0.66	C	C	C	6	1
	Responses: OV-101: HX0.5/NI0.4/NP10 OV-17: NI0.6/NP10 OV-225: NI0.5								
pentachlorobenzene	C6HCl5	0.24	0.13	0.16	C	C	C	6	1
	Responses: OV-101: HX0.3/NI0.25 OV-17: NI0.3 OV-225: NI0.25								
pentachlorobenzonitrile	C7Cl5N	0.5	0.59	0.45	C	C	P	15	2
	Responses: OV-101: NI0.5 OV-17: NI3								
pentachlorophenyl methyl ether	C7H3Cl5O	0.46	0.3	0.34	C	C	C	6	1
	Responses: OV-101: HX0.4/TR0.4 OV-17: NI0.3								
pentachlorophenyl methyl sulfide	C7H3Cl5S	0.94	0.69	0.87	C	C	C	6	1
	Responses: OV-101: HX0.7/NI0.4 OV-17: NI3 OV-225: NI0.5								
permethrin, cis-	C21H20Cl2O3	9.4	11.1	13.8	C	V #	C	6+15	2
	Responses: OV-101: NI75								

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Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
permethrin, trans-	C21H20Cl2O3	10.2	13	15	C	V #	C	6+15	2
	Responses: OV-101: NI100								
Perthane	C18H2OCl2	2.23	2.01	2.42	C	C	C	6	1
	Responses: OV-101: TR150 OV-17: NI25								
Perthane olefin	C18H19Cl	1.53	0.95	-	-	C	C	6	1
	Responses: OV-101: TR40								
phenmedipham	C16H16N2O4	0.32	-	0.41	-	-	-	-	-
	Responses: OV-101: HN(WB)1.5 OV-17: NP40								
phenothiazine	C12H9NS	1.16	-	1.56	-	-	-	-	-
	Responses: OV-101: HN(WB)1.4 OV-17: NP4								
phenothrin*	C23H26O3	5.4 11.5	4.8 10.9	6.5 15	-	-	-	-	-
	Responses: OV-101: NI500/NI(WB)40								
phenthoate	C12H17O4PS2	1.31	2.05	1.83	C	C	-	15+50	-
	Responses: OV-101: FP(WB)2/NI5/TI3 OV-17: FP2/FP(WB)2.6/NI5								
phorate	C7H17O2PS3	0.37	0.38	0.32	C	V #	V #	6	1
	Responses: OV-101: FP1/NI(WB)24/TI1 OV-17: FP0.5/NI14 OV-225: FP0.5/NI17								
phorate oxygen analog	C7H17O3PS2	0.3	0.37	0.29	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI400/TI2 OV-17: FP1 OV-225: FP0.5								
phorate oxygen analog sulfone	C7H17O5PS2	0.66	-	1.02	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: FP1/TI10 OV-17: FP6								
phorate oxygen analog sulfoxide	C7H17O4PS2	0.78	-	1.06	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI300 OV-17: FP40								
phorate sulfone	C7H17O4PS3	0.97	3.26	1.3	C	S #	S #	6-15-50	3
	Responses: OV-101: NI4/TI4 OV-17: FP2								
phorate sulfoxide	C7H17O3PS3	0.89	2.55	1.26	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: FP5/NI8/NP4 OV-17: FP6								
phosalone	C12H15ClNO4PS2	5.5	5.5	9.1	C	C	C	50	2+3
	Responses: OV-101: NP10/TR15 OV-17: NI12								
phosalone oxygen analog	C12H15ClNO5PS	3.8	-	6.2	C	-	-	-	-
	Responses: OV-101: FP600/FP(WB)8/NI160 OV-17: FP(WB)12								
phosfolan	C7H14NO3PS2	-	-	2.69	C	-	-	-	-
	Responses: OV-17: FP5								

Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
phosmet	C11H12O4NPS2	4	14.9	8.4	C	NR	-	6-15-50	3
	Responses: OV-101: NI(WB)2/NP19 OV-17: FP50/NI78 OV-225: FP50								
phosmet oxygen analog*	C11H12NO5PS	0.5	0.53	0.24	-	NR	NR	6-15-50	-
		2	0.93	0.44					
		3.1	14.8	7.1					
	Responses: OV-101: NI900/NP100 OV-17: FP150 OV-225: NI600								
phosphamidon*	C10H19ClNO5P	0.53	-	0.57	C	NR	NR	6-15-50	1-2-3
		0.67	-	0.76					
	Responses: OV-101: FP10 OV-17: FP2								
photodieldrin	C12H8Cl6O	4.4	15.5	8.5	-	C	C	15+50	2
	Responses: OV-101: TR6 OV-17: NI5								
photodieldrin B	C13H9Cl5O	1.43	-	-	-	-	-	-	-
	Responses: OV-101: TR2								
phoxim oxygen analog	C12H15N204P	0.86	-	-	C	-	-	-	-
	Responses: OV-101: FP5								
picloram methyl ester	C7H5Cl3N2O2	0.75	2.67	-	-	-	-	-	-
	Responses: OV-101: HX(WB)1.2/NI1.5/TR1								
picloram*	C6H3Cl3N2O2	0.25	-	-	-	-	-	-	-
		0.67	-	-					
	Responses: OV-101: NI100								
piperophos	C14H28NO3PS2	4.8	9.7	6.8	C	-	-	-	-
	Responses: OV-101: FP15								
pirimicarb	C11H18N4O2	0.61	-	0.73	C	-	-	-	-
	Responses: OV-17: FP(WB)2								
pirimiphos-ethyl	C13H24N3O3PS	1.14	1.03	1.14	C	C	C	15+50	3
	Responses: OV-101: FP2/TI4/TR150 OV-17: FP3/NI4								
pirimiphos-ethyl oxygen analog	C13H24N3O4P	1.01	-	1.14	C	-	-	-	-
	Responses: OV-101: FP4/FP(WB)1.6 OV-17: FP(WB)2.4								
pirimiphos-methyl	C11H20N3O3PS	0.87	-	0.92	C	C	C	15	3
	Responses: OV-101: FP2/FP(WB)1.2/NI100/NP2 OV-17: FP2/FP(WB)1.2								
PP 890	C9H10O2ClF3	1.05	1	-	-	-	-	-	-
	Responses: OV-101: NI220 OV-225: NI150								
PPG-1576	C19H17ClF3NO5	6.7	-	-	-	-	P	50	2+3
	Responses: OV-101: NI13								

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Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
PPG-2597	C20H17ClF3NO6	1.9	3.16	1.86	-	NR	NR	6-15-50	1-2-3
	Responses: OV-101: HN(WB)58/HX(WB)110/NI(WB)85/NP(WB)150 OV-17: HN(WB)120/HX(WB)66/NI(WB)78 OV-225: NI(WB)120								
PPG-847, methylated	C15H9ClF3NO3	2.15	5	2.4	-	-	-	-	-
	Responses: OV-101: HN(WB)3/HX(WB)10/NI(WB)3/NP(WB)34 OV-17: HN(WB)4/HX(WB)6.5/NI(WB)3/NP(WB)210 OV-225: NI(WB)4								
PPG-947	C17H11ClF3NO7	1.04	-	1.13	-	NR	NR	6-15-50	1-2-3
	Responses: OV-101: HN(WB)620/NI(WB)1000 OV-17: NI(WB)600								
PPG-947, methylated*	C18H13ClF3NO7	0.42 2.14	0.97 5	0.49 2.4	-	-	-	-	-
	Responses: OV-101: HN(WB)5/NI(WB)5/NP(WB)65 OV-17: HN(WB)5 OV-225: NI(WB)6								
pretilachlor	C17H26ClNO2	1.88	-	1.99	C	-	-	-	-
	Responses: OV-101: NI25 OV-17: HX10								
probenazole	C10H9NO3S	-	-	-	C	-	-	-	-
	Responses:								
prochloraz	C15H16Cl3N3O2	10.4	-	15.4	C	-	-	-	-
	Responses: OV-101: HX50/NI12								
procyazine	C10H13ClN6	1.5	7.9	-	C	-	-	-	-
	Responses:								
procymidone	C13H11Cl2NO2	1.37	3.04	1.49	C	C	P	15	-
	Responses: OV-101: NI12 OV-17: HX1 OV-225: NI7								
prodiamine	C13H17F3N4O4	0.94	0.97	0.66	C	-	-	-	-
	Responses: OV-17: NP(WB)50 OV-225: NP50								
profenofos	C11H15BrClO3PS	1.8	2.34	2.13	C	P	P	50	3
	Responses: OV-101: FP7/FP(WB)2.6/NI3 OV-17: FP5/FP(WB)2.9								
profluralin	C14H16F3N3O4	0.53	0.46	0.3	V	V	-	6	-
	Responses: OV-101: HX5/NI1/NP1 OV-17: NI0.8/NP9 OV-225: NI1								
Prolan	C15H13Cl2NO2	2.81	7.5	3.9	P	S	S	15	2
	Responses: OV-101: NI(WB)1 OV-17: NI6 OV-225: NI10								
promecarb	C12H17NO2	1.58	-	-	V	-	-	-	-
	Responses: OV-101: TI400								
prometryn	C10H19N5S	0.77	-	0.74	C	P #	P #	50	1-2-3
	Responses: OV-101: FP200/NI40/TI50 OV-17: FP20								

Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
pronamide	C12H11Cl2NO	0.51	0.84	0.4	C	P	-	15+50	-
	Responses: OV-101: HX1/NI2/NP7 OV-17: NI1/NP85 OV-225: NI3								
propachlor	C11H14ClNO	0.34	0.37	0.26	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI(WB)16/NP(WB)13 OV-17: NI5/NI(WB)5 OV-225: NI9/NI(WB)5								
propanil	C9H9Cl2NO	0.66	2.82	0.78	C	NR	NR	6-15	3
	Responses: OV-101: HX6/NI3/NI(WB)5 OV-17: NI4 OV-225: NI8								
propargite	C19H26O4S	3.8	4.8	4.3	C	C	-	15	2
	Responses: OV-101: FS45/NI(WB)230 OV-17: NI2600 OV-225: NI1300								
propazine	C9H16ClN5	0.53	0.65	0.41	C	S	NR	15+50	3
	Responses: OV-101: NI(WB)110/TI30 OV-17: NI43 OV-225: NI37								
propetamphos	C10H20NO4PS	0.48	-	0.42	C	C #	-	15+50	2+3
	Responses: OV-101: FP1.5 OV-17: FP0.5								
propham	C10H13NO2	0.13	-	0.12	C	P	P	15	-
	Responses: OV-101: NP(WB)2 OV-17: NP16								
propiconazole*	C15H17Cl2N3O2	3.06 3.21	- 5.6	- 4	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI10/NI(WB)17 OV-17: HX9								
propoxur	C11H15NO3	-	-	-	C	-	-	-	-
	Responses:								
prosulfuron*	C15H16F3N5O4S	0.18 0.44	- 2.49	- 0.64	-	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NP8 OV-17: NI120 OV-225: NI20								
prothiofos	C11H15Cl2PO2S2	1.85	1.74	1.82	C	C	C	6	2
	Responses: OV-101: FP4/NI3/NP3 OV-17: NI3 OV-225: NI1								
prothoate	C9H20NO3PS2	0.75	1.55	0.79	C	-	-	-	-
	Responses: OV-101: FP1/NI5								
pyracarbolid	C13H15NO2	1.05	-	1.43	-	-	-	-	-
	Responses: OV-101: HN(WB)1.4 OV-17: NP6								
pyrazon	C10H8ClN3O	2.67	13	8	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: HN(WB)8/HX(WB)30/NI(WB)26/NP(WB)56 OV-17: HX(WB)50 OV-225: NI3700								
pyrazon metabolite A	C16H18ClN3O6	2.62	-	-	-	-	-	-	-
	Responses: OV-101: HX(WB)260								
pyrazon metabolite B	C6H4ClN3O	0.42	-	0.9	-	NR	NR	6-15-50	1-2-3
	Responses: OV-101: HN(WB)3/HX(WB)42/NI(WB)35/NP(WB)14 OV-17: HN(WB)3/HX(WB)23/NI(WB)23/NP(WB)44								

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Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
pyrazophos	C14H20N3O5PS	8.1	-	13	C	-	-	-	-
	Responses: OV-101: FP(WB)12 OV-17: FP25								
pyrethrins*	C21H27O4	2.12	1.76	1.76	-	C	C	50	-
		2.95	2.84	2.7					
	Responses: OV-101: NI25								
pyridaphenthion	C14H17O4N2SP	4.2	14	8.7	C	-	-	-	-
	Responses: OV-101: NI(WB)16								
pyrimethanil	C12H13N3	0.67	-	-	C	S	S #	50	3
	Responses: OV-101: NP(WB)0.5								
pyrithiobac-sodium methyl ester	C14H13ClN2O4	2.51	4	4.2	-	-	-	-	-
	Responses: OV-101: HN(WB)1/HX(WB)12/NI(WB)9/NP(WB)90 OV-17: HN(WB)2/HX(WB)13/NI(WB)18/NP(WB)85 OV-225: NI(WB)13								
quinalphos	C12H15N2O3PS	1.32	2	1.64	C	C	-	15	-
	Responses: OV-101: FP5/FP(WB)2.7 OV-17: FP3/FP(WB)3								
quintozene	C6Cl5NO2	0.51	0.46	0.46	C	C	C	6	1
	Responses: OV-101: TR0.3 OV-17: NI0.5								
quizalofop ethyl ester	C19H17ClN2O4	13.6	-	25	C	-	-	-	-
	Responses: OV-101: HX70/NI80								
RH-6467*	C19H15N4ClO	7.9	-	-	S	NR	NR	6-15-50	1-2-3
		10.4	-	-					
		15	-	-					
	Responses: OV-101: NI90/NP300								
RH-9129	C19H16N3ClO2	14	-	-	V	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI40/NP190								
RH-9130	C19H16N3ClO2	12	-	-	P	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI50/NP170								
ronnel	C8H8Cl3O3PS	0.81	0.86	0.76	C	C	C	6	2
	Responses: OV-101: NI(WB)1/TI3 OV-17: FP1/NI1 OV-225: NI2								
ronnel oxygen analog	C8H8Cl3O4P	0.64	1.02	0.62	C	NR	-	6-15-50	-
	Responses: OV-101: NP3 OV-17: FP5/FP(WB)1								
RPA 203328, methylated	C10H9F3O4S	0.26	0.51	0.23	-	-	-	-	-
	Responses: OV-101: NI0.5 OV-17: NI0.4 OV-225: NI0.5 5								
RPA202248	C15H12SNO4F3	1.13	4.7	1.38	NR	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI40/NP120 OV-17: NI40/NP250 OV-225: NI60								

Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
S-bioallethrin	C19H26O3	1.37	1.22	1.12	-	C	-	50	-
	Responses: OV-101: NI2 OV-17: NI2 OV-225: NI2								
schradan	C8H24N4O3P2	0.58	-	0.52	C	NR	-	6-15-50	-
	Responses: OV-101: FP4/TI5 OV-17: FP1								
sethoxydim	C17H29NO3S	3.34	-	-	-	NR	NR	6-15-50	3
	Responses: OV-101: NI50								
sethoxydim sulfoxide	C17H29NO4S	0.84	-	-	-	NR	NR	6-15-50	3
	Responses: OV-101: NI50								
silvex	C9H7Cl3O3	0.48	-	-	-	-	-	-	-
	Responses: OV-101: NI40								
silvex methyl ester	C10H9Cl3O3	0.45	0.44	-	-	-	-	-	-
	Responses: OV-101: HX(WB)0.8/NI0.6								
simazine	C7H12ClN5	0.41	0.83	0.5	C	NR	NR	50	1-2-3
	Responses: OV-101: NI(WB)90 OV-17: NI56/NP(WB)1.5 OV-225: NI130								
simetryn	C8H15N5S	2.02	1.21	-	C	-	-	-	-
	Responses: OV-101: NP7								
Strobane*	C10H11Cl7	1.09	-	-	-	C	C	6	1
		1.32	-	-					
		1.53	-	-					
		1.8	-	-					
		1.94	-	-					
		2.09	-	-					
		2.33	-	-					
		2.69	-	-					
		3.1	-	-					
		3.7	-	-					
	Responses: OV-101: TR40								
sulfallate	C8H14ClNS2	0.38	0.44	0.36	C	C	C	6+15	2
	Responses: OV-101: NI(WB)1 OV-17: NI1 OV-225: NI1								
sulfanilamide	C6H8O2N2S	-	-	2.11	NR	NR	NR	6-15-50	1-2-3
	Responses: OV-17: NP200								
sulfotep	C8H20O5P2S2	0.34	-	0.29	C	C	P	6+15	2
	Responses: OV-101: TI0.8 OV-17: FP0.5								
Sulphenone	C12H9ClO2S	1.26	3.5	1.92	C		-	50	3
	Responses: OV-101: TR4								

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Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
sulprofos	C12H19O2PS3	2.79	-	3.5	C	-	-	-	-
	Responses: OV-101: FP(WB)47 OV-17: FP3								
sulprofos oxygen analog sulfone	C12H19O5PS2	5.1	-	10.6	C	-	-	-	-
	Responses: OV-101: FP(WB)80 OV-17: FP40/FP(WB)28/NI20/NP40								
sulprofos sulfone	C12H19O4PS3	7.2	-	13.1	C	-	-	-	-
	Responses: OV-101: FP(WB)16 OV-17: FP(WB)26								
sulprofos sulfoxide*	C12H19O3PS3	2.78	-	3.6	C	-	-	-	-
		6.1	-	11.7					
	Responses: OV-101: FP(WB)14 OV-17: FP(WB)29								
TCMTB	C9H6N2S3	1.5	4.3	2.67	C	P	P	15	-
	Responses: OV-101: NI3/NP22 OV-17: NP36 OV-225: NI12								
TDE, o,p'-	C14H10Cl4	1.9	2.46	2.19	-	C	C	6	1
	Responses: OV-101: TR2 OV-17: NI2								
TDE, o,p'-, olefin	C14H9Cl3	1.19	1.15	1.2	-	-	-	-	-
	Responses: OV-101: TR12 OV-17: NI2								
TDE, p,p'-	C14H10Cl4	2.41	3.8	2.87	C	C	C	6	1
	Responses: OV-101: TR4 OV-17: NI2								
TDE, p,p'-, olefin	C14H9Cl3	1.45	1.36	1.45	C	C	C	6	1
	Responses: OV-101: NI(WB)4 OV-17: NI4 OV-225: NI3								
tebuconazole	C16H22ClN3O	3.38	-	4.2	C	-	-	-	-
	Responses: OV-101: HX(WB)1 OV-17: HX(WB)3								
tebufenozide	C22H28N2O2	7	-	11	-	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI30/NP4000								
tebupirimfos	C13H23N2O3PS	0.63	-	-	-	V	V	6+15	2+3
	Responses: OV-101: NI16/NP1								
tebupirimfos oxygen analog	C13H23O4N2P	0.51	-	-	-	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI100/NP1								
tebuthiuron	C9H16N4OS	0.26	-	0.21	-	-	-	-	-
	Responses: OV-101: NI1000/NP(WB)0.5 OV-17: FP20								
tecnazene	C6HCl4NO2	0.29	0.26	0.24	C	C	C	6	1
	Responses: OV-101: TR0.5 OV-17: NI0.3								
teflubenzuron*	C14H6Cl2F4N2O2	-	0.11	0.09	-	NR	NR	6-15-50	1-2-3
		0.14	0.23	0.14					
	Responses: OV-101: NI(WB)1.5 OV-17: NP(WB)5								

Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
TEPP	C8H20O7P2	0.21	0.27	0.24	C	-	-	-	-
	Responses: OV-101: FP(WB)46/NI(WB)4000 OV-17: FP(WB)70/NI10000/NP(WB)70 OV-225: NI13700								
terbacil	C9H13ClN2O2	0.54	2.1	0.72	C	NR	NR	6-15	2+3
	Responses: OV-101: HN(WB)1/HX5/NI(WB)6/NP(WB)8 OV-17: HN(WB)0.6/HX(WB)5/NI(WB)2/NP(WB)8 OV-225: NI(WB)7								
terbufos	C9H21O2PS3	0.5	0.44	0.41	C	P	S	6	-
	Responses: OV-101: FP2/NI40/TI2 OV-17: FP0.5/FP(WB)1.6/NI20								
terbufos oxygen analog	C9H21O3PS2	0.42	-	0.39	C	-	NR	6-15-50	1-2-3
	Responses: OV-101: FP0.5/NI1000 OV-17: FP1/NI2500								
terbufos oxygen analog sulfone	C9H21O5PS2	0.92	2.9	1.28	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: FP7/NI40 OV-17: FP2/NI60 OV-225: FP5/NI1500								
terbufos sulfone	C9H21O4PS3	1.2	-	1.58	C	C #	C #	6-15-50	2+3
	Responses: OV-101: FP2/NI5 OV-17: FP2/NI10								
terbumeton	C10H19N5O	0.47	0.42	0.53	C	-	-	-	-
	Responses: OV-17: NP(WB)25 OV-225: NP20								
terbuthylazine	C9H16N5Cl	0.47	0.71	0.48	C	P	-	15+50	-
	Responses: OV-101: NI(WB)87/NP(WB)6 OV-17: NI250 OV-225: NI43								
terbutryn	C10H19N5S	0.84	1.08	-	C	-	-	-	-
	Responses:								
tetradifon	C12H6Cl4O2S	5.2	-	8.3	C	C	C	15	2
	Responses: OV-101: TR6 OV-17: NI5								
tetraiodoethylene	C2I4	0.55	0.86	1.04	-	P	P	6	-
	Responses: OV-101: NI(WB)3 OV-17: NI4								
tetramethrin*	C19H25NO4	4.3	-	-	C	NR	NR	6-15-50	1-2-3
		4.5	8.5	7.2					
	Responses: OV-101: NI50								
tetrasul	C12H6Cl4S	2.64	2.33	2.8	C	C	C	6	1
	Responses: OV-101: NI(WB)1 OV-17: NI5 OV-225: NI3								
tetrasul sulfoxide	C12H6Cl4OS	4.7	8.6	7.2	-	-	-	-	-
	Responses: OV-101: NI(WB)1 OV-17: NI8 OV-225: NI7								
thiabendazole	C10H7N3S	1.48	-	2.04	C	NR	-	6-15-50	-
	Responses: OV-101: NP90								

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Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
thiobencarb	C12H16ClNOS	0.94	1	0.98	C		V	15	2+3
	Responses: OV-101: NI73 OV-17: HX3								
thiometon	C6H15O2PS3	0.41	0.51	0.4	C	NR	NR	6-15-50	-
	Responses: OV-101: NI10/NI(WB)3/TI1 OV-17: FP0.4/NP(WB)1								
thionazin	C8H13N2O3PS	0.26	-	0.26	C	P	NR	15+50	-
	Responses: OV-101: FP0.5/TI1 OV-17: FP0.5								
thionazin oxygen analog	C8H13N2O4P	-	-	-	-	-	-	-	-
	Responses:								
thiophanate-methyl	C12H14N4O4S2	-	-	-	-	-	-	-	-
	Responses:								
THPI	C8H9NO2	0.21	-	-	C	NR	NR	6-15-50	-
	Responses:								
tolylfluanid	C10H13ClFNOS	1.25	-	1.41	C	-	-	-	-
	Responses: OV-101: NI3/NP70								
toxaphene*	C10H10Cl8	-	2.6	-	C	C	C	6	1
		-	2.35	-					
		-	2.14	-					
		-	1.75	-					
		1.2	2.74	-					
		1.54	3.05	-					
		1.8	3.9	-					
		2.39	4.3	-					
		2.68	4.5	-					
		3.12	5.2	-					
		3.7	5.6	-					
		4.4	6	-					
		4.6	6.4	-					
		5.1	7	-					
	Responses: OV-101: TR30								
tralkoxydim*	C20H27NO3	3.32	-	-	V	NR	NR	50	1-2-3
		6.1	1.48	4.5					
	Responses: OV-101: NI100/NP10 OV-17: NI100 OV-225: NI100								
tralomethrin	C22H19Br4NO3	27	64	44	C	V	S	15	2
	Responses: OV-101: NI30/NI(WB)1								
tri-allate	C10H16Cl3NOS	0.6	-	0.45	C	C	C	6	2
	Responses: OV-101: HX1/NI1.5								

Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
triadimefon	C14H16ClN3O2	1.05	1.64	1	C	S #	S #	50	1-2-3
Responses: OV-101: HX3/NI2/NP20/NP(WB)2 OV-17: HX5/HX5/HX(WB)3/NI(WB)2/NP2/NP(WB)3 OV-225: NI3/NP20									
triadimenol	C14H18ClN3O2	1.36	-	1.44	C	NR	NR	6-15-50	-
Responses: OV-101: HX10/NI90/NP30/NP(WB)5 OV-17: HX7/HX(WB)4/NI(WB)50/NP(WB)6									
triazamate	C13H22N4O3S	1.58	2.46	2.1	C	NR	NR	6-15-50	1-2-3
Responses: OV-101: NI15/NP25									
triazophos	C12H16N3O3PS	2.62	-	5.2	C	-	-	-	-
Responses: OV-101: FP(WB)3 OV-17: FP5/FP(WB)5									
tribufos	C12H27OPS3	1.95	1.65	1.88	C	C	P	15+50	3
Responses: OV-101: FP3/NP3/TR4 OV-17: FP3/NI3									
tributyl phosphate	C12H27O4P	0.3	-	0.23	-	R	-	50	-
Responses:									
trichlorfon	C4H8Cl3O4P	0.16	-	0.13	C	NR	NR	6-15-50	1-2-3
Responses: OV-101: NP3 OV-17: FP4									
trichloronat	C10H12Cl3O2PS	1.13	-	0.97	C	C	-	6	-
Responses: OV-101: FP3/HX2/NI2/NP3									
triclopyr methyl ester	C8H6Cl3NO3	0.36	0.36	0.39	-	-	-	-	-
Responses: OV-101: NI0.5/NI(WB)0.7/NP(WB)7									
tricyclazole	C9H7N3S	1.59	-	3.9	C	-	-	-	-
Responses: OV-101: NP500/NP(WB)8 OV-17: NP500/NP(WB)8									
tridiphane	C10H7Cl5O	0.81	0.85	0.75	C	C	-	6	1+2
Responses: OV-101: HX(WB)1/NI0.8 OV-17: HX0.8/HX(WB)0.4 OV-225: NI1									
triflumizole	C15H15ClF3N3O	1.44	2.23	1.19	C	-	-	-	-
Responses: OV-101: NP(WB)4 OV-17: HX13/HX(WB)2/NI(WB)3/NP(WB)3									
trifluralin	C13H16F3N3O4	0.34	0.27	0.17	C	C	C	6	2
Responses: OV-101: TR1 OV-17: NI0.7									
triflusulfuron methyl ester	C17H19F3N6O6S	0.3	-	0.2	V	NR	NR	6-15-50	1-2-3
Responses: OV-101: HN(WB)1/HX(WB)20/NI(WB)500/NP(WB)13 OV-17: HN(WB)1/HX(WB)35/NP(WB)11									
tris(beta-chloroethyl) phosphate	C6H12Cl3O4P	0.45	-	-	C	-	-	-	-
Responses: OV-101: FP1									
tris(chloropropyl) phosphate	C9H18Cl3O4P	0.5	2.02	0.5	C	NR	NR	6-15-50	1-2-3
Responses: OV-101: FP1									

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Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
Tycor	C9H16N4OS	0.77	1.62	0.99	C	S	S	50	3
Responses: OV-101: NI1/NP2 OV-17: NI1/NP9 OV-225: NI1									
vamidothion sulfone	C8H18NO6PS2	2.19	-	-	C	-	-	-	-
Responses:									
vernolate	C10H21NOS	0.15	-	0.09	-	P	-	15	-
Responses: OV-101: TI50 OV-17: FP11									
vinclozolin	C12H9Cl2NO3	0.69	1.15	0.64	C	C	C	15	2
Responses: OV-101: HX1.5/NI1 OV-17: HX1/NI2									
vinclozolin metabolite B	C12H11Cl2NO4	0.74	1.2	0.66	C	P #	C	6+15	2
Responses: OV-101: HN(WB)5/HX(WB)18/NI(WB)5/NP(WB)84 OV-17: HN(WB)6/HX(WB)9/NI(WB)10/NP(WB)19 OV-225: NI(WB)8									
vinclozolin metabolite E	C11H11Cl2NO2	0.89	3.02	0.93	C	S	NR	15+50	-
Responses: OV-101: HN(WB)3/HX(WB)3/NI(WB)3/NP(WB)15 OV-17: HN(WB)3/HX(WB)3/NI(WB)4/NP(WB)7 OV-225: NI(WB)9									
vinclozolin metabolite F	C11H13Cl2NO4	2.87	-	4.6	R	NR	NR	6-15-50	1-2-3
Responses: OV-101: HX(WB)160/NI(WB)57/NP(WB)140 OV-17: HN(WB)100/HX(WB)14/NI(WB)85/NP(WB)210									
vinclozolin metabolite S	C10H7Cl2NO3	0.69	2.01	0.79	V	P	V #	15	2
Responses: OV-101: HN(WB)1/HX(WB)2/NI(WB)1/NP(WB)19 OV-17: HN(WB)1/HX(WB)2/NI(WB)1/NP(WB)7 OV-225: NI(WB)2									
zoxamide*	C14H16NO2Cl3	1.44 3.9	-	-	C	C	-	50	3
Responses: OV-101:NI(WB)0.8/NP(WB)18/HX(WB)5									

Appendix I: PESTDATA Chemicals in Order by OV-101 Relative Retention Time

RRT/c OV-101	Detector Responses with OV-101 Column	RRT/c OV-225	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
0.03	TR37	-	0.02	dichlorobenzene, p-	C6H4Cl2	-	C	C	6	1
0.04	TR0.6	-	0.03	dibromochloropropane	C3H5Br2Cl	-	-	-	-	-
0.06	NI30	0.08	0.04	CGA 171683	C6H5F4N3O2	C		-	15+50	3
0.06	FS9	-	-	ethiolate	C7H15NOS	C	-	-	-	-
0.07	FP(WB)0.7	0.25	0.09	methamidophos	C2H8NO2PS	V	-	-	-	-
0.07	FP9/NI1/TI0.5	0.08	0.08	dichlorvos	C4H7Cl2O4P	C	NR	NR	6-15-50	1-2-3
0.08	TR2	-	-	1,2,3-trichlorobenzene	C6H3Cl3	-	C	P	6	1
0.09	TR5	-	-	allidochlor	C8H12ClNO	C	NR	-	6-15	1-2-3
0.1	NI220	-	-	methomyl	C5H10N202S	-	NR	NR	6-15-50	1-2-3
0.1	TR175	-	-	monuron	C9H11ClN2O	-	NR	NR	6-15-50	1-2-3
0.11	TR0.5	-	0.1	dichlobenil	C7H3Cl2N	C	P	C	15	2
0.11	TR25	-	-	neburon	C12H16Cl2N2O	C	NR	NR	6-15-50	1-2-3
0.11	NI(WB)9	0.09	0.11	diuron	C9H10Cl2N2O	C	NR	NR	6-15-50	1-2-3
0.12	TI30	-	-	EPTC	C9H19NOS	-	P	-	15	-
0.12	TR0.4	-	0.06	hexachlorocyclopentadiene	C5Cl6	-	-	-	-	-
0.13	NI14/NI(WB)24/NP35	0.15	0.1	chlorimuron ethyl ester	C15H15ClN4O6S	P	NR	-	-	-
0.13	FS10	0.23	0.11	carboxin sulfoxide	C12H13NO3S	-	NR	NR	6-15-50	1-2-3
0.13	FP2/FP(WB)1	-	0.15	mevinphos, (Z)-	C7H13O6P	C	NR	-	6-15-50	-
0.13	NP(WB)2	-	0.12	propham	C10H13NO2	C	P	P	15	-
0.14	NI(WB)1.5	0.23	0.14	teflubenzuron*	C14H6Cl2F4N2O2	-	NR	NR	6-15-50	1-2-3
0.15	FP(WB)0.9/NP3	0.64	0.19	acephate	C4H10NO3PS	C	-	-	-	-
0.15	TR7	0.24	-	hydroxy chloroneb	C7H6Cl2O2	-	NR	-	6-15	-
0.15	TI50	-	0.09	vernolate	C10H21NOS	-	P	-	15	-
0.15	NI300	0.15	0.14	dimethyl phthalate	C10H10O4	-	P	-	6+15+50	-
0.16	TR35	0.14	-	2,4,5-T BEP ester*	C17H23Cl3O3	-	-	-	-	-
0.16	FP2	-	0.13	mevinphos, (E)-	C7H13O6P	C	NR	NR	6-15-50	-
0.16	NP3	-	0.13	trichlorfon	C4H8Cl3O4P	C	NR	NR	6-15-50	1-2-3
0.17	NP200	-	0.11	CGA 236431	C8H7F3N2O2	-	-	-	-	-
0.17	NI3/NP7	0.22	0.13	3-methyl-4-nitrophenol methyl ether	C8H9O3N	-	-	-	-	-

* Multipeak chemical.

Recovery may vary with choice of Florisil elution system; see Tables 303-a, 304-a.

Appendix I: PESTDATA Chemicals in Order by OV-101 Relative Retention Time (continued)

RRT/c OV-101	Detector Responses with OV-101 Column	RRT/c OV-225	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
0.17	HX20/NI9	0.14	0.1	N-(3,4-dichlorophenyl)-N'-methylurea	C8H8Cl2N2O	-	NR	NR	6-15-50	-
0.17		-	-	metolcarb	C9H11NO2	C	-	-	-	-
0.17	HN(WB)0.4/NP7	-	0.1	pebulate	C10H21NOS	C	P	-	15	-
0.18	NP8	-	-	prosulfuron*	C15H16F3N5O4S	-	NR	NR	6-15-50	1-2-3
0.18	NI200	-	0.22	4-hydroxymethyl-3,5-dimethylphenyl methylcarbamate*	C11H15NO3	-	NR	NR	15-50	1-2-3
0.18	NI0.25	-	-	clopyralid methyl ester	C7H4Cl2NO2	-		-	50	-
0.18	NI0.3/NP0.6	0.12	0.21	etridiazole	C5H5Cl3N2OS	C	C	P	6	2
0.18	HN(WB)1/HX0.5/NI9/ NI(WB)16/NP0.9/NP(WB)1	0.27	0.14	3,5-dichloroaniline	C6H5Cl2N	S	S	S	6+15	1+2
0.19	HX(WB)1.6/NI0.6	0.18	-	dicamba methyl ester	C8H6Cl2O3	-	-	-	-	-
0.19	HX(WB)2/NI600	-	-	MCPA methyl ester	C10H11ClO3	-	-	-	-	-
0.19	HX(WB)2/NI30	-	-	mecoprop methyl ester	C11H13ClO3	-	-	-	-	-
0.19	HX1/NI1/NP1	0.22	0.14	N, N-diallyl dichloroacetamide	C8H11Cl2NO	C	S	S	15+50	2+3
0.19	NI0.5/NI(WB)0.4/NP(WB)9	0.08	0.1	2-methoxy-3,5,6-trichloropyridine	C6H4Cl3NO	C	P#	C	6+15	1+2
0.19		-	-	bis(trichloromethyl)disulfide	C2Cl6S2	-	R	-	6	-
0.19	NI3.5	0.19	-	chloroneb	C8H8Cl2O2	C	C	-	6	2
0.2	HX25/NI20	0.86	0.61	desdiethyl simazine	C3H4ClN5	-	NR	NR	6-15-50	1-2-3
0.2	HX2/NI(WB)0.8/NP(WB)4	0.18	0.2	nitrapyrin	C6H3Cl4N	C	C	V	6	2
0.2		-	0.27	1,2,4-triazole	C2H3N3	V	NR	NR	6-15-50	1-2-3
0.2	HX0.6/NI16/NP1	0.32	0.16	3,4-dichloroaniline	C6H5Cl2N	V	S	-	15	-
0.21	HX9/NI25	-	1.36	3-(3,4-dichlorophenyl)-1-methoxyurea	C8H8Cl2N2O2	R	NR	NR	6-15-50	-
0.21		-	-	THPI	C8H9NO2	C	NR	NR	6-15-50	-
0.21	FP(WB)46/NI(WB)4000	0.27	0.24	TEPP	C8H20O7P2	C	-	-	-	-
0.22	NP100	-	0.14	CGA 72903	C7H6F3N	-	-	-	-	-
0.22	NI170/NP40	0.25	0.2	3-carboxy-5-ethoxy-1,2,4-thiadiazole	C3H2N2O3S	NR	-	-	-	-
0.22	TI6	0.32	0.21	demeton-O oxygen analog	C8H19O4PS	-	-	-	-	-
0.22	HX9/NI18/NP60	0.14	0.1	3,4-dichlorophenylurea	C7H6Cl2N2O	-	NR	NR	6-15-50	-
0.22		-	-	butylate	C11H23NOS	-	-	-	-	-
0.22	NP0.5	-	0.14	CGA 150829	C5H14N4O	V	-	-	-	-
0.23	TR0.6	0.21	0.18	methyl 2,3,6-trichlorobenzoate	C8H5Cl3O2	-	-	-	-	-
0.23	NI3	-	-	disul-Na	C8H7Cl2O5S•Na	-	-	-	-	-

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Appendix I: PESTDATA Chemicals in Order by OV-101 Relative Retention Time (continued)

RRT/c OV-101	Detector Responses with OV-101 Column	RRT/c OV-225	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
0.24	NI0.2	0.15	0.22	2,3,5,6-tetrachloroanisole	C7H4Cl4O	-	C	-	6	1
0.24	HX0.3/NI0.25	0.13	0.16	pentachlorobenzene	C6HCl5	C	C	C	6	1
0.25	NI100	-	-	picloram*	C6H3Cl3N2O2	-	-	-	-	-
0.25	HN(WB)1/NI(WB)23/ NP(WB)10	0.5	0.16	cymoxanil	C7H10N4O3	V	NR	NR	6-15-50	1-2-3
0.25	HN(WB)0.5/NI(WB)4/ NP(WB)4	0.92	0.28	oxamyl oxime metabolite	C5H10N2O2S	C	NR	NR	6-15-50	1-2-3
0.25	FP(WB)0.9/TI25	1.11	0.39	omethoate	C5H12NO4PS	C	NR	NR	6-15-50	1-2-3
0.26	NP20	-	0.13	CGA 236432	C9H9F3N2O2	-	-	-	-	-
0.26	NI0.5	0.51	0.23	RPA 203328, methylated	C10H9F3O4S	-	-	-	-	-
0.26	NI1000/NP(WB)0.5	-	0.21	tebuthiuron	C9H16N4OS	-	-	-	-	-
0.26	FP0.5/TI1	-	0.26	thionazin	C8H13N2O3PS	C	P	NR	15+50	-
0.26	NI3500	-	-	diethyl phthalate	C12H14O4	-	P	P	15+50	-
0.27	NI200	-	0.31	4-hydroxymethyl-3,5-dimethylphenyl methylcarbamate*	C11H15NO3	-	NR	NR	15-50	1-2-3
0.28	FP(WB)2	-	0.36	demeton-O*	C8H19O3PS2	C	NR	-	6-15	-
0.28	HX(WB)1.6/NI2	-	-	dichlorprop methyl ester	C10H10Cl2O3	-	-	-	-	-
0.28	NP3	-	0.29	G-27550	C8H12N2O	C	-	-	-	-
0.28	FP(WB)8	0.49	0.32	metasystox thiol	C6H15O3PS2	C	-	-	-	-
0.29		-	0.25	diphenylamine	C12H11N	C	S	-	6+15	-
0.29	TR0.5	0.26	0.24	tecnazene	C6HCl4NO2	C	C	C	6	1
0.3	TR6	0.38	0.25	2,4-D methyl ester	C9H8Cl2O3	-	-	-	-	-
0.3	NI0.4	-	-	bromoxynil methyl ether	C8H5BR2ON	-	-	-	-	-
0.3	HN(WB)6/NI1	-	-	bromofenoxim methyl ether	C14H9Br2O6N3	-	-	-	-	-
0.3	HN(WB)1/HX(WB)20/ NI(WB)500/NP(WB)13	-	0.2	triflusulfuron methyl ester	C17H19F3N6O6S	V	NR	NR	6-15-50	1-2-3
0.3	HX2/NI0.4/NP8	-	-	2,4-dichloro-6-nitrobenzenamine	C6H4Cl2N2O2	-	R	-	15	2
0.3	NI400/TI2	0.37	0.29	phorate oxygen analog	C7H17O3PS2	C	NR	NR	6-15-50	1-2-3
0.3	FS2/NP15	-	-	cycloate	C11H21NOS	C	V #	S	15+50	3
0.3	HX12/NI20	0.8	0.53	desethyl simazine	C5H8ClN5	-	NR	NR	50	1-2-3
0.3		-	0.23	tributyl phosphate	C12H27O4P	-	R	-	50	-
0.31	FP6	0.49	0.32	metasystox thiono*	C6H15O3PS2	-	-	-	-	-
0.31	FP(WB)0.7/TI15	1.6	0.5	monocrotophos	C7H14NO5P	C	NR	NR	6-15-50	1-2-3

Appendix I: PESTDATA Chemicals in Order by OV-101 Relative Retention Time (continued)

RRT/c OV-101	Detector Responses with OV-101 Column	RRT/c OV-225	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
0.31	FP(WB)0.6/TI10	0.96	0.43	dicrotophos	C8H16NO5P	C	NR	-	6-15-50	-
0.32	NI(WB)0.6/NP(WB)5	0.44	0.36	3, 5, 6-trichloro-2-pyridinol methyl ester	C6H4Cl3NO	-	-	-	-	-
0.32	NI0.4	-	-	fenac methyl ester	C9H7Cl3O2	-	-	-	-	-
0.32		-	-	chlorothalonil trichloro impurity	C8HCl3N2	R	R #	NR	6-15-50	2+3
0.32		-	-	bendiocarb	C11H13NO4	C	-	-	-	-
0.32	HX2	-	-	2-chloroethyl caprate	C8H15ClO2	-	C	C	15	2
0.32	HN(WB)1.5	-	0.41	phenmedipham	C16H16N2O4	-	-	-	-	-
0.32	HX2	0.43	0.25	chlorpropham	C10H12ClNO2	C	C	C	15	2
0.33	FP0.5/HX0.3/NI0.5	0.23	0.24	chlorethoxyfos	C6H11Cl4O3PS	V	C	-	6	1
0.33	FP0.7/TI0.8	0.31	0.25	ethoprop	C8H19O2PS2	C	P #	S #	50	1-2-3
0.34	HX4/HX(WB)7/NI1	0.27	0.19	ethalfluralin	C13H14F3N3O4	C	C	C	6	2
0.34		-	0.25	2,4,5-trichloro-alpha-methylbenzene methanol	C8H7OCl3	R	R	-	15	-
0.34		-	0.36	dioxabenzofos	C8H9O3PS	C	P	-	15	-
0.34	TI0.8	-	0.29	sulfotep	C8H20O5P2S2	C	C	P	6+15	2
0.34	NI(WB)16/NP(WB)13	0.37	0.26	propachlor	C11H14ClNO	C	NR	NR	6-15-50	1-2-3
0.34	TR1	0.27	0.17	trifluralin	C13H16F3N3O4	C	C	C	6	2
0.34	NP11	-	0.32	naled	C4H7Br2Cl2O4P	C	NR	NR	6-15-50	1-2-3
0.35	HN(WB)17/NI(WB)34/ NP(WB)600	2.85	0.4	ethametsulfuron methyl ester*	C15H18N6O6S	-	NR	NR	6-15-50	1-2-3
0.35	HN(WB)30	-	-	DNOC methyl ether	C8H8N2O5	-	-	-	-	-
0.35	HN(WB)0.5/NI320/NP15	0.7	0.41	methabenzthiazuron	C10H11N3OS	C	NR	NR	6-15-50	1-2-3
0.35	NP8	0.6	0.38	2,3,5-trimethacarb	C11H15NO2	C	S #	NR	50	1-2-3
0.36	NI0.5/NI(WB)0.7/NP(WB)7	0.36	0.39	triclopyr methyl ester	C8H6Cl3NO3	-	-	-	-	-
0.37	FP(WB)0.5	0.27	0.29	cadusafos	C10H23O2PS2	C	NR	NR	6-15-50	1-2-3
0.37	HX(WB)1.5/NI(WB)2	0.28	0.18	benfluralin	C13H16F3N3O4	C	C	C	6	2
0.37	FP1/NI(WB)24/TI1	0.38	0.32	phorate	C7H17O2PS3	C	V #	V #	6	1
0.38	NI13/NP50	0.63	0.26	3-methyl-4-nitrophenol	C7H7O3N	V	NR	NR	6-15-50	1-2-3
0.38	NI(WB)1	0.44	0.36	sulfallate	C8H14ClNS2	C	C	C	6+15	2
0.39	NI(WB)20	1.16	0.54	4-chlorobenzylmethyl sulfoxide	C8H9ClOS	-	NR	NR	6-15-50	1-2-3
0.39		1.3	0.52	2,6-dichlorobenzamide	C7H5NOCl2	C	NR	NR	6-15-50	1-2-3

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Appendix I: PESTDATA Chemicals in Order by OV-101 Relative Retention Time (continued)

RRT/c OV-101	Detector Responses with OV-101 Column	RRT/c OV-225	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
0.39		-	-	carbofuran	C12H15NO3	C	-	-	-	-
0.39	NP1	0.53	0.38	fonofos oxygen analog	C10H15O2PS	V	NR	NR	6-15-50	1-2-3
0.4	NP(V)20	-	0.47	CGA 37734	C10H13NO2	C	NR	NR	6-15-50	1-2-3
0.4	HX500/NI300	-	0.71	dazomet	C5H10N2S2	S	NR	-	6-15-50	1-2-3
0.4	TR0.4	0.48	0.35	BHC, alpha-	C6H6Cl6	C	C	C	6	1
0.4	FP(WB)0.7/NI(WB)6/NP1	1.6	0.62	dimethoate	C5H12NO3PS2	C	NR	NR	6-15-50	1-2-3
0.41	FS0.5	2.71	0.81	dimethipin	C6H10O4S2	C	NR	NR	6-15-50	1-2-3
0.41	NI(WB)0.4	1.91	0.66	4-chlorobenzylmethyl sulfone	C8H9ClO2S	-	NR	NR	6-15-50	1-2-3
0.41	NI10/NI(WB)3/TI1	0.51	0.4	thiometon	C6H15O2PS3	C	NR	NR	6-15-50	-
0.41	FP(WB)0.8/TI2	0.56	0.41	demeton-S	C8H19O3PS2	C	NR	-	6-15-50	-
0.41	NI(WB)90	0.83	0.5	simazine	C7H12ClN5	C	NR	NR	50	1-2-3
0.42	HN(WB)5/NI(WB)5/ NP(WB)65	0.97	0.49	PPG-947, methylated*	C18H13ClF3NO7	-	-	-	-	-
0.42	TR10	0.74	0.33	2,4-D isopropyl ester*	C11H12Cl2O3	-	-	-	-	-
0.42	HN(WB)3/HX(WB)42/ NI(WB)35/NP(WB)14	-	0.9	pyrazon metabolite B	C6H4ClN3O	-	NR	NR	6-15-50	1-2-3
0.42	FP0.5/NI1000	-	0.39	terbufos oxygen analog	C9H21O3PS2	C	-	NR	6-15-50	1-2-3
0.42		0.26	0.33	di-allate	C10H17ClNOS	C	C	-	6	-
0.42	HX4	0.75	0.45	chlorbufam	C11H10ClNO2	C		-	15	2+3
0.42	NP100	-	0.42	melamine	C3H6N6	NR	-	-	-	-
0.42	TR0.5	0.96	0.45	dicloran	C6H4Cl2N2O2	C	S	P	15+50	2+3
0.43	HN(WB)0.4/NI(WB)26/ NP(WB)3	1.34	0.6	6-chloro-2,3-dihydro-3,3,7-methyl-5H-oxazolo(3,2-a)pyrimidin-5-one	C9H13ClN2O2	-	NR	NR	6-15-50	1-2-3
0.43	FP100	-	0.62	fenthion oxygen analog sulfoxide	C10H15O5PS	C	NR	NR	6-15-50	1-2-3
0.43	TI58/TR200	0.74	0.44	atrazine	C8H14ClN5	C	S #	NR	50	1-2-3
0.43	TR2	1.62	0.56	BHC, beta-	C6H6Cl6	C	C	C	6	1
0.44	NP8	2.49	0.64	prosulfuron*	C15H16F3N5O4S	-	NR	NR	6-15-50	1-2-3
0.44	HN(WB)0.2/NP(WB)0.5	-	0.82	isocarbamid	C8H15N3O2	C	-	-	-	-
0.44	NI1000/NP300	0.29	0.45	desmedipham	C16H16N2O4	-	-	-	-	-
0.44	HX0.8/NI0.8	1.03	-	chloramben methyl ester	C8H7Cl2NO2	-	-	-	-	-
0.45	NI8/NP35	2.25	0.71	methidathion sulfoxide	C5H8N2O4S2	-	NR	NR	6-15-50	1-2-3
0.45	HX(WB)0.8/NI0.6	0.44	-	silvex methyl ester	C10H9Cl3O3	-	-	-	-	-

Appendix I: PESTDATA Chemicals in Order by OV-101 Relative Retention Time (continued)

RRT/c OV-101	Detector Responses with OV-101 Column	RRT/c OV-225	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
0.45	HX1.5/HX2/NI110	0.59	0.46	clomazone	C12H14ClNO2	C		-	50	3
0.45	NP25	0.78	0.5	3,4,5-trimethacarb	C11H15NO2	C	NR	NR	50	1-2-3
0.45	HX0.5/NI0.25	0.25	0.33	hexachlorobenzene	C6Cl6	C	C	P	6	1
0.45	FP1	-	-	tris(beta-chloroethyl) phosphate	C6H12Cl3O4P	C	-	-	-	-
0.46	NP2	-	0.49	CGA 51702	C9H9F3N2O	-	-	-	-	-
0.46	NI1.5/NP50	0.6	0.39	furilazole	C11H13Cl2NO3	C	S	-	50	3
0.46	HX0.4/TR0.4	0.3	0.34	pentachlorophenyl methyl ether	C7H3Cl5O	C	C	C	6	1
0.46		0.49	0.31	oxydemeton-methyl	C6H15O4PS2	C	-	-	-	-
0.47		0.42	0.53	terbumeton	C10H19N5O	C	-	-	-	-
0.47	NI(WB)87/NP(WB)6	0.71	0.48	terbuthylazine	C9H16N5Cl	C	P	-	15+50	-
0.47	FP(WB)0.7/NI(WB)2/NP1	-	0.59	cyanophos	C9H10O3NSP	C	-	-	-	-
0.47	NI100/TI10	-	0.5	dioxathion	C12H26O6P2S4	V	NR	-	6-15-50	2
0.48	FP1.5	-	0.42	propetamphos	C10H20NO4PS	C	C #	-	15+50	2+3
0.48	HX4	0.91	0.48	monolinuron	C9H11ClN2O2	C	-	-	-	-
0.48	NI40	-	-	silvex	C9H7Cl3O3	-	-	-	-	-
0.48	HX0.6/TR0.5	0.69	0.47	lindane	C6H6Cl6	C	C	C	6	1
0.49	TR1	0.63	0.47	2,4,5-T methyl ester	C9H7Cl3O3	-	-	-	-	-
0.49	NI0.3	0.35	0.48	1,2,4,5-tetrachloro-3-(methylthio)= benzene	C7H4Cl4S	R	C	-	6	1
0.5	NI75	0.79	0.44	metribuzin, deaminated diketo metabolite*	C7H11N3O2	NR	NR	NR	6-15-50	1-2-3
0.5	NI900/NP100	0.53	0.24	phosmet oxygen analog*	C11H12NO5PS	-	NR	NR	6-15-50	-
0.5	NI1.4/NP34	0.69	0.48	4-(dichloroacetyl)-1-oxa-4-azapiro= [4.5]decane	C10H15Cl2NO2	C	P	-	50	3
0.5	FP1	2.02	0.5	tris(chloropropyl) phosphate	C9H18Cl3O4P	C	NR	NR	6-15-50	1-2-3
0.5	NI0.5	0.59	0.45	pentachlorobenzonitrile	C7Cl5N	C	C	P	15	2
0.5	FP2/NI40/TI2	0.44	0.41	terbufos	C9H21O2PS3	C	P	S	6	-
0.5	NI18/NP0.6	0.53	0.47	diazinon oxygen analog	C12H21N2O4P	C	NR	NR	6-15-50	1-2-3
0.5	HX0.5/TR0.4	1.71	0.67	BHC, delta-	C6H6Cl6	C	C	C	6+15	1
0.5	NI2200/NP23	2.33	0.66	ethylenethiourea	C3H6N2S	S	NR	NR	6-15-50	1-2-3
0.51	NI100/NP1	-	-	tebupirimfos oxygen analog	C13H23O4N2P	-	NR	NR	6-15-50	1-2-3
0.51	FP7/NI1000/NP(WB)3.5	0.8	0.63	etrimfos oxygen analog	C10H17N2O5P	C	-	-	-	-

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Appendix I: PESTDATA Chemicals in Order by OV-101 Relative Retention Time (continued)

RRT/c OV-101	Detector Responses with OV-101 Column	RRT/c OV-225	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
0.51	HX1/NI2/NP7	0.84	0.4	pronamide	C12H11Cl2NO	C	P	-	15+50	-
0.51	FP(WB)1/NI3/NP0.4	0.4	0.44	diazinon	C12H21N2O3PS	C	C	C	15	3
0.51	TR0.3	0.46	0.46	quintozene	C6Cl5NO2	C	C	C	6	1
0.52	NI(WB)1/TI166	0.93	0.44	dinitramine	C11H13F3N4O4	C	-	P	15	-
0.52	TI2	0.56	0.44	fonofos	C10H15OPS2	C	C	C	6	2+3
0.53	FP10	-	0.57	phosphamidon*	C10H19ClNO5P	C	NR	NR	6-15-50	1-2-3
0.53	HX3/NI1.5	0.76	0.37	fluchloralin	C12H13ClF3N3O4	C	C	-	6	2
0.53	HX5/NI1/NP1	0.46	0.3	profluralin	C14H16F3N3O4	V	V	-	6	-
0.53	NI(WB)110/TI30	0.65	0.41	propazine	C9H16ClN5	C	S	NR	15+50	3
0.54	HX2.5/NI1000	-	0.66	4-chloro-6-methoxyindole	C9H8NOCl	-	R	-	15	-
0.54	NP5/NP(WB)6	1.47	0.62	fenfuram	C12H11NO2	C	-	-	-	-
0.54	HN(WB)1/HX5/NI(WB)6/ NP(WB)8	2.1	0.72	terbacil	C9H13ClN2O2	C	NR	NR	6-15	2+3
0.54	NI(WB)15	-	1.07	4-chlorophenylurea	C7H7ClN2O	NR	NR	NR	6-15-50	1-2-3
0.54	TI2	0.6	0.46	disulfoton	C8H19O2PS3	C	P #	NR	6	1-2-3
0.55	HN(WB)17/NI(WB)34/ NP(WB)600	3.6	0.95	ethametsulfuron methyl ester*	C15H18N6O6S	-	NR	NR	6-15-50	1-2-3
0.55	NI30/NP0.4	0.8	0.63	isazofos	C9H17ClN3O3PS	C	C #	-	50	2+3
0.55	HN(WB)17/NI(WB)15/ NP(WB)9	1.41	0.9	3-ketocarbofuran	C12H12NO4	S	NR	NR	6	1
0.55	HX1/NI0.6	1.44	0.74	chlorothalonil	C8Cl4N2	S	C #	C #	6-15-50	2+3
0.55	TI5/TR11	1.71	0.66	parathion-methyl oxygen analog	C8H10NO6P	-	NR	NR	6-15-50	1-2-3
0.55	NI(WB)3	0.86	1.04	tetraiodoethylene	C2I4	-	P	P	6	-
0.55	NI2	0.92	-	dichlone	C10H4Cl2O2	P	S #	S #	6-15-50	2+3
0.56	NI(WB)2	0.49	0.47	methyl 3,5-dibromo-4-methoxy= benzoate	C9H8Br2O3	-	-	-	-	-
0.56	NI150/NP100	2.29	0.82	methidathion sulfone	C5H8N2O3S2	-	NR	NR	6-15-50	1-2-3
0.56	NI35/NP40	1.41	0.55	metribuzin, diketo metabolite	C7H12N4O2	NR	NR	NR	6-15-50	1-2-3
0.56	NI(WB)1	0.4	0.32	chlordene	C10H6Cl6	-	C	C	6	1
0.56	NI0.4	0.63	0.56	2,3,5,6-tetrachloronitroanisole	C7H3Cl4NO3	-	C	-	6	1+2
0.56	NP10	-	-	aminocarb	C11H16N2O2	C	-	-	-	-
0.57	NI0.5/NP2	1.47	0.91	metribuzin	C8H14N4OS	V	NR	NR	50	1-2-3
0.58	NP10	-	0.68	cyromazine	C6H10N6	S	-	-	-	-

Appendix I: PESTDATA Chemicals in Order by OV-101 Relative Retention Time (continued)

RRT/c OV-101	Detector Responses with OV-101 Column	RRT/c OV-225	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
0.58	FP2/NI50	0.59	0.51	etrimfos	C10H17N2O4PS	C	C	C	15	2+3
0.58	FP4/TI5	-	0.52	schradan	C8H24N4O3P2	C	NR	-	6-15-50	-
0.59	NI0.5	0.73	0.66	2,3,5,6-tetrachloroanisidine	C7H5Cl4NO	-	C	-	6	2
0.59	HX2	-	-	2-chloroethyl laurate	C14H27ClO2	-	C	C	15	2
0.6	NI200/NP200	2.65	0.9	CGA 120844	C8H9NSO3	-	NR	NR	6-15-50	1-2-3
0.6	FP20/FP400/FS1.5/ HN(WB)3.5/NI950/NP(WB)4	1.4	0.78	ethiofencarb	C10H15NO2S	C	NR	NR	6-15-50	-
0.6	FP(WB)1	-	0.54	iprobenfos	C13H21O3PS	C	-	-	-	-
0.6	HX1/NI1.5	-	0.45	tri-allate	C10H16Cl3NOS	C	C	C	6	2
0.6	NI20/NP15	1.64	0.7	ethoxyquin	C14H19N0	C	NR	NR	6-15-50	-
0.61	NI2	-	-	fluroxypyr, methylated*	C8H7O3N2Cl2F	-	-	-	-	-
0.61		-	0.73	pirimicarb	C11H18N4O2	C	-	-	-	-
0.62	TR28	0.72	-	2,4-DB methyl ester	C11H12Cl2O3	-	-	-	-	-
0.62	TR5	0.62	0.49	2,4-D isobutyl ester	C12H14Cl2O3	-	-	-	-	-
0.63	HN1/NI1.2	-	-	dinoseb methyl ether	C11H14N2O5	-	-	-	-	-
0.63	NI16/NP1	-	-	tebupirimfos	C13H23N2O3PS	-	V	V	6+15	2+3
0.64	NI1/NP6	1.06	0.7	benoxacor	C11H11Cl2NO2	C	P	C	15+50	2+3
0.64	HX1.5/NI13	1.22	0.74	cyprazine	C9H14ClN5	C	-	-	-	-
0.64	NP3	1.02	0.62	ronnel oxygen analog	C8H8Cl3O4P	C	NR	-	6-15-50	-
0.65	NI20	0.61	0.56	diisobutyl phthalate	C16H2204	-	P	-	15+50	-
0.66	FS4	-	0.64	octhilinone	C11H19NOS	C	-	-	-	-
0.66	FP1/TI10	-	1.02	phorate oxygen analog sulfone	C7H17O5PS2	C	NR	NR	6-15-50	1-2-3
0.66	HX6/NI3/NI(WB)5	2.82	0.78	propanil	C9H9Cl2NO	C	NR	NR	6-15	3
0.67	FP10	-	0.76	phosphamidon*	C10H19ClNO5P	C	NR	NR	6-15-50	1-2-3
0.67	NI100	-	-	picloram*	C6H3Cl3N2O2	-	-	-	-	-
0.67	NP(WB)0.5	-	-	pyrimethanil	C12H13N3	C	S	S #	50	3
0.67	HX8/NI5	1.44	0.69	metobromuron	C9H11BrN2O2	C	NR	NR	6-15-50	1-2-3
0.67	HX0.5/NI0.4/NP10	0.79	0.66	pentachloroaniline	C6H2Cl5N	C	C	C	6	1
0.67	FP1/FP(WB)3.5/NI1.9/TI2	0.64	0.56	dichlofenthion	C10H13Cl2O3PS	C	C	V	6	2
0.67	TR2	0.65	-	2,4,5-T isopropyl ester	C11H11Cl3O3	-	-	-	-	-
0.68	TR35	0.66	-	2,4,5-T BEP ester*	C17H23Cl3O3	-	-	-	-	-
0.68	FS25/NI4	2.89	0.93	2,3-dihydro-3,3-methyl-2-oxo-5-	C11H12O5S	-	-	-	-	-

Appendix I: PESTDATA Chemicals in Order by OV-101 Relative Retention Time (continued)

RRT/c OV-101	Detector Responses with OV-101 Column	RRT/c OV-225	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
				benzofuranyl methyl sulfonate						
0.68	TI7	1.55	0.87	malathion oxygen analog	C10H19O7PS	C	NR	NR	6-15-50	1-2-3
0.69	TR60	0.74	-	2,4-D BEP ester*	C17H24Cl2O4	-	-	-	-	-
0.69	HN(WB)1/HX(WB)2/ NI(WB)1/NP(WB)19	2.01	0.79	vinclozolin metabolite S	C10H7Cl2NO3	V	P	V #	15	2
0.69	HX1.5/NI1	1.15	0.64	vinclozolin	C12H9Cl2NO3	C	C	C	15	2
0.69		-	-	chlorthiamid	C7H5Cl2NS	-	-	-	-	-
0.7	NP13	-	0.58	CP 51214	C14H21NO3	C	NR	NR	6-15-50	1-2-3
0.7	HN(WB)0.6/NP(WB)23	-	0.8	IN-B2838	C10H15N3O3	P	NR	NR	6-15-50	1-2-3
0.71	FP5/TI12	2.95	0.96	demeton-O sulfone*	C8H19O5PS2	C	-	-	-	-
0.71	HN(WB)5/NI1	-	-	ioxynil methyl ether	C8H5I2NO	-	-	-	-	-
0.71	NI30	1.11	0.71	dimethachlor	C13H18ClNO2	C	-	-	-	-
0.71	HN(WB)0.5/NP(WB)3	-	0.95	fuberidazole	C11H8N2O	C	-	-	-	-
0.71	FP(WB)0.9/NI(WB)3/NP1.5	1.64	0.87	parathion-methyl	C8H10NO5PS	C	C	C	15	2
0.72		-	-	DDM	C13H10Cl2	-	-	-	-	-
0.72	NI(WB)19/NP(WB)14	0.98	-	dimethenamid	C12H18ClNO2S	-	NR	NR	6-15-50	1-2-3
0.72	FP(WB)2	-	1.48	oxydemeton-methyl sulfone	C6H15O5PS2	C	-	-	-	-
0.72	FP1/FP(WB)1.3/HX1.5/ NI1/NP1	0.86	0.79	chlorpyrifos-methyl	C7H7Cl3NO3PS	C	C	-	6	2
0.72	FP3	-	0.83	fenitrothion oxygen analog	C9H12NO6P	C	-	-	-	-
0.72	TR40	-	-	2,4-D n-butyl ester	C12H14Cl2O3	-	-	-	-	-
0.73	NI75	1.29	0.52	metribuzin, deaminated diketo metabolite*	C7H11N3O2	NR	NR	NR	6-15-50	1-2-3
0.73	NP6	-	0.67	CP 108064, methylated	C15H21NO4	-	-	-	-	-
0.73	NP2	-	0.89	cymiazole	C12H14N2S	-	-	-	-	-
0.74	HN(WB)5/HX(WB)18/ NI(WB)5/NP(WB)84	1.2	0.66	vinclozolin metabolite B	C12H11Cl2NO4	C	P #	C	6+15	2
0.75	HX(WB)1.2/NI1.5/TR1	2.67	-	picloram methyl ester	C7H5Cl3N2O2	-	-	-	-	-
0.75	HX5/NI9/NP5	0.88	0.67	acetochlor	C14H20NO2Cl	C	C #	P	50	3
0.75	NI2/NP2	0.8	0.68	CGA 14128	C12H21N2O4PS	C		-	50	1-2-3
0.75	FP1/NI5	1.55	0.79	prothoate	C9H20NO3PS2	C	-	-	-	-
0.75	NP60	-	1.05	carbaryl	C12H11NO2	C	-	-	-	-
0.77	NI1/NP2	1.62	0.99	Tycor	C9H16N4OS	C	S	S	50	3

Appendix I: PESTDATA Chemicals in Order by OV-101 Relative Retention Time (continued)

RRT/c OV-101	Detector Responses with OV-101 Column	RRT/c OV-225	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
0.77	FP200/NI40/TI50	-	0.74	prometryn	C10H19N5S	C	P #	P #	50	1-2-3
0.77		1.1	-	ametryn	C9H17N5S	C	-	-	-	-
0.78	NI300	-	1.06	phorate oxygen analog sulfoxide	C7H17O4PS2	C	NR	NR	6-15-50	1-2-3
0.78	FP7	-	1.12	fenthion oxygen analog	C10H15O4PS	C	NR	NR	6-15-50	1-2-3
0.78	NI0.5/NP7.5	-	-	bromoxynil butyrate	C11H9Br2NO2	-	V	-	15+50	2
0.79	NI2	-	-	fluroxypyr, methylated*	C8H7O3N2Cl2F	-	-	-	-	-
0.8	HN(WB)1.2/HX(WB)90/ NI(WB)1.5/NP(WB)10	2.1	-	bromacil methyl ether	C10H16BrN2O2	-	-	-	-	-
0.8	NP200	-	1.03	3-hydroxymethyl-2,5-dimethylphenyl methylcarbamate	C11H15NO3	-	NR	NR	6-15-50	1-2-3
0.8	NI(WB)7	1	0.72	alachlor	C14H2OClNO2	C	C	C #	50	3
0.8	NI200/NP200	-	1.17	methiocarb sulfone	C11H15NO4S	S	NR	NR	6-15-50	1-2-3
0.8	HN(WB)2/NI(WB)2/ NP(WB)17	4.8	1.36	bromacil	C9H13BrN2O2	C	NR	NR	6-15-50	1-2-3
0.8	NI(WB)5/NP3	-	0.86	parathion oxygen analog	C10H14NO6P	C	NR	NR	6-15-50	1-2-3
0.81	HX(WB)1/NI0.8	0.85	0.75	tridiphane	C10H7Cl5O	C	C	-	6	1+2
0.81	HX1000/NI1000/NP7	-	0.9	metalaxyl	C15H21NO4	C	NR	NR	6-15-50	1-2-3
0.81	HX16/NI300	0.85	-	chloroxuron	C15H15ClN2O2	C	NR	NR	6-15-50	1-2-3
0.81	NI(WB)1/TI3	0.86	0.76	ronnel	C8H8Cl3O3PS	C	C	C	6	2
0.82	TR2	0.64	0.67	chlordene, alpha-	C10H6Cl6	-	-	-	-	-
0.82	TR4	1.07	0.92	dichlorobenzophenone, o,p'-	C13H8Cl2O	-	C	C	15	2
0.83	TR5	-	-	Compound K*	C10H6Cl8	-	C	-	-	1
0.83		-	-	DDNU	C14H10Cl2	-	-	-	-	-
0.83		-	-	DDNS	C14H12Cl2	-	-	-	-	-
0.83	NI60/NP45	3.77	1.06	metribuzin, deaminated metabolite	C8H13N3OS	C	NR	NR	6-15-50	1-2-3
0.83	NI0.6	0.52	0.6	heptachlor	C10H5Cl7	C	C	C	6	1
0.84	NI50	-	-	sethoxydim sulfoxide	C17H29NO4S	-	NR	NR	6-15-50	3
0.84	NI0.6	0.65	-	chlordene epoxide	C10H6Cl6O	-	C	-	15	-
0.84		1.08	-	terbutryn	C10H19N5S	C	-	-	-	-
0.84	FP(WB)1/NP1	1.82	1.05	fenitrothion	C9H12NO5PS	C	C	C	15	2
0.85	TR28	2.13	0.95	linuron	C9H10Cl2N2O2	V	V #	V	50	3
0.86	NI5	-	-	dicofol, o,p'-*	C14H9Cl5O	C	V	S	6+15	2

Appendix I: PESTDATA Chemicals in Order by OV-101 Relative Retention Time (continued)

RRT/c OV-101	Detector Responses with OV-101 Column	RRT/c OV-225	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
0.86	HN(WB)4/HX(WB)11/ NI(WB)28/NP(WB)17	-	1.55	6-chloro-2,3-dihydro-7-hydroxy= methyl-3,3-methyl-5H-oxazolo= (3,2-a)pyrimidin-5-one	C9H13ClN2O3	-	NR	NR	6-15-50	1-2-3
0.86	FS32/NI315	1.93	1.02	ethofumesate	C13H18O5S	C	-	-	-	-
0.86	FP5	-	-	phoxim oxygen analog	C12H15N204P	C	-	-	-	-
0.87	HX7/NI1100	-	1.07	4-chlorophenoxyaniline*	C12H10ClNO	S	-	-	-	-
0.87	FP4	-	1.05	demeton-O sulfoxide	C8H15O4PS2	C	-	-	-	-
0.87	FP2/FP(WB)1.2/NI100/NP2	-	0.92	pirimiphos-methyl	C11H20N3O3PS	C	C	C	15	3
0.88		-	-	methiocarb	C11H15NO2S	C	-	-	-	-
0.88	NI30	0.92	0.84	dibutyl phthalate	C16H22O4	-	C	C	15+50	-
0.89	HN(WB)3/HX(WB)3/ NI(WB)3/NP(WB)15	3.02	0.93	vinclozolin metabolite E	C11H11Cl2NO2	C	S	NR	15+50	-
0.89	NI4/TI26	4.9	1.48	cyanazine	C9H13ClN6	C	NR	-	6-15-50	-
0.89	FP5/NI8/NP4	2.55	1.26	phorate sulfoxide	C7H17O3PS3	C	NR	NR	6-15-50	1-2-3
0.9	NP400	-	0.45	formetanate hydrochloride	C11H16ClN3O2	-	-	-	-	-
0.9	NI1/NP44	1.71	1.01	dichlofluanid	C9H11Cl2FN2O2S2	C	C #	-	15+50	2+3
0.91	TR850	-	-	diisooctyl phthalate*	C24H38O4	-	C	C	15+50	-
0.91	NI(WB)7/NP1	1.49	1.05	malathion	C10H19O6PS2	C	C	C	15+50	3
0.92	FP7/NI40	2.9	1.28	terbufos oxygen analog sulfone	C9H21O5PS2	C	NR	NR	6-15-50	1-2-3
0.93	FP(WB)5	-	1.43	des N-isopropyl isofenphos oxygen analog	C12H18NO5P	-	-	-	-	-
0.94	TR1	-	-	2,4,5-T isobutyl ester	C12H13Cl3O3	-	-	-	-	-
0.94		0.97	0.66	prodiamine	C13H17F3N4O4	C	-	-	-	-
0.94	NI73	1	0.98	thiobencarb	C12H16ClNOS	C		V	15	2+3
0.94	HX0.7/NI0.4	0.69	0.87	pentachlorophenyl methyl sulfide	C7H3Cl5S	C	C	C	6	1
0.95	FP27/NI6/TI8	1.51	1.08	chlorpyrifos oxygen analog	C9H11Cl3NO4P	C	NR	-	6-15-50	-
0.96	HX5/NI1	1.45	0.86	nitrofluorfen	C13H7ClF3NO3	C	C	C	15	2
0.96		-	1.32	carbetamide	C12H16N2O3	-	-	-	-	-
0.96	FP2/TI4	1.46	1.18	fenthion	C10H15O3PS2	C	S #	NR	6+15	1-2-3
0.97	HX13/NI40	1.46	-	methazole	C9H6Cl2N2O3	-	-	-	-	-
0.97	HN(WB)5	-	0.96	difenoxuron	C16H18N2O3	-	-	-	-	-
0.97	NI4/TI4	3.26	1.3	phorate sulfone	C7H17O4PS3	C	S #	S #	6-15-50	3
0.98	TR1	0.84	0.89	chlordene, beta-	C10H6Cl6	-	-	-	-	-

Appendix I: PESTDATA Chemicals in Order by OV-101 Relative Retention Time (continued)

RRT/c OV-101	Detector Responses with OV-101 Column	RRT/c OV-225	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
0.98	TR2	0.89	0.88	chlordene, gamma-	C10H6Cl6	-	-	-	-	-
0.98	TI340	-	-	desmethyl diphenamid	C15H15NO	-	-	-	-	-
0.98	NI(WB)4/NP2	1.91	1.07	parathion	C10H14NO5PS	C	C	C	15	2
0.99	NI1	-	-	benazolin methyl ester	C9H6O3SNCl	-	-	-	-	-
0.99	NI3/NP35	1.91	0.96	KWG 1323	C14H16ClN3O3	C	NR	NR	6-15-50	1-2-3
0.99	NI(WB)7	1.63	1.07	1-hydroxychlordene	C10H6Cl6O	-	R	-	15	-
0.99	TR3	1.25	1.08	dichlorobenzophenone, p,p'-	C13H8Cl2O	-	C	C	15	2
1	FS48/NI135	6.6	1.46	2-hydroxy-2,3-dihydro-3,3-methyl-5-benzofuranyl methyl sulfonate	C11H14O5S	-	-	-	-	-
1	NI1.5/TI3	1	1	chlorpyrifos	C9H11Cl3NO3PS	C	C	P	6	2
1.01	FP4/FP(WB)1.6	-	1.14	pirimiphos-ethyl oxygen analog	C13H24N3O4P	C	-	-	-	-
1.03	HX5/NI12	1.21	0.93	metolachlor	C15H22ClNO2	C	S #	NR	50	1-2-3
1.04	NI5	-	-	dicofol, p,p'-*	C14H9Cl5O	C	V	P #	6+15	1+2
1.04	HN(WB)620/NI(WB)1000	-	1.13	PPG-947	C17H11ClF3NO7	-	NR	NR	6-15-50	1-2-3
1.05	NI220	1	-	PP 890	C9H10O2ClF3	-	-	-	-	-
1.05	HN(WB)170/HX(WB)980/NI(WB)40/NP(WB)270	1.47	0.88	acifluorfen	C14H7ClF3NO3	-	NR	NR	6-15-50	1-2-3
1.05	HX3/NI2/NP20/NP(WB)2	1.64	1	triadimefon	C14H16ClN3O2	C	S #	S #	50	1-2-3
1.05	HN(WB)1.4	-	1.43	pyracarbolid	C13H15NO2	-	-	-	-	-
1.05	TR0.8	0.58	0.76	aldrin	C12H8Cl6	C	C	C	6	1
1.06	NI1	1.13	1	DCPA	C10H6Cl4O4	C	C	C	15	2
1.07	NI100/NP40	-	1.8	methidathion oxygen analog	C6H11N2O5PS2	-	NR	NR	6-15-50	1-2-3
1.07		-	-	3-chloro-5-methyl-4-nitro-1H-pyrazole	C4H4ClN3O2	C	-	-	-	-
1.08	TR35	0.91	-	2,4,5-T BEP ester*	C17H23Cl3O3	-	-	-	-	-
1.08	FP100/NI1000/NP40	3	1.66	fosthiazate	C9H18NO3PS2	C	NR	NR	6-15-50	1-2-3
1.08	TI6	2.33	1.3	crufomate	C12H19ClNO3P	C	NR	NR	6-15-50	-
1.09	NP33	-	0.62	fenpropimorph	C20H33NO	C		-	50	1-2-3
1.1	NI(WB)2	-	0.68	nitrothal-isopropyl	C14H17O6N	C	-	-	-	-
1.1		-	1.55	diphenamid	C16H17NO	V	NR	-	6-15	-
1.1	TR1	-	-	2,4,5-T n-butyl ester	C12H13Cl3O3	-	-	-	-	-
1.11	NI40/NP100	4.7	1.33	isoxaflutole (prop)	C15H12SNO4F3	NR	V #	S #	50	3
1.11	FP3/NI(WB)1/TI3	1.29	1.16	bromophos	C8H8BrCl2O3PS	C	C	C	6	-

Appendix I: PESTDATA Chemicals in Order by OV-101 Relative Retention Time (continued)

RRT/c OV-101	Detector Responses with OV-101 Column	RRT/c OV-225	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
1.13	NI40/NP120	4.7	1.38	RPA202248	C15H12SNO4F3	NR	NR	NR	6-15-50	1-2-3
1.13	FP3/HX2/NI2/NP3	-	0.97	trichloronat	C10H12Cl3O2PS	C	C	-	6	-
1.14	NI2/NP3	1.24	1.01	isopropalin	C15H23N3O4	C	C	-	6	-
1.14	FP2/TI4/TR150	1.03	1.14	pirimiphos-ethyl	C13H24N3O3PS	C	C	C	15+50	3
1.15	NI3	4.3	1.54	CGA 91305	C10H8Cl2N3O	V	NR	NR	6-15-50	1-2-3
1.15	NI7/NP3	1.22	0.93	butralin	C14H21N3O4	V	C	-	6+15+50	-
1.15	FP40/TI20	5.8	1.75	demeton-S sulfone	C8H19O5PS2	C	-	-	-	-
1.16	HN(WB)1.4	-	1.56	phenothiazine	C12H9NS	-	-	-	-	-
1.17	HX4	-	-	2-chloroethyl myristate	C16H31ClO2	C	V	V	15	2
1.17	FP10	1.74	1.24	isofenphos oxygen analog	C15H24NO5P	C	-	-	-	-
1.17	MC25/NI200	-	-	methyl 4-chloro-1H-indole-3-acetate	C11H10ClNO2	R	R #	NR	50	1-2-3
1.18	NP(WB)2	-	1.39	cyprodinil	C14H15N3	C	NR	NR	6-15-50	1-2-3
1.19	TR12	1.15	1.2	TDE, o,p'-, olefin	C14H9Cl3	-	-	-	-	-
1.2	FP2/NI5	-	1.58	terbufos sulfone	C9H21O4PS3	C	C #	C #	6-15-50	2+3
1.2	TR2	3.49	1.85	captan	C9H8Cl3NO2S	C	P	C	50	3
1.21	FP2	2.73	1.5	des N-isopropyl isofenphos	C12H18NO4PS	C	S	-	50	-
1.21	FP10/FP(WB)1.7/ NI(WB)2/TI4	1.58	1.29	chlorfenvinphos, alpha-	C12H14Cl3O4P	C	-	NR	6-15-50	-
1.22	NI3/NP5	1.48	1.21	pendimethalin	C13H19N3O4	C	C	P	15	2
1.23	NI(WB)1	3.01	1.94	folpet	C9H4Cl3O2NS	C	C	P	15+50	2+3
1.24	NP(WB)3	-	1.32	penconazole	C13H15Cl2N3	C	-	-	-	-
1.24	HX(WB)8/NI4	1.88	1.47	anilazine	C9H5Cl3N4	V	S	P	15+50	2+3
1.25	NI2/NP8	8	1.09	MB45950	C12H4SN4F6Cl2	S	P	V	15+50	2+3
1.25	NI3/NP70	-	1.41	tolylfluanid	C10H13ClFNOS	C	-	-	-	-
1.26	TR4	3.5	1.92	Sulphenone	C12H9ClO2S	C		-	50	3
1.27	HX12/NI19	3.39	1.42	chlorbromuron	C9H10BrClN2O2	V	V	V	50	3
1.28	HX7/NI1100	-	1.31	4-chlorophenoxyaniline*	C12H10ClNO	S	-	-	-	-
1.28	NI(WB)70/NP(WB)90	-	2.41	hexythiazox	C17H21ClN2O2S	-	S #	NR	50	2+3
1.28	NI1000	-	1.6	3-phenoxybenzenemethanol	C13H12O2	-	-	-	-	-
1.28	FP(WB)1.7/NI5	2.67	1.58	mecarbam	C10H20NO5PS2	C		-	50	-
1.29	FP2/FP(WB)1.8/HX3/ NI(WB)2/TI4	2	1.52	chlorfenvinphos, beta-	C12H14Cl3O4P	C	S #	-	50	1-2-3

Appendix I: PESTDATA Chemicals in Order by OV-101 Relative Retention Time (continued)

RRT/c OV-101	Detector Responses with OV-101 Column	RRT/c OV-225	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
1.29	HX0.7/TR1	1.22	1.15	heptachlor epoxide	C10H5Cl7O	C	C	C	6	2
1.3	NI50	8.9	-	chlorsulfuron	C12H12ClN5O4S	-	NR	NR	6-15-50	-
1.31	FP(WB)2/NI5/TI3	2.05	1.83	phenthoate	C12H17O4PS2	C	C	-	15+50	-
1.32	FP5/FP(WB)2.7	2	1.64	quinalphos	C12H15N2O3PS	C	C	-	15	-
1.33	TR1	0.94	1.05	octachlor epoxide	C10H4Cl8O	C	C	C	6	1
1.34	FP3/TI5	0.65	1.43	merphos*	C12H27PS3	-	C	C	6+15+50	3
1.35	HN(WB)4/HX(WB)40/ NI(WB)67/NP(WB)39	2.27	2.55	3-tert-butyl-5-chloro-6-hydroxy= methyluracil	C9H13ClN2O3	-	NR	NR	6-15-50	1-2-3
1.35	NI2/NP10	8.7	1.16	fipronil	C12H4Cl2F6N4OS	S	S	V	50	3
1.36	HX10/NI90/NP30/NP(WB)5	-	1.44	triadimenol	C14H18ClN3O2	C	NR	NR	6-15-50	-
1.36	FP2/NI20	1.73	1.38	isofenphos	C15H24NO4PS	C	C	-	15+50	-
1.36	NI8	1.22	-	allethrin	C19H26O3	-	C	C #	50	3
1.37	NI12	3.04	1.49	procymidone	C13H11Cl2NO2	C	C	P	15	-
1.37	NI2	1.22	1.12	S-bioallethrin	C19H26O3	-	C	-	50	-
1.37	NI60/TI10	2.85	1.9	crotoxyphos	C14H19O6P	C	NR	NR	6-15-50	1-2-3
1.39	NI1000	1.89	1.54	CGA 189138	C13H8O3Cl2	-	-	-	-	-
1.39	NI6	1.62	1.54	chlorbenside	C13H10Cl2S	C	S	P	6	1
1.4	NI500/NP500	-	-	WAK4103*	C9H9N5O3Cl	-	NR	NR	6-15-50	1-2-3
1.4	HN(WB)1.5	-	1.32	dinobuton	C14H18N2O7	C	-	-	-	-
1.4	FP5/FP(WB)1.6/NP3	3.33	2.28	methidathion	C6H11N2O4PS3	C	S	P #	50	3
1.41	HN(WB)1.6/NP(WB)51	-	2.55	IN-T3936	C10H15N3O4	S	NR	NR	6-15-50	1-2-3
1.42	NI2800	3.7	-	fenac	C8H5Cl3O2	-	NR	NR	6-15-50	-
1.43	TR2	-	-	photodieldrin B	C13H9Cl5O	-	-	-	-	-
1.44	NP(WB)4	2.23	1.19	triflumizole	C15H15ClF3N3O	C	-	-	-	-
1.44	TR60	-	-	4-(2,4-dichlorophenoxy)benzenamine	C12H9Cl2NO	-	-	-	-	-
1.44	NI(WB)0.8/NP(WB)18/ HX(WB)5	-	-	zoxamide*	C14H16NO2Cl3	C	C	-	50	3
1.45	NI(WB)4	1.36	1.45	TDE, p,p'-, olefin	C14H9Cl3	C	C	C	6	1
1.47		-	-	DDMU	C14H9Cl3	-	-	-	-	-
1.48	NP90	-	2.04	thiabendazole	C10H7N3S	C	NR	-	6-15-50	-
1.49	TR1	1.46	1.34	chlordane, trans-	C10H6Cl8	C	C	C	6	1
1.5	NI34	-	1.46	metazachlor	C14H16ClN3O	C	-	-	-	-

Transmittal No. 2000-1 (10/1999)
Form FDA 2905a (6/92)

Appendix I: PESTDATA Chemicals in Order by OV-101 Relative Retention Time (continued)

RRT/c OV-101	Detector Responses with OV-101 Column	RRT/c OV-225	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
1.5		7.9	-	procyazine	C10H13ClN6	C	-	-	-	-
1.5	NI3/NP22	4.3	2.67	TCMTB	C9H6N2S3	C	P	P	15	-
1.5	TI7	6.7	2.39	disulfoton sulfone	C8H19O4PS3	C	NR	-	6-15-50	-
1.51	FP3/NI3/TI4	1.42	1.45	bromophos-ethyl	C10H12BrCl2O3PS	C	C	P	6	-
1.52	NP(WB)6	-	1.59	paclobutrazol	C15H20ClN3O	C	-	-	-	-
1.53	NI5/NP150	6.5	2.41	CGA 94689A	C15H21NO5	V	NR	NR	6-15-50	1-2-3
1.53	TR40	0.95	-	Perthane olefin	C18H19Cl	-	C	C	6	1
1.54	TR20	3.6	-	2,4-D propylene glycol butyl ether ester*	C15H2OCl2O4	-	-	-	-	-
1.54	NI12/NP150	6.6	2.45	CGA 94689B	C15H21NO5	S	NR	NR	6-15-50	1-2-3
1.55	HX(WB)9/NI5	-	1.4	haloxyfop methyl ester	C16H13ClF3NO4	-	-	-	-	-
1.55	TR2	1.28	1.51	DDE, o,p'-	C14H8Cl4	C	C	C	6	1
1.57	NI5/NP30	1.84	1.64	cyclanilide methyl ester	C12H11Cl2NO3	-	-	-	-	-
1.57	NI(WB)1	-	1.85	oxythioquinox	C10H6N2OS2	C	-	-	-	-
1.58	NI15/NP25	2.46	2.1	triazamate	C13H22N4O3S	C	NR	NR	6-15-50	1-2-3
1.58	FP9/TI8	2.72	1.97	Gardona	C10H9Cl4O4P	C	NR	NR	6-15-50	1-2-3
1.58	TI400	-	-	promecarb	C12H17NO2	V	-	-	-	-
1.59	NI550/NP210	-	-	NTN35884*	C9H9N5O2Cl	-	NR	NR	6-15-50	1-2-3
1.59	NP500/NP(WB)8	-	3.9	tricyclazole	C9H7N3S	C	-	-	-	-
1.6	FS(WB)10/NI(WB)3	4.1	3.17	isoprothiolane	C12H18O4S2	C	-	-	-	-
1.6		-	-	MGK 264	C17H25NO2	-	-	-	-	-
1.61	HX4/TR3	3.04	2.2	ovex	C12H8Cl2O3S	C	C	C	15	2
1.64	HX1/NI1.3	1.38	1.47	endosulfan I	C9H6Cl6O3S	C	C	C	15	2
1.65		-	1.65	DDMS	C14H11Cl3	-	R	-	6	-
1.66	TR60	1.18	-	2,4-D BEP ester*	C17H24Cl2O4	-	-	-	-	-
1.66	FP8/NP8	3.7	2.41	fenamiphos	C13H22NO3PS	C	NR	NR	6-15-50	1-2-3
1.66	TR1	1.54	1.48	chlordane, cis-	C10H6Cl8	C	C	C	6	1
1.7	NP40	-	2.12	napropamide	C17H20NO2	C	-	-	-	-
1.73	HX9	1.83	1.46	butachlor	C17H26ClNO2	C	C	-	50	-
1.73	HX8/NI3	-	1.88	chlorflurecol methyl ester	C15H11ClO3	C	-	-	-	-
1.75	TR2	1.45	1.42	nonachlor, trans-	C10H5Cl9	C	C	C	6	1
1.76	NI30/NP(WB)12	4	2.08	imazalil	C14H14Cl2N2O	C	NR	NR	6-15-50	-

Appendix I: PESTDATA Chemicals in Order by OV-101 Relative Retention Time (continued)

RRT/c OV-101	Detector Responses with OV-101 Column	RRT/c OV-225	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
1.77	FP(WB)4	-	-	fenthion oxygen analog sulfone*	C10H15O6PS2	-	-	-	-	-
1.78	HX11/NI10/NP180	3.14	2	diethatyl-ethyl	C16H22ClNO3	C	NR	NR	6-15-50	1-2-3
1.79	HX540/HX(WB)50/NI45/ NP(WB)40	3.5	2.76	imazamethabenz methyl ester*	C16H20N2O3	C	-	-	-	-
1.8	NP100	-	2.96	CGA 100255	C15H12NO5	S	-	-	-	-
1.8	FP7/FP(WB)2.6/NI3	2.34	2.13	profenofos	C11H15BrClO3PS	C	P	P	50	3
1.82	TR12	2.08	1.79	2,4-D butoxyethyl ester*	C14H18Cl2O4	-	-	-	-	-
1.84	NI200/NP50	-	-	imidacloprid	C9H10ClN5O2	-	NR	NR	6-15-50	1-2-3
1.85	FP4/NI3/NP3	1.74	1.82	prothiofos	C11H15Cl2PO2S2	C	C	C	6	2
1.86	HX6/NI3	2.91	1.79	hexaconazole	C14H17Cl2N3O	C	-	-	-	-
1.87	FS50	-	-	carboxin	C12H13NO2S	C	NR	NR	6-15-50	-
1.88	NI25	-	1.99	pretilachlor	C17H26ClNO2	C	-	-	-	-
1.88	NP1	4.1	-	ethion oxygen analog	C9H22O5P2S3	C	-	-	-	-
1.9	HN(WB)58/HX(WB)110/ NI(WB)85/NP(WB)150	3.16	1.86	PPG-2597	C20H17ClF3NO6	-	NR	NR	6-15-50	1-2-3
1.9	HN(WB)0.7/NI25/NP(WB)7	7.2	2.6	myclobutanil	C15H17ClN4	C	NR	NR	6-15-50	1-2-3
1.9	TR2	2.46	2.19	TDE, o,p'-	C14H10Cl4	-	C	C	6	1
1.91	HX1/NI1.5	1.87	1.84	dieldrin	C12H8Cl6O	C	C	C	15	2
1.92	NI1.5	1.59	1.86	DDE, p,p'-	C14H8Cl4	C	C	C	6	1
1.94	HX8/NI7	-	2.45	flamprop-methyl	C17H15ClFNO3	C	-	-	-	-
1.95	FP3/TI5	1.64	1.88	merphos*	C12H27PS3	-	C	C	6+15+50	3
1.95	FP3/NP3/TR4	1.65	1.88	tribufos	C12H27OPS3	C	C	P	15+50	3
1.97	NP(WB)5	-	2.33	flusilazole	C16H15F2N3Si	C	-	-	-	-
1.97	HX4/NI4	2.48	1.96	oxadiazon	C15H18Cl2N2O3	C	C	P	15	-
1.99	TI45	-	3.8	fensulfothion oxygen analog sulfone	C11H17O7PS2	-	-	-	-	-
2	TR60	1.79	-	2,4-D BEP ester*	C17H24Cl2O4	-	-	-	-	-
2	NI900/NP100	0.93	0.44	phosmet oxygen analog*	C11H12NO5PS	-	NR	NR	6-15-50	-
2	FP600/TR10000	2.77	-	aramite*	C15H23ClO4S	C	P	NR	15	-
2	NI5	4	2.16	oxyfluorfen	C15H11ClF3NO4	C	C	C	15	2
2	FS(WB)20/NI(WB)8	3.7	2.6	bupirimate	C13H24N4SO3	C	-	-	-	-
2	NI(WB)10/NP(WB)50	3.02	3.38	kresoxim-methyl	C18H19NO4	P	C	C	15+50	3
2.02	HX7/NI7/NP(WB)8	3.4	2.03	diclobutrazol	C15H19Cl2N3O	C	NR	NR	6-15-50	1-2-3

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Appendix I: PESTDATA Chemicals in Order by OV-101 Relative Retention Time (continued)

RRT/c OV-101	Detector Responses with OV-101 Column	RRT/c OV-225	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
2.02	NP7	1.21	-	simetryn	C8H15N5S	C	-	-	-	-
2.03	NI(WB)1	3.8	2.71	nitrofen	C12H7Cl2NO3	C	C	C	15	2
2.04	TR50	1.78	1.78	2,4-D isooctyl ester*	C16H22Cl2O3	-	-	-	-	-
2.04	HN(WB)182/HX(WB)73/ NI(WB)72/NP(WB)12	1.61	2.69	cyproconazole	C15H18ClN3O	C	NR	NR	6-15-50	1-2-3
2.06	NI2/NP30	31	1.98	MB46136	C12H4SO2N4F6Cl2	S	S	V	50	2+3
2.07	NP189	-	2.92	methoprotryne	C11H21N5OS	C	-	-	-	-
2.1	NI5	1.78	1.68	2,4-D ethyl hexyl ester*	C16H22Cl2O3	-	-	-	-	-
2.12	NI25	1.76	1.76	pyrethrins*	C21H27O4	-	C	C	50	-
2.13	TR2	2.22	2.29	endrin	C12H8Cl6O	C	C #	C #	15	2
2.14	HN(WB)5/NI(WB)5/ NP(WB)65	5	2.4	PPG-947, methylated*	C18H13ClF3NO7	-	-	-	-	-
2.14	NI100	-	-	mirex, 5,10-dihydro-*	C10H2Cl10	-	-	-	-	-
2.14	FP600/TR10000	3.05	-	aramite*	C15H23ClO4S	C	P	NR	15	-
2.15	HN(WB)3/HX(WB)10/ NI(WB)3/NP(WB)34	5	2.4	PPG-847, methylated	C15H9ClF3NO3	-	-	-	-	-
2.17	NI6/TI15	4.2	3.06	carbophenothion oxygen analog	C11H16ClO3PS2	C	NR	NR	6-15-50	1-2-3
2.19		-	-	vamidothion sulfone	C8H18NO6PS2	C	-	-	-	-
2.19	NI(WB)1	4.2	2.38	binapacryl	C15H18N2O6	C	P	P	15	-
2.21	NI2/NP50	-	2.34	chlorfenapyr (prop)	C15H11BrClF3N2O	P	-	S	50	2
2.21	HX2/NI2	3.9	2.77	endosulfan II	C9H6Cl6O3S	C	C	C	15+50	2
2.22	HX9/NI6	4.1	2.99	chlorthiophos oxygen analog	C11H15Cl2O4PS	C	NR	NR	6-15-50	1-2-3
2.23	TR150	2.01	2.42	Perthane	C18H2OCl2	C	C	C	6	1
2.24	FP8	-	2.58	chlorthiophos*	C11H15Cl2O3PS2	C	C	C	6	2
2.24	NI20	-	-	metamitron	C10H10N4O	-	-	-	-	-
2.26	FP54	-	4.4	famphur oxygen analog	C10H16NO6PS	C	-	-	-	-
2.28	NI1.5	-	-	methyl 2,3,5-triiodobenzoate	C8H5I3O2	-	-	-	-	-
2.29	FP(WB)4	-	4.1	fenthion oxygen analog sulfone*	C10H15O6PS2	-	-	-	-	-
2.3	HX19/HX(WB)18/NI125	2.36	2.31	fluazifop butyl ester	C19H20F3NO4	C	C	V	15	3
2.31	NI20/NP50	15	3.93	desisopropyl iprodione	C10H6Cl2N3O3	P		-	50	1-2-3
2.31	TR70	3.26	2.61	chlorobenzilate	C16H14Cl2O3	C	C #	P #	15+50	3
2.33	TR8	5.8	-	Dilan*	C15.5H14Cl2NO2	-	P	P	15	-

Appendix I: PESTDATA Chemicals in Order by OV-101 Relative Retention Time (continued)

RRT/c OV-101	Detector Responses with OV-101 Column	RRT/c OV-225	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
2.33	TR80	2.9	2.41	chloropropylate	C17H16Cl2O3	P	C	C	15+50	3
2.35	TR4	-	-	endrin aldehyde	C12H8Cl6O	C	P	C	15+50	-
2.35	HX10	-	-	2-chloroethyl palmitate	C18H35ClO2	-	V	P	15	2
2.36	FP8	-	2.77	chlorthiophos*	C11H15Cl2O3PS2	C	C	C	6	2
2.36	NP(WB)12	-	-	etaconazole*	C14H15Cl2N3O2	C	-	-	-	-
2.37		-	-	2,4,5-T propylene glycol butyl ether esters	C15H19Cl3O4	-	-	-	-	-
2.38	NI(WB)2/TI5	3.24	3.14	leptophos photoproduct	C13H11Cl2O2PS	C	-	-	-	-
2.39	FP22	-	4.7	fenthion sulfone	C10H15O5PS2	C	NR	NR	6-15-50	1-2-3
2.4	NI120/NP50	4.3	3	imazethapyr ammonium salt methyl ester	C16H21N3O3	-	-	-	-	-
2.4	TI12	-	3.8	fensulfothion	C11H17O4PS2	C	NR	NR	6-15-50	1-2-3
2.41	NI4	-	-	2,8-dihydromirex	C10H2Cl10	-	C	-	6	-
2.41	TR4	3.8	2.87	TDE, p,p'-	C14H10Cl4	C	C	C	6	1
2.43	NP(WB)12	-	3.17	etaconazole*	C14H15Cl2N3O2	C	-	-	-	-
2.43	NP60	-	4.5	benodanil	C13H10INO	C	-	-	-	-
2.45	TR340	-	-	diisohexyl phthalate*	C20H30O4	-	C	-	15+50	-
2.46	HX9/NI9	-	2.81	flamprop-M-isopropyl	C19H19ClFNO3	C	-	-	-	-
2.47	NI100	-	-	mirex, 5,10-dihydro-*	C10H2Cl10	-	-	-	-	-
2.49	TI10	-	5	fensulfothion oxygen analog	C11H17O5PS	C	NR	-	6-15-50	-
2.5	NI1700/NP8	14	5	oxadixyl	C14H18N2O4	C	NR	NR	6-15-50	1-2-3
2.51	HN(WB)1/HX(WB)12/ NI(WB)9/NP(WB)90	4	4.2	pyrithiobac-sodium methyl ester	C14H13ClN2O4	-	-	-	-	-
2.52	HX1/NI2	3.33	2.61	nonachlor, cis-	C10H5Cl9	C	C	C	6	1
2.53	TR5	2.66	-	Compound K*	C10H6Cl8	-	C	-	-	1
2.55	NI(V)250	-	-	hydramethylnon*	C25H24F6N4	-	-	-	-	-
2.55	TR4	-	-	endrin alcohol	C12H8Cl6O	-	P	C	15+50	2+3
2.55	TR4	2.27	2.7	DDT, o,p'-	C14H9Cl5	C	C	C	6	1
2.56	FP8	-	3.16	chlorthiophos*	C11H15Cl2O3PS2	C	C	C	6	2
2.56	TR20	3.1	-	2,4,5-T isooctyl ester*	C16H21Cl3O3	-	-	-	-	-
2.56	FP(WB)1.9/NI(WB)3/NP2	3.93	3.36	ethion	C9H22O4P2S4	C	C	C	6	2
2.62	HX(WB)260	-	-	pyrazon metabolite A	C16H18ClN3O6	-	-	-	-	-

Appendix I: PESTDATA Chemicals in Order by OV-101 Relative Retention Time (continued)

RRT/c OV-101	Detector Responses with OV-101 Column	RRT/c OV-225	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
2.62	HX17	18.6	5.4	ofurace	C14H16NO3Cl	C	-	-	-	-
2.62	FP(WB)3	-	5.2	triazophos	C12H16N3O3PS	C	-	-	-	-
2.64	NI(WB)1	2.33	2.8	tetrasul	C12H6Cl4S	C	C	C	6	1
2.65	FP8/TI40	14	5	famphur	C10H16NO5PS2	C	NR	-	6-15-50	-
2.66	TR340	-	-	diisohexyl phthalate*	C20H30O4	-	C	-	15+50	-
2.67	NI7	-	-	10,10-dihydromirex	C10H2Cl10	-	C	-	6	-
2.67	HN(WB)8/HX(WB)30/ NI(WB)26/NP(WB)56	13	8	pyrazon	C10H8ClN3O	C	NR	NR	6-15-50	1-2-3
2.72	NI500/NP500	-	-	WAK4103*	C9H9N5O3Cl	-	NR	NR	6-15-50	1-2-3
2.75	NI(WB)2	1.67	2.38	chlordecone	C10H8Cl10O5	-	S #	P #	15+50	1-2-3
2.78	FP(WB)14	-	3.6	sulprofos sulfoxide*	C12H19O3PS3	C	-	-	-	-
2.79	FP(WB)47	-	3.5	sulprofos	C12H19O2PS3	C	-	-	-	-
2.8	NI9/NP7	-	3.6	fensulfothion sulfone	C11H17O5PS2	C	NR	-	6-15-50	-
2.81	TR8	7.5	-	Dilan*	C15.5H14Cl2NO2	-	P	P	15	-
2.81	NI(WB)1	7.5	3.9	Prolan	C15H13Cl2NO2	P	S	S	15	2
2.83	HX4/TR5	8.3	4	endosulfan sulfate	C9H6Cl6O4S	C	C	C	50	2
2.85	TR35	1.19	-	2,4,5-T BEP ester*	C17H23Cl3O3	-	-	-	-	-
2.85	TR5	4.7	-	chlornitrofen	C12H6Cl3NO3	C	C	C	6+15	2
2.87	HX(WB)160/NI(WB)57/ NP(WB)140	-	4.6	vinclozolin metabolite F	C11H13Cl2NO4	R	NR	NR	6-15-50	1-2-3
2.87	FP(WB)4/NI(WB)4	6.3	5.3	edifenphos	C14H15O2PS2	C	-	-	-	-
2.9	TR340	-	-	diisohexyl phthalate*	C20H30O4	-	C	-	15+50	-
2.9		-	3.7	1,1'-(2,2-dichloroethylidene)bis(2-methoxybenzene)	C16H16Cl2O2	-	R	-	-	-
2.91	TR4	3.3	-	2,4,5-T butoxyethyl ester*	C14H17Cl3O4	-	-	-	-	-
2.91	HN(WB)3/NP(WB)15	-	-	hexazinone	C12H20N4O2	P	NR	NR	6-15-50	1-2-3
2.94	TI15/TR4	4.2	3.7	carbophenothion	C11H16ClO2PS3	C	C	P	6	2
2.95	NI25	2.84	2.7	pyrethrins*	C21H27O4	-	C	C	50	-
2.96	TR20	3.4	-	2,4,5-T isooctyl ester*	C16H21Cl3O3	-	-	-	-	-
2.96	NI15/NP50	11.5	4.1	CL 202,347	C13H19N3O5	-	-	-	-	-
2.97	NI(WB)8	3.7	4.2	methoxychlor olefin	C16H14Cl2O2	C	C	C	6	2
3	NP300	-	1.18	NTN33823	C9H11N4Cl	-	NR	NR	6-15-50	1-2-3

Appendix I: PESTDATA Chemicals in Order by OV-101 Relative Retention Time (continued)

RRT/c OV-101	Detector Responses with OV-101 Column	RRT/c OV-225	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
3.03	NI170	2.74	2.88	ethephon	C2H6ClO3P	NR		-	6+15+50	1+2+3
3.06	NI10/NI(WB)17	-	-	propiconazole*	C15H17Cl2N3O2	C	NR	NR	6-15-50	1-2-3
3.06	HN(WB)1.4/NP(WB)29	-	-	IN-A3928	C11H18N4O2	S	NR	NR	6-15-50	1-2-3
3.06	NI(WB)1	7.5	4.4	Bulan	C16H15Cl2NO2	C	P	P	15	2
3.06	NI35	5.1	4.5	butyl benzyl phthalate	C19H20O4	-	C	P	15+50	-
3.1	NI900/NP100	14.8	7.1	phosmet oxygen analog*	C11H12NO5PS	-	NR	NR	6-15-50	-
3.1	FP3.5/NI3/NP(WB)3	8.2	4.6	cyanofenphos	C15H14NO2PS	C	-	-	-	-
3.1	NI(WB)2/NP(WB)220	-	3.7	fenhexamid	C14H17Cl2NO2	NR	NR	NR	6-15-50	1-2-3
3.11	NI3	-	5.4	captafol	C10H9Cl4NO2S	C	P	-	50	3
3.13	TR4	3.6	3.5	DDT, p,p'-	C14H9Cl5	C	C	C	6	1
3.14	NI4/NP40	-	-	bromoxynil octanoate	C15H17Br2NO2	-	V #	-	15+50	2
3.21	NI100	-	-	mirex, 5,10-dihydro-*	C10H2Cl10	-	-	-	-	-
3.21	NI10/NI(WB)17	5.6	4	propiconazole*	C15H17Cl2N3O2	C	NR	NR	6-15-50	1-2-3
3.22	TR60	2.09	-	2,4-D BEP ester*	C17H24Cl2O4	-	-	-	-	-
3.25	TR20	3.8	-	2,4,5-T isooctyl ester*	C16H21Cl3O3	-	-	-	-	-
3.26	NP(WB)30	5.8	4.67	clodinafop-propargyl	C17H13ClFNO4	V	V	-	50	3
3.27	TR340	-	-	diisohexyl phthalate*	C20H30O4	-	C	-	15+50	-
3.28	FS(WB)17/HN(WB)3/ NP(WB)130	-	9.4	oxycarboxin	C12H13NO4S	R	-	-	-	-
3.3	TR35	2.78	-	2,4,5-T BEP ester*	C17H23Cl3O3	-	-	-	-	-
3.3	NI(WB)0.1/NP(WB)37	-	7.5	3-desmethyl sulfentrazone	C10H8Cl2F2N4O3S	-	NR	NR	6-15-50	1-2-3
3.3	NI(WB)2	4.5	5	methoxychlor, o, p'-	C16H15Cl3O2	-	C	-	6	-
3.32	NI100/NP10	-	-	tralkoxydim*	C20H27NO3	V	NR	NR	50	1-2-3
3.34	NI50	-	-	sethoxydim	C17H29NO3S	-	NR	NR	6-15-50	3
3.36	NI5	7.3	4.8	nuarimol	C17H12ClFN2O	C	NR	C #	50	1-2-3
3.38	HX(WB)3/NI27	1.41	4.9	desmethyl norflurazon	C11H7ClF3N3O	V	NR	NR	6-15-50	1-2-3
3.38	HX(WB)1	-	4.2	tebuconazole	C16H22ClN3O	C	-	-	-	-
3.38	NI5	-	2.62	2,4,5-T ethylhexyl ester	C16H21Cl3O3	-	-	-	-	-
3.39	TR8	8.2	-	Dilan*	C15.5H14Cl2NO2	-	P	P	15	-
3.57	HX8/NI10	4.9	4.7	diclofop-methyl	C16H14Cl2O4	C	C	C	15	2
3.6	NI60	37.1	7.5	myclobutanil alcohol metabolite	C15H17ClN4O	S	NR	NR	6-15-50	1-2-3
3.6	TR5	10.3	-	endrin ketone	C12H8Cl6O	-	C	C	50	2

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Appendix I: PESTDATA Chemicals in Order by OV-101 Relative Retention Time (continued)

RRT/c OV-101	Detector Responses with OV-101 Column	RRT/c OV-225	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
3.7	FP20	-	10.1	azinphos-methyl oxygen analog	C10H12N3O4PS	C	-	-	-	-
3.74	NI5	-	-	8-monohydromirex	C10HCl11	-	C	-	6	-
3.8	FP600/FP(WB)8/NI160	-	6.2	phosalone oxygen analog	C12H15ClNO5PS	C	-	-	-	-
3.8	NI36/TI35	-	7.1	carbophenothion oxygen analog sulfone	C11H16ClO5PS2	-	-	-	-	-
3.8	NI(WB)1	24	6.3	nitralin	C13H19N3O6S	C	P	P	50	3
3.8	FS45/NI(WB)230	4.8	4.3	propargite	C19H26O4S	C	C	-	15	2
3.9	NI(WB)0.8/NP(WB)18/ HX(WB)5	-	-	zoxamide*	C14H16NO2Cl3	C	C	-	50	3
4	NI(WB)6	-	3.9	dinocap*	C18H24N2O6	C	P	P	15	2
4	NI1000/NP1000	15	4.2	KWG 1342	C14H18ClN3O3	-	-	-	-	-
4	NI(WB)2/NP19	14.9	8.4	phosmet	C11H12O4NPS2	C	NR	-	6-15-50	3
4.1	TR60	5.1	-	2,4-D BEP ester*	C17H24Cl2O4	-	-	-	-	-
4.1	NI5	1.08	0.91	dicofol, o,p'-*	C14H9Cl5O	C	V	S	6+15	2
4.1	HX15	-	-	2-chloroethyl linoleate	C20H35ClO2	-	V	P	15	2
4.2	HX13/NI15/NP15	18	6.3	iprodione*	C13H13Cl2N3O3	C	S #	NR	50	1-2-3
4.2	FP60/TI65	7.6	6.5	leptophos oxygen analog	C13H10BrCl2O3P	C	-	-	-	-
4.2	TI250	-	2.87	carbophenothion oxygen analog sulfoxide	C11H16ClO4PS2	-	-	-	-	-
4.2	NI(WB)16	14	8.7	pyridaphenthion	C14H17O4N2SP	C	-	-	-	-
4.26	NI7	-	-	10-monohydromirex	C10HCl11	-	C	-	6	-
4.3	NI100	-	-	mirex, 5,10-dihydro-*	C10H2Cl10	-	-	-	-	-
4.3	NI50	-	-	tetramethrin*	C19H25NO4	C	NR	NR	6-15-50	1-2-3
4.3	NI(WB)6	6.9	4.4	dinocap*	C18H24N2O6	C	P	P	15	2
4.3	NI(WB)3	8.4	6	benzoylprop-ethyl	C18H17Cl2NO3	P	NR	NR	6-15-50	1-2-3
4.4	NI5	1.28	1.08	dicofol, p,p'-*	C14H9Cl5O	C	V	P #	6+15	1+2
4.4	TR12	6.5	-	bromopropylate	C17H16Br2O3	C	C #	C #	15+50	1-2-3
4.4	TR6	15.5	8.5	photodieldrin	C12H8Cl6O	-	C	C	15+50	2
4.5	NI50	8.5	7.2	tetramethrin*	C19H25NO4	C	NR	NR	6-15-50	1-2-3
4.5	FP50/NP20	-	8.4	fenamiphos sulfone	C13H22NO5PS	C	NR	NR	6-15-50	1-2-3
4.5	HX50	-	5.01	norflurazon	C12H9ClF3N3O	V	NR	NR	6-15-50	-
4.5	NI0.5/TI16	10.6	6.9	EPN	C14H14NO4PS	C	C	C	15	2

Appendix I: PESTDATA Chemicals in Order by OV-101 Relative Retention Time (continued)

RRT/c OV-101	Detector Responses with OV-101 Column	RRT/c OV-225	RRT/c OV-17		Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
4.6	HX8	-	-		fenarimol metabolite C	C17H14N2OCl2	S		-	6	-
4.6	HX19	-	-		fenarimol metabolite B	C17H14N2OCl2	NR	NR	NR	6-15-50	-
4.7	NI500/NP500	-	-		WAK4103*	C9H9N5O3Cl	-	NR	NR	6-15-50	1-2-3
4.7	HN(WB)390/NI(WB)400	-	-		IN-T3937	C12H20N4O3	S	-	-	-	-
4.7	HN(WB)73/NI(WB)230	-	-	-	IN-T3935	C11H18N4O3	S	-	-	-	-
4.7	HX20/NI6	10.3	6.9		chlorthiophos sulfoxide	C11H15Cl2O4PS2	C	NR	NR	6-15-50	1-2-3
4.7	NI(WB)1	8.6	7.2		tetrasul sulfoxide	C12H6Cl4OS	-	-	-	-	-
4.7	NI8800	-	-		oryzalin	C12H8N4O6S	-	NR	NR	6-15-50	-
4.7	NI(WB)12	53	11.3		dithianon	C14H4O2N2S2	NR	-	-	-	-
4.7	TR9	7.2	7.2		methoxychlor, p, p'-	C16H15Cl3O2	C	C	C	6	2
4.8	NI(WB)6	7.7	4.8		dinocap*	C18H24N2O6	C	P	P	15	2
4.8	NI7/NI(WB)0.2/NP30	7	5.7		fenpropathrin	C22H23NO3	-	V #	V	15	2
4.8	FP15	9.7	6.8		piperophos	C14H28NO3PS2	C	-	-	-	-
4.8	NP(WB)40	6.6	6.3		cloquintocet-mexyl	C18H22ClNO3	V	NR	-	6-15-50	1-2-3
4.9	NI8	3.8	4.5		bifenthrin	C23H22ClF3O2	V	C	-	6+15	2
5	NI550/NP210	-	-		NTN35884*	C9H9N5O2Cl	-	NR	NR	6-15-50	1-2-3
5	NP50	-	7.3		fenoxycarb	C17H19NO4	C	-	-	-	-
5	HX16/NI4	14.9	8.8		bifenox	C12H9Cl2NO5	C	C	P	15+50	2+3
5.1	NI(WB)6	9.5	5.6		dinocap*	C18H24N2O6	C	P	P	15	2
5.1	FP(WB)80	-	10.6		sulprofos oxygen analog sulfone	C12H19O5PS2	C	-	-	-	-
5.1	FP3/TI20	-	9.2		carbophenothion sulfone	C11H16ClO4PS3	C	C	P	6	1
5.2	FP50/NP55	-	8.1		fenamiphos sulfoxide	C13H22N04PS	C	NR	NR	6-15-50	1-2-3
5.2	TR6	-	8.3		tetradifon	C12H6Cl4O2S	C	C	C	15	2
5.2	TI30/TR50	-	11.8		azinphos-methyl	C10H12N3O3PS2	C	NR	NR	6-15-50	1-2-3
5.3	TR35	3.28	-		2,4,5-T BEP ester*	C17H23Cl3O3	-	-	-	-	-
5.3	HX20/NI9	18.8	9.1		chlorthiophos sulfone	C11H15Cl2O5PS2	C	C	-	50	3
5.3	HX(WB)20/NI1000	-	7.5		iprodione metabolite isomer	C13H13Cl2N3O3	C	S	-	50	-
5.4	NI500/NI(WB)40	4.8	6.5		phenothrin*	C23H26O3	-	-	-	-	-
5.4	NP20	-	5.3		carbosulfan	C20H32N2O3S	P	-	-	-	-
5.4	FP3/TI35	-	4		carbophenothion sulfoxide	C11H16ClO3PS3	-	-	-	-	-
5.5	TR850	-	-		diisooctyl phthalate*	C24H38O4	-	C	C	15+50	-
5.5	NP10/TR15	5.5	9.1		phosalone	C12H15ClNO4PS2	C	C	C	50	2+3

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Appendix I: PESTDATA Chemicals in Order by OV-101 Relative Retention Time (continued)

RRT/c OV-101	Detector Responses with OV-101 Column	RRT/c OV-225	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
5.8	FP14/TI20/TR11	7.7	8.5	leptophos	C13H10BrCl2O2PS	C	C	C	6	2
5.8	NI7	2.95	5.6	mirex	C10Cl12	P	C	P	6	1
5.9	HN(WB)10.5/HX20/NI100	-	9.8	clofentezine	C14H8Cl2N4	R	S	-	15	2
6.1	NI100/NP10	1.48	4.5	tralkoxydim*	C20H27NO3	V	NR	NR	50	1-2-3
6.1	FP(WB)14	-	11.7	sulprofos sulfoxide*	C12H19O3PS3	C	-	-	-	-
6.2	TR850	-	-	diisooctyl phthalate*	C24H38O4	-	C	C	15+50	-
6.4	NI(V)200	4.5	6.1	bis(2-ethylhexyl) phthalate	C24H38O4	-	C	C	15+50	-
6.5		-	11.5	myclobutanil dihydroxy metabolite	C15H17N4O2Cl	NR	NR	NR	6-15-50	1-2-3
6.5	TI30/TR28	-	14.3	dialifor	C14H17ClNO4PS2	C	C	P	15	2
6.6	HX10/NI5	-	10.1	fenarimol	C17H12Cl2N2O	C	P #	C #	50	3
6.6	TR500	-	-	n-acetyl nitrofen	C14H11Cl2NO2	-	-	-	-	-
6.7	TR850	-	-	diisooctyl phthalate*	C24H38O4	-	C	C	15+50	-
6.7	NI1000	-	1.59	CGA 205375	C16H13N3O2Cl2	-	-	-	-	-
6.7	NI13	-	-	PPG-1576	C19H17ClF3NO5	-	-	P	50	2+3
6.9	TI58/TR200	-	14.8	azinphos-ethyl	C12H16N3O3PS2	C	P	S	50	3
7	TR35	7.7	-	2,4,5-T BEP ester*	C17H23Cl3O3	-	-	-	-	-
7	NI30/NP4000	-	11	tebufenozide	C22H28N2O2	-	NR	NR	6-15-50	1-2-3
7	NI40	-	11.4	CGA 118244	C15H13Cl2N3O3	V	NR	NR	6-15-50	1-2-3
7.2	FP(WB)16	-	13.1	sulprofos sulfone	C12H19O4PS3	C	-	-	-	-
7.3	NI10	-	-	lactofen	C19H15ClF3NO7	-	-	C	50	2+3
7.4	NI10	-	8	lambda-cyhalothrin	C23H19ClF3NO3	C	-	-	-	-
7.5	TR850	-	-	diisooctyl phthalate*	C24H38O4	-	C	C	15+50	-
7.9	NI90/NP300	-	-	RH-6467*	C19H15N4ClO	S	NR	NR	6-15-50	1-2-3
7.9	NI20	10.8	12.6	HOE-030291	C17H16Cl2O5	-	-	-	-	-
8	NI200/NP130	45	16	coumaphos oxygen analog	C14H16ClO6P	C	NR	NR	6-15-50	1-2-3
8.1	NI250	11.3	10.5	fenoxaprop ethyl ester	C18H16NO5Cl	S	V	V	50	3
8.1	FP(WB)12	-	13	pyrazophos	C14H20N3O5PS	C	-	-	-	-
9	TR850	-	-	diisooctyl phthalate*	C24H38O4	-	C	C	15+50	-
9	NI39/NP38	40	18	coumaphos	C14H16ClO5PS	C	NR	C #	6-15-50	3
9.4		-	11.8	bitertanol*	C20H23N3O2	C	-	-	-	-
9.4	NI75	11.1	13.8	permethrin, cis-	C21H20Cl2O3	C	V #	C	6+15	2
9.5	FP100/NI(WB)9/TI190	-	20.2	bensulide	C14H24NO4PS3	C	P	C	50	3

Appendix I: PESTDATA Chemicals in Order by OV-101 Relative Retention Time (continued)

RRT/c OV-101	Detector Responses with OV-101 Column	RRT/c OV-225	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
9.7		-	12.5	bitertanol*	C20H23N3O2	C	-	-	-	-
9.7	TR7	-	-	hexachlorophene dimethyl ether	C15H10Cl6O2	-	NR	NR	6-15	-
9.8	NI1000/NP70	-	-	fenbuconazole	C19H17ClN4	C	NR	NR	6-15-50	1-2-3
10.2	TR60	13	-	2,4-D BEP ester*	C17H24Cl2O4	-	-	-	-	-
10.2	NI100	13	15	permethrin, trans-	C21H20Cl2O3	C	V #	C	6+15	2
10.4	NI90/NP300	-	-	RH-6467*	C19H15N4ClO	S	NR	NR	6-15-50	1-2-3
10.4	NI15/NP125	12.8	8.9	acrinathrin	C26H21F6NO5	V	V	V#	15	2
10.4	HX50/NI12	-	15.4	prochloraz	C15H16Cl3N3O2	C	-	-	-	-
10.5	TR850	-	-	diisooctyl phthalate*	C24H38O4	-	C	C	15+50	-
11.5	NI500/NI(WB)40	10.9	15	phenothrin*	C23H26O3	-	-	-	-	-
11.7	HX30/NI30	-	-	cyfluthrin*	C22H18Cl2FNO3	C	P	-	15	-
12	NI50	8.9	6.1	CGA 205374	C16H11N3O2Cl2	-	NR	NR	6-15-50	1-2-3
12	NI50/NP170	-	-	RH-9130	C19H16N3ClO2	P	NR	NR	6-15-50	1-2-3
12	NI(V)330	-	-	di-n-octyl phthalate	C24H38O4	-	C	C	15+50	-
12.4	NP(WB)14	-	-	flumetsulam, methylated	C13H11F2N5O2S	-	-	-	-	-
12.5	HX30/NI30	-	-	cyfluthrin*	C22H18Cl2FNO3	C	P	-	15	-
12.8	HX30/NI30	-	-	cyfluthrin*	C22H18Cl2FNO3	C	P	-	15	-
13	NI(WB)2	13	16	hexachlorophene	C13H6Cl6O2	-	NR	NR	6-15-50	-
13.6	HX70/NI80	-	25	quizalofop ethyl ester	C19H17ClN2O4	C	-	-	-	-
14	NI40/NP190	-	-	RH-9129	C19H16N3ClO2	V	NR	NR	6-15-50	1-2-3
14	HX9/NI22	-	-	alpha-cypermethrin	C22H19Cl2O3N	C	C	-	-	2
14	NI(WB)90	-	-	azafenidin	C15H13Cl2N3O2	V	-	-	-	-
14.1	NI90	33	23	cypermethrin*	C22H19Cl2NO3	C	C	C	15	2
14.7	NI40/NI(WB)15	36.9	21.4	flucythrinate*	C26H23F2NO4	C	C	-	15	2+3
15	NI90/NP300	-	-	RH-6467*	C19H15N4ClO	S	NR	NR	6-15-50	1-2-3
15.1	NI90	36	25	cypermethrin*	C22H19Cl2NO3	C	C	C	15	2
16.1	NI40/NI(WB)15	42	24	flucythrinate*	C26H23F2NO4	C	C	-	15	2+3
16.3	HX400/NI1500	24	-	fluridone	C19H14F3NO	-	NR	NR	6-15-50	-
17	NI200	-	6.2	deltamethrin, trans-*	C22H19Br2NO3	-	P #	NR	15	2
17.1	NI1300	-	21	deltamethrin*	C22H19Br2NO3	C	S #	P	15	2
20.3	NI90	44	35	fenvalerate*	C25H22ClNO3	C	C	C	15	2
20.7	NI500/NP500	-	-	WAK4103*	C9H9N5O3Cl	-	NR	NR	6-15-50	1-2-3

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Appendix I: PESTDATA Chemicals in Order by OV-101 Relative Retention Time (continued)

RRT/c OV-101	Detector Responses with OV-101 Column	RRT/c OV-225	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
22.5	NI90	51	40	fenvalerate*	C25H22ClNO3	C	C	C	15	2
22.5	NI90	-	-	esfenvalerate	C25H22ClNO3	C	C	C	15	2
23	NI150/NP300	57	43	PB-7, methylated	C20H25ClN2O3S	-	-	-	-	-
25		59	38	fluvalinate*	C26H22ClF3N2O3	C	C	-	15	2
25	NI300/NP500	87	46	PB-9	C19H25ClN2O2S	V	NR	NR	6-15-50	1-2-3
27	NI1300	-	35	deltamethrin*	C22H19Br2NO3	C	S #	P	15	2
27	NI30/NI(WB)1	64	44	tralomethrin	C22H19Br4NO3	C	V	S	15	2
29	NI200	-	20	deltamethrin, trans-*	C22H19Br2NO3	-	P #	NR	15	2
29	NI1300	19.9	38	deltamethrin*	C22H19Br2NO3	C	S #	P	15	2
31	NI200	19.7	38	deltamethrin, trans-*	C22H19Br2NO3	-	P #	NR	15	2
32	NI(V)250	4.5	-	hydramethylnon*	C25H24F6N4	-	-	-	-	-
44	NI(V)250	53	-	hydramethylnon*	C25H24F6N4	-	-	-	-	-

Appendix I: PESTDATA Chemicals in Order by OV-225 Relative Retention Time

RRT/c OV-225	Detector Responses with OV-225 Column	RRT/c OV-101	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
0.08		-	-	2,4-D BEP ester*	C17H24Cl2O4	-	-	-	-	-
0.08	NI40	0.06	0.04	CGA 171683	C6H5F4N3O2	C		-	15+50	3
0.08		0.19	0.1	2-methoxy-3,5,6-trichloropyridine	C6H4Cl3NO	C	P#	C	6+15	1+2
0.08	FP(WB)0.7	0.07	0.08	dichlorvos	C4H7Cl2O4P	C	NR	NR	6-15-50	1-2-3
0.09	NI27	0.11	0.11	diuron	C9H10Cl2N2O	C	NR	NR	6-15-50	1-2-3
0.11		-	0.09	teflubenzuron*	C14H6Cl2F4N2O2	-	NR	NR	6-15-50	1-2-3
0.12	NI0.2	0.18	0.21	etridiazole	C5H5Cl3N2OS	C	C	P	6	2
0.13	NI0.25	0.24	0.16	pentachlorobenzene	C6HCl5	C	C	C	6	1
0.14		0.16	-	2,4,5-T BEP ester*	C17H23Cl3O3	-	-	-	-	-
0.14	NI12	0.17	0.1	N-(3,4-dichlorophenyl)-N'-methylurea	C8H8Cl2N2O	-	NR	NR	6-15-50	-
0.14	NI6	0.22	0.1	3,4-dichlorophenylurea	C7H6Cl2N2O	-	NR	NR	6-15-50	-
0.15		0.13	0.1	chlorimuron ethyl ester	C15H15ClN4O6S	P	NR	-	-	-
0.15	NI0.2	0.24	0.22	2,3,5,6-tetrachloroanisole	C7H4Cl4O	-	C	-	6	1
0.15		0.15	0.14	dimethyl phthalate	C10H10O4	-	P	-	6+15+50	-
0.18		0.19	-	dicamba methyl ester	C8H6Cl2O3	-	-	-	-	-
0.18	NI0.9	0.2	0.2	nitrapyrin	C6H3Cl4N	C	C	V	6	2
0.19		0.19	-	chloroneb	C8H8Cl2O2	C	C	-	6	2
0.19		-	-	molinate	C9H17NOS	-	-	-	-	-
0.21		0.23	0.18	methyl 2,3,6-trichlorobenzoate	C8H5Cl3O2	-	-	-	-	-
0.22	NI2	0.17	0.13	3-methyl-4-nitrophenol methyl ether	C8H9O3N	-	-	-	-	-
0.22	NI1	0.19	0.14	N, N-diallyl dichloroacetamide	C8H11Cl2NO	C	S	S	15+50	2+3
0.23		0.14	0.14	teflubenzuron*	C14H6Cl2F4N2O2	-	NR	NR	6-15-50	1-2-3
0.23	FP0.5/NI0.3	0.33	0.24	chlorethoxyfos	C6H11Cl4O3PS	V	C	-	6	1
0.23	FS25	0.13	0.11	carboxin sulfoxide	C12H13NO3S	-	NR	NR	6-15-50	1-2-3
0.24		0.15	-	hydroxy chloroneb	C7H6Cl2O2	-	NR	-	6-15	-
0.24		-	-	carbofuran-3-keto-7-phenol	C10H10O3	-	-	-	-	-
0.25	NI100	0.22	0.2	3-carboxy-5-ethoxy-1,2,4-thiadiazole	C3H2N2O3S	NR	-	-	-	-
0.25	FP4	0.07	0.09	methamidophos	C2H8NO2PS	V	-	-	-	-
0.25	NI0.3	0.45	0.33	hexachlorobenzene	C6Cl6	C	C	P	6	1

* Multipeak chemical.

\# Recovery may vary with choice of Florisil elution system; see Tables 303-a, 304-a.

Appendix I: PESTDATA Chemicals in Order by OV-225 Relative Retention Time (continued)

RRT/c OV-225	Detector Responses with OV-225 Column	RRT/c OV-101	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
0.26		0.42	0.33	di-allate	C10H17ClNOS	C	C	-	6	-
0.26		0.29	0.24	tecnazene	C6HCl4NO2	C	C	C	6	1
0.27	FP(WB)1	0.37	0.29	cadusafos	C10H23O2PS2	C	NR	NR	6-15-50	1-2-3
0.27	NI0.4	0.34	0.19	ethalfluralin	C13H14F3N3O4	C	C	C	6	2
0.27		0.34	0.17	trifluralin	C13H16F3N3O4	C	C	C	6	2
0.27	NI20/NI(WB)25	0.18	0.14	3,5-dichloroaniline	C6H5Cl2N	S	S	S	6+15	1+2
0.27	NI13700	0.21	0.24	TEPP	C8H20O7P2	C	-	-	-	-
0.28	NI2	0.37	0.18	benfluralin	C13H16F3N3O4	C	C	C	6	2
0.29	NI1000	0.44	0.45	desmedipham	C16H16N2O4	-	-	-	-	-
0.3		0.46	0.34	pentachlorophenyl methyl ether	C7H3Cl5O	C	C	C	6	1
0.31		0.33	0.25	ethoprop	C8H19O2PS2	C	P #	S #	50	1-2-3
0.32		0.22	0.21	demeton-O oxygen analog	C8H19O4PS	-	-	-	-	-
0.32	NI30	0.2	0.16	3,4-dichloroaniline	C6H5Cl2N	V	S	-	15	-
0.35	NI0.3	0.49	0.48	1,2,4,5-tetrachloro-3-(methylthio)= benzene	C7H4Cl4S	R	C	-	6	1
0.36		0.36	0.39	triclopyr methyl ester	C8H6Cl3NO3	-	-	-	-	-
0.37	FP0.5	0.3	0.29	phorate oxygen analog	C7H17O3PS2	C	NR	NR	6-15-50	1-2-3
0.37	NI9/NI(WB)5	0.34	0.26	propachlor	C11H14ClNO	C	NR	NR	6-15-50	1-2-3
0.38		0.3	0.25	2,4-D methyl ester	C9H8Cl2O3	-	-	-	-	-
0.38	FP0.5/NI17	0.37	0.32	phorate	C7H17O2PS3	C	V #	V #	6	1
0.4	NI0.3	0.56	0.32	chlordene	C10H6Cl6	-	C	C	6	1
0.4	FP6/NI4.5	0.51	0.44	diazinon	C12H21N2O3PS	C	C	C	15	3
0.42	NP20	0.47	0.53	terbumeton	C10H19N5O	C	-	-	-	-
0.43		0.32	0.25	chlorpropham	C10H12ClNO2	C	C	C	15	2
0.44		0.32	0.36	3, 5, 6-trichloro-2-pyridinol methyl ester	C6H4Cl3NO	-	-	-	-	-
0.44		0.45	-	silvex methyl ester	C10H9Cl3O3	-	-	-	-	-
0.44		0.5	0.41	terbufos	C9H21O2PS3	C	P	S	6	-
0.44	NI1	0.38	0.36	sulfallate	C8H14ClNS2	C	C	C	6+15	2
0.46	NI1	0.53	0.3	profluralin	C14H16F3N3O4	V	V	-	6	-
0.46		0.51	0.46	quintozene	C6Cl5NO2	C	C	C	6	1
0.48		0.4	0.35	BHC, alpha-	C6H6Cl6	C	C	C	6	1

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Appendix I: PESTDATA Chemicals in Order by OV-225 Relative Retention Time (continued)

RRT/c OV-225	Detector Responses with OV-225 Column	RRT/c OV-101	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
0.49		0.31	0.32	metasystox thiono*	C6H15O3PS2	-	-	-	-	-
0.49	NI(WB)1.5	0.56	0.47	methyl 3,5-dibromo-4-methoxy= benzoate	C9H8Br2O3	-	-	-	-	-
0.49		0.28	0.32	metasystox thiol	C6H15O3PS2	C	-	-	-	-
0.49		0.46	0.31	oxydemeton-methyl	C6H15O4PS2	C	-	-	-	-
0.5	NI(WB)70	0.25	0.16	cymoxanil	C7H10N4O3	V	NR	NR	6-15-50	1-2-3
0.51	NI0.5	0.26	0.23	RPA 203328, methylated	C10H9F3O4S	-	-	-	-	-
0.51		0.41	0.4	thiometon	C6H15O2PS3	C	NR	NR	6-15-50	-
0.52		0.83	0.6	heptachlor	C10H5Cl7	C	C	C	6	1
0.53	NI600	0.5	0.24	phosmet oxygen analog*	C11H12NO5PS	-	NR	NR	6-15-50	-
0.53	NI60	0.5	0.47	diazinon oxygen analog	C12H21N2O4P	C	NR	NR	6-15-50	1-2-3
0.53		0.39	0.38	fonofos oxygen analog	C10H15O2PS	V	NR	NR	6-15-50	1-2-3
0.56		0.52	0.44	fonofos	C10H15OPS2	C	C	C	6	2+3
0.56		0.41	0.41	demeton-S	C8H19O3PS2	C	NR	-	6-15-50	-
0.58		1.05	0.76	aldrin	C12H8Cl6	C	C	C	6	1
0.59	NI150	0.45	0.46	clomazone	C12H14ClNO2	C		-	50	3
0.59	NI30	0.58	0.51	etrimfos	C10H17N2O4PS	C	C	C	15	2+3
0.59		0.5	0.45	pentachlorobenzonitrile	C7Cl5N	C	C	P	15	2
0.6	NI1.6	0.46	0.39	furilazole	C11H13Cl2NO3	C	S	-	50	3
0.6	NP10	0.35	0.38	2,3,5-trimethacarb	C11H15NO2	C	S #	NR	50	1-2-3
0.6		0.54	0.46	disulfoton	C8H19O2PS3	C	P #	NR	6	1-2-3
0.61		0.65	0.56	diisobutyl phthalate	C16H2204	-	P	-	15+50	-
0.62		-	-	2,4-D isopropyl ester*	C11H12Cl2O3	-	-	-	-	-
0.62		0.62	0.49	2,4-D isobutyl ester	C12H14Cl2O3	-	-	-	-	-
0.63		0.49	0.47	2,4,5-T methyl ester	C9H7Cl3O3	-	-	-	-	-
0.63	NI250	0.38	0.26	3-methyl-4-nitrophenol	C7H7O3N	V	NR	NR	6-15-50	1-2-3
0.63	NI0.4	0.56	0.56	2,3,5,6-tetrachloronitroanisole	C7H3Cl4NO3	-	C	-	6	1+2
0.64		0.82	0.67	chlordene, alpha-	C10H6Cl6	-	-	-	-	-
0.64	FP5	0.15	0.19	acephate	C4H10NO3PS	C	-	-	-	-
0.64		0.67	0.56	dichlofenthion	C10H13Cl2O3PS	C	C	V	6	2
0.65		1.34	1.43	merphos*	C12H27PS3	-	C	C	6+15+50	3
0.65		0.84	-	chlordene epoxide	C10H6Cl6O	-	C	-	15	-

Appendix I: PESTDATA Chemicals in Order by OV-225 Relative Retention Time (continued)

RRT/c OV-225	Detector Responses with OV-225 Column	RRT/c OV-101	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
0.65	NI37	0.53	0.41	propazine	C9H16ClN5	C	S	NR	15+50	3
0.65		0.67	-	2,4,5-T isopropyl ester	C11H11Cl3O3	-	-	-	-	-
0.66		0.68	-	2,4,5-T BEP ester*	C17H23Cl3O3	-	-	-	-	-
0.69	NI1.6	0.5	0.48	4-(dichloroacetyl)-1-oxa-4-azapiro=[4.5]decane	C10H15Cl2NO2	C	P	-	50	3
0.69	NI0.5	0.94	0.87	pentachlorophenyl methyl sulfide	C7H3Cl5S	C	C	C	6	1
0.69		0.48	0.47	lindane	C6H6Cl6	C	C	C	6	1
0.7	NI2550	0.35	0.41	methabenzthiazuron	C10H11N3OS	C	NR	NR	6-15-50	1-2-3
0.71	NI43	0.47	0.48	terbuthylazine	C9H16N5Cl	C	P	-	15+50	-
0.72		0.62	-	2,4-DB methyl ester	C11H12Cl2O3	-	-	-	-	-
0.73	NI0.5	0.59	0.66	2,3,5,6-tetrachloroanisidine	C7H5Cl4NO	-	C	-	6	2
0.74		0.69	-	2,4-D BEP ester*	C17H24Cl2O4	-	-	-	-	-
0.74		0.42	0.33	2,4-D isopropyl ester*	C11H12Cl2O3	-	-	-	-	-
0.74		0.43	0.44	atrazine	C8H14ClN5	C	S #	NR	50	1-2-3
0.75		0.42	0.45	chlorbufam	C11H10ClNO2	C		-	15	2+3
0.76		0.53	0.37	fluchloralin	C12H13ClF3N3O4	C	C	-	6	2
0.78	NP200	0.45	0.5	3,4,5-trimethacarb	C11H15NO2	C	NR	NR	50	1-2-3
0.79	NI160	0.5	0.44	metribuzin, deaminated diketo metabolite*	C7H11N3O2	NR	NR	NR	6-15-50	1-2-3
0.79	NI0.5	0.67	0.66	pentachloroaniline	C6H2Cl5N	C	C	C	6	1
0.8		0.51	0.63	etrimfos oxygen analog	C10H17N2O5P	C	-	-	-	-
0.8	NI20	0.55	0.63	isazofos	C9H17ClN3O3PS	C	C #	-	50	2+3
0.8	NI2	0.75	0.68	CGA 14128	C12H21N2O4PS	C		-	50	1-2-3
0.8	NI80	0.3	0.53	desethyl simazine	C5H8ClN5	-	NR	NR	50	1-2-3
0.83	NI130	0.41	0.5	simazine	C7H12ClN5	C	NR	NR	50	1-2-3
0.84		0.98	0.89	chlordene, beta-	C10H6Cl6	-	-	-	-	-
0.84	NI3	0.51	0.4	pronamide	C12H11Cl2NO	C	P	-	15+50	-
0.85	NI1	0.81	0.75	tridiphane	C10H7Cl5O	C	C	-	6	1+2
0.85		0.81	-	chloroxuron	C15H15ClN2O2	C	NR	NR	6-15-50	1-2-3
0.86		0.72	0.79	chlorpyrifos-methyl	C7H7Cl3NO3PS	C	C	-	6	2
0.86	NI20	0.2	0.61	desdiethyl simazine	C3H4ClN5	-	NR	NR	6-15-50	1-2-3
0.86		0.55	1.04	tetraiodoethylene	C2I4	-	P	P	6	-

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RRT/c OV-225	Detector Responses with OV-225 Column	RRT/c OV-101	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
0.86	NI2	0.81	0.76	ronnel	C8H8Cl3O3PS	C	C	C	6	2
0.88	NI5	0.75	0.67	acetochlor	C14H20NO2Cl	C	C #	P	50	3
0.89	NI1	0.98	0.88	chlordene, gamma-	C10H6Cl6	-	-	-	-	-
0.91		1.08	-	2,4,5-T BEP ester*	C17H23Cl3O3	-	-	-	-	-
0.91	NI180	0.48	0.48	monolinuron	C9H11ClN2O2	C	-	-	-	-
0.92	NI(WB)13	0.25	0.28	oxamyl oxime metabolite	C5H10N2O2S	C	NR	NR	6-15-50	1-2-3
0.92		0.55	-	dichlone	C10H4Cl2O2	P	S #	S #	6-15-50	2+3
0.92		0.88	0.84	dibutyl phthalate	C16H22O4	-	C	C	15+50	-
0.93	NI600	2	0.44	phosmet oxygen analog*	C11H12NO5PS	-	NR	NR	6-15-50	-
0.93	NI1	0.52	0.44	dinitramine	C11H13F3N4O4	C	-	P	15	-
0.94		1.33	1.05	octachlor epoxide	C10H4Cl8O	C	C	C	6	1
0.95		1.53	-	Perthane olefin	C18H19Cl	-	C	C	6	1
0.96		0.31	0.43	dicrotophos	C8H16NO5P	C	NR	-	6-15-50	-
0.96		0.42	0.45	dicloran	C6H4Cl2N2O2	C	S	P	15+50	2+3
0.97	NI(WB)6	0.42	0.49	PPG-947, methylated*	C18H13ClF3NO7	-	-	-	-	-
0.97	NP50	0.94	0.66	prodiamine	C13H17F3N4O4	C	-	-	-	-
0.98		0.72	-	dimethenamid	C12H18ClNO2S	-	NR	NR	6-15-50	1-2-3
1	NI150	1.05	-	PP 890	C9H10O2ClF3	-	-	-	-	-
1		0.94	0.98	thiobencarb	C12H16ClNOS	C		V	15	2+3
1	NI6	0.8	0.72	alachlor	C14H2OClNO2	C	C	C #	50	3
1		1	1	chlorpyrifos	C9H11Cl3NO3PS	C	C	P	6	2
1.02		0.64	0.62	ronnel oxygen analog	C8H8Cl3O4P	C	NR	-	6-15-50	-
1.03		1.14	1.14	pirimiphos-ethyl	C13H24N3O3PS	C	C	C	15+50	3
1.03		0.44	-	chloramben methyl ester	C8H7Cl2NO2	-	-	-	-	-
1.06	NI2	0.64	0.7	benoxacor	C11H11Cl2NO2	C	P	C	15+50	2+3
1.07		0.82	0.92	dichlorobenzophenone, o,p'-	C13H8Cl2O	-	C	C	15	2
1.08		4.1	0.91	dicofol, o,p'-*	C14H9Cl5O	C	V	S	6+15	2
1.08		0.84	-	terbutryn	C10H19N5S	C	-	-	-	-
1.1		0.77	-	ametryn	C9H17N5S	C	-	-	-	-
1.11	NI20	0.71	0.71	dimethachlor	C13H18ClNO2	C	-	-	-	-
1.11	FP5	0.25	0.39	omethoate	C5H12NO4PS	C	NR	NR	6-15-50	1-2-3
1.13	NI1	1.06	1	DCPA	C10H6Cl4O4	C	C	C	15	2

Appendix I: PESTDATA Chemicals in Order by OV-225 Relative Retention Time (continued)

RRT/c OV-225	Detector Responses with OV-225 Column	RRT/c OV-101	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
1.15		0.69	0.64	vinclozolin	C12H9Cl2NO3	C	C	C	15	2
1.15		1.19	1.2	TDE, o,p'-, olefin	C14H9Cl3	-	-	-	-	-
1.16	NI(WB)55	0.39	0.54	4-chlorobenzylmethyl sulfoxide	C8H9ClOS	-	NR	NR	6-15-50	1-2-3
1.18		1.66	-	2,4-D BEP ester*	C17H24Cl2O4	-	-	-	-	-
1.19		2.85	-	2,4,5-T BEP ester*	C17H23Cl3O3	-	-	-	-	-
1.2	NI(WB)8	0.74	0.66	vinclozolin metabolite B	C12H11Cl2NO4	C	P #	C	6+15	2
1.21	NI9	1.03	0.93	metolachlor	C15H22ClNO2	C	S #	NR	50	1-2-3
1.21		2.02	-	simetryn	C8H15N5S	C	-	-	-	-
1.22	NI8	1.15	0.93	butralin	C14H21N3O4	V	C	-	6+15+50	-
1.22	NI2	1.37	1.12	S-bioallethrin	C19H26O3	-	C	-	50	-
1.22		0.64	0.74	cyprazine	C9H14ClN5	C	-	-	-	-
1.22		1.29	1.15	heptachlor epoxide	C10H5Cl7O	C	C	C	6	2
1.22		1.36	-	allethrin	C19H26O3	-	C	C #	50	3
1.24	NI2	1.14	1.01	isopropalin	C15H23N3O4	C	C	-	6	-
1.25		0.99	1.08	dichlorobenzophenone, p,p'-	C13H8Cl2O	-	C	C	15	2
1.28		4.4	1.08	dicofol, p,p'-*	C14H9Cl5O	C	V	P #	6+15	1+2
1.28		1.55	1.51	DDE, o,p'-	C14H8Cl4	C	C	C	6	1
1.29	NI160	0.73	0.52	metribuzin, deaminated diketo metabolite*	C7H11N3O2	NR	NR	NR	6-15-50	1-2-3
1.29	NI6	1.11	1.16	bromophos	C8H8BrCl2O3PS	C	C	C	6	-
1.3		0.39	0.52	2,6-dichlorobenzamide	C7H5NOCl2	C	NR	NR	6-15-50	1-2-3
1.34	NI(WB)51	0.43	0.6	6-chloro-2,3-dihydro-3,3,7-methyl-5H-oxazolo(3,2-a)pyrimidin-5-one	C9H13ClN2O2	-	NR	NR	6-15-50	1-2-3
1.36	NI3	1.45	1.45	TDE, p,p'-, olefin	C14H9Cl3	C	C	C	6	1
1.38		1.64	1.47	endosulfan I	C9H6Cl6O3S	C	C	C	15	2
1.4		0.6	0.78	ethiofencarb	C10H15NO2S	C	NR	NR	6-15-50	-
1.41	NI200	3.38	4.9	desmethyl norflurazon	C11H7ClF3N3O	V	NR	NR	6-15-50	1-2-3
1.41	NI15	0.56	0.55	metribuzin, diketo metabolite	C7H12N4O2	NR	NR	NR	6-15-50	1-2-3
1.41	NI(WB)80	0.55	0.9	3-ketocarbofuran	C12H12NO4	S	NR	NR	6	1
1.42		-	-	2,4-D propylene glycol butyl ether ester*	C15H2OCl2O4	-	-	-	-	-
1.42		1.51	1.45	bromophos-ethyl	C10H12BrCl2O3PS	C	C	P	6	-

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RRT/c OV-225	Detector Responses with OV-225 Column	RRT/c OV-101	RRT/c OV-17	Name	Molecular Formula	Recoveries				
						302	303	304	Ethers	CH_2Cl_2
1.44		0.67	0.69	metobromuron	C9H11BrN2O2	C	NR	NR	6-15-50	1-2-3
1.44		0.55	0.74	chlorothalonil	C8Cl4N2	S	C #	C #	6-15-50	2+3
1.45	NI1.5	0.96	0.86	nitrofluorfen	C13H7ClF3NO3	C	C	C	15	2
1.45		1.75	1.42	nonachlor, trans-	C10H5Cl9	C	C	C	6	1
1.46		0.97	-	methazole	C9H6Cl2N2O3	-	-	-	-	-
1.46		1.49	1.34	chlordane, trans-	C10H6Cl8	C	C	C	6	1
1.46		0.96	1.18	fenthion	C10H15O3PS2	C	S #	NR	6+15	1-2-3
1.47	NI(WB)300	1.05	0.88	acifluorfen	C14H7ClF3NO3	-	NR	NR	6-15-50	1-2-3
1.47		0.54	0.62	fenfuram	C12H11NO2	C	-	-	-	-
1.47	NI0.7	0.57	0.91	metribuzin	C8H14N4OS	V	NR	NR	50	1-2-3
1.48	NI100	6.1	4.5	tralkoxydim*	C20H27NO3	V	NR	NR	50	1-2-3
1.48	NI3	1.22	1.21	pendimethalin	C13H19N3O4	C	C	P	15	2
1.49	NI44	0.91	1.05	malathion	C10H19O6PS2	C	C	C	15+50	3
1.51		-	-	2,4-D ethyl hexyl ester*	C16H22Cl2O3	-	-	-	-	-
1.51		0.95	1.08	chlorpyrifos oxygen analog	C9H11Cl3NO4P	C	NR	-	6-15-50	-
1.54		1.66	1.48	chlordane, cis-	C10H6Cl8	C	C	C	6	1
1.55		0.75	0.79	prothoate	C9H20NO3PS2	C	-	-	-	-
1.55		0.68	0.87	malathion oxygen analog	C10H19O7PS	C	NR	NR	6-15-50	1-2-3
1.58	NI5	1.21	1.29	chlorfenvinphos, alpha-	C12H14Cl3O4P	C	-	NR	6-15-50	-
1.59		1.92	1.86	DDE, p,p'-	C14H8Cl4	C	C	C	6	1
1.6	FP3	0.31	0.5	monocrotophos	C7H14NO5P	C	NR	NR	6-15-50	1-2-3
1.6		0.4	0.62	dimethoate	C5H12NO3PS2	C	NR	NR	6-15-50	1-2-3
1.61		2.04	2.69	cyproconazole	C15H18ClN3O	C	NR	NR	6-15-50	1-2-3
1.62	NI1	0.77	0.99	Tycor	C9H16N4OS	C	S	S	50	3
1.62		0.43	0.56	BHC, beta-	C6H6Cl6	C	C	C	6	1
1.62		1.39	1.54	chlorbenside	C13H10Cl2S	C	S	P	6	1
1.63	NI1	0.99	1.07	1-hydroxychlordene	C10H6Cl6O	-	R	-	15	-
1.64		1.95	1.88	merphos*	C12H27PS3	-	C	C	6+15+50	3
1.64	NI3/NP20	1.05	1	triadimefon	C14H16ClN3O2	C	S #	S #	50	1-2-3
1.64	FP1/NI11	0.71	0.87	parathion-methyl	C8H10NO5PS	C	C	C	15	2
1.64	NI30	0.6	0.7	ethoxyquin	C14H19N0	C	NR	NR	6-15-50	-
1.65		1.95	1.88	tribufos	C12H27OPS3	C	C	P	15+50	3

Appendix I: PESTDATA Chemicals in Order by OV-225 Relative Retention Time (continued)

RRT/c OV-225	Detector Responses with OV-225 Column	RRT/c OV-101	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
1.67		-	1.44	2,4-D butoxyethyl ester*	C14H18Cl2O4	-	-	-	-	-
1.67	NI6	2.75	2.38	chlordecone	C10H8Cl10O5	-	S #	P #	15+50	1-2-3
1.71		0.9	1.01	dichlofluanid	C9H11Cl2FN2O2S2	C	C #	-	15+50	2+3
1.71		0.55	0.66	parathion-methyl oxygen analog	C8H10NO6P	-	NR	NR	6-15-50	1-2-3
1.71		0.5	0.67	BHC, delta-	C6H6Cl6	C	C	C	6+15	1
1.73		1.36	1.38	isofenphos	C15H24NO4PS	C	C	-	15+50	-
1.74	NI1	1.85	1.82	prothiofos	C11H15Cl2PO2S2	C	C	C	6	2
1.74	FP5	1.17	1.24	isofenphos oxygen analog	C15H24NO5P	C	-	-	-	-
1.76		2.12	1.76	pyrethrins*	C21H27O4	-	C	C	50	-
1.78		2.04	1.78	2,4-D isooctyl ester*	C16H22Cl2O3	-	-	-	-	-
1.78		2.1	1.68	2,4-D ethyl hexyl ester*	C16H22Cl2O3	-	-	-	-	-
1.79		2	-	2,4-D BEP ester*	C17H24Cl2O4	-	-	-	-	-
1.82		0.84	1.05	fenitrothion	C9H12NO5PS	C	C	C	15	2
1.83	NI14	1.73	1.46	butachlor	C17H26ClNO2	C	C	-	50	-
1.84	NI6	1.57	1.64	cyclanilide methyl ester	C12H11Cl2NO3	-	-	-	-	-
1.87		1.91	1.84	dieldrin	C12H8Cl6O	C	C	C	15	2
1.88		1.24	1.47	anilazine	C9H5Cl3N4	V	S	P	15+50	2+3
1.89	NI1000	1.39	1.54	CGA 189138	C13H8O3Cl2	-	-	-	-	-
1.91	NI3/NP65	0.99	0.96	KWG 1323	C14H16ClN3O3	C	NR	NR	6-15-50	1-2-3
1.91	NI(WB)2	0.41	0.66	4-chlorobenzylmethyl sulfone	C8H9ClO2S	-	NR	NR	6-15-50	1-2-3
1.91	FP2/NI6	0.98	1.07	parathion	C10H14NO5PS	C	C	C	15	2
1.93	FS56/NI638	0.86	1.02	ethofumesate	C13H18O5S	C	-	-	-	-
2	FP4/NI5	1.29	1.52	chlorfenvinphos, beta-	C12H14Cl3O4P	C	S #	-	50	1-2-3
2		1.32	1.64	quinalphos	C12H15N2O3PS	C	C	-	15	-
2.01	NI(WB)2	0.69	0.79	vinclozolin metabolite S	C10H7Cl2NO3	V	P	V #	15	2
2.01		2.23	2.42	Perthane	C18H2OCl2	C	C	C	6	1
2.02		0.5	0.5	tris(chloropropyl) phosphate	C9H18Cl3O4P	C	NR	NR	6-15-50	1-2-3
2.03	NI1.5	-	-	fenson	C12H10O3ClS	-	-	-	-	-
2.05		1.31	1.83	phenthoate	C12H17O4PS2	C	C	-	15+50	-
2.08		1.82	1.79	2,4-D butoxyethyl ester*	C14H18Cl2O4	-	-	-	-	-
2.09		3.22	-	2,4-D BEP ester*	C17H24Cl2O4	-	-	-	-	-
2.1	NI(WB)3.8	0.8	-	bromacil methyl ether	C10H16BrN2O2	-	-	-	-	-

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Appendix I: PESTDATA Chemicals in Order by OV-225 Relative Retention Time (continued)

RRT/c OV-225	Detector Responses with OV-225 Column	RRT/c OV-101	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
2.1	NI(WB)7	0.54	0.72	terbacil	C9H13ClN2O2	C	NR	NR	6-15	2+3
2.13		0.85	0.95	linuron	C9H10Cl2N2O2	V	V #	V	50	3
2.22		2.13	2.29	endrin	C12H8Cl6O	C	C #	C #	15	2
2.23		1.44	1.19	triflumizole	C15H15ClF3N3O	C	-	-	-	-
2.25	NI30	0.45	0.71	methidathion sulfoxide	C5H8N2O4S2	-	NR	NR	6-15-50	1-2-3
2.27	NI(WB)630	1.35	2.55	3-tert-butyl-5-chloro-6-hydroxy= methyluracil	C9H13ClN2O3	-	NR	NR	6-15-50	1-2-3
2.27		2.55	2.7	DDT, o,p'-	C14H9Cl5	C	C	C	6	1
2.29	NI400	0.56	0.82	methidathion sulfone	C5H8N2O3S2	-	NR	NR	6-15-50	1-2-3
2.33	NI3	2.64	2.8	tetrasul	C12H6Cl4S	C	C	C	6	1
2.33		1.08	1.3	crufomate	C12H19ClNO3P	C	NR	NR	6-15-50	-
2.33	NI6250	0.5	0.66	ethylenethiourea	C3H6N2S	S	NR	NR	6-15-50	1-2-3
2.34		1.8	2.13	profenofos	C11H15BrClO3PS	C	P	P	50	3
2.36	NI3000	2.3	2.31	fluazifop butyl ester	C19H20F3NO4	C	C	V	15	3
2.46		1.58	2.1	triazamate	C13H22N4O3S	C	NR	NR	6-15-50	1-2-3
2.46		1.9	2.19	TDE, o,p'-	C14H10Cl4	-	C	C	6	1
2.48		1.97	1.96	oxadiazon	C15H18Cl2N2O3	C	C	P	15	-
2.49	NI20	0.44	0.64	prosulfuron*	C15H16F3N5O4S	-	NR	NR	6-15-50	1-2-3
2.55		0.89	1.26	phorate sulfoxide	C7H17O3PS3	C	NR	NR	6-15-50	1-2-3
2.65	NI300	0.6	0.9	CGA 120844	C8H9NSO3	-	NR	NR	6-15-50	1-2-3
2.66		-	-	2,4,5-T butoxyethyl ester*	C14H17Cl3O4	-	-	-	-	-
2.66		2.53	-	Compound K*	C10H6Cl8	-	C	-	-	1
2.67		0.75	-	picloram methyl ester	C7H5Cl3N2O2	-	-	-	-	-
2.67	HN4	1.28	1.58	mecarbam	C10H20NO5PS2	C		-	50	-
2.69		-	-	2,4,5-T isooctyl ester*	C16H21Cl3O3	-	-	-	-	-
2.71	FS2	0.41	0.81	dimethipin	C6H10O4S2	C	NR	NR	6-15-50	1-2-3
2.72		1.58	1.97	Gardona	C10H9Cl4O4P	C	NR	NR	6-15-50	1-2-3
2.73		1.21	1.5	des N-isopropyl isofenphos	C12H18NO4PS	C	S	-	50	-
2.74		3.03	2.88	ethephon	C2H6ClO3P	NR		-	6+15+50	1+2+3
2.77		2	-	aramite*	C15H23ClO4S	C	P	NR	15	-
2.78		3.3	-	2,4,5-T BEP ester*	C17H23Cl3O3	-	-	-	-	-
2.82	NI8	0.66	0.78	propanil	C9H9Cl2NO	C	NR	NR	6-15	3

Appendix I: PESTDATA Chemicals in Order by OV-225 Relative Retention Time (continued)

RRT/c OV-225	Detector Responses with OV-225 Column	RRT/c OV-101	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
2.84		2.95	2.7	pyrethrins*	C21H27O4	-	C	C	50	-
2.85	NI(WB)780	0.35	0.4	ethametsulfuron methyl ester*	C15H18N6O6S	-	NR	NR	6-15-50	1-2-3
2.85		1.37	1.9	crotoxyphos	C14H19O6P	C	NR	NR	6-15-50	1-2-3
2.89	FS63/NI10	0.68	0.93	2,3-dihydro-3,3-methyl-2-oxo-5-benzofuranyl methyl sulfonate	C11H12O5S	-	-	-	-	-
2.9	FP5/NI1500	0.92	1.28	terbufos oxygen analog sulfone	C9H21O5PS2	C	NR	NR	6-15-50	1-2-3
2.9		2.33	2.41	chloropropylate	C17H16Cl2O3	P	C	C	15+50	3
2.91		1.86	1.79	hexaconazole	C14H17Cl2N3O	C	-	-	-	-
2.95		0.71	0.96	demeton-O sulfone*	C8H19O5PS2	C	-	-	-	-
2.95		5.8	5.6	mirex	C10Cl12	P	C	P	6	1
3	FP15/NI300	1.08	1.66	fosthiazate	C9H18NO3PS2	C	NR	NR	6-15-50	1-2-3
3.01		1.23	1.94	folpet	C9H4Cl3O2NS	C	C	P	15+50	2+3
3.02	NI(WB)9	0.89	0.93	vinclozolin metabolite E	C11H11Cl2NO2	C	S	NR	15+50	-
3.02	NI(WB)10	2	3.38	kresoxim-methyl	C18H19NO4	P	C	C	15+50	3
3.04	NI7	1.37	1.49	procymidone	C13H11Cl2NO2	C	C	P	15	-
3.04	NI3	1.61	2.2	ovex	C12H8Cl2O3S	C	C	C	15	2
3.05		2.14	-	aramite*	C15H23ClO4S	C	P	NR	15	-
3.1		2.56	-	2,4,5-T isooctyl ester*	C16H21Cl3O3	-	-	-	-	-
3.14	NI14	1.78	2	diethatyl-ethyl	C16H22ClNO3	C	NR	NR	6-15-50	1-2-3
3.16	NI(WB)120	1.9	1.86	PPG-2597	C20H17ClF3NO6	-	NR	NR	6-15-50	1-2-3
3.24	NI5	2.38	3.14	leptophos photoproduct	C13H11Cl2O2PS	C	-	-	-	-
3.26		0.97	1.3	phorate sulfone	C7H17O4PS3	C	S #	S #	6-15-50	3
3.26		2.31	2.61	chlorobenzilate	C16H14Cl2O3	C	C #	P #	15+50	3
3.28		5.3	-	2,4,5-T BEP ester*	C17H23Cl3O3	-	-	-	-	-
3.3		2.91	-	2,4,5-T butoxyethyl ester*	C14H17Cl3O4	-	-	-	-	-
3.33	NI3	2.52	2.61	nonachlor, cis-	C10H5Cl9	C	C	C	6	1
3.33	NI50	1.4	2.28	methidathion	C6H11N2O4PS3	C	S	P #	50	3
3.39		1.27	1.42	chlorbromuron	C9H10BrClN2O2	V	V	V	50	3
3.4		2.96	-	2,4,5-T isooctyl ester*	C16H21Cl3O3	-	-	-	-	-
3.4	NI7	2.02	2.03	diclobutrazol	C15H19Cl2N3O	C	NR	NR	6-15-50	1-2-3
3.49		1.2	1.85	captan	C9H8Cl3NO2S	C	P	C	50	3
3.5		1.79	2.76	imazamethabenz methyl ester*	C16H20N2O3	C	-	-	-	-

Appendix I: PESTDATA Chemicals in Order by OV-225 Relative Retention Time (continued)

RRT/c OV-225	Detector Responses with OV-225 Column	RRT/c OV-101	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
3.5		1.26	1.92	Sulphenone	C12H9ClO2S	C		-	50	3
3.6	NI(WB)780	0.55	0.95	ethametsulfuron methyl ester*	C15H18N6O6S	-	NR	NR	6-15-50	1-2-3
3.6		1.54	-	2,4-D propylene glycol butyl ether ester*	C15H2OCl2O4	-	-	-	-	-
3.6		3.13	3.5	DDT, p,p'-	C14H9Cl5	C	C	C	6	1
3.7		2	2.6	bupirimate	C13H24N4SO3	C	-	-	-	-
3.7	NP6	1.66	2.41	fenamiphos	C13H22NO3PS	C	NR	NR	6-15-50	1-2-3
3.7	NI9	2.97	4.2	methoxychlor olefin	C16H14Cl2O2	C	C	C	6	2
3.7		1.42	-	fenac	C8H5Cl3O2	-	NR	NR	6-15-50	-
3.77	NI60	0.83	1.06	metribuzin, deaminated metabolite	C8H13N3OS	C	NR	NR	6-15-50	1-2-3
3.8		3.25	-	2,4,5-T isooctyl ester*	C16H21Cl3O3	-	-	-	-	-
3.8		4.9	4.5	bifenthrin	C23H22ClF3O2	V	C	-	6+15	2
3.8	NI3	2.03	2.71	nitrofen	C12H7Cl2NO3	C	C	C	15	2
3.8		2.41	2.87	TDE, p,p'-	C14H10Cl4	C	C	C	6	1
3.9		2.21	2.77	endosulfan II	C9H6Cl6O3S	C	C	C	15+50	2
3.93	FP2/NI8	2.56	3.36	ethion	C9H22O4P2S4	C	C	C	6	2
4	NI(WB)13	2.51	4.2	pyrithiobac-sodium methyl ester	C14H13ClN2O4	-	-	-	-	-
4		2	2.16	oxyfluorfen	C15H11ClF3NO4	C	C	C	15	2
4		1.76	2.08	imazalil	C14H14Cl2N2O	C	NR	NR	6-15-50	-
4.1	FP6	2.22	2.99	chlorthiophos oxygen analog	C11H15Cl2O4PS	C	NR	NR	6-15-50	1-2-3
4.1		1.6	3.17	isoprothiolane	C12H18O4S2	C	-	-	-	-
4.1		1.88	-	ethion oxygen analog	C9H22O5P2S3	C	-	-	-	-
4.2		2.17	3.06	carbophenothion oxygen analog	C11H16ClO3PS2	C	NR	NR	6-15-50	1-2-3
4.2		2.94	3.7	carbophenothion	C11H16ClO2PS3	C	C	P	6	2
4.2		2.19	2.38	binapacryl	C15H18N2O6	C	P	P	15	-
4.3	NI160	2.4	3	imazethapyr ammonium salt methyl ester	C16H21N3O3	-	-	-	-	-
4.3		1.15	1.54	CGA 91305	C10H8Cl2N3O	V	NR	NR	6-15-50	1-2-3
4.3	NI12	1.5	2.67	TCMTB	C9H6N2S3	C	P	P	15	-
4.5		32	-	hydramethylnon*	C25H24F6N4	-	-	-	-	-
4.5	NI23	3.3	5	methoxychlor, o, p'-	C16H15Cl3O2	-	C	-	6	-
4.5		6.4	6.1	bis(2-ethylhexyl) phthalate	C24H38O4	-	C	C	15+50	-

Appendix I: PESTDATA Chemicals in Order by OV-225 Relative Retention Time (continued)

RRT/c OV-225	Detector Responses with OV-225 Column	RRT/c OV-101	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
4.7	NI60	1.13	1.38	RPA202248	C15H12SNO4F3	NR	NR	NR	6-15-50	1-2-3
4.7	NI40	1.11	1.33	isoxaflutole (prop)	C15H12SNO4F3	NR	V #	S #	50	3
4.7		2.85	-	chlornitrofen	C12H6Cl3NO3	C	C	C	6+15	2
4.8		5.4	6.5	phenothrin*	C23H26O3	-	-	-	-	-
4.8		-	-	Dilan*	C15.5H14Cl2NO2	-	P	P	15	-
4.8	NI1300	3.8	4.3	propargite	C19H26O4S	C	C	-	15	2
4.8	NI(WB)12	0.8	1.36	bromacil	C9H13BrN2O2	C	NR	NR	6-15-50	1-2-3
4.9	NI12	3.57	4.7	diclofop-methyl	C16H14Cl2O4	C	C	C	15	2
4.9		0.89	1.48	cyanazine	C9H13ClN6	C	NR	-	6-15-50	-
5	NI(WB)6	2.14	2.4	PPG-947, methylated*	C18H13ClF3NO7	-	-	-	-	-
5	NI(WB)4	2.15	2.4	PPG-847, methylated	C15H9ClF3NO3	-	-	-	-	-
5.1		4.1	-	2,4-D BEP ester*	C17H24Cl2O4	-	-	-	-	-
5.1		3.06	4.5	butyl benzyl phthalate	C19H20O4	-	C	P	15+50	-
5.3		-	-	Dilan*	C15.5H14Cl2NO2	-	P	P	15	-
5.5		5.5	9.1	phosalone	C12H15ClNO4PS2	C	C	C	50	2+3
5.6		3.21	4	propiconazole*	C15H17Cl2N3O2	C	NR	NR	6-15-50	1-2-3
5.8		2.33	-	Dilan*	C15.5H14Cl2NO2	-	P	P	15	-
5.8	FP60	1.15	1.75	demeton-S sulfone	C8H19O5PS2	C	-	-	-	-
5.8	NI(WB)5	3.26	4.67	clodinafop-propargyl	C17H13ClFNO4	V	V	-	50	3
6.3		2.87	5.3	edifenphos	C14H15O2PS2	C	-	-	-	-
6.5	NI5	1.53	2.41	CGA 94689A	C15H21NO5	V	NR	NR	6-15-50	1-2-3
6.5		4.4	-	bromopropylate	C17H16Br2O3	C	C #	C #	15+50	1-2-3
6.6	FS175/NI400	1	1.46	2-hydroxy-2,3-dihydro-3,3-methyl-5-benzofuranyl methyl sulfonate	C11H14O5S	-	-	-	-	-
6.6	NI8	1.54	2.45	CGA 94689B	C15H21NO5	S	NR	NR	6-15-50	1-2-3
6.6	NI(WB)5	4.8	6.3	cloquintocet-mexyl	C18H22ClNO3	V	NR	-	6-15-50	1-2-3
6.7		1.5	2.39	disulfoton sulfone	C8H19O4PS3	C	NR	-	6-15-50	-
6.9		4.3	4.4	dinocap*	C18H24N2O6	C	P	P	15	2
7	NI10	4.8	5.7	fenpropathrin	C22H23NO3	-	V #	V	15	2
7.2		1.9	2.6	myclobutanil	C15H17ClN4	C	NR	NR	6-15-50	1-2-3
7.2		4.7	7.2	methoxychlor, p, p'-	C16H15Cl3O2	C	C	C	6	2
7.3		3.36	4.8	nuarimol	C17H12ClFN2O	C	NR	C #	50	1-2-3

Appendix I: PESTDATA Chemicals in Order by OV-225 Relative Retention Time (continued)

RRT/c OV-225	Detector Responses with OV-225 Column	RRT/c OV-101	RRT/c OV-17		Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
7.5		2.81	-		Dilan*	C15.5H14Cl2NO2	-	P	P	15	-
7.5	NI10	2.81	3.9		Prolan	C15H13Cl2NO2	P	S	S	15	2
7.5	NI6	3.06	4.4		Bulan	C16H15Cl2NO2	C	P	P	15	2
7.6		4.2	6.5		leptophos oxygen analog	C13H10BrCl2O3P	C	-	-	-	-
7.7		7	-		2,4,5-T BEP ester*	C17H23Cl3O3	-	-	-	-	-
7.7		4.8	4.8		dinocap*	C18H24N2O6	C	P	P	15	2
7.7		5.8	8.5	1	leptophos	C13H10BrCl2O2PS	C	C	C	6	2
7.9		1.5	-		procyazine	C10H13ClN6	C	-	-	-	-
8	NI6	1.25	1.09		MB45950	C12H4SN4F6Cl2	S	P	V	15+50	2+3
8.2		3.39	-		Dilan*	C15.5H14Cl2NO2	-	P	P	15	-
8.2		3.1	4.6		cyanofenphos	C15H14NO2PS	C	-	-	-	-
8.3		2.83	4		endosulfan sulfate	C9H6Cl6O4S	C	C	C	50	2
8.4	NI6	4.3	6		benzoylprop-ethyl	C18H17Cl2NO3	P	NR	NR	6-15-50	1-2-3
8.5		4.5	7.2		tetramethrin*	C19H25NO4	C	NR	NR	6-15-50	1-2-3
8.6	NI7	4.7	7.2		tetrasul sulfoxide	C12H6Cl4OS	-	-	-	-	-
8.7	NI10	1.35	1.16		fipronil	C12H4Cl2F6N4OS	S	S	V	50	3
8.9	NI500	12	6.1		CGA 205374	C16H11N3O2Cl2	-	NR	NR	6-15-50	1-2-3
8.9		1.3	-		chlorsulfuron	C12H12ClN5O4S	-	NR	NR	6-15-50	-
9.5		5.1	5.6		dinocap*	C18H24N2O6	C	P	P	15	2
9.7		4.8	6.8		piperophos	C14H28NO3PS2	C	-	-	-	-
10.3	FP15	4.7	6.9		chlorthiophos sulfoxide	C11H15Cl2O4PS2	C	NR	NR	6-15-50	1-2-3
10.3		3.6	-		endrin ketone	C12H8Cl6O	-	C	C	50	2
10.6		4.5	6.9		EPN	C14H14NO4PS	C	C	C	15	2
10.8	NI20	7.9	12.6		HOE-030291	C17H16Cl2O5	-	-	-	-	-
10.9		11.5	15		phenothrin*	C23H26O3	-	-	-	-	-
11.1		9.4	13.8		permethrin, cis-	C21H20Cl2O3	C	V #	C	6+15	2
11.3		8.1	10.5		fenoxaprop ethyl ester	C18H16NO5Cl	S	V	V	50	3
11.5	NI60	2.96	4.1		CL 202,347	C13H19N3O5	-	-	-	-	-
12.8	NI40	10.4	8.9		acrinathrin	C26H21F6NO5	V	V	V #	15	2
13		10.2	-		2,4-D BEP ester*	C17H24Cl2O4	-	-	-	-	-
13		10.2	15		permethrin, trans-	C21H20Cl2O3	C	V #	C	6+15	2
13	NI3700	2.67	8		pyrazon	C10H8ClN3O	C	NR	NR	6-15-50	1-2-3

Appendix I: PESTDATA Chemicals in Order by OV-225 Relative Retention Time (continued)

RRT/c OV-225	Detector Responses with OV-225 Column	RRT/c OV-101	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
13	NI1200	13	16	hexachlorophene	C13H6Cl6O2	-	NR	NR	6-15-50	-
14	NI4500	2.5	5	oxadixyl	C14H18N2O4	C	NR	NR	6-15-50	1-2-3
14		4.2	8.7	pyridaphenthion	C14H17O4N2SP	C	-	-	-	-
14		2.65	5	famphur	C10H16NO5PS2	C	NR	-	6-15-50	-
14.8	NI600	3.1	7.1	phosmet oxygen analog*	C11H12NO5PS	-	NR	NR	6-15-50	-
14.9		5	8.8	bifenox	C12H9Cl2NO5	C	C	P	15+50	2+3
14.9	FP50	4	8.4	phosmet	C11H12O4NPS2	C	NR	-	6-15-50	3
15		2.31	3.93	desisopropyl iprodione	C10H6Cl2N3O3	P		-	50	1-2-3
15	NP1000	4	4.2	KWG 1342	C14H18ClN3O3	-	-	-	-	-
15.5		4.4	8.5	photodieldrin	C12H8Cl6O	-	C	C	15+50	2
18	NI75	4.2	6.3	iprodione*	C13H13Cl2N3O3	C	S #	NR	50	1-2-3
18.1		-	-	carbofuran-7-phenol-DNP ether	C16H14N2O6	-	-	-	-	-
18.6	NI44	2.62	5.4	ofurace	C14H16NO3Cl	C	-	-	-	-
18.8	FP39	5.3	9.1	chlorthiophos sulfone	C11H15Cl2O5PS2	C	C	-	50	3
19.7		31	38	deltamethrin, trans-*	C22H19Br2NO3	-	P #	NR	15	2
19.9		29	38	deltamethrin*	C22H19Br2NO3	C	S #	P	15	2
24		16.3	-	fluridone	C19H14F3NO	-	NR	NR	6-15-50	-
24	NI21	3.8	6.3	nitralin	C13H19N3O6S	C	P	P	50	3
29		-	-	cypermethrin*	C22H19Cl2NO3	C	C	C	15	2
31	NI30	2.06	1.98	MB46136	C12H4SO2N4F6Cl2	S	S	V	50	2+3
33		14.1	23	cypermethrin*	C22H19Cl2NO3	C	C	C	15	2
36		15.1	25	cypermethrin*	C22H19Cl2NO3	C	C	C	15	2
36.9		14.7	21.4	flucythrinate*	C26H23F2NO4	C	C	-	15	2+3
37.1		3.6	7.5	myclobutanil alcohol metabolite	C15H17ClN40	S	NR	NR	6-15-50	1-2-3
40	NI100	9	18	coumaphos	C14H16ClO5PS	C	NR	C #	6-15-50	3
42		16.1	24	flucythrinate*	C26H23F2NO4	C	C	-	15	2+3
44		20.3	35	fenvalerate*	C25H22ClNO3	C	C	C	15	2
45	NI150	8	16	coumaphos oxygen analog	C14H16ClO6P	C	NR	NR	6-15-50	1-2-3
51		22.5	40	fenvalerate*	C25H22ClNO3	C	C	C	15	2
53		44	-	hydramethylnon*	C25H24F6N4	-	-	-	-	-
53		4.7	11.3	dithianon	C14H4O2N2S2	NR	-	-	-	-
56		-	35	fluvalinate*	C26H22ClF3N2O3	C	C	-	15	2

Appendix I: PESTDATA Chemicals in Order by OV-225 Relative Retention Time (continued)

RRT/c OV-225	Detector Responses with OV-225 Column	RRT/c OV-101	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
57	NI300	23	43	PB-7, methylated	C20H25ClN2O3S	-	-	-	-	-
59		25	38	fluvalinate*	C26H22ClF3N2O3	C	C	-	15	2
64		27	44	tralomethrin	C22H19Br4NO3	C	V	S	15	2
87	NI500	25	46	PB-9	C19H25ClN2O2S	V	NR	NR	6-15-50	1-2-3

Appendix I: PESTDATA Chemicals in Order by OV-17 Relative Retention Time

RRT/c OV-17	Detector Responses with OV-17 Column	RRT/c OV-101	RRT/c OV-225	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
0.02	NI4	0.03	-	dichlorobenzene, p-	C6H4Cl2	-	C	C	6	1
0.02	NI0.1	-	-	hexachloroethane	C2Cl6	-	-	-	-	-
0.03	NI0.2	0.04	-	dibromochloropropane	C3H5Br2Cl	-	-	-	-	-
0.04	NI10	0.06	0.08	CGA 171683	C6H5F4N3O2	C		-	15+50	3
0.04	NI0.1	-	-	hexachlorobutadiene	C4Cl6	-	V #	P	6	1
0.06	NI0.8	0.12	-	hexachlorocyclopentadiene	C5Cl6	-	-	-	-	-
0.07	NI0.1	-	-	1,2,3,5-tetrachlorobenzene	C6H2Cl4	-	P #	-	6	1
0.07	NI0.2	-	-	1,2,4,5-tetrachlorobenzene	C6H2Cl4	-	-	-	-	-
0.07	NP(WB)1.5	-	-	4-chlorobenzeneamine	C6H6ClN	S	NR	NR	6-15-50	1-2-3
0.08	FP2.5	0.07	0.08	dichlorvos	C4H7Cl2O4P	C	NR	NR	6-15-50	1-2-3
0.09	NP(WB)5	-	0.11	teflubenzuron*	C14H6Cl2F4N2O2	-	NR	NR	6-15-50	1-2-3
0.09	FP1/FP(WB)0.6	0.07	0.25	methamidophos	C2H8NO2PS	V	-	-	-	-
0.09	FP11	0.15	-	vernolate	C10H21NOS	-	P	-	15	-
0.09	NI0.2	-	-	1,2,3,4-tetrachlorobenzene	C6H2Cl4	-	-	-	-	-
0.1	NI(WB)1.4/NP23	0.13	0.15	chlorimuron ethyl ester	C15H15ClN4O6S	P	NR	-	-	-
0.1	HX1.5	0.19	0.08	2-methoxy-3,5,6-trichloropyridine	C6H4Cl3NO	C	P #	C	6+15	1+2
0.1	NI5	0.17	0.14	N-(3,4-dichlorophenyl)-N'-methylurea	C8H8Cl2N2O	-	NR	NR	6-15-50	-
0.1	NI4	0.22	0.14	3,4-dichlorophenylurea	C7H6Cl2N2O	-	NR	NR	6-15-50	-
0.1	NI0.6	0.11	-	dichlobenil	C7H3Cl2N	C	P	C	15	2
0.1	NP6	0.17	-	pebulate	C10H21NOS	C	P	-	15	-
0.11	NP20	0.17	-	CGA 236431	C8H7F3N2O2	-	-	-	-	-
0.11	FP0.4	-	-	chlormephos	C5H12ClO2PS2	C	-	-	-	-
0.11	FS30	0.13	0.23	carboxin sulfoxide	C12H13NO3S	-	NR	NR	6-15-50	1-2-3
0.11	NI12	0.11	0.09	diuron	C9H10Cl2N2O	C	NR	NR	6-15-50	1-2-3
0.12	NI0.2	-	-	hexachloronorbornadiene	C7H2Cl6	-	-	-	-	-
0.12	NP16	0.13	-	propham	C10H13NO2	C	P	P	15	-
0.13	NP8	0.26	-	CGA 236432	C9H9F3N2O2	-	-	-	-	-
0.13	NI1/NP2	0.17	0.22	3-methyl-4-nitrophenol methyl ether	C8H9O3N	-	-	-	-	-
0.13	FP2	0.16	-	mevinphos, (E)-	C7H13O6P	C	NR	NR	6-15-50	-

* Multipeak chemical.

Recovery may vary with choice of Florisil elution system; see Tables 303-a, 304-a.

Appendix I: PESTDATA Chemicals in Order by OV-17 Relative Retention Time (continued)

RRT/c OV-17	Detector Responses with OV-17 Column	RRT/c OV-101	RRT/c OV-225	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
0.13	FP4	0.16	-	trichlorfon	C4H8Cl3O4P	C	NR	NR	6-15-50	1-2-3
0.14	NP(WB)5	0.14	0.23	teflubenzuron*	C14H6Cl2F4N2O2	-	NR	NR	6-15-50	1-2-3
0.14	NP50	0.22	-	CGA 72903	C7H6F3N	-	-	-	-	-
0.14	NI1	0.19	0.22	N, N-diallyl dichloroacetamide	C8H11Cl2NO	C	S	S	15+50	2+3
0.14	NP2	-	-	fluometuron	C10H11F3N2O	-	-	-	-	-
0.14	NP1	0.22	-	CGA 150829	C5H14N4O	V	-	-	-	-
0.14	HN(WB)0.3/HX(WB)2/NI8/ NI(WB)14/NP8/NP(WB)0.4	0.18	0.27	3,5-dichloroaniline	C6H5Cl2N	S	S	S	6+15	1+2
0.14		0.15	0.15	dimethyl phthalate	C10H10O4	-	P	-	6+15+50	-
0.15	FP2/FP(WB)0.6	0.13	-	mevinphos, (Z)-	C7H13O6P	C	NR	-	6-15-50	-
0.16	HN(WB)3/NI(WB)120/ NP(WB)7	0.25	0.5	cymoxanil	C7H10N4O3	V	NR	NR	6-15-50	1-2-3
0.16	NI0.3	0.24	0.13	pentachlorobenzene	C6HCl5	C	C	C	6	1
0.16	NI13/NP8	0.2	0.32	3,4-dichloroaniline	C6H5Cl2N	V	S	-	15	-
0.17	NI0.7	0.34	0.27	trifluralin	C13H16F3N3O4	C	C	C	6	2
0.18	FP(WB)2	-	-	metasystox thiono*	C6H15O3PS2	-	-	-	-	-
0.18	HX25	-	-	metoxuron	C10H13ClN2O2	V	NR	NR	6-15-50	1-2-3
0.18	NI0.3	0.23	0.21	methyl 2,3,6-trichlorobenzoate	C8H5Cl3O2	-	-	-	-	-
0.18	HX(WB)1	0.37	0.28	benfluralin	C13H16F3N3O4	C	C	C	6	2
0.19	HX6	0.34	0.27	ethalfluralin	C13H14F3N3O4	C	C	C	6	2
0.19	FP(WB)0.6	0.15	0.64	acephate	C4H10NO3PS	C	-	-	-	-
0.2	FP2	-	-	demeton-O*	C8H19O3PS2	C	NR	-	6-15	-
0.2	NI160/NP30	0.22	0.25	3-carboxy-5-ethoxy-1,2,4-thiadiazole	C3H2N2O3S	NR	-	-	-	-
0.2		-	-	4-chlorobiphenyl	C12H9Cl	-	-	-	-	-
0.2	HN(WB)1/HX(WB)35/ NP(WB)11	0.3	-	triflusulfuron methyl ester	C17H19F3N6O6S	V	NR	NR	6-15-50	1-2-3
0.2		-	-	epoxyhexachloronorbornene	C7H2Cl6O	-	-	-	-	-
0.2	HX(WB)0.6	0.2	0.18	nitrapyrin	C6H3Cl4N	C	C	V	6	2
0.21	FP20	0.26	-	tebuthiuron	C9H16N4OS	-	-	-	-	-
0.21	FP25	0.22	0.32	demeton-O oxygen analog	C8H19O4PS	-	-	-	-	-
0.21	HX(WB)0.8/NI0.4/NP0.5	0.18	0.12	etridiazole	C5H5Cl3N2OS	C	C	P	6	2
0.22	NI150	0.18	-	4-hydroxymethyl-3,5-dimethylphenyl methylcarbamate*	C11H15NO3	-	NR	NR	15-50	1-2-3

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Appendix I: PESTDATA Chemicals in Order by OV-17 Relative Retention Time (continued)

RRT/c OV-17	Detector Responses with OV-17 Column	RRT/c OV-101	RRT/c OV-225	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
0.22	NI0.2	-	-	octachlorocyclopentane	C5Cl8	-	-	-	-	-
0.22	HX(WB)0.3	0.24	0.15	2,3,5,6-tetrachloroanisole	C7H4Cl4O	-	C	-	6	1
0.23	NI0.4	0.26	0.51	RPA 203328, methylated	C10H9F3O4S	-	-	-	-	-
0.23	NI0.2	-	-	heptachloronorbornene	C7H3Cl7	-	-	-	-	-
0.23		0.3	-	tributyl phosphate	C12H27O4P	-	R	-	50	-
0.24	FP150	0.5	0.53	phosmet oxygen analog*	C11H12NO5PS	-	NR	NR	6-15-50	-
0.24	FP0.5/HX0.3/NI0.3	0.33	0.23	chlorethoxyfos	C6H11Cl4O3PS	V	C	-	6	1
0.24	NI0.3	0.29	0.26	tecnazene	C6HCl4NO2	C	C	C	6	1
0.24	FP(WB)70/NI10000/ NP(WB)70	0.21	0.27	TEPP	C8H20O7P2	C	-	-	-	-
0.25		0.3	0.38	2,4-D methyl ester	C9H8Cl2O3	-	-	-	-	-
0.25		0.34	-	2,4,5-trichloro-alpha-methylbenzene methanol	C8H7OCl3	R	R	-	15	-
0.25	FP1	0.33	0.31	ethoprop	C8H19O2PS2	C	P #	S #	50	1-2-3
0.25		0.29	-	diphenylamine	C12H11N	C	S	-	6+15	-
0.25	NI80	0.32	0.43	chlorpropham	C10H12ClNO2	C	C	C	15	2
0.26	NI5/NP20	0.38	0.63	3-methyl-4-nitrophenol	C7H7O3N	V	NR	NR	6-15-50	1-2-3
0.26	NI5/NI(WB)5	0.34	0.37	propachlor	C11H14ClNO	C	NR	NR	6-15-50	1-2-3
0.26	FP0.5	0.26	-	thionazin	C8H13N2O3PS	C	P	NR	15+50	-
0.27		0.2	-	1,2,4-triazole	C2H3N3	V	NR	NR	6-15-50	1-2-3
0.28	FP3	-	-	demeton-O sulfone*	C8H19O5PS2	C	-	-	-	-
0.28	HN(WB)0.4/NI(WB)3/ NP(WB)3	0.25	0.92	oxamyl oxime metabolite	C5H10N2O2S	C	NR	NR	6-15-50	1-2-3
0.29	FP(WB)0.4/NI(WB)12/ NP(WB)0.5	0.37	0.27	cadusafos	C10H23O2PS2	C	NR	NR	6-15-50	1-2-3
0.29	FP0.5	0.34	-	sulfotep	C8H20O5P2S2	C	C	P	6+15	2
0.29	NP3	0.28	-	G-27550	C8H12N2O	C	-	-	-	-
0.29	FP1	0.3	0.37	phorate oxygen analog	C7H17O3PS2	C	NR	NR	6-15-50	1-2-3
0.3	NI0.8/NP9	0.53	0.46	profluralin	C14H16F3N3O4	V	V	-	6	-
0.31	NI150	0.27	-	4-hydroxymethyl-3,5-dimethylphenyl methylcarbamate*	C11H15NO3	-	NR	NR	15-50	1-2-3
0.31	FP(WB)4	0.46	0.49	oxydemeton-methyl	C6H15O4PS2	C	-	-	-	-
0.32	FP(WB)2	0.31	0.49	metasystox thiono*	C6H15O3PS2	-	-	-	-	-

Appendix I: PESTDATA Chemicals in Order by OV-17 Relative Retention Time (continued)

RRT/c OV-17	Detector Responses with OV-17 Column	RRT/c OV-101	RRT/c OV-225	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
0.32	NI0.4	0.56	0.4	chlordene	C10H6Cl6	-	C	C	6	1
0.32	FP(WB)0.8	0.28	0.49	metasystox thiol	C6H15O3PS2	C	-	-	-	-
0.32	FP20/NI8	0.34	-	naled	C4H7Br2Cl2O4P	C	NR	NR	6-15-50	1-2-3
0.32	FP0.5/NI14	0.37	0.38	phorate	C7H17O2PS3	C	V #	V #	6	1
0.33		0.42	0.74	2,4-D isopropyl ester*	C11H12Cl2O3	-	-	-	-	-
0.33		0.42	0.26	di-allate	C10H17ClNOS	C	C	-	6	-
0.33	HX0.3/NI0.3	0.45	0.25	hexachlorobenzene	C6Cl6	C	C	P	6	1
0.34	NI0.3	0.46	0.3	pentachlorophenyl methyl ether	C7H3Cl5O	C	C	C	6	1
0.35	NI0.3	0.4	0.48	BHC, alpha-	C6H6Cl6	C	C	C	6	1
0.36	FP2	0.28	-	demeton-O*	C8H19O3PS2	C	NR	-	6-15	-
0.36		0.32	0.44	3, 5, 6-trichloro-2-pyridinol methyl ester	C6H4Cl3NO	-	-	-	-	-
0.36	FP0.7	0.34	-	dioxabenzofos	C8H9O3PS	C	P	-	15	-
0.36	NI1	0.38	0.44	sulfallate	C8H14ClNS2	C	C	C	6+15	2
0.37	HX3/NI0.5	0.53	0.76	fluchloralin	C12H13ClF3N3O4	C	C	-	6	2
0.38	NP4	0.35	0.6	2,3,5-trimethacarb	C11H15NO2	C	S #	NR	50	1-2-3
0.38	FP4/FP(WB)1	0.39	0.53	fonofos oxygen analog	C10H15O2PS	V	NR	NR	6-15-50	1-2-3
0.39		0.36	0.36	triclopyr methyl ester	C8H6Cl3NO3	-	-	-	-	-
0.39	NI1/NP1.8	0.46	0.6	furilazole	C11H13Cl2NO3	C	S	-	50	3
0.39	FP1/NI2500	0.42	-	terbufos oxygen analog	C9H21O3PS2	C	-	NR	6-15-50	1-2-3
0.39	FP5/FP(WB)1.1	0.25	1.11	omethoate	C5H12NO4PS	C	NR	NR	6-15-50	1-2-3
0.4	HN(WB)30	0.35	2.85	ethametsulfuron methyl ester*	C15H18N6O6S	-	NR	NR	6-15-50	1-2-3
0.4	NI1/NP85	0.51	0.84	pronamide	C12H11Cl2NO	C	P	-	15+50	-
0.4	FP0.4/NP(WB)1	0.41	0.51	thiometon	C6H15O2PS3	C	NR	NR	6-15-50	-
0.41	NP24	0.35	0.7	methabenzthiazuron	C10H11N3OS	C	NR	NR	6-15-50	1-2-3
0.41	NP40	0.32	-	phenmedipham	C16H16N2O4	-	-	-	-	-
0.41	FP0.5/FP(WB)1.6/NI20	0.5	0.44	terbufos	C9H21O2PS3	C	P	S	6	-
0.41	NI43	0.53	0.65	propazine	C9H16ClN5	C	S	NR	15+50	3
0.41	FP0.8/FP(WB)0.8	0.41	0.56	demeton-S	C8H19O3PS2	C	NR	-	6-15-50	-
0.42	FP0.5	0.48	-	propetamphos	C10H20NO4PS	C	C #	-	15+50	2+3
0.42	NP9	0.42	-	melamine	C3H6N6	NR	-	-	-	-
0.43	FP1/FP(WB)0.8	0.31	0.96	dicrotophos	C8H16NO5P	C	NR	-	6-15-50	-

Appendix I: PESTDATA Chemicals in Order by OV-17 Relative Retention Time (continued)

RRT/c OV-17	Detector Responses with OV-17 Column	RRT/c OV-101	RRT/c OV-225	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
0.44	NI150	0.5	0.79	metribuzin, deaminated diketo metabolite*	C7H11N3O2	NR	NR	NR	6-15-50	1-2-3
0.44	FP150	2	0.93	phosmet oxygen analog*	C11H12NO5PS	-	NR	NR	6-15-50	-
0.44	NI1	0.52	0.93	dinitramine	C11H13F3N4O4	C	-	P	15	-
0.44	NI20	0.43	0.74	atrazine	C8H14ClN5	C	S #	NR	50	1-2-3
0.44	FP0.7	0.52	0.56	fonofos	C10H15OPS2	C	C	C	6	2+3
0.44	FP0.7/FP(WB)0.9/NI4/ NP0.25	0.51	0.4	diazinon	C12H21N2O3PS	C	C	C	15	3
0.45	NP430	0.9	-	formetanate hydrochloride	C11H16ClN3O2	-	-	-	-	-
0.45	NI3	0.5	0.59	pentachlorobenzonitrile	C7Cl5N	C	C	P	15	2
0.45	NI1000/NP218	0.44	0.29	desmedipham	C16H16N2O4	-	-	-	-	-
0.45		0.6	-	tri-allate	C10H16Cl3NOS	C	C	C	6	2
0.45	HN(WB)0.4	0.42	0.75	chlorbufam	C11H10ClNO2	C		-	15	2+3
0.45	NI0.4	0.42	0.96	dicloran	C6H4Cl2N2O2	C	S	P	15+50	2+3
0.46	HX2/NP11	0.45	0.59	clomazone	C12H14ClNO2	C		-	50	3
0.46	FP1	0.54	0.6	disulfoton	C8H19O2PS3	C	P #	NR	6	1-2-3
0.46	NI0.5	0.51	0.46	quintozene	C6Cl5NO2	C	C	C	6	1
0.47	NI(WB)1	0.56	0.49	methyl 3,5-dibromo-4-methoxy= benzoate	C9H8Br2O3	-	-	-	-	-
0.47		0.49	0.63	2,4,5-T methyl ester	C9H7Cl3O3	-	-	-	-	-
0.47	NP100	0.4	-	CGA 37734	C10H13NO2	C	NR	NR	6-15-50	1-2-3
0.47	NI30/NP0.6	0.5	0.53	diazinon oxygen analog	C12H21N2O4P	C	NR	NR	6-15-50	1-2-3
0.47	NI0.4	0.48	0.69	lindane	C6H6Cl6	C	C	C	6	1
0.48	NI1.1/NP1.2	0.5	0.69	4-(dichloroacetyl)-1-oxa-4-azapiro= [4.5]decane	C10H15Cl2NO2	C	P	-	50	3
0.48	HX(WB)0.3	0.49	0.35	1,2,4,5-tetrachloro-3-(methylthio)= benzene	C7H4Cl4S	R	C	-	6	1
0.48	NI250	0.47	0.71	terbuthylazine	C9H16N5Cl	C	P	-	15+50	-
0.48	HX18	0.48	0.91	monolinuron	C9H11ClN2O2	C	-	-	-	-
0.49	HN(WB)5	0.42	0.97	PPG-947, methylated*	C18H13ClF3NO7	-	-	-	-	-
0.49	NP3	0.46	-	CGA 51702	C9H9F3N2O	-	-	-	-	-
0.49		0.62	0.62	2,4-D isobutyl ester	C12H14Cl2O3	-	-	-	-	-
0.5		0.5	2.02	tris(chloropropyl) phosphate	C9H18Cl3O4P	C	NR	NR	6-15-50	1-2-3

Appendix I: PESTDATA Chemicals in Order by OV-17 Relative Retention Time (continued)

RRT/c OV-17	Detector Responses with OV-17 Column	RRT/c OV-101	RRT/c OV-225	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
0.5	FP2/FP(WB)0.8	0.31	1.6	monocrotophos	C7H14NO5P	C	NR	NR	6-15-50	1-2-3
0.5	NP10	0.45	0.78	3,4,5-trimethacarb	C11H15NO2	C	NR	NR	50	1-2-3
0.5	NI56/NP(WB)1.5	0.41	0.83	simazine	C7H12ClN5	C	NR	NR	50	1-2-3
0.5	FP7/FP(WB)13	0.47	-	dioxathion	C12H26O6P2S4	V	NR	-	6-15-50	2
0.51		-	-	4,4'-dichlorobiphenyl	C12H8Cl2	-	-	-	-	-
0.51	NI50	0.58	0.59	etrimfos	C10H17N2O4PS	C	C	C	15	2+3
0.52	NI150	0.73	1.29	metribuzin, deaminated diketo metabolite*	C7H11N3O2	NR	NR	NR	6-15-50	1-2-3
0.52		0.39	1.3	2,6-dichlorobenzamide	C7H5NOCl2	C	NR	NR	6-15-50	1-2-3
0.52	FP1	0.58	-	schradan	C8H24N4O3P2	C	NR	-	6-15-50	-
0.53	NP(WB)25	0.47	0.42	terbumeton	C10H19N5O	C	-	-	-	-
0.53	HN(WB)0.1/HX12	0.3	0.8	desethyl simazine	C5H8ClN5	-	NR	NR	50	1-2-3
0.54	FP2	0.6	-	iprobenfos	C13H21O3PS	C	-	-	-	-
0.54	NI(WB)16	0.39	1.16	4-chlorobenzylmethyl sulfoxide	C8H9ClOS	-	NR	NR	6-15-50	1-2-3
0.55	NI6/NP20	0.56	1.41	metribuzin, diketo metabolite	C7H12N4O2	NR	NR	NR	6-15-50	1-2-3
0.56	HX(WB)0.5	0.56	0.63	2,3,5,6-tetrachloronitroanisole	C7H3Cl4NO3	-	C	-	6	1+2
0.56	NI1	0.43	1.62	BHC, beta-	C6H6Cl6	C	C	C	6	1
0.56	FP0.8/HX(WB)2	0.67	0.64	dichlofenthion	C10H13Cl2O3PS	C	C	V	6	2
0.56		0.65	0.61	diisobutyl phthalate	C16H2204	-	P	-	15+50	-
0.57	FP2	0.53	-	phosphamidon*	C10H19ClNO5P	C	NR	NR	6-15-50	1-2-3
0.58	NP24	0.7	-	CP 51214	C14H21NO3	C	NR	NR	6-15-50	1-2-3
0.59	FP(WB)0.7/NP(WB)1	0.47	-	cyanophos	C9H10O3NSP	C	-	-	-	-
0.6	HN(WB)0.4/HX(WB)4/ NI(WB)36/NP(WB)2	0.43	1.34	6-chloro-2,3-dihydro-3,3,7-methyl-5H-oxazolo(3,2-a)pyrimidin-5-one	C9H13ClN2O2	-	NR	NR	6-15-50	1-2-3
0.6	NI0.4	0.83	0.52	heptachlor	C10H5Cl7	C	C	C	6	1
0.61	HN(WB)0.1/HX25	0.2	0.86	desdiethyl simazine	C3H4ClN5	-	NR	NR	6-15-50	1-2-3
0.62	NP50	-	-	CGA 27092	C8H7F3N2O	-	-	-	-	-
0.62	NP14	1.09	-	fenpropimorph	C20H33NO	C		-	50	1-2-3
0.62		0.54	1.47	fenfuram	C12H11NO2	C	-	-	-	-
0.62	FP135	0.43	-	fenthion oxygen analog sulfoxide	C10H15O5PS	C	NR	NR	6-15-50	1-2-3
0.62	FP5/FP(WB)1	0.64	1.02	ronnel oxygen analog	C8H8Cl3O4P	C	NR	-	6-15-50	-
0.62	FP0.8/FP(WB)0.8/NI5	0.4	1.6	dimethoate	C5H12NO3PS2	C	NR	NR	6-15-50	1-2-3

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Appendix I: PESTDATA Chemicals in Order by OV-17 Relative Retention Time (continued)

RRT/c OV-17	Detector Responses with OV-17 Column	RRT/c OV-101	RRT/c OV-225	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
0.63		0.51	0.8	etrimfos oxygen analog	C10H17N2O5P	C	-	-	-	-
0.63	FP(WB)1/NI10/NP0.2	0.55	0.8	isazofos	C9H17ClN3O3PS	C	C #	-	50	2+3
0.64	NI120	0.44	2.49	prosulfuron*	C15H16F3N5O4S	-	NR	NR	6-15-50	1-2-3
0.64	HX1/NI2	0.69	1.15	vinclozolin	C12H9Cl2NO3	C	C	C	15	2
0.64		0.66	-	octhilinone	C11H19NOS	C	-	-	-	-
0.66	HN(WB)6/HX(WB)9/ NI(WB)10/NP(WB)19	0.74	1.2	vinclozolin metabolite B	C12H11Cl2NO4	C	P #	C	6+15	2
0.66	HX0	0.54	-	4-chloro-6-methoxyindole	C9H8NOCl	-	R	-	15	-
0.66	HX(WB)0.6	0.59	0.73	2,3,5,6-tetrachloroanisidine	C7H5Cl4NO	-	C	-	6	2
0.66	NP(WB)50	0.94	0.97	prodiamine	C13H17F3N4O4	C	-	-	-	-
0.66	NI(WB)0.8	0.41	1.91	4-chlorobenzylmethyl sulfone	C8H9ClO2S	-	NR	NR	6-15-50	1-2-3
0.66	FP10	0.55	1.71	parathion-methyl oxygen analog	C8H10NO6P	-	NR	NR	6-15-50	1-2-3
0.66	NI0.6/NP10	0.67	0.79	pentachloroaniline	C6H2Cl5N	C	C	C	6	1
0.66	NI4500/NP64	0.5	2.33	ethylenethiourea	C3H6N2S	S	NR	NR	6-15-50	1-2-3
0.67	NP6	0.73	-	CP 108064, methylated	C15H21NO4	-	-	-	-	-
0.67	NI0.6	0.82	0.64	chlordene, alpha-	C10H6Cl6	-	-	-	-	-
0.67	NI5	0.75	0.88	acetochlor	C14H20NO2Cl	C	C #	P	50	3
0.67	NI0.5	0.5	1.71	BHC, delta-	C6H6Cl6	C	C	C	6+15	1
0.68	NP2	0.58	-	cyromazine	C6H10N6	S	-	-	-	-
0.68	NI0.6/NP2	0.75	0.8	CGA 14128	C12H21N2O4PS	C		-	50	1-2-3
0.68	NP(WB)65	1.1	-	nitrothal-isopropyl	C14H17O6N	C	-	-	-	-
0.69	HX10	0.67	1.44	metobromuron	C9H11BrN2O2	C	NR	NR	6-15-50	1-2-3
0.7	NI1/NP7	0.64	1.06	benoxacor	C11H11Cl2NO2	C	P	C	15+50	2+3
0.7	NI12/NP15	0.6	1.64	ethoxyquin	C14H19N0	C	NR	NR	6-15-50	-
0.71	NI8/NP10	0.45	2.25	methidathion sulfoxide	C5H8N2O4S2	-	NR	NR	6-15-50	1-2-3
0.71	NI10	0.71	1.11	dimethachlor	C13H18ClNO2	C	-	-	-	-
0.71	FS(WB)80/HN(WB)0.4/ HX500/NI300	0.4	-	dazomet	C5H10N2S2	S	NR	-	6-15-50	1-2-3
0.72	NP1000	-	-	1-methyl cyromazine	C7H13N6	-	-	-	-	-
0.72	NI6	0.8	1	alachlor	C14H2OClNO2	C	C	C #	50	3
0.72	HN(WB)0.6/HX(WB)5/ NI(WB)2/NP(WB)8	0.54	2.1	terbacil	C9H13ClN2O2	C	NR	NR	6-15	2+3

Appendix I: PESTDATA Chemicals in Order by OV-17 Relative Retention Time (continued)

RRT/c OV-17	Detector Responses with OV-17 Column	RRT/c OV-101	RRT/c OV-225	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
0.73	FP(WB)2	0.61	-	pirimicarb	C11H18N4O2	C	-	-	-	-
0.74		0.64	1.22	cyprazine	C9H14ClN5	C	-	-	-	-
0.74	FP20	0.77	-	prometryn	C10H19N5S	C	P #	P #	50	1-2-3
0.74	HX1/NI2	0.55	1.44	chlorothalonil	C8Cl4N2	S	C #	C #	6-15-50	2+3
0.75	HX0.8/HX(WB)0.4	0.81	0.85	tridiphane	C10H7Cl5O	C	C	-	6	1+2
0.76	FP2	0.67	-	phosphamidon*	C10H19ClNO5P	C	NR	NR	6-15-50	1-2-3
0.76	NI1	1.05	0.58	aldrin	C12H8Cl6	C	C	C	6	1
0.76	FP1/NI1	0.81	0.86	ronnel	C8H8Cl3O3PS	C	C	C	6	2
0.78	NP133	0.6	1.4	ethiofencarb	C10H15NO2S	C	NR	NR	6-15-50	-
0.78	NI4	0.66	2.82	propanil	C9H9Cl2NO	C	NR	NR	6-15	3
0.79	HN(WB)1/HX(WB)2/ NI(WB)1/NP(WB)7	0.69	2.01	vinclozolin metabolite S	C10H7Cl2NO3	V	P	V #	15	2
0.79	FP(WB)1.5	0.72	0.86	chlorpyrifos-methyl	C7H7Cl3NO3PS	C	C	-	6	2
0.79		0.75	1.55	prothoate	C9H20NO3PS2	C	-	-	-	-
0.8	HN(WB)0.2/NP(WB)13	0.7	-	IN-B2838	C10H15N3O3	P	NR	NR	6-15-50	1-2-3
0.81	FS1.5	0.41	2.71	dimethipin	C6H10O4S2	C	NR	NR	6-15-50	1-2-3
0.82	NI200/NP35	0.56	2.29	methidathion sulfone	C5H8N2O3S2	-	NR	NR	6-15-50	1-2-3
0.82		-	-	butylisodecyl phthalate	C22H34O4	-	-	-	-	-
0.82	NP7	0.44	-	isocarbamid	C8H15N3O2	C	-	-	-	-
0.83	FP10	0.72	-	fenitrothion oxygen analog	C9H12NO6P	C	-	-	-	-
0.84		0.88	0.92	dibutyl phthalate	C16H22O4	-	C	C	15+50	-
0.86		-	-	2,4,5-T butyl esters*	C12H13Cl3O3	-	-	-	-	-
0.86	HX3	0.96	1.45	nitrofluorfen	C13H7ClF3NO3	C	C	C	15	2
0.86	FP4/NI15	0.8	-	parathion oxygen analog	C10H14NO6P	C	NR	NR	6-15-50	1-2-3
0.87	NI3	0.94	0.69	pentachlorophenyl methyl sulfide	C7H3Cl5S	C	C	C	6	1
0.87	FP5	0.68	1.55	malathion oxygen analog	C10H19O7PS	C	NR	NR	6-15-50	1-2-3
0.87	FP2/FP(WB)1/NI3	0.71	1.64	parathion-methyl	C8H10NO5PS	C	C	C	15	2
0.88	NI1	0.98	0.89	chlordene, gamma-	C10H6Cl6	-	-	-	-	-
0.88	HN(WB)48/HX(WB)390/ NI(WB)27/NP(WB)1000	1.05	1.47	acifluorfen	C14H7ClF3NO3	-	NR	NR	6-15-50	1-2-3
0.89	NI1	0.98	0.84	chlordene, beta-	C10H6Cl6	-	-	-	-	-
0.89	NP(WB)2	0.73	-	cymiazole	C12H14N2S	-	-	-	-	-

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RRT/c OV-17	Detector Responses with OV-17 Column	RRT/c OV-101	RRT/c OV-225	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
0.89	NP(WB)10	-	-	isoproturon	C12H18N2O	S	-	-	-	-
0.9	HN(WB)3/HX(WB)23/ NI(WB)23/NP(WB)44	0.42	-	pyrazon metabolite B	C6H4ClN3O	-	NR	NR	6-15-50	1-2-3
0.9	NI100/NP300	0.6	2.65	CGA 120844	C8H9NSO3	-	NR	NR	6-15-50	1-2-3
0.9	NI1000/NP50	0.81	-	metalaxyl	C15H21NO4	C	NR	NR	6-15-50	1-2-3
0.9	HN(WB)15/NI(WB)80/ NP(WB)30	0.55	1.41	3-ketocarbofuran	C12H12NO4	S	NR	NR	6	1
0.91	HX2	4.1	1.08	dicofol, o,p'-*	C14H9Cl5O	C	V	S	6+15	2
0.91	NI0.4/NP11	0.57	1.47	metribuzin	C8H14N4OS	V	NR	NR	50	1-2-3
0.91	FP(WB)10	-	-	formothion	C6H12NO4PS2	C	NR	NR	6-15-50	1-2-3
0.92	FP2/FP(WB)1.2	0.87	-	pirimiphos-methyl	C11H20N3O3PS	C	C	C	15	3
0.92	NI2	0.82	1.07	dichlorobenzophenone, o,p'-	C13H8Cl2O	-	C	C	15	2
0.93	HN(WB)3/HX(WB)3/ NI(WB)4/NP(WB)7	0.89	3.02	vinclozolin metabolite E	C11H11Cl2NO2	C	S	NR	15+50	-
0.93	FS54/NI4.5	0.68	2.89	2,3-dihydro-3,3-methyl-2-oxo-5-benzofuranyl methyl sulfonate	C11H12O5S	-	-	-	-	-
0.93	HX7	1.03	1.21	metolachlor	C15H22ClNO2	C	S #	NR	50	1-2-3
0.93	NI6/NP15	1.15	1.22	butralin	C14H21N3O4	V	C	-	6+15+50	-
0.95	HN(WB)30	0.55	3.6	ethametsulfuron methyl ester*	C15H18N6O6S	-	NR	NR	6-15-50	1-2-3
0.95	NP5.5	0.71	-	fuberidazole	C11H8N2O	C	-	-	-	-
0.95		0.85	2.13	linuron	C9H10Cl2N2O2	V	V #	V	50	3
0.96	FP3	0.71	2.95	demeton-O sulfone*	C8H19O5PS2	C	-	-	-	-
0.96	NI1/NP5	0.99	1.91	KWG 1323	C14H16ClN3O3	C	NR	NR	6-15-50	1-2-3
0.96	NP16	0.97	-	difenoxuron	C16H18N2O3	-	-	-	-	-
0.97		1.13	-	trichloronat	C10H12Cl3O2PS	C	C	-	6	-
0.98	HX3	0.94	1	thiobencarb	C12H16ClNOS	C		V	15	2+3
0.99	NI1/NP9	0.77	1.62	Tycor	C9H16N4OS	C	S	S	50	3
1	HX5/HX5/HX(WB)3/ NI(WB)2/NP2/NP(WB)3	1.05	1.64	triadimefon	C14H16ClN3O2	C	S #	S #	50	1-2-3
1	FP2/NI2	1	1	chlorpyrifos	C9H11Cl3NO3PS	C	C	P	6	2
1	NI1	1.06	1.13	DCPA	C10H6Cl4O4	C	C	C	15	2
1.01	NI3/NP15	1.14	1.24	isopropalin	C15H23N3O4	C	C	-	6	-
1.01		0.9	1.71	dichlofluanid	C9H11Cl2FN2O2S2	C	C #	-	15+50	2+3

Appendix I: PESTDATA Chemicals in Order by OV-17 Relative Retention Time (continued)

RRT/c OV-17	Detector Responses with OV-17 Column	RRT/c OV-101	RRT/c OV-225	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
1.02	FS65/NI333	0.86	1.93	ethofumesate	C13H18O5S	C	-	-	-	-
1.02	FP6	0.66	-	phorate oxygen analog sulfone	C7H17O5PS2	C	NR	NR	6-15-50	1-2-3
1.03	NP600	0.8	-	3-hydroxymethyl-2,5-dimethylphenyl methylcarbamate	C11H15NO3	-	NR	NR	6-15-50	1-2-3
1.04	NI4	0.55	0.86	tetraiodoethylene	C2I4	-	P	P	6	-
1.05		-	-	2,4,5-T butyl esters*	C12H13Cl3O3	-	-	-	-	-
1.05		0.87	-	demeton-O sulfoxide	C8H15O4PS2	C	-	-	-	-
1.05	NI0.6	1.33	0.94	octachlor epoxide	C10H4Cl8O	C	C	C	6	1
1.05	NP(WB)8	-	-	norea	C13H15N2O	C	-	-	-	-
1.05	FP3/FP(WB)1.1	0.84	1.82	fenitrothion	C9H12NO5PS	C	C	C	15	2
1.05	FP3/NI7	0.91	1.49	malathion	C10H19O6PS2	C	C	C	15+50	3
1.05		0.75	-	carbaryl	C12H11NO2	C	-	-	-	-
1.06	FP40	0.78	-	phorate oxygen analog sulfoxide	C7H17O4PS2	C	NR	NR	6-15-50	1-2-3
1.06	NI10/NP125	0.83	3.77	metribuzin, deaminated metabolite	C8H13N3OS	C	NR	NR	6-15-50	1-2-3
1.07		0.87	-	4-chlorophenoxyaniline*	C12H10ClNO	S	-	-	-	-
1.07	NI1	0.99	1.63	1-hydroxychlordene	C10H6Cl6O	-	R	-	15	-
1.07	FP2/NI4	0.98	1.91	parathion	C10H14NO5PS	C	C	C	15	2
1.07	NI(WB)40	0.54	-	4-chlorophenylurea	C7H7ClN2O	NR	NR	NR	6-15-50	1-2-3
1.08	HX3	4.4	1.28	dicofol, p,p'-*	C14H9Cl5O	C	V	P #	6+15	1+2
1.08		0.95	1.51	chlorpyrifos oxygen analog	C9H11Cl3NO4P	C	NR	-	6-15-50	-
1.08	NI2	0.99	1.25	dichlorobenzophenone, p,p'-	C13H8Cl2O	-	C	C	15	2
1.09	NI1/NP6	1.25	8	MB45950	C12H4SN4F6Cl2	S	P	V	15+50	2+3
1.12	NI2	1.37	1.22	S-bioallethrin	C19H26O3	-	C	-	50	-
1.12	FP9	0.78	-	fenthion oxygen analog	C10H15O4PS	C	NR	NR	6-15-50	1-2-3
1.13	NI(WB)600	1.04	-	PPG-947	C17H11ClF3NO7	-	NR	NR	6-15-50	1-2-3
1.14	FP(WB)2.4	1.01	-	pirimiphos-ethyl oxygen analog	C13H24N3O4P	C	-	-	-	-
1.14	FP3/NI4	1.14	1.03	pirimiphos-ethyl	C13H24N3O3PS	C	C	C	15+50	3
1.15	HX0.9/NI2	1.29	1.22	heptachlor epoxide	C10H5Cl7O	C	C	C	6	2
1.16	NI1/NP5	1.35	8.7	fipronil	C12H4Cl2F6N4OS	S	S	V	50	3
1.16	FP3/NI2	1.11	1.29	bromophos	C8H8BrCl2O3PS	C	C	C	6	-
1.17		0.8	-	methiocarb sulfone	C11H15NO4S	S	NR	NR	6-15-50	1-2-3
1.18	NP1000	3	-	NTN33823	C9H11N4Cl	-	NR	NR	6-15-50	1-2-3

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Appendix I: PESTDATA Chemicals in Order by OV-17 Relative Retention Time (continued)

RRT/c OV-17	Detector Responses with OV-17 Column	RRT/c OV-101	RRT/c OV-225	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
1.18	FP3	0.96	1.46	fenthion	C10H15O3PS2	C	S #	NR	6+15	1-2-3
1.19	HX13/HX(WB)2/NI(WB)3/NP(WB)3	1.44	2.23	triflumizole	C15H15ClF3N3O	C	-	-	-	-
1.2	NI2	1.19	1.15	TDE, o,p'-, olefin	C14H9Cl3	-	-	-	-	-
1.21	NI1.5/NP6	1.22	1.48	pendimethalin	C13H19N3O4	C	C	P	15	2
1.24	FP7	1.17	1.74	isofenphos oxygen analog	C15H24NO5P	C	-	-	-	-
1.26	FP6	0.89	2.55	phorate sulfoxide	C7H17O3PS3	C	NR	NR	6-15-50	1-2-3
1.28	FP2/NI60	0.92	2.9	terbufos oxygen analog sulfone	C9H21O5PS2	C	NR	NR	6-15-50	1-2-3
1.29	FP(WB)2/NI3	1.21	1.58	chlorfenvinphos, alpha-	C12H14Cl3O4P	C	-	NR	6-15-50	-
1.3	FP2	0.97	3.26	phorate sulfone	C7H17O4PS3	C	S #	S #	6-15-50	3
1.3	FP2/NI3	1.08	2.33	crufomate	C12H19ClNO3P	C	NR	NR	6-15-50	-
1.31		1.28	-	4-chlorophenoxyaniline*	C12H10ClNO	S	-	-	-	-
1.32	HX3/HX(WB)2/NI(WB)2/NP(WB)3	1.24	-	penconazole	C13H15Cl2N3	C	-	-	-	-
1.32		0.96	-	carbetamide	C12H16N2O3	-	-	-	-	-
1.32	HN(WB)1/NP(WB)50	1.4	-	dinobuton	C14H18N2O7	C	-	-	-	-
1.33	NI40/NP200	1.11	4.7	isoxaflutole (prop)	C15H12SNO4F3	NR	V #	S #	50	3
1.34	NI0.6	1.49	1.46	chlordane, trans-	C10H6Cl8	C	C	C	6	1
1.36	NP250	0.21	-	3-(3,4-dichlorophenyl)-1-methoxyurea	C8H8Cl2N2O2	R	NR	NR	6-15-50	
1.36	HN(WB)1/HX(WB)8/NI(WB)6/NP(WB)5	0.8	4.8	bromacil	C9H13BrN2O2	C	NR	NR	6-15-50	1-2-3
1.38	NI40/NP250	1.13	4.7	RPA202248	C15H12SNO4F3	NR	NR	NR	6-15-50	1-2-3
1.38	FP2	1.36	1.73	isofenphos	C15H24NO4PS	C	C	-	15+50	-
1.39	NP(WB)10	1.18	-	cyprodinil	C14H15N3	C	NR	NR	6-15-50	1-2-3
1.4	HX(WB)6/NI4.5	1.55	-	haloxyfop methyl ester	C16H13ClF3NO4	-	-	-	-	-
1.41		1.25	-	tolylfluanid	C10H13ClFNOS	C	-	-	-	-
1.42	HX0.4/NI0.8	1.75	1.45	nonachlor, trans-	C10H5Cl9	C	C	C	6	1
1.42		1.27	3.39	chlorbromuron	C9H10BrClN2O2	V	V	V	50	3
1.43	FP25	1.34	0.65	merphos*	C12H27PS3	-	C	C	6+15+50	3
1.43	FP(WB)12	0.93	-	des N-isopropyl isofenphos oxygen analog	C12H18NO5P	-	-	-	-	-
1.43	NP6	1.05	-	pyracarbolid	C13H15NO2	-	-	-	-	-
1.44	NI5	-	1.67	2,4-D butoxyethyl ester*	C14H18Cl2O4	-	-	-	-	-

Appendix I: PESTDATA Chemicals in Order by OV-17 Relative Retention Time (continued)

RRT/c OV-17	Detector Responses with OV-17 Column	RRT/c OV-101	RRT/c OV-225	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
1.44	HX7/HX(WB)4/NI(WB)50/NP(WB)6	1.36	-	triadimenol	C14H18ClN3O2	C	NR	NR	6-15-50	-
1.45	FP(WB)0.3	1.51	1.42	bromophos-ethyl	C10H12BrCl2O3PS	C	C	P	6	-
1.45	NI4	1.45	1.36	TDE, p,p'-, olefin	C14H9Cl3	C	C	C	6	1
1.46	FS88/NI96	1	6.6	2-hydroxy-2,3-dihydro-3,3-methyl-5-benzofuranyl methyl sulfonate	C11H14O5S	-	-	-	-	-
1.46	HX5/NP4	1.5	-	metazachlor	C14H16ClN3O	C	-	-	-	-
1.46	HX9	1.73	1.83	butachlor	C17H26ClNO2	C	C	-	50	-
1.47	HX1/NI2	1.64	1.38	endosulfan I	C9H6Cl6O3S	C	C	C	15	2
1.47	NP20	1.24	1.88	anilazine	C9H5Cl3N4	V	S	P	15+50	2+3
1.48	NI5	-	-	2,4-D isooctyl ester*	C16H22Cl2O3	-	-	-	-	-
1.48	HX6	0.89	4.9	cyanazine	C9H13ClN6	C	NR	-	6-15-50	-
1.48	FP(WB)3	0.72	-	oxydemeton-methyl sulfone	C6H15O5PS2	C	-	-	-	-
1.48	NI0.8	1.66	1.54	chlordane, cis-	C10H6Cl8	C	C	C	6	1
1.49	HX1	1.37	3.04	procymidone	C13H11Cl2NO2	C	C	P	15	-
1.5	FP3	1.21	2.73	des N-isopropyl isofenphos	C12H18NO4PS	C	S	-	50	-
1.51	NI1	1.55	1.28	DDE, o,p'-	C14H8Cl4	C	C	C	6	1
1.52	FP4/FP(WB)2/NI3	1.29	2	chlorfenvinphos, beta-	C12H14Cl3O4P	C	S #	-	50	1-2-3
1.54	NI1000	1.39	1.89	CGA 189138	C13H8O3Cl2	-	-	-	-	-
1.54		1.15	4.3	CGA 91305	C10H8Cl2N3O	V	NR	NR	6-15-50	1-2-3
1.54	HX3/NI1	1.39	1.62	chlorbenside	C13H10Cl2S	C	S	P	6	1
1.55	HN(WB)3/HX(WB)17/NI(WB)19/NP(WB)12	0.86	-	6-chloro-2,3-dihydro-7-hydroxy=methyl-3,3-methyl-5H-oxazolo=(3,2-a)pyrimidin-5-one	C9H13ClN2O3	-	NR	NR	6-15-50	1-2-3
1.55	NP(WB)25	1.1	-	diphenamid	C16H17NO	V	NR	-	6-15	-
1.56	NP4	1.16	-	phenothiazine	C12H9NS	-	-	-	-	-
1.58	FP2/NI10	1.2	-	terbufos sulfone	C9H21O4PS3	C	C #	C #	6-15-50	2+3
1.58	FP3/FP(WB)1.9	1.28	2.67	mecarbam	C10H20NO5PS2	C		-	50	-
1.59	NI1000	6.7	-	CGA 205375	C16H13N3O2Cl2	-	-	-	-	-
1.59	HX7/HX(WB)3/NI(WB)85/NP(WB)5	1.52	-	paclobutrazol	C15H20ClN3O	C	-	-	-	-
1.6	NI1000	1.28	-	3-phenoxybenzenemethanol	C13H12O2	-	-	-	-	-
1.64	NI6/NP30	1.57	1.84	cyclanilide methyl ester	C12H11Cl2NO3	-	-	-	-	-

Appendix I: PESTDATA Chemicals in Order by OV-17 Relative Retention Time (continued)

RRT/c OV-17	Detector Responses with OV-17 Column	RRT/c OV-101	RRT/c OV-225	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
1.64	FP3/FP(WB)3	1.32	2	quinalphos	C12H15N2O3PS	C	C	-	15	-
1.65		1.65	-	DDMS	C14H11Cl3	-	R	-	6	-
1.66	FP10/NI120/NP7	1.08	3	fosthiazate	C9H18NO3PS2	C	NR	NR	6-15-50	1-2-3
1.68		2.1	1.78	2,4-D ethyl hexyl ester*	C16H22Cl2O3	-	-	-	-	-
1.75	FP5	1.15	5.8	demeton-S sulfone	C8H19O5PS2	C	-	-	-	-
1.76		2.12	1.76	pyrethrins*	C21H27O4	-	C	C	50	-
1.78	NI5	2.04	1.78	2,4-D isooctyl ester*	C16H22Cl2O3	-	-	-	-	-
1.79	NI5	1.82	2.08	2,4-D butoxyethyl ester*	C14H18Cl2O4	-	-	-	-	-
1.79	NP(WB)23	1.86	2.91	hexaconazole	C14H17Cl2N3O	C	-	-	-	-
1.8	FP125/NI200/NP40	1.07	-	methidathion oxygen analog	C6H11N2O5PS2	-	NR	NR	6-15-50	1-2-3
1.82	NI3	1.85	1.74	prothiofos	C11H15Cl2PO2S2	C	C	C	6	2
1.83	FP2/FP(WB)2.6/NI5	1.31	2.05	phenthoate	C12H17O4PS2	C	C	-	15+50	-
1.84	HX1.5/NI1	1.91	1.87	dieldrin	C12H8Cl6O	C	C	C	15	2
1.85		1.57	-	oxythioquinox	C10H6N2OS2	C	-	-	-	-
1.85	NI2	1.2	3.49	captan	C9H8Cl3NO2S	C	P	C	50	3
1.86	HN(WB)120/HX(WB)66/ NI(WB)78	1.9	3.16	PPG-2597	C20H17ClF3NO6	-	NR	NR	6-15-50	1-2-3
1.86	NI1	1.92	1.59	DDE, p,p'-	C14H8Cl4	C	C	C	6	1
1.88	FP25	1.95	1.64	merphos*	C12H27PS3	-	C	C	6+15+50	3
1.88		1.73	-	chlorflurecol methyl ester	C15H11ClO3	C	-	-	-	-
1.88	FP3/NI3	1.95	1.65	tribufos	C12H27OPS3	C	C	P	15+50	3
1.9	FP10/FP(WB)3	1.37	2.85	crotoxyphos	C14H19O6P	C	NR	NR	6-15-50	1-2-3
1.92		1.26	3.5	Sulphenone	C12H9ClO2S	C		-	50	3
1.94	NI9	1.23	3.01	folpet	C9H4Cl3O2NS	C	C	P	15+50	2+3
1.96	NI2	1.97	2.48	oxadiazon	C15H18Cl2N2O3	C	C	P	15	-
1.97	FP10/FP(WB)3	1.58	2.72	Gardona	C10H9Cl4O4P	C	NR	NR	6-15-50	1-2-3
1.98	NI2/NP10	2.06	31	MB46136	C12H4SO2N4F6Cl2	S	S	V	50	2+3
1.99	HX10	1.88	-	pretilachlor	C17H26ClNO2	C	-	-	-	-
2	NI11/NP200	1.78	3.14	diethatyl-ethyl	C16H22ClNO3	C	NR	NR	6-15-50	1-2-3
2.03	HN(WB)1.3/HX7/HX4/ HX(WB)2/NI(WB)4/NP(WB)8	2.02	3.4	diclobutrazol	C15H19Cl2N3O	C	NR	NR	6-15-50	1-2-3
2.04		1.48	-	thiabendazole	C10H7N3S	C	NR	-	6-15-50	-

Appendix I: PESTDATA Chemicals in Order by OV-17 Relative Retention Time (continued)

RRT/c OV-17	Detector Responses with OV-17 Column	RRT/c OV-101	RRT/c OV-225	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
2.08	HX19/HX(WB)2/NI(WB)5/NP(WB)10	1.76	4	imazalil	C14H14Cl2N2O	C	NR	NR	6-15-50	-
2.1		1.58	2.46	triazamate	C13H22N4O3S	C	NR	NR	6-15-50	1-2-3
2.11	FP7	-	-	jodfenphos	C8H8Cl2IO3PS	C	-	-	-	-
2.11	NP200	-	-	sulfanilamide	C6H8O2N2S	NR	NR	NR	6-15-50	1-2-3
2.12		1.7	-	napropamide	C17H20NO2	C	-	-	-	-
2.13	FP5/FP(WB)2.9	1.8	2.34	profenofos	C11H15BrClO3PS	C	P	P	50	3
2.16	HX9/NI2/NP350	2	4	oxyfluorfen	C15H11ClF3NO4	C	C	C	15	2
2.19	NI2	1.9	2.46	TDE, o,p'-	C14H10Cl4	-	C	C	6	1
2.2	HX5	1.61	3.04	ovex	C12H8Cl2O3S	C	C	C	15	2
2.28	FP(WB)2.4/NI10	1.4	3.33	methidathion	C6H11N2O4PS3	C	S	P #	50	3
2.29	NI2	2.13	2.22	endrin	C12H8Cl6O	C	C #	C #	15	2
2.31	HX40	2.3	2.36	fluazifop butyl ester	C19H20F3NO4	C	C	V	15	3
2.33	HX24/HX(WB)5/NP(WB)6	1.97	-	flusilazole	C16H15F2N3Si	C	-	-	-	-
2.34	NI2/NP50	2.21	-	chlorfenapyr (prop)	C15H11BrClF3N2O	P	-	S	50	2
2.38	NI22/NP(WB)100	2.19	4.2	binapacryl	C15H18N2O6	C	P	P	15	-
2.38	HX2/NI5	2.75	1.67	chlordecone	C10H8Cl10O5	-	S #	P #	15+50	1-2-3
2.39	FP7	1.5	6.7	disulfoton sulfone	C8H19O4PS3	C	NR	-	6-15-50	-
2.4	HN(WB)5	2.14	5	PPG-947, methylated*	C18H13ClF3NO7	-	-	-	-	-
2.4	HN(WB)4/HX(WB)6.5/NI(WB)3/NP(WB)210	2.15	5	PPG-847, methylated	C15H9ClF3NO3	-	-	-	-	-
2.41	NI5/NP38	1.53	6.5	CGA 94689A	C15H21NO5	V	NR	NR	6-15-50	1-2-3
2.41	FS(WB)90/NI(WB)80/NP(WB)80	1.28	-	hexythiazox	C17H21ClN2O2S	-	S #	NR	50	2+3
2.41	FP10/NP3	1.66	3.7	fenamiphos	C13H22NO3PS	C	NR	NR	6-15-50	1-2-3
2.41	NI15	2.33	2.9	chloropropylate	C17H16Cl2O3	P	C	C	15+50	3
2.42	NI25	2.23	2.01	Perthane	C18H2OCl2	C	C	C	6	1
2.44	HX60	-	-	imazamethabenz methyl ester*	C16H20N2O3	C	-	-	-	-
2.45	NI10/NP75	1.54	6.6	CGA 94689B	C15H21NO5	S	NR	NR	6-15-50	1-2-3
2.45		1.94	-	flamprop-methyl	C17H15ClFNO3	C	-	-	-	-
2.51	FP3	-	-	mephosfolan	C8H16NO3PS2	C	-	-	-	-
2.55	HN(WB)13/HX(WB)99/NI(WB)35/NP(WB)290	1.35	2.27	3-tert-butyl-5-chloro-6-hydroxy= methyluracil	C9H13ClN2O3	-	NR	NR	6-15-50	1-2-3

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Appendix I: PESTDATA Chemicals in Order by OV-17 Relative Retention Time (continued)

RRT/c OV-17	Detector Responses with OV-17 Column	RRT/c OV-101	RRT/c OV-225		Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
2.55	HN(WB)1.2/NP(WB)53	1.41	-		IN-T3936	C10H15N3O4	S	NR	NR	6-15-50	1-2-3
2.58	FP5	2.24	-		chlorthiophos*	C11H15Cl2O3PS2	C	C	C	6	2
2.6	HX10/HX(WB)4/NI(WB)21/ NP75/NP(WB)6	1.9	7.2		myclobutanil	C15H17ClN4	C	NR	NR	6-15-50	1-2-3
2.6	NP(WB)300	2	3.7		bupirimate	C13H24N4SO3	C	-	-	-	-
2.61	NI2	2.52	3.33		nonachlor, cis-	C10H5Cl9	C	C	C	6	1
2.61	NI15	2.31	3.26	-	chlorobenzilate	C16H14Cl2O3	C	C #	P #	15+50	3
2.62		3.38	-		2,4,5-T ethylhexyl ester	C16H21Cl3O3	-	-	-	-	-
2.67	NP36	1.5	4.3		TCMTB	C9H6N2S3	C	P	P	15	-
2.69		2.04	1.61		cyproconazole	C15H18ClN3O	C	NR	NR	6-15-50	1-2-3
2.69	FP5	-	-		phosfolan	C7H14NO3PS2	C	-	-	-	-
2.7		2.95	2.84		pyrethrins*	C21H27O4	-	C	C	50	-
2.7	NI2	2.55	2.27		DDT, o,p'-	C14H9Cl5	C	C	C	6	1
2.71	NI3	2.03	3.8		nitrofen	C12H7Cl2NO3	C	C	C	15	2
2.76	HX60	1.79	3.5		imazamethabenz methyl ester*	C16H20N2O3	C	-	-	-	-
2.77	FP5	2.36	-		chlorthiophos*	C11H15Cl2O3PS2	C	C	C	6	2
2.77	HX3/NI2	2.21	3.9		endosulfan II	C9H6Cl6O3S	C	C	C	15+50	2
2.8	NI5	2.64	2.33		tetrasul	C12H6Cl4S	C	C	C	6	1
2.81		2.46	-		flamprop-M-isopropyl	C19H19ClFNO3	C	-	-	-	-
2.87	FP15	4.2	-		carbophenothion oxygen analog sulfoxide	C11H16ClO4PS2	-	-	-	-	-
2.87	NI2	2.41	3.8		TDE, p,p'-	C14H10Cl4	C	C	C	6	1
2.88		3.03	2.74		ethephon	C2H6ClO3P	NR		-	6+15+50	1+2+3
2.92	NP(WB)8	2.07	-		methoprotryne	C11H21N5OS	C	-	-	-	-
2.96	NI1000/NP150	1.8	-		CGA 100255	C15H12NO5	S	-	-	-	-
2.99	FP10/HX11	2.22	4.1		chlorthiophos oxygen analog	C11H15Cl2O4PS	C	NR	NR	6-15-50	1-2-3
3	NI120/NP70	2.4	4.3		imazethapyr ammonium salt methyl ester	C16H21N3O3	-	-	-	-	-
3.06	FP15	2.17	4.2		carbophenothion oxygen analog	C11H16ClO3PS2	C	NR	NR	6-15-50	1-2-3
3.14	NI5	2.38	3.24		leptophos photoproduct	C13H11Cl2O2PS	C	-	-	-	-
3.16	FP5	2.56	-		chlorthiophos*	C11H15Cl2O3PS2	C	C	C	6	2
3.17	HX7	2.43	-		etaconazole*	C14H15Cl2N3O2	C	-	-	-	-

Appendix I: PESTDATA Chemicals in Order by OV-17 Relative Retention Time (continued)

RRT/c OV-17	Detector Responses with OV-17 Column	RRT/c OV-101	RRT/c OV-225	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
3.17		1.6	4.1	isoprothiolane	C12H18O4S2	C	-	-	-	-
3.36	FP4/FP(WB)2.3/NI8	2.56	3.93	ethion	C9H22O4P2S4	C	C	C	6	2
3.38	NI(WB)15/NP(WB)40	2	3.02	kresoxim-methyl	C18H19NO4	P	C	C	15+50	3
3.5	NI150	-	-	dinocap*	C18H24N2O6	C	P	P	15	2
3.5	FP3	2.79	-	sulprofos	C12H19O2PS3	C	-	-	-	-
3.5	NI2	3.13	3.6	DDT, p,p'-	C14H9Cl5	C	C	C	6	1
3.6	FP(WB)29	2.78	-	sulprofos sulfoxide*	C12H19O3PS3	C	-	-	-	-
3.6	FP100	2.8	-	fensulfothion sulfone	C11H17O5PS2	C	NR	-	6-15-50	-
3.7		2.9	-	1,1'-(2,2-dichloroethylidene)bis(2-methoxybenzene)	C16H16Cl2O2	-	R	-	-	-
3.7	FP8	2.94	4.2	carbophenothion	C11H16ClO2PS3	C	C	P	6	2
3.7	NI(WB)5	3.1	-	fenhexamid	C14H17Cl2NO2	NR	NR	NR	6-15-50	1-2-3
3.8	FP6	1.99	-	fensulfothion oxygen analog sulfone	C11H17O7PS2	-	-	-	-	-
3.8	FP6/FP(WB)7	2.4	-	fensulfothion	C11H17O4PS2	C	NR	NR	6-15-50	1-2-3
3.9	NI150	4	-	dinocap*	C18H24N2O6	C	P	P	15	2
3.9	NP500/NP(WB)8	1.59	-	tricyclazole	C9H7N3S	C	-	-	-	-
3.9	NI6	2.81	7.5	Prolan	C15H13Cl2NO2	P	S	S	15	2
3.93		2.31	15	desisopropyl iprodione	C10H6Cl2N3O3	P		-	50	1-2-3
4	HX9	3.21	5.6	propiconazole*	C15H17Cl2N3O2	C	NR	NR	6-15-50	1-2-3
4	FP20	5.4	-	carbophenothion sulfoxide	C11H16ClO3PS3	-	-	-	-	-
4	HX6/NI6	2.83	8.3	endosulfan sulfate	C9H6Cl6O4S	C	C	C	50	2
4.1	FP(WB)8	2.29	-	fenthion oxygen analog sulfone*	C10H15O6PS2	-	-	-	-	-
4.1	NI20/NP100	2.96	11.5	CL 202,347	C13H19N3O5	-	-	-	-	-
4.2	HN(WB)2/HX(WB)13/ NI(WB)18/NP(WB)85	2.51	4	pyrithiobac-sodium methyl ester	C14H13ClN2O4	-	-	-	-	-
4.2	HX(WB)3	3.38	-	tebuconazole	C16H22ClN3O	C	-	-	-	-
4.2	NI200/NP50	4	15	KWG 1342	C14H18ClN3O3	-	-	-	-	-
4.2	NI10	2.97	3.7	methoxychlor olefin	C16H14Cl2O2	C	C	C	6	2
4.3	NI2600	3.8	4.8	propargite	C19H26O4S	C	C	-	15	2
4.4	NI150	4.3	6.9	dinocap*	C18H24N2O6	C	P	P	15	2
4.4	FP(WB)12	2.26	-	famphur oxygen analog	C10H16NO6PS	C	-	-	-	-
4.4	NI5	3.06	7.5	Bulan	C16H15Cl2NO2	C	P	P	15	2

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Appendix I: PESTDATA Chemicals in Order by OV-17 Relative Retention Time (continued)

RRT/c OV-17	Detector Responses with OV-17 Column	RRT/c OV-101	RRT/c OV-225	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
4.5	NI100	6.1	1.48	tralkoxydim*	C20H27NO3	V	NR	NR	50	1-2-3
4.5	HX5/HX(WB)20	4.9	3.8	bifenthrin	C23H22ClF3O2	V	C	-	6+15	2
4.5		2.43	-	benodanil	C13H10INO	C	-	-	-	-
4.5		3.06	5.1	butyl benzyl phthalate	C19H20O4	-	C	P	15+50	-
4.6	HN(WB)100/HX(WB)14/ NI(WB)85/NP(WB)210	2.87	-	vinclozolin metabolite F	C11H13Cl2NO4	R	NR	NR	6-15-50	1-2-3
4.6		3.1	8.2	cyanofenphos	C15H14NO2PS	C	-	-	-	-
4.67	NI(WB)20	3.26	5.8	clodinafop-propargyl	C17H13ClFNO4	V	V	-	50	3
4.7	HX10	3.57	4.9	diclofop-methyl	C16H14Cl2O4	C	C	C	15	2
4.7	FP20	2.39	-	fenthion sulfone	C10H15O5PS2	C	NR	NR	6-15-50	1-2-3
4.8	NI150	4.8	7.7	dinocap*	C18H24N2O6	C	P	P	15	2
4.8	HX4	3.36	7.3	nuarimol	C17H12ClFN2O	C	NR	C #	50	1-2-3
4.9	HX(WB)3	3.38	1.41	desmethyl norflurazon	C11H7ClF3N3O	V	NR	NR	6-15-50	1-2-3
5	NP14	2.5	14	oxadixyl	C14H18N2O4	C	NR	NR	6-15-50	1-2-3
5	NI9	3.3	4.5	methoxychlor, o, p'-	C16H15Cl3O2	-	C	-	6	-
5	FP5	2.49	-	fensulfothion oxygen analog	C11H17O5PS	C	NR	-	6-15-50	-
5	FP50/FP(WB)7	2.65	14	famphur	C10H16NO5PS2	C	NR	-	6-15-50	-
5.01	NP(WB)10	4.5	-	norflurazon	C12H9ClF3N3O	V	NR	NR	6-15-50	-
5.2	HX20/NI35/NP200	-	-	iprodione*	C13H13Cl2N3O3	C	S #	NR	50	1-2-3
5.2	FP5/FP(WB)5	2.62	-	triazophos	C12H16N3O3PS	C	-	-	-	-
5.3		5.4	-	carbosulfan	C20H32N2O3S	P	-	-	-	-
5.3	FP(WB)8	2.87	6.3	edifenphos	C14H15O2PS2	C	-	-	-	-
5.4	HX51	2.62	18.6	ofurace	C14H16NO3Cl	C	-	-	-	-
5.4	NI5	3.11	-	captafol	C10H9Cl4NO2S	C	P	-	50	3
5.6	NI150	5.1	9.5	dinocap*	C18H24N2O6	C	P	P	15	2
5.6	NI4	5.8	2.95	mirex	C10Cl12	P	C	P	6	1
5.7	NI13	4.8	7	fenpropathrin	C22H23NO3	-	V #	V	15	2
6	NI8	4.3	8.4	benzoylprop-ethyl	C18H17Cl2NO3	P	NR	NR	6-15-50	1-2-3
6.1	NI200	12	8.9	CGA 205374	C16H11N3O2Cl2	-	NR	NR	6-15-50	1-2-3
6.1		6.4	4.5	bis(2-ethylhexyl) phthalate	C24H38O4	-	C	C	15+50	-
6.2		17	-	deltamethrin, trans-*	C22H19Br2NO3	-	P #	NR	15	2
6.2	FP(WB)12	3.8	-	phosalone oxygen analog	C12H15ClNO5PS	C	-	-	-	-

Appendix I: PESTDATA Chemicals in Order by OV-17 Relative Retention Time (continued)

RRT/c OV-17	Detector Responses with OV-17 Column	RRT/c OV-101	RRT/c OV-225		Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
6.3	HX20/NI35/NP200	4.2	18		iprodione*	C13H13Cl2N3O3	C	S #	NR	50	1-2-3
6.3	NI7	3.8	24		nitralin	C13H19N3O6S	C	P	P	50	3
6.3	NI(WB)20	4.8	6.6		cloquintocet-mexyl	C18H22ClNO3	V	NR	-	6-15-50	1-2-3
6.5		5.4	4.8	-	phenothrin*	C23H26O3	-	-	-	-	-
6.5		4.2	7.6		leptophos oxygen analog	C13H10BrCl2O3P	C	-	-	-	-
6.8		4.8	9.7		piperophos	C14H28NO3PS2	C	-	-	-	-
6.9	FP25/HX17	4.7	10.3		chlorthiophos sulfoxide	C11H15Cl2O4PS2	C	NR	NR	6-15-50	1-2-3
6.9	FP50/NI9	4.5	10.6		EPN	C14H14NO4PS	C	C	C	15	2
7.1	FP150	3.1	14.8		phosmet oxygen analog*	C11H12NO5PS	-	NR	NR	6-15-50	-
7.1	FP(WB)24	3.8	-		carbophenothion oxygen analog sulfone	C11H16ClO5PS2	-	-	-	-	-
7.2		4.5	8.5		tetramethrin*	C19H25NO4	C	NR	NR	6-15-50	1-2-3
7.2	NI8	4.7	8.6		tetrasul sulfoxide	C12H6Cl4OS	-	-	-	-	-
7.2	NI7	4.7	7.2		methoxychlor, p, p'-	C16H15Cl3O2	C	C	C	6	2
7.3	NP50	5	-		fenoxycarb	C17H19NO4	C	-	-	-	-
7.5	FS(WB)1500/NI(WB)0.1/ NP(WB)73	3.3	-		3-desmethyl sulfentrazone	C10H8Cl2F2N4O3S	-	NR	NR	6-15-50	1-2-3
7.5	NP375	3.6	37.1		myclobutanil alcohol metabolite	C15H17ClN40	S	NR	NR	6-15-50	1-2-3
7.5	HX80	5.3	-		iprodione metabolite isomer	C13H13Cl2N3O3	C	S	-	50	-
8	HX30	7.4	-		lambda-cyhalothrin	C23H19ClF3NO3	C	-	-	-	-
8	HX(WB)50	2.67	13		pyrazon	C10H8ClN3O	C	NR	NR	6-15-50	1-2-3
8.1	FP(WB)28/NP45	5.2	-		fenamiphos sulfoxide	C13H22N04PS	C	NR	NR	6-15-50	1-2-3
8.3	NI5	5.2	-		tetradifon	C12H6Cl4O2S	C	C	C	15	2
8.4	FP(WB)20/NP60	4.5	-		fenamiphos sulfone	C13H22NO5PS	C	NR	NR	6-15-50	1-2-3
8.4	FP50/NI78	4	14.9		phosmet	C11H12O4NPS2	C	NR	-	6-15-50	3
8.5	FP(WB)15/NI12	5.8	7.7		leptophos	C13H10BrCl2O2PS	C	C	C	6	2
8.5	NI5	4.4	15.5		photodieldrin	C12H8Cl6O	-	C	C	15+50	2
8.7		4.2	14		pyridaphenthion	C14H17O4N2SP	C	-	-	-	-
8.8		5	14.9		bifenox	C12H9Cl2NO5	C	C	P	15+50	2+3
8.9	NI25/NP100	10.4	12.8		acrinathrin	C26H21F6NO5	V	V	V #	15	2
9.1	FP100/HX22	5.3	18.8		chlorthiophos sulfone	C11H15Cl2O5PS2	C	C	-	50	3
9.1	NI12	5.5	5.5		phosalone	C12H15ClNO4PS2	C	C	C	50	2+3

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Appendix I: PESTDATA Chemicals in Order by OV-17 Relative Retention Time (continued)

RRT/c OV-17	Detector Responses with OV-17 Column	RRT/c OV-101	RRT/c OV-225	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
9.2	FP30	5.1	-	carbophenothion sulfone	C11H16ClO4PS3	C	C	P	6	1
9.4	HN(WB)4	3.28	-	oxycarboxin	C12H13NO4S	R	-	-	-	-
9.8	NP165	5.9	-	clofentezine	C14H8Cl2N4	R	S	-	15	2
10.1		6.6	-	fenarimol	C17H12Cl2N2O	C	P #	C #	50	3
10.1	FP(WB)42	3.7	-	azinphos-methyl oxygen analog	C10H12N3O4PS	C	-	-	-	-
10.5		8.1	11.3	fenoxaprop ethyl ester	C18H16NO5Cl	S	V	V	50	3
10.6	FP40/FP(WB)28/NI20/NP40	5.1	-	sulprofos oxygen analog sulfone	C12H19O5PS2	C	-	-	-	-
11		7	-	tebufenozide	C22H28N2O2	-	NR	NR	6-15-50	1-2-3
11.3	NP(WB)120	4.7	53	dithianon	C14H4O2N2S2	NR	-	-	-	-
11.4		7	-	CGA 118244	C15H13Cl2N3O3	V	NR	NR	6-15-50	1-2-3
11.5	NI1000/NP1000	6.5	-	myclobutanil dihydroxy metabolite	C15H17N4O2Cl	NR	NR	NR	6-15-50	1-2-3
11.7	FP(WB)29	6.1	-	sulprofos sulfoxide*	C12H19O3PS3	C	-	-	-	-
11.8	NP(WB)200	9.4	-	bitertanol*	C20H23N3O2	C	-	-	-	-
11.8		5.2	-	azinphos-methyl	C10H12N3O3PS2	C	NR	NR	6-15-50	1-2-3
12.5	NP(WB)200	9.7	-	bitertanol*	C20H23N3O2	C	-	-	-	-
12.6	NI70	7.9	10.8	HOE-030291	C17H16Cl2O5	-	-	-	-	-
13	FP25	8.1	-	pyrazophos	C14H20N3O5PS	C	-	-	-	-
13.1	FP(WB)26	7.2	-	sulprofos sulfone	C12H19O4PS3	C	-	-	-	-
13.8		9.4	11.1	permethrin, cis-	C21H20Cl2O3	C	V #	C	6+15	2
14.3	FP25/FP(WB)31	6.5	-	dialifor	C14H17ClNO4PS2	C	C	P	15	2
14.8	FP(WB)26/NI20	6.9	-	azinphos-ethyl	C12H16N3O3PS2	C	P	S	50	3
15		11.5	10.9	phenothrin*	C23H26O3	-	-	-	-	-
15		10.2	13	permethrin, trans-	C21H20Cl2O3	C	V #	C	6+15	2
15.4		10.4	-	prochloraz	C15H16Cl3N3O2	C	-	-	-	-
16	FP75/NI50/NP40	8	45	coumaphos oxygen analog	C14H16ClO6P	C	NR	NR	6-15-50	1-2-3
16	NI400	13	13	hexachlorophene	C13H6Cl6O2	-	NR	NR	6-15-50	-
18	FP50/FP(WB)26/NI38/NP34	9	40	coumaphos	C14H16ClO5PS	C	NR	C #	6-15-50	3
20		29	-	deltamethrin, trans-*	C22H19Br2NO3	-	P #	NR	15	2
20.2	FP100	9.5	-	bensulide	C14H24NO4PS3	C	P	C	50	3
21		17.1	-	deltamethrin*	C22H19Br2NO3	C	S #	P	15	2
21.4		14.7	36.9	flucythrinate*	C26H23F2NO4	C	C	-	15	2+3
23		14.1	33	cypermethrin*	C22H19Cl2NO3	C	C	C	15	2

Appendix I: PESTDATA Chemicals in Order by OV-17 Relative Retention Time (continued)

RRT/c OV-17	Detector Responses with OV-17 Column	RRT/c OV-101	RRT/c OV-225	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
24		16.1	42	flucythrinate*	C26H23F2NO4	C	C	-	15	2+3
25		15.1	36	cypermethrin*	C22H19Cl2NO3	C	C	C	15	2
25		13.6	-	quizalofop ethyl ester	C19H17ClN2O4	C	-	-	-	-
35		-	56	fluvalinate*	C26H22ClF3N2O3	C	C	-	15	2
35		27	-	deltamethrin*	C22H19Br2NO3	C	S #	P	15	2
35		20.3	44	fenvalerate*	C25H22ClNO3	C	C	C	15	2
38		25	59	fluvalinate*	C26H22ClF3N2O3	C	C	-	15	2
38		31	19.7	deltamethrin, trans-*	C22H19Br2NO3	-	P #	NR	15	2
38		29	19.9	deltamethrin*	C22H19Br2NO3	C	S #	P	15	2
40		22.5	51	fenvalerate*	C25H22ClNO3	C	C	C	15	2
43	NI200/NP500	23	57	PB-7, methylated	C20H25ClN2O3S	-	-	-	-	-
44		27	64	tralomethrin	C22H19Br4NO3	C	V	S	15	2
46	NI250/NP750	25	87	PB-9	C19H25ClN2O2S	V	NR	NR	6-15-50	1-2-3

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APPENDIX II: PROTOCOLS AND REPORTING FORMS FOR TESTING CHEMICALS THROUGH PAM I MULTIRESIDUE METHODS

INTRODUCTION: MULTIRESIDUE METHOD TESTING

Use of any multiresidue method (MRM) is supported by available information about how potential residues behave through the steps of the method. To provide that support for PAM I MRMs, additional chemicals are continually tested through the method steps and the resulting data compiled in a single database. All PAM I tables in Chapters 3 and 4 and Appendix I are produced from that database. This Appendix provides directions for performing such tests and forms for reporting results.

The effort spent on the testing of MRMs and compilation of results is justified by the advantages such compilations offer the analytical chemist. When analytical behavior data for numerous chemicals through the method in use are known, the analyst is better equipped to identify residues that may be present in a sample of unknown treatment history. In situations where the likelihood of some particular residue is known, the data lists for several methods can be consulted to help choose which method should be used.

Regulatory agencies often must assess the incidence of residue occurrence. This effort is also assisted by compilations of method behavior data. The absence of many chemicals from the sample can be ascertained when it is known that those chemicals could have been detected had they been present.

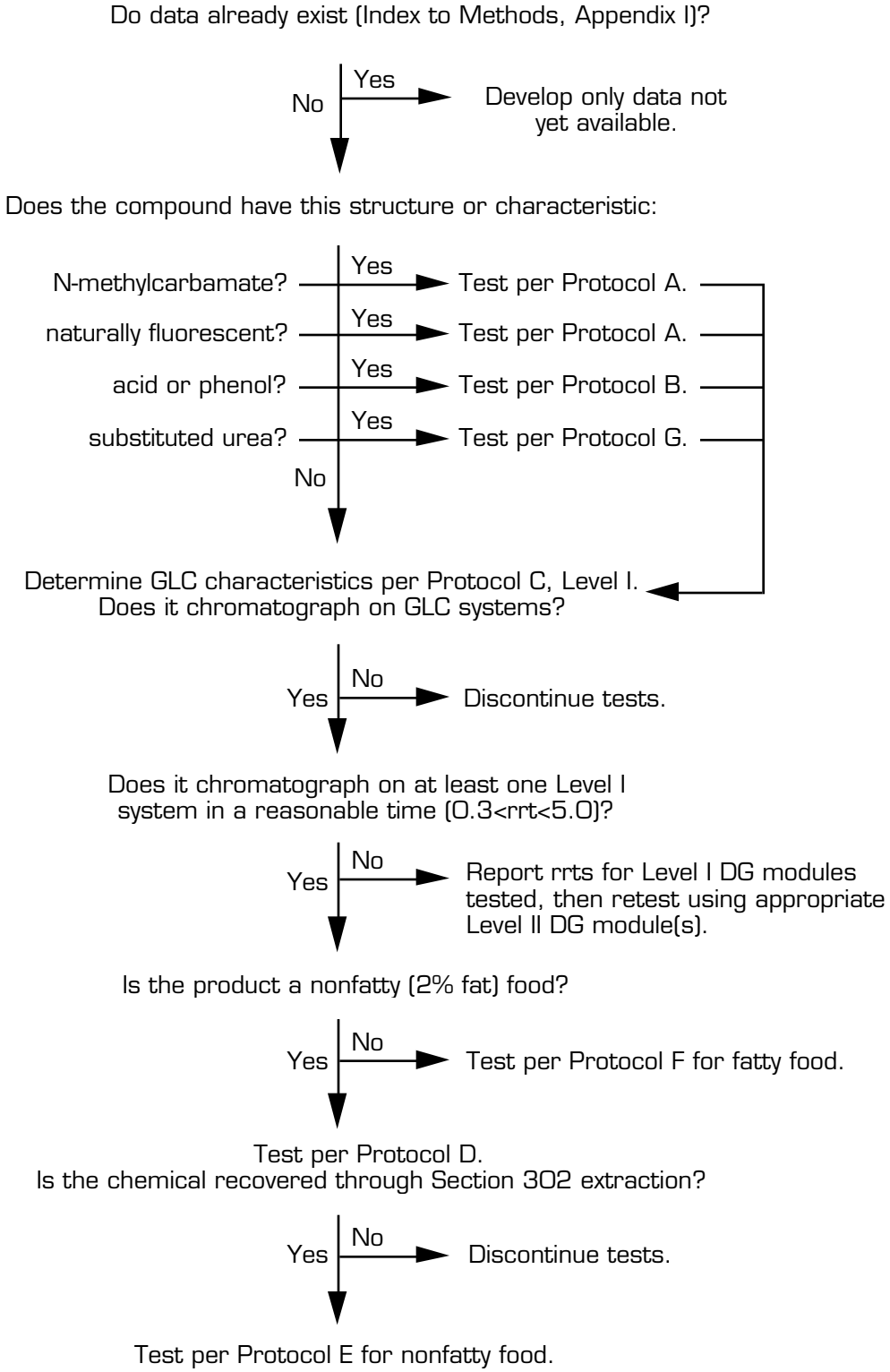
It has been found advisable to define protocols for developing data on MRM behavior. In order to compile data into usable formats, it is imperative that all contributing laboratories perform the tests uniformly. The goal of this method-testing is not to find the optimum conditions for the one chemical currently being tested, but to be able to describe how the chemical will behave when determined by the precisely defined method.

This Appendix includes one protocol for determining GLC characteristics of chemicals and six protocols for testing their behavior through individual MRMs. Forms for reporting the results of testing by each protocol are also included. Each protocol references the PAM I method(s) involved, the types of chemicals to which it applies, and the PAM I table(s) in which previously collected data are published.

Some PAM I MRMs are applicable to a wide variety of residues, while others are targeted to those with specific chemical structures. A Decision Tree is included in this Appendix to direct the user to the most appropriate protocol(s) for each chemical being tested. Follow the Decision Tree in deciding which protocol(s) to use.

Follow the steps of these protocols, in the order written. Report data on a copy of the appropriate form and send it to: PAM I Editors, HFS-337, Food and Drug Administration, 200 C Street SW, Washington, DC 20204.

Decision Tree for MRM Testing



SUGGESTIONS FOR PRODUCING QUALITY DATA

The following suggestions were developed in response to data received and to specific questions that have been raised.

Decisions on What Protocols to Follow

- As directed in the first step of the Decision Tree, review existing data on the chemical before performing experiments. Examine PAM I Index to Methods for entries, then review the details in appropriate Chapter 3 or 4 table(s); review Appendix I for available GLC data. If these sources reveal gaps in the data, perform the tests necessary to provide missing data, but do not repeat experiments unless specifically asked to do so or unless current data reflect variability.

- Develop GLC data (Protocol C) with system(s) likely to detect the chemical; *i.e.*, of the DG modules listed, choose at least one whose detector is selective to elements in the molecule. The electron capture detector may not be suitable to examine uncleaned extracts from Section 302 (Protocol D), so DG1, 13, and 18 are often insufficient. If an electron capture detector is the only one that responds to the chemical, apply caution when injecting extracts from Section 302.

- Choose the GLC system that provides the best chromatography and sensitivity for examining solutions from recovery studies; it is not necessary to re-examine the extracts by multiple GLC systems.

- The Decision Tree provides basic criteria for making decisions about which methods to test. Where situations exist that make testing or continuation of testing illogical, suspend testing and report the reasons. Examples:

  – If previous studies with radiolabelled chemical clearly show that the residue will partition into the (discarded) water layer of the method, method recovery tests need not be run; however, collection of GLC data should still be attempted.

  – If the only commodity of interest is fatty, do not attempt to perform tests on the product with methods designed for nonfatty foods. If the commodity is meat, use only Section 304 E1 (Protocol F) or, if the chemical is an acid or phenol, Section 402 E1 (Protocol B).

  – Suspend testing if the GLC tests (Protocol C) indicate that even the most sensitive GLC system is insensitive to the compound. As a general rule, suspend testing if the minimum weight of compound that causes 10% full scale deflection (FSD) is equivalent to ≥10 times the tolerance for an injection of the normal mg sample equivalent described in the method.

  – If the method being tested proves not amenable to analysis of a particular commodity, as evidenced by severe emulsions, failure to form distinct phases, *etc.*, suspend testing.

Proper Application of Methods

- Do not combine a GLC column that contains cyano groups with a nitrogen-selective detector.

- Adjust GLC column temperature to establish the correct relative retention time (rrt) of the marker chemical(s). Absolute retention times (min) are provided in DG modules to provide an indication of normal behavior, but the temperature should not be adjusted to achieve this.

- Measure retention times on GLC columns from the solvent front wherever possible for both the compound being tested and the marker compound. If the instrumentation in use precludes measurement from the solvent front, state this fact in the report.

- When submitting chromatograms with reporting forms, label them clearly: indicate how many nanograms (ng) or picograms (pg) are represented by the peak. Do not label a chromatogram of a standard in ppm. For chromatograms that result from recovery studies, indicate on the chromatogram label how much sample weight equivalent (mg) was injected.

- For accurate quantitation, detector response to the reference standard should be within ±25% of the response to the analyte in the sample, based on observable (on-scale) peak heights. Usual GLC linearity does not support quantitation that compares responses differing in size by more than that amount.

- Make sure that chromatograms do not overlap; allow peaks from one injection to elute before the next injection is made.

- When the chromatogram contains multiple peaks representing different components in the standard, report the rrt of each peak >5% FSD.

- It is not necessary to evaporate the solvent from the fortification solution once it has been added to the sample, unless there is some reason to expect it to interfere with the analysis. It is preferable for the fortifying solution to be made from the same solvent used to extract the sample.

- Use only the 60/100 mesh PR grade Florisil specified by Sections 303 and 304 (Protocols E and F). Other grades of commercially available Florisil are likely to result in different elution patterns.

- During tests of elution from Florisil, do not add reference material to the Florisil column in a polar solvent, which will affect elution pattern.

- Test Florisil elution by both ethyl ether/petroleum ether (Sections 303 C1, 304 C1 and C3) and methylene chloride (303 C2, 304 C2 and C4) elution systems when following the steps of Protocols E and F.

- Even when Florisil elution tests with reference standards indicate no elution with later eluants, examine by GLC all the Florisil eluates of the recovery test from the fortified sample. Sometimes the presence of extract changes the Florisil elution pattern.

PROTOCOL A: PROCEDURE FOR TESTING CHEMICALS THROUGH SECTION 401

BACKGROUND

Methods: Section 401 E1 + C1 + DL1 or DL2

Chemical Type: Applicable to chemicals with N-methylcarbamate structure (DL1) and to some chemicals that are naturally fluorescent (DL2).

Commodity Type: Applicable to nonfatty foods and to certain fatty foods such as soybeans and nuts.

PAM I Tables: Tables 401-a, 401-b

DATA DEVELOPMENT

HPLC Analytical Behavior

N-Methylcarbamates: Set up HPLC with post-column fluorescence labeling and fluorescence detector, as described in Section 401 DL1; check for proper operation using system suitability test described.

Fluorescent Pesticides: Set up HPLC and fluorescence detector, as described in Section 401 DL2.

Develop information on test chemical(s) as follows:

- Dissolve reference standard in methanol to prepare stock solutions. Dilute with methanol for HPLC working standards.
- Determine amount (ng) of chemical that causes detector response of 50% full scale deflection (FSD) on recorder or printer/plotter. Note peak shape of response to determine adequacy of chromatography.
- Determine linear response range of detector to chemical.
- Determine stability of chemical in methanol:
  - Short term. Prepare 1 μg/mL methanol solution of chemical. Use actinic glassware. Inject 5-10 μL injections of this solution into HPLC system over 8 hr to measure short term stability of chemical in solution. Report results as peak height (mm) response.
  - Long term. Using same solution as above, inject 10 μL into system once a day for 1-2 wk to measure long term stability of solution. Store solution on laboratory bench during day and in refrigerator overnight. Report results as peak height (mm) response to test chemical, normalized to peak height for carbofuran standard injected on same day.

- Calculate retention time of chemical relative to carbofuran on the HPLC system.

Recovery of Chemical Through Cleanup Column

Prepare fortification solution by diluting stock solution with methanol.

- Initially determine that charcoal/silanized Celite column has proper elution characteristics as described in Section 401 C1.

- In duplicate, add 25 μg pesticide to newly prepared charcoal/silanized Celite column. Then elute as described in method. After collection of eluate (20 mL methylene chloride + 125 mL toluene/acetonitrile) in round-bottom (r-b) flask, momentarily stop flow, remove bottom flask and replace with second r-b flask. Elute column with additional 100 mL toluene/acetonitrile. Evaporate solvents in both flasks to dryness as described in method. Dissolve residue in first flask to appropriate volume with appropriate solvent. Dissolve residue in second flask with 5 mL solvent. Determine percentage of total added pesticide eluted in each eluate.

- Continue recovery studies with food products only if combined recoveries from charcoal column are >50%. If ≥10% of pesticide elutes in second flask, collect separate additional 100 mL eluting solution in recovery studies with food products.

Recovery Through Complete Method

- Select representative food sample. Analyze by Section 401 to ensure that there are no interferences with chemical being tested. Simultaneously, analyze reagent blank for further information on source of possible interferences.

- Fortify duplicate 150 g portions of chopped food product, while it is in homogenizer, at about 0.05 ppm; analyze as above.

- Fortify duplicate 150 g samples at level near tolerance or, if no tolerance exists, at about 0.25 ppm; analyze as above.

REPORTING RESULTS

Report all results on copy of Reporting Form A. An asterisk (*) appears on form wherever name of tested chemical should be entered.

REPORTING FORM A: BEHAVIOR THROUGH SECTION 401

The following data resulted from testing the chemical * through PAM I Section 401 E1 + C1 + DL1 or DL2, according to Appendix II, Protocol A.

Name: *

Alternative Names:

Reference Standard (source and number):

Molecular Formula:

Structure:

Comments:

Results of HPLC Tests

The following HPLC system was used:

Analytical Column:
Guard Column:
Mobile Phase:

For natural fluorescence, DL2:

* fluoresces at excitation and emission wavelengths of _____ and _____ nm, respectively.

Peak Characteristics:

	DL1	DL2
Peak shape	_____	_____
Retention time (relative to carbofuran)	_____	_____
ng causing 50% FSD	_____	_____
Linear range	_____	_____

Results of Stability in Methanol Studies

Short Term Study		Long Term Study	
Time	Peak Ht (mm)	Day	Peak Ht (mm)
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____

Recovery Through Charcoal/Silanized Celite Column

µg * added to column: _____

Percent recovered from charcoal/silanized Celite column:

Methylene chloride + toluene/acetonitrile		Additional 100 mL toluene/acetonitrile	
Trial 1	Trial 2	Trial 1	Trial 2
_____	_____	_____	_____

Recovery Through Complete Method

Food sample:

Does sample extract cause any interference with test chemical?

Does reagent blank cause any interference with test chemical?

Determinative step used for recovery test:

Duplicate 150 g samples fortified at _____ ppm and _____ ppm.

Percent * recovered:

_____ ppm		_____ ppm	
Trial 1	Trial 2	Trial 1	Trial 2
_____	_____	_____	_____

Additional Data on Crop Used as Samples

Pesticide residues found:

Unidentified peaks (specify determinative step used and list peaks by retention time relative to appropriate chemical).

Information submitted by:

Address:

Phone: ( )

Date:

PROTOCOL B: PROCEDURE FOR TESTING CHEMICALS THROUGH SECTION 402

BACKGROUND

Method: Section 402

Chemical Type: Applicable to chemicals with acid or phenol structure. Chemicals are methylated before determination by GLC, because esters and ethers are more easily chromatographed than acids and phenols.

Commodity Type: Applicable to wide variety of commodities, both fatty and nonfatty. Different extraction steps are used depending on commodity.

PAM I Tables: Table 402-a

DATA DEVELOPMENT

Gas Chromatography

Because the method being tested includes methylation of the analyte, GLC characteristics of both the acid/phenol and its methylation product are collected.

Perform the following operations:

- Dissolve methyl ester/ether reference standard, if available, in 10% acetone/isooctane (v/v) to prepare stock standard solution. Dilute with isooctane.
- Follow directions in Protocol C for chromatography of methyl ester/ether reference standard. Report results on Reporting Form C.
- Stop work if methyl ester/ether does not cause response on any detector when chromatographed on any appropriate column.
- Dissolve acid/phenol reference standard in acetone to prepare stock standard solution. Dilute as needed with acetone. Also dilute with 50% methylene chloride/hexane to prepare solution suitable for testing recovery through gel permeation chromatography (GPC). (Note: 2,4,5-T will not dissolve in 50% methylene chloride/hexane, so solutions in that solvent mixture must be prepared by diluting acetone stock solution.)
- Follow directions in Protocol C for chromatography of acid/phenol reference standard. Report results on separate copy of Reporting Form C.
- Methylate 100 µg acid/phenol reference standard in acetone solution according to procedure described in Section 402 C1b. Dilute as needed with hexane. If any other methylation procedure is used, describe procedure on Reporting Form B.
- Follow directions in Protocol C for chromatography of methylated reference standard. Report results on separate copy of Reporting Form C (Note: if ester/ether reference standard is available, these results should

verify retention times and response characteristics previously found. Note this on Reporting Form prepared for methyl ether/ester reference standard test results.)

- Determine efficiency of methylation by direct comparison to methyl ester/ether reference standard, if available. Report percentage conversion to methylated product on Reporting Form B. If methyl ester/ether reference standard is not available, assume complete methylation of chemical for calculating amount of reference standard causing 50% FSD.

Stop work if acid/phenol cannot be methylated or if there is no GLC response to ester/ether.

Recovery Through GPC and Florisil

Methyl Ester/Ether Reference Standards

- In duplicate, place 1-100 μg methyl ester/ether reference standard, in 1-10 mL hexane, on Florisil column prepared as described in Section 402 C1c. (Use Florisil that has been shown to permit elution of both heptachlor epoxide and endrin by eluant 2 [Section 204].)

- Elute columns with 35 mL eluant 1, 60 mL eluant 2, and 100 mL ethyl ether, and determine percentage recovered in each eluate.

- If total recovery is <30%, report results on Reporting Form B and terminate work.

Acid/Phenol Reference Standards

- Place 100 μg acid/phenol reference standard, dissolved in 50% methylene chloride/hexane, on calibrated GPC column and elute as directed in Section 402 C1a.

- Methylate collected fraction according to C1b.

- Determine percentage recovery by comparison to methyl ester/ether reference standard, if available, or to reference standard previously methylated in laboratory.

- If recoveries are <30%, report results on Reporting Form B and terminate work.

Recovery Through Complete Method

- Select one representative fatty and one nonfatty food. Analyze by Section 402, using extraction module appropriate to commodity, to ensure that there are no interferences with chemical being tested. Simultaneously, analyze reagent blank for further information on source of possible interferences.

- Fortify duplicate portions of food samples with 1-2 mL acetone solution of acid/phenol reference standard at tolerance level or, if no tolerance exists, at 0.05 ppm.

- Fortify duplicate portions of food samples with 1-2 mL acetone solution of acid/phenol reference standard at 10 times tolerance levels or, if no tolerance exists, at 0.5 ppm.

- Perform complete analysis as described in Section 402, using extraction module appropriate to commodity. Calculate percentage recovered against methyl ester/ether reference standard, if available, or reference standard previously methylated in laboratory, if necessary.

REPORTING RESULTS

Report all results on copy of Reporting Form B. An asterisk (*) appears on form wherever name of tested chemical should be entered.

REPORTING FORM B: BEHAVIOR THROUGH SECTION 402

The following data resulted from testing the chemical * through PAM I Section 402, according to Appendix II, Protocol B.

GLC characteristics of this chemical have been reported on Form C for both the acid/phenol and the methyl ester/ether. The ester/ether was:

___ available as reference standard

___ prepared by methylating the acid/phenol

Name: *

Reference Standard (source and number):

Acid/phenol:

Methyl ester/ether:

Methylation

Acid/phenol reference standard dissolved in:

µg * methylated:

Methylation procedure used:

Percent conversion to methyl ester/ether, as measured against methyl ester/ether reference standard: _____

Comments:

Recovery Through GPC

µg acid/phenol reference standard (in 50% methylene chloride/hexane) added to column: ____

Percent found in collect fraction: _____

Recovery Through Florisil

µg methyl ester/ether of * added to column: _____

Percent eluted with eluant 1, eluant 2, and 100 mL ethyl ether:

eluant 1 _____

eluant 2 _____

100 mL ethyl ether _____

Recovery Through Complete Method

Method for Fatty Foods

Fatty food sample:

Does sample extract cause any interference with test chemical?

Does reagent blank cause any interference with test chemical?

Duplicate 100 g samples fortified at _____ ppm and _____ ppm.

Percent recovered using eluant 1, eluant 2, and 100 mL ethyl ether:

	_____ ppm		_____ ppm	
	Trial 1	Trial 2	Trial 1	Trial 2
eluant 1	_____	_____	_____	_____
eluant 2	_____	_____	_____	_____
100 mL ethyl ether	_____	_____	_____	_____

Method for Nonfatty Foods

Nonfatty food sample:

Does sample extract cause any interference with test chemical?

Does reagent blank cause any interference with test chemical?

Duplicate 100 g samples fortified at _____ ppm and _____ ppm.

Percent recovered using eluant 1, eluant 2, and 100 mL ethyl ether:

	_____ ppm		_____ ppm	
	Trial 1	Trial 2	Trial 1	Trial 2
eluant 1	_____	_____	_____	_____
eluant 2	_____	_____	_____	_____
100 mL ethyl ether	_____	_____	_____	_____

Information submitted by:

Address:

Phone: ( )

Date:

PROTOCOL C: PROCEDURE FOR DEVELOPING GLC DATA

BACKGROUND

Methods: Section 302 DG1-DG23; GLC systems are used with Sections 302, 303, 304, and 402 methods.

Chemical Type: Applicable to chemicals that can be vaporized at temperatures about 250° C without degradation. Most pesticides and their related chemicals that meet this criterion can be chromatographed and detected by at least one of the GLC systems DG1-DG23.

PAM I Tables: Appendix I (PESTDATA)

DATA DEVELOPMENT

For each GLC DG module tested:

- Dissolve reference standard in pesticide grade solvent to prepare stock standard solution. Isooctane is preferred, but acetone may be required for dissolution.
- Set up GLC system as described in specified DG module (Section 302). Check rrts of marker compounds and adjust column temperature to match conditions specified.
- Inject aliquots of test solution into GLC.
- Calculate retention time (relative to marker compound specified in DG module).
- Calculate ng standard that causes 50% FSD response. Do not inject >1000 ng (1 μg).
- Test chemical on one or more of these systems:

  Level I:

  All chemicals:

  DG 1 100% methyl siloxane (*e.g.*, DB-1), 200° C, EC

  DG13 50% phenyl, 50% methyl siloxane(*e.g.*, DB-17), 200° C, EC

  DG18 50% cyanopropylphenyl, 50% methyl siloxane (*e.g.*, DB-225), 200° C, EC

  Chemicals containing halogen:

  DG 3 100% methyl siloxane (*e.g.*, DB-1), 200° C, ElCD-X

  DG16 50% phenyl, 50% methyl siloxane (*e.g.*, DB-17), 200° C, ElCD-X

Chemicals containing phosphorus:

DG 2 100% methyl siloxane (*e.g.*, DB-1), 200° C, FPD-P

DG14 50% phenyl, 50% methyl siloxane (*e.g.*, DB-17), 200° C, FPD-P

DG19 50% cyanopropylphenyl, 50% methyl siloxane (*e.g.*, DB-225), 200° C, FPD-P

▶

Chemicals containing sulfur:

DG15 50% phenyl, 50% methyl siloxane (*e.g.*, DB-17), 200° C, FPD-S

▶

Chemicals containing nitrogen:

DG 4 100% methyl siloxane (*e.g.*, DB-1), 200° C, ElCD-N

DG 5 100% methyl siloxane (*e.g.*, DB-1), 200° C, N/P

DG17 50% phenyl, 50% methyl siloxane (*e.g.*, DB-17), 200° C, N/P

▶

Chemicals with no heteroatom to which element-selective detectors respond:

DG 6 100% methyl siloxane (*e.g.*, DB-1), 130° C, FID

Level II:

If chemical chromatographs on system described in module(s) of Level I, but rrt is <0.3, rechromatograph at lower column temperature, *e.g.*:

DG 7 100% methyl siloxane (*e.g.*, DB-1), 130° C, EC

DG 8 100% methyl siloxane (*e.g.*, DB-1), 130° C, FPD-P

DG 9 100% methyl siloxane (*e.g.*, DB-1), 130° C, ElCD-X

If chemical chromatographs on system described in module(s) of Level I, but rrt is >5.0, rechromatograph at higher column temperature, *e.g.*:

DG10 100% methyl siloxane (*e.g.*, DB-1), 230° C, EC

DG11 100% methyl siloxane (*e.g.*, DB-1), 230° C, FPD-P

DG12 100% methyl siloxane (*e.g.*, DB-1), 230° C, ElCD-X

REPORTING RESULTS

Report results for each DG module on copy of Reporting Form C. An asterisk (*) appears on form wherever name of tested chemical should be entered.

REPORTING FORM C: GLC DATA

The following GLC data resulted from testing the chemical * on systems described in PAM I Section 302 DG modules, according to directions in Appendix II, Protocol C.

Name: *

Alternative Names:

Reference Standard (source and number):

Molecular Formula:

Structure:

Comments:

Results for DG module (Section 302): DG ____

Standard reference material dissolved in

Brief details about GLC system used:

Column:

- Length:
- id:
- Film thickness:
- Carrier gas:
- Flow rate:
- Makeup gas:
- Flow rate:
- Retention time (relative to _____) of _____ (marker compound): ◀

Detector:

- Temperature:
- Other conditions:
- Detector response to ____ ng _____ (marker compound):_____% FSD

Behavior of * :

- Retention time (relative to _____):
- ng required for 50% FSD:

Information submitted by:

Address:

Phone: ( )

Date:

PROTOCOL D: PROCEDURE FOR TESTING CHEMICALS THROUGH SECTION 302 E1, E2

BACKGROUND

Methods: Section 302 E1, E2; use determinative step found useful for chemical in Protocol C and considered suitable for extract according to Section 302 recommendations.

Chemical Type: Applicable to nonionic pesticides; detection of particular chemicals is dependent on determinative step(s) used to examine extract.

Commodity Type: Applicable to all nonfatty foods, although some commodities contain co-extractives that interfere with some determinative steps.

PAM I Tables: Table 302-a, Appendix I (PESTDATA)

DATA DEVELOPMENT

Prepare fortification solution by diluting stock solution with acetone.

Recovery Through Complete Method Without Cleanup

- Select representative nonfatty food. Analyze by Section 302 to ensure that there are no interferences with chemical being tested. Simultaneously, analyze reagent blank for further information on source of possible interferences.

- Fortify duplicate 100 g portions at 0.05-0.1 ppm. Analyze duplicate fortified samples as described in Section 302 E1 or E2.

- Fortify duplicate 100 g portions at tolerance level for chemical or, if no tolerance exists, at five times level used in first fortification. Analyze duplicate fortified samples.

Recovery Through Florisil

If the chemical being tested can only be determined by electron capture detector, or if that is the only detector available, then the extract must be cleaned up by Florisil chromatography (Section 302 C5 or C1) before determination.

- To determine recovery through Florisil column, use only Florisil that has been shown to permit elution of heptachlor epoxide in 6% ethyl ether/petroleum ether or methylene chloride eluate 2 and elution of endrin in 15% ethyl ether/petroleum ether or eluate 2 (Section 204).

- Add 10-100 μg analyte in 1-10 mL solution to each of two Florisil columns. Elute duplicate columns according to directions of Section 302 C1 or C5. If recovery is <30%, report results, then terminate work.

Recovery Through Complete Method with Florisil Cleanup

- Select representative nonfatty food sample. Analyze by Section 302, with cleanup step being studied, to ensure that there are no interferences with chemical being tested. Simultaneously, analyze reagent blank for further information on source of possible interferences.

- Fortify duplicate100 g portions at about 0.05 ppm and analyze with method of Section 302, using E1 or E2 with cleanup C1 or C5.

- Fortify duplicate 100 g portions at tolerance level or, if no tolerance exists, at about 0.5 ppm; analyze as above.

REPORTING RESULTS

Report all results on copy of Reporting Form D. An asterisk (*) appears on form wherever name of tested chemical should be entered.

REPORTING FORM D: BEHAVIOR THROUGH SECTION 302

The following data resulted from testing the chemical * through PAM I Section 302 E1/E2 (without cleanup and/or with cleanup C1 or C5), according to Appendix II, Protocol D. GLC characteristics of this chemical have been reported on Reporting Form C.

Name: *

Reference Standard (source and number):

Recovery Through Method Without Cleanup

Nonfatty food sample:

Does sample extract cause any interference with test chemical?

Does reagent blank cause any interference with test chemical?

Duplicate 100 g sample fortified at _____ ppm and _____ ppm.

Percent * recovered:

_____ ppm		_____ ppm	
Trial 1	Trial 2	Trial 1	Trial 2
_____	_____	_____	_____

(Optional) Recovery Through Method with Florisil Column Cleanup

Recovery Through Florisil

Cleanup step tested: Section 302 C _____

µg * added to column: _____

Percent eluted with C1 eluant (50 mL 50% methylene chloride/1.5% acetonitrile/ 48.5% hexane):

_____

Percent eluted with C5 eluants:

200 mL 15% ethyl ether/petroleum ether (EE/PE) _____

200 mL 50% EE/PE _____

Recovery Through Complete Method

Nonfatty food sample:

Does sample extract cause any interference with test chemical?

Does reagent blank cause any interference with test chemical?

Duplicate _____ g sample fortified at _____ ppm and _____ ppm.

Percent recovered using C1 eluant:

	_____ ppm		_____ ppm	
	Trial 1	Trial 2	Trial 1	Trial 2
	_____	_____	_____	_____

Percent recovered using C5 eluants:

	_____ ppm		_____ ppm	
	Trial 1	Trial 2	Trial 1	Trial 2
15% EE/PE	_____	_____	_____	_____
50% EE/PE	_____	_____	_____	_____

Information submitted by:

Address:

Phone: ( )

Date:

PROTOCOL E: PROCEDURE FOR TESTING CHEMICALS THROUGH SECTION 303

BACKGROUND

Methods: Section 303 E1-E5 + C1 and C2

Chemical Type: Generally applicable to relatively nonpolar chemicals, although many chemicals are recovered through this method. Do not assume too readily that a chemical is too polar to be recovered.

Commodity Type: Applicable to nonfatty foods; with special extractions for samples of low (<75%) moisture foods, high (5-15%) and very high (15-30%) sugar foods, and eggs.

PAM I Tables: Table 303-a, Appendix I (PESTDATA)

DATA DEVELOPMENT

Prepare fortification solution by diluting stock solution with petroleum ether or hexane.

Recovery Through Cleanup Column

- To determine recovery through Florisil column, use only Florisil that has been shown to permit elution of heptachlor epoxide in 6% ethyl ether/petroleum ether or methylene chloride eluate 2 and endrin in 15% ethyl ether/petroleum ether or eluate 2 (Section 204).

- Add 10-100 µg analyte in 1-10 mL solution to each of four Florisil columns. Elute duplicate columns according to directions of Section 303 C1 and C2, respectively. If recoveries through both elution systems are <30%, report results, then terminate work.

Recovery Through Complete Method

- Select representative nonfatty food sample. Analyze by Section 303 to ensure that there are no interferences with chemical being tested. Simultaneously, analyze reagent blank for further information on source of possible interferences.

- Fortify duplicate 100 g portions at about 0.05 ppm and analyze with method of Section 303, using extraction module appropriate to commodity and cleanup C1.

- Fortify duplicate 100 g portions at tolerance level or, if no tolerance exists, at about 0.5 ppm; analyze as above.

- If chemical is recovered through method, and if it was previously found to be eluted from Florisil using methylene chloride eluants of C2, then repeat recovery experiments through whole method using extraction module appropriate to commodity and cleanup C2.

REPORTING RESULTS

Report all results on copy of Reporting Form E. An asterisk (*) appears on form wherever name of tested chemical should be entered.

REPORTING FORM E: BEHAVIOR THROUGH SECTION 303

The following data resulted from testing the chemical * through PAM I Section 303 E1-E5 + C1 and C2, according to Appendix II, Protocol E. GLC characteristics have been reported on Reporting Form C.

Name: *

Reference Standard (source and number):

Recovery Through Florisil

µg * added to column: _____

Percent eluted with C1 eluants:

6% _____
15% _____
50% _____

Percent eluted with C2 eluants:

1 _____
2 _____
3 _____

Recovery Through Complete Method

Nonfatty food sample: Extraction used: 303 E ___

Does sample extract cause any interference with test chemical?

Does reagent blank cause any interference with test chemical?

Duplicate _____ g sample fortified at _____ ppm and _____ ppm.

Percent recovered using C1 eluants:

	_____ ppm		_____ ppm	
	Trial 1	Trial 2	Trial 1	Trial 2
6%	_____	_____	_____	_____
15%	_____	_____	_____	_____
50%	_____	_____	_____	_____

Percent recovered using C2 eluants:

	_____ ppm		_____ ppm	
	Trial 1	Trial 2	Trial 1	Trial 2
1	_____	_____	_____	_____
2	_____	_____	_____	_____
3	_____	_____	_____	_____

Information submitted by:

Address:

Phone: ( )

Date:

PROTOCOL F: PROCEDURE FOR TESTING CHEMICALS THROUGH SECTION 304

BACKGROUND

Methods: Section 304 E1-E5 + C1-C4

Chemical Type: Generally applicable to relatively nonpolar chemicals, although many chemicals can be recovered through this method. Do not assume too readily that a chemical is too polar to be recovered. Very nonpolar chemicals may be only partially recovered through acetonitrile/petroleum ether partitioning steps of C1-C4.

Commodity Type: Applicable to fatty foods, with special extraction steps for removal of fat from different fatty commodities.

PAM I Tables: Table 304-a, Appendix I (PESTDATA)

DATA DEVELOPMENT

Prepare fortification solution by diluting stock solution with petroleum ether or hexane.

Recovery Through Cleanup Column

- To determine recovery through Florisil column, use only Florisil that has been shown to permit elution of heptachlor epoxide in 6% ethyl ether/petroleum ether or eluate 2 and endrin in 15% ethyl ether/petroleum ether or eluate 2 (Section 204).

- Add 10-100 µg analyte in 1-10 mL solution to each of four Florisil columns. Elute duplicate columns according to directions of Section 304 C1 and C2, respectively. If recoveries through both elution systems are <30%, report results, then terminate work.

- If chemical elutes in 6% ethyl ether/petroleum ether (C1) or eluate 1 (C2), rerun Florisil column experiment (duplicates) and elute according to directions of Section 304 C3 and/or C4, *i.e.*, elute with 250 mL petroleum ether before elution with first eluant of each system.

Recovery Through Complete Method

- Select one representative fatty food sample. Analyze by Section 304 to ensure that there are no interferences with chemical being tested. Simultaneously, analyze reagent blank for further information on source of possible interferences.

- Fortify duplicate 100 g portions at about 0.05 ppm and analyze with method of Section 304, using extraction module appropriate to the commodity and cleanup C1.

- Fortify duplicate 100 g portions at tolerance level or, if no tolerance exists, as about 0.5 ppm; analyze as above.

- If chemical is recovered through method, and if it was previously found to be eluted from Florisil using methylene chloride eluants of C2, then repeat recovery experiments through whole method using extraction module appropriate to commodity and cleanup C2.

REPORTING RESULTS

Report all results on copy of Reporting Form F. An asterisk (*) appears on form wherever name of tested chemical should be entered.

REPORTING FORM F: BEHAVIOR THROUGH SECTION 304

The following data resulted from testing the chemical * through PAM I Section 304 E1-E5 + C1-C4, according to Appendix II, Protocol F. GLC characteristics have been reported on Reporting Form C.

Name: *

Reference Standard (source and number):

Recovery Through Florisil

µg * added to column: _____

Percent eluted with C1 eluants:

6% _____
15% _____
50% _____

Percent eluted with C2 eluants:

1 _____
2 _____
3 _____

For chemicals that elute in 6% ethyl ether/petroleum ether (C1) or Eluant 1 (C2) only:

µg * added to column: _____

Percent eluted with C3 and C4 eluants:

PE _____
6% _____

PE _____
1% _____

Recovery Through Complete Method

Fatty food sample: Extraction used: 304 E ___

Does sample extract cause any interference with test chemical?

Does reagent blank cause any interference with test chemical?

Duplicate _____ g sample fortified at _____ ppm and _____ ppm.

Percent recovered using C1 eluants:

	_____ ppm		_____ ppm	
	Trial 1	Trial 2	Trial 1	Trial 2
6%	_____	_____	_____	_____
15%	_____	_____	_____	_____
50%	_____	_____	_____	_____

Percent recovered using C2 eluants:

	_____ ppm		_____ ppm	
	Trial 1	Trial 2	Trial 1	Trial 2
1	_____	_____	_____	_____
2	_____	_____	_____	_____
3	_____	_____	_____	_____

Information submitted by:

Address:

Phone: ( )

Date:

PROTOCOL G: PROCEDURE FOR TESTING CHEMICALS THROUGH SECTION 403

BACKGROUND

Methods: Section 403 E1 + C1 + DL3

Chemical Type: Applicable to chemicals with phenylurea structure; it may also be applicable to ureas substituted with other constituents that can be degraded photolytically to form primary amines.

Commodity Type: Applicable to nonfatty foods.

PAM I Tables: Table 403-a

DATA DEVELOPMENT

HPLC Analytical Behavior

Phenylureas: Set up HPLC with post-column photodegradation, derivatization, and fluorescence detection as described in Section 403 DL3. Check for proper operation using system suitability test described.

Develop information on test chemical(s) as follows:

- Dissolve reference standard in methanol to prepare 1 mg/mL stock solution. Dilute with methanol for HPLC working standards. Dilute with methylene chloride to 20 μg/mL, then dilute 1 mL to 5 mL, to perform recovery through Florisil column.
- Determine amount (ng) of chemical that causes detector response of 50% full scale deflection (FSD) on recorder or printer/plotter. Note peak shape and measure asymmetry (Section 602 C).
- Determine linear response range of detector to chemical.
- Calculate retention time of chemical relative to diuron on HPLC system.

Recovery of Chemical Through Florisil Cleanup Column

- Prepare duplicate Florisil columns as directed in method (Section 403 C1).
- After washing columns with 30 mL methylene chloride, transfer 20 μg chemical in 5 mL methylene chloride to each column.
- Elute columns as described and proceed with HPLC determination.
- Continue with recovery through entire procedure only if recovery from Florisil column is >50%.

Recovery Through Complete Method

- Select appropriate nonfatty food sample. Analyze by Section 403 to ensure that there are no interferences with chemical being tested. Simultaneously, analyze reagent blank for further information on source of possible interferences.

- Fortify duplicate 50 g portions of chopped food product, while it is in homogenizer, at about 0.05 ppm; analyze as above.

- Fortify duplicate 50 g portions at level near tolerance or, if no tolerance exists, at about 0.25 ppm; analyze as above.

REPORTING RESULTS

Report all results on copy of Reporting Form G. An asterisk (*) appears on form wherever name of tested chemical should be entered.

REPORTING FORM G: BEHAVIOR THROUGH SECTION 403

The following data resulted from testing the chemical * through PAM I Section 403 E1 + C1 + DL3, according to Appendix II, Protocol G.

Name: *

Alternative Names:

Reference Standard (source and number):

Molecular Formula:

Structure:

Comments:

Results of HPLC Tests

The following HPLC system was used:

Analytical column:
Guard column:
Mobile phase:
Detector type:
Detector excitation wavelength:
Detector emission wavelength:

Peak characteristics:

Peak shape (asymmetry):
Retention time relative to diuron:
ng causing 50% FSD response:
Linear range:

Recovery Through Florisil Column

µg * added to column: _____

Percentage recovered from Florisil column:

Trial 1	Trial 2
_____	_____

Recovery Through Complete Method

Food sample:

Does sample extract cause any interference with test chemical?

Does reagent blank cause any interference with test chemical?

Duplicate 50 g samples fortified at 0.05 ppm.

Percent recovered:

Trial 1	Trial 2
_____	_____

Duplicate 50 g samples fortified at _____ ppm.

Percent recovered:

Trial 1	Trial 2
_____	_____

Additional Data on Crop Used as Samples:

Unidentified peaks: rrt relative to diuron.

Any additional residues detected:

Information submitted by:

Address:

Phone: ( )

Date:

Pesticide Analytical Manual Volume II

Alphabetical Index

This index to Pesticide Analytical Manual Volume II (PAM II), methods for residues of individual pesticides, is alphabetical by name. Included as names are those used in Code of Federal Regulations Title 40 (40 CFR) and those treated as "preferred names" by U.S. Food and Drug Administration, with the former considered the main entry; FDA names cross-reference the 40 CFR name unless they are the same. For additional names for these and other pesticides, see *FDA Glossary of Pesticide Chemicals*, also available on the WWW site maintained by FDA's Center for Food Safety and Applied Nutrition.

Dates in this index are the transmittal dates of the PAM II published methods. Asterisks indicate that additional or new method(s) have been submitted to EPA but not compiled in PAM II. Users with a need for such methods may request copies from:

(Mail)	Mark Law US EPA/OPP/BEAD/ACB Environmental Science Center 701 Mapes Road Fort Meade, MD 20755-5350
(Telephone)	(410) 305-2915 (TTY)*
(Fax)	(410) 305-3091
(Email)	ResidueMethods@epa.gov

In this index, many names appear in different order from the previous (published) index, which was alphabetized by hand with leading numbers and other characters ignored. In this index, names are alphabetized by computer using all characters, according to the following sequence:

space ! + # $ % & + ( ) * + , - . / 0 1 2 3 4 5 6 7 8 9 : ; < = > ? @
A B C D E F G H I J K L M N O P Q R S T U V W X Y Z [\ ]

If needed to permit a long chemical name to fit on additional lines, an "=" is used where no space or hyphen occurs in the actual name at that point.

*Requestors who do not use TTY should first dial 711 to be connected with their state's Relay Service, or dial the Relay Service directly. The telephone number is in the user's local directory. The call will be answered by a live operator, to whom the requestor must give the EPA phone number above. The operator will complete the connection and assist in the conversation.

* Method(s) have been submitted to EPA but not compiled in PAM II; request copies from EPA.

Pesticide Analytical Manual Volume II

Numerical Index

This index to Pesticide Analytical Manual Volume II (PAM II), methods for residues of individual pesticides, is in order by the section of Code of Federal Regulations Title 40 (40 CFR) in which tolerances for that pesticide are listed. Parts 180 (tolerances for pesticide chemicals in or on raw agricultural commodities), 185 (food additives in food for human consumption) and 186 (feed additives permitted in animal feed) are included.

Each entry in this index also includes references to other 40 CFR parts for the chemical.

Dates in this index are the transmittal dates of the PAM II published methods. Asterisks indicate that additional or new method(s) have been submitted to EPA but not compiled in PAM II. Users with a need for such methods may request copies from:

(Mail)	Mark Law US EPA/OPP/BEAD/ACB Environmental Science Center 701 Mapes Road Fort Meade, MD 20755-5350
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Title 40, Part 180: Tolerances and exemptions from tolerances for pesticide chemicals in or on raw agricultural commodities

Sec. No.	Name	Date
180.103	captan	11/01/75*
180.106	diuron (186.2550)	07/01/67
180.108	acephate (revoked, 186.100)	10/77
180.110	maneb	07/01/67
180.111	malathion (185.3850, 185.7000, 186.3850)	11/01/75*
180.113	allethrin	07/01/67
180.114	ferbam	07/01/67
180.116	ziram	11/01/75
180.117	S-ethyl dipropylthiocarbamate	11/01/73
180.120	methoxychlor	11/01/75
180.121	parathion	11/01/75
180.121	parathion methyl homolog	11/01/75
180.127	piperonyl butoxide (185.4900, 186.4900)	11/01/75
180.128	pyrethrins (185.5200, 186.5200)	11/01/75
180.129	phenylphenol, o-	11/01/75
180.130	hydrogen cyanide (185.3600)	07/01/67
180.132	thiram	11/01/75
180.133	lindane	11/01/75
180.136	basic copper carbonate	11/01/75
180.142	2,4-D	07/01/67*
180.143	dipropyl isocinchomeronate	11/01/75
180.144	cyhexatin (185.1350, 186.1350)	06/86
180.145	fluorine compounds (185.3375)	11/01/75*
180.151	ethylene oxide (185.2850)	07/01/67
180.152	sodium dimethyldithiocarbamate	11/01/75
180.153	diazinon (185.1750, 186.1750)	11/01/75
180.154	O,O-dimethyl S-((4-oxo-1,2,3-benzotriazin-3(4H)-yl)methyl) phosphorodithioate	11/01/75
180.155	1-naphthaleneacetic acid	11/01/75
180.157	methyl 3-((dimethoxyphosphinyl)oxy)-2-butenoate	11/01/75
180.163	1,1-bis(p-chlorophenyl)-2,2,2-trichloroethanol	07/01/67
180.167	nicotine	11/01/75
180.169	carbaryl (186.550)	11/01/75
180.172	dodine	11/01/75*
180.173	ethion (185.2750, 186.2750)	07/01/67
180.174	tetradifon	07/01/67
180.175	maleic hydrazide (185.3900)	11/01/75
180.176	coordination product of zinc ion and maneb (185.6300, 186.6300)	07/01/67
180.178	ethoxyquin	11/01/75

* Method(s)have been submitted to EPA but not compiled in PAM II; request copies from EPA.

180.241	S-(O,O-diisopropyl phosphorodithioate) of N-(2-mercaptoethyl) benzenesulfonamide	11/01/73
180.242	thiabendazole (185.5550, 186.5550)	07/88
180.243	propazine	11/85
180.245	streptomycin	01/02/69
180.249	alachlor	07/01/69*
180.252	2-chloro-1-(2,4,5-trichlorophenyl) vinyl dimethyl phosphate (186.950)	07/01/69*
180.253	methomyl (185.4100)	07/01/70
180.254	carbofuran (185.600, 186.600)	08/87
180.257	chloroneb	07/01/69
180.258	ametryn	11/85
180.259	propargite (185.5000, 186.5000)	11/78
180.261	N-(mercaptomethyl)phthalimide S-(O,O-dimethyl phosphorodithioate)	007/78
180.262	ethoprop	11/01/73*
180.263	phosalone (185.4800, 186.4800)	07/01/70*
180.267	captafol	07/78
180.268	barban	07/01/70
180.269	aldicarb (185.150, 186.150)	11/84
180.272	S,S,S-tributyl phosphorotrithioate (186.5800)	07/01/70*
180.274	propanil	11/01/73
180.275	chlorothalonil	07/01/70
180.276	formetanate hydrochloride	11/01/73*
180.278	phenmedipham	12/31/74
180.287	amitraz (memo)	08/87*
180.288	2-(thiocyanomethylthio)benzothiazole	07/78
180.289	methanearsonic acid	11/01/75
180.291	pentachloronitrobenzene	11/01/73
180.292	picloram	07/78
180.293	endothall	11/01/73
180.294	benomyl (185.350, 186.350)	11/78*
180.296	dimethyl phosphate of 3-hydroxy-N-methyl-cis-crotonamide (185.2250)	12/31/74
180.297	N-1-naphthylphthalamic acid	07/01/67
180.298	methidathion	11/78*
180.299	dimethyl phosphate of 3-hydroxy-N-methyl-cis-crotonamide	12/31/74
180.300	ethephon (185.2700, 186.2700)	12/31/74*
180.301	carboxin	10/77
180.303	oxamyl (186.4575)	10/77
180.304	oryzalin (185.4550)	10/77
180.305	3,4,5-trimethylphenyl methylcarbamate and 2,3,5-trimethylphenyl methylcarbamate	12/31/74
180.307	2-((4-chloro-6-(ethylamino)-s-triazin-2-yl)amino)-2-methylpropionitrile	12/31/74
180.309	naphthaleneacetamide, alpha-	12/31/74
180.311	cacodylic acid	12/31/74
180.312	4-aminopyridine	12/31/74

180.314	S–2,3,3-trichloroallyl diisopropylthiocarbamate	12/31/74
180.315, 180.108	methamidophos	12/31/74
180.316	pyrazon	12/31/74
180.317	3,5-dichloro-N-(1,1-dimethyl-2-propynyl)benzamide	12/31/74
180.318	4-(2-methyl-4-chlorophenoxy) butyric acid	12/31/74
180.324	bromoxynil	08/87
180.325	2-(m-chlorophenoxy)propionic acid	12/31/74
180.328	N,N-diethyl-2-(1-naphthalenyloxy)propionamide	10/77
180.330	S-(2-(ethylsulfinyl)ethyl) O,O-dimethyl phosphorothioate (186.3050)	11/01/75*
180.331	4-(2,4-dichlorophenoxy)butyric acid	11/01/75
180.332	4-amino-6-(1,1-dimethylethyl)-3-(methylthio)-1,2,4-triazin-5-(4H)-one (185.250, 186.250)	11/01/75*
180.337	oxytetracycline	07/01/67
180.337	oxytetracycline hydrochloride	07/01/67
180.338	6-methyl-1,3-dithiolo[4,5-b]quinoxalin-2-one	10/77
180.339	2-methyl-4-chlorophenoxyacetic acid	11/01/75
180.341	2,4-dinitro-6-octylphenyl crotonate and 2,6-dinitro-4-octylphenyl crotonate (186.2400)	10/77
180.342	chlorpyrifos (185.1000, 186.1000)	10/77
180.344	4,6-dinitro-o-cresol	10/77
180.345	ethofumesate	05/84
180.346	oxadiazon	10/77
180.349	ethyl 3-methyl-4-(methylthio)phenyl (1-methylethyl)phosphoramidate (185.2950, 186.2950)	06/85
180.350	nitrapyrin	11/85
180.352	terbufos	10/77*
180.353	desmedipham	10/77
180.355	bentazon (186.375)	11/78
180.356	norflurazon	10/77*
180.359	methoprene (185.4150, 186.4150)	11/78
180.360	asulam	09/83
180.361	pendimethalin	10/77
180.362	hexakis(2-methyl-2-phenylpropyl)distannoxane (185.3550, 186.3550)	10/77*
180.364	glyphosate	06/86*
180.367	N-octyl bicycloheptene dicarboximide (185.4500)	10/77
180.368	metolachlor	12/82
180.369	difenzoquat methyl sulfate	10/77
180.370	5-ethoxy-3-(trichloromethyl)-1,2,4-thiadiazole	12/82
180.371	thiophanate-methyl (186.5700)	11/78*
180.372	2,6-dimethyl-4-tridecylmorpholine	12/82
180.377	diflubenzuron (186.2000)	12/82
180.378	permethrin	11/84
180.379	cyano(3-phenoxyphenyl)methyl 4-chloro-alpha-(1-methylethyl)benzeneacetate	12/82*
180.380	3-(3,5-dichlorophenyl)-5-ethenyl-5-methyl-2,4-oxazolidinedione	12/82

180.436	cyfluthrin	*
180.437	methyl 2-(4-isopropyl-4-methyl-5-oxo-2-imidazolin-2-yl)-p-toluate and methyl (±)-6-(4-isopropyl-4-methyl-5-oxo-2-imidazolin-2-yl)-m-toluate	*
180.438	cyhalothrin, lambda- (185.1310)	*
180.439	thifensulfuron methyl ester	*
180.440	tefluthrin	*
180.441	quizalofop ethyl	*
180.442	bifenthrin	*
180.443	myclobutanil (185.4350, 186.4350)	*
180.445	bensulfuron methyl ester	*
180.446	clofentezine (185.—) (several methods)	*
180.447	imazethapyr ammonium salt	09/91
180.449	Avermectin B1 (several methods)	*
180.451	tribenuron methyl ester	*
180.452	primisulfuron-methyl	*
180.454	nicosulfuron	*
180.455	procymidone (ref to PAM I)	*
180.456	oxadixyl	*
180.457	(1,1'-biphenyl)-4-yloxy-alpha-(1,1-dimethylethyl)-1H-1,2,4-triazole-1-ethanol, beta- (185.—, 186.—)	*
180.458	clethodim (186.1075)	*
180.459	triasulfuron	*
180.460	4-(dichloroacetyl)-3,4-dihydro-3-methyl-2H-1,4-benzoxazine	*
180.461	cadusafos	*
180.462	pyridate	*
180.463	3,7-dichloro-8-quinolinecarboxylic acid	*
180.464	dimethenamid	*
180.465	4-(dichloroacetyl)-1-oxa-4-azaspiro[4.5]decane	*
180.466	fenpropathrin	*
180.467	carbon disulfide	*
180.468	flumetsulam	*
180.470	acetochlor	*
180.471	3-(dichloroacetyl)-5-(2-furanyl)-2,2-dimethyloxazolidine	*
180.472	imidacloprid	*
180.473	glufosinate-ammonium	11/28/00
180.474	tebuconazole	*
180.477	flumiclorac pentyl ester (186.3325)	*
180.478	rimsulfuron	*
180.479	halosulfuron-methyl	*
180.480	fenbuconazole	*
180.482	benzoic acid, 3,5-dimethyl-1-(1,1-dimethylethyl)-2-(4-ethylbenzoyl)hydrazide	*
180.483	O-(2-(1,1-dimethylethyl)-5-pyrimidinyl) O-ethyl O-(1-methylethyl) phosphorothioate	*
180.488	hexaconazole	*

180.489	sulfosate (removed)	*
180.495	spinosad	*
180.501	ethyl (2E,4E)-3,7,11-trimethyl-2,4-dodecadienoate, (R)- and (S)-	*
180.502	aminoethoxyvinylglycine	*
180.515	carfentrazone-ethyl	*
180.516	fludioxonil	*
180.521, 180.522	methyl bromide	*
180.525	resmethrin	08/87
180.530	2,2-dimethyl-1,3-benzodioxol-4-ol methylcarbamate	*
180.533	esfenvalerate	*
180.540	O,O-dimethyl O-4-nitro-m-tolyl phosphorothioate (185.2200)	*
180.541	propetamphos (185.5100, 186.5100)	06/86
180.543	diclosulam	*
180.553	fenhexamid	*
180.554	kresoxim-methyl	*
180.555	trifloxystrobin	*

Title 40, Part 185: Food additives permitted in food for human consumption

Sec. No.	Name	Date
185.—	(1,1'-biphenyl)-4-yloxy-alpha-(1,1-dimethylethyl)-1H-1,2,4-triazole-1-ethanol, beta-	*
185.—	clofentezine (several methods)	*
185.1000	chlorpyrifos	10/77
185.1000	triclopyr	06/86
185.1050	chlorpyrifos-methyl	07/88
185.1200	copper	11/01/75
185.1310	cyhalothrin, lambda-	*
185.1350	cyhexatin	06/86
185.150	aldicarb	11/84
185.1750	diazinon	11/01/75
185.1900	2,2-dichlorovinyl dimethyl phosphate	06/85
185.2200	O,O-dimethyl O-4-nitro-m-tolyl phosphorothioate	*
185.2250	dimethyl phosphate of 3-hydroxy-N-methyl-cis-crotonamide	12/31/74
185.250	4-amino-6-(1,1-dimethylethyl)-3-(methylthio)-1,2,4-triazin-5-(4H)-one	11/01/75*
185.2500	diquat	06/01/68
185.2600	endosulfan	07/01/70
185.2700	ethephon	12/31/74*
185.2750	ethion	07/01/67
185.2850	ethylene oxide	07/01/67
185.2950	ethyl 3-methyl-4-(methylthio)phenyl (1-methylethyl)phosphoramidate	06/85
185.3250	fluazifop-butyl	06/85
185.3375	fluorine compounds	11/01/75*
185.350	benomyl	11/78*
185.3550	hexakis(2-methyl-2-phenylpropyl)distannoxane	10/77*
185.3575	hexazinone	09/83
185.3600	hydrogen cyanide	07/01/67
185.3850, 185.7000	malathion	11/01/75*
185.3900	maleic hydrazide	11/01/75
185.4000	metalaxyl	11/84*
185.4100	methomyl	07/01/70
185.4150	methoprene	11/78
185.4350	myclobutanil	*
185.4500	N-octyl bicycloheptene dicarboximide	10/77
185.4550	oryzalin	10/77
185.4800	phosalone	07/01/70*
185.4900	piperonyl butoxide	11/01/75
185.4950	pirimiphos-methyl	06/85*
185.5000	propargite	11/78
185.5100	propetamphos	06/86

185.5200	pyrethrins	11/01/75
185.5550	thiabendazole	07/88
185.5950	triforine	09/83
185.600	carbofuran	08/87
185.6300	coordination product of zinc ion and maneb	07/01/67

Title 40, Part 186: Feed additives permitted in animal feed

Sec. No.	Name	Date
186.—	(1,1'-biphenyl)-4-yloxy-alpha-(1,1-dimethylethyl)-1H-1,2,4-triazole-1-ethanol, beta-	*
186.100	acephate	10/77
186.1000	chlorpyrifos	10/77
186.1000	triclopyr	06/86
186.1050	chlorpyrifos-methyl	07/88
186.1075	clethodim	*
186.1350	cyhexatin	06/86
186.150	aldicarb	11/84
186.1750	diazinon	11/01/75
186.1950	O,O-diethyl S-(2-(ethylthio)ethyl) phosphorodithioate	07/01/70
186.2000	diflubenzuron	12/82
186.2050	dimethipin	06/85
186.2100	dimethoate	11/01/73
186.2325	dimethyl (2,2,2-trichloro-1-hydroxyethyl)phosphonate	07/01/70
186.2400	2,4-dinitro-6-octylphenyl crotonate and 2,6-dinitro-4-octylphenyl crotonate	10/77
186.2450	dioxathion	11/01/73
186.250	4-amino-6-(1,1-dimethylethyl)-3-(methylthio)-1,2,4-triazin-5-(4H)-one	11/01/75*
186.2500	diquat	06/01/68
186.2550	diuron	07/01/67
186.2700	ethephon	12/31/74*
186.2750	ethion	07/01/67
186.2950	ethyl 3-methyl-4-(methylthio)phenyl (1-methylethyl)phosphoramidate	06/85
186.3050	S-(2-(ethylsulfinyl)ethyl) O,O-dimethyl phosphorothioate	11/01/75*
186.3250	fluazifop-butyl	06/85
186.3325	flumiclorac pentyl ester	*
186.3350	fluometuron	07/01/70
186.3400	fluvalinate, (alpha RS,2R)-	09/89*
186.350	benomyl	11/78*
186.3550	hexakis(2-methyl-2-phenylpropyl)distannoxane	10/77*
186.3575	hexazinone	09/83
186.3650	imazalil	06/85
186.375	bentazon	11/78
186.3850	malathion	11/01/75*
186.4150	methoprene	11/78
186.4350	myclobutanil	*
186.450	sec-butylamine	12/31/74
186.4575	oxamyl	10/77
186.4750	phorate	11/01/73
186.4800	phosalone	07/01/70*
186.4900	piperonyl butoxide	11/01/75

186.4950	pirimiphos-methyl	06/85*
186.4975	profenofos	06/85*
186.5000	propargite	11/78
186.5100	propetamphos	06/86
186.5200	pyrethrins	11/01/75
186.5350	simazine	11/85
186.550	carbaryl	11/01/75
186.5550	thiabendazole	07/88
186.5600	thidiazuron	11/84
186.5650	thiodicarb	06/86
186.5700	thiophanate-methyl	11/78*
186.5800	S,S,S-tributyl phosphorotrithioate	07/01/70*
186.5950	triforine	09/83
186.600	carbofuran	08/87
186.6300	coordination product of zinc ion and maneb	07/1/67
186.950	2-chloro-1-(2,4,5-trichlorophenyl) vinyl dimethyl phosphate	07/1/69*

Pesticide Analytical Manual Volume I

9/96 Revisions

The following pages contain corrections or changes for PAM I. Print these pages and use them to replace the same current pages in PAM I 3rd edition (published Jan., 1994).

Each set of two pages is intended to appear on two sides of the same paper, but Acrobat Reader does not offer a feature that facilitates printing on both sides of the page. It may be necessary to print one page at a time and turn the paper over to print the second page on the reverse side.

Material Transmitted	PAM I Page	Page of this File
1. Title Page and Masthead	i, ii (not numbered on page)	2, 3
2. Introduction	v, vi	4, 5
3. Section 102, pages 3 and 4	102-3, 102-4	6, 7
4. Section 105, pages 3 and 4	105-3, 105-4	8, 9
5. Section 204, pages 7 and 8	204-7, 204-8	10, 11
6. Section 404, pages 11 and 12	404-11, 404-12	12, 13
7. Section 605, pages 5 and 6	605-5, 605-6	14, 15
8. Appendix II, Report Form C	Appendix II-17 and Appendix II-18	16, 17

Explanations of changes:

Title and masthead reflect this current revision.

Introduction includes information about the FDA web site and the electronic files now available.

Table 102-b, page 102-4, contains a new sentence to clarify that brine is considered inedible and that it and other inedible media are discarded during preparation of processed food.

Table 105-a, page 105-4, corrects a previously inaccurate limit of quantitation (Lq) for MBC.

Paragraph 5, page 204-7, corrects a previously inaccurate statement about the eluant in which malathion elutes from Florisil.

Figure 404-b, page 404-12, corrects a previously inaccurate label for thiabendazole in both “B” chromatograms.

Figure 605-c, page 605-6, corrects a previously inaccurate label for the excitation filter.

Reporting Form C, Appendix II-17, clarifies a previously confusing entry area.

PESTICIDE ANALYTICAL MANUAL

VOLUME I: Multiresidue Methods

U.S. Department of Health and Human Services • Public Health Service
Food and Drug Administration

PESTICIDE ANALYTICAL MANUAL VOLUME I

3rd Edition, 1994

Revised, September, 1996

MANAGING EDITORS

Management Methods Branch
Office of the Associate Commissioner for Management and Operations

Design and Format: R.O.W. Sciences, Inc.
under contract to U.S. Food and Drug Administration

Transmittal No. 96-1 (9/96)
Form FDA 2905a (6/92)

PESTICIDE ANALYTICAL MANUAL

INTRODUCTION

The Food and Drug Administration (FDA) is responsible under the Federal Food, Drug, and Cosmetic Act for enforcing tolerances established by the Environmental Protection Agency (EPA) for amounts of pesticide residues that may legally remain on food (including animal feed). In meeting this responsibility, FDA collects and analyzes food from commercial channels of trade for determining compliance with EPA tolerances. The residue data gathered under this regulatory monitoring program are also used for evaluating the extent and significance of pesticide residues in the food supply.

The Pesticide Analytical Manual (PAM) is published by FDA as a repository of the analytical methods used in FDA laboratories to examine food for pesticide residues for regulatory purposes.[1] The manual is organized according to the scope of the analytical methods:

Volume I contains multiresidue methods (MRMs) that are used by FDA on a routine basis, because of their efficiency and broad applicability, especially for analyzing foods of unknown pesticide treatment history.

Volume II contains methods designed for the analysis of commodities for residues of only a single compound (although some methods are capable of determining several related compounds). These methods are most often used when the likely residue is known to the chemist and/or when the residue of interest cannot be determined by common MRMs.

PAM is designed to be used by analysts experienced in trace residue analysis. All of the techniques employed are subject to potential interferences from reagents, apparatus, containers, contaminated air supply, and handling by personnel. The experienced analyst is alert for these possibilities and recognizes the need to confirm results by other techniques that measure different chemical or physical properties of the analyte.

Experienced residue analysts are aware that no report of validation in another laboratory can substitute for verification that the method does indeed work in the analyst's own laboratory. The analyst should verify method performance in each particular application by a trial of the method that includes examination of reagent and sample blanks and measurement of the recovery of added analyte. The editors invite analysts to report results of their experiences with PAM methods.

Revisions

Starting with transmittal 96-1 (9/96), revisions of PAM I will be issued in two ways: (1) changes in most manual sections will be distributed as hard (paper) copies, with symbols ▶ or ◀ marking lines that have been changed, and (2) updates to

[1] 40 CFR 180.101 (c)

the tables in Chapters 3 and 4, Appendix I, and the indices to methods, names, and CAS Registry numbers will be issued only *via* Internet. No hard copies will be distributed for the latter updated sections, but updates will be available more frequently than in the past.

Chapter tables of contents will include the date on which each section within the chapter was transmitted; dates associated with those sections distributed only electronically will reflect the most recent version at the time the table of contents issued.

Internet Access to PAM I Files

PAM I is now available *via* Internet as Adobe Acrobat "portable document format" (pdf) files. Pdf format permits the user to read and print the document from any computer using appropriate free software.

To obtain a copy of PAM I files, use one of these routes:

1) Go to the World Wide Web site at: http://vm.cfsan.fda.gov. Follow these links: Center for Food Safety and Applied Nutrition/Pesticides and Chemical Contaminants/ Pesticide Analytical Manual. A link on the resulting page displays the PAM I Table of Contents with links to currently available files. Follow the instructions for downloading.

2) FTP to ftp.cfsan.fda.gov. Go to the directory public/pam. Download ReadMe.txt and other file(s) as desired; use ascii protocol during download of ReadMe.txt and binary protocol for pdf files.

Adobe Acrobat Reader is required to view and print pdf files. (ReadMe can be read with any word processor.) Download a copy of this free software from Adobe's web site at http://www.adobe.com/acrobat/readstep.html. A link to that site is provided on the PAM I page. Choose the version of Acrobat Reader appropriate to your own computer system.

Peanuts	Whole peanut meat (kernel) after removing hulls.
Peanut hulls	Whole commodity after removing peanut meat.
Dates and olives	Whole commodity after removing and discarding stems and stones or pits.
Pineapples	Whole commodity after removing and discarding crowns (leaves at top of fruit).
Avocados and mangoes	Whole commodity after removing and discarding stones.
Bananas	Whole commodity including peel after removing and discarding crown tissue and stalk.
Miscellaneous raw fruits and vegetables not previously included	Whole commodity after removing and discarding obviously decomposed or withered leaves, stems, stones or pits, shells or husks; if commodity has adhering amounts of soil, remove by lightly rinsing in running water.
Almond hulls	Whole commodity after removing shell and nutmeat.
Cereal grains group	Whole commodity (grain) except for fresh corn (including sweet corn). Include kernels plus cob after removing and discarding husk.
Eggs	Whole commodity after removing and discarding shells.
Fish	Edible portion of the commodity after removing and discarding heads, tails, scales, fins, viscera, bones (if inedible), and skin (if inedible).
Crab (hard shell)	Edible portion of commodity after removing and discarding shells, gills, and viscera.
Crab (soft shell)	Edible portion of commodity after removing and discarding gills.
Shrimp and crayfish	Edible portion of commodity after removing and discarding heads, shells, and inedible tails of shrimp.
Lobster	Edible portion of commodity including tomalley (liver) after removing and discarding shells and stomachs (hard sac near head).
Oyster, clam, and other shellfish	Edible portion of commodity including the liquor, after removing and discarding shells.
Rabbits and other game	Edible portion of commodity after removing and discarding bones.

Processed Foods

In the absence of EPA regulations, FDA also developed the instructions listed in Table 102-b on the portion of processed food to be analyzed for tolerance enforcement purposes. These instructions, like the ones for raw agricultural commodities, ensure uniformity and consistency in FDA analysis of processed food for pesticide residues. The instructions take a practical approach for sample preparation of processed food; *e.g.*, fruit juice concentrates that are normally reconstituted before consumption are also reconstituted prior to analysis for pesticide residues. Therefore:

- Follow the directions in Table 102-b to prepare test samples of processed foods.

Table 102-b: Portion of Processed Food to be Analyzed for Pesticide Residues

Processed food consisting of one ingredient and sold in a ready-to-eat form (*e.g.*, canned fruits packed in syrup or their own juice, canned vegetables packed in water or brine, or frozen fruits or vegetables, dried fruits, single-strength juices, catsup)	Analyze the whole processed commodity including any liquid or other edible media in which the commodity is packed. Discard inedible media, *e.g.*, brine.
Processed food consisting primarily of one ingredient and sold in a form requiring further preparation before it is ready to eat (*e.g.*, fruit juice concentrates, dehydrated vegetables, and powdered potatoes)	Analyze the whole processed commodity after compensating for or reconstituting to the commodity's normal moisture content.
Processed food in a form not ready to eat, used as an ingredient or component of other food (*e.g.*, flour, tomato concentrates such as paste, and citrus oils)	Analyze the whole processed commodity on an "as is" basis.
Cheese	Analyze the whole commodity including natural cheese rind after removing and discarding waxed or oiled rinds.
Frozen seafood (*e.g.*, fish or shrimp)	Analyze the edible portion after thawing; discard water.
Canned seafood	Analyze the edible portion including edible liquor and media, such as oil, broth, or sauces in which commodity is packed. Discard media that is not edible.
Frog legs	Analyze the edible portion of commodity after removing and discarding bones.

1) Determinative step sensitivity to any particular residue. A distinct Lq applies to each residue determinable by a particular MRM, because the sensitivity of the determinative step to each compound may be different.

2) Limited detector sensitivity. Not all individual detectors are capable of reaching the sensitivity specified; in such cases, the Lq will be higher than targeted.

3) Greater detector sensitivity. Directions here recommend sensitivity at which detectors should be operated, even though some are capable of greater sensitivity. However, operation at conditions that produce recommended sensitivity may sometimes be precluded by other disadvantages in detector performance. For example, many models of ^{63}Ni electron capture detectors are not linear at conditions that produce sensitivity of 50% FSD to 1.5 ng chlorpyrifos, as is recommended for other detectors; most are linear, however, at conditions that produce 50% FSD to 0.15 ng chlorpyrifos. The rules in Section 105 C specify that, in this situation, the laboratory should operate at the greater sensitivity in order to work in a linear range, then proportionately reduce the weight of sample equivalent injected in order to maintain Lqs consistent with those achieved by other laboratories.

4) Other improvements that affect determinative step. Wide bore capillary GLC columns (Section 502 C) permit analytes to elute in a tighter band than was possible with packed column chromatography. When detector response is measured in terms of peak height, use of capillary columns results in an apparent improvement of response. Injection of a smaller amount of equivalent sample, as directed in Section 105 C, is appropriate and, at the same time, beneficial to the longevity of the column.

5) Excessive interferences from sample co-extractives. Interferences from sample co-extractives raise the Lq of a method by masking the detector response to the residue or by preventing injection of the specified sample equivalent without undesirable damage to the system. Additional procedures to clean up the sample extract prior to determination may improve the Lq by removing these interferences.

Table 105-a: Examples of Method Specifications Used to Calculate Lqs

PAM I Method[1]	Recommended Mg Injected	Recommended Sensitivity[2]	Lq (marker compound)[3]
302 E1+DG2 (FPD-P)	20 mg	1.5 ng chlorpyrifos	0.015 ppm chlorpyrifos
302 E3+C1+DG3 (ElCD-X)	20 mg	1.5 ng chlorpyrifos	0.015 ppm chlorpyrifos
302+E1+C3+DL1	116 mg	10 ng carbofuran	0.017 ppm carbofuran
303 E1+C1+DG1 (EC)	20 mg 2 mg	1.5 ng chlorpyrifos 0.15 ng chlorpyrifos	0.015 ppm chlorpyrifos 0.015 ppm chlorpyrifos
304 E4+C2+DG1 (EC)	10 mg (cheese with 30% fat)	1.5 ng chlorpyrifos	0.03 ppm chlorpyrifos, whole product basis
401 E1+C1+DL1	200 mg	10 ng carbofuran	0.01 ppm carbofuran
402 E1+C1+DG3 (fatty foods)	5 mg Eluate 1	1.5 ng chlorpyrifos (0.2 ng PCP methyl ether)	0.008 ppm PCP methyl ether
	10 mg Eluate 2	1.5 ng chlorpyrifos (0.5 ng 2,4,5-T methyl ester)	0.01 ppm 2,4,5-T methyl ester
402 E2+C1+DG3 (nonfatty foods)	10 mg Eluate 1	1.5 ng chlorpyrifos (0.2 ng PCP methyl ether)	0.004 ppm PCP methyl ether
	20 mg Eluate 2	1.5 ng chlorpyrifos (0.5 ng 2,4,5-T methyl ester)	0.005 ppm 2,4,5-T methyl ester
403 E1+C1+DL3	800 mg	40 ng diuron	0.01 ppm diuron
▶ 404 E1+DL5	125 mg	62.5 ng MBC	0.1 ppm MBC
404 E1+DL7	125 mg	6.25 ng thiabendazole (fluorescence detector)	0.01 ppm thiabendazole

[1] Parenthetical codes indicate the detector used in the GLC determinative step.
[2] Ng marker compound that causes detector response of 50% FSD; where residues targeted by the method are different from the marker compound, weight of example target that caused 50% FSD is also listed.
[3] Calculated by formula in Section 105 B; note that sensitivity is divided by 5 to produce ng causing 10% FSD.

- Elute each column with 200 mL 6% ethyl ether/petroleum ether. (Collect rinses with this eluate.)

- Change receivers; elute each column with 200 mL 15% ethyl ether/petroleum ether.

- Change receivers; elute each column with 200 mL 50% ethyl ether/petroleum ether.

- Concentrate each eluate, dilute to volume with hexane, and inject about 5 µL into appropriate GLC systems to determine recoveries. Dilute 1.0 mL each standard solutions A and B to 10 mL and use diluted solution as GLC reference standard.

- Consider Florisil lot acceptable if one of three columns permits complete recovery of test compounds and exhibits proper elution pattern (heptachlor, heptachlor epoxide, chlorpyrifos, and fonofos in 6% eluate; dieldrin, endosulfan I, parathion-methyl, and pirimiphos-methyl in 15% eluate; malathion and endosulfan sulfate in 50% eluate; and endosulfan II in both 15 and 50% eluates). Acceptable recovery is >80% for all compounds except heptachlor, and 60-90% for heptachlor. In subsequent use of lot of Florisil, use same weight as that in column with acceptable elution. ◀

- If none of the three columns exhibits proper elution but a consistent relationship exists between weight and elution, test additional columns of weights 3 g above or 3 g below that calculated using LA Value. If these columns also do not exhibit proper elution, it is best to use a different lot of Florisil.

If acceptable weight of Florisil is determined, test that column size further with following procedures:

- Repeat elution tests above, using 1.0 mL each solutions C and D. Elute column with 250 mL petroleum ether, followed by 6, 15, and 50% ethyl ether/petroleum ether eluants; collect each eluate separately. Determine recoveries of pesticides and verify accuracy of elution pattern using gas chromatographic measurement.

- Transfer each eluate quantitatively to separate tared 20 mL beaker. Evaporate solvent on steam bath or hot plate until constant weight is attained to measure amount of butterfat recovered in each eluate. Acceptable lots of Florisil typically permit about 0.3 mg (range 0-1.7 mg) butterfat to elute in petroleum ether eluate, 0.1 (0-0.4) mg in 6% ethyl ether/petroleum ether, 82 (40-135) mg in 15%, and 105 (60-172) mg in 50%.

- Repeat elution tests above, using 1.0 mL each solutions A and B and eluting with Eluants 1, 2, and 3 instead of ethyl ether/petroleum ether eluants.

It is acceptable, once the Florisil lot has been tested and appropriate weight of Florisil determined, to measure and record height of column produced by specified weight; subsequent columns may then be prepared by measuring height rather than weight.

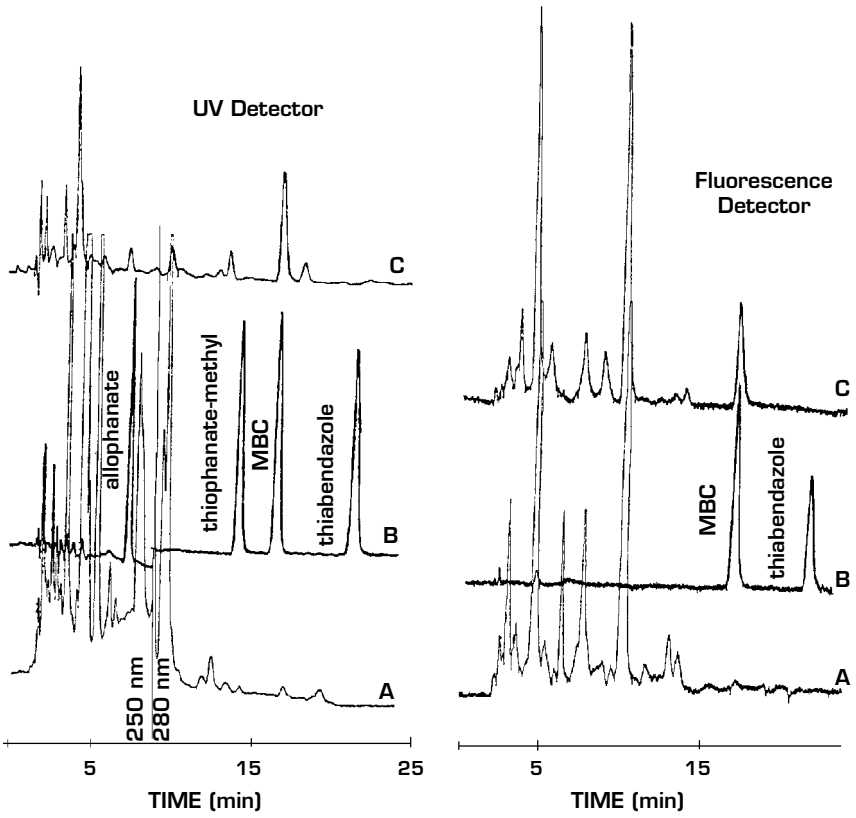
- Inject at least three 25 µL portions 2.5 ng/µL mixed standard solution. Determine following parameters:
  1) retention time and peak height for each peak; relative standard deviations (RSD) for repetitive retention times and peak height measurements
  2) column efficiency (N) for thiabendazole peak
  3) asymmetry factor (As) for thiabendazole peak
- Inject each of three different concentrations of mixed standard solutions (10-100 ng/25 µL injection). Plot peak height vs. amount injected.
- HPLC systems adequate for analysis for benzimidazoles will meet following minimum criteria: retention times of about 8, 15, 18, and 23 min for allophanate, thiophanate-methyl, MBC, and thiabendazole, respectively (retention times may vary among columns but should remain constant for particular column); RSD <0.5% for retention times and <3% for peak heights of individual peaks in three consecutive chromatograms; N>12,000 and As <1.3 for thiabendazole peak.
- Examine systems not meeting these criteria for problems, using various troubleshooting sections of Chapter 6. Correct problems uncovered by troubleshooting until system meets criteria defined above.
- System will typically respond linearly to 10-100 ng of each compound, but linear range may vary among systems. Perform quantitative analyses only within calculated linear range of system as determined above. Dilute sample extracts as needed to permit injection of analyte level within linear range. Adjust amounts injected so that peak heights of analyte and reference standard do not differ >25% from one another.

Directions

See Figure 404-b for typical chromatograms produced by HPLC system.

- To extract from E1-E3 (dissolved in 4.0 mL methanol), add 6.0 mL ion pairing solution; mix. Residue *must* be dissolved in methanol prior to adding ion pairing solution.
- Filter through 0.45 µm porosity membrane; filter will plug as solution is applied, so filter only volume needed for HPLC determination, about 1 mL.
- Inject 25 µL sample solution and chromatograph as described in System Operation.
- Compare chromatographic response (peak retention times, heights, and/or areas) with that of standard solution and calculate residue amount.
- If further dilutions are necessary, use mixture of 4:6 methanol:ion pairing solution as diluent.
- To convert calculated MBC (MW 191.2) to equivalent benomyl (MW 290.4), multiply by 1.52.
- To convert calculated MBC to equivalent thiophanate-methyl (MW 342.4), multiply by 1.79.
- Peaks of 50% FSD at conditions established for screening analysis are equivalent to about 0.5 ppm each of thiophanate-methyl, allophanate, and thiabendazole; MBC peak of 50% FSD at these conditions represents about 0.3 ppm.

Figure 404-b
Chromatograms of Benzimidazole Compounds



Chromatograms of: (A) peach extract partitioned from the acidic phase of 404 E1, (B) standard solution, (C) peach extract partitioned from basic phase of 404 E1. HPLC operation as directed in DL5. Sample contains 0.14 ppm field-incurred MBC.

ALTERNATIVES:

DL6 HPLC, CONCENTRATED ION PAIR MOBILE PHASE, UV AND FLUORESCENCE DETECTOR

Reference

Gilvydis, D.M., and Walters, S.M. (Aug. 1989) "Modification of LIB 3217 for Carbendazim (MBC) in Green and Roasted Coffee Beans," LIB 3353, FDA, Rockville, MD

Principles

Concentration of ion pairing reagent is increased eight times to increase k′ values of analytes and improve separation from early eluting co-extractives.

Additional Reagents

ion pairing solution, 32.7 mM 1-decanesulfonate, sodium salt. Pipet 7.0 mL phosphoric acid into 200 mL HPLC grade water; dissolve 8.0 g 1-decanesulfonate, sodium salt in this mixture. Pipet 10.0 mL triethylamine into solution and dilute to 1 L with HPLC grade water. Filter through <1 µm porosity membrane. (pH of solution should be about 2.4.)

Gradient elution is possible provided the solvents do not absorb. At very sensitive settings, changes in RI, as caused by gradient elution or pressure and flow changes, can produce baseline shifts with some types of detector cells.

The fixed wavelength detector is less versatile but is much less expensive and often gives less noise than the continuously variable wavelength spectrophotometric detector. As mentioned above, the great advantage of the variable wavelength detector is the ability to optimize sensitivity and/or selectivity for each analyte by detection at the most favorable wavelength.

Multichannel or Photodiode Array Detectors

In a photodiode array detector, polychromatic radiation is passed through the detector flow cell, and emerging radiation is diffracted by a grating so that it falls on an array of photodiodes. Each diode receives a different narrow wavelength band. The complete array of diodes is scanned by a microprocessor many times a second. The resulting spectra may be displayed on a cathode ray tube monitor and/or stored in the instrument for transfer to a recorder or printer. The detector is best used in conjunction with a computerized data station, which allows various post-run manipulations, such as identity confirmation by comparison of spectra with a library of standard spectra recalled from disk storage. Detection can be made at a single wavelength or at a number of wavelengths simultaneously, or wavelength changes can be programmed to occur at specified points during the run. Absorbance ratios at selected wavelengths (*e.g.*, 254 and 280 nm) can be displayed for each peak, which aids in determining identity and the presence of unresolved components.

Applications

The UV detector has been the most widely used for pesticide residue determination. Section 404 uses UV and fluorescence detectors to determine benzimidazole residues, whereas other references describe combinations of UV and photoconductivity [1-3]; the photodiode array is applicable to determining paraquat and diquat [4].

Problems, Maintenance, and Troubleshooting

Air bubbles in UV flow cells can produce a series of very fast noise spikes on the chromatogram, or pronounced baseline drift. Falsely high absorbance readings can be caused by impure or improperly prepared mobile phase, large air bubbles in the flow cell, a misaligned flow cell, or dirty end windows. Gas bubbles develop in the detector cell because they are pumped through the system or the solvent is degassed in the detector. Prevent bubbles from being pumped through the system by eliminating system leaks, expelling air from the pumping system, avoiding very volatile solvents, and not stirring the mobile phase reservoir too vigorously. Prevent solvent degassing in the sample cell by degassing the mobile phase prior to use. If the cell has no back pressure valve, raise cell pressure above atmospheric by attaching ≥10' spiral steel or Teflon tubing to the detector outlet to act as a flow restrictor, and placing the tubing outlet above the detector. The tubing must not shut off flow completely, as too great a pressure increase could shatter the cell windows.

To dissolve gas bubbles lodged in the cell, briefly increase cell back pressure by holding a piece of rubber septum over the detector outlet or by connecting a syringe to the outlet. With aqueous systems, it may be necessary to fill the cell with methanol and repeat application of back pressure.

Protect the detector from temperature fluctuations by placing the system away from direct sunlight and drafts, and regularly monitor flow rate and pressure for change.

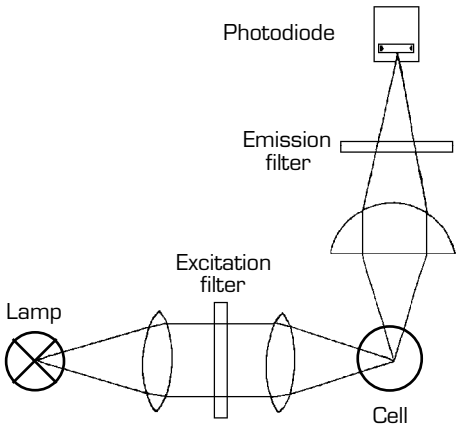
Detector response can drop because dirt in the cell or a bad source lamp reduces the level of radiation reaching the photocell. Some detectors have a meter that allows easy determination of light level. If it is low, clean the detector or change the source lamp. (Avoid eye damage by not viewing the light directly.) Consult the detector manual for the proper procedure for changing the lamp and cleaning the cell. The average life of a 254 nm lamp is approximately 5000 hr, but it should be replaced as soon as aging begins to cause significant intensity changes. Some cells can be taken apart, the optical components cleaned with a suitable solvent and dried, and the cell re-assembled. Others cannot be taken apart and are cleaned by flushing the cells with a series of solvents delivered from a 50 mL glass syringe, *e.g.*, acetone, 6 M nitric acid, distilled water, and acetone, then drying with a flow of clean, dry nitrogen before reconnection to the column. If necessary, allow 6 M nitric acid to stand in the cell overnight. To remove particles most effectively, draw nitric acid through the cell with a syringe in a direction opposite to the normal flow.

605 B: FLUORESCENCE DETECTORS

Fluorescence detectors provide two to three orders of magnitude more sensitivity than UV detection. Selectivity is also excellent because of the choice of excitation and emission wavelengths and the fact that only a small fraction of all compounds naturally fluoresce.

The simplest type of instrumentation is a fixed wavelength fluorometer with bandpass filters for both excitation and emission. More convenient and versatile fluorometric detectors can operate at variable wavelengths. These are equipped with monochromators to select excitation and emission wavelengths. Most compounds that fluoresce naturally have a rigid, planar conjugated cyclic structure. Nonfluorescent compounds can be detected if they are first converted to fluorescent compounds by pre- and post-column derivatization.

Figure 605-c
Fluorescence HPLC Detector



[Reprinted with permission of John Wiley and Sons, Inc., from Meyer, V.R. (1988) *Practical High Performance Liquid Chromatography,* Figure 5.10, page 74.]

Detector Design

Figure 605-c is a schematic diagram of a simple filter fluorometer detector. Light from a mercury lamp passes through a filter that selects the excitation wavelength. An interference filter providing a 10-20 nm

REPORTING FORM C: GLC DATA

The following GLC data resulted from testing the chemical * on systems described in PAM I Section 302 DG modules, according to directions in Appendix II, Protocol C.

Name: *

Alternative Names:

Reference Standard (source and number):

Molecular Formula:

Structure:

Comments:

Results for DG module (Section 302): DG ____

Standard reference material dissolved in

Brief details about GLC system used:

Column:

Length:

id:

Film thickness:

Carrier gas:

Flow rate:

Makeup gas:

Flow rate:

Retention time (relative to _____) of _____ (marker compound): ◀

Detector:

Temperature:

Other conditions:

Detector response to ____ ng _____:_____% FSD

Behavior of * :

Retention time (relative to _____):

ng required for 50% FSD:

Information submitted by:

Address:

Phone: ( )

Date:

Pesticide Analytical Manual Volume I
10/97 Revisions

The following pages contain corrections or changes for PAM I. Print these pages and use them to replace the same current pages in the current PAM I 3rd edition (published 1/94, revised 9/96).

Each set of two pages is intended to appear on two sides of the same paper, but Acrobat Reader does not offer a feature that facilitates printing on both sides of the page. It may be necessary to print one page at a time and turn the paper over to print the second page on the reverse side.

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12. Pages 302-57 and 302-58	302-57, 302-58	32-33
13. Pages 302-63 to 302-70	302-63 to 302-70	34-41
14. Appendix II, pages 15-16	Appendix II-15 and Appendix II-16	42-43

Explanations of changes:

Tables of Contents for Chapters 1, 2, and 3 now include the date of the current version for each section within the chapter.

Introduction includes revised directions for downloading PAM I from the World Wide Web; it is no longer possible to use FTP to obtain a copy of PAM I.

Section 105, page 2, is revised to clarify the formula for calculating limits of quantitation and to specify the use of methyl siloxane columns in such calculations for GLC analyses.

Pages 302-3 and 302-23 are revised to remove references to the DEGS packed GLC column, which is now considered obsolete (it is no longer commercially available).

Pages 302-27, 302-33, 302-51, and 302-57 are revised to include a new system suitability test for GLC systems used for organophosphorus residues.

Pages 302-63 through 302-70 are revised to include a statement that DEGS columns are now considered obsolete.

Appendix II, pages 15-16 are revised to remove the requirement to collect GLC data on DEGS columns.

PESTICIDE ANALYTICAL MANUAL

VOLUME I: Multiresidue Methods

U.S. Department of Health and Human Services • Public Health Service
Food and Drug Administration

PESTICIDE ANALYTICAL MANUAL VOLUME I

3rd Edition, 1994

Revised, September, 1996
Revised, October, 1997

MANAGING EDITORS

Management Methods Branch
Office of the Associate Commissioner for Management and Operations

Design and Format: R.O.W. Sciences, Inc.
under contract to U.S. Food and Drug Administration

PESTICIDE ANALYTICAL MANUAL

INTRODUCTION

The Food and Drug Administration (FDA) is responsible under the Federal Food, Drug, and Cosmetic Act for enforcing tolerances established by the Environmental Protection Agency (EPA) for amounts of pesticide residues that may legally remain on food (including animal feed). In meeting this responsibility, FDA collects and analyzes food from commercial channels of trade for determining compliance with EPA tolerances. The residue data gathered under this regulatory monitoring program are also used for evaluating the extent and significance of pesticide residues in the food supply.

The Pesticide Analytical Manual (PAM) is published by FDA as a repository of the analytical methods used in FDA laboratories to examine food for pesticide residues for regulatory purposes.[1] The manual is organized according to the scope of the analytical methods:

Volume I contains multiresidue methods (MRMs) that are used by FDA on a routine basis, because of their efficiency and broad applicability, especially for analyzing foods of unknown pesticide treatment history.

Volume II contains methods designed for the analysis of commodities for residues of only a single compound (although some methods are capable of determining several related compounds). These methods are most often used when the likely residue is known to the chemist and/or when the residue of interest cannot be determined by common MRMs.

PAM is designed to be used by analysts experienced in trace residue analysis. All of the techniques employed are subject to potential interferences from reagents, apparatus, containers, contaminated air supply, and handling by personnel. The experienced analyst is alert for these possibilities and recognizes the need to confirm results by other techniques that measure different chemical or physical properties of the analyte.

Experienced residue analysts are aware that no report of validation in another laboratory can substitute for verification that the method does indeed work in the analyst's own laboratory. The analyst should verify method performance in each particular application by a trial of the method that includes examination of reagent and sample blanks and measurement of the recovery of added analyte. The editors invite analysts to report results of their experiences with PAM methods.

Revisions

Starting with transmittal 96-1 (9/96), revisions of PAM I have been issued in two ways: (1) changes in most manual sections will be distributed as hard (paper) copies, with symbols ▶ or ◀ marking lines that have been changed, and (2) updates to the tables

[1] 40 CFR 180.101 (c)

in Chapters 3 and 4, Appendix I, and the indices to methods, names, and CAS Registry numbers will be issued only *via* Internet. No hard copies will be distributed for the latter updated sections, but updates will be available more frequently than in the past.

As chapter tables of contents are revised, they will include the date on which each section within the chapter was transmitted; dates associated with those sections distributed only electronically will reflect the most recent version at the time the table of contents issued.

Internet Access to PAM I Files

PAM I is now available *via* Internet as Adobe Acrobat "portable document format" (pdf) files. Pdf format permits the user to read and print the document from any computer using appropriate free software.

▶ To obtain a copy of PAM I files, go to the World Wide Web site at: http://vm.cfsan.fda.gov/~frf/pami1.html. The resulting page describes PAM and provides links to currently available files. Follow the instructions for downloading.

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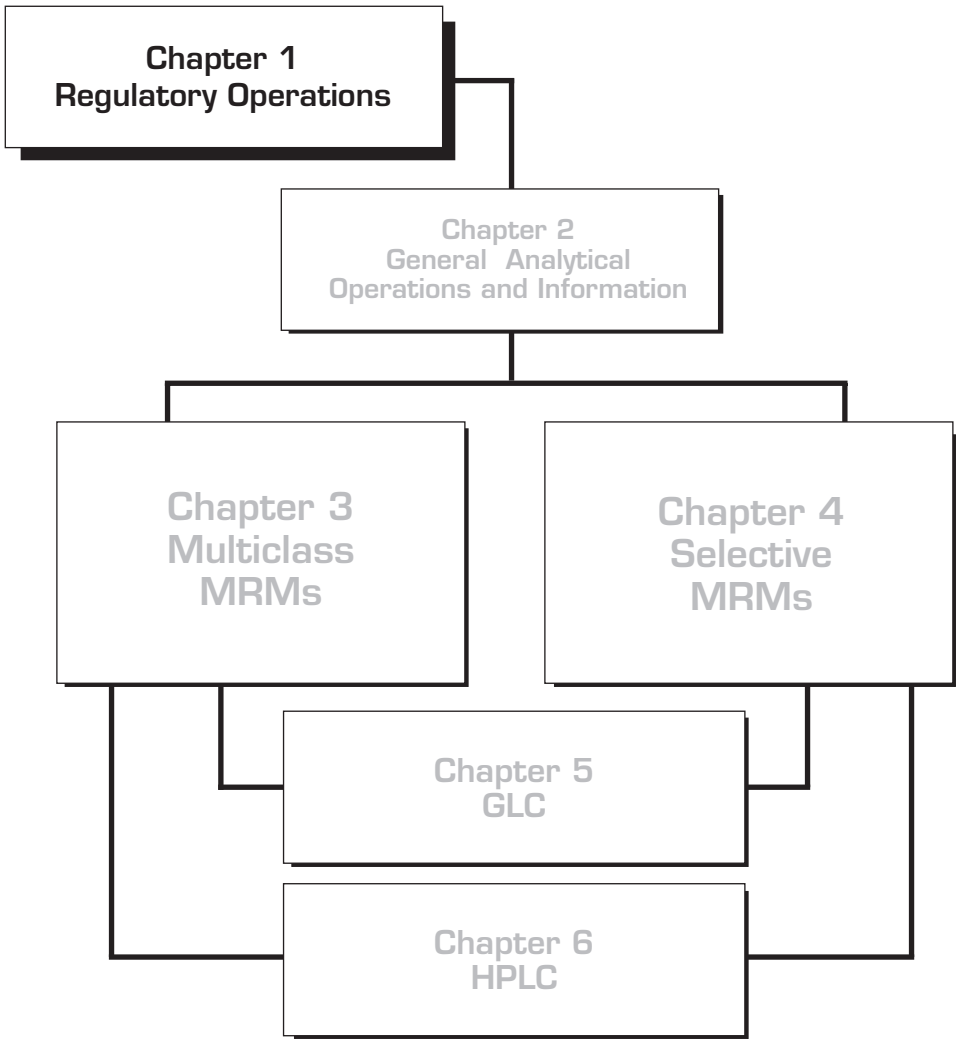


Table of Contents

105: ANALYTICAL LIMITS OF QUANTITATION

105 A: DEFINITION

FDA defines limit of quantitation (Lq) as the lowest level of residue that can be quantitated by a given method and whose identity can be confirmed in regulatory laboratories operating under routine conditions. Levels less than the Lq are defined as trace.

When MRMs are used, a separate Lq applies to each residue determined by the method because each represents a different analytical situation.

The following factors must be specified in order to define the analytical situation; only then can an Lq be calculated:

1) Analytical method used

2) Sample (matrix) type

3) Sample weight equivalent introduced to the determinative step

4) Sensitivity of the determinative step to the analyte; sensitivity is dependent on the following instrumental conditions:

   a) Determinative technique (In MRMs, the determinative step is usually GLC or HPLC; operational parameters must be defined as part of the method description.)

   b) Range of analyte weight that produces a linear detector response

   c) Overall condition of the system

   d) Amplification and/or attenuation of the detector signal

   e) Characteristics of the signal processing or recording device

   f) Chromatographic elution characteristics of the analyte

105 B: CALCULATION

FDA Lqs for each method are arrived at by (1) specifying a sample weight equivalent to be examined by the determinative step (the amount chosen must be compatible with long-term instrument stability); (2) establishing a recommended determinative step sensitivity that is stable, reproducible, and achievable by all laboratories; and (3) establishing a response equivalent to 10% of full scale deflection (FSD) on the signal-processing device as the minimum considered quantifiable and confirmable. FDA methods applied according to these guidelines are capable of analyzing for most residues at levels well below established tolerances.

Determinative step sensitivity is established by reference to a "marker compound"; *i.e.*, the instrumental parameters are adjusted to cause a specified response to a specified quantity of the marker compound. This approach makes it possible for different laboratories to achieve approximately the same Lq even though the instrument settings may be different for each. Lq for the marker compound can then be calculated with the formula below for any particular method. Lqs for all other compounds recovered through the method will vary according to the determinative step sensitivities for each.

▶ With these guidelines established, Lq for a method is calculated thus:

$$\text{ng 50\% FSD} = \text{ng analyte injected} \times \frac{\text{ng marker specified}}{\text{ng marker injected}} \times \frac{\text{marker peak height}}{\text{analyte peak height}}$$

$$\text{ng 10\% FSD} = \text{ng 50\% FSD}/5$$

$$\text{Lq} = (\text{ng 10\% FSD})/(\text{mg sample injected})$$

▶ Round the Lq result following the guidance for significant figures and reporting analytical results in Section 104, page 104-3. For general purposes, results at or below 0.010 ppm are deemed to have an Lq of 0.010 ppm.

105 C: IMPLEMENTATION

Guidelines for applying analytical methods are required to provide consistency among laboratories performing regulatory analyses. Otherwise, variations in the amount of sample equivalent injected and/or the sensitivity of the determinative step can cause different Lqs in different laboratories. Lqs that result from following FDA guidelines are adequate for the enforcement of tolerances and, in most cases, are sufficient to determine residues below the tolerance level so that data on incidence and levels of residues in foods and feeds can be collected.

The following rules are established to maintain consistent Lqs among FDA laboratories:

- ▶ Establish the sensitivity recommended in each determinative step method module (*e.g.*, Section 302 DG1-DG12, Section 401 DL1). Note that the requirement for GC determinations to be based on columns of 100% methyl siloxane is in effect as of FY'98 (October 1, 1997); prior to that time, other DG modules may have been used to calculate Lq.

- Inject a volume of extract containing the equivalent sample weight recommended for each method (*e.g.*, Section 302, Determination).

- If one of the recommended specifications above cannot be achieved, or if changing one is advisable for any reason, adjust the other parameter to maintain the targeted limit of quantitation. Section 105 D describes factors that may cause problems in specific situations.

Table 105-a lists examples of Lqs that can be calculated from the recommended sample weight equivalent and determinative step sensitivity for particular PAM I methods. The list is not exhaustive but does illustrate the way in which the Lq for any method in PAM I can be calculated.

105 D: FACTORS AFFECTING TARGET LIMITS OF QUANTITATION

The following factors, individually or in combination, may reduce the certainty of quantitation and/or identification of a residue in any specific analytical situation. They may also cause the Lq to differ from the recommended limit defined by the formula above and by Table 105-a. Measures taken to compensate for one factor may trigger the influence of another.

1) Determinative step sensitivity to any particular residue. A distinct Lq applies to each residue determinable by a particular MRM, because the sensitivity of the determinative step to each compound may be different.

2) Limited detector sensitivity. Not all individual detectors are capable of reaching the sensitivity specified; in such cases, the Lq will be higher than targeted.

3) Greater detector sensitivity. Directions here recommend sensitivity at which detectors should be operated, even though some are capable of greater sensitivity. However, operation at conditions that produce recommended sensitivity may sometimes be precluded by other disadvantages in detector performance. For example, many models of ^{63}Ni electron capture detectors are not linear at conditions that produce sensitivity of 50% FSD to 1.5 ng chlorpyrifos, as is recommended for other detectors; most are linear, however, at conditions that produce 50% FSD to 0.15 ng chlorpyrifos. The rules in Section 105 C specify that, in this situation, the laboratory should operate at the greater sensitivity in order to work in a linear range, then proportionately reduce the weight of sample equivalent injected in order to maintain Lqs consistent with those achieved by other laboratories.

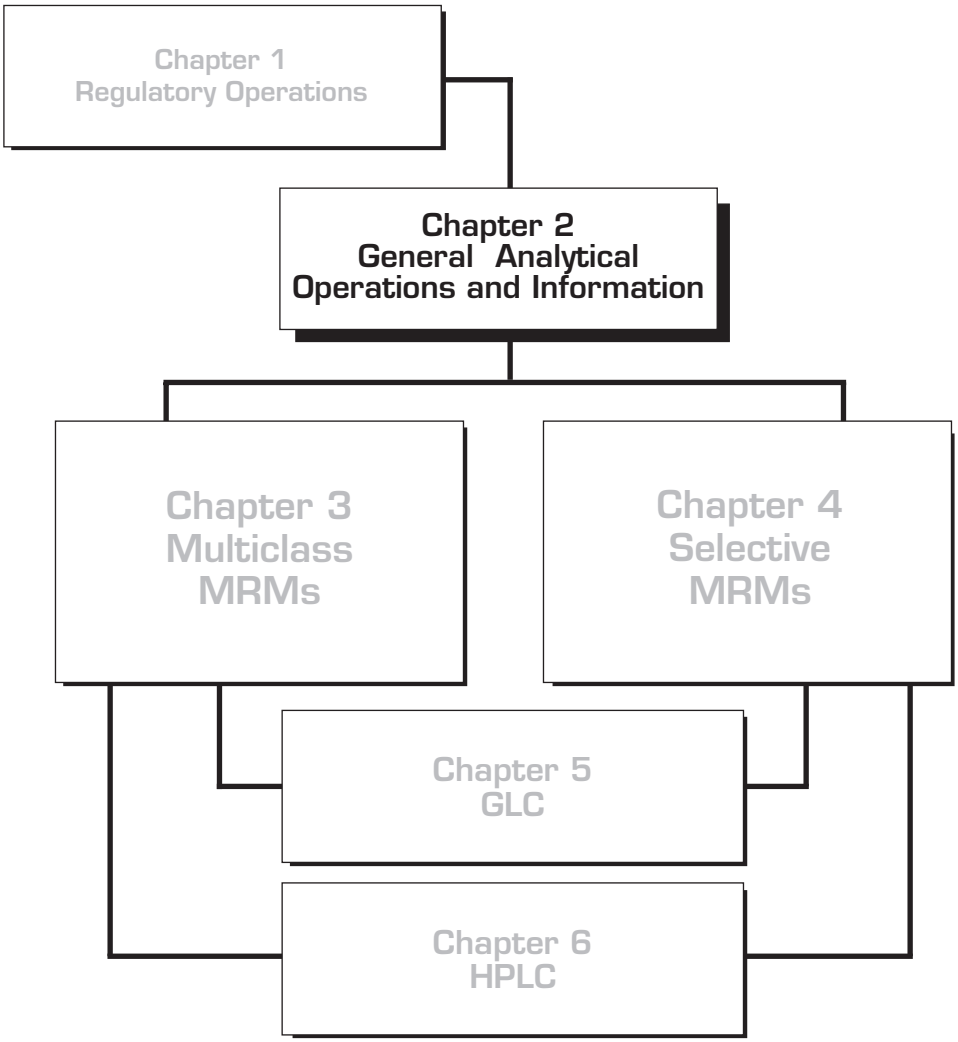
4) Other improvements that affect determinative step. Wide bore capillary GLC columns (Section 502 C) permit analytes to elute in a tighter band than was possible with packed column chromatography. When detector response is measured in terms of peak height, use of capillary columns results in an apparent improvement of response. Injection of a smaller amount of equivalent sample, as directed in Section 105 C, is appropriate and, at the same time, beneficial to the longevity of the column.

5) Excessive interferences from sample co-extractives. Interferences from sample co-extractives raise the Lq of a method by masking the detector response to the residue or by preventing injection of the specified sample equivalent without undesirable damage to the system. Additional procedures to clean up the sample extract prior to determination may improve the Lq by removing these interferences.

Table 105-a: Examples of Method Specifications Used to Calculate Lqs

PAM I Method[1]		Recommended Mg Injected	Recommended Sensitivity[2]	Lq (marker compound)[3]
302 E1+DG2 (FPD-P)		20 mg	1.5 ng chlorpyrifos	0.015 ppm chlorpyrifos
302 E3+C1+DG3 (ElCD-X)		20 mg	1.5 ng chlorpyrifos	0.015 ppm chlorpyrifos
302+E1+C3+DL1		116 mg	10 ng carbofuran	0.017 ppm carbofuran
303 E1+C1+DG1 (EC)	OR	20 mg 2 mg	1.5 ng chlorpyrifos 0.15 ng chlorpyrifos	0.015 ppm chlorpyrifos 0.015 ppm chlorpyrifos
304 E4+C2+DG1 (EC)		10 mg (cheese with 30% fat)	1.5 ng chlorpyrifos	0.03 ppm chlorpyrifos, whole product basis
401 E1+C1+DL1		200 mg	10 ng carbofuran	0.01 ppm carbofuran
402 E1+C1+DG3 (fatty foods)		5 mg Eluate 1	1.5 ng chlorpyrifos (0.2 ng PCP methyl ether)	0.008 ppm PCP methyl ether
		10 mg Eluate 2	1.5 ng chlorpyrifos (0.5 ng 2,4,5-T methyl ester)	0.01 ppm 2,4,5-T methyl ester
402 E2+C1+DG3 (nonfatty foods)		10 mg Eluate 1	1.5 ng chlorpyrifos (0.2 ng PCP methyl ether)	0.004 ppm PCP methyl ether
		20 mg Eluate 2	1.5 ng chlorpyrifos (0.5 ng 2,4,5-T methyl ester)	0.005 ppm 2,4,5-T methyl ester
403 E1+C1+DL3		800 mg	40 ng diuron	0.01 ppm diuron
404 E1+DL5		125 mg	62.5 ng MBC	0.1 ppm MBC
404 E1+DL7		125 mg	6.25 ng thiabendazole (fluorescence detector)	0.01 ppm thiabendazole

[1] Parenthetical codes indicate the detector used in the GLC determinative step.
[2] Ng marker compound that causes detector response of 50% FSD; where residues targeted by the method are different from the marker compound, weight of example target that caused 50% FSD is also listed.
[3] Calculated by formula in Section 105 B; note that sensitivity is divided by 5 to produce ng causing 10% FSD.



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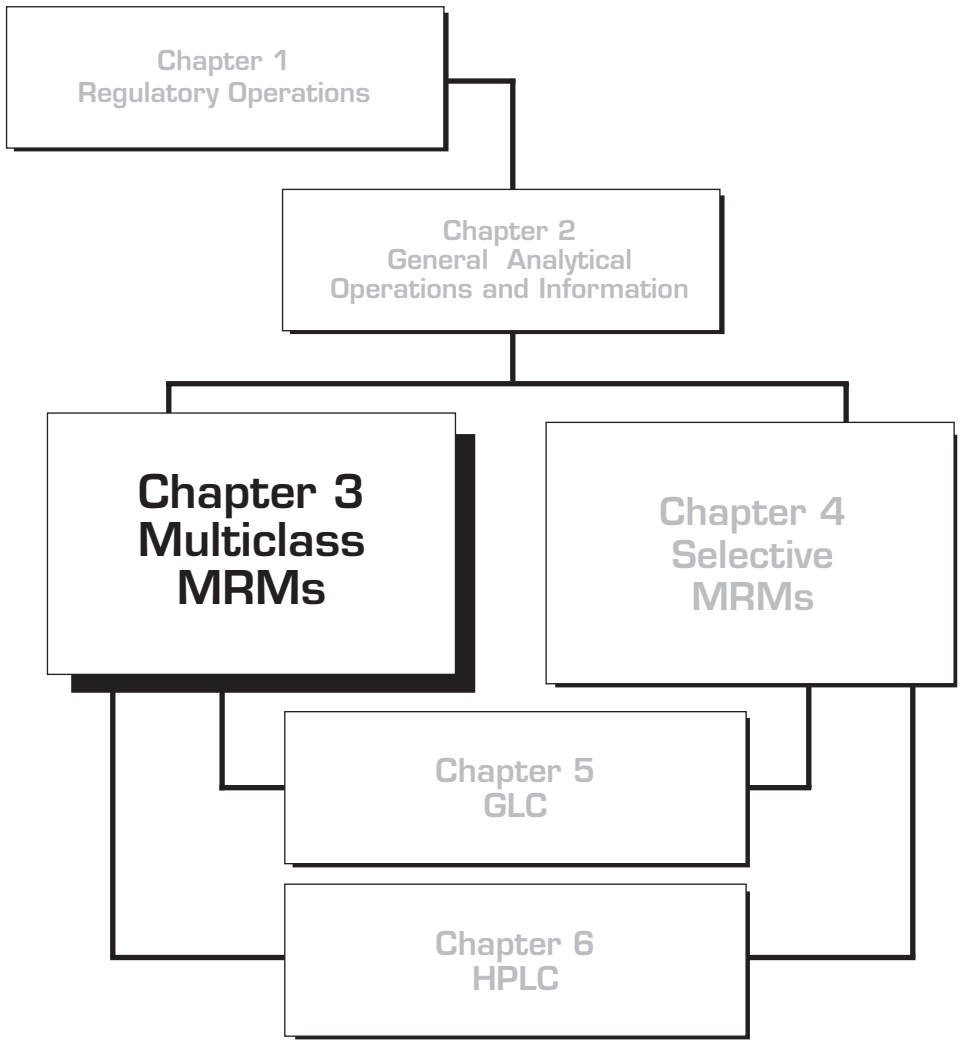

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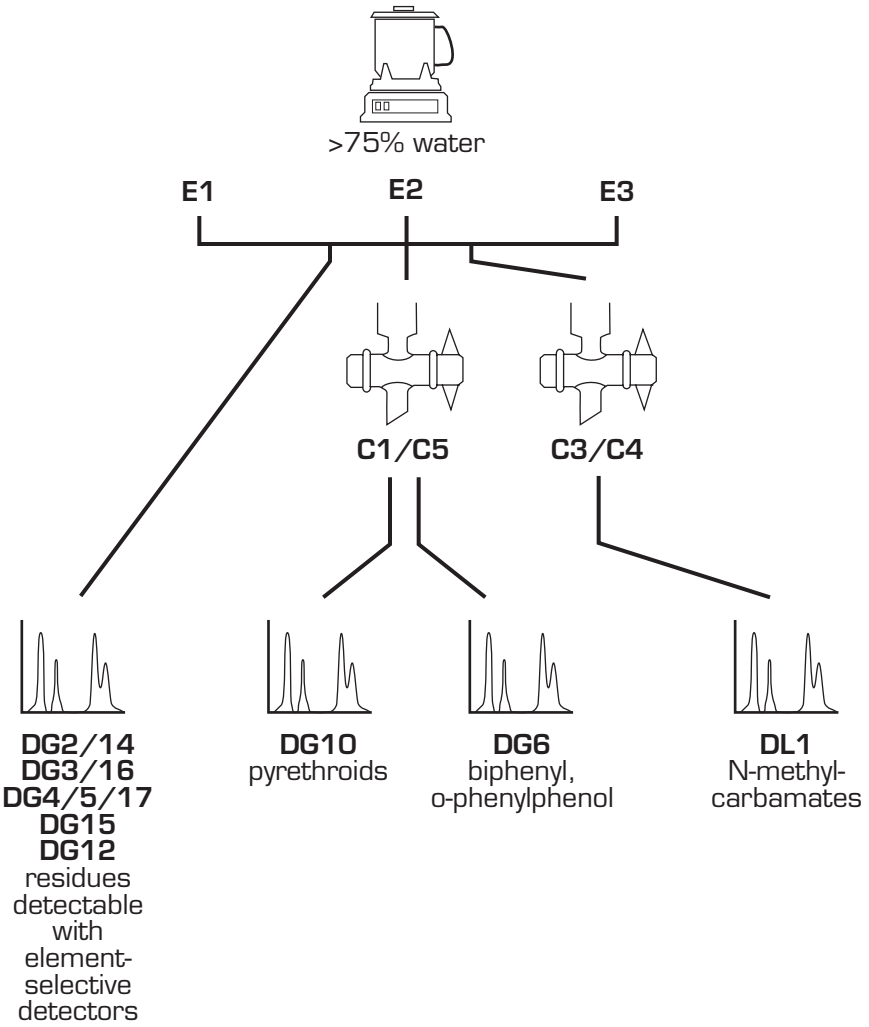
DG21	(p. 302-65)	GLC, DEGS column, 180°, FPD-S	polar residues with sulfur
DG22	(p. 302-67)	GLC, DEGS column, 180°, ElCD-X	polar residues with halogen
DG23	(p. 302-69)	GLC, DEGS column, 180°, N/P detector	polar residues with nitrogen or phosphorus
DL1	(p. 401-9)	HPLC, post-column hydrolysis and derivatization, fluorescence detection	N-methylcarbamates

Figure 302-a
Recommended Approach: Nonfatty Foods



◀

VALIDATION

Many combinations of method modules are possible. The following combinations have undergone interlaboratory validation and are recommended for use:

E1 + DG2, DG3

Validation report:

Sawyer, L.D. (1985) *J. Assoc. Off. Anal. Chem.* **68**, 64-71. Collaborative study leading to AOAC official final action status for acephate, α-BHC, chlorpyrifos, dieldrin, monocrotophos, and omethoate in lettuce, strawberries, and tomatoes.

AOAC official method reference: *Official Methods of Analysis of the AOAC* (1990) 15th ed., 985.22.

E1 + C3 + DL1

Validation report:

Pardue, J.R. (April 1987) "Recoveries of N-Methyl Carbamates Using a Combination of the Luke (PAM I, 232.4) and Krause (PAM I, 242.24b, 242.25) Procedures," LIB 3138, FDA, Rockville, MD

E2 + C1 + [temperature programmed GLC systems equivalent to] DG1, DG7, DG10, and DG16

Validation report:

Griffitt, K.R., and Szorik, M.M. (Sept 1989) "The Analysis of 127 Total Diet Items for Chlorinated Residues Using Luke/Solid Phase Extracts," LIB 3366, FDA, Rockville, MD

DETERMINATION

Inject concentrated extract equivalent to 20 mg (whole high moisture product) into the following GLC systems for determination of residues. (Although AOAC collaborative study for this method involved injection of 12 mg sample equivalent, experience since then has proven that GLC systems can tolerate routine injections equivalent to 20 mg of most nonfatty foods.)

Extract not cleaned up prior to determination:

DG2 or DG14	organophosphorus residues; large amounts of sulfur may interfere
DG3 or DG16	organohalogen residues
DG4 or	organonitrogen residues; selective to nitrogen, but co-extractives may contain nitrogen
DG5 or DG17	organonitrogen and organophosphorus residues
DG15	organosulfur residues; large amounts of phosphorus may interfere ◀
DG12	late eluting organohalogen residues, especially pyrethroids

Additional recommended determinations:

Extract not cleaned up prior to determination:

DG8	early eluting organophosphorus residues
DG11	late eluting organophosphorus residues
DG9	early eluting organohalogen residues

Extract cleaned up on Florisil column, C1 or C5:

DG1 or DG13	residues with halogen, sulfur, or other moieties
DG7	early eluting residues with halogen, sulfur, or other moieties
DG10	late eluting residues, especially synthetic pyrethroids
DG6	o-phenylphenol and biphenyl

Inject concentrated extract equivalent to about 58-116 mg (whole high moisture product) cleaned up by C3 (charcoal/Celite column) or C4 (C-18 cartridge) into following HPLC system:

DL1	N-methylcarbamates (determinative step described in Section 401)

For accurate quantitation, reference standards should be dissolved in same solvent as concentrated extract, only peaks >10% FSD should be measured, and peak sizes of residue and reference standard should match within ±25%.

See Chapter 5 for additional information about operation of GLC systems; Section 504 provides information about quantitation of residues.

See Chapter 6 for additional information about operation of HPLC systems; Section 606 provides information about quantitation of residues.

See Section 205 for additional information about reference standards.

See Section 104 for additional information about reporting residues and determining compliance with regulations.

See Section 105 for additional information about analytical limits of quantitation.

CONFIRMATION

After residues have been tentatively identified and quantitated by comparison to appropriate reference standards, confirm identity according to principles discussed in Section 103. Use appropriate tables of data (PESTDATA, tables accompanying each method, Index to Methods) to choose the most appropriate determinative steps and/or alternative methods for confirmation.

DG2 GLC, 100% METHYL SILOXANE, 200° C, FPD-P

Applicability

Determinative step is applicable to residues containing phosphorus. It is particularly useful for residues such as organophosphate pesticides.

Column

Wide bore capillary, 30 m × 0.53 mm id, coated with 100% methyl substituted polysiloxane liquid phase, 1-1.5 µm film thickness, bonded and cross-linked. For example, DB-1; see Table 502-a for equivalents. See Section 502 C, Recommended Operating Procedure for Wide Bore Columns.

Column Operating Conditions:

200° C isothermal; if necessary, adjust temperature so that relative retention time to chlorpyrifos (rrt_c) of ethion is 2.56±0.05.

Carrier gas: helium; adjust flow rate so that chlorpyrifos elutes in about 4.0±0.5 min (about 20 mL/min).

Injector temperature: 220-250° C

Detector

Flame photometric, phosphorus mode (FPD-P)

Detector Operating Conditions:

225-250° C

See Section 503 C for other information about FPD operation.

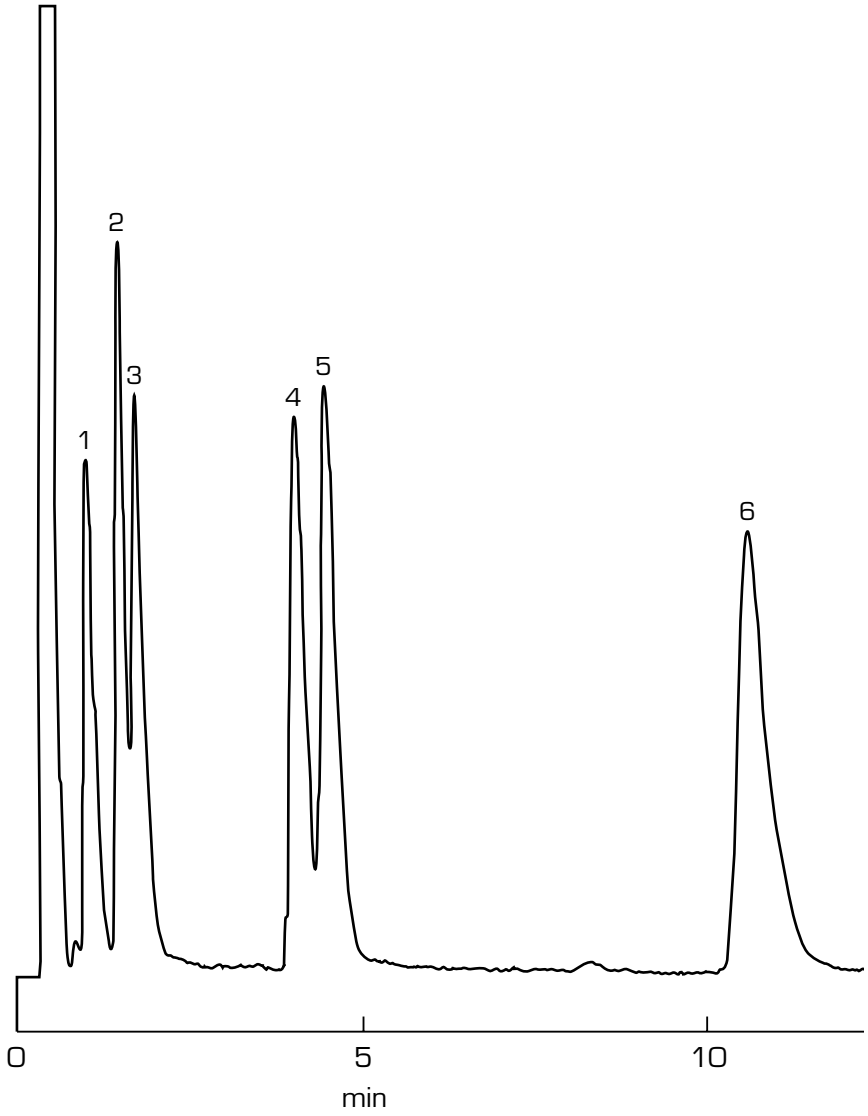
Set detector electronics (amplification, attenuation) so that response to 1.5 ng chlorpyrifos is 50% full scale deflection (FSD).

A properly functioning system will give a response to 1.5 ng omethoate of ≥50% FSD. ◀

Other Considerations

Rrt_cs and ng required to cause 50% FSD response are listed in Appendix I, PESTDATA (many data in PESTDATA were collected using equivalent packed column).

Example chromatogram is on next page.



DG2

Chromatogram of: 1) 0.85 ng acephate, 2) 1.73 ng omethoate, 3) 0.68 ng monocrotophos, 4) 1.30 ng malathion, 5) 1.27 ng chlorpyrifos, and 6) 1.26 ng ethion at the conditions described; helium carrier gas flow was 15 mL/min, with 15 mL/min make-up gas being added before the detector. Detector gas flows: 100 mL/min hydrogen, 130 mL/min air.

DG5 GLC, 100% METHYL SILOXANE, 200° C, N/P

Applicability

Determinative step is applicable to residues containing nitrogen. It is particularly useful for residues such as triazines and triazoles.

Column: Wide bore capillary, 30 m × 0.53 mm id, coated with 100% methyl substituted polysiloxane liquid phase, 1-1.5 µm film thickness, bonded and cross-linked. For example, DB-1; see Table 502-a for equivalents. See Section 502 C, Recommended Operating Procedure for Wide Bore Columns.

Column Operating Conditions:

200° C isothermal; if necessary, adjust temperature so that relative retention time to chlorpyrifos (rrt_c) of ethion is 2.56±0.05.

Carrier gas: helium; adjust flow rate so that chlorpyrifos elutes in about 4.0±0.5 min (about 20 mL/min).

Injector temperature: 220-250° C

Detector

Alkali bead detector, nitrogen selective (N/P)

Detector Operating Conditions:

250° C

See Section 503 E for other information about N/P detector operation.

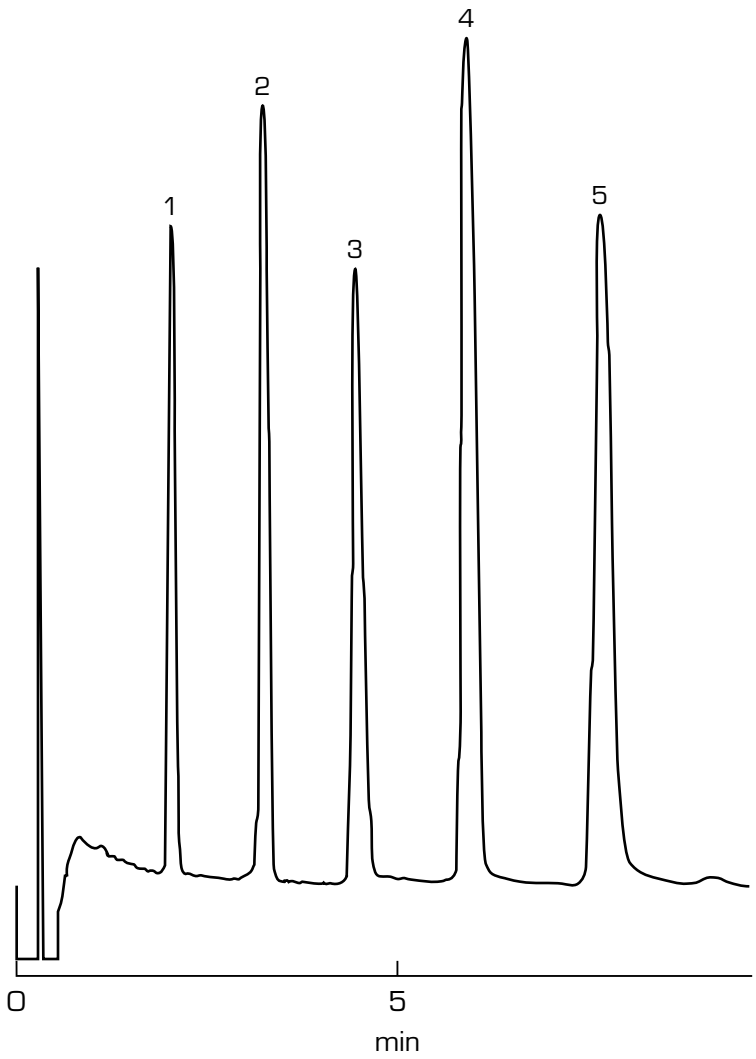
Set detector electronics (amplification, attenuation) so that response to 1.5 ng chlorpyrifos is 50% full scale deflection (FSD).

A properly functioning system will give a response to 1.5 ng omethoate of ≥50% FSD. ◀

Other Considerations

Rrt_cs and ng required to cause 50% FSD response are listed in Appendix I, PESTDATA (many data in PESTDATA were collected using equivalent packed column).

Example chromatogram is on next page.



DG5

Chromatogram of: 1) 1.0 ng atrazine, 2) 7.5 ng carbaryl, 3) 1.5 ng chlorpyrifos, 4) 2.5 ng procyazine, and 5) 5.0 ng imazalil at the conditions described.

DG14 GLC, 50% PHENYL, 50% METHYL SILOXANE, 200° C, FPD-P

Applicability

Determinative step is applicable to residues containing phosphorus. It is particularly useful for residues such as organophosphate pesticides.

Column

Wide bore capillary, 30 mm × 0.53 mm id, coated with 50% phenyl, 50% methyl substituted polysiloxane liquid phase, 1-1.5 µm film thickness, bonded and cross-linked. For example, DB-17; see Table 502-a for equivalents. See Section 502 C, Recommended Operating Procedure for Wide Bore Columns.

Column Operating Conditions:

200° C, isothermal; if necessary, adjust temperature so that relative retention time to chlorpyrifos (rrt_c) of ethion is 3.36±0.07.

Carrier gas: helium; adjust flow rate so that chlorpyrifos elutes in about 4±0.5 min (about 20 mL/min).

Injector temperature: 250° C

Detector

Flame photometric, phosphorus mode (FPD-P)

Detector Operating Conditions:

225-250° C

See Section 503 C for other information about FPD operation.

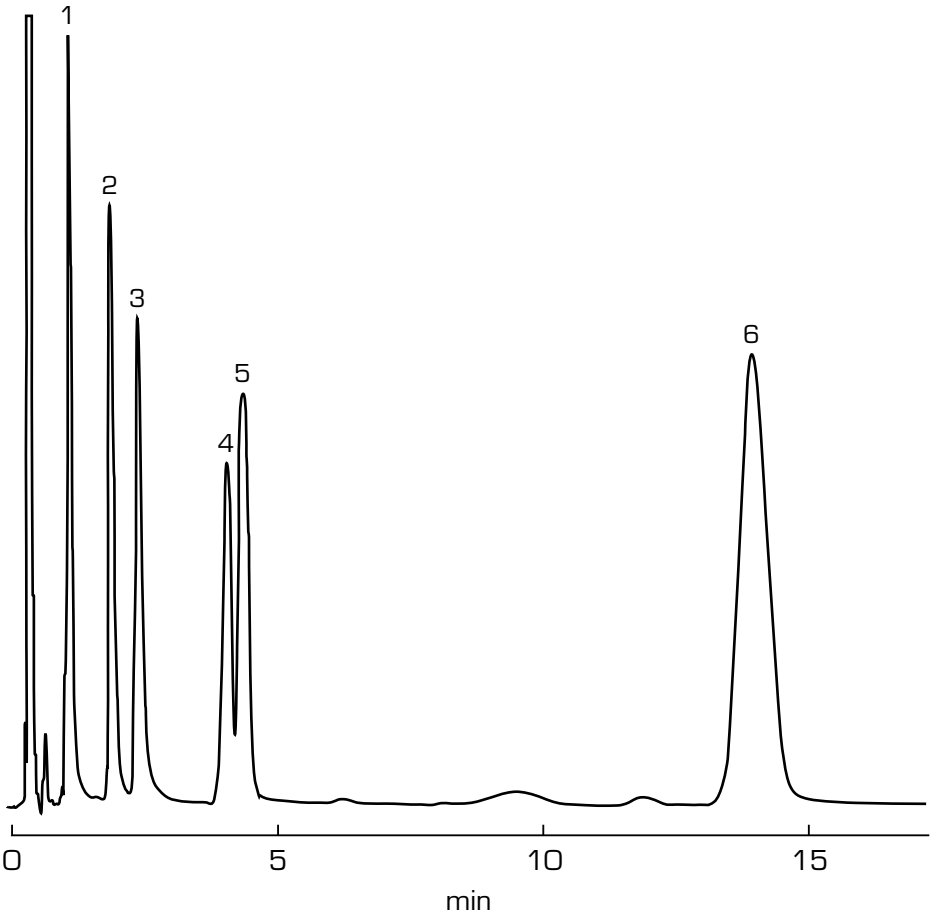
Set detector electronics (amplification, attenuation) so that response to 1.5 ng chlorpyrifos is 50% full scale deflection (FSD).

A properly functioning system will give a response to 1.5 ng omethoate of ≥50% FSD. ◀

Other Considerations

Rrt_cs and ng required to cause 50% FSD response are listed in Appendix I, PESTDATA (many data in PESTDATA were collected using equivalent packed column).

Example chromatogram is on next page.



DG14

Chromatogram of: 1) 1.0 ng acephate, 2) 1.5 ng omethoate, 3) 1.0 ng monocrotophos, 4) 1.0 ng pirimiphos-methyl, 5) 1.0 ng chlorpyrifos, and 6) 3.0 ng ethion at the conditions described.

DG17 GLC, 50% PHENYL, 50% METHYL SILOXANE, 200° C, N/P

Applicability

Determinative step is applicable to residues containing nitrogen. It is particularly useful for residues such as triazines, triazoles, and THPI (captan metabolite).

Column

Wide bore capillary, 30 m m × 0.53 mm id, coated with 50% phenyl, 50% methyl substituted polysiloxane liquid phase, 1-1.5 µm film thickness, bonded and cross-linked. For example, DB-17; see Table 502-a for equivalents. See Section 502 C, Recommended Operating Procedure for Wide Bore Columns.

Column Operating Conditions:

200° C, isothermal; if necessary, adjust temperature so that relative retention time to chlorpyrifos (rrt_c) of ethion is 3.36±0.07.

Carrier gas: helium; adjust flow rate so that chlorpyrifos elutes in about 4±0.5 min (about 20 mL/min).

Injector temperature: 250° C

Detector

Alkali bead detector, nitrogen selective (N/P)

Detector Operating Conditions:

250° C

3.7±0.1 mL/min hydrogen and 110 mL/min air

See Section 503 E for other information about N/P detector operation.

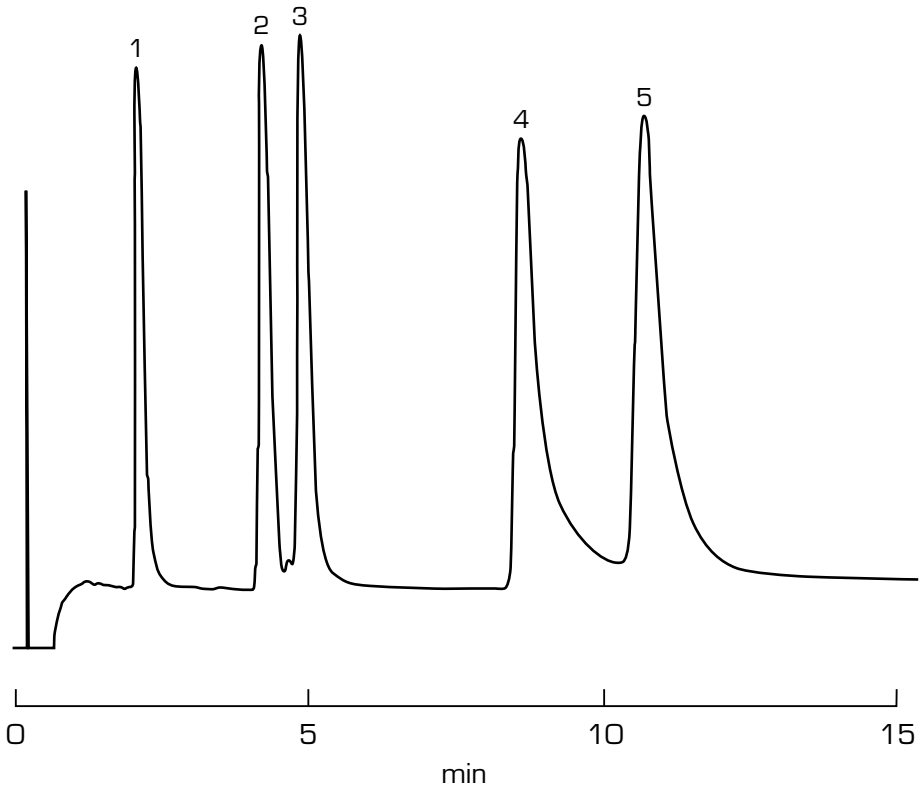
Set detector electronics (amplification, attenuation) so that response to 1.5 ng chlorpyrifos is 50% full scale deflection (FSD).

A properly functioning system will give a response to 1.5 ng omethoate of ≥50% FSD. ◀

Other Considerations

Rrt_cs and ng required to cause 50% FSD response are listed in Appendix I, PESTDATA (many data in PESTDATA were collected using equivalent packed column).

Example chromatogram is on next page.



DG17

Chromatogram of: 1) 1.5 ng atrazine, 2) 1.5 ng chlorpyrifos, 3) 15.0 ng carbaryl, 4) 10.0 ng imazalil, and 5) 5.0 ng procyazine at the conditions described.

DG20 GLC, DEGS, 180° C, FPD-P

NOTICE: Because DEGS column packing is no longer available commercially, FDA laboratories may use it for confirmatory analyses only as of Nov., 1997. ◀

Applicability

Determinative step is applicable to residues containing phosphorus. It is particularly useful for residues such as polar organophosphate pesticides and their metabolites.

Column

4' × 2 mm id 2% DEGS (stabilized) on Chromosorb W AW, 80/100 mesh. Packing is no longer commercially available but is still used in some laboratories.

Column Operating Conditions:

180° C, isothermal; if necessary, adjust temperature so that relative retention time to chlorpyrifos (rrt_c) of parathion is 2.50±0.05.

Carrier gas: nitrogen/helium; adjust flow rate so that chlorpyrifos elutes in about 2-2.5 min (about 30 mL/min).

Injector Temperature: 190° C (not more than 10° C above column temperature)

Column Conditioning: With column disconnected from detector, degas column with nitrogen at 60 mL/min for 0.5 hr. After degassing, program temperature at 1-2° C/min to 230° C. Condition with carrier flow at 230° C for 16 hr.

Detector

Flame photometric, phosphorus mode (FPD-P)

Detector Operating Conditions:

225-250° C

See Section 503 C for other information about FPD operation.

Set detector electronics (amplification, attenuation) so that response to 1.5 ng chlorpyrifos is 50% full scale deflection (FSD).

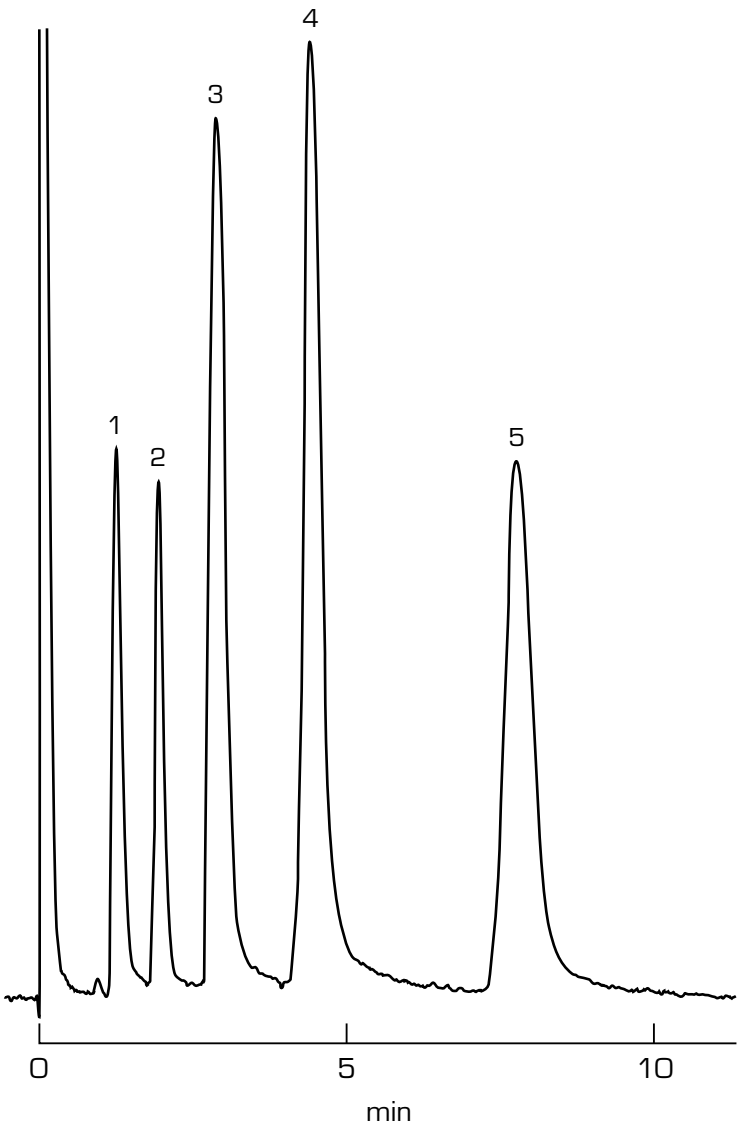
Other Considerations

When using a DEGS column, it is necessary to ascertain that polar chemicals are chromatographing properly. Adjust the system of DEGS column and FPD-P detector to result in a peak of 50% FSD for 1.5 ng chlorpyrifos. A DEGS column on which polar chemicals chromatograph properly will produce the same size peak in response to ≤8 ng monocrotophos. Do not use a DEGS column on which monocrotophos cannot be seen.

Rrt_cs and ng required to cause 50% FSD response are listed in Appendix I, PESTDATA.

DEGS columns may not tolerate injection of extracts from fatty foods; use caution if such determination is necessary.

Example chromatogram is on next page.



DG20

Chromatogram of: 1) 0.73 ng methamidophos, 2) 1.35 ng chlorpyrifos, 3) 3.72 ng acephate, 4) 7.12 ng omethoate, and 5) 5.40 ng monocrotophos.

DG21 GLC, DEGS, 180° C, FPD-S

NOTICE: Because DEGS column packing is no longer available commercially, FDA laboratories may use it for confirmatory analyses only as of Nov., 1997. ◀

Applicability

Determinative step is applicable to residues containing sulfur. It is particularly useful for residues such as polar organothiophosphate pesticides and their metabolites.

Column

4' × 2 mm id 2% DEGS (stabilized) on Chromosorb W AW, 80/100 mesh. Packing is no longer commercially available but is still used in some laboratories.

Column Operating Conditions:

180° C, isothermal; if necessary, adjust temperature so that relative retention time to chlorpyrifos (rrt_c) of parathion is 2.50±0.05.

Carrier gas: nitrogen/helium; adjust flow rate so that chlorpyrifos elutes in about 2-2.5 min (about 30 mL/min).

Injector Temperature: 190° C (not more than 10° C above column temperature)

Column Conditioning: With column disconnected from detector, degas column with nitrogen at 60 mL/min for 0.5 hr. After degassing, program temperature at 1-2° C/min to 230° C. Condition with carrier flow at 230° C for 16 hr.

Detector

Flame photometric, sulfur mode (FPD-S)

Detector Operating Conditions:

225-250° C

See Section 503 C for other information about FPD operation.

Set detector electronics (amplification, attenuation) to produce greatest possible response (50% full scale deflection [FSD] to 15 ng chlorpyrifos is reasonable).

Other Considerations

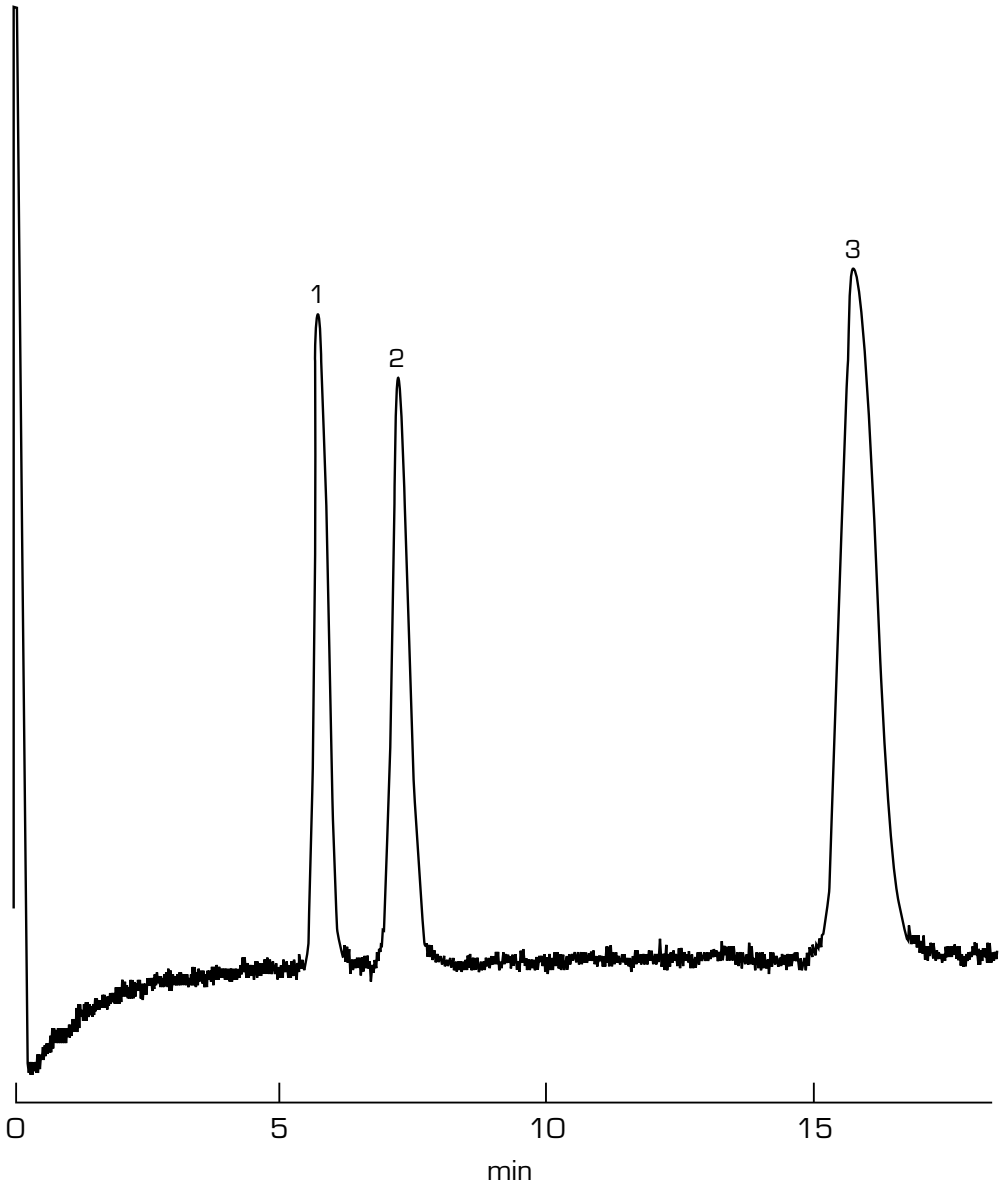
When using a DEGS column, it is necessary to ascertain that polar chemicals are chromatographing properly. Adjust the system of DEGS column and FPD-P detector to result in a peak of 50% FSD for 1.5 ng chlorpyrifos. A DEGS column on which polar chemicals chromatograph properly will produce the same size peak in response to ≤8 ng monocrotophos. Do not use a DEGS column on which monocrotophos cannot be seen.

Detector is not linear; quantitation of residues may be calculated from calibration curve (response *vs* amount injected).

Rrt_cs and ng required to cause 50% FSD response are listed in Appendix I, PESTDATA.

DEGS columns may not tolerate injection of extracts from fatty foods; use caution if such determination is necessary.

Example chromatogram is on next page.



DG21

Chromatogram of: 1) 14.4 ng ethofumesate, 2) 12.6 ng metribuzin, and 3) 48.8 ng propargite at the conditions described, except that the detector was at 200° C. Using this system, 1.64 ng chlorpyrifos caused 37% FSD response.

DG22 GLC, DEGS, 180° C, ELCD-X

NOTICE: Because DEGS column packing is no longer available commercially, FDA laboratories may use it for confirmatory analyses only as of Nov., 1997. ◀

Applicability

Determinative step is applicable to residues containing halogen. It is particularly useful for residues such as oxadiazon, which co-elutes with dieldrin on some other columns. This system also separates p,p′ -DDE from dieldrin when they occur in the same extract, *e.g.*, analysis of root crops with Section 302.

Column

4′ × 2 mm id 2% DEGS

Column Operating Conditions:

180° C, isothermal

Carrier gas: nitrogen/helium; adjust flow rate so that chlorpyrifos elutes in about 2-2.5 min (about 30 mL/min).

Injector Temperature: 190° C (not more than 10° C above column temperature)

Column Conditioning: With column disconnected from detector, degas column with nitrogen at 60 mL/min for 0.5 hr. After degassing, program temperature at 1-2° C/min to 230° C. Condition with carrier flow at 230° C for 16 hr.

Detector

Electroconductivity, halogen mode (ElCD-X)

Detector Operating Conditions:

base temperature 250° C, furnace temperature 900° C (or as specified in operating manual)

Reactor gas: hydrogen, flow as required by specific detector model; see operating manual

See Section 503 D for other information about ElCD operation.

Set detector electronics (amplification, attenuation) so that response to 1.5 ng chlorpyrifos is 50% full scale deflection (FSD).

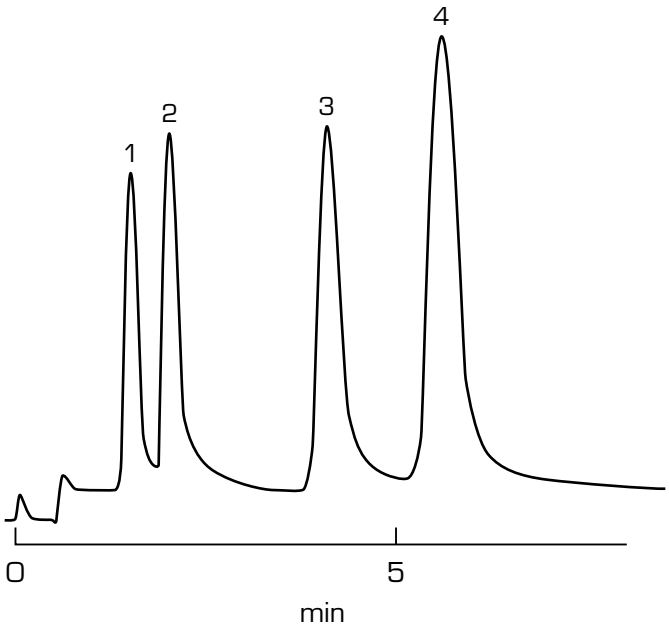
Other Considerations

When using a DEGS column, it is necessary to ascertain that polar chemicals are chromatographing properly. Adjust the system of DEGS column and FPD-P detector to result in a peak of 50% FSD for 1.5 ng chlorpyrifos. A DEGS column on which polar chemicals chromatograph properly will produce the same size peak in response to ≤8 ng monocrotophos. Do not use a DEGS column on which monocrotophos cannot be seen.

Rrt_cs and ng required to cause 50% FSD response are listed in Appendix I, PESTDATA.

DEGS columns may not tolerate injection of extracts from fatty foods; use caution if such determination is necessary.

Example chromatogram is on next page.



DG22

Chromatogram of: 1) 1.11 ng trichloronat, 2) 1.71 ng ronnel oxygen analog, 3) 4.06 ng oxadiazon, and 4) 4.59 ng procymidone at the conditions described.

DG23 GLC, DEGS, 180° C, N/P

NOTICE: Because DEGS column packing is no longer available commercially, FDA laboratories may use it for confirmatory analyses only as of Nov., 1997. ◀

Applicability

Determinative step is applicable to residues containing nitrogen. It is particularly useful for polar residues such as carbamates and organophosphate pesticide metabolites.

Column

4′ ×2 mm id 2% DEGS (stabilized) on Chromosorb W AW, 80/100 mesh. Packing is no longer commercially available but is still used in some laboratories.

Column Operating Conditions:

180° C, isothermal; if necessary, adjust temperature so that relative retention time to chlorpyrifos (rrt_c) of parathion is 2.50±0.05.

Carrier gas: nitrogen/helium; adjust flow rate so that chlorpyrifos elutes in about 2-2.5 min (about 30 mL/min).

Injector Temperature: 190° C (not more than 20° C above column temperature)

Column Conditioning: With column disconnected from detector, degas column with nitrogen at 60 mL/min for 0.5 hr. After degassing, program temperature at 1-2° C/min to 230° C. Condition with carrier flow at 230° C for 16 hr.

Detector

Alkali bead detector, nitrogen selective (N/P)

Detector Operating Conditions:

250° C

See Section 503 E for other information about N/P detector operation.

Set detector electronics (amplification, attenuation) so that response to 1.5 ng chlorpyrifos is 50% full scale deflection (FSD).

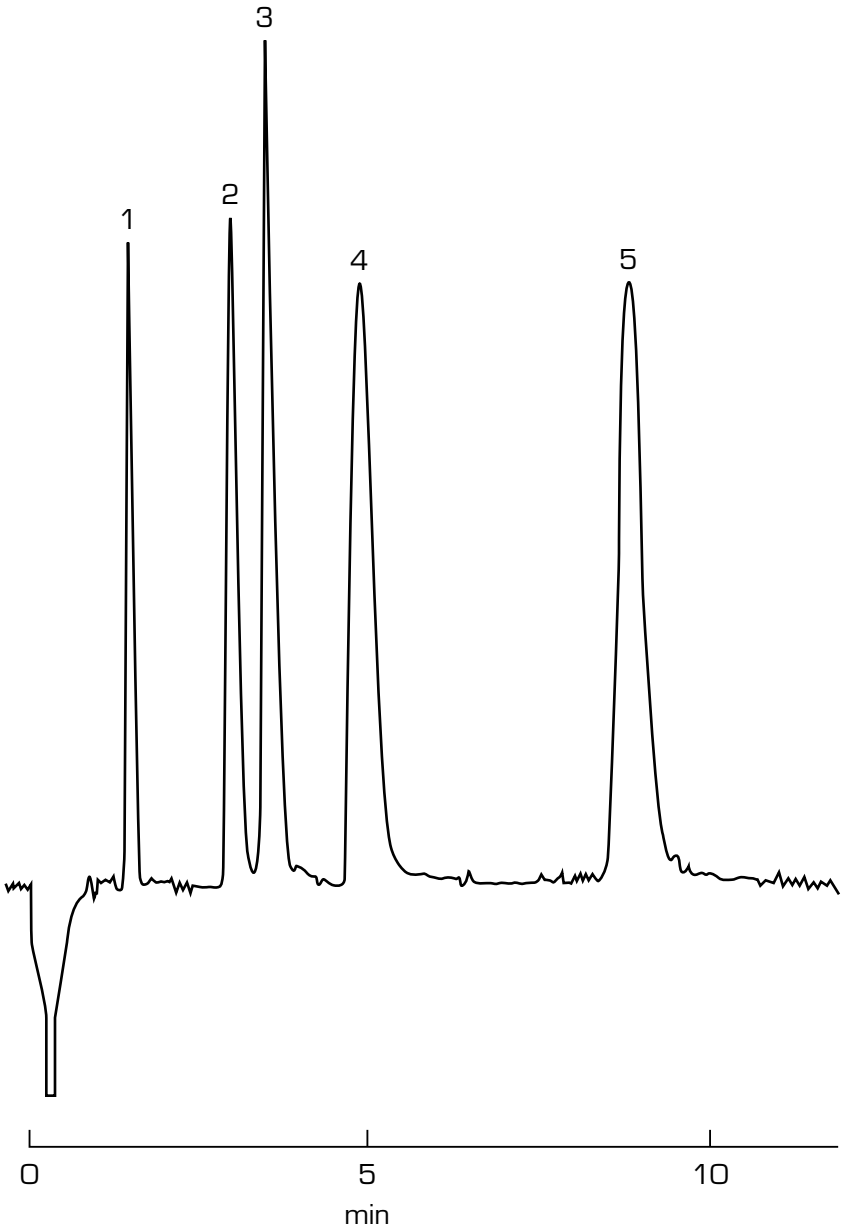
Other Considerations

When using a DEGS column, it is necessary to ascertain that polar chemicals are chromatographing properly. Adjust the system of DEGS column and FPD-P detector to result in a peak of 50% FSD for 1.5 ng chlorpyrifos. A DEGS column on which polar chemicals chromatograph properly will produce the same size peak in response to ≤8 ng monocrotophos. Do not use a DEGS column on which monocrotophos cannot be seen.

Rrt_cs and ng required to cause 50% FSD response are listed in Appendix I, PESTDATA.

DEGS columns may not tolerate injection of extracts from fatty foods; use caution if such determination is necessary.

Example chromatogram is on next page.



DG23

Chromatogram of: 1) 1.5 ng diphenylamine, 2) 1.5 ng chlorpyrifos, 3) 2.05 ng aminocarb, 4) 3.25 ng oxythioquinox, and 5) 7.5 ng napropamide at the conditions described.

PROTOCOL C: PROCEDURE FOR DEVELOPING GLC DATA

BACKGROUND

Methods: Section 302 DG1-DG23; GLC systems are used with Sections 302, 303, 304, and 402 methods.

Chemical Type: Applicable to chemicals that can be vaporized at temperatures about 250° C without degradation. Most pesticides and their related chemicals that meet this criterion can be chromatographed and detected by at least one of the GLC systems DG1-DG23.

PAM I Tables: Appendix I (PESTDATA)

DATA DEVELOPMENT

For each GLC DG module tested:

- Dissolve reference standard in pesticide grade solvent to prepare stock standard solution. Isooctane is preferred, but acetone may be required for dissolution.
- Set up GLC system as described in specified DG module (Section 302). Check rrts of marker compounds and adjust column temperature to match conditions specified.
- Inject aliquots of test solution into GLC.
- Calculate retention time (relative to marker compound specified in DG module).
- Calculate ng standard that causes 50% FSD response. Do not inject >1000 ng (1 μg).
- Test chemical on one or more of these systems:

  Level I:

  All chemicals:

  DG 1 100% methyl siloxane (*e.g.*, DB-1), 200° C, EC

  DG13 50% phenyl, 50% methyl siloxane(*e.g.*, DB-17), 200° C, EC

  DG18 50% cyanopropylphenyl, 50% methyl siloxane (*e.g.*, DB-225), 200° C, EC

  Chemicals containing halogen:

  DG 3 100% methyl siloxane (*e.g.*, DB-1), 200° C, ElCD-X

  DG16 50% phenyl, 50% methyl siloxane (*e.g.*, DB-17), 200° C, ElCD-X

Chemicals containing phosphorus:

DG 2 100% methyl siloxane (*e.g.*, DB-1), 200° C, FPD-P

DG14 50% phenyl, 50% methyl siloxane (*e.g.*, DB-17), 200° C, FPD-P

DG19 50% cyanopropylphenyl, 50% methyl siloxane (*e.g.*, DB-225), 200° C, FPD-P

▶

Chemicals containing sulfur:

DG15 50% phenyl, 50% methyl siloxane (*e.g.*, DB-17), 200° C, FPD-S

▶

Chemicals containing nitrogen:

DG 4 100% methyl siloxane (*e.g.*, DB-1), 200° C, ElCD-N

DG 5 100% methyl siloxane (*e.g.*, DB-1), 200° C, N/P

DG17 50% phenyl, 50% methyl siloxane (*e.g.*, DB-17), 200° C, N/P

▶

Chemicals with no heteroatom to which element-selective detectors respond:

DG 6 100% methyl siloxane (*e.g.*, DB-1), 130° C, FID

Level II:

If chemical chromatographs on system described in module(s) of Level I, but rrt is <0.3, rechromatograph at lower column temperature, *e.g.*:

DG 7 100% methyl siloxane (*e.g.*, DB-1), 130° C, EC

DG 8 100% methyl siloxane (*e.g.*, DB-1), 130° C, FPD-P

DG 9 100% methyl siloxane (*e.g.*, DB-1), 130° C, ElCD-X

If chemical chromatographs on system described in module(s) of Level I, but rrt is >5.0, rechromatograph at higher column temperature, *e.g.*:

DG10 100% methyl siloxane (*e.g.*, DB-1), 230° C, EC

DG11 100% methyl siloxane (*e.g.*, DB-1), 230° C, FPD-P

DG12 100% methyl siloxane (*e.g.*, DB-1), 230° C, ElCD-X

REPORTING RESULTS

Report results for each DG module on copy of Reporting Form C. An asterisk (*) appears on form wherever name of tested chemical should be entered.

Pesticide Analytical Manual Volume I
10/1999 Revisions

The following pages contain corrections or changes for PAM I. Print these pages and use them to replace the same current pages in the current PAM I 3rd edition (published 1/94, revised 9/96 and 10/99).

Each set of two pages is intended to appear on two sides of the same paper. Different versions of Acrobat Reader vary in their ability to print on both sides of the page. It may be necessary to print one page at a time and turn the paper over to print the second page on the reverse side.

This transmittal does not include updated tables, indices, or appendices. Those files can be found at http://vm.cfsan.fda.gov/~frf/pami2.html#tables.

Material Transmitted	PAM I Page	Page of this File
1. Title Page	i, ii (not numbered on page)	2, 3
2. Pages 205-1 to 205-2	205-1 to 205-2	4-5
3. Chapter 3 Table of Contents	300-1 to 300-6	6-11
4. Pages 302-1 to 302-70	302-1 to 302-70	12-81
8. Pages 303-1 to 303-2	303-1 to 303-2	82-83
9. Page 304-1 to 304-2	304-1 to 304-2	84-85

Explanations of changes:

Section 205, page 2, is revised to clarify the directions for weighings of reference standards based on their purity.

Pages 301-1 through 300-5 are revised to include new method modules and to remove references to the DEGS packed GLC column, which is now considered obsolete (it is no longer commercially available).

Pages 302-1through 302-70 now include three additional extraction modules (E5-E7) and one additional cleanup module (C6). Determination modules DG20-DG23 have been removed because the DEGS column is now considered obsolete.

Pages 303-2 and 304-2 have been revised to reflect changes in previous page numbers and to remove DEGS column data.

PESTICIDE ANALYTICAL MANUAL

VOLUME I: Multiresidue Methods

U.S. Department of Health and Human Services • Public Health Service
Food and Drug Administration

PESTICIDE ANALYTICAL MANUAL VOLUME I

3rd Edition, 1994

Revised, September, 1996
Revised, October, 1997
Revised, October 1999

Design and Format: R.O.W. Sciences, Inc.
under contract to U.S. Food and Drug Administration

Transmittal No. 2000-1 (10/1999)
Form FDA 2905a (6/92)

205: REFERENCE STANDARDS

The purity of reference standards and use of appropriate preparation and storage techniques for standard solutions significantly affect analytical results. Reliable and accurate data can be obtained only if correct analytical standard solutions are used for identification and quantitation. Each laboratory's quality assurance program plan (Section 206) should include an element on reference standards and standard solutions. Standard operating procedures (SOPs) should include protocols for obtaining, labeling, storing, and handling standards. This section provides rudimentary information that may be incorporated, as appropriate, into such documentation.

205 A: SOURCES

Reference standards are currently available from several commercial sources, including companies that supply only reference standards, suppliers of specialty laboratory chemicals, and suppliers of chromatographic equipment. Each company publishes lists of reference standards for pesticides, related metabolites, and certain industrial chemicals. Eligible laboratories, mainly Federal Government laboratories, may also obtain reference standards for some chemicals from a repository maintained, under contract, by EPA; eligibility is determined by EPA.

Reference standards in "neat" (undiluted) form, preferably certified by EPA, should be used whenever possible. If neat standards are not available, certified solutions of standards may be used.

205 B: EQUIPMENT AND SOLVENTS

Equipment

Equipment used for preparation and storage of reference standards and solutions includes the following essential, but not all-inclusive, items:

1) analytical balance calibrated for accuracy of ±0.05 mg

2) explosion-resistant refrigerator/freezer, used only to store standards

3) standard solution storage containers:

   a) amber colored, screw-cap bottles, 1 and 2 oz

   b) Teflon-lined caps for bottles

   c) vials for working standards

4) desiccators to store reference standards. Larger vials containing desiccant can be used as individual desiccators for vials of standards.

5) appropriate volumetric glassware, pipets, or microliter syringes

Solvents

Pesticide residue quality solvents are essential for preparation of reference standard solutions. Solvents should be checked before use for the presence of interfering substances by injecting the solvent into the determinative system(s) to be used.

Choice of solvent is sometimes restricted by solubility and stability of the particular chemical. The following solvents, in order of preference, should be used to prepare standard solutions, if suitable for the particular chemical: isooctane (2,2,4-trimethylpentane), hexane, acetone, isopropanol, and toluene.

205 C: STORAGE

Reference standards must be stored properly to prevent undesirable reactions, such as oxidation, re-arrangement, or hydrolysis. Improper storage can lead to loss of integrity of previously acceptable standards. Storage conditions must also prevent the possibility of external contamination. Storage requirements are dependent on the chemical and physical properties of the chemical of interest and are much more stringent for volatile, reactive, or unstable compounds. Review the physical and chemical properties of each compound to determine which storage conditions are appropriate. Minimum requirements for long term storage of analytical reference standards follow:

- If at all possible, store reference standards in tightly sealed containers under desiccation in a freezer.
- Store more stable compounds, such as organochlorine pesticides, in a refrigerator if freezer is not available.

Reference standards that have been stored in refrigerators or freezers must be brought to room temperature in a desiccator prior to weighing.

205 D: PURITY

The analyst is responsible for knowing the purity of the reference standard used to obtain reported data. Follow these rules for recording information about reference standard purity:

- Standards with known purity ≥99%: weight may be recorded as measured; it is not necessary to correct for purity.
- Standards with purity <99%: apply appropriate correction factor to measured weights.
- Technical standards with unknown purity (use only if this is the only available reference standard): record weight as measured, do not correct for purity, but include a note on the source and unknown purity of this standard with the results of any analysis whose results rely on this standard.

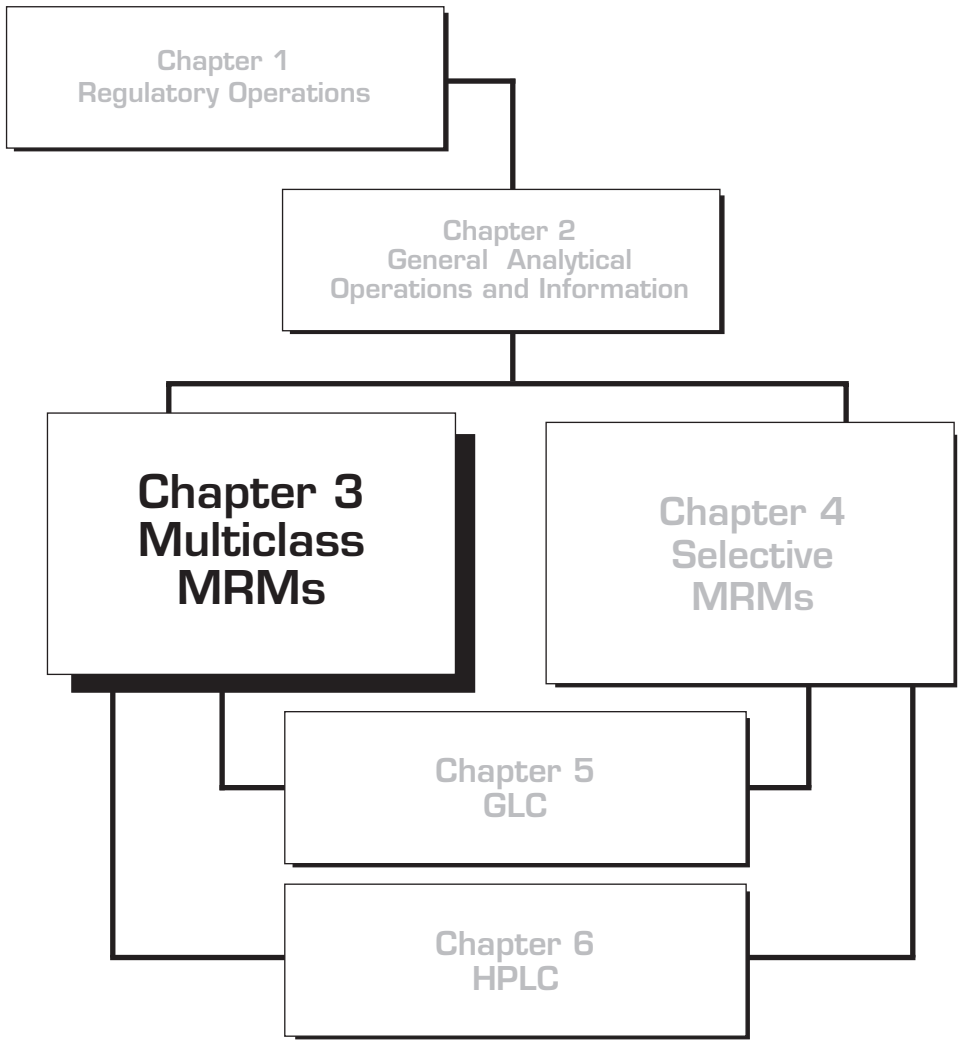


Table of Contents

Figures

Tables

302: METHOD I FOR NONFATTY FOODS

BASIC REFERENCES

Luke, M.A., *et al.* (1975) *J. Assoc. Off. Anal. Chem.* **58**, 1020-1026

Luke, M.A., *et al.* (1981) *J. Assoc. Off. Anal. Chem.* **64**, 1187-1195

GENERAL PRINCIPLES

Residues are extracted from nonfatty foods by blending with acetone or water/acetone, then transferred from the filtered aqueous extract into organic solvent. The extract is cleaned up if necessary and examined by various determinative steps; the amount of cleanup necessary is dictated by the determinative step(s) to be used and by the type of commodity being analyzed.

APPLICABILITY

Consult Guide to PAM I for additional information pertinent to the appropriate application of multiresidue methodology.

Method is applicable to nonionic residues in nonfatty foods. Cleanup steps may be needed for particularly dirty extracts or for examination by less selective detectors; some residues may be lost during cleanup. Extract is amenable to examination by many determinative steps, and the residues covered by a particular analysis are dependent on the number of different determinative steps used. See Tables 302-a and 302-b, following the method description, for results of recovery tests.

METHOD MODULES

Choose from these method modules, using Figure 302-a for guidance:

Extraction (E)			Recommended Use	
E1	(p. 302-7)	Extraction with acetone, liquid-liquid partitioning with petroleum ether/methylene chloride	nonfatty, high moisture commodities	
E2	(p. 302-9)	Extraction with acetone, removal of water with 40 g Hydromatrix	nonfatty, high moisture commodities	
E3	(p. 302-11)	Extraction with acetone, removal of water with 25 g Hydromatrix	alternative to E2 for reduction in solvent use	
E4	(p. 302-13)	Extraction with water/acetone, liquid-liquid partitioning with petroleum ether/methylene chloride	nonfatty, low moisture commodities	
E5	(p. 302-15)	Extraction with acetone, liquid-liquid partitioning with acetone/methylene chloride	alternative to E1 for relatively polar residues	◀
E6	(p. 302-16)	Extraction with water/acetone, liquid-liquid partitioning with acetone/methylene chloride	alternative to E4 for relatively polar residues	◀
E7	(p. 302-17)	Extraction with acetone and solid phase extraction cartridges, liquid-liquid partitioning	nonfatty, high moisture commodities for relatively polar residues	◀

Cleanup (C)

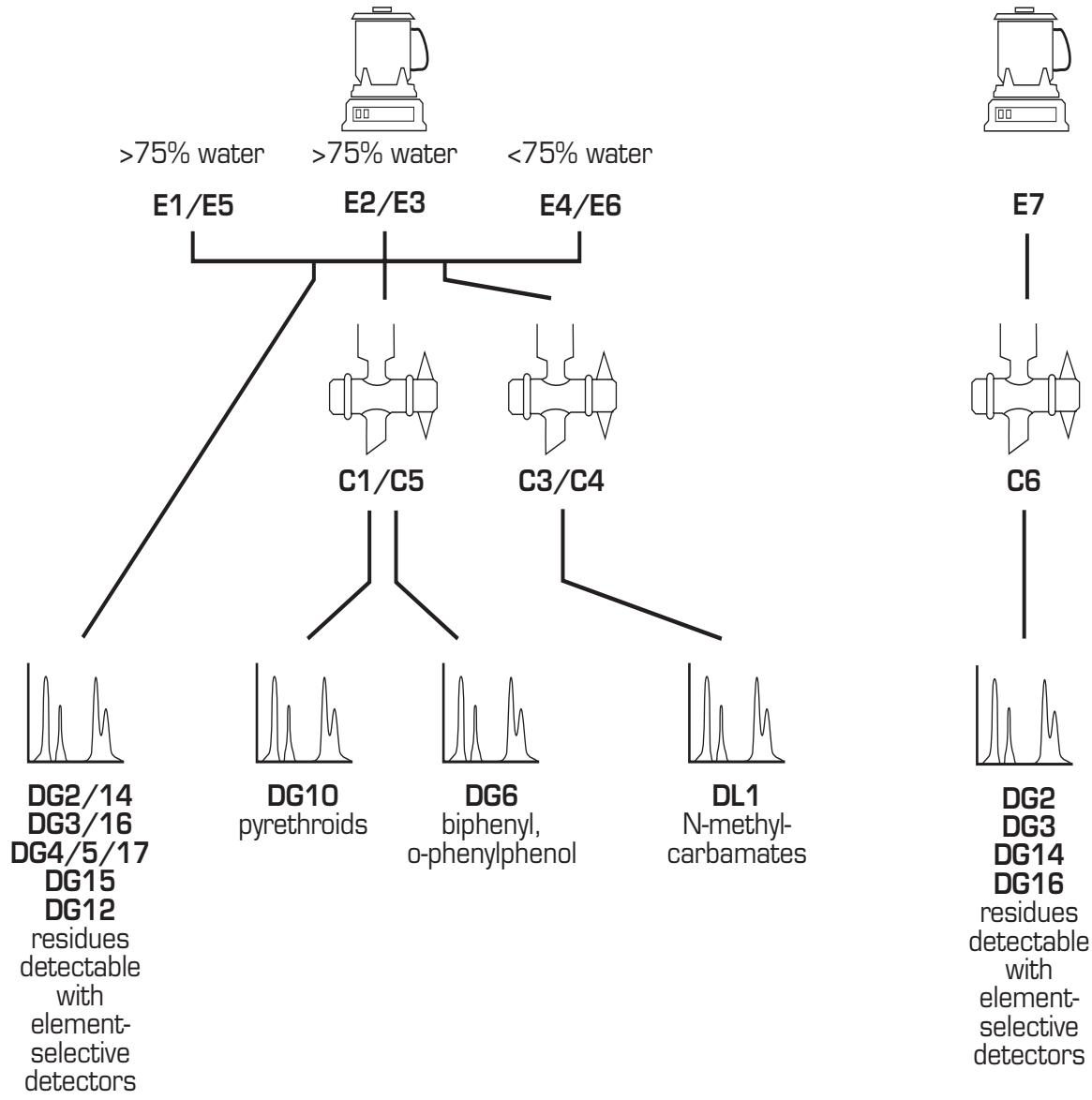
C1	(p. 302-21)	Florisil column (4 g) cleanup, with one methylene chloride eluant	relatively nonpolar residues
C2	(p. 302-23)	Charcoal/Celite/magnesium oxide column cleanup	polar residues
C3	(p. 302-25)	Charcoal/silanized Celite column cleanup	before HPLC determination for N-methylcarbamates
C4	(p. 302-27)	C-18 cartridge cleanup	before HPLC determination for N-methylcarbamates
C5	(p. 302-29)	Florisil column cleanup, with mixed ether eluants	relatively nonpolar residues
▶ **C6**	(p. 302-31)	SAX/PSA cartridge cleanup	polar and nonpolar residues

Determination (D)		**Recommended Use**	
DG 1	(p. 302-33)	GLC, 100% methyl siloxane column, 200°, EC detector	residues with halogen. sulfur, other moities
DG 2	(p. 302-35)	GLC, 100% methyl siloxane column, 200°, FPD-P	residues with phosphorus
DG 3	(p. 302-37)	GLC, 100% methyl siloxane column, 200°, ELCD-X	residues with halogen
DG 4	(p. 302-39)	GLC, 100% methyl siloxane column, 200°, ELCD-N	residues with nitrogen
DG 5	(p. 302-41)	GLC, 100% methyl siloxane column, 200°, N/P detector	residues with nitrogen or phosphorus
DG 6	(p. 302-43)	GLC, 100% methyl siloxane column, 160°, FID	biphenyl, o-phenylphenol
DG 7	(p. 302-45)	GLC, 100% methyl siloxane column, 130°, EC detector	early eluting residues with halogen, sulfur, other moieties
DG 8	(p. 302-47)	GLC, 100% methyl siloxane column, 130°, FPD-P	early eluting residues with phosphorus
DG 9	(p. 302-49)	GLC, 100% methyl siloxane column, 130°, ELCD-X	early eluting residues with halogen
DG10	(p. 302-51)	GLC, 100% methyl siloxane column, 230°, EC detector other moieties	late eluting residues with halogen, sulfur,
DG11	(p. 302-53)	GLC, 100% methyl siloxane column, 230°, FPD-P	late eluting residues with phosphorus
DG12	(p. 302-55)	GLC, 100% methyl siloxane column, 230°, ELCD-X	late eluting residues with halogen
DG13	(p. 302-57)	GLC, 50% phenyl, 50% methyl siloxane column, 200°, EC detector	residues with halogen, sulfur, other moieties

DG14 (p. 302-59)	GLC, 50% phenyl, 50% methyl siloxane column, 200°, FPD-P	residues with phosphorus
DG15 (p. 302-61)	GLC, 50% phenyl, 50% methyl siloxane column, 200°, FPD-S	residues with sulfur
DG16 (p. 302-63)	GLC, 50% phenyl, 50% methyl siloxane column, 200°, ELCD-X	residues with halogen
DG17 (p. 302-65)	GLC, 50% phenyl, 50% methyl siloxane column, 200°, N/P detector	residues with nitrogen or phosphorus
DG18 (p. 302-67)	GLC, 50% cyanopropylphenyl, 50% methyl siloxane column, 200°, EC detector	residues with halogen, sulfur, other moieties
DG19 (p. 302-69)	GLC, 50% cyanopropylphenyl, 50% methyl siloxane column, 200°, FPD-P	residues with phosphorus

►

Figure 302
Recommended Approach: Nonfatty Foods



VALIDATION

Many combinations of method modules are possible. The following combinations have undergone interlaboratory validation and are recommended for use:

E1 + DG2, DG3

Validation report:

> Sawyer, L.D. (1985) *J. Assoc. Off. Anal. Chem.* **68**, 64-71. Collaborative study lead-ing to AOAC official final action status for acephate, a-BHC, chlorpyrifos, dieldrin, monocrotophos, and omethoate in lettuce, strawberries, and tomatoes.
>
> AOAC official method reference: *Official Methods of Analysis of the AOAC* (1990) 15th ed., 985.22.

E1 + C3 + DL1

Validation report:

> Pardue, J.R. (April 1987) "Recoveries of N-Methyl Carbamates Using a Combination of the Luke (PAM I, 232.4) and Krause (PAM I, 242.24b, 242.25) Procedures," LIB 3138, FDA, Rockville, MD

E2 + C1 + [temperature programmed GLC systems equivalent to] DG1, DG7, DG10, and DG16

Validation report:

> Griffitt, K.R., and Szorik, M.M. (Sept 1989) "The Analysis of 127 Total Diet Items for Chlorinated Residues Using Luke/Solid Phase Extracts," LIB 3366, FDA, Rockville, MD

E1 EXTRACTION WITH ACETONE, LIQUID-LIQUID PARTITIONING WITH PETROLEUM ETHER/METHYLENE CHLORIDE

References

Luke, M.A., *et al.* (1975) *J. Assoc. Off. Anal. Chem.* **58**, 1020-1026

Luke, M.A., *et al.* (1981) *J. Assoc. Off. Anal. Chem.* **64**, 1187-1195

Principles

Nonfatty sample is blended with acetone and filtered. Most nonionic residues are extracted into aqueous acetone solution. Residues are transferred from aqueous acetone to methylene chloride/petroleum ether by partitioning, with salt added to aqueous layer after the first partitioning to aid transfer. Concentration step is repeated in the presence of petroleum ether to remove all traces of methylene chloride, then repeated again to produce final extract in acetone solution.

Apparatus

blender, high speed; explosion-proof Waring Blendor, 1 qt jar

Büchner funnel (Büchner), porcelain, 12 cm diameter

filter paper, Shark Skin®, to fit Büchner

long-stemmed funnel, glass, 4" diameter

Kuderna-Danish concentrator (K-D), 500 mL, with Snyder column, two-ball micro-Snyder column, graduated receiving flask

separatory funnel (separator), 1 L

Reagents

acetone, distilled from all-glass apparatus

boiling chips, 20-30 mesh carborundum

glass wool, Pyrex, see Section 204 for handling directions

methylene chloride, distilled from all-glass apparatus

petroleum ether, distilled from all-glass apparatus

sodium chloride, reagent grade

sodium sulfate, anhydrous, granular, reagent grade; see Section 204 for handling directions

Directions

- Prewash filter paper with acetone to remove contaminants.
- Weigh 100 g chopped or blended sample into blender jar, add 200 mL acetone, and blend 2 min at high speed.
- Filter with suction through 12 cm Büchner fitted with Shark Skin® paper; collect extract in 500 mL suction flask. Filtration is normally complete in <1 min. Continuation of vacuum for excessive period can reduce volume of extract and cause error in calculation.
- Place 80 mL sample extract in 1 L separator, and add 100 mL petroleum ether and 100 mL methylene chloride. Shake vigorously 1 min.
- Transfer lower aqueous layer to second 1 L separator.

- Dry upper layer of first separator by passing through about 1.5" sodium sulfate supported on washed glass wool in 4" funnel, collecting in K-D. (If extract will be cleaned up directly with C3, charcoal/Celite column, collect in vacuum rotary evaporator flask.)
- To separator with aqueous phase, add 7 g sodium chloride and shake vigorously 30 sec until most of the sodium chloride is dissolved.
- Add 100 mL methylene chloride, shake 1 min, and dry lower organic phase through same sodium sulfate.
- Extract aqueous phase with additional 100 mL methylene chloride and dry as above. Rinse sodium sulfate with about 50 mL methylene chloride.

  (If extract will be cleaned up directly with C3, proceed to concentration step described there instead of evaporating in K-D as follows.)
- Add boiling chips to K-D and concentrate solvent in K-D; start evaporation slowly by placing only receiver tube into steam. After 100-150 mL has evaporated, concentrator may be exposed to more steam. When liquid level in hot concentrator tube is about 2 mL, add 100 mL petroleum ether through Snyder column and reconcentrate to about 2 mL. Add 50 mL petroleum ether and repeat concentration step. Add 20 mL acetone, and reconcentrate to about 2 mL. Do not allow solution to go to dryness during any of the concentration steps. Adjust volume of extract to suitable definite volume with acetone.
- Calculate equivalent sample weight in final solution:

$$\frac{\text{mg sample equivalent}}{\mu\text{L final extract}} = 100 \times \frac{80}{200 + W - 10} \times \frac{1}{\text{mL final volume}}$$

where:

100 = g sample analyzed

80 = mL filtered extract taken for liquid-liquid partitioning

200 = mL acetone blended with 100 g sample

W = amount (mL) of water present in sample (Section 201; if data are not available for particular raw agricultural commodity, use 85%)

10 = adjustment for water/acetone volume contraction.

Thus, when sample contains 85% water (85 mL/100 g) and final extract volume is 7 mL, each µL contains:

$$100 \times \frac{80}{200 + 85 - 10} \times \frac{1}{7} = \frac{4.15 \text{ mg sample equivalent}}{\mu\text{L final extract}}$$

- Extract may be suitable, as is, for determination by GLC with selective detectors (*e.g.*, DG2, DG3). If co-extractives interfere with determination or adversely affect chromatography, clean up extract with C1, C2, or C5 prior to determination.
- Clean up extract with C1 or C5 prior to determination by electron capture (DG1, DG7, *etc.*) or flame ionization detectors (DG6). Clean up extract with C3 or C4 prior to determination by DL1 for N-methylcarbamates.

E2 EXTRACTION WITH ACETONE, REMOVAL OF WATER WITH 40 G HYDROMATRIX

References

Luke, M.A., *et al.* (1975) *J. Assoc. Off. Anal. Chem.* **58**, 1020-1026

Luke, M.A., *et al.* (1981) *J. Assoc. Off. Anal. Chem.* **64**, 1187-1195

Hopper, M.L. (1988) *J. Assoc. Off. Anal. Chem.* **71**, 731-734

Principles

Nonfatty sample is blended with acetone and filtered. Most nonionic residues are extracted from nonfatty foods into aqueous acetone solution. Water is removed from aqueous acetone solution by passing it through a column of specially treated diatomaceous earth (Hydromatrix). Residues are eluted from column with methylene chloride. Up to 13.3 mL water, from 40 mL aqueous acetone extractant, is adsorbed by the column, which is re-usable.

Apparatus

blender, high speed; explosion-proof Waring Blendor, 1 qt jar

Büchner funnel (Büchner), porcelain, 12 cm diameter

filter paper, Shark Skin®, to fit Büchner

chromatographic column, 25 mm id × 500 mm, Teflon stopcock

long-stemmed funnel, glass, 4" diameter

powder funnel, glass, 4" diameter

Kuderna-Danish concentrator (K-D), 500 mL, with Snyder column, two-ball micro-Snyder column, graduated receiving flask

sieve, No. 30

Reagents

acetone, distilled from all-glass apparatus

buffer solution: 0.1 M (13.6 g/L) potassium phosphate monobasic (KH_2PO_4) in water

Hydromatrix material (pelletized diatomaceous earth), Part No. 0019-8003, Analytichem International, Harbor City, CA; also available through Varian

methylene chloride, distilled from all-glass apparatus

potassium phosphate monobasic, certified ACS grade

wire gauze, 40 mesh stainless steel

Directions

- Prepare Hydromatrix column:
  - Cut two pieces stainless steel gauze into circles of diameter slightly larger than chromatographic column id. Place one circle in bottom of column.
  - Place 50 g Hydromatrix material on No. 30 sieve and sieve thoroughly to remove fines.

  - Pour 40 g sieved Hydromatrix material into column with aid of powder funnel. Tap end of column lightly on benchtop to settle material. Place second stainless steel gauze circle on top of material in column.
  - With stopcock fully open, wash column with 150 mL buffer solution.
  - After buffer solution has passed into column and flow has slowed to 3-5 mL/min, wash column with 300 mL acetone. Adjust flow to 50-60 mL/min after first 100 mL acetone has eluted.
  - Wash column with 300 mL methylene chloride. Re-adjust flow to 50-60 mL/min after first 100 mL methylene chloride has eluted.

- Prewash filter paper with acetone to remove artifacts.
- Weigh 100 g chopped or blended sample into blender jar, add 200 mL acetone, and blend 2 min at high speed.
- Filter with suction through 12 cm Büchner fitted with Shark Skin® paper; collect extract in 500 mL suction flask. Filtration is normally complete in <1 min. Continuation of vacuum for excessive period can reduce volume of extract and cause error in calculation.
- Prewash Hydromatrix column with 200 mL acetone followed by 200 mL methylene chloride immediately before each use. Discard wash solvents.
- Place K-D under column. (If extract will be cleaned up directly with C3, charcoal/Celite column, collect in vacuum rotary evaporator flask.) Trans fer 40 mL filtered acetone extract to top of column. Let extract pass into column until flow rate has slowed to <1 mL/min. Let column equilibrate 3 min at <1 mL/min.
- Add 50 mL methylene chloride to column. After that has passed into column, add another 50 mL methylene chloride. After that has passed into column, add another 200 mL methylene chloride.
- Collect eluate until flow rate has decreased to slow drip (about 1 mL/min). Total elution time is 6-8 min.

  (If extract will be cleaned up directly with C3, proceed to concentration step described there instead of evaporating in K-D as follows.)
- Add boiling chips to K-D and concentrate solvent in K-D; start evaporation slowly by placing only receiver tube into steam. After 100-150 mL has evaporated, concentrator may be exposed to more steam. When liquid level in hot concentrator tube is about 2 mL, add 100 mL petroleum ether through Snyder column and reconcentrate to about 2 mL. Add 50 mL petroleum ether and repeat concentration step. Add 20 mL acetone, and reconcentrate to about 2 mL. Do not allow solution to go to dryness during any of the concentration steps. Adjust volume of extract to suitable definite volume with acetone.
- If extract will be cleaned up directly with C1, Florisil column, it is not necessary to reconcentrate repeatedly (as above) to remove all traces of methylene chloride. Instead, add boiling chips and concentrate solvent in K-D to <5 mL. Without allowing K-D to cool, add 50 mL acetone through Snyder column, and reconcentrate to suitable definite volume; allow to cool.

- Calculate equivalent sample weight in final solution:

$$\frac{\text{mg sample equivalent}}{\mu\text{L final extract}} = 100 \times \frac{40}{200 + W - 10} \times \frac{1}{\text{mL final volume}}$$

where:

100 = g sample analyzed

40 = mL filtered extract taken for Hydromatrix partitioning

200 = mL acetone blended with 100 g sample

W = amount (mL) of water present in sample (Section 201; if data are not available for particular raw agricultural commodity, use 85%)

10 = adjustment for water/acetone volume contraction.

Thus, when sample contains 85% water (85 mL/100 g) and final extract volume is 5 mL, each µL contains:

$$100 \times \frac{40}{200 + 85 - 10} \times \frac{1}{5} = \frac{2.9 \text{ mg sample equivalent}}{\mu\text{L final extract}}$$

- Extract may be suitable, as is, for determination by GLC with selective detectors (*e.g.*, DG2, DG3). If co-extractives interfere with determination or adversely affect chromatography, clean up extract with C1, C2, or C5 prior to determination.
- Clean up extract with C1 or C5 prior to determination by electron capture (DG1, DG7, *etc.*) or flame ionization detectors (DG6). Clean up extract with C3 or C4 prior to determination by DL1 for N-methyl-carbamates.
- Re-use Hydromatrix column without further rinsing, unless any adsorbed color elutes from column (after about 20 uses). When this occurs, restore column as follows:
  - Do not change stopcock setting. Flow rate will change due to different solvent densities, but this is of no consequence.
  - Wash column with 200 mL acetone, followed by sufficient volume (200-300 mL) buffer solution to remove any color left on column. Once color has been removed, elute with 300 mL acetone followed by 200 mL methylene chloride. Column is now ready for re-use.

ALTERNATIVE:

E3 EXTRACTION WITH ACETONE, REMOVAL OF WATER WITH 25 G HYDROMATRIX

Reference

Palmer, R.E., and Hopper, M.L. (Nov. 1991) "Miniaturized Solid Phase Partition Column for Determination of Organochlorine and Organophosphate Pesticides with PAM I 232.4 (Luke procedure) Acetone Filtrate," LIB 3613, FDA, Rockville, MD

Principles

Smaller size column of Hydromatrix reduces solvent use by 40% over E2, while still removing water from same amount of extract. However, solution eluting from 25 g Hydromatrix column may be cloudy, probably from a small amount of water; this disappears during concentration. The 25 g column may also have a shorter lifetime than the 40 g column. Results using the 25 g column may be somewhat less reliable for certain chemicals; *e.g.*, p,p'-dicofol and dicloran are recovered less reproducibly, and >0.4 ppm methamidophos may be only partially recovered; elution with 300 mL methylene chloride permits complete recovery of the latter.

Directions

- Follow directions of E2, except:
  - Prepare Hydromatrix column from 25 g material instead of 40 g.
  - Prewash Hydromatrix column with 100 mL acetone followed by 100 mL methylene chloride immediately before each use.
  - After transferring 40 mL filtered acetone extract to top of column, elute with 25, 25, and 150 mL methylene chloride, instead of volumes used in E2.
  - Because amount of original sample and amount of filtered acetone extract transferred to Hydromatrix column are the same as in E2, mg sample equivalent is the same as E2.

E4 EXTRACTION WITH WATER/ACETONE, LIQUID-LIQUID PARTITIONING WITH PETROLEUM ETHER/METHYLENE CHLORIDE

Reference

Luke, M.A., and Doose, G.M. (1983) *Bull. Environ. Contam. Toxicol.* **30**, 110-116

Principles

Low moisture nonfatty sample is blended with 35% water/acetone and filtered; the presence of water in the extractant facilitates extraction of residues from the dry product and dilutes co-extractives. Most nonionic residues are extracted into aqueous acetone solution. Residues are transferred from aqueous acetone to organic solvent methylene chloride/petroleum ether by partitioning, with salt added to the aqueous layer after the first partitioning to aid transfer.

Apparatus

blender, high speed; explosion-proof Waring Blendor, 1 qt jar

Büchner funnel (Büchner), porcelain, 12 cm diameter

filter paper, Shark Skin®, to fit Büchner

long-stemmed funnel, glass, 4" diameter

grinder, suitable for reducing dry products to <20 mesh

Kuderna-Danish concentrator (K-D), 500 mL, with Snyder column, two-ball micro-Snyder column, graduated receiving flask

separatory funnel (separator), 1 L

Reagents

acetone, distilled from all-glass apparatus

boiling chips, 20-30 mesh carborundum (optional)

glass wool, Pyrex; see Section 204 for handling directions

methylene chloride, distilled from all-glass apparatus

petroleum ether, distilled from all-glass apparatus

sodium chloride, reagent grade

sodium sulfate, anhydrous, granular, reagent grade; see Section 204 for handling directions

35% (v/v) water/acetone

Directions

- Prewash filter paper with acetone to remove artifacts.
- Grind sample containing <10% fat or oil to <20 mesh.
- Weigh 15 g ground sample into blender jar, add 350 mL 35% water/acetone, and blend 2 min at high speed.
- Filter with suction through 12 cm Büchner fitted with Shark Skin® paper; collect extract in 500 mL suction flask. Filtration is normally complete in <1 min. Continuation of vacuum for excessive period can reduce volume of extract and cause error in calculation.
- Place 80 mL sample extract in 1 L separator containing 100 mL methylene chloride. Add 100 mL petroleum ether and shake vigorously 1 min.

- Transfer lower aqueous layer to second 1 L separator.
- Dry upper organic layer of first separator by passing through about 1.5" sodium sulfate supported on washed glass wool in 4" funnel, collecting in K-D. (If extract will be cleaned up directly with C3, charcoal/Celite column, collect in vacuum rotary evaporator flask.)
- To separator with aqueous phase, add 7 g sodium chloride and shake vigorously 30 sec until most of the sodium chloride is dissolved.
- Add 100 mL methylene chloride, shake 1 min, and dry lower organic phase through same sodium sulfate.
- Extract aqueous phase with additional 100 mL methylene chloride and dry as above. Rinse sodium sulfate with about 50 mL methylene chloride.

  (If extract will be cleaned up directly with C3, proceed to concentration step described there instead of evaporating in K-D as follows.)
- Add boiling chips to K-D and concentrate solvent in K-D; start evaporation slowly by placing only receiver tube into steam. After 100-150 mL has evaporated, concentrator may be exposed to more steam. When liquid level in hot concentrator tube is about 2 mL, add 100 mL petroleum ether through Snyder column and reconcentrate to about 2 mL. Add 50 mL petroleum ether and repeat concentration step. Add 20 mL acetone, and reconcentrate to about 2 mL. Do not allow solution to go to dryness during any of the concentration steps. Adjust volume of extract to suitable definite volume with acetone.
- Calculate equivalent sample weight in final solution:

$$\frac{\text{mg sample equivalent}}{\mu\text{L final extract}} = 15 \times \frac{80}{350} \times \frac{1}{\text{mL final volume}}$$

  where:

  15 = g sample analyzed

  80 = mL filtered extract taken for liquid-liquid partitioning

  Thus, when final extract volume is 2 mL, each µL contains:

$$15 \times \frac{80}{350} \times \frac{1}{2} = \frac{1.7 \text{ mg sample equivalent}}{\mu\text{L final extract}}$$

- Extract may be suitable, as is, for determination by GLC with selective detectors (*e.g.*, DG2, DG3). If co-extractives interfere with determination or adversely affect chromatography, clean up extract with C1, C2, or C5 prior to determination.
- Clean up extract with C1 or C5 prior to determination by electron capture (DG1, DG7, *etc.*) or flame ionization detectors (DG6). Clean up extract with C3 or C4 prior to determination by DL1 for N-methylcarbamates.

ALTERNATIVE: ◀

E5 EXTRACTION WITH ACETONE, LIQUID-LIQUID PARTITIONING WITH ACETONE/METHYLENE CHLORIDE

Reference

Luke, M. A., and Doose, G. M. (1983) *Bull. Environ. Contam. Toxicol.* **30**, 110-116

Principle

Polar pesticides such as methamidophos exhibit variable recoveries when petroleum ether/dichloromethane is used in partitioning. Better recoveries are obtained when acetone is substituted for petroleum ether. Transfer of polar pesticides from the aqueous phase to the organic layer is further facilitated by adding sodium chloride before, rather than after, the first partitioning step.

Directions

- Follow directions of E1 through blending and filtering. Then:
  - Place 80 mL sample extract in 1 L separator, and add 100 mL acetone, 100 mL methylene chloride, and 7 g sodium chloride. Shake vigorously 1 min.
  - Transfer lower aqueous layer to second 1 L separator.
  - Dry upper organic layer of first separator by passing through about 1.5" sodium sulfate supported on washed glass wool in 4" funnel, collecting in K-D. (If extract will be cleaned up directly with C3, charcoal/ silanized Celite column, collect in vacuum rotary evaporator flask.)
  - Add 100 mL methylene chloride, shake 1 min, and dry lower organic phase through same sodium sulfate.
- Continue as in E1, "Extract aqueous phase with additional 100 mL methylene chloride..."

ALTERNATIVE: ◀

E6 EXTRACTION WITH WATER/ACETONE, LIQUID-LIQUID PARTITIONING WITH ACETONE/METHYLENE CHLORIDE

Reference

Luke, M. A., and Doose, G. M. (1983) *Bull. Environ. Contam. Toxicol.* **30**, 110-116

Principle

Polar pesticides such as methamidophos exhibit variable recoveries when petroleum ether/methylene chloride is used in partitioning. Better recoveries are obtained when acetone is substituted for petroleum ether. Transfer of polar pesticides from the aqueous phase to the organic layer is further facilitated by adding sodium chloride before, rather than after, the first partitioning step.

Directions

- Follow directions of E4 through blending and filtering. Then:
  - Place 80 mL sample extract in 1 L separator containing 100 mL methylene chloride. Add 100 mL acetone and 7 g sodium chloride and shake vigorously 1 min.
  - Transfer lower aqueous layer to second 1 L separator.
  - Dry upper organic layer of first separator by passing through about 1.5" sodium sulfate supported on washed glass wool in 4" funnel, collecting in K-D. (If extract will be cleaned up directly with C3, charcoal/silanized Celite column, collect in vacuum rotary evaporator flask.)
  - Add 100 mL methylene chloride, shake 1 min, and dry lower organic phase through same sodium sulfate.
- Continue as in E4, "Extract aqueous phase with additional 100 mL methyl ene chloride..."

E7 EXTRACTION WITH ACETONE AND SOLID PHASE EXTRACTION CARTRIDGES, LIQUID-LIQUID PARTITIONING

Reference

Luke, M. A., *et al.* (Sept. 1994) "An Improved Variation of the Luke Multiresidue Pesticide Procedure for the Analysis of Fruits and Vegetables Using Solid Phase Extraction Cartridges and Element Selective Gas Chromatographic Detectors," LIB 3896, FDA, Rockville, MD

Apparatus

blender, high speed; explosion-proof Waring Blendor, 1 qt jar

Büchner funnel (Büchner), porcelain, 12 cm diameter

filter paper, Shark Skin®, to fit Büchner

500 mL suction flask

long-stemmed funnel, glass, 4" diameter

Kuderna-Danish concentrator (K-D), 500 mL, with Snyder column, two-ball micro-Snyder column, graduated receiving flask

separatory funnel (separator), 1 L

75 mL Bond Elut reservoir or equivalent

25 mm syringe filter, 0.45 µm Nylon 66, with 1 µm prefilter

tC-18 Solid Phase Extraction (SPE) cartridge, 500 mg

Reagents

acetone, distilled from all-glass apparatus

boiling chips, 20-30 mesh carborundum

eluant, water/acetone, 30% (v/v)

glass wool, Pyrex, see Section 204 for handling directions

methylene chloride, distilled from all-glass apparatus

petroleum ether, distilled from all-glass apparatus

sodium chloride, reagent grade

sodium sulfate, anhydrous, granular, reagent grade; see Section 204 for handling directions

Directions

- Prewash filter paper with acetone to remove contaminants.
- Weigh 100 g chopped or blended sample into blender jar, add 200 mL acetone, and blend 2 min at high speed.
- Filter with suction through 12 cm Büchner fitted with Shark Skin® paper; collect extract in 500 mL suction flask. Continuation of vacuum for excessive period can reduce volume of extract and cause error in calculation.
- Attach 0.45 µm Nylon cartridge filter to bottom of 75 mL reservoir; attach tC-18 SPE cartridge to outlet of cartridge filter.
- Wash system with 40 mL acetone, followed by 10 mL eluant. Discard washes.

- Measure 40 mL sample extract and place into reservoir. Elute extract at 3 to 5 mL/min, with air pressure, into 1 L separatory funnel; do not allow level of extract to go below bottom of reservoir.
- Rinse graduated cylinder used for transfer with 10 mL 30% water/acetone; place rinse into reservoir and elute to column dryness.
- Add 50 mL acetone and 100 mL methylene chloride to separatory funnel and shake vigorously 1 min. Let separator stand 5-10 min to allow layers to separate.
- Dry lower organic layer of first separator by passing through about 1.5" sodium sulfate supported on washed glass wool in 4" funnel, collecting in K-D.
- Add 100 mL acetone and 100 mL methylene chloride to separator and repeat shaking. Let separator stand 5-10 min.
- Drain lower organic layer through sodium sulfate into separator. (Sugar content of fruit samples may result in aqueous phase's being the lower layer. In that case, add 5-10 mL methylene chloride and repeat shaking.) Rinse sodium sulfate with about 50 mL methylene chloride.
- Add boiling chips to K-D and concentrate solvent in K-D; start evaporation slowly by placing only receiver tube into steam. After 100-150 mL has evaporated, concentrator may be exposed to more steam. Concentrate solvent to 2-3 mL. After cooling, remove tube from K-D and adjust volume to 5 mL with acetone.
- Calculate equivalent sample weight in final solution:

$$\frac{\text{mg sample equivalent}}{\mu\text{L final extract}} = 100 \times \frac{40}{200 + W - 10} \times \frac{1}{\text{mL final volume}}$$

where:

100 = g sample analyzed

40 = mL filtered extract taken for liquid-liquid partitioning

200 = mL acetone blended with 100 g sample

W = amount (mL) of water present in sample (Section 201; if data are not available for particular raw agricultural commodity, use 85%)

10 = adjustment for water/acetone volume contraction.

Thus, when sample contains 85% water (85 mL/100 g) and final extract volume is 5 mL, each uL contains:

$$100 \times \frac{40}{200 + 85 - 10} \times \frac{1}{5} = \frac{2.9 \text{ mg sample equivalent}}{\mu\text{L final extract}}$$

- Clean up extract with C6 prior to determination.

C1 FLORISIL COLUMN (4 G) CLEANUP, WITH ONE METHYLENE CHLORIDE ELUANT

References

Griffitt, K.R., *et al.* (July 1983) "Miniaturized Florisil Column Cleanup of Chlorinated and Organophosphate Eluates in Total Diet Samples," LIB 2722, FDA, Rockville, MD

Griffitt, K.R., and Szorik, M.M. (Sept. 1989) "The Analysis of 127 Total Diet Items for Chlorinated Residues Using Luke/Solid Phase Extracts," LIB 3366, FDA, Rockville, MD

Principle

Residues in solution are separated from sample co-extractives on a small column of Florisil adsorbent, eluting with a single eluant.

Apparatus

chromatographic column, 10 mm id × 300 mm, Teflon stopcock, coarse porosity fritted disc

Kuderna-Danish concentrator (K-D), 125 or 250 mL, with Snyder column, two-ball micro-Snyder column, graduated or volumetric receiving flask

Reagents

acetonitrile, distilled from all-glass apparatus; see Section 204 for distillation directions

Florisil, PR grade; see Section 204 for handling and testing directions and calculation of lauric acid value

hexane, distilled from all-glass apparatus

sodium sulfate, anhydrous, granular, reagent grade; see Section 204 for handling directions

eluant: 50% methylene chloride/1.5% acetonitrile/48.5% hexane (v/v/v). Pipet 15 mL acetonitrile into 500 mL methylene chloride and dilute with hexane. Allow mixture to reach room temperature and adjust to 1 L with hexane.

Directions

- Place activated Florisil (weight = 110/lauric acid value × 4 g) in 10 mm chromatographic column; add about 2 cm sodium sulfate. Completely open stopcock and tap column to settle adsorbent. Prewet column with 15 mL hexane. Do not allow column to go dry. Place K-D with volumetric or graduated receiving flask under column to receive eluate.
- Dilute extract with hexane to produce solution of 10% acetone/hexane. Volumes depend on concentration of extract, volume taken for cleanup; *e.g.*, dilute 1 mL E1 extract, previously concentrated to 7 mL acetone, to 10 mL with hexane.
- Transfer solution to Florisil column, letting it pass through at about 5 mL/min. Rinse container with two 3 mL portions hexane, transfer rinsings to column, and rinse walls of chromatographic tube with additional small portions hexane.
- Elute column at about 5 mL/min with 50 mL eluant.

- Add boiling chip to K-D and concentrate eluate to suitable definite volume. For example, if 1 mL E1 extract (equivalent to 4.15 mg/mL) was cleaned up, concentrate Florisil eluate to 1 mL for same final concentration.

  When volume <5 mL is needed, use two-ball micro-Snyder or micro-Vigreaux column during evaporation.

- Use appropriate determinative steps, such as DG1 or DG13, DG6, DG7, and DG10, to identify and measure residues.

C2 CHARCOAL/CELITE/MAGNESIUM OXIDE COLUMN CLEANUP

References

Luke, M.A., and Doose, G.M. (1983) *Bull. Environ. Contam. Toxicol.* **30**, 110-116

Hardy, R.P. (Fall 1984) "Recoveries of Organophosphorus Compounds Through the Modified Storherr Method Using Charcoal Columns With and Without Magnesium Oxide," LIB 2860, FDA, Rockville, MD

Principles

Polar residues in solution are separated from sample co-extractives on a column of charcoal/Celite/magnesium oxide; cleanup may be necessary for subsequent examination of extract with selective detectors. Aromatic residues are not eluted with this system and must be determined in extract cleaned up by C1, Florisil column. Magnesium oxide may be eliminated to prevent destruction of sensitive residues (*e.g.*, acephate) without diminishing recoveries of other residues normally eluted.

Apparatus

chromatographic column, 22 mm id × 300 mm, Teflon stopcock, coarse porosity fritted disc

Kuderna-Danish concentrator (K-D), 500 mL, with Snyder column, two-ball micro-Snyder column, graduated or volumetric receiving flask

Reagents

acetone, distilled from all-glass apparatus

adsorbent mixture, 1:4:2 (w/w/w) charcoal/Celite 545/magnesium oxide or 1:4 (w/w) charcoal/Celite 545

Celite 545. To prepare, slurry about 500 g with distilled water, heat on steam bath about 30 min, and filter with suction. Dry overnight at l05-l30° C and pulverize to pass No. 60 sieve. Store in closed jar.

charcoal, Darco G60 or Norite S.G. Extra

glass wool, Pyrex; see Section 204 for handling directions

magnesium oxide, 200 mesh, adsorptive grade (optional)

methylene chloride, distilled from all-glass apparatus

eluant: 2:1 (v/v) acetone/methylene chloride

Directions

- Place about 1" Celite 545 in column, then add 6 g adsorbent mixture, and top with large plug glass wool.
- Tamp column down firmly and add about 25 mL methylene chloride. Force solvent through column with air pressure until top of solvent reaches top of column. Discard solvent.
- Transfer sample extract quantitatively to column with small portions methylene chloride and force solvent through as before, collecting in K-D.
- Elute with 200 mL 2:1 acetone/methylene chloride; force through as before.

- Mix contents of K-D, add boiling chips, and concentrate solvent; start evaporation slowly by placing only receiver tube into steam. After 100-150 mL has evaporated, concentrator may be exposed to more steam. When liquid level in hot concentrator tube is about 2 mL, add 100 mL petroleum ether through Snyder column and reconcentrate to about 2 mL. Add 50 mL petroleum ether and repeat concentration step. Add 20 mL acetone, and reconcentrate to about 2 mL. Do not allow solution to go to dryness during any of the concentration steps. Adjust volume of extract to suitable definite volume with acetone.
- If magnesium oxide is not used, a white precipitate may form if extract is concentrated to <2 mL; this should not affect GLC.
- Use appropriate determinative steps or confirmatory steps, such as GLC with mass spectrometric detection.

C3 CHARCOAL/SILANIZED CELITE COLUMN CLEANUP

References

Krause, R.T. (1980) *J. Assoc. Off. Anal. Chem.* **63**, 1114-1124

Pardue, J.R. (May 1987) "Recoveries of N-Methyl Carbamates Using a Combination of the Luke (PAM I, 232.4) and Krause (PAM I, 242.24b, 242.25) Procedures," LIB 3138, FDA, Rockville, MD

Principle

Residues in solution are separated from sample co-extractives on a column of charcoal and Celite, cleaning up the extract sufficiently for subsequent determination by HPLC system DL1.

Apparatus

chromatographic column, 22 mm id × 300 mm, Teflon stopcock, coarse porosity fritted disc

evaporator, vacuum rotary, as described in Section 401 E1

flasks, round-bottom (r-b), 250 and 500 mL, 1 L

magnetic stirrer, star, 10 mm diameter × 8 mm

vacuum adapter, side arm, with Ts bottom joint to fit in 500 mL r-b flask

Reagents

acetonitrile, distilled from all-glass apparatus; see Section 204 for distillation directions

Celite 545, silanized and prepared for use as directed in Section 401 C1

charcoal (Nuchar S-N), produced by Westvaco Corp. and available from Eastman Kodak, Cat. No. 118 0454, purified as directed in Section 401 C1

glass wool, Pyrex; see Section 204 for handling directions

methanol, distilled from all-glass apparatus

methylene chloride, distilled from all-glass apparatus

toluene, distilled from all-glass apparatus

eluant: 25% (v/v) toluene/acetonitrile

Directions

- Test charcoal/silanized Celite column as described in Section 401 C1.
- To the extract in r-b flask, add star magnetic stirrer. Place 250 mL T 24/40 trap on 1 L r-b flask and attach to vacuum rotary evaporator.
- Circulate refrigerated (-15° C) 1+1 water/ethylene glycol through evaporator condensing coils; maintain receiving flask at -15° C by immersion in refrigerated bath.
- Apply vacuum slowly to minimize frothing by regulating with needle valve. After full vacuum is applied, slowly place flask in 35° C water bath.
- Remove r-b flask from evaporator immediately after last traces of solution have evaporated and add 10 mL methylene chloride to r-b flask.

- Fit one-hole No. 5 rubber stopper onto tip of chromatographic column, add side arm vacuum adapter and 500 mL r-b flask, open stopcock, and connect apparatus to vacuum line.
- Place 0.5 g silanized Celite 545 in chromatographic column, tamp, add 5 g charcoal/Celite 545 (1+4) mixture, and tamp again. Add 1-2 cm glass wool plug on top of adsorbent.
- Prewash column with 50 mL 25% toluene/acetonitrile eluant. Close stopcock when prewash solution is about 0.5 cm from top of glass wool.
- Disconnect vacuum, discard solution in r-b flask, and reconnect flask to apparatus.
- Transfer 10 mL methylene chloride extract to column and let pass through column at 5 mL/min.
- Wash 1 L r-b flask with 10 mL methylene chloride and then with 25 mL eluant. Transfer each separately to column and elute each to top of glass wool before adding next solution.
- Add 100 mL eluant and elute column at 5 mL/min. Turn off stopcock when top of eluant reaches top of glass wool.
- Evaporate solution in 500 mL r-b flask just to dryness using vacuum evaporator as above. Remove flask from evaporator immediately after all solution has evaporated.
- Immediately pipet 5 mL methanol into 500 mL r-b flask to dissolve residue. Cleaned up extract contains concentration of sample equivalent (mg/µL) equal to amount of sample in extract taken for cleanup, divided by 5. For example, if entire E1 extract of commodity with 85% water is used, 29 g sample equivalent is cleaned up, *i.e.*, $100\,g \times 80/(200+85-10)$; final concentration of cleaned up extract is 5.8 mg/µL (29 g/5 mL).
- Use determinative step DL1 or DL2 (Section 401) to determine N-methylcarbamates, except use 20 µL injection loop instead of 10 µL loop specified.

C4 C-18 CARTRIDGE CLEANUP

Reference

Sharp, K.B., and Bramlett, C.L. (Dec. 1983) "Analysis for Carbamate Residues in Fresh Produce," LIB 2778, FDA, Rockville, MD

Principle

Residues in solution are separated from sample co-extractives on a C-18 solid phase extraction cartridge, cleaning up the extract sufficiently for subsequent determination by HPLC system DL1.

Apparatus

Kuderna-Danish concentrator (K-D), 500 mL, with Snyder column, two-ball micro-Snyder column, graduated receiving flask

volumetric flask, 5 mL

Reagents

cartridge (solid phase extraction type), C-18, 2.8 mL

methanol, distilled from all-glass apparatus

Directions

- Concentrate extract in K-D to 2 mL. Evaporate almost to dryness (about 0.1 mL) under current of nitrogen.
- Prewet C-18 cartridge with methanol and discard solvent.
- Dissolve residue in receiving flask with 2 mL methanol and transfer quantitatively onto prewet C-18 cartridge. Collect eluate from cartridge in 5 mL volumetric flask.
- Elute cartridge with additional methanol until collected volume is almost 5 mL; add methanol to make volume 5.0 mL. Cleaned up extract contains concentration of sample equivalent (mg/µL) equal to amount of sample in extract taken for cleanup, divided by 5. For example, if entire E1 extract of commodity with 85% water is used, 29 g sample equivalent is cleaned up, *i.e.*, 100 g × 80/(200 + 85 - 10); final concentration of cleaned up extract is 5.8 mg/µL (29 g/5 mL).
- Use determinative step DL1 or DL2 (Section 401) to determine N-methylcarbamates, except use 20 µL injection loop instead of 10 µL loop specified.

C5 FLORISIL COLUMN CLEANUP, WITH MIXED ETHER ELUANTS

Reference

Luke, M.A., *et al.* (1975) *J. Assoc. Off. Anal. Chem.* **58**, 1020-1026

Principles

Residues in solution are separated from sample co-extractives on a column of Florisil adsorbent; cleanup is usually necessary for subsequent examination of extract with DG1, electron capture detector.

Apparatus

chromatographic column, 22 mm id × 300 mm, Teflon stopcock, coarse porosity fritted disc

graduated cylinder (graduate), glass-stoppered (g-s), 100 mL

Kuderna-Danish concentrator (K-D), 500 mL, with Snyder column, two-ball micro-Snyder column, volumetric or graduated receiving flask

Reagents

boiling chips, 20-30 mesh carborundum

eluants: 15% (v/v) ethyl ether/petroleum ether
50% (v/v) ethyl ether/petroleum ether

ethyl ether, distilled from all-glass apparatus, with 2% ethanol as preservative; see Section 204 for peroxide test

Florisil, PR grade; see Section 204 for handling and testing directions and calculation of lauric acid value

petroleum ether, distilled from all-glass apparatus

sodium sulfate, anhydrous, granular, reagent grade; see Section 204 for handling directions

Directions

- Place activated Florisil (4" or weight determined by lauric acid value) in 22 mm id column; add about 0.5" sodium sulfate. Prewet column with 40-50 mL petroleum ether. Place K-D with volumetric or graduated receiving flask under column to receive eluate.
- Dilute concentrated extract to 10 mL with acetone and transfer to 100 mL g-s graduate, using petroleum ether to rinse. Dilute to 100 mL with petroleum ether; stopper and mix well.
- Transfer diluted extract solution to column letting it pass through at about 5 mL/min.
- Elute column at about 5 mL/min with 200 mL 15% ethyl ether/petroleum ether eluant.
- Change K-Ds and elute at about 5 mL/min with 200 mL 50% ethyl ether/petroleum ether eluant.
- Add boiling chips to K-Ds and concentrate to suitable definite volume. For example, if entire E1 extract of commodity with 85% water is used, and final volume is 5 mL, final concentration of cleaned up extract is 5.8 mg/µL, *i.e.*, 100 g × 80/(200 + 85 - 10) = 29 g; 29 g/5 mL = 5.8 mg/µL.

- When volume <5 mL is needed, use two-ball micro-Snyder or micro-Vigreaux column during final evaporation in receiving flask.
- Use appropriate determinative steps, such as DG1 or DG13, DG6, DG7, and DG10, to identify and measure residues.

C6 SAX/PSA CARTRIDGE CLEANUP

Reference

Luke, M. A., *et al.* (Sept. 1994) "An Improved Variation of the Luke Multiresidue Pesticide Procedure for the Analysis of Fruits and Vegetables Using Solid Phase Extraction Cartridges and Element Selective Gas Chromatographic Detectors," LIB 3896, FDA, Rockville, MD

Principle

SAX and PSA cartridges provide the improved cleanup required for determination with capillary and megabore GC columns; both polar and nonpolar residues can be recovered.

Apparatus

75 mL Bond Elut reservoir or equivalent

25 mm syringe filter, 0.45 µm Nylon 66 with 1 µm prefilter

SAX SPE cartridge or equivalent, 500 mg

PSA SPE cartridge or equivalent, 500 mg

Kuderna-Danish concentrator (K-D), 500 mL, with Snyder column, two-ball micro-Snyder column, graduated or volumetric receiving flask

Reagents

acetone, distilled from all-glass apparatus

petroleum ether, distilled from all-glass apparatus

acetone+petroleum ether, 1+2

Directions

- Attach 0.45 µm filter to bottom of 75 mL reservoir. Attach SAX or equivalent cartridge to filter, and attach PSA or equivalent cartridge to first cartridge.
- Wash cartridges with 40 mL acetone; follow with 10 mL acetone+petroleum ether. Discard washes.
- Dilute the 5.0 mL concentrated acetone extract from E7 with 10 mL petroleum ether and mix. Transfer to reservoir, and elute dropwise with air pressure.
- Rinse tube with five 10 mL portions acetone+petroleum ether. Elute each rinse when the previous solvent has reached top of column.
- Mix contents of K-D, add boiling chips, and concentrate solvent; start evaporation slowly by placing only receiver tube into steam. When liquid level in hot concentrator tube is about 2 mL, add 100 mL petroleum ether through Snyder column and reconcentrate to about 2 mL. Add 50 mL petroleum ether and repeat concentration step. Carefully add 25 mL acetone and reconcentrate to about 2 mL. Do not allow solution to go to dryness during any of the concentration steps. Adjust volume of extract to suitable definite volume with acetone.
- Use appropriate determinative steps, such as DG2, DG3, DG14, or DG16, to identify and measure residues.

DETERMINATION

Inject concentrated extract equivalent to 20 mg (whole high moisture product) into the following GLC systems for determination of residues. (Although AOAC collaborative study for this method involved injection of 12 mg sample equivalent, experience since then has proven that GLC systems can tolerate routine injections equivalent to 20 mg of most nonfatty foods.)

Extract not cleaned up prior to determination:

DG2 or DG14	organophosphorus residues; large amounts of sulfur may interfere
DG3 or DG16	organohalogen residues
DG4 or	organonitrogen residues; selective to nitrogen, but co-extractives may contain nitrogen
DG5 or DG17	organonitrogen and organophosphorus residues
DG15	organosulfur residues; large amounts of phosphorus may interfere
DG12	late eluting organohalogen residues, especially pyrethroids

Additional recommended determinations:

Extract not cleaned up prior to determination:

DG8	early eluting organophosphorus residues
DG11	late eluting organophosphorus residues
DG9	early eluting organohalogen residues

Extract cleaned up on Florisil column, C1 or C5:

DG1 or DG13	residues with halogen, sulfur, or other moieties
DG7	early eluting residues with halogen, sulfur, or other moieties
DG10	late eluting residues, especially synthetic pyrethroids
DG6	o-phenylphenol and biphenyl

Inject concentrated extract equivalent to about 58-116 mg (whole high moisture product) cleaned up by C3 (charcoal/Celite column) or C4 (C-18 cartridge) into following HPLC system:

DL1	N-methylcarbamates (determinative step described in Section 401)

For accurate quantitation, reference standards should be dissolved in same solvent as concentrated extract, only peaks >10% FSD should be measured, and peak sizes of residue and reference standard should match within ±25%.

See Chapter 5 for additional information about operation of GLC systems; Section 504 provides information about quantitation of residues.

See Chapter 6 for additional information about operation of HPLC systems; Section 606 provides information about quantitation of residues.

See Section 205 for additional information about reference standards.

See Section 104 for additional information about reporting residues and determining compliance with regulations.

See Section 105 for additional information about analytical limits of quantitation.

CONFIRMATION

After residues have been tentatively identified and quantitated by comparison to appropriate reference standards, confirm identity according to principles discussed in Section 103. Use appropriate tables of data (PESTDATA, tables accompanying each method, Index to Methods) to choose the most appropriate determinative steps and/or alternative methods for confirmation.

DG1 GLC, 100% METHYL SILOXANE, 200° C, EC

Applicability

Determinative step is applicable to residues containing halogen, sulfur, or other electrophilic moieties. It is a general purpose system, but subject to interferences by nonpesticides.

Column

Wide bore capillary, 30 m × 0.53 mm id, coated with 100% methyl substituted polysiloxane liquid phase, 1-1.5 µm film thickness, bonded and cross-linked. For example, DB-1; see Table 502-a for equivalents. See Section 502 C, Recommended Operating Procedure for Wide Bore Columns.

Column Operating Conditions:

200° C isothermal; if necessary, adjust temperature so that relative retention time to chlorpyrifos (rrt_c) of p,p'-DDT is 3.1 ± 0.06.

Carrier gas: helium; adjust flow rate so that chlorpyrifos elutes in about 4.0 ± 0.5 min (about 20 mL/min).

Injector temperature: 220-250° C

Detector

Electron Capture (EC)

Detector Operating Conditions:

350° C

Make-up gas: nitrogen or argon/methane (95:5), at 30 mL/min

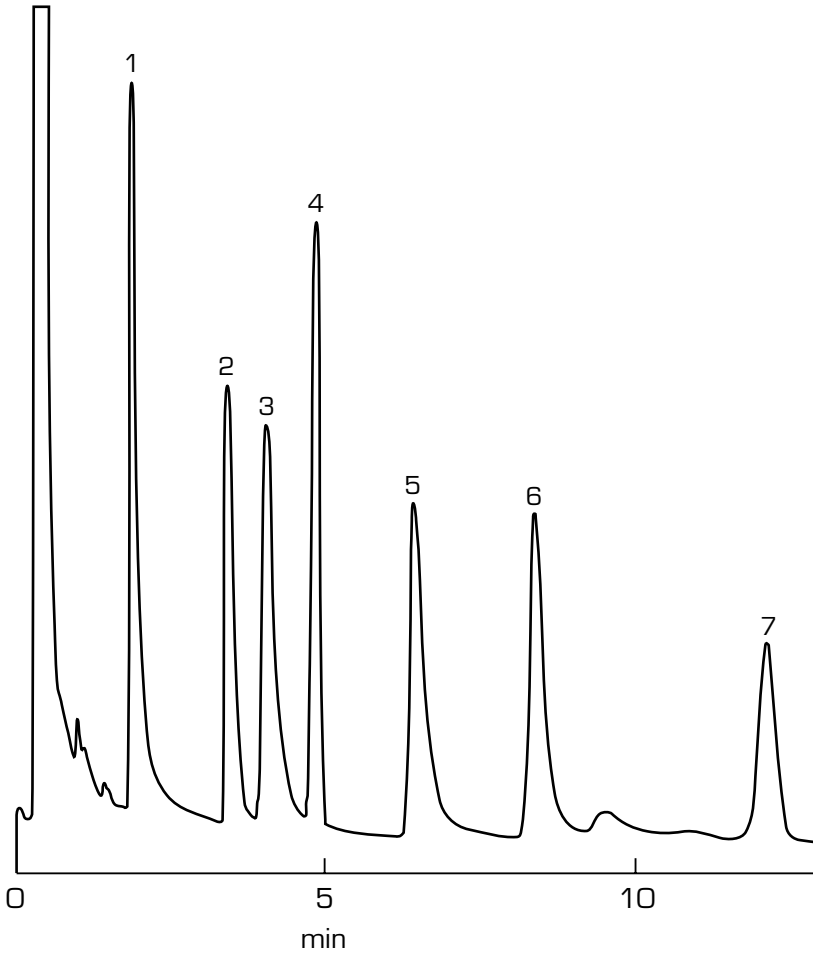
See Section 503 B for other information about EC detector operation.

Set detector electronics (amplification, attenuation) so that response to 0.15 ng chlorpyrifos (or an amount within the detector's linear range) is 50% full scale deflection (FSD).

Other Considerations

Rrt_cs and ng required to cause 50% FSD response are listed in Appendix I, PESTDATA (many data in PESTDATA were collected using equivalent packed column). Response data in Appendix I are based on detector sensitivity of 50% FSD to 1.5 ng chlorpyrifos.

Example chromatogram is on next page. Also see Figures 504-c, d, e, and f.



DG1

Chromatogram of: 1) 0.15 ng dicloran, 2) 0.10 ng heptachlor, 3) 0.19 ng chlorpyrifos, 4) 0.31 ng captan, 5) 0.14 ng endosulfan I, 6) 0.18 ng endrin, and 7) 0.20 ng p,p′-DDT at the conditions described.

DG2 GLC, 100% METHYL SILOXANE, 200° C, FPD-P

Applicability

Determinative step is applicable to residues containing phosphorus. It is particularly useful for residues such as organophosphate pesticides.

Column

Wide bore capillary, 30 m × 0.53 mm id, coated with 100% methyl substituted polysiloxane liquid phase, 1-1.5 µm film thickness, bonded and cross-linked. For example, DB-1; see Table 502-a for equivalents. See Section 502 C, Recommended Operating Procedure for Wide Bore Columns.

Column Operating Conditions:

200° C isothermal; if necessary, adjust temperature so that relative retention time to chlorpyrifos (rrt_c) of ethion is 2.56 ± 0.05.

Carrier gas: helium; adjust flow rate so that chlorpyrifos elutes in about 4.0 ± 0.5 min (about 20 mL/min).

Injector temperature: 220-250° C

Detector

Flame photometric, phosphorus mode (FPD-P)

Detector Operating Conditions:

225-250° C

See Section 503 C for other information about FPD operation.

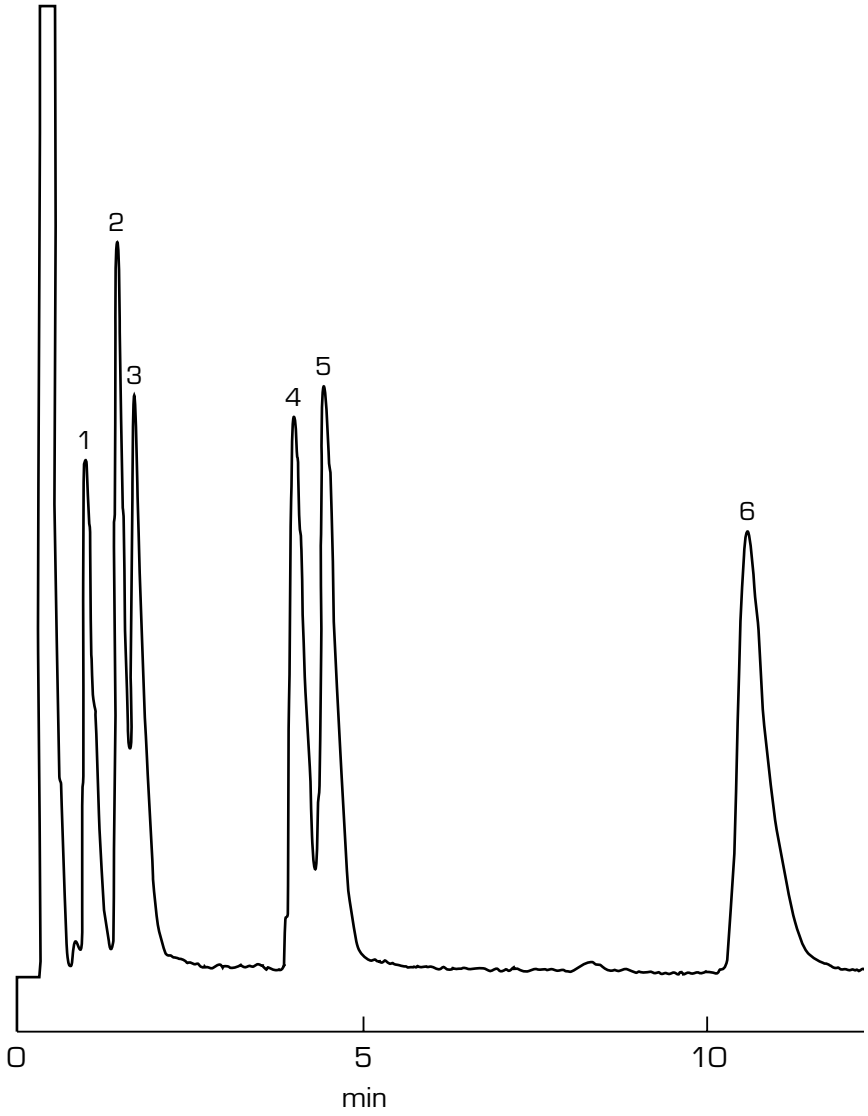
Set detector electronics (amplification, attenuation) so that response to 1.5 ng chlorpyrifos is 50% full scale deflection (FSD).

A properly functioning system will give a response to 1.5 ng omethoate of ≥50% FSD.

Other Considerations

Rrt_cs and ng required to cause 50% FSD response are listed in Appendix I, PESTDATA (many data in PESTDATA were collected using equivalent packed column).

Example chromatogram is on next page.



DG2

Chromatogram of: 1) 0.85 ng acephate, 2) 1.73 ng omethoate, 3) 0.68 ng monocrotophos, 4) 1.30 ng malathion, 5) 1.27 ng chlorpyrifos, and 6) 1.26 ng ethion at the conditions described; helium carrier gas flow was 15 mL/min, with 15 mL/min make-up gas being added before the detector. Detector gas flows: 100 mL/min hydrogen, 130 mL/min air.

DG3 GLC, 100% METHYL SILOXANE, 200° C, ELCD-X

Applicability

Determinative step is applicable to residues containing halogen. It is particularly useful for residues such as chlorinated hydrocarbon pesticides and polychlorinated biphenyls.

Column

Wide bore capillary, 30 m × 0.53 mm id, coated with 100% methyl substituted polysiloxane liquid phase, 1-1.5 µm film thickness, bonded and cross-linked. For example, DB-1; see Table 502-a for equivalents. See Section 502 C, Recommended Operating Procedure for Wide Bore Columns.

Column Operating Conditions:

200° C isothermal; if necessary, adjust temperature so that relative retention time to chlorpyrifos (rrt_c) of p,p'-DDT is 3.1 ± 0.06.

Carrier gas: helium; adjust flow rate so that chlorpyrifos elutes in about 4.0 ± 0.5 min (about 20 mL/min).

Injector temperature: 220-250° C

Detector

Electroconductivity, halogen mode (ELCD-X)

Detector Operating Conditions:

Base temperature 250° C; furnace temperature 900° C (or as specified in operating manual)

Reactor gas: hydrogen, flow as required by specific detector model; see operating manual

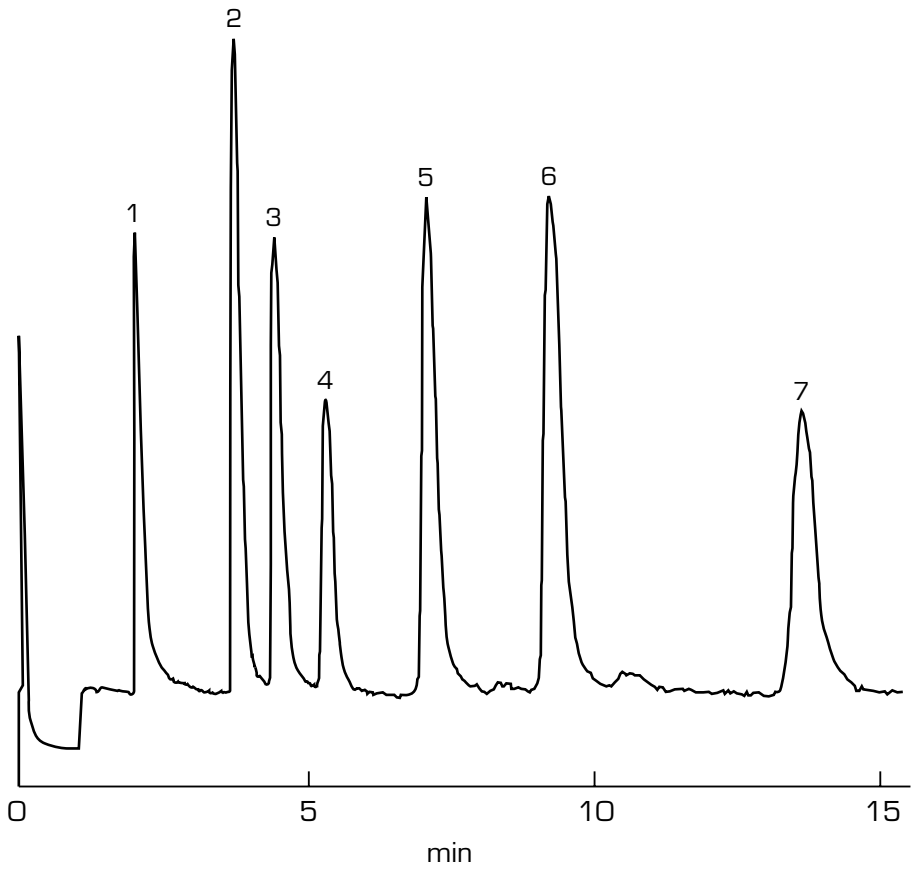
See Section 503 D for other information about ELCD operation.

Set detector electronics (amplification, attenuation) so that response to 1.5 ng chlorpyrifos is 50% full scale deflection (FSD).

Other Considerations

Rrt_cs and ng required to cause 50% FSD response are listed in Appendix I, PESTDATA (many data in PESTDATA were collected using equivalent packed column).

Example chromatogram is on next page.



DG3

Chromatogram of: 1) 1.44 ng dicloran, 2) 0.98 ng heptachlor, 3) 1.87 ng chlorpyrifos, 4) 2.99 ng captan, 5) 1.37 ng endosulfan I, 6) 1.77 ng endrin, and 7) 1.91 ng p,p'-DDT at the conditions described. Hydrogen reactor gas flow: 40 mL/min, n-propanol electrolyte: 0.3 mL/min.

DG4 GLC, 100% METHYL SILOXANE, 200° C, ELCD-N

Applicability

Determinative step is applicable to residues containing nitrogen. It may be useful for confirmation of residues such as triazines (atrazine, simazine, *etc.*) and triazoles (propiconazole, diclobutrazole, *etc.*).

Column

Wide bore capillary, 30 m × 0.53 mm id, coated with 100% methyl substituted polysiloxane liquid phase, 1-1.5 µm film thickness, bonded and cross-linked. For example, DB-1; see Table 502-a for equivalents. See Section 502 C, Recommended Operating Procedure for Wide Bore Columns.

Column Operating Conditions:

200° C isothermal

Carrier gas: helium; adjust flow rate so that chlorpyrifos elutes in about 4.0 ± 0.5 min (about 20 mL/min).

Injector temperature: 220-250° C

Detector

Electroconductivity, nitrogen mode (ELCD-N)

Detector Operating Conditions:

Base temperature 250° C; furnace temperature 900° C (or as specified in operating manual)

Reactor gas: hydrogen, flow as required by specific detector model; see operating manual

See Section 503 D for other information about ELCD operation.

Set detector electronics (amplification, attenuation) so that response to 1.5 ng chlorpyrifos is 50% full scale deflection (FSD).

Other Considerations

Rrt_cs and ng required to cause 50% FSD response are listed in Appendix I, PESTDATA (many data in PESTDATA were collected using equivalent packed column).

No chromatogram currently available.

DG5 GLC, 100% METHYL SILOXANE, 200° C, N/P

Applicability

Determinative step is applicable to residues containing nitrogen. It is particularly useful for residues such as triazines and triazoles.

Column:

Wide bore capillary, 30 m × 0.53 mm id, coated with 100% methyl substituted polysiloxane liquid phase, 1-1.5 µm film thickness, bonded and cross-linked. For example, DB-1; see Table 502-a for equivalents. See Section 502 C, Recommended Operating Procedure for Wide Bore Columns.

Column Operating Conditions:

200° C isothermal; if necessary, adjust temperature so that relative retention time to chlorpyrifos (rrt_c) of ethion is 2.56 ± 0.05.

Carrier gas: helium; adjust flow rate so that chlorpyrifos elutes in about 4.0 ± 0.5 min (about 20 mL/min).

Injector temperature: 220-250° C

Detector

Alkali bead detector, nitrogen selective (N/P)

Detector Operating Conditions:

250° C

See Section 503 E for other information about N/P detector operation.

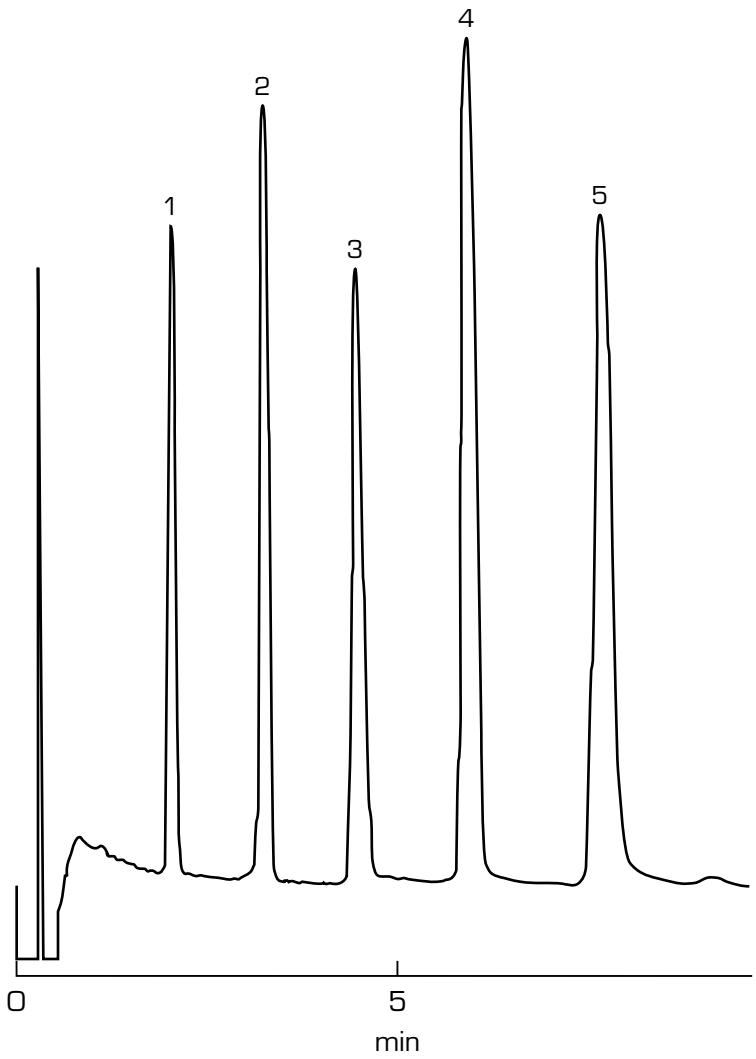
Set detector electronics (amplification, attenuation) so that response to 1.5 ng chlorpyrifos is 50% full scale deflection (FSD).

A properly functioning system will give a response to 1.5 ng omethoate of ≥50% FSD.

Other Considerations

Rrt_cs and ng required to cause 50% FSD response are listed in Appendix I, PESTDATA (many data in PESTDATA were collected using equivalent packed column).

Example chromatogram is on next page.



DG5

Chromatogram of: 1) 1.0 ng atrazine, 2) 7.5 ng carbaryl, 3) 1.5 ng chlorpyrifos, 4) 2.5 ng procyazine, and 5) 5.0 ng imazalil at the conditions described.

DG6 GLC, 100% METHYL SILOXANE, 130° C, FID

Applicability

Determinative step is applicable to residues containing no elements to which element-selective detectors respond. It is particularly useful for residues such as biphenyl and o-phenylphenol.

Column

Wide bore capillary, 30 m ¥ 0.53 mm id, coated with 100% methyl substituted polysiloxane liquid phase, 1-1.5 µm film thickness, bonded and cross-linked. For example, DB-1; see Table 502-a for equivalents. See Section 502 C, Recommended Operating Procedure for Wide Bore Columns.

Column Operating Conditions:

130° C isothermal

Carrier gas: helium, about 20 mL/min. At these conditions, chlorpyrifos elutes in about 16 min, and biphenyl and o-phenylphenol elute in <2 min.

Injector temperature: 220-250° C

Detector

Flame ionization detector (FID)

Detector Operating Conditions:

300° C

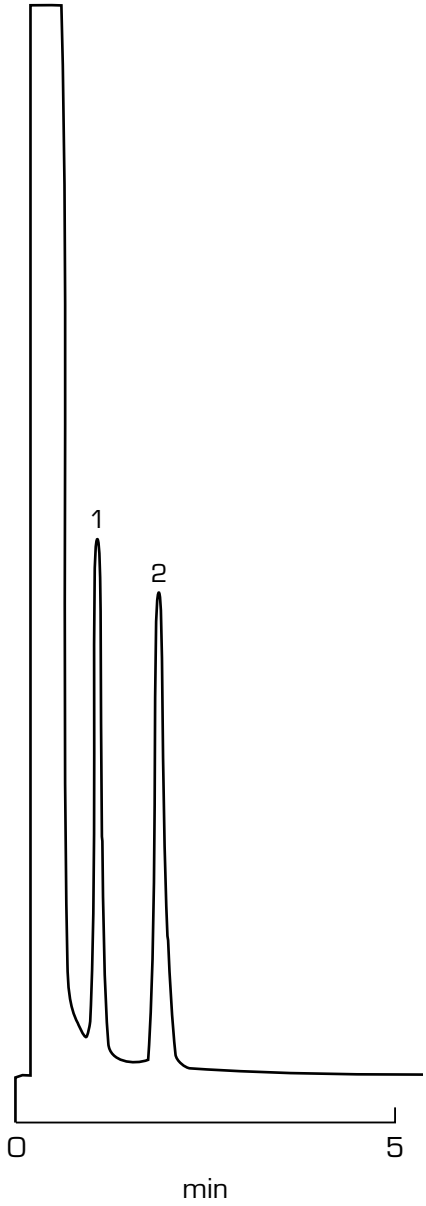
detector gases: hydrogen, 30 mL/min, air, 300 mL/min

set detector electronics (amplification, attenuation) so that response to 50 ng o-phenylphenol is 50% full scale deflection (FSD).

Other Considerations

FID is nonselective and will respond to large quantities of any co-extractive.

Example chromatogram is on next page.



DG6

Chromatogram of: 1) 20 ng biphenyl and 2) 53 ng o-phenylphenol at the conditions described.

DG7 GLC, 100% METHYL SILOXANE, 130° C, EC

Applicability

Determinative step is applicable to residues of high volatility (early elution) and containing halogen, sulfur, or other electrophilic moieties. It is particularly useful for residues such as benfluralin and sulfallate.

Column

Wide bore capillary, 30 m × 0.53 mm id, coated with 100% methyl substituted polysiloxane liquid phase, 1-1.5 µm film thickness, bonded and cross-linked. For example, DB-1; see Table 502-a for equivalents. See Section 502 C, Recommended Operating Procedure for Wide Bore Columns.

Column Operating Conditions:

130° C isothermal

Carrier gas: helium; adjust flow rate while column temperature is 200° C so that chlorpyrifos elutes in about 4.0 ± 0.5 min; then change column temperature without changing flow controller.

Injector temperature: 220-250° C

Detector

Electron Capture (EC)

Detector Operating Conditions:

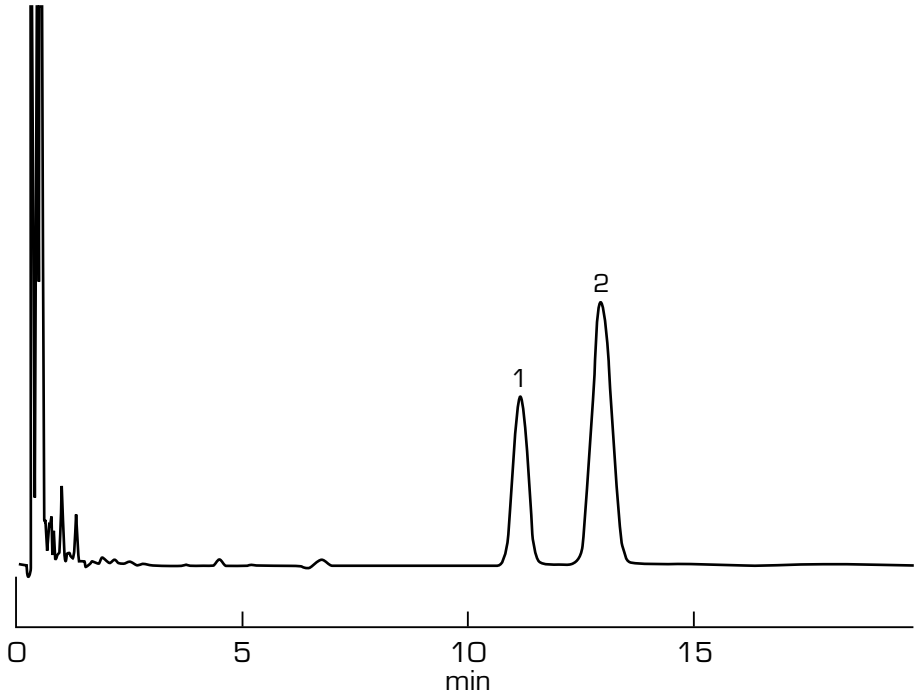
350° C

Make-up gas: nitrogen or argon/methane (95:5), at 30 mL/min

See Section 503 B for other information about EC detector operation.

While column temperature is 200° C, set detector electronics (amplification, attenuation) so that response to 0.15 ng chlorpyrifos (or an amount within the detector's linear range) is 50% full scale deflection; then change column temperature without changing electronics.

Example chromatogram is on next page.



DG7

Chromatogram of: 1) 0.18 ng benfluralin and 2) 0.09 ng sulfallate at the conditions described.

DG8 GLC, 100% METHYL SILOXANE, 130° C, FPD-P

Applicability

Determinative step is applicable to residues of high volatility (early elution) and containing phosphorus. It is particularly useful for residues such as mevinphos, acephate, demeton, and dicrotophos.

Column

Wide bore capillary, 30 m × 0.53 mm id, coated with 100% methyl substituted polysiloxane liquid phase, 1-1.5 µm film thickness, bonded and cross-linked. For example, DB-1; see Table 502-a for equivalents. See Section 502 C, Recommended Operating Procedure for Wide Bore Columns.

Column Operating Conditions:

130° C isothermal

Carrier gas: helium; adjust flow rate while column temperature is 200° C so that chlorpyrifos elutes in about 4.0 ± 0.5 min; then change column temperature without changing flow controller.

Injector temperature: 220-250° C

Detector

Flame photometric, phosphorus mode (FPD-P)

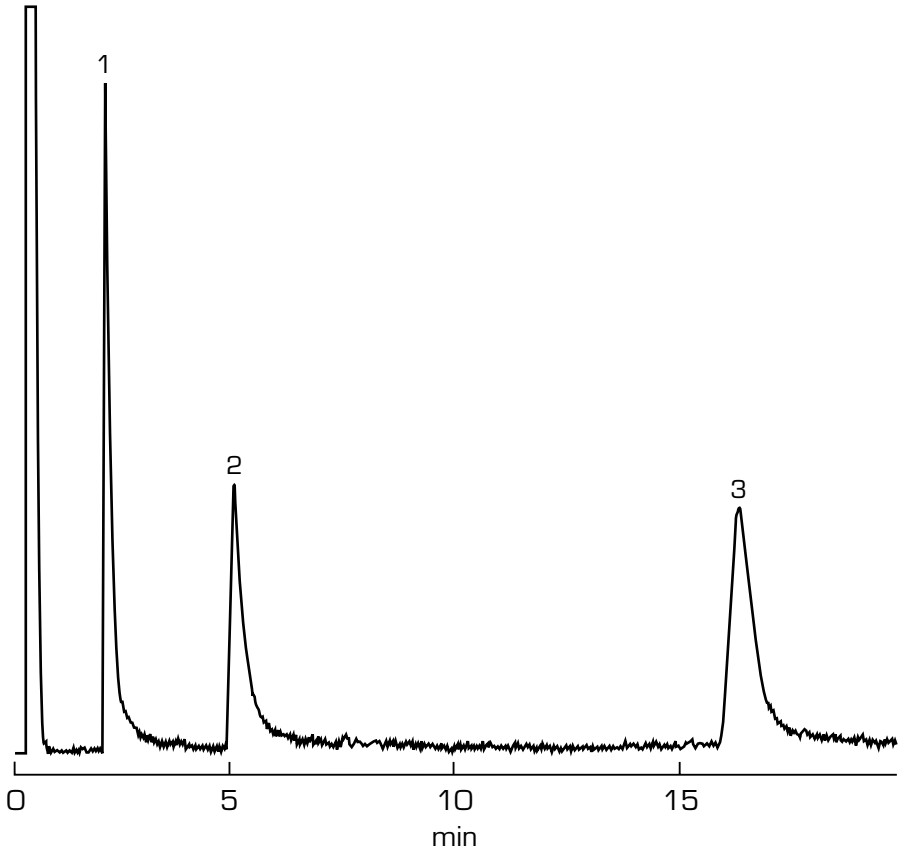
Detector Operating Conditions:

225-250° C

See Section 503 C for other information about FPD operation.

While column temperature is 200° C, set detector electronics (amplification, attenuation) so that response to 1.5 ng chlorpyrifos is 50% full scale deflection; then change column temperature without changing electronics.

Example chromatogram is on next page.



DG8

Chromatogram of: 1) 2.0 ng methamidophos, 2) 2.0 ng acephate, and 3) 4.0 ng dicrotophos at the conditions described.

DG9 GLC, 100% METHYL SILOXANE, 130° C, ELCD-X

Applicability

Determinative step is applicable to residues of high volatility (early elution) and containing halogen. It is particularly useful for residues such as the methyl esters of dicamba, MCPA, mecoprop, dichlorprop, and silvex.

Column

Wide bore capillary, 30 m × 0.53 mm id, coated with 100% methyl substituted polysiloxane liquid phase, 1-1.5 µm film thickness, bonded and cross-linked. For example, DB-1; see Table 502-a for equivalents. See Section 502 C, Recommended Operating Procedure for Wide Bore Columns.

Column Operating Conditions:

130° C isothermal

Carrier gas: helium, about 20 mL/min

Injector temperature: 220-250° C

Detector

Electroconductivity, halogen mode (ELCD-X)

Detector Operating Conditions:

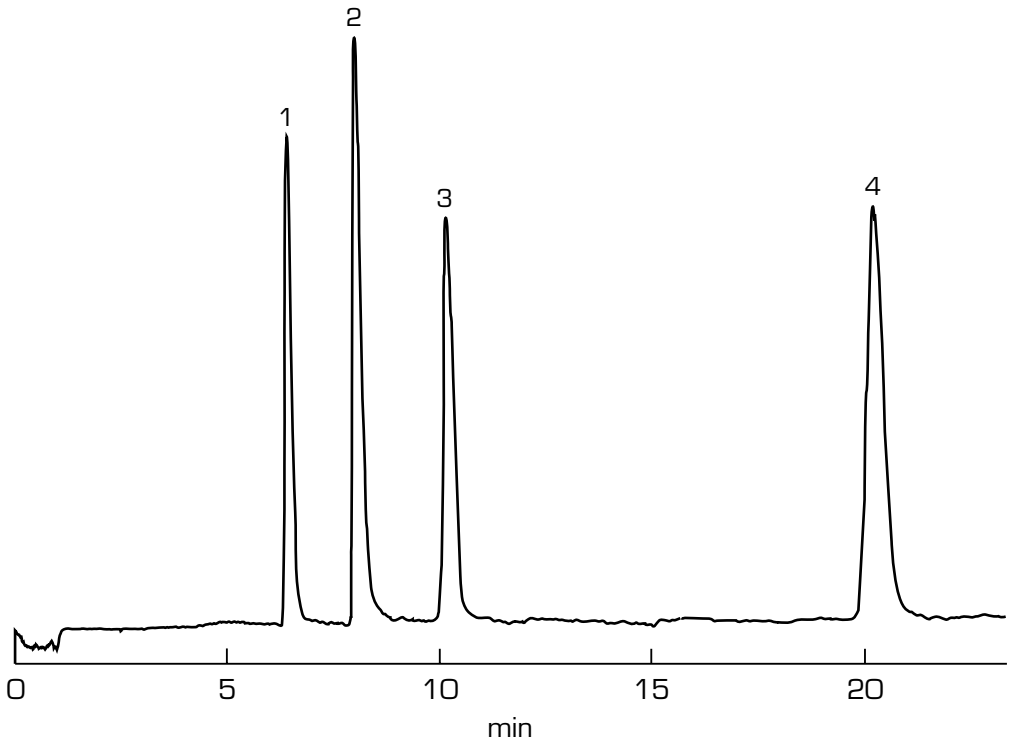
Base temperature 250° C; furnace temperature 900° C (or as specified in operating manual)

Reactor gas: hydrogen, flow as required by specific detector model; see operating manual

See Section 503 D for other information about ELCD operation.

Set detector electronics (amplification, attenuation) so that response to 0.5 ng pentachlorobenzene is 50% full scale deflection.

Example chromatogram is on next page.



DG9

Chromatogram of: 1) 1.0 ng dicamba methyl ester, 2) 3.0 ng MCPA methyl ester, 3) 1.5 ng dichlorprop methyl ester, and 4) 2.0 ng silvex methyl ester at the conditions described, except that carrier gas was hydrogen at 25 mL/min. Hydrogen reactor gas flow: 35 mL/min, n-propanol electrolyte 0.5 mL/min. Pentachlorobenzene eluted in 6.9 min at these conditions, and 0.3 ng pentachlorobenzene caused 40% FSD detector response.

DG10 GLC, 100% METHYL SILOXANE, 230° C, EC

Applicability

Determinative step is applicable to residues of low volatility (late elution) and containing halogen, sulfur, or other electrophilic moieties. It is particularly useful for residues such as pyrethroids, with halogen (permethrin, fenvalerate, deltamethrin) or without halogen (tetramethrin).

Column

Wide bore capillary, 30 m × 0.53 mm id, coated with 100% methyl substituted polysiloxane liquid phase, 1-1.5 µm film thickness, bonded and cross-linked. For example, DB-1; see Table 502-a for equivalents. See Section 502 C, Recommended Operating Procedure for Wide Bore Columns.

Column Operating Conditions:

230° C isothermal; if necessary, adjust temperature so that relative retention time (rrt) to phosalone of cis permethrin is about 1.55.

Carrier gas: helium; adjust flow rate so that phosalone elutes in about 8 min (about 18 mL/min).

Injector temperature: 250° C

Detector

Electron Capture (EC)

Detector Operating Conditions:

350° C

Make-up gas: nitrogen or argon/methane (95:5), at 30 mL/min

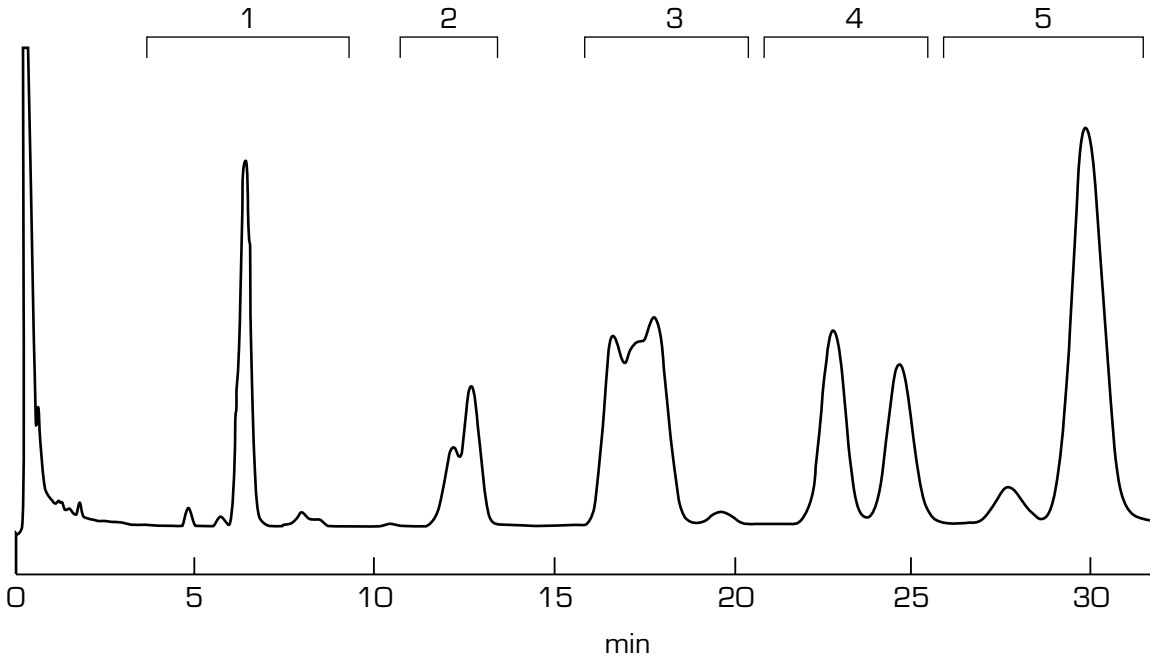
See Section 503 B for other information about EC detector operation.

Set detector electronics (amplification, attenuation) so that response to 0.5 ng phosalone is 50% full scale deflection (FSD).

Other Considerations

Detector sensitivity must be sufficient to measure residues of pyrethroids at ≤0.1 ppm, where some tolerances are set.

Example chromatogram is on next page.



DG10

Chromatogram of: 1) 3.5 ng tetramethrin, 2) 2.3 ng permethrin, 3) 2.1 ng cypermethrin, 4) 1.9 ng fenvalerate, and 5) 2.2 ng deltamethrin at the conditions described.

DG11 GLC, 100% METHYL SILOXANE, 230° C, FPD-P

Applicability

Determinative step is applicable to residues of low volatility (late elution) and containing phosphorus. It is particularly useful for residues such as some organophosphorus pesticides, their oxygen analog sulfones and sulfoxides, and aryl phosphate industrial chemicals.

Column

Wide bore capillary, 30 m × 0.53 mm id, coated with 100% methyl substituted polysiloxane liquid phase, 1-1.5 µm film thickness, bonded and cross-linked. For example, DB-1; see Table 502-a for equivalents. See Section 502 C, Recommended Operating Procedure for Wide Bore Columns.

Column Operating Conditions:

230° C isothermal; if necessary, adjust temperature so that relative retention time (rrt) to phosalone of coumaphos is about 1.56.

Carrier gas: helium; adjust flow rate so that phosalone elutes in about 8.5 min (about 18 mL/min).

Injector temperature: 250° C

Detector

Flame photometric, phosphorus mode (FPD-P)

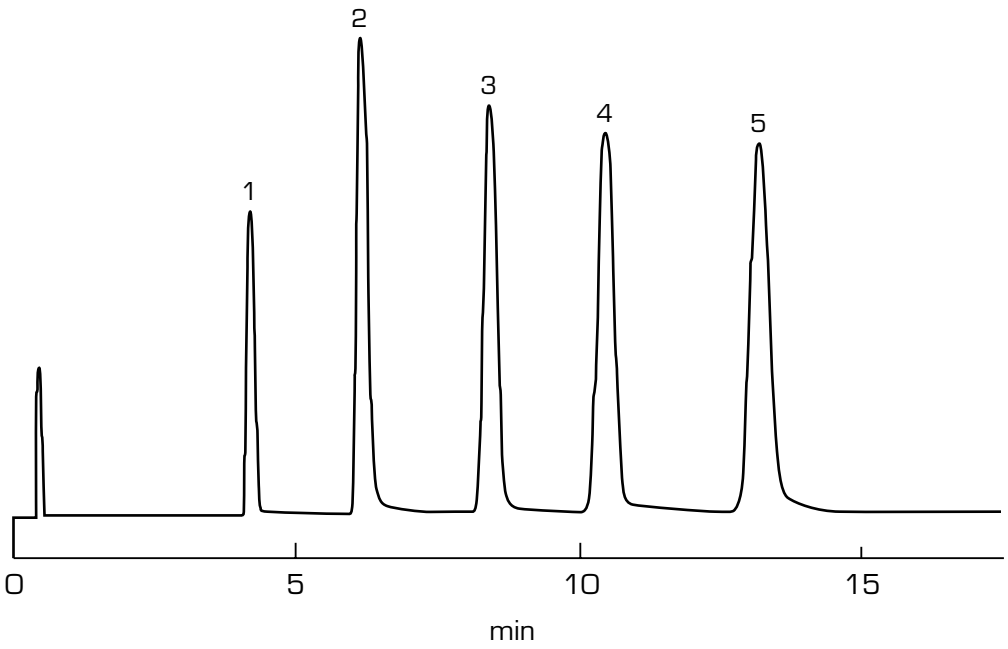
Detector Operating Conditions:

225-250° C

See Section 503 C for other information about FPD operation.

Set detector electronics (amplification, attenuation) so that response to 7.5 ng phosalone is 50% full scale deflection (FSD).

Example chromatogram is on next page.



DG11

Chromatogram of: 1) 1.38 ng ethion, 2) 20.8 ng azinphos-methyl oxygen analog, 3) 7.28 ng phosalone, 4) 7.79 ng pyrazophos, and 5) 10.1 ng coumaphos at the conditions described.

DG12 GLC, 100% METHYL SILOXANE, 230° C, ELCD-X

Applicability

Determinative step is applicable to residues of low volatility (late elution) and containing halogen. It is particularly useful for residues such as halogenated pyrethroids (cyfluthrin, alpha-cypermethrin).

Column

Wide bore capillary, 30 m × 0.53 mm id, coated with 100% methyl substituted polysiloxane liquid phase, 1-1.5 µm film thickness, bonded and cross-linked. For example, DB-1; see Table 502-a for equivalents. See Section 502 C, Recommended Operating Procedure for Wide Bore Columns.

Column Operating Conditions:

230° C isothermal; if necessary, adjust temperature so that relative retention time (rrt) to phosalone of cis permethrin is about 1.55.

Carrier gas: helium or hydrogen; adjust flow rate so that phosalone elutes in about 8 min.

Injector temperature: 250° C

Detector

Electroconductivity, halogen mode (ELCD-X)

Detector Operating Conditions:

Base temperature 250° C; furnace temperature 900° C (or as specified in operating manual)

Reactor gas: hydrogen, flow as required by specific detector model; see operating manual

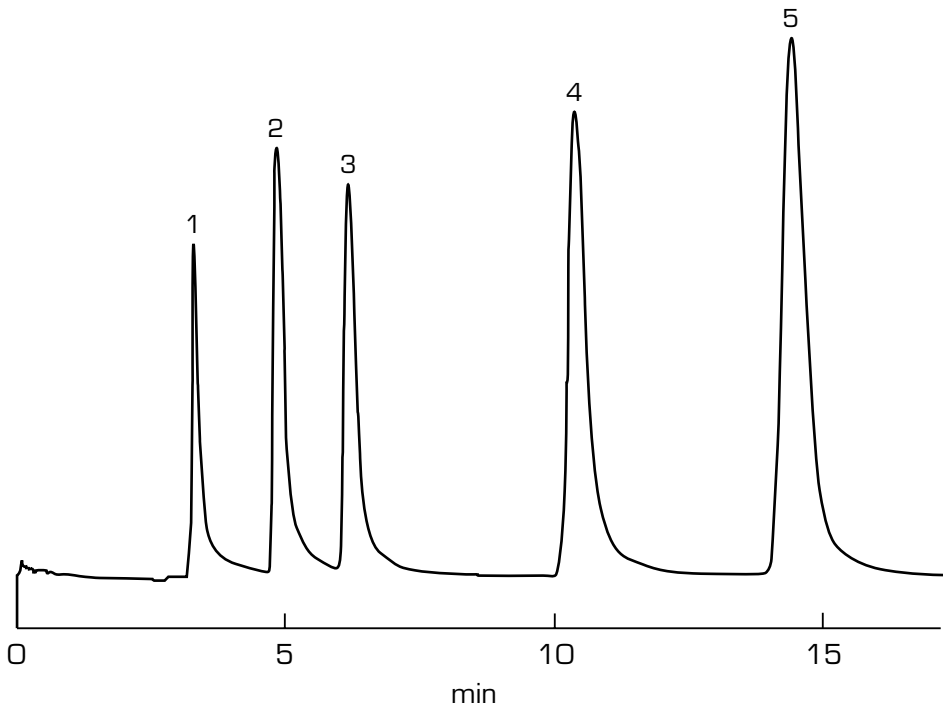
See Section 503 D for other information about ELCD operation.

Set detector electronics (amplification, attenuation) so that response to 18 ng phosalone is 50% full scale defection (FSD).

Other Considerations

Detector sensitivity can probably not be increased to match that of DG10, for the same residues.

Example chromatogram is on next page.



DG12

Chromatogram of: 1) 8.72 ng ofurace, 2) 9.96 ng iprodione, 3) 17.86 ng phosalone, 4) 11.01 ng prochloraz, and 5) 21.06 ng alpha-cypermethrin at the conditions described.

DG13 GLC, 50% PHENYL, 50% METHYL SILOXANE, 200° C, EC

Applicability

Determinative step is applicable to residues containing halogen, sulfur, or other electrophilic moieties. It is a general purpose system, but subject to interferences by nonpesticides.

Column

Wide bore capillary, 30 m m × 0.53 mm id, coated with 50% phenyl, 50% methyl substituted polysiloxane liquid phase, 1-1.5 µm film thickness, bonded and cross-linked. For example, DB-17; see Table 502-a for equivalents. See Section 502 C, Recommedned Operating Procedure for Wide Bore Columns.

Column Operating Conditions:

200° C, isothermal; if necessary, adjust temperature so that relative retention time to chlorpyrifos (rrt_c) of p,p'-DDT is 3.5 ± 0.07 or rrt_c of ethion is 3.36 ± 0.07.

Carrier gas: helium; adjust flow rate so that chlorpyrifos elutes in about 4 ± 0.5 min (about 20 mL/min).

Injector temperature: 250° C

Detector

Electron Capture (EC)

Detector Operating Conditions:

350° C

Make-up gas: nitrogen or argon/methane (95:5), at 30 mL/min

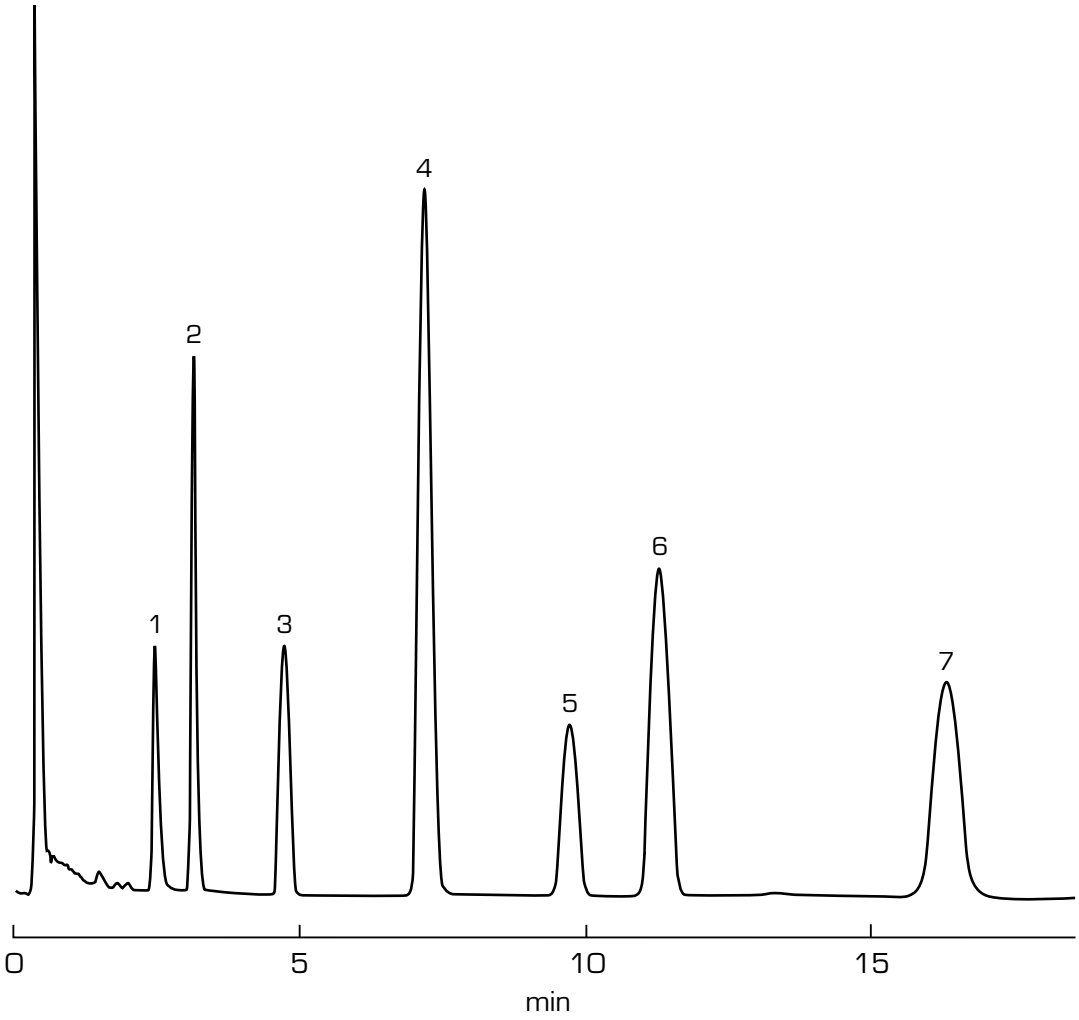
See Section 503 B for other information about EC detector operation.

Set detector electronics (amplification, attenuation) so that response to 0.15 ng chlorpyrifos (on an amount within the detector's linear range) is 50% full scale deflection (FSD).

Other Considerations

Rrt_cs and ng required to cause 50% FSD response are listed in Appendix I, PESTDATA (many data in PESTDATA were collected using equivalent packed column). Response data in Appendix I are based on detector sensitivity of 50% FSD to 1.5 ng chlorpyrifos.

Example chromatogram is on next page.



DG13

Chromatogram of: 1) 0.048 ng dicloran, 2) 0.049 ng heptachlor, 3) 0.15 ng chlorpyrifos, 4) 0.23 ng endosulfan I, 5) 0.22 ng captan, 6) 0.24 ng endrin, and 7) 0.24 ng p,p′-DDT at the conditions described.

DG14 GLC, 50% PHENYL, 50% METHYL SILOXANE, 200° C, FPD-P

Applicability

Determinative step is applicable to residues containing phosphorus. It is particularly useful for residues such as organophosphate pesticides.

Column

Wide bore capillary, 30 mm × 0.53 mm id, coated with 50% phenyl, 50% methyl substituted polysiloxane liquid phase, 1-1.5 µm film thickness, bonded and cross-linked. For example, DB-17; see Table 502-a for equivalents. See Section 502 C, Recommended Operating Procedure for Wide Bore Columns.

Column Operating Conditions:

200° C, isothermal; if necessary, adjust temperature so that relative retention time to chlorpyrifos (rrt_c) of ethion is 3.36 ± 0.07.

Carrier gas: helium; adjust flow rate so that chlorpyrifos elutes in about 4 ± 0.5 min (about 20 mL/min).

Injector temperature: 250° C

Detector

Flame photometric, phosphorus mode (FPD-P)

Detector Operating Conditions:

225-250° C

See Section 503 C for other information about FPD operation.

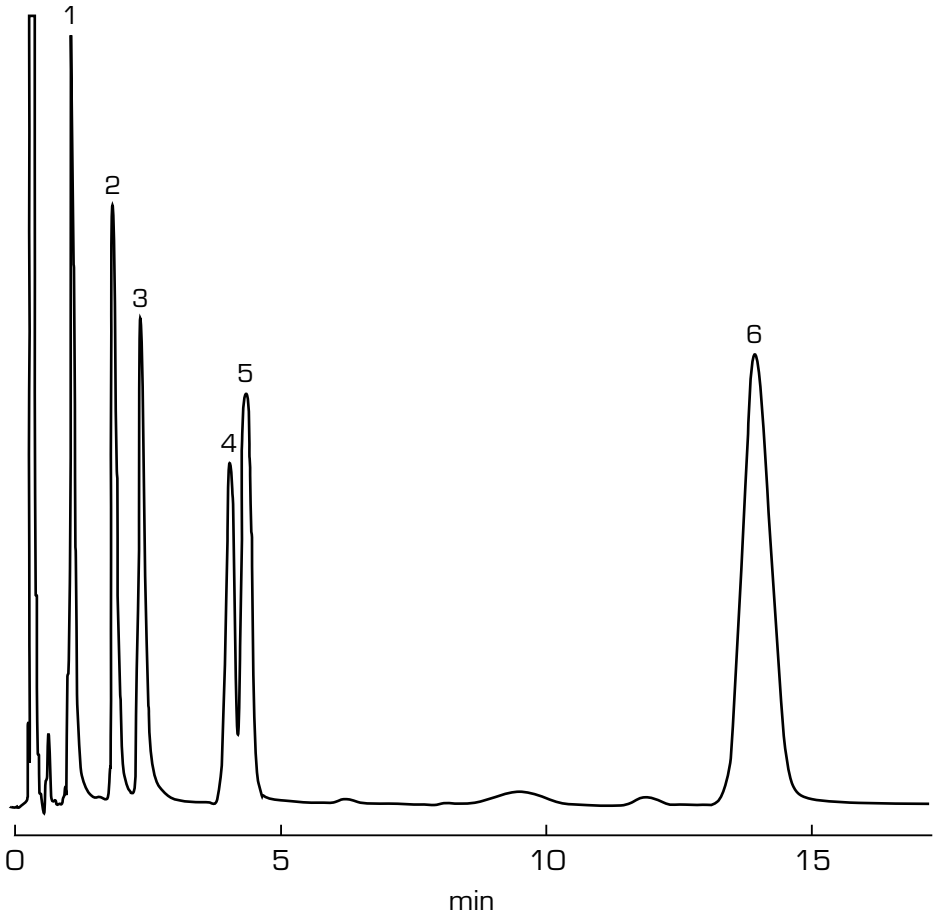
Set detector electronics (amplification, attenuation) so that response to 1.5 ng chlorpyrifos is 50% full scale deflection (FSD).

A properly functioning system will give a response to 1.5 ng omethoate of ≥50% FSD.

Other Considerations

Rrt_cs and ng required to cause 50% FSD response are listed in Appendix I, PESTDATA (many data in PESTDATA were collected using equivalent packed column).

Example chromatogram is on next page.



DG14

Chromatogram of: 1) 1.0 ng acephate, 2) 1.5 ng omethoate, 3) 1.0 ng monocrotophos, 4) 1.0 ng pirimiphos-methyl, 5) 1.0 ng chlorpyrifos, and 6) 3.0 ng ethion at the conditions described.

DG15 GLC, 50% PHENYL, 50% METHYL SILOXANE, 230° C, FPD-S

Applicability

Determinative step is applicable to residues containing sulfur. It is particularly useful for residues such as propargite, thiabendazole, and ethofumesate.

Column

Wide bore capillary, 30 m m × 0.53 mm id, coated with 50% phenyl, 50% methyl substituted polysiloxane liquid phase, 1-1.5 µm film thickness, bonded and cross-linked. For example, DB-17; see Table 502-a for equivalents. See Section 502 C, Recommended Operating Procedure for Wide Bore Columns.

Column Operating Conditions:

200° C, isothermal; if necessary, adjust temperature so that relative retention time to chlorpyrifos (rrt_c) of ethion is 3.36 ± 0.07.

Carrier gas: helium; adjust flow rate so that chlorpyrifos elutes in about 4 ± 0.5 min (about 20 mL/min).

Injector temperature: 250° C

Detector

Flame photometric, sulfur mode (FPD-S)

Detector Operating Conditions:

225-250° C

See Section 503 C for other information about FPD operation.

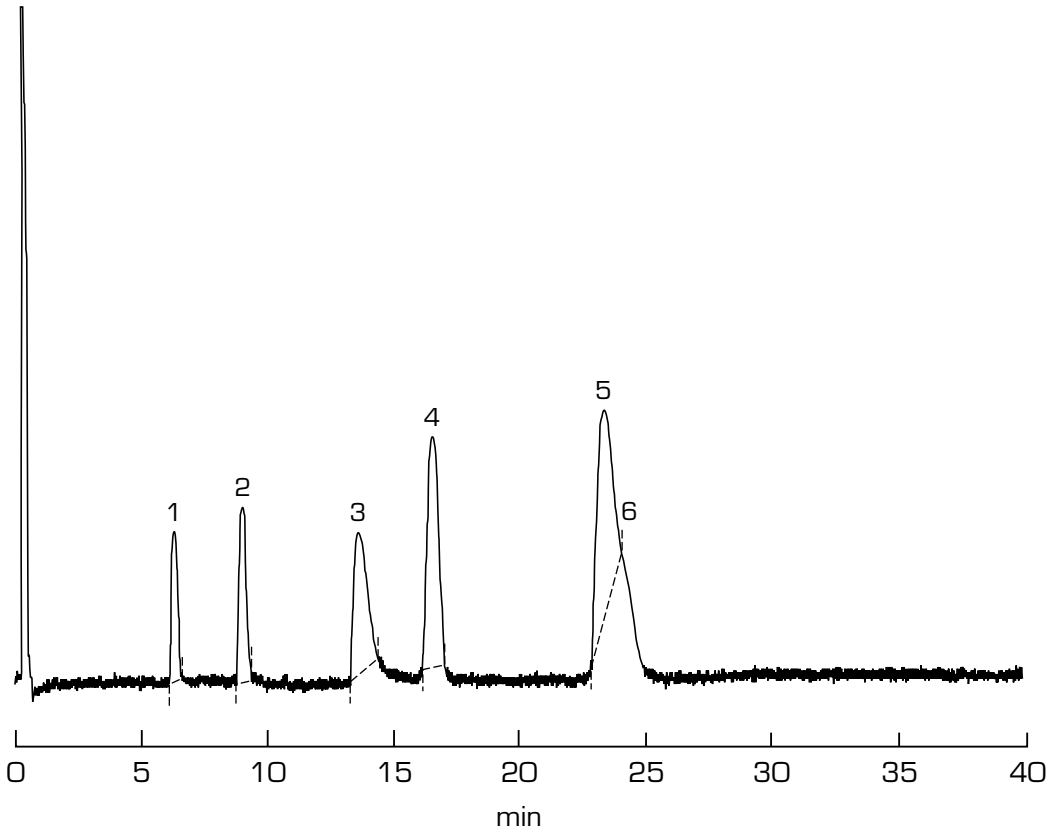
Set detector electronics (amplification, attenuation) to produce greatest possible response (50% full scale deflection [FSD]) to 15 ng chlorpyrifos is reasonable).

Other Considerations

Detector is not linear; quantitation of residues may be calculated from calibration curve (response vs amount injected).

Rrt_cs and ng required to cause 50% FSD response are listed in Appendix I, PESTDATA (many data in PESTDATA were collected using equivalent packed column).

Example chromatogram is on next page.



DG15

Chromatogram of: 1) 2.5 ng ethofumesate, 2) 5.0 ng endosulfan I, 3) 12.5 ng thiabendazole, 4) 10.0 ng endosulfan II, 5) 15.0 ng propargite, and 6) 15.0 ng endosulfan sulfate at the conditions described. Using this system, 5.0 ng chlorpyrifos caused about 50% FSD response.

DG16 GLC, 50% PHENYL, 50% METHYL SILOXANE, 200° C, ELCD-X

Applicability

Determinative step is applicable to residues containing halogen. It is particularly useful for residues such as chlorinated hydrocarbon pesticides and polychlorinated biphenyls.

Column

Wide bore capillary, 30 m m × 0.53 mm id, coated with 50% phenyl, 50% methyl substituted polysiloxane liquid phase, 1-1.5 µm film thickness, bonded and cross-linked. For example, DB-17; see Table 502-a for equivalents. See Section 502 C, Recommended Operating Procedure for Wide Bore Columns.

Column Operating Conditions:

200° C, isothermal; if necessary, adjust temperature so that relative retention time to chlorpyrifos (rrt_c) of p,p¢-DDT is 3.5 ± 0.07.

Carrier gas: helium; adjust flow rate so that chlorpyrifos elutes in about 4 ± 0.5 min (about 20 mL/min).

Injector temperature: 250° C

Detector

Electroconductivity, halogen mode (ELCD-X)

Detector Operating Conditions:

Base temperature 250° C; furnace temperature 900° C (or as specified in operating manual)

Reactor gas: hydrogen, flow as required by specific detector model; see operating manual

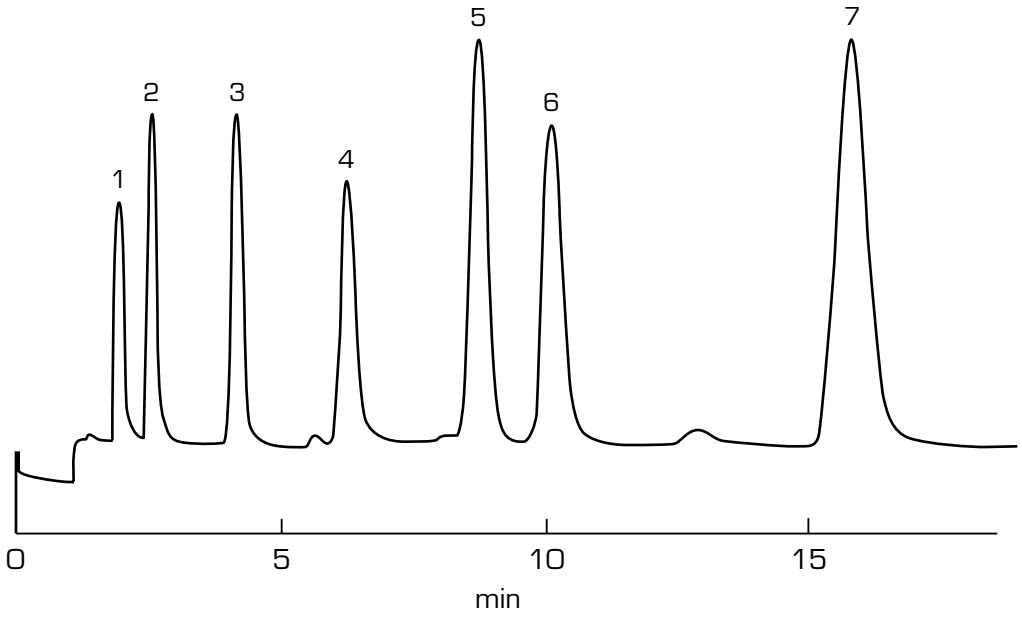
See Section 503 D for other information about ELCD operation.

Set detector electronics (amplification, attenuation) so that response to 1.5 ng chlorpyrifos is 50% full scale deflection (FSD).

Other Considerations

Rrt_cs and ng required to cause 50% FSD response are listed in Appendix I, PESTDATA (many data in PESTDATA were collected using equivalent packed column).

Example chromatogram is on next page.



DG16

Chromatogram of: 1) 0.85 ng dicloran, 2) 0.58 ng heptachlor, 3) 1.65 ng chlorpyrifos, 4) 1.01 ng endosulfan I, 5) 4.58 ng captan, 6) 1.56 ng endrin, and 7) 3.56 ng p,p'-DDT at the conditions described.

DG17 GLC, 50% PHENYL, 50% METHYL SILOXANE, 200° C, N/P

Applicability

Determinative step is applicable to residues containing nitrogen. It is particularly useful for residues such as triazines, triazoles, and THPI (captan metabolite).

Column

Wide bore capillary, 30 m m × 0.53 mm id, coated with 50% phenyl, 50% methyl substituted polysiloxane liquid phase, 1-1.5 µm film thickness, bonded and cross-linked. For example, DB-17; see Table 502-a for equivalents. See Section 502 C, Recommended Operating Procedure for Wide Bore Columns.

Column Operating Conditions:

200° C, isothermal; if necessary, adjust temperature so that relative retention time to chlorpyrifos (rrt_c) of ethion is 3.36 ± 0.07.

Carrier gas: helium; adjust flow rate so that chlorpyrifos elutes in about 4 ± 0.5 min (about 20 mL/min).

Injector temperature: 250° C

Detector

Alkali bead detector, nitrogen selective (N/P)

Detector Operating Conditions:

250° C

3.7 ± 0.1 mL/min hydrogen and 110 mL/min air

See Section 503 E for other information about N/P detector operation.

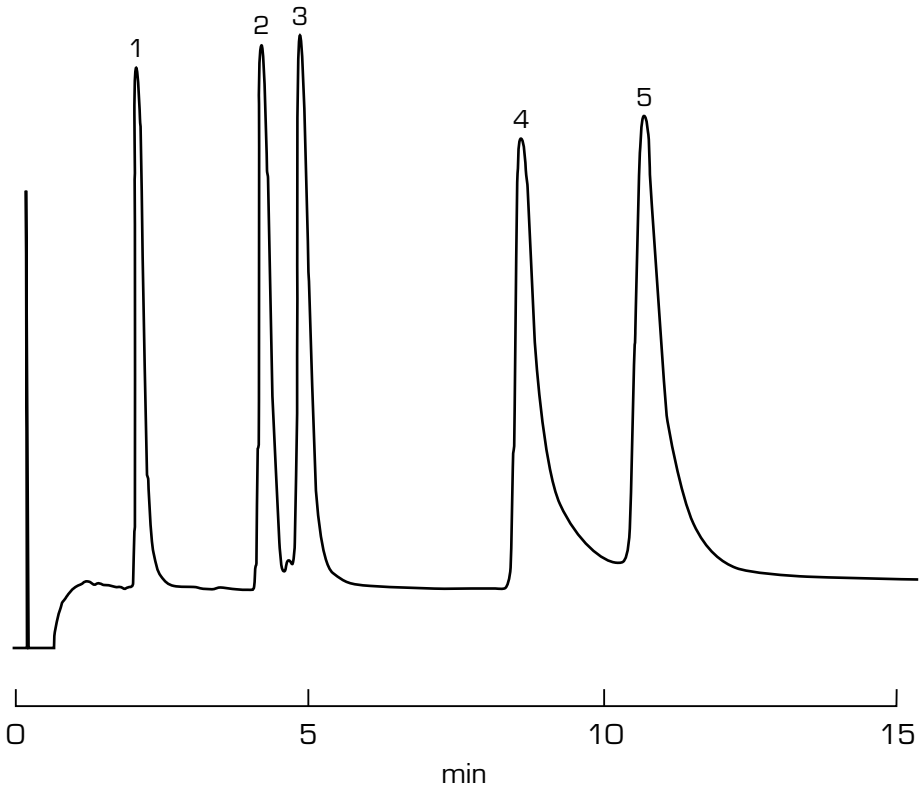
Set detector electronics (amplification, attenuation) so that response to 1.5 ng chlorpyrifos is 50% full scale deflection (FSD).

A properly functioning system will give a response to 1.5 ng omethoate of ≥50% FSD.

Other Considerations

Rrt_cs and ng required to cause 50% FSD response are listed in Appendix I, PESTDATA (many data in PESTDATA were collected using equivalent packed column).

Example chromatogram is on next page.



DG17

Chromatogram of: 1) 1.5 ng atrazine, 2) 1.5 ng chlorpyrifos, 3) 15.0 ng carbaryl, 4) 10.0 ng imazalil, and 5) 5.0 ng procyazine at the conditions described.

DG18 GLC, 50% CYANOPROPYLPHENYL, 50% METHYL SILOXANE, 200° C, EC

Applicability

Determinative step is applicable to residues containing halogen, sulfur, or other electrophilic moieties. It is a general purpose system, subject to interferences from nonpesticides; it is particularly useful for separating BHC isomers and hexachlorobenzene.

Column

Wide bore capillary, 30 m × 0.53 mm id, coated with 50% cyanopropylphenyl, 50% methyl substituted polysiloxane liquid phase, 1-1.5 µm film thickness, bonded and cross-linked. For example, DB-225; see Table 502-a for equivalents. See Section 502 C, Recommended Operating Procedure for Wide Bore Columns.

Column Operating Conditions:

200° C, isothermal; if necessary, adjust temperature so that relative retention time to chlorpyrifos (rrt_c) of lindane is 0.69 ± 0.02 and p,p¢-DDT is 3.6 ± 0.06 or rrt_c of ethion is 3.9 ± 0.1.

Carrier gas: helium; adjust flow rate so that chlorpyrifos elutes in about 5.5 ± 0.5 min (about 20 mL/min).

Injector temperature: 250° C

Detector

Electron Capture (EC)

Detector Operating Conditions:

350° C

Make-up gas: nitrogen or argon/methane (95:5), a 30 mL/min

See Section 503 B for other information about EC detector operation.

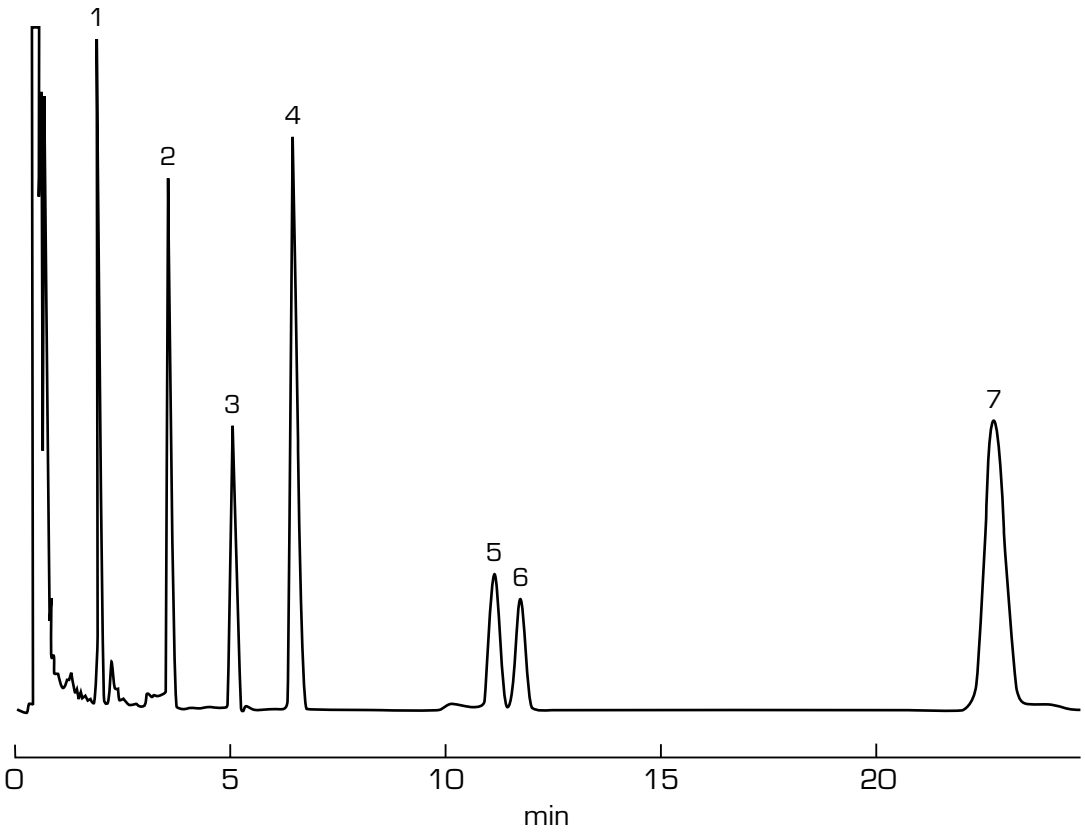
Set detector electronics (amplification, attenuation) so that response to 0.15 ng chlorpyrifos (on an amount within the detector's linear range) is 50% full scale deflection (FSD).

Other Considerations

Columns containing cyano moieties in the phase must not be connected to nitrogen selective or electrolytic conductivity detectors, so this column cannot be used with a different detector to confirm residues tentatively identified using this system.

Rrt_cs and ng required to cause 50% FSD response are listed in Appendix I, PESTDATA (many data in PESTDATA were collected using equivalent packed column). Response data in Appendix I are based on detector sensitivity of 50% FSD to 1.5 ng chlorpyrifos.

Example chromatogram is on next page.



DG18

Chromatogram of: 1) 0.032 ng hexachlorobenzene, 2) 0.049 ng α-BHC, 3) 0.056 lindane, 4) 0.15 ng chlorpyrifos, 5) 0.054 ng β-BHC, 6) 0.054 ng δ-BHC, and 7) 0.201 ng p,p′-DDT at the conditions described.

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Form FDA 2905a (6/92)

DG19 GLC, 50% CYANOPROPYLPHENYL, 50% METHYL SILOXANE, 200° C, FPD-P

Applicability

Determinative step is applicable to residues containing phosphorus. It is particularly useful for residues such as organophosphate pesticides.

Column

Wide bore capillary, 30 m × 0.53 mm id, coated with 50% cyanopropylphenyl, 50% methyl substituted polysiloxane liquid phase, 1-1.5 µm film thickness, bonded and cross-linked. For example, DB-225; see Table 502-a for equivalents. See Section 502 C, Recommended Operating Procedure for Wide Bore Columns.

Column Operating Conditions:

200° C, isothermal; if necessary, adjust temperature so that relative retention time to chlorpyrifos (rrt_c) of ethion is 3.9 ± 0.1.

Carrier gas: helium; adjust flow rate so that chlorpyrifos elutes in about 5.5 ± 0.5 min (about 20 mL/min).

Injector temperature: 250° C

Detector

Flame photometric, phosphorus mode (FPD-P)

Detector Operating Conditions:

225-250° C

See Section 503 C for other information about FPD operation.

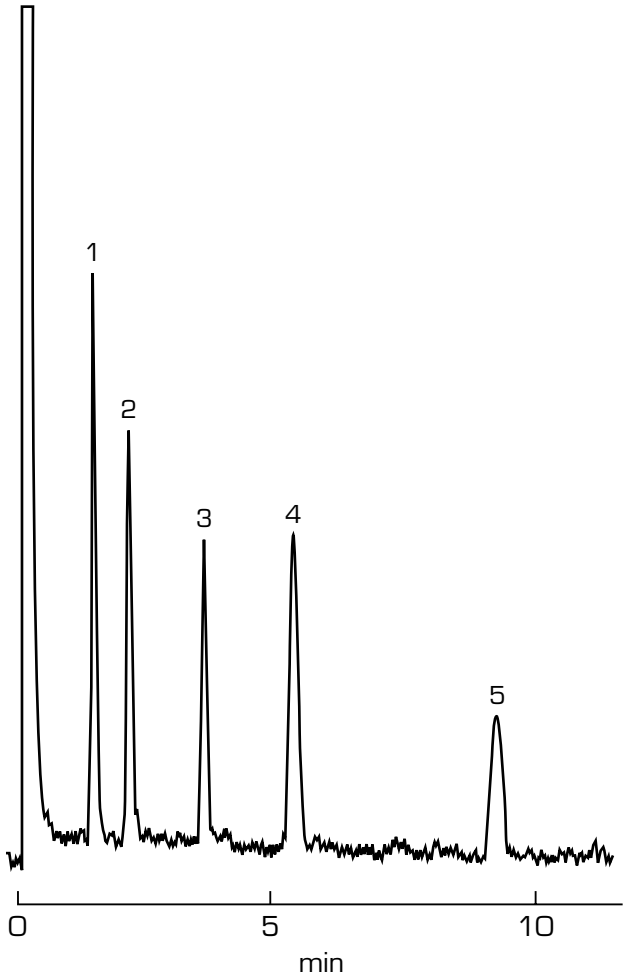
Set detector electronics (amplification, attenuation) so that response to 1.5 ng chlorpyrifos is 50% full scale deflection (FSD).

Other Considerations

Columns containing cyano moieties in the phase must not be connected to nitrogen selective or electrolytic conductivity detectors, so this column cannot be used with a different detector to confirm residues tentatively identified using this system.

Rrt_cs and ng required to cause 50% FSD response are listed in Appendix I, PESTDATA (many data in PESTDATA were collected using equivalent packed column).

Example chromatogram is on next page.



DG19

Chromatogram of: 1) 0.5 ng methamidophos, 2) 1.0 ng diazinon, 3) 1.0 ng acephate, 4) 1.5 ng chlorpyrifos, and 5) 1.0 ng monocrotophos at the conditions described.

303: METHOD II FOR NONFATTY FOODS

BASIC REFERENCE

Mills, P.A., *et al.* (1963) *J. Assoc. Off. Agric. Chem.* **46**, 186-191

GENERAL PRINCIPLES

Residues are extracted by blending with acetonitrile or water and acetonitrile, then transferred into petroleum ether by liquid-liquid partitioning. Subsequent cleanup of the extract with Florisil column chromatography results in an extract suitable for determination by GLC; two elution systems produce different elution patterns, useful in confirmatory or additional analyses.

The amount of sample represented in the final solution is calculated from the aliquot of acetonitrile extract used and the proportion of petroleum ether retrieved from the partitioning step; this calculation is valid only when the original filtered extract is homogeneous. Variations in the extraction step are used for products of high (>5%) sugar content to ensure homogeneity.

APPLICABILITY

Consult Guide to PAM I for additional information pertinent to the appropriate application of multiresidue methodology.

Method is generally applicable to relatively nonpolar residues in nonfatty commodities, i.e., fruits and vegetables containing ≤2 g fat in 100 g sample. Extraction E1 is applicable to products with high moisture (>75%) content; that extraction is also applicable to eggs if sample size is reduced (Extraction E2). Extraction E3 is applicable to dry products (<75% water), E4 to products with 5-15% sugar, and E5 to products with >15% sugar. See Section 201 for percentages fat, water, and sugar of many commodities. Florisil cleanup step prevents applicability to very polar residues. See Table 303-a, following the method description, for results of recovery tests.

METHOD MODULES

Choose from these method modules, using Figure 303-a for guidance:

Extraction (E)			**Recommended Use**
E1	(p. 303-7)	Extraction with acetonitrile, partition into petroleum ether with high moisture	fruits and vegetables (>75%), and low sugar (<5%), low fat (<2%)
E2	(p. 303-8)	Extraction from eggs with acetonitrile, partition into petroleum ether	whole eggs
E3	(p. 303-9)	Extraction with water/acetonitrile, partition into petroleum ether	dried egg whites, grains, and other foods with low moisture (<75%), low fat (<2%)
E4	(p. 303-9)	Extraction with acetonitrile and water, partition into petroleum ether	fruits and other foods with high sugar (5-15%)
E5	(p. 303-10)	Extraction with heated acetonitrile and water, partition into petroleum ether	fruits and other foods with very high sugar (>15%)

Cleanup (C)

C1	(p. 303-13)	Florisil column cleanup, with three ethyl ether/petroleum ether eluants	for relatively nonpolar residues
C2	(p. 303-14)	Florisil column cleanup, with three methylene chloride eluants	alternative to C1, some additional residues recovered

Determination (D)

(See Section 302 for full details of GLC modules.)

DG 1	(p. 302-33)	GLC, 100% methyl siloxane column, 200°, EC detector	residues with halogen, sulfur, other moieties
DG 2	(p. 302-35)	GLC, 100% methyl siloxane column, 200°, FPD-P	residues with phosphorus
DG 3	(p. 302-37)	GLC, 100% methyl siloxane column, 200°, ELCD-X	residues with halogen
DG 4	(p. 302-39)	GLC, 100% methyl siloxane column, 200°, ELCD-N	residues with nitrogen
DG 5	(p. 302-41)	GLC, 100% methyl siloxane column, 200°, N/P detector	residues with nitrogen or phosphorus
DG 7	(p. 302-45)	GLC, 100% methyl siloxane column, 130°, EC detector	early eluting residues with halogen, sulfur, other moieties
DG10	(p. 302-51)	GLC, 100% methyl siloxane column, 230°, EC detector	late eluting residues with halogen, sulfur, other moieties
DG12	(p. 302-55)	GLC, 100% methyl siloxane column, 230°, ELCD-X	late eluting residues with halogen
DG13	(p. 302-57)	GLC, 50% phenyl, 50% methyl siloxane column, 200°, EC detector	residues with halogen, sulfur, other moieties
DG14	(p. 302-59)	GLC, 50% phenyl, 50% methyl siloxane column, 200°, FPD-P	residues with phosphorus
DG16	(p. 302-63)	GLC, 50% phenyl, 50% methyl siloxane column, 200°, ELCD-X	residues with halogen
DG17	(p. 302-65)	GLC, 50% phenyl, 50% methyl siloxane column, 200°, N/P detector	residues with nitrogen or phosphorus

304: METHOD FOR FATTY FOODS

BASIC REFERENCE

Mills, P.A. (1959) *J. Assoc. Off. Agric. Chem.* **42,** 734-740

GENERAL PRINCIPLES

Fat and residues are extracted from fatty foods and dissolved in an organic solvent. Residues are separated from the extracted fat to produce a cleaned up extract solution suitable for determination by gas chromatography.

APPLICABILITY

Consult Guide to PAM I for additional information pertinent to the appropriate application of multiresidue methodology.

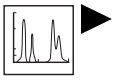
Method is applicable to moderately nonpolar residues in fatty foods. Residue polarity will affect recovery in Cleanups 1 and 2; neither very nonpolar nor very polar residues are recovered completely. See Table 304-a, following the method description, for results of recovery tests.

METHOD MODULES

Choose from these method modules, using Figure 304-a for guidance:

Extraction (E)		Recommended Use	
E1	(p. 304-5)	Extraction of fat with sodium sulfate, petroleum ether	animal tissues, fatty fish
E2	(p. 304-7)	Small scale extraction of fat with sodium sulfate, petroleum ether	animal tissues, fatty fish
E3	(p. 304-9)	Extraction of fat by filtering	butter, oils
E4	(p. 304-11)	Extraction of fat with solvents from denatured product	cheese, milk, egg yolks, dried whole eggs
E5	(p. 304-13)	Extraction of fat with solvents	oilseeds, high fat feeds or feed materials, grains, nuts
Cleanup (C)			
C1	(p. 304-15)	Acetonitrile-petroleum ether partitioning, Florisil column cleanup, three mixed ether eluants	for relatively few samples
C2	(p. 304-17)	Acetonitrile-petroleum ether partitioning, Florisil column cleanup, three methylene chloride eluants	for better cleanup than C1
C3	(p. 304-18)	Acetonitrile-petroleum ether partitioning, Florisil column cleanup, petroleum ether and three mixed ether eluants	to separate PCBs from most pesticides
C4	(p. 304-19)	Acetonitrile-petroleum ether partitioning, Florisil column cleanup, petroleum ether and three methylene chloride eluants	to separate PCBs from most pesticides
C5	(p. 304-21)	Gel permeation chromatography (GPC)	for efficient analysis of many samples (can be automated)

C6	(p. 304-24)	GPC, Florisil column (4 g) cleanup, three methylene chloride eluants	when C5 provides insufficient cleanup
C7	(p. 304-27)	Florisil column (4 g) cleanup, two mixed ether eluants, optional alkaline hydrolysis	to decrease time, solvent use compared to C1
C8	(p. 304-29)	Dispersion on alumina, Florisil column cleanup, three mixed ether eluants	to reduce time compared to C1; screening test only
C9	(p. 304-31)	Dispersion on alumina, Florisil columncleanup, three methylene chloride eluants	to reduce time compared to C3; screening test only



Determinations (D)

(See Section 302 for full details of GLC modules.)

DG 1	(p. 302-33)	GLC, 100% methyl siloxane column, 200°, EC detector	residues with halogen, sulfur, other moieties
DG 2	(p. 302-35)	GLC, 100% methyl siloxane column, 200°, FPD-P	residues with phosphorus
DG 3	(p. 302-37)	GLC, 100% methyl siloxane column, 200°, ELCD-X	residues with halogen
DG 4	(p. 302-39)	GLC, 100% methyl siloxane column, 200°, ELCD-N	residues with nitrogen
DG 5	(p. 302-41)	GLC, 100% methyl siloxane column, 200°, N/P	residues with nitrogen or phosphorus
DG 7	(p. 302-45)	GLC, 100% methyl siloxane column, 130°, EC detector	early eluting residues with halogen, sulfur, other moieties
DG10	(p. 302-51)	GLC, 100% methyl siloxane column, 230°, EC detector	late eluting residues with halogen, sulfur, other moieties
DG12	(p. 302-55)	GLC, 100% methyl siloxane column, 230°, ELCD-X	late eluting residues with halogen
DG13	(p. 302-57)	GLC, 50% phenyl, 50% methyl siloxane column, 200°, EC detector	residues with halogen, sulfur, other moieties
DG14	(p. 302-59)	GLC, 50% phenyl, 50% methyl siloxane column, 200°, FPD-P	residues with phosphorus
DG16	(p. 302-63)	GLC, 50% phenyl, 50% methyl siloxane column, 200°, ELCD-X	residues with halogen
DG17	(p. 302-65)	GLC, 50% phenyl, 50% methyl siloxane column, 200°, N/P detector	residues with nitrogen or phosphorus

Index to PAM I Methods, by Chemicals Tested for Recovery

Codes: C: complete (recovery); P: partial; S: small; V: variable; R: recovered; NR: not recovered

Index to Names Used for Chemicals in PAM I

demeton-S sulfone: **Use:** demeton-S sulfone
demeton-S sulfoxide: **Use:** demeton-S sulfoxide
demeton-S-methyl: **Use:** metasystox thiol
demeton-S-methyl sulfoxide: **Use:** oxydemeton-methyl
demeton-S-methylsulphon: **Use:** oxydemeton-methyl sulfone
Demosan: **Use:** chloroneb
Derosal: **Use:** carbendazim
des N-isopropyl isofenphos: **Use:** des N-isopropyl isofenphos
des N-isopropyl isofenphos oxygen analog: **Use:** des N-isopropyl isofenphos oxygen analog
desdiethyl simazine: **Use:** desdiethyl simazine
desdiisopropyl propazine: **Use:** desdiethyl simazine
desethyl simazine: **Use:** desethyl simazine
desethyldesisopropyl atrazine: **Use:** desdiethyl simazine
desisopropyl atrazine: **Use:** desethyl simazine
desmedipham: **Use:** desmedipham
desmethyl diphenamid: **Use:** desmethyl diphenamid
desmethyl norflurazon: **Use:** desmethyl norflurazon
Dessin: **Use:** dinobuton
Devrinol: **Use:** napropamide
di(2-ethylhexyl) phthalate: **Use:** bis(2-ethylhexyl) phthalate
di(n-butyl) phthalate: **Use:** dibutyl phthalate
di-allate: **Use:** di-allate
di-n-octyl phthalate: **Use:** di-n-octyl phthalate
dialifor: **Use:** dialifor
dialifos: **Use:** dialifor
diazinon: **Use:** diazinon
diazinon metabolite: **Use:** CGA 14128
diazinon metabolite: **Use:** GS-31144
diazinon metabolite (hydrolysis product): **Use:** G-27550
diazinon oxon: **Use:** diazinon oxygen analog
diazinon oxygen analog: **Use:** diazinon oxygen analog
diazoxon: **Use:** diazinon oxygen analog
Dibrom: **Use:** naled
dibromochloropropane: **Use:** dibromochloropropane
dibutalin: **Use:** butralin
dibutyl 1,2-benzenedicarboxylate: **Use:** dibutyl phthalate
dibutyl phthalate: **Use:** dibutyl phthalate
dicamba: **Use:** dicamba
dicamba methyl ester: **Use:** dicamba methyl ester
Dicarbam: **Use:** carbaryl
dichlobenil: **Use:** dichlobenil
dichlobenil metabolite: **Use:** 2,6-dichlorobenzamide
dichlofenthion: **Use:** dichlofenthion
dichlofluanid: **Use:** dichlofluanid
dichlone: **Use:** dichlone
dichlormid: **Use:** N, N-diallyl dichloroacetamide
dichlorobenzene, p-: **Use:** dichlorobenzene, p-
dichlorobenzophenone, o,p'-: **Use:** dichlorobenzophenone, o,p'-
dichlorobenzophenone, p,p'-: **Use:** dichlorobenzophenone, p,p'-
dichlorodiphenyldichloroethane: **Use:** TDE, p,p'-
dichlorofenthion: **Use:** dichlofenthion
dichlorprop: **Use:** dichlorprop
dichlorprop methyl ester: **Use:** dichlorprop methyl ester
dichlorvos: **Use:** dichlorvos
diclobutrazol: **Use:** diclobutrazol
diclofop: **Use:** diclofop
diclofop-methyl: **Use:** diclofop-methyl
diclofop-methyl metabolite: **Use:** diclofop
diclofop-methyl metabolite: **Use:** HOE-038182
diclofop-methyl metabolite: **Use:** HOE-030291
dicloran: **Use:** dicloran
Dicloran impurity: **Use:** 2,4-dichloro-6-nitrobenzenamine
dicofol breakdown product: **Use:** dichlorobenzophenone, p,p'-
dicofol breakdown product: **Use:** dichlorobenzophenone, o,p'-
dicofol, o,p'-: **Use:** dicofol, o,p'-
dicofol, p,p'-: **Use:** dicofol, p,p'-
dicophane: **Use:** DDT, p,p'-
dicrotophos: **Use:** dicrotophos
Dicuran: **Use:** chlorotoluron
dieldrin: **Use:** dieldrin
dieldrin photoproduct: **Use:** photodieldrin
diethamine: **Use:** dinitramine
diethatyl-ethyl: **Use:** diethatyl-ethyl
diethyl ((dimethoxyphosphinothioyl)thio)butanedioate: **Use:** malathion
diethyl ((dimethoxyphosphinyl)thio)butanedioate: **Use:** malathion oxygen analog
diethyl (4-methyl-1,3-dithiolan-2-ylidene)phosphoramidate: **Use:** mephosfolan
diethyl 1,3-dithiolan-2-ylidenephosphoramidate: **Use:** phosfolan
diethyl 2-(ethylthio)ethyl phosphate: **Use:** demeton-O oxygen analog
diethyl 4-nitrophenyl phosphate: **Use:** parathion oxygen analog
diethyl 6-methyl-2-(1-methylethyl)-4-pyrimidinyl phosphate: **Use:** diazinon oxygen analog
diethyl phthalate: **Use:** diethyl phthalate
diethyl pyrazinyl phosphate: **Use:** thionazin oxygen analog
diethyl(3-chloro-4-methyl-2-oxo-2H-1-benzopyran-7-yl) phosphate: **Use:** coumaphos oxygen analog
diethylhexyl phthalate: **Use:** bis(2-ethylhexyl) phthalate
difenoconazole metabolite: **Use:** CGA 189138
difenoconazole metabolite: **Use:** CGA 205374
difenoconazole metabolite: **Use:** CGA 205375
difenoxuron: **Use:** difenoxuron
Difolatan: **Use:** captafol
diisobutyl phthalate: **Use:** diisobutyl phthalate
diisohexyl phthalate: **Use:** diisohexyl phthalate
diisooctyl 1,2-benzenedicarboxylate: **Use:** diisooctyl phthalate
diisooctyl phthalate: **Use:** diisooctyl phthalate
Dilan: **Use:** Dilan
Dimecron: **Use:** phosphamidon
dimephenthoate: **Use:** phenthoate
dimethachlor: **Use:** dimethachlor
dimethametryn: **Use:** dimethametryn
dimethazone: **Use:** clomazone
dimethenamid: **Use:** dimethenamid
dimethipin: **Use:** dimethipin
dimethoate: **Use:** dimethoate
dimethoate oxygen analog: **Use:** omethoate
dimethomorph (prop): **Use:** dimethomorph (prop)
dimethoxon: **Use:** omethoate
dimethyl (1,2-phenylenebis(iminocarbonothioyl))bis(carbamate): **Use:** thiophanate-methyl
dimethyl (1,2-phenylenebis(iminocarbonyl))bis(carbamate): **Use:** allophanate
dimethyl (2,2,2-trichloro-1-hydroxyethyl)phosphonate: **Use:** trichlorfon
dimethyl 1-methyl-3-(methylamino)-3-oxo-1-propenyl phosphate: **Use:** monocrotophos
dimethyl 1-methyl-N,N-(dimethylamino)-3-oxo-1-propenyl phosphate, (E)-: **Use:** dicrotophos
dimethyl 2,3,5,6-tetrachloro-1,4-benzenedicarboxylate: **Use:** DCPA

S-ethyl bis(2-methylpropyl)carbamothioate: **Use:** butylate
S-ethyl cyclohexylethylcarbamothioate: **Use:** cycloate
S-ethyl cyclohexylethylthiocarbamate: **Use:** cycloate
S-ethyl diethylcarbamothioate: **Use:** ethiolate
S-ethyl dipropylcarbamothioate: **Use:** EPTC
S-ethyl hexahydro-1H-azepine-1-carbothioate: **Use:** molinate
S-propyl butylethylcarbamothioate: **Use:** pebulate
S-propyl dipropylcarbamothioate: **Use:** vernolate
Safrotin: **Use:** propetamphos
Salithion: **Use:** dioxabenzofos
SAN 371F: **Use:** oxadixyl
SAN 619 F: **Use:** cyproconazole
SAN-582H: **Use:** dimethenamid
Sandofan: **Use:** oxadixyl
Santoquin: **Use:** ethoxyquin
Satisfar: **Use:** etrimfos
Saturn: **Use:** thiobencarb
Savey: **Use:** hexythiazox
schradan: **Use:** schradan
Scout: **Use:** tralomethrin
SD 11831: **Use:** nitralin
SD 15418: **Use:** cyanazine
SD 43775: **Use:** fenvalerate
SD 8447: **Use:** Gardona
Sector: **Use:** butralin
Selecron: **Use:** profenofos
Sencor: **Use:** metribuzin
Sencor, deaminated diketo metabolite: **Use:** metribuzin, deaminated diketo metabolite
Sencor, deaminated metabolite: **Use:** metribuzin, deaminated metabolite
Sencor, diketo metabolite: **Use:** metribuzin, diketo metabolite
SES: **Use:** disul-Na
sesone: **Use:** disul-Na
sethoxydim: **Use:** sethoxydim
sethoxydim sulfoxide: **Use:** sethoxydim sulfoxide
Sevin: **Use:** carbaryl
Sicarol: **Use:** pyracarbolid
siduron: **Use:** siduron
Silosan: **Use:** pirimiphos-methyl
silvex: **Use:** silvex
silvex methyl ester: **Use:** silvex methyl ester
simazine: **Use:** simazine
simazine metabolite: **Use:** desdiethyl simazine
simazine metabolite: **Use:** desethyl simazine
simetryn: **Use:** simetryn
Sinbar: **Use:** terbacil
Sinox: **Use:** DNOC
Sipcam: **Use:** hexythiazox
SN 100309: **Use:** pyrimethanil
sodium 2,2-dimethyl-4,6-dioxo-5-(1-((2-propenyloxy)imino)=butyl)cyclohexanecarboxylate ion(1-): **Use:** alloxydim-sodium
sodium 2-(2,4-dichlorophenoxy)ethanyl hydrogen sulfate: **Use:** disul-Na
sodium 2-chloro-6-((4,6-dimethoxy-2-pyrimidinyl)thio)benzoate: **Use:** pyrithiobac-sodium
sodium salt of ((2-(ethoxymethyl) (2-ethyl-6-methylphenyl)=amino)-2-oxoethyl)sulfinyl)acetic acid: **Use:** CP 97290
sodium salt of ((ethoxymethyl)(2-(1-hydroxyethyl)-6-methylphenyl)amino)oxoacetic acid: **Use:** CP 108669
sodium salt of ((ethoxymethyl)(2-ethyl-6-(hydroxymethyl)=phenyl)amino)oxoacetic acid: **Use:** CP 106077
sodium salt of (ethoxymethyl) (2-ethyl-6-methylphenyl)=amino)oxoacetic acid: **Use:** CP 95200
sodium salt of 2-((2-ethyl-6-methylphenyl) (ethoxymethyl)amino)-2-oxoethanesulfonic acid: **Use:** CP 92429
sodium salt of 2-((ethoxymethyl)(2-(1-hydroxyethyl)-6-methyl=phenyl)amino)-2-oxoethanesulfonic acid: **Use:** CP 106070
Sofac: **Use:** cyfluthrin
Solgard: **Use:** pirimiphos-ethyl
Solicam: **Use:** norflurazon
Sonalan: **Use:** ethalfluralin
Sonar X: **Use:** fluridone
Sonax: **Use:** etaconazole
Spectracide: **Use:** diazinon
Spike: **Use:** tebuthiuron
Splendor: **Use:** tralkoxydim
Sportak: **Use:** prochloraz
Spot Kleen: **Use:** thiophanate-methyl
Stalker: **Use:** chlorfenapyr (prop)
Stam F-34: **Use:** propanil
Standak: **Use:** aldoxycarb
Staple: **Use:** pyrithiobac-sodium
stirofos: **Use:** Gardona
Stomp: **Use:** pendimethalin
Stop-scald: **Use:** ethoxyquin
Strobane: **Use:** Strobane
Subdue: **Use:** metalaxyl
Suffix: **Use:** benzoylprop-ethyl
sulfallate: **Use:** sulfallate
sulfanilamide: **Use:** sulfanilamide
sulfentrazone metabolite: **Use:** 3-desmethyl sulfentrazone
sulfocarb: **Use:** aldoxycarb
sulfotep: **Use:** sulfotep
Sulphenone: **Use:** Sulphenone
sulprofos: **Use:** sulprofos
sulprofos oxygen analog sulfone: **Use:** sulprofos oxygen analog sulfone
sulprofos sulfone: **Use:** sulprofos sulfone
sulprofos sulfoxide: **Use:** sulprofos sulfoxide
Sumi-alpha: **Use:** esfenvalerate
Sumicidin: **Use:** fenvalerate
Sumilex: **Use:** procymidone
Sumisclex: **Use:** procymidone
Sumithion: **Use:** fenitrothion
Sumithrin: **Use:** phenothrin
Summit: **Use:** triadimenol
Suncide: **Use:** propoxur
Super-Suffix: **Use:** flamprop-methyl
Supermethrin: **Use:** cypermethrin
Supracide: **Use:** methidathion
Surecide: **Use:** cyanofenphos
Surflan: **Use:** oryzalin
Sutan: **Use:** butylate
systam: **Use:** schradan
Systhane: **Use:** myclobutanil
Systox thiol: **Use:** demeton-S
Systox thiol sulfone: **Use:** demeton-S sulfone
Systox thiono: **Use:** demeton-O
Systox thiono oxygen analog: **Use:** demeton-O oxygen analog
Systox thiono sulfone: **Use:** demeton-O sulfone
Talstar: **Use:** bifenthrin

Tomahawk: **Use:** pirimiphos-methyl
Top Hand: **Use:** acetochlor
Top Notch: **Use:** acetochlor
Topas: **Use:** penconazole
Topaz: **Use:** penconazole
Topaze: **Use:** penconazole
Topsin M: **Use:** thiophanate-methyl
Torak: **Use:** dialifor
Tordon: **Use:** picloram
Toxakil: **Use:** toxaphene
toxaphene: **Use:** toxaphene
tralkoxydim: **Use:** tralkoxydim
tralomethrin: **Use:** tralomethrin
tralomethrin metabolite: **Use:** deltamethrin
tralomethrin metabolite: **Use:** deltamethrin, trans-
Tramat: **Use:** ethofumesate
Treflan: **Use:** trifluralin
tri(beta-chloroethyl) phosphate: **Use:** tris(beta-chloroethyl) phosphate
tri(N-butyl) phosphate: **Use:** tributyl phosphate
tri-allate: **Use:** tri-allate
triadimefon: **Use:** triadimefon
triadimefon metabolite: **Use:** KWG 1342
triadimefon metabolite: **Use:** triadimenol
triadimefon metabolite: **Use:** KWG 1323
triadimenol: **Use:** triadimenol
triadimenol metabolite: **Use:** KWG 1342
triasulfuron metabolite: **Use:** CGA 150829
triasulfuron metabolite: **Use:** CGA 195654
triasulfuron metabolite: **Use:** CGA 161149
triazamate: **Use:** triazamate
triazamate (prop): **Use:** triazamate
Triazid: **Use:** amitraz
triazophos: **Use:** triazophos
tribufos: **Use:** tribufos
Tribunil: **Use:** methabenzthiazuron
tributyl phosphate: **Use:** tributyl phosphate
tributyl phosphorotrithioate: **Use:** merphos
trichlorfon: **Use:** trichlorfon
trichlorobenzyl chloride metabolite: **Use:** 2,3,6-TBA
trichloronat: **Use:** trichloronat
trichlorophenyl ethanol: **Use:** 2,4,5-trichloro-alpha-methylbenzenemethanol
triclopyr: **Use:** triclopyr
triclopyr metabolite: **Use:** 3,5,6-trichloro-2-pyridinol
triclopyr metabolite: **Use:** 2-methoxy-3,5,6-trichloropyridine
triclopyr methyl ester: **Use:** triclopyr methyl ester
tricyclazole: **Use:** tricyclazole
tricyclazone: **Use:** tricyclazole
tridiphane: **Use:** tridiphane
triflumizole: **Use:** triflumizole
trifluralin: **Use:** trifluralin
triflusulfuron methyl ester: **Use:** triflusulfuron methyl ester
triflusulfuron-methyl: **Use:** triflusulfuron methyl ester
Trifmine: **Use:** triflumizole
trimethacarb metabolite: **Use:** 4-hydroxymethyl-3,5-dimethylphenyl methylcarbamate
trimethacarb metabolite : **Use:** 3-hydroxymethyl-4,5-dimethylphenyl methylcarbamate
trimethacarb metabolite : **Use:** 3-hydroxymethyl-2,5-dimethylphenyl methylcarbamate
Trimidal: **Use:** nuarimol
Triminol: **Use:** nuarimol
triphenyl phosphate: **Use:** triphenyl phosphate
tris(2-chloroethyl) phosphate: **Use:** tris(beta-chloroethyl) phosphate
tris(beta-chloroethyl) phosphate: **Use:** tris(beta-chloroethyl) phosphate
tris(chloropropyl) phosphate: **Use:** tris(chloropropyl) phosphate
Tritex: **Use:** alloxydim-sodium
Trithion: **Use:** carbophenothion
Trithion oxygen analog: **Use:** carbophenothion oxygen analog
Trithion oxygen analog sulfone: **Use:** carbophenothion oxygen analog sulfone
Trithion oxygen analog sulfoxide: **Use:** carbophenothion oxygen analog sulfoxide
Trithion sulfone: **Use:** carbophenothion sulfone
Trithion sulfoxide: **Use:** carbophenothion sulfoxide
Tritisan: **Use:** quintozene
Triumph: **Use:** isazofos
Tropotox: **Use:** MCPB
Truban: **Use:** etridiazole
Trysben: **Use:** 2,3,6-TBA
Tsumacide: **Use:** metolcarb
Tunic: **Use:** methazole
Tupersan: **Use:** siduron
Tycor: **Use:** Tycor
UC-21865: **Use:** aldoxycarb
UC21149: **Use:** aldicarb
Ultracide: **Use:** methidathion
Unden: **Use:** propoxur
Uniroyal D-014: **Use:** propargite
Usb 3584: **Use:** dinitramine
Valexon: **Use:** phoxim
vamidothion metabolite: **Use:** vamidothion sulfone
Van Dyk 264: **Use:** MGK 264
Vangard: **Use:** etaconazole
Vapona: **Use:** dichlorvos
Vapotone: **Use:** TEPP
VC-13: **Use:** dichlofenthion
Vegadex: **Use:** sulfallate
Velpar: **Use:** hexazinone
Verdict: **Use:** haloxyfop methyl ester
Vernam: **Use:** vernolate
vernolate: **Use:** vernolate
Vigil: **Use:** diclobutrazol
vinclozolin: **Use:** vinclozolin
vinclozolin metabolite B: **Use:** vinclozolin metabolite B
vinclozolin metabolite B, methylated: **Use:** vinclozolin
vinclozolin metabolite D: **Use:** 3,5-dichloroaniline
vinclozolin metabolite E: **Use:** vinclozolin metabolite E
vinclozolin metabolite S: **Use:** vinclozolin metabolite S
vinclozoline metabolite F: **Use:** vinclozolin metabolite F
Viran: **Use:** parathion
Vitavax: **Use:** carboxin
Volaton: **Use:** phoxim
Vondcaptan: **Use:** captan
Voronit: **Use:** fuberidazole
Vydate: **Use:** oxamyl
WAK3745: **Use:** NTN35884
Wakil: **Use:** oxadixyl
Warbex: **Use:** famphur
Waylay: **Use:** napropamide
Weed B Gon: **Use:** 2,4-D

Weedone: **Use:** 2,4,5-T
Whip: **Use:** fenoxaprop ethyl ester
Wipeout: **Use:** hydramethylnon
WL 41706: **Use:** fenpropathrin
WL 85871: **Use:** alpha-cypermethrin
XMC: **Use:** XMC
XRD 498: **Use:** flumetsulam
Xymiazole: **Use:** cymiazole
Zeldox: **Use:** hexythiazox
zeta-cypermethrin: **Use:** cypermethrin
Zinophos: **Use:** thionazin
Zobar: **Use:** 2,3,6-TBA
Zolone: **Use:** phosalone
Zorial: **Use:** norflurazon

Index to CAS Registry Numbers for Chemicals in PAM I

n/a = number not available

Index to Subjects Covered in PAM I

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Table 302-a: Recovery of Chemicals Through Method 302 (E1-E3 + DG1-DG19)
(acetone extraction, partitioning or Hydromatrix removal of water, GLC determination with various columns and detectors)

Chemical	Recovery[1]	Notes[2]
(4-chlorophenyl)-urea	NR	
1,2,4,5-tetrachloro-3-(methylthio)benzene	R	
1,2,4-triazole	V	N detector required.
2,3,5-trimethacarb	C	N detector required.
2,4,5-trichloro-alpha-methylbenzenemethanol	R	
2,4-dichloro-6-nitrobenzenamine	C (110%)	n=1
2,6-dichlorobenzamide	C	
2-chloroethyl myristate	C	
2-methoxy-3,5,6-trichloropyridine	C	Low temperature column recommended.
3,4,5-trimethacarb	C	N detector required.
3,4-dichloroaniline	V (44-84%)	
3,5-dichloroaniline	S (15-62%)	
3-(3,4-dichlorophenyl)-1-methoxyurea	R	GLC not reliable for quantitation.
3-carboxy-5-ethoxy-1,2,4-thiadiazole	NR	
3-chloro-5-methyl-4-nitro-1H-pyrazole	C	OV-101 peak tails severely.
3-ketocarbofuran	S (0-150%)	
3-methyl-4-nitrophenol	V (65-153%)	Interferences from sample extract may have caused variable results.
4'-hydroxy bifenthrin	C	High temperature GLC column required.
4-(dichloroacetyl)-1-oxa-4-azapiro[4.5]decane	C	Low level (0.05 ppm) fortification in corn grain obscured by matrix.
4-(phenylamino)phenol	C	
4-chlorobenzenamine	S (23-43%)	
4-chlorophenoxyaniline	S (10-29%)	Poor EC detector sensitivity; halogen-selective detector required.
6-benzyladenine	C	N detector required.
acephate	C	Wide bore or DEGS column required.
acetochlor	C	
acrinathrin	V (80-136%)	

[1] Codes: C: complete (>80%); P: partial (50-80%); S: small (<50%); V: variable (approximate percentage when known); R: recovered but no quantitative information available; NR: not recovered.

[2] Notes assume that extract is examined by GLC with columns at 200° C and, at a minimum, halogen-selective detector (DG3 or 16) and phosphorus-selective detectors (DG2 or 14 or 19). Notes indicate those chemicals that can be determined only by use of columns, temperatures, and/or detectors other than the minimal ones.

Table 302-a: Recovery Through 302 (E1-E3 + DG1-DG19)

Chemical	Recovery[1]	Notes[2]
alachlor	C	
aldrin	C	
allidochlor	C	Low temperature DEGS column used.
alpha-cypermethrin	C	
ametryn	C	N or S detector required.
aminocarb	C	N detector required.
amitraz	S (0-70%)	N detector, high temperature column required.
anilazine	V	GLC response variable.
aramite	C	
atrazine	C	
azinphos-ethyl	C	
azinphos-methyl	C	DEGS column unsuitable.
azinphos-methyl oxygen analog	C	
bendiocarb	C	N detector required.
benfluralin	C	
benodanil	C	
benoxacor	C	
bensulide	C	Results may be variable with certain GLC systems.
benzoylprop-ethyl	P (79%)	
BF 490-1	C	
BF 490-2	C	
BF 490-9	C	
BHC, alpha-	C	
BHC, beta-	C	
BHC, delta-	C	
bifenox	C	
bifenthrin	V (66-133%)	
binapacryl	C	N detector required.
biphenyl	C	FID required.
bitertanol	C	GLC with high temperature column, N/P detector required.
bromacil	C	
bromophos	C	
bromophos-ethyl	C	

Table 302-a: Recovery Through 302 (E1-E3 + DG1-DG19)

Chemical	Recovery[1]	Notes[2]
bromopropylate	C	
bromuconazole	C	
BTS 27919	C	N detector required.
Bulan	C	
bupirimate	C	N or S detector required.
butachlor	C	
butralin	V (77-90%)	N detector required.
butylate	V (73-99%)	N detector required.
cadusafos	C	
captafol	C	
captan	C	
carbaryl	C	N detector required for GLC determination; GLC not the method of choice.
carbetamide	C	N detector required.
carbofuran	C	N detector required for GLC determination; GLC not the method of choice.
carbophenothion	C	
carbophenothion oxygen analog	C	
carbophenothion sulfone	C	
carbosulfan	P (47-75%)	N or S detector required.
carboxin	C	N or S detector required.
CGA 100255	S (37-146%)	N detector required, but response is poor.
CGA 118244	V	
CGA 14128	C	
CGA 150829	V (40-111%)	N detector required; wide bore or DEGS column recommended.
CGA 171683	C	N detector, wide bore or DEGS column required.
CGA 37734	C	N detector required but response variable.
CGA 91305	V	
CGA 94689A	V (44-108%)	N detector required.
CGA 94689B	S (39-94%)	N detector required, but response varies widely with different columns.
CGA-232449	C	Needs N detector.
chlorbenside	C	
chlorbromuron	V (73-100%)	

Table 302-a: Recovery Through 302 (E1-E3 + DG1-DG19)

Chemical	Recovery[1]	Notes[2]
chlorbufam	C	
chlordane	C	
chlordane, cis-	C	
chlordane, trans-	C	
chlordimeform hydrochloride	P (80%)	
chlorethoxyfos	V (64-119%)	Recoveries performed with electron capture detector.
chlorfenapyr	P (73-82%)	
chlorfenvinphos, alpha-	C	
chlorfenvinphos, beta-	C	
chlorflurecol methyl ester	C	
chlorimuron ethyl ester	P (69-70%)	
chlormephos	C	Low temperature column required.
chlornitrofen	C (80%)	
chlorobenzilate	C	
chloroneb	C	Low temperature column required.
chloropropylate	P (64%)	
chlorothalonil	S	Recovery may be 0%.
chlorothalonil trichloro impurity	R	
chloroxuron	C	
chlorpropham	C	
chlorpyrifos	C	
chlorpyrifos oxygen analog	C	Wide bore or DEGS column recommended.
chlorpyrifos-methyl	C	
chlorthiophos	C	
chlorthiophos oxygen analog	C	
chlorthiophos sulfone	C	
chlorthiophos sulfoxide	C	
clodinafop-propargyl	V	Recovery test yielded very high recoveries (>200%) from wheat.
clofentezine	R	Degrades on GLC in presence of extract.
clomazone	C	
cloquintocet-mexyl	V (57-137%)	
coumaphos	C	

Table 302-a: Recovery Through 302 (E1-E3 + DG1-DG19)

Chemical	Recovery[1]	Notes[2]
coumaphos oxygen analog	C	
CP 51214	C	
crotoxyphos	C	
crufomate	C	
cyanazine	C	
cyanofenphos	C	
cyanophos	C	
cycloate	C	N or S detector required.
cycluron	C	N detector required.
cyfluthrin	C	High temperature column required.
cymoxanil	V (70-107%)	N detector required; GLC rrts and responses variable.
cypermethrin	C	
cyprazine	C	
cyproconazole	C	
cyprodinil	C	Needs N detector
cyromazine	S (16-20%)	
dazomet	S (<10%)	
DCPA	C	
DDE, o,p'-	C	
DDE, p,p'-	C	
DDT, o,p'-	C	
DDT, p,p'-	C	
deltamethrin	C	
demeton-O	C	
demeton-O sulfone	C	
demeton-O sulfoxide	C	
demeton-S	C	
demeton-S sulfone	C	Wide bore or DEGS column recommended.
demeton-S sulfoxide	C	Wide bore or DEGS column required.
des N-isopropyl isofenphos	C	
desisopropyl iprodione	P (67-84%)	
desmethyl norflurazon	V (63-200%)	
di-allate	C	

Table 302-a: Recovery Through 302 (E1-E3 + DG1-DG19)

Chemical	Recovery[1]	Notes[2]
dialifor	C	
diazinon	C	
diazinon oxygen analog	C (80%)	
dichlobenil	C	Low temperature column required.
dichlofenthion	C	
dichlofluanid	C	
dichlone	P (55%)	May break down.
dichlorvos	C	Low temperature column required; wide bore or DEGS recommended.
diclobutrazol	C	Wide bore column recommended.
diclofop-methyl	C	
dicloran	C	
dicofol, o,p'-	C	
dicofol, p,p'-	C	
dicrotophos	C	Wide bore or DEGS column required.
dieldrin	C	
diethatyl-ethyl	C	
difenoxuron	R	79-95% recovered at 1 and 5.5 ppm, but subject to interferences.
dimethachlor	C	
dimethametryn	C	N or S detector required.
dimethipin	C	
dimethoate	C	Wide bore or DEGS column recommended.
dimethomorph (prop)	V (87-133%)	High temperature column required.
dinitramine	C	N detector required.
dinobuton	C	
dinocap	C	N detector required.
dioxabenzofos	C	
dioxacarb	C	N detector required; used Megabore Carbowax column.
dioxathion	V (72-94%)	
diphenamid	V (57-155%)	N detector required.
diphenyl 2-ethylhexyl phosphate	C	mean recovery 104.2%, n=15
diphenylamine	C	N detector required.
disulfoton	C	

Table 302-a: Recovery Through 302 (E1-E3 + DG1-DG19)

Chemical	Recovery[1]	Notes[2]
disulfoton sulfone	C	
disulfoton sulfoxide	C	Wide bore or DEGS column required.
dithianon	NR	Breaks down in presence of extract.
diuron	C	Low temperature column required.
edifenphos	C	High recovery (113-121%) reported.
endosulfan I	C	
endosulfan II	C	
endosulfan sulfate	C	
endrin	C	
endrin aldehyde	C	
EPN	C	
esfenvalerate	C	High temperature column required.
etaconazole	C	Wide bore column recommended.
ethalfluralin	C	
ethephon	NR	
ethiofencarb	C	N or S detector required; responses variable.
ethiolate	C	Low temperature column, N or S detector required.
ethion	C	
ethion oxygen analog	C	
ethirimol	P (73%)	
ethofumesate	C	S selective detector required.
ethoprop	C	
ethoxyquin	C	N detector required.
ethyl p-toluene sulfonamide	C	N or S detector required.
ethylenethiourea	S (0-48%)	Short, low temperature DEGS or wide bore column, N or S detector required.
etridiazole	C	Low temperature column recommended.
etrimfos	C	
etrimfos oxygen analog	C	
famphur	C	
famphur oxygen analog	C	Quantitation affected by poor GLC.
fenamiphos	C	
fenamiphos sulfone	C	

Table 302-a: Recovery Through 302 (E1-E3 + DG1-DG19)

Chemical	Recovery[1]	Notes[2]
fenamiphos sulfoxide	C	
fenarimol	C	
fenarimol metabolite B	NR	
fenarimol metabolite C	S (6%)	
fenbuconazole	C	
fenfuram	C	
fenitrothion	C	
fenitrothion oxygen analog	C	
fenoxaprop ethyl ester	S (0-40%)	
fenoxycarb	C	N detector required.
fenpropimorph	C	N detector required.
fensulfothion	C	
fensulfothion oxygen analog	C	
fensulfothion sulfone	C	
fenthion	C	
fenthion oxygen analog	C	
fenthion oxygen analog sulfoxide	C	
fenthion sulfone	C	
fenvalerate	C	High temperature column required.
fipronil	S (0-72%)	Corn forage sample interfered with determination.
flamprop-M-isopropyl	C	
flamprop-methyl	C	
fluazifop butyl ester	C (78-112%)	
fluchloralin	C	
flucythrinate	C	High temperature column required.
fludioxonil	V (49-121%)	Requires N detector.
flusilazole	C	Wide bore column recommended.
fluvalinate	C	High temperature column required.
FOE 5043	C	
folpet	C	
fonofos	C	
fonofos oxygen analog	V (57-108%)	
formothion	C	

Table 302-a: Recovery Through 302 (E1-E3 + DG1-DG19)

Chemical	Recovery[1]	Notes[2]
fosthiazate	C	
fuberidazole	C	May break down in solution. Temp program separated from interference in tomato.
furilazole	C	
G-27550	C	N detector required.
Gardona	C	
heptachlor	C	
heptachlor epoxide	C	
heptenophos	C	
hexachlorobenzene	C	
hexaconazole	C	
hexazinone	P (57-76%)	N detector required; high temperature column may be needed.
imazalil	C	Wide bore column recommended.
imazamethabenz methyl ester	C	N detector required, though halogen-selective detector may respond.
IN-A3928	S (23-39%)	
IN-B2838	P (75-84%)	
IN-T3935	S (20%)	
IN-T3936	S (29-34%)	
IN-T3937	S (25%)	N detector required.
iprobenfos	C	
iprodione	C	
iprodione metabolite isomer	C	
isazofos	C	
isocarbamid	C	
isofenphos	C	
isofenphos oxygen analog	C	
isopropalin	C	N detector required.
isoprothiolane	C	
isoproturon	S (44-67%)	GLC poor; requires wide bore column; compound may degrade.
isoxaben	C	N detector required.
isoxaflutole	NR	Crop interference may have prevented measurement of recovery.

Table 302-a: Recovery Through 302 (E1-E3 + DG1-DG19)

Chemical	Recovery[1]	Notes[2]
jodfenphos	C	
kresoxim-methyl	P (73-89%)	
KWG 1323	C	
lambda-cyhalothrin	C	
lenacil	C	N detector required.
leptophos	C	
leptophos oxygen analog	C	
leptophos photoproduct	C	
lindane	C	
linuron	V (57-101%)	
malathion	C	
malathion oxygen analog	C	
MB 46513	C	
MB45950	S (0-35%)	
MB46136	S (0-19%)	
mecarbam	C	
mefluidide	R	123% recovered of 3 ppm added; subject to interference, poor GLC.
melamine	NR	
mephosfolan	C	
metalaxyl	C	N detector required but response variable.
metasystox thiol	C	
metazachlor	C	
methabenzthiazuron	C (85-86%)	
methamidophos	V	For complete recovery, use variation from PAM I 302 E5/E6
methidathion	C	
methiocarb	C	N or S detector required for GLC determination; GLC not the method of choice.
methiocarb sulfone	S	Some reports of no recovery; N or S detector required.
methiocarb sulfoxide	P (60-80%)	GLC not preferred, requires N or S detector, wide bore or DEGS column.
methoprotryne	C	Wide bore column recommended.
methoxychlor olefin	C	

Table 302-a: Recovery Through 302 (E1-E3 + DG1-DG19)

Chemical	Recovery[1]	Notes[2]
methoxychlor, p, p'-	C	
methyl 4-chloro-1H-indole-3-acetate	R	
metobromuron	C	
metolachlor	C	
metolcarb	C	N detector, wide bore, DEGS, or low temperature OV-17 column required.
metoxuron	V (73-110%)	Requires low temperature column.
metribuzin	V	N or S detector required.
metribuzin, deaminated diketo metabolite	NR	
metribuzin, deaminated metabolite	C	N or S detector required.
metribuzin, diketo metabolite	NR	
mevinphos, (E)-	C	Wide bore or DEGS column required for separation from (Z)-.
mevinphos, (Z)-	C	Wide bore or DEGS column required for separation from (E)-.
MGK 264	C	
mirex	P (71-83%)	
molinate	C	Recovery tested at 0.053 and 0.264 ppm.
monocrotophos	C	Response enhanced by co-extractives. Wide bore or DEGS column required.
monolinuron	C	
myclobutanil	C	Wide bore column recommended.
myclobutanil alcohol metabolite	S (30-55%)	Poor N/P detector sensitivity.
myclobutanil dihydroxy metabolite	NR	
N, N-diallyl dichloroacetamide	C	
naled	C	May break down to dichlorvos on GLC column. Wide bore or DEGS column required.
napropamide	C	N detector, wide bore or DEGS column required.
neburon	C	
nitralin	C	N or S detector required.
nitrapyrin	C	
nitrofen	C	
nitrofluorfen	C	
nitrothal-isopropyl	C	N detector required.
nonachlor, cis-	C	

Table 302-a: Recovery Through 302 (E1-E3 + DG1-DG19)

Chemical	Recovery[1]	Notes[2]
nonachlor, trans-	C	
norea	C	N detector required.
norflurazon	V (60-200%)	
nuarimol	C	
octachlor epoxide	C	
octhilinone	C	N or S detector required.
ofurace	C	
omethoate	C	Wide bore or DEGS column required.
ovex	C	
oxadiazon	C	
oxadixyl	C	N detector required.
oxamyl oxime metabolite	C	Lower temperature column needed to separate from coextractives.
oxycarboxin	R	Matrix enhancement of response causes high results.
oxydemeton-methyl	C	Wide bore or DEGS column required.
oxydemeton-methyl sulfone	C	Wide bore or DEGS column required; poor GLC makes quantitation questionable.
oxyfluorfen	C	Poor N/P detector sensitivity.
oxythioquinox	C	N or S detector required; wide bore or short DEGS column recommended.
paclobutrazol	C	Wide bore column recommended.
parathion	C	
parathion oxygen analog	C	
parathion-methyl	C	
PB-9	V (106-215%)	
pebulate	C	
penconazole	C	Wide bore column recommended.
pendimethalin	C	N detector required.
pentachloroaniline	C	
pentachlorobenzene	C	
pentachlorobenzonitrile	C	
pentachlorophenyl methyl ether	C	
pentachlorophenyl methyl sulfide	C	
permethrin, cis-	C	High temperature column recommended.

Table 302-a: Recovery Through 302 (E1-E3 + DG1-DG19)

Chemical	Recovery[1]	Notes[2]
permethrin, trans-	C	High temperature column recommended.
Perthane	C	
phenthoate	C	
phenylphenol, o-	C	FID required.
phorate	C	
phorate oxygen analog	C	
phorate oxygen analog sulfone	C	
phorate oxygen analog sulfoxide	C	GLC retention times and responses variable.
phorate sulfone	C	GLC variable.
phorate sulfoxide	C	GLC retention times and responses variable.
phosalone	C	
phosalone oxygen analog	C	Poor GLC detector sensitivity.
phosfolan	C	
phosmet	C	
phosphamidon	C	
phoxim	C	Low temperature column required; degrades at 200°.
phoxim oxygen analog	C	
piperophos	C	
pirimicarb	C	N detector required.
pirimiphos-ethyl	C	
pirimiphos-ethyl oxygen analog	C	
pirimiphos-methyl	C	
pretilachlor	C	
probenazole	C	N or S detector required; FPD-S more sensitive than N/P.
prochloraz	C	High temperature column required.
procyazine	C	
procymidone	C	
prodiamine	C	Recoveries of 0.5 and 1 ppm from apples: 110, 125%, respectively.
profenofos	C	
profluralin	V (40-90%)	
Prolan	P (58%)	

Table 302-a: Recovery Through 302 (E1-E3 + DG1-DG19)

Chemical	Recovery[1]	Notes[2]
promecarb	V	N detector required; GLC not determinative step of choice.
prometryn	C	N or S detector required.
pronamide	C	
propachlor	C	
propanil	C	
propargite	C	S detector required.
propazine	C	
propetamphos	C	
propham	C	N detector required; low temperature column recommended.
propiconazole	C	Wide bore column recommended.
propoxur	C	N detector required for GLC.
prothiofos	C	
prothoate	C	
PYPAC	V (144-162%)	Low temperature column, N detector required.
pyracarbolid	C	N detector required.
pyrazon	C	Wide bore column recommended.
pyrazophos	C	
pyridaphenthion	C	S detector is less sensitive than FPD or N/P.
pyrimethanil	C	
pyriproxyfen	C	N detector required.
quinalphos	C	
quintozene	C	
quizalofop ethyl ester	C	Wide bore column recommended.
RH-6467	S (0-17%)	
RH-9129	V (68-92%)	
RH-9130	P (48-71%)	
ronnel	C	
ronnel oxygen analog	C	
RPA202248	NR	
schradan	C	
SDS-67131	C	
simazine	C	

Table 302-a: Recovery Through 302 (E1-E3 + DG1-DG19)

Chemical	Recovery[1]	Notes[2]
simetryn	C	N or S detector required.
sulfallate	C	
sulfanilamide	NR	
sulfotep	C	Wide bore or DEGS column recommended.
Sulphenone	C	
sulprofos	C	
sulprofos oxygen analog sulfone	C	
sulprofos sulfone	C	
sulprofos sulfoxide	C	
TCMTB	C	
TDE, p,p'-	C	
TDE, p,p'-, olefin	C	
tebuconazole	C	
tecnazene	C	
tefluthrin	C	Recovery tested at 0.275 and 1.374 ppm.
TEPP	C	
terbacil	C	
terbufos	C	
terbufos oxygen analog	C	
terbufos oxygen analog sulfone	C	
terbufos sulfone	C	
terbumeton	C	Recoveries of 0.5 and 1 ppm from apples: about 120%.
terbuthylazine	C	
terbutryn	C	N or S detector required.
tetradifon	C	
tetramethrin	C	
tetrasul	C	
thiabendazole	C	N or S detector required for GC determination.
thiazopyr	C	Recovery at 0.5 ppm; interferences prevented measurement at 0.1 ppm.
thiobencarb	C	
thiometon	C	Degrades while standing in extract.
thionazin	C	

Table 302-a: Recovery Through 302 (E1-E3 + DG1-DG19)

Chemical	Recovery[1]	Notes[2]
THPI	C	N detector, wide bore or DEGS column required.
tolylfluanid	C	
toxaphene	C	
tralkoxydim	V (38-106%)	Recoveries of two OV-101 peaks are different from one another.
tralomethrin	C	
tri-allate	C	
triadimefon	C	Wide bore column recommended.
triadimenol	C	Wide bore column recommended.
triazamate	C	
triazophos	C	
tribufos	C	
trichlorfon	C	Often converts to dichlorvos on GLC column. Wide bore or DEGS column required.
tricyclazole	C	N or S detector required; wide bore column recommended.
tridiphane	C	
trietazine	C	Recovery tested at 0.11 and 0.55 ppm.
triflumizole	C	Wide bore column recommended.
trifluralin	C	
triflusulfuron methyl ester	V (67-106%)	
triphenyl phosphate	C	
tris(2-ethylhexyl) phosphate	C (68-112%)	mean recovery 97.6%, n=11
tris(beta-chloroethyl) phosphate	C	
tris(chloropropyl) phosphate	C	
Tycor	C	May break down in solution. Temp program separated from interference in tomato.
vamidothion sulfone	C	
vinclozolin	C	
vinclozolin metabolite B	C	Severely subject to influence of matrix; levels <1.0 ppm had very high recovery.
vinclozolin metabolite E	C	Severely subject to influence of matrix; levels <1.0 ppm had very high recovery.
vinclozolin metabolite F	R	Poor chromatography, influence of matrix prevent quantitation of recovery.
vinclozolin metabolite S	V (59-137%)	

Table 302-b: Recovery of Chemicals Through Method 302 (E1-E3 + C5 + DG1-DG19)
(acetone extraction, partitioning or Hydromatrix removal of water, Florisil column cleanup, GLC determination with various columns and detectors)

Chemical	Recovery[1,2]	Eluant, C5[3]	Notes[4]
2,3-dihydro-3,3-methyl-2-oxo= -5-benzofuranyl methyl sulfonate	NR	15	No elution from Florisil only in 15% EE/PE; 50% not tested.
2,6-dichlorobenzamide	NR	50	83% elution from Florisil only in 200 mL ethyl ether.
2-hydroxy-2,3-dihydro-3,3-methyl= -5-benzofuranyl methyl sulfonate	NR	15	No elution from Florisil only in 15% EE/PE; 50% not tested.
2-methoxy-3,5,6-trichloropyridine	P (65%)	15	Low temperature column recommended.
acetochlor	C	15+50	80% eluted in 15% EE/PE.
alachlor	P (68%)	15	
aldrin	C	15	
allethrin	C (80%)	15	
atrazine	C	15+50	Eluted in 50% EE/PE.
azinphos-ethyl	C	15	
BHC, alpha-	C	15	
BHC, beta-	C	15	
BHC, delta-	C	15	
bifenthrin	C	15	
binapacryl	C (83%)	15	
bioresmethrin	NR	15	Some elution from Florisil in 200 mL 50% EE/PE, more in 200 mL 75% EE/PE.
biphenyl	C	15	FID required.
bromophos	C	15	
bromopropylate	C (80%)	50	
bupirimate	S (10-30%)	15	
captafol	NR	15	Some elution from Florisil in 50% EE/PE after 15%.

[1] Codes: C: complete (>80%); P: partial (50-80%); S: small (<50%); V: variable (approximate percentage when known); R: recovered but no quantitative information available; NR: not recovered.

[2] Recovery results refer to complete method; blank entry in this column indicates Florisil elution was tested but not complete method.

[3] Eluants(s) in which chemical is eluted from Florisil, according to directions in 302 C5, *i.e.*, 15 and 50% ethyl ether/petroleum ether (EE/PE). Entries for chemicals not recovered indicate which eluants were used in tests.

[4] "Florisil only" refers to tests in which elution patterns were tested by added reference standard solutions directly to Florisil column.

Table 302-b: Recovery Through 302 (E1-E3 + C5 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C5[3]	Notes[4]
captan	S (25%)	15	Additional elution from Florisil in 50% EE/PE after 15%.
carfentrazone ethyl ester	C	15	Some additional elution in 50% EE/PE possible.
chlorbenside	C	15	
chlordane	C	15	
chlordane, cis-	C	15	
chlordane, trans-	C	15	
chlordimeform hydrochloride	NR	15-50	
chlorfenapyr	C	15+50	
chlorflurecol methyl ester	NR	15	No elution from Florisil only in 15% EE/PE; 50% not tested.
chlornitrofen	C	15	
chlorothalonil	C (81%)	15	Additional elution from Florisil in 200 mL 50% EE/PE.
chlorpyrifos	C	15	
chlorpyrifos oxygen analog	NR	15	Elution from Florisil with 50% EE/PE not tested.
chlorpyrifos-methyl	C	15	
chlorthiophos oxygen analog	NR	15	No elution from Florisil only in 15% EE/PE; 50% not tested.
chlorthiophos sulfone	S (8%)	15	
chlorthiophos sulfoxide	NR	15	No elution from Florisil only in 15% EE/PE; 50% not tested.
clofentezine	S (27-50%)	15	Complete elution from Florisil only; degrades on GLC in presence of extract.
cymiazole	NR	6-15-50	No elution from Florisil only in 6, 15, or 50% EE/PE or 100% EE.
cypermethrin	C	15	
DCPA	C	15	
DDE, o,p'-	C	15	
DDE, p,p'-	C	15	
DDT, o,p'-	C	15	
DDT, p,p'-	C	15	
deltamethrin	C	15	Very poor EC detector sensitivity.
diazinon	C	15	
dichlofluanid	C	15+50	60% eluted in 15% EE/PE

Table 302-b: Recovery Through 302 (E1-E3 + C5 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C5[3]	Notes[4]
diclobutrazol	P	15	56-70% elution from Florisil only; 50% not tested; wide bore column recommended.
dicloran	C	15	
dicofol, o,p'-	C	15	
dicofol, p,p'-	C	15	May be variable.
dieldrin	C	15	
difenoxuron	NR	15	No elution from Florisil only in 15% EE/PE; 50% not tested.
dimethachlor	NR	15	No elution from Florisil only in 15% EE/PE; 50% not tested.
dinobuton	C (83%)	15	
endosulfan I	C	15	
endosulfan II	C	15	
endosulfan sulfate	C	15	Additional elution possible in 50% EE/PE.
endrin	C	15	
esfenvalerate	C	15	
etaconazole	S	15	30% elution from Florisil only; 50% not tested; wide bore column recommended.
fenfuram	P (45%)	15	Partial recovery also when Florisil eluted with MeCl2 eluant #3.
fenpropathrin	C	15	
fenson	C	15	
fensulfothion sulfone	NR	50	No elution from Florisil only in 50% EE/PE.
fenvalerate	C	15	
flamprop-M-isopropyl	NR	15	Complete elution from Florisil only in 50% EE/PE plus additional EE.
flamprop-methyl	NR	6-15-50	No elution from Florisil only in 6, 15, or 50% EE/PE; complete elution with EE.
fluchloralin	C	15	
flucythrinate	C	15+50	About 80% eluted in 15% EE/PE
flusilazole	S	15	35-44% elution from Florisil only; wide bore column recommended.
fluvalinate	C	15	
FOE 5043 thioglycolate sulfoxide	NR		not eluted from Florisil
folpet	C	15+50	78% eluted in 15% EE/PE
fonofos	C	15	

Table 302-b: Recovery Through 302 (E1-E3 + C5 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C5[3]	Notes[4]
fuberidazole	NR	15	No elution from Florisil only in 15% EE/PE; 50% not tested.
heptachlor	C	15	Elution from Florisil not always complete.
heptachlor epoxide	C	15	
hexachlorobenzene	C	15	Recovery test run with 6,15,50% EE/PE, elution in 6%.
hexaconazole	NR	15	
imazalil	NR	15	No elution from Florisil only in 15% EE/PE; 50% not tested.
iprodione	S (24-97%)	15	Complete elution from Florisil requires more polar eluants.
isocarbamid	NR	15	No elution from Florisil only in 15% EE/PE; 50% not tested.
lindane	C	15	
metalaxyl	NR	15	
methabenzthiazuron	NR	15	No elution from Florisil only in 15% EE/PE; 50% not tested.
methoprotryne	NR	15	No elution from Florisil only in 15% EE/PE; 50% not tested.
methyl 3,5-dichlorobenzoate	C	15	
mirex	C	15	
myclobutanil	NR	15	No elution from Florisil only in 15% EE/PE; 50% not tested.
nitrapyrin	C	15	
nitrofen	C	15	
nitrothal-isopropyl	C	15	
nonachlor, trans-	C	15	
norflurazon	NR	15	Elution from Florisil with 50% EE/PE not tested.
ovex	C	15	
oxadiazon	C	15	
oxythioquinox	C (79-96%)	15	
paclobutrazol	P	15	44-55% elution from Florisil only; 50% not tested; wide bore column recommended.
parathion	C	15	
parathion-methyl	C	15	

Table 302-b: Recovery Through 302 (E1-E3 + C5 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C5[3]	Notes[4]
penconazole	S	15	42-54% elution from Florisil only; 50% not tested; wide bore column recommended.
pentachloroaniline	C	15	Recovery test run with 6,15,50% EE/PE, elution in 6%.
pentachlorobenzene	C	15	Recovery test run with 6,15,50% EE/PE, elution in 6%.
pentachlorophenyl methyl sulfide	C	15	Recovery test run with 6,15,50% EE/PE, elution in 6%.
permethrin, cis-	C	15	High temperature column recommended.
permethrin, trans-	C	15	High temperature column recommended.
phenmedipham	NR	15	No elution from Florisil only in 15% EE/PE; 50% not tested.
phenothrin	P (60%)	15	No additional elution in 200 mL 50% EE/PE; wide bore column recommended.
phenylphenol, o-	C	15	FID required.
phorate sulfoxide	NR	15-50	Recovery test included elution from Florisil with 50% EE/PE only.
pirimicarb	S (25%)	15	
pretilachlor		15	Elution from Florisil only complete in 15% EE/PE.
procymidone	C	15	
prodiamine		15	Elution from Florisil only complete in 15% EE/PE.
propargite	C	15	
propham	C	15	N detector required; low temperature column recommended.
propiconazole	P	15	46-50% elution from Florisil only; 50% not tested; wide bore column recommended.
pyrethrins	C	15+50	Most eluted in 15% EE/PE.
quintozene	C	15	
quizalofop ethyl ester	C	15	
simazine	P (69%)	15	
TDE, p,p'-	C	15	
tebufenozide	NR	15	
TEPP	NR	15	
terbumeton	NR	15-50	No elution from Florisil only in 15 or 50% EE/PE.
terbuthylazine	C	15	

Table 302-b: Recovery Through 302 (E1-E3 + C5 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C5[3]	Notes[4]
tetradifon	C	15	
thiometon	C	15	
THPI	NR	15-50	Only small amount recovered (5%) in subsequent elution with 200 mL EE .
toxaphene	C	15	
tralomethrin	C	15	
triadimefon	S (7%)	15	72-84% elution from Florisil only; 50% not tested; wide bore column recommended.
triadimenol	S	15	40-45% elution from Florisil only; 50% not tested; wide bore column recommended.
tricyclazole	NR	15	No elution from Florisil only in 15% EE/PE; 50% not tested.
triflumizole	P	15	46-52% elution from Florisil only; 50% not tested; wide bore column recommended.
vinclozolin	C	15	

Table 302-c: Recovery of Chemicals Through Method 302 (E1-E3 + C3 + DL1)

(acetone extraction, partitioning or Hydromatrix removal of water, charcoal/silanized Celite column cleanup, HPLC with post-column hydrolysis and derivatization, fluorescence detection)

Chemical	Recovery[1]	Rrt[2]	ng[3]	Notes
2,3,5-trimethacarb	C			
3,4,5-trimethacarb	C			
3-hydroxycarbofuran	C	0.6	10	mean recovery 97.6%, n=45
aldicarb	C	0.83	14	mean recovery 89.2%, n=210
aldicarb sulfoxide	C	0.33	9	mean recovery 98.6%, n=108
aldoxycarb	C	0.4	9	mean recovery 102%, n=111
aminocarb	C			
bufencarb	C	1.44	19	Major peak is listed. mean recovery 97.4%, n=27
butocarboxim	S (0-108%)	0.75	15	mean recovery 56.1%, n=22
carbaryl	C	1.06	7	mean recovery 98.1%, n=147
carbofuran	C	1	10	mean recovery 97.4%, n=121
dioxacarb	P (72%)	0.67	15	
isoprocarb	C	1.13	8	
methiocarb	C	1.26	10	mean recovery 99.9%, n=67
methomyl	C	0.46	10	mean recovery 94.1%, n=128
metolcarb	C	0.85	10	mean recovery 90.7%, n=12
oxamyl	C	0.44	10	mean recovery 94.3%, n=41
promecarb	C	1.31	10	mean recovery 99.9%, n=29
propoxur	C	0.98	8	mean recovery 92.2%, n=48
XMC	C	1.06	10	mean recovery 95.6%, n=28

[1] Codes: C: complete (>80%); P: partial (50-80%); S: small (<50%); V: variable (approximate percentage when known); R: recovered but no quantitative information available; NR: not recovered.

[2] Retention time, relative to carbofuran, on C-8 column described in DL1.

[3] ng that cause 50% full scale deflection detector response in DL1.

Table 302-d: Recovery of Chemicals Through Method 302 (E2/E3 + C1 + DG1-DG19)
(acetone extraction, Hydromatrix removal of water, Florisil cleanup with one methylene chloride eluant, GLC determination with various columns and detectors)

Chemical	Recovery[1]	Notes[2]
1,2,3,5-tetrachlorobenzene	V (63-141%)	mean recovery 91.6%, n=16
2,3,5,6-tetrachloroaniline	C (67-110%%)	mean recovery 85.5%, n=10
2,4-dichloro-6-nitrobenzenamine	V (65-123%)	mean 91%, n=7
aldrin	C	mean recovery 86.9%, n=16
allethrin	C	mean recovery 91.8%, n=4
alpha-cypermethrin	C	mean recovery 98.0%, n=15
azafenidin	V (45-160%)	High Temperature column required.
BHC, alpha-	V (68-89.5%)	mean recovery 79.0%, n=2
bifenthrin	C (59-110%)	mean recovery 91.4%, n=15
bromopropylate	NR	
butachlor	C	mean recovery 91.7%, n=2
captafol	C	mean recovery 101.7%, n=4
captan	V (0-139%)	mean recovery 65.2%, n=20
carbaryl	C	recovery 107%, n=1
chlordane	P (64%)	mean recovery 64.4%, n=2
chlordane, cis-	C	
chlordane, trans-	C	
chlorobenzilate	NR	mean recovery 5.5%, n=11
chlorothalonil	S (0-93%)	mean recovery 36.9%, n=17
chlorpropham	V (76-95%)	mean recovery 86.1%, n=2
chlorpyrifos	C	mean recovery 88.9%, n=27
chlorpyrifos-methyl	V (54-116%)	mean recovery 80.6%, n=16
clodinafop-propargyl	V (56-104%)	
cloquintocet-mexyl	NR	Not eluted from Florisil only.
cyfluthrin	V (60-117%)	mean recovery 86.5%, n=15
cypermethrin	C	
DCPA	P	mean recovery 77.0%, n=4
DDE, o,p'-	C	

[1] Codes: C: complete (>80%); P: partial (50-80%); S: small (<50%); V: variable (approximate percentage when known); R: recovered but no quantitative information available; NR: not recovered.

[2] Notes assume that extract is examined by GLC with columns at 200° C and, at a minimum, halogen-selective detector (DG3 or 16) and phosphorus-selective detectors (DG2 or 14 or 19). Notes indicate those chemicals that can be determined only by use of columns, temperatures, and/or detectors other than the minimal ones.

Table 302-d: Recovery Through 302 (E2/E3 + C1 + DG1-DG19)

Chemical	Recovery[1]	Notes[2]
DDE, p,p'-	C	mean recovery 83.7%, n=6
DDT, o,p'-	V (58-111%)	mean recovery 86.3%, n=15
DDT, p,p'-	C	mean recovery 91.4%, n=2
deltamethrin	C	mean recovery 88.5%, n=5
diazinon	C	
dichlone	P	recovery 58.6%, n=1
diclofop-methyl	V (56-135%)	mean recovery 88.2%, n=29
dicloran	V (57-118%)	mean recovery 82.1%, n=14
dicofol, o,p'-	C	
dicofol, p,p'-	C	mean recovery 107%, n=4
dieldrin	C	mean recovery 88.4%, n=146
endosulfan I	V (64-89%)	mean recovery 76.2%, n=4
endosulfan II	C	mean recovery 93.6%, n=4
endosulfan sulfate	C	mean recovery 91.9%, n=28
endrin	C	mean recovery 99.1%, n=2
esfenvalerate	V (70-138%)	mean recovery 95.9%, n=16
fenarimol	S (0-33%)	mean recovery 13%, n=21
fenhexamid	NR	Not recovered from Florisil only.
fenoxaprop ethyl ester	C	mean recovery 97.8%, n=2
fenvalerate	V (65-162%)	mean recovery 93.8%, n=21
fluchloralin	C	mean recovery 91.8%, n=16
fluvalinate	C (64-113%)	mean recovery 93.4%, n=10
folpet	C	mean recovery 120%, n=4
haloxyfop methyl ester	C	recovery 126%, n=1
heptachlor	C	mean recovery 81.7%, n=2
heptachlor epoxide	V (58-118%)	mean recovery 87.3%, n=49
hexachlorobenzene	C	
hexythiazox	V (36-89%)	
iprodione	S (0-95%)	mean recovery 32.4%, n=26. Complete recovery requires 50% EE/PE eluant.
iprodione metabolite isomer	V (32-149%)	mean recovery 85.0%, n=24
isopropalin	C	mean recovery 85.8%, n=4
lambda-cyhalothrin	C	mean recovery 106%, n=16, range 87-133%
lindane	C	mean recovery 84.7%, n=54

Table 302-d: Recovery Through 302 (E2/E3 + C1 + DG1-DG19)

Chemical	Recovery[1]	Notes[2]
linuron	C	mean recovery 88.3%, n=18
methamidophos	C	mean recovery 91.2%, n=1
methoxychlor olefin	C (63-104%)	mean recovery 88.2%, n=10
methoxychlor, o, p'-	C	72-123% recoveries, TDS
methoxychlor, p, p'-	V (76-130%)	mean recovery 100.9%, n=19
metolachlor	NR	
mirex	V (37-110%)	mean recovery 79.3%, n=15
nitrapyrin	V (69-123%)	mean recovery 96.1%, n=2
nonachlor, cis-	C	
nonachlor, trans-	C	
nuarimol	NR	
octachlor epoxide	C	mean recovery 91.5%, n=23
oxadiazon	C	mean recovery 89.4%, n=2
oxyfluorfen	C	
parathion	C	mean recovery 117%, n=1
pentachloroaniline	C	
pentachlorobenzene	C	
pentachlorophenyl methyl ether	C	mean recovery 89.5%, n=1
pentachlorophenyl methyl sulfide	V (49-112%)	mean recovery 80.7%, n=15
permethrin, cis-	C	mean recovery 91.6%, n=4
permethrin, trans-	C	mean recovery 93.5%, n=4
phenylphenol, o-	V (76-129%)	FID required; mean recovery 97.6%, n=16
phosalone	V (27-116%)	mean recovery 75.4%, n=16
procymidone	C	mean recovery 90.7%, n=3
propanil	C	mean recovery 100.2%, n=2
propargite	V (71-125%)	mean recovery 93%, n=23
prothiofos	V (36-127%)	mean recovery 75.8%, n=21
pyrethrins	C	mean recovery 83.5%, n=6
quintozene	P	mean recovery 79.6%, n=14
sulfallate	V (39-87%)	mean recovery 58.9%, n=5
TDE, o,p'-	V (70-145%)	mean recovery 97.7%, n=17
TDE, p,p'-	C	
TDE, p,p'-, olefin	V (41-128%)	mean recovery 78.4%, n=21
tecnazene	C	mean recovery 83.2%, n=1

Table 302-d: Recovery Through 302 (E2/E3 + C1 + DG1-DG19)

Chemical	**Recovery[1]**	**Notes[2]**
tetradifon	C	mean recovery 111%, n=4
thiobencarb	C	mean recovery 90.4%, n=2
toxaphene	C	mean recovery 94.1%, n=5
tralomethrin	C (67-103%)	mean recovery 87.5%, n=11
tridiphane	V (54-110%)	mean recovery 85.1%, n=16
trifluralin	P	mean recovery 57.0%, n=3
vinclozolin	V (61-109%)	mean recovery 86.8%, n=14

Table 302-e: Recovery of Chemicals Through Method 302 (E1/E4 + C4 + DL1)
(acetone extraction, partitioning to remove water, C-18 cartridge cleanup, HPLC with post-column derivatization and fluorescence detection)

Chemical	Recovery[1]	Rrt[2]	ng[3]	Notes
3-hydroxycarbofuran	C	0.6	10	mean recovery 94.7%, n=2
aldicarb	C	0.83	14	mean recovery 87.4%, n=8
aldicarb sulfoxide	C	0.33	9	mean recovery 89.6%, n=9
aldoxycarb	V (70-104%)	0.4	9	mean recovery 88.7%, n=8
bitertanol	C			GLC with high temperature column, N/P detector required.
bufencarb	C	1.44	19	Major peak is listed. recovery 107%, n=1
carbaryl	C	1.06	7	mean recovery 88.9%, n=45
carbofuran	C	1	10	mean recovery 96.2%, n=3
dioxacarb	C	0.67	15	recovery 91.1%, n=1
methiocarb	C	1.26	10	mean recovery 97.9%, n=5
methiocarb sulfoxide	S	0.64	12	recovery 42.0%, n=1
methomyl	C	0.46	10	mean recovery 96.4%, n=36
oxamyl	C	0.44	10	mean recovery 95.8%, n=33
phenylphenol, o-	C			DL2 required; mean recovery 86.9%, n=8.
piperonyl butoxide	C			mean recovery 91.8%, n=5
pronamide	C			
propoxur	C	0.98	8	mean recovery 85.2%, n=6

[1] Codes: C: complete (>80%); P: partial (50-80%); S: small (<50%); V: variable (approximate percentage when known); R: recovered but no quantitative information available; NR: not recovered.

[2] Retention time, relative to carbofuran, on C-8 column described in DL1.

[3] ng that cause 50% full scale deflection detector response in DL1.

Table 302-f: Recovery of Chemicals Through Method 302 (E7 + C6 + DG1-DG3, DG6-DG7, DG10, DG13-DG14, or DG16)
(acetone extraction and solid phase extraction cartridges, liquid-liquid partitioning, SAX cartridge cleanup, GLC determination)

Chemical	Recovery[1]	Notes
3-hydroxycarbofuran	C	Determination by DL1.
acephate	C	
aldicarb	C	Determination by DL1.
aldicarb sulfoxide	C	Determination by DL1.
alpha-cypermethrin	C	
atrazine	C	
azinphos-methyl	C	Recoveries tend to be >100%.
BHC, alpha-	C	
BHC, beta-	C	Recoveries tend to be >100%.
BHC, delta-	C	
bitertanol	C	GLC with high temperature column, N/P detector required.
carbaryl	C	Determination by DL1.
carbendazim	C	Determined by UV detector at 280 nm.
carbofuran	C	Determination by DL1.
carbophenothion	C	
chlorothalonil	C	based on two recoveries
chloroxuron	C	Determination by DL3.
chlorpropham	C	
chlorpyrifos	C	
chlorpyrifos-methyl	C	
chlorthiophos	C	
cyanazine	C	
cyfluthrin	C	
DCPA	C	
DDE, o,p'-	C	
DDE, p,p'-	C	
DDT, o,p'-	C	
DDT, p,p'-	C	

[1] Codes: C: complete (>80%); P: partial (50-80%); S: small (<50%); V: variable (approximate percentage when known); R: recovered but no quantitative information available; NR: not recovered.

Table 302-f: Recovery Through 302 (E7 + C6 + DG1-DG3, DG6-DG7, DG10, DG13-DG14, or DG16)

Chemical	Recovery[1]	Notes
deltamethrin	C	
demeton-O	C	
demeton-S sulfone	C	Recoveries tend to be >100%.
diazinon	C	
dichlofluanid	C	Recoveries tend to be >100%.
dichlorvos	P (75%)	
dicloran	C	
dicofol, p,p'-	C	
dicrotophos	C	
dieldrin	C	
dimethoate	C	
dioxathion	C	
diphenylamine	C	N detector required.
disulfoton sulfone	C	
diuron	C	Determination by DL3.
endosulfan I	C	
endosulfan II	C	
endosulfan sulfate	C	
endrin	C	
EPN	C	
esfenvalerate	C	
etaconazole	C	
ethion	C	
fenamiphos	P (79%)	
fenarimol	V (79, 99%)	
fenpropimorph	C	N detector required
fenthion	C	
fenuron	C	Determination by DL3.
fluridone	P (65%)	
folpet	C	
hexachlorobenzene	P (76%)	
imazalil	C	
iprodione	C	
lindane	C	

Table 302-f: Recovery Through 302 (E7 + C6 + DG1-DG3, DG6-DG7, DG10, DG13-DG14, or DG16)

Chemical	Recovery[1]	Notes
linuron	C	Determination by DL3.
malathion	C	
methamidophos	C	
methidathion	C	
methiocarb	C	Determination by DL1.
methomyl	C	Determination by DL1.
methoxychlor, o, p'-	C	
methoxychlor, p, p'-	C	
metobromuron	C	Determination by DL3.
metoxuron	C	Determination by DL3.
mevinphos, (E)-	C	
mevinphos, (Z)-	C	
monocrotophos	C	
monolinuron	C	Determination by DL3.
monuron	C	Determination by DL3.
myclobutanil	C	
neburon	C	Determination by DL3.
omethoate	C	Wide bore or DEGS column required.
oryzalin	C	N or S detector required.
oxamyl	C	Determination by DL1.
parathion	C	
parathion oxygen analog	C	
parathion-methyl	C	
parathion-methyl oxygen analog		C
penconazole	C	
pentachloroaniline	C	
permethrin, cis-	C	
permethrin, trans-	C	
phenylphenol, o-	C	
phorate sulfone	C	
phosalone	C	
phosmet	C	
pirimiphos-ethyl	C	
pirimiphos-methyl	C	

Table 302-f: Recovery Through 302 (E7 + C6 + DG1-DG3, DG6-DG7, DG10, DG13-DG14, or DG16)

Chemical	Recovery[1]	Notes
procymidone	C	
propanil	C	
propiconazole	C	
prothiofos	C	
pyrazophos	C	
pyridaphenthion	C	
quintozene	C	
ronnel	C (122%)	n=1
simazine	C	
sulfotep	C	
TDE, p,p'-	C	
tetradifon	C	
thiabendazole	C	Determined by UV detector at 280 nm; confirm, increase sensitivity with DL7.
THPI	C	
triadimefon	C	
triadimenol	C	
triazophos	C	
vinclozolin	C	

Table 303-a: Recovery of Chemicals Through Method 303 (E1-E5 + C1 or C2 + DG1-DG19)

(acetonitrile or water/acetonitrile extraction, partitioning into petroleum ether, Florisil column cleanup, GLC determination with various columns and detectors)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
1,1'-(2,2-dichloroethylidene)= bis(2-methoxybenzene)	R			
1,2,3,5-tetrachlorobenzene	P (75%) C	6	 1	
1,2,3-trichlorobenzene	C	6	1	Elutes in PE forerun.
1,2,4,5-tetrachloro-3-(methyl= thio)benzene	C	6	1	
1,2,4-triazole	NR	6-15-50	1-2-3	
1-hydroxychlordene	R	15		
10,10-dihydromirex	C	6		
10-monohydromirex	C	6		
2,3,5,6-tetrachloroaniline	R			
2,3,5,6-tetrachloroanisidine	C	6	2	
2,3,5,6-tetrachloroanisole	C	6	1	
2,3,5,6-tetrachloronitroanisole	C	6	1+2	
2,3,5-trimethacarb	S (18%) NR	50	 1-2-3	
2,4,5-trichloro-alpha-methyl= benzenemethanol	R	15		

[1] Codes: C: complete (>80%); P: partial (50-80%); S: small (<50%); V: variable (approximate percentage when known); R: recovered but no quantitative information available; NR: not recovered.

[2] Recovery results refer to complete method; blank entry in this column indicates Florisil elution was tested but not complete method. Separate results are listed for C1 and C2 only if recovery is affected by Florisil elution system.

[3] Eluants(s) in which chemical is eluted from Florisil, according to directions in 303 C1, *i.e.*, 6, 15, and 50% ethyl ether/ petroleum ether (EE/PE). Entries for chemicals not recovered indicate which eluants were used in tests.

[4] Eluants(s) in which chemical is eluted from Florisil, according to directions in 303 C2, *i.e.*, methylene chloride (CH_2Cl_2) eluants #1, 2, and 3. Entries for chemicals not recovered indicate which eluants were used in tests.

[5] "Florisil only" refers to tests in which elution patterns were tested by added reference standard solutions directly to Florisil column.

[6] Reference to petroleum ether (PE) forerun refers to Florisil elution performed as in 304 C3 or C4; not usually used in analysis of nonfatty foods.

Table 303-a: Recovery Through 303 (E1-E5 + C1 or C2 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
2,4-dichloro-6-nitrobenzenamine	R	15	2	Complete elution from Florisil only in 15% EE/PE or CH_2Cl_2 eluant #2.
2,6-dichlorobenzamide	NR	6-15-50	1-2-3	
2,8-dihydromirex	C	6		
2-chloroethyl caprate	C	15	2	
2-chloroethyl laurate	C	15	2	
2-chloroethyl linoleate	V (36-114%)	15	2	
2-chloroethyl myristate	V (48-112%)	15	2	
2-chloroethyl palmitate	V (38-107%)	15	2	
2-methoxy-3,5,6-trichloropyridine	P(60-78%) C	6+15	 1+2	
3,4,5-trimethacarb		50		Partial (20-35%) elution from Florisil only in 50% EE/PE.
	NR		1-2-3	
3,4-dichloroaniline	S (8%)	15		35% elution from Florisil only in 15% EE/PE.
3,4-dichlorophenylurea	NR	6-15-50		
3,5-dichloroaniline	S (12-48%)	6+15	1+2	Partial (73%) elution from Florisil only in 15% EE/PE.
3-(3,4-dichlorophenyl)-1-methoxyurea	NR	6-15-50		
3-desmethyl sulfentrazone	NR	6-15-50	1-2-3	
3-hydroxymethyl-2,5-dimethyl=phenyl methylcarbamate	NR	6-15-50	1-2-3	
3-ketocarbofuran	NR	6	1	60% recovered from Florisil only in 6% EE/PE or CH_2Cl_2 #1; also elutes with PE.
3-methyl-4-nitrophenol	NR	6-15-50	1-2-3	
3-tert-butyl-5-chloro-6-hydroxy=methyluracil	NR	6-15-50	1-2-3	

Table 303-a: Recovery Through 303 (E1-E5 + C1 or C2 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
4-(dichloroacetyl)-1-oxa-4-azapiro[4.5]decane	P (50-62%)	50	3	Complete elution from Florisil only in 50% and in CH_2Cl_2 eluant #3.
4-chloro-6-methoxyindole	R	15		
4-chlorobenzylmethyl sulfone	NR	6-15-50	1-2-3	
4-chlorobenzylmethyl sulfoxide	NR	6-15-50	1-2-3	
4-hydroxymethyl-3,5-dimethyl=phenyl methylcarbamate	NR	15-50	1-2-3	<20% elution from Florisil only in 15+50% EE/PE; <10% in CH_2Cl_2 eluants 1,2,3.
6-chloro-2,3-dihydro-3,3,7-methyl-5H-oxazolo(3,2-a)pyrimidin-5-one	NR	6-15-50	1-2-3	
6-chloro-2,3-dihydro-7-hydroxy methyl-3,3-methyl-5H-oxazolo=(3,2-a)pyrimidin-5-one	NR	6-15-50	1-2-3	
6-chloronicotinic acid	NR	6-15-50	1-2-3	
8-monohydromirex	C	6		
acetochlor	C (80-86%) P (55-68%)	50	3	Complete elution from Florisil only in CH_2Cl_2 eluant #3.
acifluorfen	NR	6-15-50	1-2-3	
acrinathrin	V(67-100%) V(66-96%)	15	2	
alachlor	C	50	3	16% elution from Florisil only in CH_2Cl_2 eluant #3.
aldrin	C	6	1	
allethrin	C	50	3	
allidochlor	NR	6-15	1-2-3	
alpha-cypermethrin	C		2	
anilazine	S (4-88%)	15+50	2+3	
aramite	P	15		Poor GLCsensitivity.

Table 303-a: Recovery Through 303 (E1-E5 + C1 or C2 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
Aroclor 1016	C	6	1	Elutes in PE forerun.
Aroclor 1221	C	6	1	Elutes in PE forerun.
Aroclor 1242	C	6	1	Elutes in PE forerun.
Aroclor 1248	C	6	1	Elutes in PE forerun.
Aroclor 1254	C	6	1	Elutes in PE forerun.
Aroclor 1260	C	6	1	Elutes in PE forerun.
Aroclor 1262	C	6	1	Elutes in PE forerun.
Aroclor 1268	C	6		Elutes in PE forerun.
Aroclor 4465	C	6	1	Elutes in PE forerun.
atrazine	S (25%) NR	50	 1-2-3	
azinphos-ethyl	P (50%)	50	3	49-79% elution from Florisil only in CH_2Cl_2 eluant #3.
azinphos-methyl	NR	6-15-50	1-2-3	
benfluralin	C	6	2	
benoxacor	P	15+50	2+3	60-75% elution from Florisil only in EE/PE; 40-80% in CH_2Cl_2 eluants.
bensulide	P (70%)	50	3	14% elution from Florisil only in CH_2Cl_2 eluant #3.
benzoylprop-ethyl	NR	6-15-50	1-2-3	
BHC, alpha-	C	6	1	Partially elutes in PE forerun.
BHC, beta-	C	6	1	
BHC, delta-	C	6+15	1	EE/PE elution variable.
bifenox	C	15+50	2+3	
bifenthrin	C	6+15	2	
binapacryl	P	15		

Table 303-a: Recovery Through 303 (E1-E5 + C1 or C2 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
bis(2-ethylhexyl) phthalate	C	15+50		Poor EC detector sensitivity.
bis(trichloromethyl)disulfide	R	6		
bromacil	NR	6-15-50	1-2-3	
bromophos	C	6		
bromophos-ethyl	C	6		
bromopropylate	C NR	15+50	 1-2-3	
bromoxynil butyrate	V (20-143%)	15+50	2	
bromoxynil octanoate	V (70-127%) S (15-42%)	15+50	 2	
Bulan	P (60%)	15	 2	 Complete elution from Florisil only in CH_2Cl_2 eluant #2.
butachlor	C	50		Also complete (83%) recovery from Florisil only in CH_2Cl_2 eluant 3.
butralin	C	6+15+50		Elution from Florisil variable.
butyl benzyl phthalate	C	15+50		
cadusafos	NR	6-15-50	1-2-3	
captafol	P (75-80%)	50	3	
captan	P (75%) P (50%)	50	 3	
captan epoxide	NR	6-15		
carbophenothion	C	6	 2	Elution from Florisil may be variable. <60% elution from Florisil only in CH_2Cl_2 eluant #2.
carbophenothion oxygen analog	NR	6-15-50	1-2-3	
carbophenothion sulfone	C (80%)	6	1	Elutes in PE forerun.
carboxin	NR	6-15-50		

Table 303-a: Recovery Through 303 (E1-E5 + C1 or C2 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
carboxin sulfoxide	NR	6-15-50	1-2-3	
CGA 118244	NR	6-15-50	1-2-3	
CGA 120844	NR	6-15-50	1-2-3	
CGA 14128		50	1-2-3	9-22% elution from Florisil only in EE/PE; no elution in CH_2Cl_2 eluants.
CGA 171683		15+50	3	Complete elution from Florisil only in 15+50% EE/PE, 70% in CH_2Cl_2 eluant #3.
CGA 205374	NR	6-15-50	1-2-3	
CGA 37734	NR	6-15-50	1-2-3	
CGA 91305	NR	6-15-50	1-2-3	
CGA 94689A	NR	6-15-50	1-2-3	
CGA 94689B	NR	6-15-50	1-2-3	
chlorbenside	S	6	1	Recovery 25-85% using EE/PE eluants; may be better with CH_2Cl_2.
chlorbromuron	V (45-67%)	50	3	Complete elution from Florisil only.
chlorbufam		15	2+3	Complete elution from Florisil only in 15% EE/PE, 77% in CH_2Cl_2 eluants 2+3.
chlordane	C	6	1	
chlordane, cis-	C	6	1	May elute in PE forerun.
chlordane, trans-	C	6	1	
chlordecone	S (45%) NR	15+50	 1-2-3	Elution from Florisil variable.
chlordene	C	6	1	Elutes in PE forerun.
chlordene epoxide	C	15		
chlorethoxyfos	C	6	1	

Table 303-a: Recovery Through 303 (E1-E5 + C1 or C2 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
chlorfenvinphos, beta-	S (0-49%)	50		
	NR		1-2-3	
chlorimuron ethyl ester	NR			Variable (75-92%) elution from Florisil only in 50% EE/PE.
chlornitrofen	C	6+15	2	Variable elution from Florisil in EE/PE.
chlorobenzilate	C	15+50		
	NR		3	Some variable elution from Florisil only in eluant #3.
chloroneb	C	6	2	82% elution from Florisil only in CH_2Cl_2 eluant #2.
chloropropylate	C	15+50		
			3	Some variable elution from Florisil only in CH_2Cl_2 eluant #3.
chlorothalonil	NR	6-15-50		
	C		2+3	
chlorothalonil trichloro impurity	NR	6-15-50		
	R		2+3	
chloroxuron	NR	6-15-50	1-2-3	
chlorpropham	C	15	2	
chlorpyrifos	C	6	2	
chlorpyrifos oxygen analog	NR	6-15-50		
chlorpyrifos-methyl	C	6	2	
chlorsulfuron	NR	6-15-50		
chlorthiophos	C	6		
			2	11% elution from Florisil only in CH_2Cl_2 eluant #2.
chlorthiophos oxygen analog	NR	6-15-50	1-2-3	
chlorthiophos sulfone		50		55% elution from Florisil only in 50% EE/PE.
	C		3	

Table 303-a: Recovery Through 303 (E1-E5 + C1 or C2 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
chlorthiophos sulfoxide	NR	6-15-50	1-2-3	
clofentezine	S (15-24%)	15	2	Complete elution from Florisil only; degrades on GLC in extract.
clomazone		50	3	88% elution from Florisil only in 50% EE/PE, 54-74% in CH_2Cl_2 eluant #3.
clopyralid methyl ester		50		17% elution from Florisil only in 50% EE/PE.
Compound K	C		1	
coumaphos	NR	6-15-50	3	Complete elution from Florisil only in CH_2Cl_2 eluant #3.
coumaphos oxygen analog	NR	6-15-50	1-2-3	
CP 51214	NR	6-15-50	1-2-3	
crotoxyphos	NR	6-15-50	1-2-3	
crufomate	NR	6-15-50		
cyanazine	NR	6-15-50		
cycloate	V (43-65%) C	15+50	3	
cyfluthrin	P (60%)	15		
cymoxanil	NR	6-15-50	1-2-3	Not eluted from Florisil.
cypermethrin	C	15	2	
cyproconazole	NR	6-15-50	1-2-3	
cyprodinil	NR	6-15-50	1-2-3	Not eluted from Florisil.
dazomet	NR	6-15-50	1-2-3	
DCPA	C	15	2	
DDE, o,p'-	C	6	1	Partially elutes in PE forerun.
DDE, p,p'-	C	6	1	Elutes in PE forerun.

Table 303-a: Recovery Through 303 (E1-E5 + C1 or C2 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
DDMS	R	6		
DDT, o,p'-	C	6	1	
DDT, p,p'-	C	6	1	
deltamethrin	S (32-65%)	15		Very poor EC detector sensitivity.
	C		2	
deltamethrin, trans-	P (50-67%) V (47-142%)	15	2	
demeton-O	NR	6-15		
demeton-S	NR	6-15-50		
des N-isopropyl isofenphos	S (30%)	50		
desdiethyl simazine	NR	6-15-50	1-2-3	
desethyl simazine		50		43% elution from Florisil only in 50% EE/PE.
	NR		1-2-3	
desisopropyl iprodione		50	1-2-3	17% eluted from Florisil only with 50% EE/PE; not eluted with CH_2Cl_2 eluants.
desmethyl norflurazon	NR	6-15-50	1-2-3	
di-allate	C	6		
di-n-octyl phthalate	C	15+50		Poor and variable EC detector sensitivity.
dialifor	C	15		
			2	Complete elution from Florisil only in CH_2Cl_2 eluant #2.
diazinon	C	15	3	
diazinon oxygen analog	NR	6-15-50	1-2-3	
dibutyl phthalate	C	15+50		
dichlobenil	P	15	2	
dichlofenthion	C	6	2	

Table 303-a: Recovery Through 303 (E1-E5 + C1 or C2 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
dichlofluanid	C V (51-91%)	15+50	2+3	Elution from Florisil variable in CH_2Cl_2 eluants.
dichlone	NR S (30%)	6-15-50	2+3	Elution from Florisil variable.
dichlorobenzene, p-	C	6	1	
dichlorobenzophenone, o,p'-	C	15	2	
dichlorobenzophenone, p,p'-	C	15	2	
dichlorvos	NR	6-15-50	1-2-3	
diclobutrazol	NR	6-15-50	1-2-3	
diclofop-methyl	C	15	2	
dicloran	S (35%)	15+50	2+3	
dicofol, o,p'-	V (50-100%)	6+15	2	Elution from Florisil may be variable.
dicofol, p,p'-	V (68-99%) V (78-90%)	6+15	1+2	Elution from Florisil variable.
dicrotophos	NR	6-15-50		
dieldrin	C	15	2	
diethatyl-ethyl	NR	6-15-50	1-2-3	
diethyl phthalate	P	15+50		Poor EC detector sensitivity.
diisobutyl phthalate	P (75%)	15+50		About 80% elution from Florisil only in 15+50% EE/PE.
diisohexyl phthalate	C	15+50		Poor EC detector sensitivity.
diisooctyl phthalate	C	15+50		Poor EC detector sensitivity.
Dilan	P (65%)	15		
dimethenamid	NR	6-15-50	1-2-3	
dimethipin	NR	6-15-50	1-2-3	
dimethoate	NR	6-15-50	1-2-3	

Table 303-a: Recovery Through 303 (E1-E5 + C1 or C2 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
dimethomorph (prop)	NR	6-15-50	1-2-3	Recovery tested using high temperature column.
dimethyl phthalate	P	6+15+50		Partial elution from Florisil only in all EE/PE; poor EC detector sensitivity.
dinocap	P	15		75% elution from Florisil only in 15% EE/PE.
			2	Complete elution from Florisil only in CH_2Cl_2 eluant #2.
dioxabenzofos	P (72%)	15		
dioxathion	NR	6-15-50		
			2	45% elution from Florisil only in CH_2Cl_2 eluant #2.
diphenamid	NR	6-15		
diphenylamine	S (<20%)	6+15		<10% elution from Florisil only in each 6 and 15% EE/PE.
disulfoton	P (50-74%)	6		25-40% elution from Florisil only in 6% EE/PE.
	NR		1-2-3	
disulfoton sulfone	NR	6-15-50		
diuron	NR	6-15-50	1-2-3	
endosulfan I	C	15	2	
endosulfan II	C	15+50	2	
endosulfan sulfate	C	50	2	
endrin	C	15		
	V		2	60-90% elution from Florisil only in CH_2Cl_2 eluant #2.
endrin alcohol	P (50%)	15+50		
			2+3	Partial (48%) elution from Florisil only in CH_2Cl_2 eluant #2, 28% in #3.
endrin aldehyde	P (50%)	15+50		
endrin ketone	C	50	2	

Table 303-a: Recovery Through 303 (E1-E5 + C1 or C2 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
EPN	C	15		
			2	71% elution from Florisil only in CH_2Cl_2 eluant #2.
EPTC	P (63%)	15		
esfenvalerate	C	15	2	
ethalfluralin	C	6	2	Elution from Florisil in CH_2Cl_2 eluants may be variable.
ethametsulfuron methyl ester	NR	6-15-50	1-2-3	
ethephon		6+15+50	1+2+3	5-25% eluted from Florisil only in each eluate.
ethiofencarb	NR	6-15-50		
ethion	C	6		
			2	60-90% elution from Florisil only in CH_2Cl_2 eluant #2.
ethoprop	P (55%)	50		
	NR		1-2-3	
ethoxyquin	NR	6-15-50		
ethylenethiourea	NR	6-15-50	1-2-3	
etridiazole	C	6		Other data show poor recovery through C1.
			2	Percent elution from Florisil only varies in different reports.
etrimfos	C	15	2+3	
famphur	NR	6-15-50		
fenac	NR	6-15-50		
fenamiphos	NR	6-15-50	1-2-3	
fenamiphos sulfone	NR	6-15-50	1-2-3	
fenamiphos sulfoxide	NR	6-15-50	1-2-3	
fenarimol	P (60%)	50		Quantitation may be influenced by presence of sample extract.
	S (40%)		3	

Table 303-a: Recovery Through 303 (E1-E5 + C1 or C2 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
fenarimol metabolite B	NR	6-15-50		
fenarimol metabolite C		6		17% elution from Florisil only in 6% EE/PE; no elution in 15 or 50% EE/PE.
fenbuconazole	NR	6-15-50	1-2-3	
fenitrothion	C	15	2	
fenoxaprop ethyl ester	V (58-125%)	50	3	Partial (70%) elution from Florisil only in either elution system.
fenpropathrin	V (43-71%)	15		Complete (111-116%) elution from Florisil only in 15% EE/PE.
	P (55-65%)		2	Partial (56-62%) elution from Florisil only in CH_2Cl_2 eluant #2.
fenpropimorph		50		Partial (49-63%) elution from Florisil only in 50% EE/PE.
			1-2-3	Not recovered from Florisil only in CH_2Cl_2 eluates.
fensulfothion	NR	6-15-50	1-2-3	
fensulfothion oxygen analog	NR	6-15-50		
fensulfothion sulfone	NR	6-15-50		
fenthion	S (45%)	6+15		
	NR		1-2-3	
fenthion oxygen analog	NR	6-15-50	1-2-3	
fenthion oxygen analog sulfoxide	NR	6-15-50	1-2-3	
fenthion sulfone	NR	6-15-50	1-2-3	
fenvalerate	C	15	2	
fipronil	S (21-41%)	50	3	
fluazifop butyl ester	C	15	3	Poor EC detector sensitivity with OV-225.
fluchloralin	C	6	2	

Table 303-a: Recovery Through 303 (E1-E5 + C1 or C2 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
flucythrinate	C	15		
			2+3	Elution from Florisil only 95% in CH_2Cl_2 eluant 2, 6% in eluant 3.
fluridone	NR	6-15-50		
fluvalinate	C	15		
			2	Complete elution in Florisil only in CH_2Cl_2 eluant 2.
folpet	C	15+50		
	C (80%)		2+3	Complete elution from Florisil only in CH_2Cl_2 eluants #2 & 3.
fonofos	C	6	2+3	
fonofos oxygen analog	NR	6-15-50	1-2-3	
formothion	NR	6-15-50	1-2-3	
fosthiazate	NR	6-15-50	1-2-3	
furilazole	S (28-50%)	50	3	Complete elution from Florisil only in 50% EE/PE, CH_2Cl_2 #3.
Gardona	NR	6-15-50	1-2-3	
GS-31144	NR	6-15-50	1-2-3	
heptachlor	C	6	1	
heptachlor epoxide	C	6	2	
hexachlorobenzene	C	6	1	Elutes in PE forerun.
hexachlorobutadiene	V (62-88%)	6		Elutes in PE forerun.
	P (78%)		1	
hexachlorophene	NR	6-15-50		
hexachlorophene dimethyl ether	NR	6-15		
hexazinone	NR	6-15-50	1-2-3	
hexythiazox	S (2-20%)	50		Florisil pattern and recoveries vary; may elute in 15%, may be complete.
	C		2+3	

Table 303-a: Recovery Through 303 (E1-E5 + C1 or C2 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
hydroxy chloroneb	NR	6-15		
imazalil	NR	6-15-50		
imidacloprid	NR	6-15-50	1-2-3	
IN-A3928	NR	6-15-50	1-2-3	
IN-B2838	NR	6-15-50	1-2-3	
IN-T3936	NR	6-15-50	1-2-3	
iprodione	S (5-56%) NR	50	 1-2-3	
iprodione metabolite isomer	S (21-100%)	50		
isazofos	C	50		Recovery test performed on corn grain and beef liver.
	P		2+3	
isofenphos	C	15+50		
isopropalin	C	6		
isoxaflutole (prop)	V (60-120%)	50		Complete elution from Florisil only in 50% EE/PE
	NR		3	32-56% elution from Florisil only in CH_2Cl_2 eluant 3.
Korax	NR	6-15		
KWG 1323	NR	6-15-50	1-2-3	
leptophos	C	6	2	
lindane	C	6	1	
linuron	V (42-64%) S (19-33%)	50	 3	
malathion	C	15+50	3	Elution from Florisil variable in EE/PE eluants.
malathion oxygen analog	NR	6-15-50	1-2-3	
MB45950	P (50-73%)	15+50	2+3	
MB46136	S (28-55%)	50	2+3	

Table 303-a: Recovery Through 303 (E1-E5 + C1 or C2 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
mecarbam		50		Partial (43%) elution from Florisil only in 50% EE/PE.
merphos	C	6+15+50	3	Elution from Florisil variable. Partial (48%) elution from Florisil only in CH_2Cl_2 eluant #3.
metalaxyl	NR	6-15-50	1-2-3	
methabenzthiazuron	NR	6-15-50	1-2-3	
methidathion	S (35%)	50	3	Complete elution from Florisil only in CH_2Cl_2 eluant #3.
methidathion oxygen analog	NR	6-15-50	1-2-3	
methidathion sulfone	NR	6-15-50	1-2-3	
methidathion sulfoxide	NR	6-15-50	1-2-3	
methiocarb sulfone	NR	6-15-50	1-2-3	
methomyl	NR	6-15-50	1-2-3	
methoxychlor olefin	C	6	2	
methoxychlor, o, p'-	C	6		
methoxychlor, p, p'-	C	6	2	
methyl 4-chloro-1H-indole-3-acetate		R	50	
	NR		1-2-3	Not eluted from Florisil only in CH_2Cl_2 eluants.
metobromuron	NR	6-15-50	1-2-3	
metolachlor	S (28-70%)	50		
	NR		1-2-3	
metoxuron	NR	6-15-50	1-2-3	
metribuzin	NR	50	1-2-3	Complete elution from Florisil only in 50% EE/PE; may be S recovery thru method.
metribuzin, deaminated diketo metabolite		NR	6-15-50	1-2-3
metribuzin, deaminated metabolite	NR	6-15-50	1-2-3	

Table 303-a: Recovery Through 303 (E1-E5 + C1 or C2 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
metribuzin, diketo metabolite	NR	6-15-50	1-2-3	
mevinphos, (E)-	NR	6-15-50		
mevinphos, (Z)-	NR	6-15-50		
mirex	C	6	1	Elutes in PE forerun.
monocrotophos	NR	6-15-50	1-2-3	
monuron	NR	6-15-50	1-2-3	
myclobutanil	NR	6-15-50	1-2-3	
myclobutanil alcohol metabolite	NR	6-15-50	1-2-3	
myclobutanil dihydroxy metabolite	NR	6-15-50	1-2-3	
N, N-diallyl dichloroacetamide	S (41-51%)	15+50	2+3	Complete elution from Florisil only in 15+50% EE/PE, CH_2Cl_2 eluants 2+3.
N-(3,4-dichlorophenyl)-N'-methylurea		NR	6-15-50	
naled	NR	6-15-50	1-2-3	
neburon	NR	6-15-50	1-2-3	
nitralin	P (60%)	50		50-80% elution from Florisil only in 50% EE/PE.
			3	75% elution from Florisil only in CH_2Cl_2 eluant #3.
nitrapyrin	C	6	2	Complete elution from Florisil only in 6% EE/PE or CH_2Cl_2 eluant #2.
nitrofen	C	15	2	
nitrofluorfen	C	15	2	
nonachlor, cis-	C	6	1	
nonachlor, trans-	C	6	1	Elutes in PE forerun.
norflurazon	NR	6-15-50		
NTN33823	NR	6-15-50	1-2-3	
NTN35884	NR	6-15-50	1-2-3	

Table 303-a: Recovery Through 303 (E1-E5 + C1 or C2 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
nuarimol		50		35% elution from Florisil only in 50% EE/PE.
	NR		1-2-3	
octachlor epoxide	C	6	1	
omethoate	NR	6-15-50	1-2-3	
oryzalin	NR	6-15-50		
ovex	C	15	2	
oxadiazon	C	15		
oxadixyl	NR	6-15-50	1-2-3	
oxamyl oxime metabolite	NR	6-15-50	1-2-3	
oxyfluorfen	C	15	2	Poor N/P detector sensitivity.
parathion	C	15	2	
parathion oxygen analog	NR	6-15-50	1-2-3	
parathion-methyl	C	15	2	
parathion-methyl oxygen analog	NR	6-15-50	1-2-3	
PB-9	NR	6-15-50	1-2-3	
pebulate	P (70%)	15		68% elution from Florisil only in 15% EE/PE; none eluted in 50%.
pendimethalin	C	15	2	
pentachloroaniline	C	6	1	
pentachlorobenzene	C	6	1	Elutes in PE forerun.
pentachlorobenzonitrile	C	15	2	
pentachlorophenyl methyl ether	C	6	1	
pentachlorophenyl methyl sulfide	C	6	1	

Table 303-a: Recovery Through 303 (E1-E5 + C1 or C2 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
permethrin, cis-	V (60-115%)	6+15		Complete elution from Florisil only in 6+15% EE/PE; high temp column recommended.
	C		2	High temperature column recommended.
permethrin, trans-	V (60-115%)	6+15		Complete elution from Florisil only in 6+15% EE/PE; high temp column recommended.
	C		2	High temperature column recommended.
Perthane	C	6	1	
Perthane olefin	C	6	1	
phenthoate	C	15+50		
phorate	V (40-75%)	6		Elution from Florisil quite variable.
	C		1	
phorate oxygen analog	NR	6-15-50	1-2-3	
phorate oxygen analog sulfone	NR	6-15-50	1-2-3	
phorate oxygen analog sulfoxide	NR	6-15-50	1-2-3	
phorate sulfone	NR	6-15-50		
	S (34-38%)		3	38% elution from Florisil only in CH_2Cl_2 eluant #3.
phorate sulfoxide	NR	6-15-50	1-2-3	
phosalone	C	50	2+3	
phosmet	NR	6-15-50		
			3	Partial (60%) elution from Florisil only in CH_2Cl_2 eluant #3.
phosmet oxygen analog	NR	6-15-50		
phosphamidon	NR	6-15-50	1-2-3	
photodieldrin	C	15+50	2	
pirimiphos-ethyl	C	15+50	3	
pirimiphos-methyl	C	15	3	

Table 303-a: Recovery Through 303 (E1-E5 + C1 or C2 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
PPG-2597	NR	6-15-50	1-2-3	
PPG-947	NR	6-15-50	1-2-3	
procymidone	C (84%)	15		
profenofos	P (65%)	50		Partial (56%) elution from Florisil only in 50% EE/PE.
			3	Partial (38%) elution from Florisil only in CH_2Cl_2 eluant #3.
profluralin	V (70-100%)	6		Complete elution from Florisil only in 6% EE/PE.
Prolan	S (40%)	15		
			2	Complete elution from Florisil only in CH_2Cl_2 eluant #2.
prometryn	P (50%)	50		Variable (22-67%) elution from Florisil only in 50% EE/PE.
	NR		1-2-3	
pronamide	P (63-71%)	15+50		39% elution from Florisil only in 6% EE/PE, 24% in 50% EE/PE.
propachlor	NR	6-15-50	1-2-3	Trace amount may be eluted in CH_2Cl_2 eluant #3.
propanil	NR	6-15		
			3	Partial (41%) elution from Florisil only in CH_2Cl_2 eluant #3.
propargite	C	15	2	
propazine	S (41%)	15+50		Complete but variable elution from Florisil only in 15%+50% EE/PE.
			3	Also elution of trace amount from Florisil only in CH_2Cl_2 eluant #2.
propetamphos	C (80%) P (50%)	15+50	2+3	

Table 303-a: Recovery Through 303 (E1-E5 + C1 or C2 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
propham	P (53%)	15		Addn 8-16% elution from Florisil only in 50% EE/PE; none in 50% thru method.
propiconazole	NR	6-15-50	1-2-3	
prosulfuron	NR	6-15-50	1-2-3	
prothiofos	C C	6	 2	 79% elution from Florisil only in CH_2Cl_2 eluant #2.
pyrazon	NR	6-15-50	1-2-3	
pyrazon metabolite B	NR	6-15-50	1-2-3	
pyrethrins	C	50		
pyrimethanil	S (11-51%)	50	3	Complete elution from Florisil only in 50% EE/PE or CH_2Cl_2 eluant #3.
quinalphos	C	15		
quintozene	C	6	1	
RH-6467	NR	6-15-50	1-2-3	
RH-9129	NR	6-15-50	1-2-3	
RH-9130	NR	6-15-50	1-2-3	
ronnel	C	6	2	
ronnel oxygen analog	NR	6-15-50		
RPA202248	NR	6-15-50	1-2-3	
S-bioallethrin	C	50		
schradan	NR	6-15-50		
sethoxydim	NR	6-15-50	3	CH_2Cl_2 elution from Florisil only tested with eluant #3 only, not #1 or #2.
sethoxydim sulfoxide	NR	6-15-50	3	CH_2Cl_2 elution from Florisil only tested with eluant #3 only, not #1 or #2.

Table 303-a: Recovery Through 303 (E1-E5 + C1 or C2 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
simazine	NR	50		Complete elution from Florisil only in 50% EE/PE.
	NR		1-2-3	
Strobane	C	6	1	
sulfallate	C	6+15	2	Elution with EE/PE may be variable.
sulfanilamide	NR	6-15-50	1-2-3	
sulfotep	C	6+15		Wide bore column recommended.
			2	50% elution from Florisil only in CH_2Cl_2 eluant #2.
Sulphenone		50	3	Complete elution from Florisil only in 20+25% EE/PE or in CH_2Cl_2 eluant #3.
TCMTB	P (50-67%)	15		P (61-62%) elution from Florisil only in 15% EE/PE; no elution in 50% EE/PE.
TDE, o,p'-	C	6	1	
TDE, p,p'-	C	6	1	
TDE, p,p'-, olefin	C	6	1	Partially elutes in PE forerun.
tebufenozide	NR	6-15-50	1-2-3	
tebupirimfos	V (50-115%)	6+15	2+3	Elution from Florisil only also variable.
tebupirimfos oxygen analog	NR	6-15-50	1-2-3	
tecnazene	C	6	1	
teflubenzuron	NR	6-15-50	1-2-3	
terbacil	NR	6-15		
			2+3	30% elution from Florisil only in CH_2Cl_2 eluant #2, 13% in eluant #3.
terbufos	P (62%)	6		
terbufos oxygen analog sulfone	NR	6-15-50	1-2-3	

Table 303-a: Recovery Through 303 (E1-E5 + C1 or C2 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
terbufos sulfone	NR	6-15-50		
	C (77-86%)		2+3	Elution from Florisil only in CH_2Cl_2 eluant #2 (46%) and #3 (37%).
terbuthylazine	P (57%)	15+50		
tetradifon	C	15	2	
tetraiodoethylene	P (65%)	6		
tetramethrin	NR	6-15-50	1-2-3	Trace amount may elute from Florisil in CH_2Cl_2 eluant #3.
tetrasul	C	6	1	
thiabendazole	NR	6-15-50		
thiobencarb		15	2+3	40% elution from Florisil only in 15% EE/PE; 42% inCH_2Cl_2 #2, 11% in CH_2Cl_2 #3.
thiometon	NR	6-15-50		
thionazin	P (59%)	15+50		Complete (80%) elution from Florisil only in 15% and/or 50% EE/PE.
THPI	NR	6-15-50		
toxaphene	C	6	1	
tralkoxydim		50		20% elution from Florisil only in 50% EE/PE.
	NR		1-2-3	
tralomethrin	V (50-100%)	15	2	
tri-allate	C	6	2	
triadimefon	S (27-40%)	50		35% elution from Florisil only in 50% EE/PE.
	NR		1-2-3	No elution from Florisil in CH_2Cl_2 eluants.
triadimenol	NR	6-15-50		
triazamate	NR	6-15-50	1-2-3	

Table 303-a: Recovery Through 303 (E1-E5 + C1 or C2 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
tribufos	C	15+50	3	Partial, variable elution from Florisil only in CH_2Cl_2 eluant #3.
tributyl phosphate	R	50		
trichlorfon	NR	6-15-50	1-2-3	
trichloronat	C	6		
tridiphane	C	6	1+2	20% elution from Florisil only in CH_2Cl_2 eluant #1, 80% in #2.
trifluralin	C	6	2	
triflusulfuron methyl ester	NR	6-15-50	1-2-3	
tris(chloropropyl) phosphate	NR	6-15-50	1-2-3	
Tycor	S (1-19%)	50	3	Complete elution from Florisil only in 50% EE/PE, 50-60% in CH_2Cl_2 #3.
vernolate	P (65%)	15		
vinclozolin	C	15	2	
vinclozolin metabolite B	P (55-66%) V (60-105%)	6+15	2	
vinclozolin metabolite E	S (9-39%)	15+50		
vinclozolin metabolite F	NR	6-15-50	1-2-3	
vinclozolin metabolite S	P (55-70%)	15	2	
WAK4103	NR	6-15-50	1-2-3	

Table 304-a: Recovery of Chemicals Through Method 304 (E1-E5 + C1-C4 + DG1-DG19)
(extraction of fat from fatty products, acetonitrile/petroleum ether partitioning, Florisil column cleanup, GLC determination with various columns and detectors)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
1,2,3-trichlorobenzene	P (60%)	6		Elutes in PE forerun.
			1	Complete elution from Florisil only in CH_2Cl_2 eluant #1; elutes in PE forerun.
1,2,4-triazole	NR	6-15-50	1-2-3	
2,3,5-trimethacarb		50		50% elution from Florisil only in 50% EE/PE eluant.
	NR		1-2-3	
2,4-dichloro-6-nitrobenzenamine		15	2	
2,6-dichlorobenzamide	NR	6-15-50	1-2-3	
2-chloroethyl caprate	C	15	2	
2-chloroethyl laurate	C	15	2	
2-chloroethyl linoleate	P (73-80%)	15	2	
2-chloroethyl myristate	V (42-80%)	15	2	
2-chloroethyl palmitate	P (50-59%)	15	2	
2-methoxy-3,5,6-trichloropyridine	C	6+15	1+2	
3,4,5-trimethacarb		50		Partial (20-35%) elution from Florisil only in 50% EE/PE.
	NR		1-2-3	
3,4-dichlorophenylurea	NR	6-15-50		
3,5-dichloroaniline	S (22-43%)	15	2	
3-(3,4-dichlorophenyl)-1-methoxyurea		NR	6-15-50	

[1] Codes: C: complete (>80%); P: partial (50-80%); S: small (<50%); V: variable (approximate percentage when known); R: recovered but no quantitative information available; NR: not recovered.

[2] Recovery results refer to complete method; blank entry in this column indicates Florisil elution was tested but not complete method. Separate results are listed for C1 and C2 only if recovery is affected by Florisil elution system used.

[3] Eluants(s) in which chemical is eluted from Florisil, according to directions in 304 C1, *i.e.*, 6, 15, and 50% ethyl ether/petroleum ether (EE/PE). Entries for chemicals not recovered indicate which eluants were used in tests.

[4] Eluants(s) in which chemical is eluted from Florisil, according to directions in 304 C2, *i.e.*, methylene chloride (CH_2Cl_2) eluants #1, 2, and 3. Entries for chemicals not recovered indicate which eluants were used in tests.

[5] "Florisil only" refers to tests in which elution patterns were tested by added reference standard solutions directly to Florisil column.

[6] Reference to petroleum ether (PE) forerun refers to Florisil elution performed as in 304 C3 or C4.

Table 304-a: Recovery Through 304 (E1-E5 + C1-C4 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
3-desmethyl sulfentrazone	NR	6-15-50	1-2-3	
3-hydroxymethyl-2,5-dimethyl= phenyl methylcarbamate	NR	6-15-50	1-2-3	
3-ketocarbofuran	NR	6	1	60% recovered from Florisil only in 6% EE/PE or CH_2Cl_2 #1; also elutes with PE.
3-methyl-4-nitrophenol	NR	6-15-50	1-2-3	
3-tert-butyl-5-chloro-6-hydroxy= methyluracil	NR	6-15-50	1-2-3	
4-chlorobenzylmethyl sulfone	NR	6-15-50	1-2-3	
4-chlorobenzylmethyl sulfoxide	NR	6-15-50	1-2-3	
4-hydroxymethyl-3,5-dimethyl= phenyl methylcarbamate	NR	15-50	1-2-3	<20% elution from Florisil only in 15+50% EE/PE; <10% in CH_2Cl_2 eluants 1,2,3.
6-chloro-2,3-dihydro-3,3,7-methyl-5H-oxazolo(3,2-a)pyrimidin-5-one	NR	6-15-50	1-2-3	
6-chloro-2,3-dihydro-7-hydroxy= methyl-3,3-methyl-5H-oxazolo= (3,2-a)pyrimidin-5-one	NR	6-15-50	1-2-3	
6-chloronicotinic acid	NR	6-15-50	1-2-3	
acetochlor	P (52-70%)	15+50	2+3	Complete elution from Florisil only in 50% EE/PE or CH_2Cl_2 eluant #3.
acifluorfen	NR	6-15-50	1-2-3	
acrinathrin	NR			Complete elution from Florisil only in 15% EE/PE.
	V(27-80%)		2	
alachlor	C	50		
	S (23%)		3	16% elution from Florisil only in CH_2Cl_2 eluant #3.
aldrin	C	6	1	
allethrin	C	50		Elution from Florisil in EE/PE may be variable.
	P (66-75%)		3	

Table 304-a: Recovery Through 304 (E1-E5 + C1-C4 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
anilazine	P	15+50	2+3	
aramite	NR			
Aroclor 1016	C	6	1	Elutes in PE forerun.
Aroclor 1221	C	6	1	Elutes in PE forerun.
Aroclor 1242	C	6	1	Elutes in PE forerun.
Aroclor 1248	C	6	1	Elutes in PE forerun.
Aroclor 1254	C	6	1	Elutes in PE forerun.
Aroclor 1260	C	6	1	Elutes in PE forerun.
Aroclor 1262	C	6	1	Elutes in PE forerun.
Aroclor 4465	C	6	1	Elutes in PE forerun.
atrazine	NR		1-2-3	
azinphos-ethyl	S (14%)	50	3	49-79% elution from Florisil only in CH_2Cl_2 eluant #3.
azinphos-methyl	NR	6-15	1-2-3	
benfluralin	C	6	2	
benoxacor	C	15+50	2+3	
bensulide	C	50	3	14% elution from Florisil only in CH_2Cl_2 eluant #3.
benzoylprop-ethyl	NR	6-15-50	1-2-3	
BHC, alpha-	C	6	1	Partially elutes in PE forerun.
BHC, beta-	C	6	1	
BHC, delta-	C	6+15	1	EE/PE elution variable.
bifenox	P (51-78%)	15+50	2+3	51-58% elution from Florisil with EE/PE; 56-78% with CH_2Cl_2.
bifenthrin		6+15	2	

Table 304-a: Recovery Through 304 (E1-E5 + C1-C4 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
binapacryl	P (65%)	15		
bis(2-ethylhexyl) phthalate	C	15+50		Poor EC detector sensitivity.
bromacil	NR	6-15-50	1-2-3	
bromophos	C	6		
bromophos-ethyl	P (59-78%)	6		
bromopropylate	C NR	15+50	 1-2-3	
Bulan	P (75%)	15	 2	 Complete elution from Florisil only in CH_2Cl_2 eluant #2.
butyl benzyl phthalate	P (70%)	15+50		Complete elution from Florisil only in15+50% EE/PE.
cadusafos	NR	6-15-50	1-2-3	
captan	C (80%)	50		
carbophenothion	P (60%)	6	 2	Elution from Florisil may be variable. <60% elution from Florisil only in CH_2Cl_2 eluant #2.
carbophenothion oxygen analog	NR	6-15-50	1-2-3	
carbophenothion sulfone	P (66%)	6	1	Elutes in PE forerun.
carboxin	NR	6-15-50		
carboxin sulfoxide	NR	6-15-50	1-2-3	
CGA 118244	NR	6-15-50	1-2-3	
CGA 120844	NR	6-15-50	1-2-3	
CGA 14128		50	1-2-3	9-22% elution from Florisil only in EE/PE; not recovered in CH_2Cl_2 eluants.
CGA 171683		15+50	3	Complete elution from Florisil only in 15+50% EE/PE, 70% in CH_2Cl_2 eluant #3.
CGA 205374	NR	6-15-50	1-2-3	

Table 304-a: Recovery Through 304 (E1-E5 + C1-C4 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
CGA 37734	NR	6-15-50	1-2-3	
CGA 91305	NR	6-15-50	1-2-3	
CGA 94689A	NR	6-15-50	1-2-3	
CGA 94689B	NR	6-15-50	1-2-3	
chlorbenside	P	6	1	Recovery 50% using EE/PE eluants; may be better with CH_2Cl_2.
chlorbromuron	V (44-100%)	50	3	Complete elution from Florisil only.
chlorbufam		15	2+3	Complete elution from Florisil only in 15% EE/PE, 77% in CH_2Cl_2 eluants 2+3.
chlordane	C	6	1	
chlordane, cis-	C	6	1	May elute in PE forerun.
chlordane, trans-	C	6	1	
chlordecone	P NR	15+50	 1-2-3	Elution from Florisil variable.
chlordene	C	6	1	Elutes in PE forerun.
chlorfenapyr (prop)	S (30-50%)	50	2	Complete elution from Florisil only in 50% EE/PE and CH_2Cl_2 eluant 2.
chlorfenvinphos, alpha-	NR	6-15-50		
chlornitrofen	C	6+15	2	Variable elution from Florisil in EE/PE.
chlorobenzilate	P (75%) NR	15+50	 3	Some variable elution from Florisil in CH_2Cl_2 eluant #3.
chloroneb			2	82% elution from Florisil only in CH_2Cl_2 eluant #2.
chloropropylate	C	15+50	 3	Some variable elution from Florisil in CH_2Cl_2 eluant #3.

Table 304-a: Recovery Through 304 (E1-E5 + C1-C4 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
chlorothalonil	NR C (80-90%)	6-15-50	 2+3	
chlorothalonil trichloro impurity	NR	6-15-50		
chloroxuron	NR	6-15-50	1-2-3	
chlorpropham	C	15	2	
chlorpyrifos	P (74-83%)	6	2	
chlorsulfuron	NR	6-15-50		
chlorthiophos	C	6	2	11% elution from Florisil only in CH_2Cl_2 eluant #2.
chlorthiophos oxygen analog	NR	6-15-50	1-2-3	
chlorthiophos sulfone		50		55% elution from Florisil only in 50% EE/PE.
chlorthiophos sulfoxide	NR	6-15-50	1-2-3	
clomazone		50	3	88% elution from Florisil only in 50% EE/PE, 54-74% in CH_2Cl_2 eluant #3.
clopyralid methyl ester		50		17% elution from Florisil only in 50% EE/PE.
coumaphos	NR C (76-93%)	6-15-50	 3	High temperature or short column GLC needed.
coumaphos oxygen analog	NR	6-15-50	1-2-3	
CP 51214	NR	6-15-50	1-2-3	
crotoxyphos	NR	6-15-50	1-2-3	
crufomate	NR	6-15-50		
cycloate	S (39-61%) S (24-37%)	15+50	 3	
cymoxanil	NR	6-15-50	1-2-3	Not eluted from Florisil.
cypermethrin	C (81%)	15	2	

Table 304-a: Recovery Through 304 (E1-E5 + C1-C4 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
cyproconazole	NR	6-15-50	1-2-3	
cyprodinil	NR	6-15-50	1-2-3	Not eluted from Florisil.
DCPA	C	15	2	
DDE, o,p'-	C	6	1	Partially elutes in PE forerun.
DDE, p,p'-	C	6	1	Elutes in PE forerun.
DDT, o,p'-	C	6	1	
DDT, p,p'-	C	6	1	
deltamethrin	P (77-80%)	15	2	Very poor EC detector sensitivity.
deltamethrin, trans-	NR			Partial (33%) elution from Florisil only in 15% EE/PE, complete in CH_2Cl_2 eluant #2.
desdiethyl simazine	NR	6-15-50	1-2-3	
desethyl simazine		50		43% elution from Florisil only in 50% EE/PE.
	NR		1-2-3	
desmethyl norflurazon	NR	6-15-50	1-2-3	
di-n-octyl phthalate	C	15+50		Poor and variable EC detector sensitivity.
dialifor	P (50%)	15		
			2	Complete elution from Florisil only in CH_2Cl_2 eluant #2.
diazinon	C	15	3	
diazinon oxygen analog	NR	6-15-50	1-2-3	
dibutyl phthalate	C	15+50		
dichlobenil	C (80%)	15	2	
dichlofenthion	V (69-89%)	6		
			2	
dichlone	NR	6-15-50		
	S (25%)		2+3	Elution from Florisil variable.

Table 304-a: Recovery Through 304 (E1-E5 + C1-C4 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
dichlorobenzene, p-	C	6	1	
dichlorobenzophenone, o,p'-	C	15	2	
dichlorobenzophenone, p,p'-	C	15	2	
dichlorvos	NR	6-15-50	1-2-3	
diclobutrazol	NR	6-15-50	1-2-3	
diclofop-methyl	C	15	2	
dicloran	P (50%)	15+50	2+3	
dicofol, o,p'-	S (25-50%)	6+15	2	Elution from Florisil may be variable.
dicofol, p,p'-	P (61-85%) S (36-58%)	6+15	 1+2	Elution from Florisil variable.
dieldrin	C	15	2	
diethatyl-ethyl	NR	6-15-50	1-2-3	
diethyl phthalate	P	15+50		Poor EC detector sensitivity.
diisobutyl phthalate		15+50		About 80% elution from Florisil only in 15+50% EE/PE.
diisohexyl phthalate		15+50		Complete elution from Florisil only in 15+50% EE/PE; poor EC sensitivity.
diisooctyl phthalate	C	15+50		Poor EC detector sensitivity.
Dilan	P (65%)	15		
dimethenamid	NR	6-15-50	1-2-3	
dimethipin	NR	6-15-50	1-2-3	
dimethoate	NR	6-15-50	1-2-3	
dimethomorph (prop)	NR	6-15-50	1-2-3	
dimethyl phthalate		6+15+50		Partial elution from Florisil only in all EE/PE; poor EC detector sensitivity.

Table 304-a: Recovery Through 304 (E1-E5 + C1-C4 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
dinitramine	P (78-80%)	15		Some elution from Florisil in 6% EE/PE.
dinocap	P (60%)	15		75% elution from Florisil only in 15% EE/PE.
			2	Elution from Florisil only, complete in CH_2Cl_2 eluant #2.
dioxathion			2	45% elution from Florisil only in CH_2Cl_2 eluant #2.
disulfoton		6		25-40% elution from Florisil only in 6% EE/PE.
	NR		1-2-3	
diuron	NR	6-15-50	1-2-3	
endosulfan I	C	15	2	
endosulfan II	C	15+50	2	
endosulfan sulfate	C	50	2	
endrin	C	15		
	V		2	60-90% elution from Florisil only in CH_2Cl_2 eluant #2.
endrin alcohol	C	15+50		
			2+3	Partial (48%) elution from Florisil only in CH_2Cl_2 eluant #2, 28% in #3.
endrin aldehyde	C	15+50		
endrin ketone	C	50	2	
EPN	C	15		
			2	71% elution from Florisil only in CH_2Cl_2 eluant #2.
esfenvalerate	C	15	2	
ethalfluralin	C	6	2	Elution from Florisil in CH_2Cl_2 eluants may be variable.
ethametsulfuron methyl ester	NR	6-15-50	1-2-3	
ethephon		6+15+50	1+2+3	5-25% eluted from Florisil only in each eluant.

Table 304-a: Recovery Through 304 (E1-E5 + C1-C4 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
ethiofencarb	NR	6-15-50		
ethion	C	6		
			2	60-90% elution from Florisil only in CH_2Cl_2 eluant #2.
ethoprop	S (45%)	50		
	NR		1-2-3	
ethoxyquin	NR	6-15-50		
ethylenethiourea	NR	6-15-50	1-2-3	
etridiazole	P (68-73%)	6		Other data shows poor recovery through C1.
			2	Percent elution from Florisil only varies in different reports.
etrimfos	C	15	2+3	
fenac	NR	6-15-50		
fenamiphos	NR	6-15-50	1-2-3	
fenamiphos sulfone	NR	6-15-50	1-2-3	
fenamiphos sulfoxide	NR	6-15-50	1-2-3	
fenarimol	C	50		Quantitation may be influenced by presence of sample extract.
	V (72-110%)		3	
fenarimol metabolite B	NR	6-15-50		
fenarimol metabolite C		6		17% elution from Florisil only in 6% EE/PE; no elution in 15 or 50% EE/PE.
fenbuconazole	NR	6-15-50	1-2-3	
fenitrothion	C	15	2	
fenoxaprop ethyl ester	V (65-110%)	50	3	Partial (70%) elution from Florisil only in either elution system.

Table 304-a: Recovery Through 304 (E1-E5 + C1-C4 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
fenpropathrin	V (59-114%)	15		Elution from Florisil only, complete (111-116%) in 15% EE/PE.
	V (58-91%)		2	Elution from Florisil only, partial (56-62%) in CH_2Cl_2 eluant #2.
fenpropimorph		50		Partial (49-63%) elution from Florisil only in 50% EE/PE.
			1-2-3	Not recovered from Florisil only in CH_2Cl_2 eluates.
fensulfothion	NR	6-15-50	1-2-3	
fenthion	NR	6-15	1-2-3	
fenthion oxygen analog	NR	6-15-50	1-2-3	
fenthion oxygen analog sulfoxide	NR	6-15-50	1-2-3	
fenthion sulfone	NR	6-15-50	1-2-3	
fenvalerate		15		Complete elution from Florisil only in 15% EE/PE.
	C (81%)		2	
fipronil	V (55-97%)	50	3	
fluazifop butyl ester	V (50-110%)	15	3	Poor EC detector sensitivity with OV-225.
fluridone	NR	6-15-50		
folpet	P (50%)	15+50		
			2+3	Complete elution from Florisil only in CH_2Cl_2 eluants #2 & 3.
fonofos	C	6	2+3	
fonofos oxygen analog	NR	6-15-50	1-2-3	
formothion	NR	6-15-50	1-2-3	
fosthiazate	NR	6-15-50	1-2-3	
Gardona	NR	6-15-50	1-2-3	
GS-31144	NR	6-15-50	1-2-3	
heptachlor	C	6	1	

Table 304-a: Recovery Through 304 (E1-E5 + C1-C4 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
heptachlor epoxide	C	6	2	
hexachlorobenzene	P (60%)	6		Loss in partitioning from PE to acetonitrile/water; elutes in PE forerun.
			1	Complete elution from Florisil only in CH_2Cl_2 eluant #1; elutes in PE forerun.
hexachlorobutadiene	P (63%)		1	Elutes in PE forerun.
hexachlorophene	NR	6-15-50		
hexachlorophene dimethyl ether	NR	6-15		
hexazinone	NR	6-15-50	1-2-3	
hexythiazox	NR	6-15-50	1-2-3	Complete elution from Florisil only in 15+50% EE/PE, CH_2Cl_2 eluants 2+3.
imazalil	NR	6-15-50		
imidacloprid	NR	6-15-50	1-2-3	
IN-A3928	NR	6-15-50	1-2-3	
IN-B2838	NR	6-15-50	1-2-3	
IN-T3936	NR	6-15-50	1-2-3	
iprodione		50		Partial (4-19%) elution from Florisil only in 50% EE/PE.
	NR		1-2-3	
isoxaflutole (prop)	S (37-126%)	50		Complete elution from Florisil only in 50% EE/PE.
	NR			32-56% elution from Florisil only in CH_2Cl_2 eluant 3.
KWG 1323	NR	6-15-50	1-2-3	
lactofen	C	50	2+3	
leptophos	C	6	2	
lindane	C	6	1	
linuron	V (42-62%)	50		
			3	

Table 304-a: Recovery Through 304 (E1-E5 + C1-C4 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
malathion	C	15+50	3	Variable elution from Florisil in EE/PE eluants.
malathion oxygen analog	NR	6-15-50	1-2-3	
MB45950	V (60-190%)	15+50	2+3	
MB46136	V (54-140%)	50	2+3	
mecarbam		50		Partial (43%) elution from Florisil only in 50% EE/PE.
merphos	C	6+15+50		Variable elution from Florisil in EE/PE eluants.
			3	Partial (48%) elution from Florisil only in CH_2Cl_2 eluant #3.
metalaxyl	NR	6-15-50	1-2-3	
methabenzthiazuron	NR	6-15-50	1-2-3	
methidathion	P (50%) C (80%)	50	 3	
methidathion oxygen analog	NR	6-15-50	1-2-3	
methidathion sulfone	NR	6-15-50	1-2-3	
methidathion sulfoxide	NR	6-15-50	1-2-3	
methiocarb sulfone	NR	6-15-50	1-2-3	
methomyl	NR	6-15-50	1-2-3	
methoxychlor olefin	C	6	2	
methoxychlor, p, p'-	C	6	2	
methyl 4-chloro-1H-indole-3-acetate	NR		1-2-3	Not eluted from Florisil only in CH_2Cl_2 eluants.
metobromuron	NR	6-15-50	1-2-3	
metolachlor	NR		1-2-3	
metoxuron	NR	6-15-50	1-2-3	
metribuzin	NR	50	1-2-3	Complete elution from Florisil only in 50% EE/PE; may be S recovery thru method.

Table 304-a: Recovery Through 304 (E1-E5 + C1-C4 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
metribuzin, deaminated diketo metabolite	NR	6-15-50	1-2-3	
metribuzin, deaminated metabolite	NR	6-15-50	1-2-3	
metribuzin, diketo metabolite	NR	6-15-50	1-2-3	
mevinphos, (E)-	NR	6-15-50		
mirex	P (75%)	6	1	Loss in partitioning from PE to acetonitrile/water; elutes in PE forerun.
monocrotophos	NR	6-15-50	1-2-3	
monuron	NR	6-15-50	1-2-3	
myclobutanil	NR	6-15-50	1-2-3	
myclobutanil alcohol metabolite	NR	6-15-50	1-2-3	
myclobutanil dihydroxy metabolite	NR	6-15-50	1-2-3	
N, N-diallyl dichloroacetamide	S (32-47%)	15+50	2+3	Complete elution from Florisil only in 15+50% EE/PE, CH_2Cl_2 eluants 2+3.
N-(3,4-dichlorophenyl)-N'-methylurea	NR	6-15-50		
naled	NR	6-15-50	1-2-3	
neburon	NR	6-15-50	1-2-3	
nitralin	P (70%)	50	3	50-80% elution from Florisil only in 50% EE/PE. 75% elution from Florisil only in CH_2Cl_2 eluant #3.
nitrapyrin	V (32-111%)	6	2	Complete elution from Florisil only in 6% EE/PE or CH_2Cl_2 eluant #2.
nitrofen	C	15	2	
nitrofluorfen	C	15	2	
nonachlor, cis-	C	6	1	

Table 304-a: Recovery Through 304 (E1-E5 + C1-C4 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
nonachlor, trans-	C	6	1	Elutes in PE forerun.
norflurazon	NR	6-15-50		
NTN33823	NR	6-15-50	1-2-3	
NTN35884	NR	6-15-50	1-2-3	
nuarimol	C	50		35% elution from Florisil only in 50% EE/PE.
	NR		1-2-3	
octachlor epoxide	C	6	1	
omethoate	NR	6-15-50	1-2-3	
oryzalin	NR	6-15-50		
ovex	C	15	2	
oxadiazon	P (75%)	15		
oxadixyl	NR	6-15-50	1-2-3	
oxamyl oxime metabolite	NR	6-15-50	1-2-3	
oxyfluorfen	C	15	2	
parathion	C	15	2	
parathion oxygen analog	NR	6-15-50	1-2-3	
parathion-methyl	C	15	2	
parathion-methyl oxygen analog	NR	6-15-50	1-2-3	
PB-9	NR	6-15-50	1-2-3	
pendimethalin	P (33-56%)	15		
	P (66-82%)		2	
pentachloroaniline	C	6	1	
pentachlorobenzene	C	6	1	Elutes in PE forerun.
pentachlorobenzonitrile	P (60%)	15		
			2	Complete elution from Florisil only in CH_2Cl_2 eluant #2.
pentachlorophenyl methyl ether	C	6	1	

Table 304-a: Recovery Through 304 (E1-E5 + C1-C4 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
pentachlorophenyl methyl sulfide	C	6	1	
permethrin, cis-		6+15		Complete elution from Florisil only in 6+15% EE/PE; high temp column recommended.
	C (82%)		2	High temperature column recommended.
permethrin, trans-		6+15		Complete elution from Florisil only in 6+15% EE/PE; high temp col recommended.
	C (82%)		2	High temperature column recommended.
Perthane	C	6	1	
Perthane olefin	C	6	1	
phorate	V (80%)	6		Elution from Florisil quite variable, may be 0%.
	C		1	
phorate oxygen analog	NR	6-15-50	1-2-3	
phorate oxygen analog sulfone	NR	6-15-50	1-2-3	
phorate oxygen analog sulfoxide	NR	6-15-50	1-2-3	
phorate sulfone	NR	6-15-50		
	S (12-20%)		3	38% elution from Florisil only in eluant 3.
phorate sulfoxide	NR	6-15-50	1-2-3	
phosalone	C	50	2+3	
phosmet			3	Partial (60%) elution from Florisil only in CH_2Cl_2 eluant #3.
phosmet oxygen analog	NR	6-15-50		
phosphamidon	NR	6-15-50	1-2-3	
photodieldrin	C	15+50	2	
pirimiphos-ethyl	C	15+50	3	
pirimiphos-methyl	C	15	3	

Table 304-a: Recovery Through 304 (E1-E5 + C1-C4 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
PPG-1576	P	50	2+3	72-85% elution from Florisil only in EE/PE; 54-75% in CH_2Cl_2 eluants.
PPG-2597	NR	6-15-50	1-2-3	
PPG-947	NR	6-15-50	1-2-3	
procymidone	P (76%)	15		
profenofos	P (50%)	50		Partial (56%) elution from Florisil only in 50% EE/PE.
			3	Partial (38%) elution from Florisil only in CH_2Cl_2 eluant #3.
Prolan	S (25%)	15		
			2	Complete elution from Florisil only in CH_2Cl_2 eluant #2.
prometryn	P (70%)	50		Variable (22-67%) elution from Florisil only in 50% EE/PE.
	NR		1-2-3	
propachlor	NR	6-15-50	1-2-3	Trace amount may be eluted in CH_2Cl_2 eluant #3.
propanil	NR	6-15		
			3	Partial (41%) elution from Florisil only in CH_2Cl_2 eluant #3.
propargite		15	2	Complete elution from Florisil only in 15% EE/PE or CH_2Cl_2 eluant #2.
propazine	NR	15+50		Complete but variable elution from Florisil only in 15%+50% EE/PE.
			3	Also elution of trace amount from Florisil only in CH_2Cl_2 eluant #2.
propham	P (80%)	15		Addition 8-16% elution from Florisil only in 50% EE/PE; none in 50% thru method.
propiconazole	NR	6-15-50	1-2-3	

Table 304-a: Recovery Through 304 (E1-E5 + C1-C4 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
prosulfuron	NR	6-15-50	1-2-3	
prothiofos	C	6		
			2	79% elution from Florisil only in CH_2Cl_2 eluant #2.
pyrazon	NR	6-15-50	1-2-3	
pyrazon metabolite B	NR	6-15-50	1-2-3	
pyrethrins	C	50		
pyrimethanil	S (0-40%)	50	3	Complete elution from Florisil only in 50% EE/PE or CH_2Cl_2 eluant 3.
	P (75-82%	50	3	
quintozene	C	6	1	
RH-6467	NR	6-15-50	1-2-3	
RH-9129	NR	6-15-50	1-2-3	
RH-9130	NR	6-15-50	1-2-3	
ronnel	C	6	2	
RPA202248	NR	6-15-50	1-2-3	
sethoxydim	NR	6-15-50	3	CH_2Cl_2 elution from Florisil tested with eluant #3 only, not #1 or #2.
sethoxydim sulfoxide	NR	6-15-50	3	CH_2Cl_2 elution from Florisil tested with eluant #3 only, not #1 or #2.
simazine		50		Complete elution from Florisil only in 50%EE/PE.
	NR		1-2-3	
Strobane	C	6	1	
sulfallate	C	6+15	2	Elution with EE/PE may be variable.
sulfanilamide	NR	6-15-50	1-2-3	

Table 304-a: Recovery Through 304 (E1-E5 + C1-C4 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
sulfotep	P (65-70%)	6+15	2	65% elution from Florisil with EE/PE; 70% with CH_2Cl_2; need wide bore column.
Sulphenone		50	3	Complete elution from Florisil only in 20+25% EE/PE or in CH_2Cl_2 eluant #3.
TCMTB	P (61-62%)	15		P (61-62%) elution from Florisil only in 15% EE/PE; no elution in 50% EE/PE.
TDE, o,p'-	C	6	1	
TDE, p,p'-	C	6	1	
TDE, p,p'-, olefin	C	6	1	Partially elutes in PE forerun.
tebufenozide	NR	6-15-50	1-2-3	
tebupirimfos	V (57-171%)	6+15	2+3	Elution from Florisil only also variable.
tebupirimfos oxygen analog	NR	6-15-50	1-2-3	
tecnazene	C	6	1	
teflubenzuron	NR	6-15-50	1-2-3	
terbacil	NR	6-15	2+3	30% elution from Florisil only in CH_2Cl_2 eluant #2, 13% in eluant #3.
terbufos	S (16%)	6		
terbufos oxygen analog	NR	6-15-50	1-2-3	
terbufos oxygen analog sulfone	NR	6-15-50	1-2-3	No elution from Florisil in either elution system.
terbufos sulfone	NR C	6-15-50	2+3	Elution from Florisil only in CH_2Cl_2 eluant #2 (46%) and #3 (37%).
tetradifon	C	15	2	
tetraiodoethylene	P (65%)	6		

Table 304-a: Recovery Through 304 (E1-E5 + C1-C4 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
tetramethrin	NR	6-15-50	1-2-3	May be elution of trace amount from Florisil in CH_2Cl_2 eluant #3.
tetrasul	C	6	1	
thiobencarb	V (<50-86%)	15		40% elution from Florisil only in 15% EE/PE.
			2+3	42% elution from Florisil only in CH_2Cl_2 #2, 11% in CH_2Cl_2 #3.
thiometon	NR	6-15-50		
thionazin	NR	15+50		Complete (80%) elution from Florisil only in 15% and/or 50% EE/PE.
THPI	NR	6-15-50		
toxaphene	C	6	1	
tralkoxydim		50		20% elution from Florisil only in 50% EE/PE.
	NR		1-2-3	
tralomethrin	S (0-50%)	15	2	
tri-allate	C	6	2	
triadimefon	S (13-62%)	50		35% elution from Florisil only in 50% EE/PE.
	NR		1-2-3	No elution from Florisil in CH_2Cl_2 eluants.
triadimenol	NR	6-15-50		
triazamate	NR	6-15-50	1-2-3	
tribufos	P (60%)	15+50		
			3	Partial, variable elution from Florisil only in eluant #3.
trichlorfon	NR	6-15-50	1-2-3	
trichloronat		6		Complete elution from Florisil only in 6% EE/PE.
tridiphane		6	1+2	20% elution from Florisil only in CH_2Cl_2 eluant #1, 80% in #2.

Table 304-a: Recovery Through 304 (E1-E5 + C1-C4 + DG1-DG19)

Chemical	Recovery[1,2]	Eluant, C1[3]	Eluant, C2[4]	Notes[5,6]
trifluralin	C	6	2	
triflusulfuron methyl ester	NR	6-15-50	1-2-3	
tris(chloropropyl) phosphate	NR	6-15-50	1-2-3	
Tycor	S (12-162%)	50	3	Complete elution from Florisil only in 50% EE/PE, 50-60% in CH_2Cl_2 #3.
vinclozolin	C	15	2	
vinclozolin metabolite B	C	6+15	2	Recovery in 6% EE/PE; other studies showed split into 15%.
vinclozolin metabolite E	NR	6-15-50		
vinclozolin metabolite F	NR	6-15-50	1-2-3	
vinclozolin metabolite S	V (47-81%) C	15	 2	
WAK4103	NR	6-15-50	1-2-3	

Table 304-b: Recovery of Chemicals Through Method 304 (E1-E5 + C6 + DG1-DG19)
(extraction of fat from fatty products, cleanup with gel permeation and Florisil column chromatography, GLC determination with various columns and detectors)

Chemical	Recovery[1]	Eluant[2]	Notes
1,2,3,5-tetrachlorobenzene	V (41-138%)	1	mean recovery 85.9%, n=15
1,2,4,5-tetrachloro-3-(methylthio)= benzene	C	1	mean recovery 86%, n=11
2,3,5,6-tetrachloroanisidine	V (47-108%)	2	mean recovery 82.8%, n=10
2,3,5,6-tetrachloroanisole	C	1	mean recovery 89.6%, n=10
2,3,5,6-tetrachloronitroanisole	V (47-135%)	1+2	mean recovery 76.4%, n=10
2-chloroethyl linoleate	V (0-102%)	2	mean recovery 66.86%, n=7
2-chloroethyl palmitate	V (0-105%)	2	mean recovery 68.0%, n=7
alachlor	S (0-121%)	3	mean recovery 47.6%, n=13
aldrin	C	1	mean recovery 85.2%, n=21
alpha-cypermethrin	C	2	mean recovery 89.3%, n=12
anilazine	S (4-87%)	2+3	mean recovery 32%, n=11
bromopropylate	NR	1-2-3	
captan	S (0-88%)	3	mean recovery 33.4%, n=13
carbophenothion	NR	1-2-3	mean recovery 3%, n=12
chlorfenvinphos, beta-	NR	1-2-3	mean recovery 2%, n=13
chlornitrofen	C	2	69-114% recovered, TDS
chlorobenzilate	NR	1-2-3	
chlorothalonil	S (0-86%)	2+3	mean recovery 37%, n=26
chlorpropham	C	2	mean recovery 82%, n=14
chlorpyrifos	C	2	mean recovery 83%, n=39
chlorpyrifos-methyl	C	2	mean recovery 83.4%, n=14
cyfluthrin	P	2	mean recovery 77.8%, n=14
DDE, p,p'-	P	1	recovery 75.3%, n=1
DDT, o,p'-	C	1	mean recovery 88.2%, n=13
diazinon	C	3	69-114% recovery, TDS
dichlofenthion	C	2	mean recovery 87.5%, n=12
diclofop-methyl	C	2	mean recovery 88.1%, n=25
dicloran	V (49-111%)	2+3	mean recovery 76.8%, n=12
dieldrin	C	2	mean recovery 87.7%, n=130

[1] Codes: C: complete (>80%); P: partial (50-80%); S: small (<50%); V: variable (approximate percentage when known); R: recovered but no quantitative information available; NR: not recovered.

[2] Eluants(s) in which chemical is eluted from Florisil, according to directions in 304 C6, *i.e.*, methylene chloride (CH_2Cl_2) eluants #1, 2, and 3. Entries for chemicals not recovered indicate which eluants were used in tests.

Table 304-b: Recovery Through 304 (E1-E5 + C6 + DG1-DG19)

Chemical	Recovery[1]	Eluant[2]	Notes
endosulfan I	C	2	mean recovery 89.0%, n=12
endosulfan sulfate	C	2	mean recovery 88.9%, n=27
endrin	C	2	recovery 82.7%, n=1
esfenvalerate	C	2	mean recovery 88.8%, n=12
ethion oxygen analog	NR	1-2-3	
fenarimol	S (0-58%)	3	mean recovery 13.5%, n=13
fenthion	NR	1-2-3	mean recovery 0.5%, n=13
fenvalerate	V (69-130%)	2	mean recovery 91%, n=14
fluchloralin	C	2	mean recovery 81%, n=11
haloxyfop methyl ester	C	2+3	mean recovery 91.3%, n=4 , 80% eluant 2 remainder in 3.
heptachlor	C	1	recovery 96.7%, n=1
heptachlor epoxide	C	2	mean recovery 84.4%, n=28
iprodione	S (0-52%)	3	mean recovery 16.2%, n=16 ; trace amount eluated by elauant 3.
iprodione metabolite isomer	V (12-120%)	3	mean recovery 73.2%, n=28 .
lindane	C	1	mean recovery 87.2%, n=40
linuron	V (43-114%)	3	mean recovery 73.5%, n=24
mecarbam	V (13-92%)	3	mean recovery 71.6%, n=15 .
methidathion	C	3	
methoxychlor, o, p'-	C	2	83-124% recoveries, TDS
methoxychlor, p, p'-	C	2	mean recovery 88.2%, n=12
metolachlor	NR	1-2-3	
mirex	C	1	mean recovery 89.4%, n=17
nonachlor, cis-	C	1	
nuarimol	NR	1-2-3	
octachlor epoxide	C	1	mean recovery 90.5%, n=32
parathion	C	2	mean recovery 81.7%, n=82
pentachlorophenyl methyl sulfide	C	1	mean recovery 84.7%, n=17
Perthane	C	1	mean recovery 87.5%, n=15
phenthoate	P		mean recovery 76.0%, n=12; eluant data to be tested.
phosalone	S (17-78%)	2+3	mean recovery 39.8%, n=16
phosmet	S	3	37%, 67% recoveries, TDS
pirimiphos-ethyl	V (29-109%)	3	mean recovery 67.2%, n=13

Table 304-b: Recovery Through 304 (E1-E5 + C6 + DG1-DG19)

Chemical	Recovery[1]	Eluant[2]	Notes
propargite	P	2	mean recovery 79.5%, n=12
prothiofos	P	2	mean recovery 71.9%, n=13
pyrazophos	C		recovery 107%, n=1 ; eluant data to be tested.
sulprofos	NR	1-2-3	
TDE, o,p'-	C	1	mean recovery 95.9%, n=15
TDE, p,p'-	C	1	mean recovery102.4%, n=14
TDE, p,p'-, olefin	C	1	mean recovery 86.0%, n=13
tecnazene	C	1	58-108% recoveries, TDS
tetradifon	C	2	
tridiphane	C	1+2	mean recovery 84.5%, n=13
vinclozolin	C	2	mean recovery 83.3%, n=12

Table 304-c: Recovery of Chemicals Through Method 304 (E2 + C7 + DG1-DG19)
(extraction of fat from fatty products, Florisil column cleanup, GLC determination with various columns and detectors)

Chemical	Recovery[1, 2]	Eluant[3]	Notes
aldrin	C	6	mean recovery 94.8%, n=19
Aroclor 1254	C	6	mean recovery 80.0%, n=18
chlordane	C	6	recovery 83.0%, n=1
chlordane, cis-	C	6	recovery 93.0%, n=1
chlordane, trans-	C	6	recovery 124%, n=1
chlorpyrifos	C	6	mean recovery 88.0%, n=12
DDE, p,p'-	C	6	recovery 107%, n=1
DDT, p,p'-	C	6	mean recovery 87.2%, n=8
diazinon	C	15	mean recovery 107%, n=1
dieldrin	C	15	mean recovery 97.1%, n=31
heptachlor epoxide	C	6	
nonachlor, cis-	C	6	recovery 103%, n=1
nonachlor, trans-	S(0.8-32%)	6	mean recovery 16.4%, n=2
octachlor epoxide	C	6	recovery 83.0%, n=1
pentachlorobenzene	C	6	recovery 100%, n=1
TDE, p,p'-	V (59-93%)	6	mean recovery 75.5%, n=4

[1] Codes: C: complete (>80%); P: partial (50-80%); S: small (<50%); V: variable (approximate percentage when known); R: recovered but no quantitative information available; NR: not recovered.

[2] Recovery results do not include the optional alkaline hydrolysis step.

[3] Eluants(s) in which chemical is eluted from Florisil, according to directions in 304 C7, *i.e.*, 6 and 15% ethyl ether/petroleum ether (EE/PE). Entries for chemicals not recovered indicate which eluants were used in tests.

Table 401-a: Recovery of Chemicals Through Method 401 (E1-E2 + C1 + DL1)
(methanol extraction, cleanup with partitioning and charcoal/Celite column, HPLC with post-column derivatization and fluorescence detection)

Chemical	Recovery[1]	Rrt[2]	ng[3]	Notes
2,3,5-trimethacarb	C			
3,4,5-trimethacarb	C			
3-hydroxycarbofuran	C	0.6	10	
3-hydroxymethyl-2,5-dimethyl= phenyl methylcarbamate	P (70%)			
3-hydroxymethyl-4,5-dimethyl= phenyl methylcarbamate	C			
3-ketocarbofuran	V (67-110%)	0.85	11	
4-hydroxymethyl-3,5-dimethyl= phenyl methylcarbamate	C			
aldicarb	C	0.83	14	
aldicarb sulfoxide	P (50-60%)	0.33	9	
aldoxycarb	C	0.4	9	
bendiocarb	C	1	10	
bufencarb	C	1.44	19	Major peak is listed.
butocarboxim	C	0.75	15	
carbaryl	C	1.06	7	
carbofuran	C	1	10	
dioxacarb	C	0.67	15	
ethiofencarb	P (70-82%)	1.1	15	Breaks down to 2 peaks; other rrt 0.5.
fenobucarb	C	1.47	10	
isoprocarb	C	1.13	8	
methiocarb	C	1.26	10	
methiocarb sulfone	C	0.79	11	
methiocarb sulfoxide	C	0.64	12	
methomyl	C	0.46	10	32% recovery from peanuts.
metolcarb	C	0.85	10	
oxamyl	C	0.44	10	
promecarb	C	1.31	10	

[1] Codes: C: complete (>80%); P: partial (50-80%); S: small (<50%); V: variable (approximate percentage when known); R: recovered but no quantitative information available; NR: not recovered.

[2] Retention time, relative to carbofuran, on C-8 column described in DL1.

[3] ng that cause 50% full scale deflection detector response in DL1.

Table 401-a: Recovery Through Method 401 (E1-E2 + C1 + DL1)

Chemical	Recovery[1]	Rrt[2]	ng[3]	Notes
propoxur	C	0.98	8	
thiodicarb	P (40-60%)	0.99	11	Recovery C if analytical breakdown product (methomyl) also measured.
XMC	C	1.06	10	

Table 401-b: Recovery of Chemicals Through Method 401 (E1-E2 + C1 + DL2)

(methanol extraction, cleanup with partitioning and charcoal/Celite column, HPLC with fluorescence detection)

Chemical	Recovery[1]	Rrt[2]	ng[3]	Notes[4]
carbaryl	C	1.06	3	Ex L 288, Em L 330.
carbofuran	C	1	90	Ex L 288, Em L 330.
CGA 161149	V (43-99%)	0.73	10	Ex L 288, Em L 330.
CGA 195654	S (15-132%)	0.57	300	Ex L 288, Em L 330.
dioxacarb	C	0.67	180	Ex L 265, Em L 294.
fluometuron	V (60-100%)	1.09	50	Ex L 288, Em L 330. Low level residues may be obscured by matrix interferences.
isoprocarb	C	1.13	370	Ex L 264, Em L 292.
naphthaleneacetamide	P (77%)	0.75	3	Ex L 288, Em L 320. For C rec., elute charcoal with additional 100 mL petr ether
napropamide	C	1.36	4	Ex L 288, Em L 330.
phosalone	C	1.7	90	Ex L 288, Em L 330.
phosalone oxygen analog	C	1.3	90	Ex L 288, Em L 330.
piperonyl butoxide	C	1.74	5	Ex 288, Em L 330.
propoxur	C	0.98	40	Ex L 276, Em L 300.

[1] Codes: C: complete (>80%); P: partial (50-80%); S: small (<50%); V: variable (approximate percentage when known); R: recovered but no quantitative information available; NR: not recovered.

[2] Retention time, relative to carbofuran, on C-8 column described in DL1/DL2.

[3] ng that cause 50% full scale deflection detector response in DL2.

[4] Excitation (Ex) and emission (Em) wavelengths found optimum for the chemical.

Table 402-a: Recovery of Chemicals Through Method 402 (E1-E7 + C1 + DG1 or DG3 or DG4)
(extraction from acidified mixture, GPC, methylation, and Florisil cleanup, determination by GLC)

Chemical	Recovery[1-3]	Notes[4,5]
2,3,5,6-tetrachlorotere= phthalic acid	E1: NR E2: NR	Methylated completely, but did not elute from GPC.
2,3,5-triiodobenzoic acid	E1: V (66-86%) E2: V (79-138%)	No ester reference standard.
2,3,6-TBA	E1: C E2: C	
2,3-dihydro-3,3-methyl-2-oxo-5-benzofuranyl methyl sulfonate	E1: NR E2: NR	Chemical did not methylate.
2,4,5-T	E1: P E2: P	79% mean recovery, 31% CV, n=270, nonfat and fat.
2,4-D	E1: P E2: P	72% mean recovery, 34% CV, n=186, nonfat and fat.
2,4-DB	E1: C E2: C	
2-hydroxy-2,3-dihydro-3,3-methyl-5-benzofuranyl methyl sulfonate	E1: NR E2: NR	Chemical did not methylate.
3,5,6-trichloro-2-pyridinol	NR	Some (<20%) recovered in 100mL ethyl ether.
3,5-dibromo-4-hydroxy= benzoic acid	S (0-42%)	
3-carboxy-5-ethoxy-1,2,4-thiadiazole	NR	Methyl ester not eluted from Florisil column.
3-chlorosulfonamide acid	NR	Complete recovery from Florisil only, 14% from GPC.
3-methyl-4-nitrophenol		Methyl ether completely eluted from Florisil, but only 30% from GPC.

[1] Codes: C: complete (>80%); P: partial (50-80%); S: small (<50%); V: variable (approximate percentage when known); R: recovered but no quantitative information available; NR: not recovered.

[2] Extraction module used during testing (*e.g.*, E1) is indicated with each result.

[3] Florisil eluted with Eluant 1 and Eluant 2 only; chemicals eluted in ethyl ether (EE) are considered NR through basic method as normally performed.

[4] Ester/ether elutes from Florisil with Eluant 2 unless otherwise noted.

[5] When no reference material available for ester/ether, recoveries calculated against acid/phenol methylated per method.

Table 402-a: Recovery Through 402 (E1-E7 + C1 + DG1 or DG3 or DG4)

Chemical	Recovery[1-3]	Notes[4,5]
4-chlorobenzoic acid	E1: S (27-66%) E2: S (2-76%)	Low temperature column needed to detect methyl ester.
4-CPA	E1: S (32-69%) E2: C	Chromatographs only on wide bore GLC. No ester reference standard.
6-chloropicolinic acid	NR	Methylates, but methyl ester does not elute from Florisil.
AC 263,222 ammonium salt	NR	Methyl ester not eluted from Florisil.
acifluorfen	E1: P (54-69%)	
alloxydim-sodium	E1: NR E2: NR	Does not methylate.
arsanilic acid		Compound did not methylate under method conditions.
benazolin	E1: NR E2: NR	28-32% recovered if Florisil eluted with additional 100 mL EE. Complete recovery if Florisil eluted with additional 100 mL EE.
bifenox	E1: C E2: C	Parent is methyl ether.
bromacil	E2: NR	Complete recovery if Florisil eluted with additional 100 mL EE.
bromofenoxim	E1: P (57-86%) E2: C	No ether reference standard.
bromoxynil	E1: P (50-68%) E2: C	No ether reference standard.
chloramben	E1: S (40-43%) E2: P (49-59%)	
chloroxuron	E1: NR E2: NR	Does not methylate.
clofencet potassium salt	NR	Does not methylate.
cloprop	E1: P (50-66%) E2: C	Chromatographs only on wide bore GLC. No ester reference standard.

Table 402-a: Recovery Through 402 (E1-E7 + C1 + DG1 or DG3 or DG4)

Chemical	Recovery[1-3]	Notes[4,5]
CP 106070	NR	Does not methylate.
CP 106077	NR	Some methylation, but does not elute from GPC.
CP 108064	E1: NR	NR through method even in 100 mL EE; recovered from GPC only, Florisil only.
	E2: NR	Complete recovery if Florisil eluted with additional 100 mL EE.
CP 108669	NR	Some methylation, but does not elute from GPC.
CP 92429	NR	Does not methylate.
CP 95200	NR	Some methylation, but does not elute from GPC.
CP 97290	NR	Does not methylate.
cyclanilide	E1: C E2: V (45-67%)	
dicamba	E1: P (71-76%) E2: C	
dichlorprop	E1: C (80%) E2: C (72-104%)	No ester reference standard.
diclofop	E1: S (43-51%) E2: V (81-200%)	
dinoseb	E1: NR E2: NR	Does not methylate.
disul-Na	E1: NR	Does not methylate; parent 50% recovered if Florisil eluted with 100 mL EE.
	E2: NR	Does not methylate; complete recovery of parent with 100 mL EE.
DNOC	E1: S (45-50%)	Nitrogen detector required. No ether reference standard.
	E2: C	
dodine	E1: NR E2: NR	Does not methylate.
fenac	E1: C (74-92%) E2: C	No ester reference standard.
flumetsulam	E1: NR E2: NR	Methylated product not soluble in hexane.

Table 402-a: Recovery Through 402 (E1-E7 + C1 + DG1 or DG3 or DG4)

Chemical	Recovery[1-3]	Notes[4,5]
fluroxypyr	E1: S (23-30%)	Two peaks result; 3-7% more eluted with100 mL EE. No ester ref std.
	E2: P (64-77%)	Two peaks result; complete recovery with 100 mL EE. No ester ref std.
haloxyfop	E2: P (54%)	Florisil elution with 100 mL EE not tested.
HOE-038182	E1: NR E2: S (30-41%)	Methylation was complete, but ester not recovered. Elution from Florisil only with eluant #2 + 100 mL EE.
HOE-099730	NR	Does not methylate.
imazamox	NR	Methyl ester not eluted fromFlorisil.
ioxynil	E1: C (80-87%) E2: C	No ether reference standard.
iprodione urea	NR	Methyl ether not eluted from Florisil.
MCPA	E1: C (78-89%) E2: C	
MCPB	E1: C (70-106%)	Chromatographs only on wide bore GLC. No ether reference standard.
	E2: C	
mecoprop	E1: C (73-84%)	Wide bore GLC recommended. No ester reference standard.
	E2: C	
PB-7	E1: NR	Complete recovery if Florisil eluted with additional 100 mL EE.
	E2: NR	
pentachlorophenol	E1: P	70% mean recovery, 31% CV, n=275, nonfat and fat.
	E2: P	
picloram	E1: NR	6-10% recovered if Florisil eluted with additional 100 mL EE.
	E2: NR	Complete recovery if Florisil eluted with additional 100 mL EE.
PPG-947	E1: P (49-78%)	Two peaks from methylation; only one seen by halogen detector.
pyrithiobac-sodium	E1: S (7-13%)	Additional 31-34% recovered if Florisil eluted with 100 ml EE.

Table 402-a: Recovery Through 402 (E1-E7 + C1 + DG1 or DG3 or DG4)

Chemical	Recovery[1-3]	Notes[4,5]
RPA203328	NR	Small (0-34%) recovery in 100 mL ethyl ether.
silvex	E1: C E2: C	
triadimenol	E1: NR E2: NR	Methyl ether not eluted from Florisil.
triclopyr	E1: C E2: C	Recovery from fatty foods may be <50%.
vinclozolin metabolite B	E1: S (26-43%) E2: S (27-43%)	Methylated product is parent vinclozolin; 62% recovery through Florisil only.

Table 403-a: Recovery of Chemicals Through Method 403 (E1 + C1 + DL3 and DL4)
(methanol extraction, cleanup by partitioning and Florisil chromatography, HPLC with post-column photolysis and derivatization, fluorescence detection)

		DL3: Methanol/ Water Mobile Phase		DL4: Acetonitrile/ Water Mobile Phase	
Chemical	**Recovery[1]**	**Rrt[2]**	**Notes[3]**	**Rrt[2]**	**Notes[3]**
chlorbromuron	C	1.16	LD 0.006, LQ 0.022	1.28	LD 0.003, LQ 0.011
chlorotoluron	C	0.87		0.91	
chloroxuron	C	1.25	LD 0.002, LQ 0.008	1.28	LD 0.001, LQ 0.003
diuron	C	1	LD 0.002, LQ 0.007	1	LD 0.001, LQ 0.003
fenuron	C	0.42		0.49	
fluometuron	C	0.87	LD 0.002, LQ 0.006	0.93	LD 0.001, LQ 0.003
isoproturon	C	0.96		1	
linuron	C	1.12	LD 0.004, LQ 0.014	1.23	LD 0.005, LQ 0.017
metobromuron	C	0.91	LD 0.004, LQ 0.015	1.04	LD 0.004, LQ 0.014
metoxuron	C	0.62		0.67	
monolinuron	C	0.91		0.99	
monuron	C	0.72		0.75	
neburon	C	1.34		1.43	
siduron	C	1.08		1.16	

[1] Codes: C: complete (>80%); P: partial (50-80%); S: small (<50%); V: variable (approximate percentage when known); R: recovered but no quantitative information available; NR: not recovered.

[2] Retention time, relative to diuron, on the HPLC system described.

[3] LD, limit of detection: concentration (ppm) of phenylurea found to cause response three times baseline noise; LQ, limit of quantitation: concentration (ppm) found to cause response 10 times baseline noise.

Table 404-a: Recovery of Chemicals Through Method 404 (E1-E3 + DL5)
(methanol extraction, partitioning into methylene chloride, HPLC with ion pairing mobile phase and UV and fluorescence detection)

Chemical	Recovery[1]	Notes
allophanate	C	Determined by UV detector at 250 nm.
benomyl	C	Determined as MBC (carbendazim) by UV detector at 280 nm.
MBC[2]	C	Determined by UV detector at 280 nm.
thiabendazole	C	Determined by UV detector at 280 nm; confirm, increase sensitivity with DL7.
thiophanate-methyl	C	Determined by UV at 280 nm; degrades in extract, must be determined quickly.

[1] Codes: C: complete (>80%); P: partial (50-80%); S: small (<50%); V: variable (approximate percentage when known); R: recovered but no quantitative information available; NR: not recovered.

[2] Residue may result from use of: (1) benomyl, never found as a residue itself, because it is rapidly converted to MBC; (2) thiophanate-methyl, which degrades slowly to MBC; or (3) carbendazim, as MBC is called when used as a fungicide itself (not registered in the U.S.).

APPENDIX I: PESTDATA

Caution: Use this table only as a quick reference for tentative identification of residues found in samples analyzed by the most commonly used PAM I multiresidue methods. Always compare the residue to a standard chromatographed in your own gas chromatograph. Apply appropriate confirmatory tests to verify tentative identification. Note that PESTDATA cannot and does not contain all details; consult PAM I tables that accompany each method for more definitive information about the behavior of the compound through the steps of the methods.

NOTATIONS AND ABBREVIATIONS USED IN PESTDATA

(In all categories a hyphen indicates absence of data)

Name

Preferred name for each chemical. "*" indicates chemicals with multiple GLC peaks. Chlordane, Strobane, toxaphene and all Aroclors are listed only in the table ordered by name and do not appear in the tables ordered by relative retention times.

Molecular Formula

Numbers are not subscripted. Averages are used for multicomponent chemicals.

RRT/c

Columns list retention times (relative to chlorpyrifos) on GLC column indicated. Conditions under which these data were gathered are described in these Section 302 DG modules:

GLC Column	Section 302 DG modules
OV-101	DG1-DG5
OV-17	DG13-DG17
OV-225	DG18, DG19

Note that headers in these tables refer to GLC columns by the names used for packed columns, despite DG1-DG19's descriptions of wide bore capillary column systems, because so many of these rrts were developed using packed columns. Rrt data for equivalent packed and capillary columns are expected to be essentially identical and are combined in PESTDATA.

Responses

Data specify column and detector used. Numbers refer to weight (ng) that causes detector response of approximately 50% full scale deflection (FSD) on the recording device. Response values collected when the detector was combined with a wide bore capillary column include the notation "(WB)." Codes refer to detectors and operating conditions described in these Section 302 DG modules, except that all response values are based on detector sensitivity of 50% FSD to 1.5 ng chlorpyrifos:

Code	Detector	Section 302 DG modules
TR	tritium electron capture	none - obsolete detector
TI	thermionic (KCl)	none - obsolete detector
FP	flame photometric, phosphorus	DG2, DG14, DG19
FS	flame photometric, sulfur	DG15
NI	^{63}Ni electron capture	DG1, DG13, DG18
NP	nitrogen/phosphorus	DG5, DG17
HX	electroconductivity (halogen mode)	DG3, DG16
HN	electroconductivity (nitrogen mode)	DG4
MC	microcoulometric	none - obsolete detector

NOTES: Response values are approximate and can vary dramatically on different chromatographs. Most response values represent rounded-off or averaged values; some were collected under conditions different from those suggested in references.

Recoveries

Data on the recovery of the compound through several PAM I methods are listed in columns with the following headings. See the appropriate PAM I table(s) for more details, such as partial recoveries through Florisil.

Heading	Common Name	PAM I Section	PAM I Table
302	Luke (Los Angeles)	302 E1-E3, no cleanup	302-a
303	Mills, Onley, Gaither	303 E1-E5 + C1 or C2	303-a
304	Mills fatty food	304 E1-E5 + C1-C4	304-a
Ethers	Florisil elution system	303 C1, 304 C1 and C3	303-a, 304-a
CH_2Cl_2	alternative Florisil elution	303 C2, 304 C2 and C4	303-a, 304-a

Recovery codes have the following meanings: C: complete (>80%); P: partial (50-80%); S: small (<50%); V: variable; R: recovered but no quantitative information available; NR: not recovered.

Transmittal No. 2000-1 (10/1999)
Form FDA 2905a (6/92)

Appendix I: PESTDATA Chemicals in Order by Chemical Name

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
1,1'-(2,2-dichloroethylidene)bis(2-methoxybenzene)	C16H16Cl2O2	2.9	-	3.7	-	R	-	-	-
	Responses:								
1,2,3,4-tetrachlorobenzene	C6H2Cl4	-	-	0.09	-	-	-	-	-
	Responses: OV-17: NI0.2								
1,2,3,5-tetrachlorobenzene	C6H2Cl4	-	-	0.07	-	P #	-	6	1
	Responses: OV-17: NI0.1								
1,2,3-trichlorobenzene	C6H3Cl3	0.08	-	-	-	C	P	6	1
	Responses: OV-101: TR2								
1,2,4,5-tetrachloro-3-(methylthio)=benzene	C7H4Cl4S	0.49	0.35	0.48	R	C	-	6	1
	Responses: OV-101: NI0.3 OV-17: HX(WB)0.3 OV-225: NI0.3								
1,2,4,5-tetrachlorobenzene	C6H2Cl4	-	-	0.07	-	-	-	-	-
	Responses: OV-17: NI0.2								
1,2,4-triazole	C2H3N3	0.2	-	0.27	V	NR	NR	6-15-50	1-2-3
	Responses: DEGS: NP3								
1-hydroxychlordene	C10H6Cl6O	0.99	1.63	1.07	-	R	-	15	-
	Responses: OV-101: NI(WB)7 OV-17: NI1 OV-225: NI1								
1-methyl cyromazine	C7H13N6	-	-	0.72	-	-	-	-	-
	Responses: OV-17: NP1000								
10,10-dihydromirex	C10H2Cl10	2.67	-	-	-	C	-	6	-
	Responses: OV-101: NI7								
10-monohydromirex	C10HCl11	4.26	-	-	-	C	-	6	-
	Responses: OV-101: NI7								
2,3,5,6-tetrachloroanisidine	C7H5Cl4NO	0.59	0.73	0.66	-	C	-	6	2
	Responses: OV-101: NI0.5 OV-17: HX(WB)0.6 OV-225: NI0.5								
2,3,5,6-tetrachloroanisole	C7H4Cl4O	0.24	0.15	0.22	-	C	-	6	1
	Responses: OV-101: NI0.2 OV-17: HX(WB)0.3 OV-225: NI0.2								
2,3,5,6-tetrachloronitroanisole	C7H3Cl4NO3	0.56	0.63	0.56	-	C	-	6	1+2
	Responses: OV-101: NI0.4 OV-17: HX(WB)0.5 OV-225: NI0.4								
2,3,5,6-tetrafluoro-4-hydroxymethyl=benzoic acid	C8H4OF4	-	-	-	-	-	-	-	-
	Responses:								

* Multipeak chemical.

\# Recovery may vary with choice of Florisil elution system; see Tables 303-a, 304-a.

Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
2,3,5-trimethacarb	C11H15NO2	0.35	0.6	0.38	C	S #	NR	50	1-2-3
	Responses: OV-101: NP8 OV-17: NP4 OV-225: NP10								
2,3-dihydro-3,3-methyl-2-oxo-5-benzofuranyl methyl sulfonate	C11H12O5S	0.68	2.89	0.93	-	-	-	-	-
	Responses: OV-101: FS25/NI4 OV-17: FS54/NI4.5 OV-225: FS63/NI10								
2,4,5-T BEP ester*	C17H23Cl3O3	0.16	0.14	-	-	-	-	-	-
		0.68	0.66	-					
		1.08	0.91	-					
		2.85	1.19	-					
		3.3	2.78	-					
		5.3	3.28	-					
		7	7.7	-					
	Responses: OV-101: TR35								
2,4,5-T butoxyethyl ester*	C14H17Cl3O4	-	2.66	-	-	-	-	-	-
		2.91	3.3	-					
	Responses: OV-101: TR4								
2,4,5-T butyl esters*	C12H13Cl3O3	-	-	1.05	-	-	-	-	-
		-	-	0.86					
	Responses:								
2,4,5-T ethylhexyl ester	C16H21Cl3O3	3.38	-	2.62	-	-	-	-	-
	Responses: OV-101: NI5								
2,4,5-T isobutyl ester	C12H13Cl3O3	0.94	-	-	-	-	-	-	-
	Responses: OV-101: TR1								
2,4,5-T isooctyl ester*	C16H21Cl3O3	-	2.69	-	-	-	-	-	-
		2.56	3.1	-					
		2.96	3.4	-					
		3.25	3.8	-					
	Responses: OV-101: TR20								
2,4,5-T isopropyl ester	C11H11Cl3O3	0.67	0.65	-	-	-	-	-	-
	Responses: OV-101: TR2								
2,4,5-T methyl ester	C9H7Cl3O3	0.49	0.63	0.47	-	-	-	-	-
	Responses: OV-101: TR1								
2,4,5-T n-butyl ester	C12H13Cl3O3	1.1	-	-	-	-	-	-	-
	Responses: OV-101: TR1								
2,4,5-T propylene glycol butyl ether esters	C15H19Cl3O4	2.37	-	-	-	-	-	-	-
	Responses:								

Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
2,4,5-trichloro-alpha-methylbenzene=methanol	C8H7OCl3	0.34	-	0.25	R	R	-	15	-
	Responses:								
2,4-D BEP ester*	C17H24Cl2O4	-	0.08	-	-	-	-	-	-
		0.69	0.74	-					
		1.66	1.18	-					
		2	1.79	-					
		3.22	2.09	-					
		4.1	5.1	-					
		10.2	13	-					
	Responses: OV-101: TR60								
2,4-D butoxyethyl ester*	C14H18Cl2O4	-	1.67	1.44	-	-	-	-	-
		1.82	2.08	1.79					
	Responses: OV-101: TR12 OV-17: NI5								
2,4-D ethyl hexyl ester*	C16H22Cl2O3	-	1.51	-	-	-	-	-	-
		2.1	1.78	1.68					
	Responses: OV-101: NI5								
2,4-D isobutyl ester	C12H14Cl2O3	0.62	0.62	0.49	-	-	-	-	-
	Responses: OV-101: TR5								
2,4-D isooctyl ester*	C16H22Cl2O3	-	-	1.48	-	-	-	-	-
		2.04	1.78	1.78					
	Responses: OV-101: TR50 OV-17: NI5								
2,4-D isopropyl ester*	C11H12Cl2O3	-	0.62	-	-	-	-	-	-
		0.42	0.74	0.33					
	Responses: OV-101: TR10								
2,4-D methyl ester	C9H8Cl2O3	0.3	0.38	0.25	-	-	-	-	-
	Responses: OV-101: TR6								
2,4-D n-butyl ester	C12H14Cl2O3	0.72	-	-	-	-	-	-	-
	Responses: OV-101: TR40								
2,4-D propylene glycol butyl ether ester*	C15H2OCl2O4	-	1.42	-	-	-	-	-	-
		1.54	3.6	-					
	Responses: OV-101: TR20								
2,4-DB methyl ester	C11H12Cl2O3	0.62	0.72	-	-	-	-	-	-
	Responses: OV-101: TR28								
2,4-dichloro-6-nitrobenzenamine	C6H4Cl2N2O2	0.3	-	-	-	R	-	15	2
	Responses: OV-101: HX2/NI0.4/NP8								

Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
2,6-dichlorobenzamide	C7H5NOCl2	0.39	1.3	0.52	C	NR	NR	6-15-50	1-2-3
Responses:									
2,8-dihydromirex	C10H2Cl10	2.41	-	-	-	C	-	6	-
Responses: OV-101: NI4									
2-chloroethyl caprate	C8H15ClO2	0.32	-	-	-	C	C	15	2
Responses: OV-101: HX2									
2-chloroethyl laurate	C14H27ClO2	0.59	-	-	-	C	C	15	2
Responses: OV-101: HX2									
2-chloroethyl linoleate	C20H35ClO2	4.1	-	-	-	V	P	15	2
Responses: OV-101: HX15									
2-chloroethyl myristate	C16H31ClO2	1.17	-	-	C	V	V	15	2
Responses: OV-101: HX4									
2-chloroethyl palmitate	C18H35ClO2	2.35	-	-	-	V	P	15	2
Responses: OV-101: HX10									
2-hydroxy-2,3-dihydro-3,3-methyl-5-benzofuranyl methyl sulfonate	C11H14O5S	1	6.6	1.46	-	-	-	-	-
Responses: OV-101: FS48/NI135 OV-17: FS88/NI96 OV-225: FS175/NI400									
2-methoxy-3,5,6-trichloropyridine	C6H4Cl3NO	0.19	0.08	0.1	C	P #	C	6+15	1+2
Responses: OV-101: NI0.5/NI(WB)0.4/NP(WB)9 OV-17: HX1.5									
3, 5, 6-trichloro-2-pyridinol methyl ester	C6H4Cl3NO	0.32	0.44	0.36	-	-	-	-	-
Responses: OV-101: NI(WB)0.6/NP(WB)5									
3,4,5-trimethacarb	C11H15NO2	0.45	0.78	0.5	C	NR	NR	50	1-2-3
Responses: OV-101: NP25 OV-17: NP10 OV-225: NP200									
3,4-dichloroaniline	C6H5Cl2N	0.2	0.32	0.16	V	S	-	15	-
Responses: OV-101: HX0.6/NI16/NP1 OV-17: NI13/NP8 OV-225: NI30									
3,4-dichlorophenylurea	C7H6Cl2N2O	0.22	0.14	0.1	-	NR	NR	6-15-50	-
Responses: OV-101: HX9/NI18/NP60 OV-17: NI4 OV-225: NI6									
3,5-dichloroaniline	C6H5Cl2N	0.18	0.27	0.14	S	S	S	6+15	1+2
Responses: OV-101: HN(WB)1/HX0.5/NI9/NI(WB)16/NP0.9/NP(WB)1 OV-17: HN(WB)0.3/HX(WB)2/NI8/NI(WB)14/NP8/NP(WB)0.4 OV-225: NI20/NI(WB)25									
3-(3,4-dichlorophenyl)-1-methoxyurea	C8H8Cl2N2O2	0.21	-	1.36	R	NR	NR	6-15-50	-
Responses: OV-101: HX9/NI25 OV-17: NP250									
3-aminophenol	C6H7NO	-	-	-	-	-	-	-	-
Responses:									

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Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
3-carboxy-5-ethoxy-1,2,4-thiadiazole	C3H2N2O3S	0.22	0.25	0.2	NR	-	-	-	-
Responses: OV-101: NI170/NP40 OV-17: NI160/NP30 OV-225: NI100 DEGS: NI200/NP40									
3-chloro-5-methyl-4-nitro-1H-pyrazole	C4H4ClN3O2	1.07	-	-	C	-	-	-	-
Responses:									
3-desmethyl sulfentrazone	C10H8Cl2F2N4O3S	3.3	-	7.5	-	NR	NR	6-15-50	1-2-3
Responses: OV-101: NI(WB)0.1/NP(WB)37 OV-17: FS(WB)1500/NI(WB)0.1/NP(WB)73									
3-hydroxycarbofuran	C12H15NO4	-	-	-	-	-	-	-	-
Responses:									
3-hydroxymethyl-2,5-dimethylphenyl methylcarbamate	C11H15NO3	0.8	-	1.03	-	NR	NR	6-15-50	1-2-3
Responses: OV-101: NP200 OV-17: NP600									
3-ketocarbofuran	C12H12NO4	0.55	1.41	0.9	S	NR	NR	6	1
Responses: OV-101: HN(WB)17/NI(WB)15/NP(WB)9 OV-17: HN(WB)15/NI(WB)80/NP(WB)30 OV-225: NI(WB)80									
3-methyl-4-nitrophenol	C7H7O3N	0.38	0.63	0.26	V	NR	NR	6-15-50	1-2-3
Responses: OV-101: NI13/NP50 OV-17: NI5/NP20 OV-225: NI250									
3-methyl-4-nitrophenol methyl ether	C8H9O3N	0.17	0.22	0.13	-	-	-	-	-
Responses: OV-101: NI3/NP7 OV-17: NI1/NP2 OV-225: NI2									
3-phenoxybenzenemethanol	C13H12O2	1.28	-	1.6	-	-	-	-	-
Responses: OV-101: NI1000 OV-17: NI1000									
3-tert-butyl-5-chloro-6-hydroxy=methyluracil	C9H13ClN2O3	1.35	2.27	2.55	-	NR	NR	6-15-50	1-2-3
Responses: OV-101: HN(WB)4/HX(WB)40/NI(WB)67/NP(WB)39 OV-17: HN(WB)13/HX(WB)99/NI(WB)35/NP(WB)290 OV-225: NI(WB)630									
4,4'-dichlorobiphenyl	C12H8Cl2	-	-	0.51	-	-	-	-	-
Responses:									
4-(2,4-dichlorophenoxy)=benzenamine	C12H9Cl2NO	1.44	-	-	-	-	-	-	-
Responses: OV-101: TR60									
4-(dichloroacetyl)-1-oxa-4-azapiro=[4.5]decane	C10H15Cl2NO2	0.5	0.69	0.48	C	P	-	50	3
Responses: OV-101: NI1.4/NP34 OV-17: NI1.1/NP1.2 OV-225: NI1.6									
4-chloro-6-methoxyindole	C9H8NOCl	0.54	-	0.66	-	R	-	15	-
Responses: OV-101: HX2.5/NI1000 OV-17: HX0									
4-chlorobenzeneamine	C6H6ClN	-	-	0.07	S	NR	NR	6-15-50	1-2-3
Responses:OV-17:NP(WB)1.5									

Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
4-chlorobenzylmethyl sulfone	C8H9ClO2S	0.41	1.91	0.66	-	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI(WB)0.4 OV-17: NI(WB)0.8 OV-225: NI(WB)2								
4-chlorobenzylmethyl sulfoxide	C8H9ClOS	0.39	1.16	0.54	-	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI(WB)20 OV-17: NI(WB)16 OV-225: NI(WB)55								
4-chlorobiphenyl	C12H9Cl	-	-	0.2	-	-	-	-	-
	Responses:								
4-chlorophenoxyaniline*	C12H10ClNO	0.87	-	1.07	S	-	-	-	-
		1.28	-	1.31					
	Responses: OV-101: HX7/NI1100								
4-chlorophenylurea	C7H7ClN2O	0.54	-	1.07	NR	NR	NR	6-15-50	1-2-3
	Responses:OV-101:NI(WB)15 OV-17:NI(WB)40								
4-hydroxymethyl-3,5-dimethylphenyl methylcarbamate*	C11H15NO3	0.18	-	0.22	-	NR	NR	15-50	1-2-3
		0.27	-	0.31					
	Responses: OV-101: NI200 OV-17: NI150								
6-chloro-2,3-dihydro-3,3,7-methyl-5H-oxazolo(3,2-a)pyrimidin-5-one	C9H13ClN2O2	0.43	1.34	0.6	-	NR	NR	6-15-50	1-2-3
	Responses: OV-101: HN(WB)0.4/NI(WB)26/NP(WB)3 OV-17: HN(WB)0.4/HX(WB)4/NI(WB)36/NP(WB)2 OV-225: NI(WB)51								
6-chloro-2,3-dihydro-7-hydroxy=methyl-3,3-methyl-5H-oxazolo=(3,2-a)pyrimidin-5-one	C9H13ClN2O3	0.86	-	1.55	-	NR	NR	6-15-50	1-2-3
	Responses: OV-101: HN(WB)4/HX(WB)11/NI(WB)28/NP(WB)17 OV-17: HN(WB)3/HX(WB)17/NI(WB)19/NP(WB)12								
6-chloronicotinic acid*	C6H4NO2Cl	-	-	-	-	NR	NR	6-15-50	1-2-3
		-	-	-					
	Responses: DEGS: HX40/NI11/NP66								
8-monohydromirex	C10HCl11	3.74	-	-	-	C	-	6	-
	Responses: OV-101: NI5								
acephate	C4H10NO3PS	0.15	0.64	0.19	C	-	-	-	-
	Responses: OV-101: FP(WB)0.9/NP3 OV-17: FP(WB)0.6 OV-225: FP5								
acetochlor	C14H20NO2Cl	0.75	0.88	0.67	C	C #	P	50	3
	Responses: OV-101: HX5/NI9/NP5 OV-17: NI5 OV-225: NI5								
acifluorfen	C14H7ClF3NO3	1.05	1.47	0.88	-	NR	NR	6-15-50	1-2-3
	Responses: OV-101: HN(WB)170/HX(WB)980/NI(WB)40/NP(WB)270 OV-17: HN(WB)48/HX(WB)390/NI(WB)27/NP(WB)1000 OV-225: NI(WB)300								
acrinathrin	C26H21F6NO5	10.4	12.8	8.9	V	V	V #	15	2
	Responses: OV-101: NI15/NP125 OV-17: NI25/NP100 OV-225: NI40								

Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
alachlor	C14H2OClNO2	0.8	1	0.72	C	C	C #	50	3
	Responses: OV-101: NI(WB)7 OV-17: NI6 OV-225: NI6								
aldrin	C12H8Cl6	1.05	0.58	0.76	C	C	C	6	1
	Responses: OV-101: TR0.8 OV-17: NI1								
allethrin	C19H26O3	1.36	1.22	-	-	C	C #	50	3
	Responses: OV-101: NI8								
allidochlor	C8H12ClNO	0.09	-	-	C	NR	-	6-15	1-2-3
	Responses: OV-101: TR5								
alpha-cypermethrin	C22H19Cl2O3N	14	-	-	C	C	-	-	2
	Responses: OV-101: HX9/NI22								
ametryn	C9H17N5S	0.77	1.1	-	C	-	-	-	-
	Responses:								
aminocarb	C11H16N2O2	0.56	-	-	C	-	-	-	-
	Responses: OV-101: NP10								
amitraz	C19H23N3	-	-	-	S	-	-	-	-
	Responses:								
anilazine	C9H5Cl3N4	1.24	1.88	1.47	V	S	P	15+50	2+3
	Responses: OV-101: HX(WB)8/NI4 OV-17: NP20								
aramite*	C15H23ClO4S	2	2.77	-	C	P	NR	15	-
		2.14	3.05	-					
	Responses: OV-101: FP600/TR10000								
Aroclor 1016*	CHCl (mix)	0.2	0.24	-	-	C	C	6	1
		0.3	0.3	-					
		0.39	0.4	-					
		0.44	0.46	-					
		0.52	0.51	-					
		0.59	0.54	-					
		0.68	0.61	-					
		0.73	0.68	-					
		0.87	0.85	-					
		1	0.9	-					
		1.07	0.98	-					
		1.3	1.09	-					
	Responses:								

Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
Aroclor 1221*	C12H8.8Cl1.2	0.21	-	-	-	C	C	6	1
		0.27	-	-					
		0.32	-	-					
		0.37	-	-					
		0.4	-	-					
		0.53	-	-					
		0.6	0.15	-					
		0.65	0.21	-					
		0.7	0.24	-					
		0.77	0.3	-					
		0.9	0.4	-					
		1.01	0.43	-					
		1.3	0.46	-					
		1.45	0.5	-					
		1.55	0.54	-					
		1.8	0.61	-					
		1.9	0.68	-					
		2.12	0.92	-					
		2.26	1.04	-					
		2.7	1.16	-					
		3.16	1.24	-					
	Responses: OV-101: TR40								
Aroclor 1242*	C12H7Cl3	0.4	0.24	-	-	C	C	6	1
		0.52	0.3	-					
		0.58	0.4	-					
		0.68	0.46	-					
		0.73	0.54	-					
		0.88	0.61	-					
		0.98	0.68	-					
		1.05	0.85	-					
		1.24	0.9	-					
		1.42	0.98	-					
		1.52	1.1	-					
		1.77	1.36	-					
		1.87	1.59	-					
		2.24	1.75	-					
		2.61	2.01	-					
	Responses: OV-101: TR50								

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Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
Aroclor 1248*	C12H6.1Cl3.9	0.52	0.4	-	-	C	C	6	1
		0.58	0.47	-					
		0.68	0.54	-					
		0.82	0.61	-					
		0.87	0.68	-					
		0.98	0.85	-					
		1.05	0.9	-					
		1.25	0.98	-					
		1.42	1.1	-					
		1.52	1.18	-					
		1.77	1.37	-					
		1.88	1.6	-					
		2.24	1.75	-					
		2.59	2.01	-					
		3.1	2.72	-					
	Responses: OV-101: TR50								
Aroclor 1254*	C12H5Cl5	-	-	0.35	-	C	C	6	1
		0.89	0.68	0.48					
		1	0.85	0.63					
		1.07	0.9	0.81					
		1.3	0.99	0.97					
		1.55	1.1	1.3					
		1.82	1.17	1.43					
		1.92	1.39	1.84					
		2.24	1.48	1.98					
		2.68	1.6	2.26					
		3.14	1.75	2.55					
		3.7	2.03	2.91					
		4.2	2.46	3.3					
		4.4	2.79	4.3					
		5	3.8	4.8					
		5.9	4.3	5.2					
	Responses: OV-101: TR30								
Aroclor 1260*	C12H3.7Cl6.3	-	1.09	-	-	C	C	6	1
		1.31	1.17	-					
		1.53	1.61	-					
		1.9	1.81	-					
		2.11	2.06	-					
		2.25	2.18	-					
		2.68	2.45	-					
		2.9	2.76	-					

Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
Aroclor 1260* (cont'd)		3.2	3.12	-					
		3.6	3.48	-					
		4.2	3.7	-					
		5	4.3	-					
		5.9	5.2	-					
		6.6	5.8	-					
		8	7.2	-					
		9.3	8.8	-					
	Responses: OV-101: TR20								
Aroclor 1262*	C12H3.3Cl6.7	1.29	-	-	-	C	C	6	1
		1.53	-	-					
		1.89	-	-					
		2.11	-	-					
		2.26	-	-					
		2.66	-	-					
		2.88	-	-					
		3.12	-	-					
		3.6	-	-					
		4.2	-	-					
		5	-	-					
		5.9	-	-					
		6.5	-	-					
		6.7	-	-					
		8	-	-					
		9.3	-	-					
	Responses: OV-101: TR20								
Aroclor 1268*	C12H1Cl9	3.8	-	-	-	C	-	6	-
		4.7	-	-					
		5.4	-	-					
		7.3	-	-					
		8.7	-	-					
		10	-	-					
		13	-	-					
		16.2	-	-					
	Responses: OV-101: NI40								
Aroclor 4465*	CHCl (MIX)	2.08	-	-	-	C	C	6	1
		2.22	-	-					
		2.67	-	-					
		2.88	-	-					
		3.11	-	-					

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Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
Aroclor 4465* (cont'd)		3.6	-	-					
		4.2	-	-					
		4.5	-	-					
		5	-	-					
		5.4	-	-					
		5.9	-	-					
		6.5	-	-					
		6.6	-	-					
		8	-	-					
		9.3	-	-					
		12.1	-	-					
	Responses: OV-101: TR40								
atrazine	C8H14ClN5	0.43	0.74	0.44	C	S #	NR	50	1-2-3
	Responses: OV-101: TI58/TR200 OV-17: NI20								
azafenidin	C15H13Cl2N3O2	14	-	-	V	-	-	-	-
	Responses: OV-101:NI(WB)90								
azinphos-ethyl	C12H16N3O3PS2	6.9	-	14.8	C	P	S	50	3
	Responses: OV-101: TI58/TR200 OV-17: FP(WB)26/NI20								
azinphos-methyl	C10H12N3O3PS2	5.2	-	11.8	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: TI30/TR50								
azinphos-methyl oxygen analog	C10H12N3O4PS	3.7	-	10.1	C	-	-	-	-
	Responses: OV-101: FP20 OV-17: FP(WB)42								
benazolin methyl ester	C9H6O3SNCl	0.99	-	-	-	-	-	-	-
	Responses: OV-101: NI1								
bendiocarb	C11H13NO4	0.32	-	-	C	-	-	-	-
	Responses: DEGS: NP13								
benfluralin	C13H16F3N3O4	0.37	0.28	0.18	C	C	C	6	2
	Responses: OV-101: HX(WB)1.5/NI(WB)2 OV-17: HX(WB)1 OV-225: NI2								
benodanil	C13H10INO	2.43	-	4.5	C	-	-	-	-
	Responses: OV-101: NP60								
benoxacor	C11H11Cl2NO2	0.64	1.06	0.7	C	P	C	15+50	2+3
	Responses: OV-101: NI1/NP6 OV-17: NI1/NP7 OV-225: NI2								
bensulide	C14H24NO4PS3	9.5	-	20.2	C	P	C	50	3
	Responses: OV-101: FP100/NI(WB)9/TI190 OV-17: FP100								

Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
benzoylprop-ethyl	C18H17Cl2NO3	4.3	8.4	6	P	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI(WB)3 OV-17: NI8 OV-225: NI6								
BHC, alpha-	C6H6Cl6	0.4	0.48	0.35	C	C	C	6	1
	Responses: OV-101: TR0.4 OV-17: NI0.3								
BHC, beta-	C6H6Cl6	0.43	1.62	0.56	C	C	C	6	1
	Responses: OV-101: TR2 OV-17: NI1								
BHC, delta-	C6H6Cl6	0.5	1.71	0.67	C	C	C	6+15	1
	Responses: OV-101: HX0.5/TR0.4 OV-17: NI0.5								
bifenox	C12H9Cl2NO5	5	14.9	8.8	C	C	P	15+50	2+3
	Responses: OV-101: HX16/NI4								
bifenthrin	C23H22ClF3O2	4.9	3.8	4.5	V	C	-	6+15	2
	Responses: OV-101: NI8 OV-17: HX5/HX(WB)20								
binapacryl	C15H18N2O6	2.19	4.2	2.38	C	P	P	15	-
	Responses: OV-101: NI(WB)1 OV-17: NI22/NP(WB)100								
bis(2-ethylhexyl) phthalate	C24H38O4	6.4	4.5	6.1	-	C	C	15+50	-
	Responses: OV-101: NI(V)200								
bis(trichloromethyl)disulfide	C2Cl6S2	0.19	-	-	-	R	-	6	-
	Responses:								
bitertanol*	C20H23N3O2	9.4	-	11.8	C	-	-	-	-
		9.7	-	12.5					
	Responses: OV-17: NP(WB)200								
bromacil	C9H13BrN2O2	0.8	4.8	1.36	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: HN(WB)2/NI(WB)2/NP(WB)17 OV-17: HN(WB)1/HX(WB)8/NI(WB)6/NP(WB)5 OV-225: NI(WB)12								
bromacil methyl ether	C10H16BrN2O2	0.8	2.1	-	-	-	-	-	-
	Responses: OV-101: HN(WB)1.2/HX(WB)90/NI(WB)1.5/NP(WB)10 OV-225: NI(WB)3.8								
bromofenoxim methyl ether	C14H9Br2O6N3	0.3	-	-	-	-	-	-	-
	Responses: OV-101: HN(WB)6/NI1								
bromophos	C8H8BrCl2O3PS	1.11	1.29	1.16	C	C	C	6	-
	Responses: OV-101: FP3/NI(WB)1/TI3 OV-17: FP3/NI2 OV-225: NI6								
bromophos-ethyl	C10H12BrCl2O3PS	1.51	1.42	1.45	C	C	P	6	-
	Responses: OV-101: FP3/NI3/TI4 OV-17: FP(WB)0.3								
bromopropylate	C17H16Br2O3	4.4	6.5	-	C	C #	C #	15+50	1-2-3
	Responses: OV-101: TR12								

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Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
bromoxynil butyrate	C11H9Br2NO2	0.78	-	-	-	V	-	15+50	2
	Responses: OV-101: NI0.5/NP7.5								
bromoxynil methyl ether	C8H5BR2ON	0.3	-	-	-	-	-	-	-
	Responses: OV-101: NI0.4								
bromoxynil octanoate	C15H17Br2NO2	3.14	-	-	-	V #	-	15+50	2
	Responses: OV-101: NI4/NP40								
BTS 27271-HCl	C10H14N2•HCl	-	-	-	-	-	-	-	-
	Responses:								
BTS 27919	C9H11NO	-	-	-	C	-	-	-	-
	Responses:								
bufencarb*	C13H19NO2	-	-	-	-	-	-	-	-
		-	-	-					
		-	-	-					
		-	-	-					
		-	-	-					
	Responses:								
Bulan	C16H15Cl2NO2	3.06	7.5	4.4	C	P	P	15	2
	Responses: OV-101: NI(WB)1 OV-17: NI5 OV-225: NI6								
bupirimate	C13H24N4SO3	2	3.7	2.6	C	-	-	-	-
	Responses: OV-101: FS(WB)20/NI(WB)8 OV-17: NP(WB)300								
butachlor	C17H26ClNO2	1.73	1.83	1.46	C	C	-	50	-
	Responses: OV-101: HX9 OV-17: HX9 OV-225: NI14								
butralin	C14H21N3O4	1.15	1.22	0.93	V	C	-	6+15+50	-
	Responses: OV-101: NI7/NP3 OV-17: NI6/NP15 OV-225: NI8								
butyl benzyl phthalate	C19H20O4	3.06	5.1	4.5	-	C	P	15+50	-
	Responses: OV-101: NI35								
butylate	C11H23NOS	0.22	-	-	-	-	-	-	-
	Responses:								
butylisodecyl phthalate	C22H34O4	-	-	0.82	-	-	-	-	-
	Responses:								
cadusafos	C10H23O2PS2	0.37	0.27	0.29	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: FP(WB)0.5 OV-17: FP(WB)0.4/NI(WB)12/NP(WB)0.5 OV-225: FP(WB)1								
captafol	C10H9Cl4NO2S	3.11	-	5.4	C	P	-	50	3
	Responses: OV-101: NI3 OV-17: NI5								

Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
captan	C9H8Cl3NO2S	1.2	3.49	1.85	C	P	C	50	3
	Responses: OV-101: TR2 OV-17: NI2								
carbaryl	C12H11NO2	0.75	-	1.05	C	-	-	-	-
	Responses: OV-101: NP60								
carbetamide	C12H16N2O3	0.96	-	1.32	-	-	-	-	-
	Responses:								
carbofuran	C12H15NO3	0.39	-	-	C	-	-	-	-
	Responses:								
carbofuran-3-keto-7-phenol	C10H10O3	-	0.24	-	-	-	-	-	-
	Responses:								
carbofuran-7-phenol-DNP ether	C16H14N2O6	-	18.1	-	-	-	-	-	-
	Responses:								
carbophenothion	C11H16ClO2PS3	2.94	4.2	3.7	C	C	P	6	2
	Responses: OV-101: TI15/TR4 OV-17: FP8								
carbophenothion oxygen analog	C11H16ClO3PS2	2.17	4.2	3.06	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI6/TI15 OV-17: FP15								
carbophenothion oxygen analog sulfone	C11H16ClO5PS2	3.8	-	7.1	-	-	-	-	-
	Responses: OV-101: NI36/TI35 OV-17: FP(WB)24								
carbophenothion oxygen analog sulfoxide	C11H16ClO4PS2	4.2	-	2.87	-	-	-	-	-
	Responses: OV-101: TI250 OV-17: FP15								
carbophenothion sulfone	C11H16ClO4PS3	5.1	-	9.2	C	C	P	6	1
	Responses: OV-101: FP3/TI20 OV-17: FP30								
carbophenothion sulfoxide	C11H16ClO3PS3	5.4	-	4	-	-	-	-	-
	Responses: OV-101: FP3/TI35 OV-17: FP20								
carbosulfan	C20H32N2O3S	5.4	-	5.3	P	-	-	-	-
	Responses: OV-101: NP20								
carboxin	C12H13NO2S	1.87	-	-	C	NR	NR	6-15-50	-
	Responses: OV-101: FS50								
carboxin sulfoxide	C12H13NO3S	0.13	0.23	0.11	-	NR	NR	6-15-50	1-2-3
	Responses: OV-101: FS10 OV-17: FS30 OV-225: FS25								
CGA 100255	C15H12NO5	1.8	-	2.96	S	-	-	-	-
	Responses: OV-101: NP100 OV-17: NI1000/NP150								

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Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
CGA 118244	C15H13Cl2N3O3	7	-	11.4	V	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI40								
CGA 120844	C8H9NSO3	0.6	2.65	0.9	-	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI200/NP200 OV-17: NI100/NP300 OV-225: NI300								
CGA 14128	C12H21N2O4PS	0.75	0.8	0.68	C		-	50	1-2-3
	Responses: OV-101: NI2/NP2 OV-17: NI0.6/NP2 OV-225: NI2 D								
CGA 150829	C5H14N4O	0.22	-	0.14	V	-	-	-	-
	Responses: OV-101: NP0.5 OV-17: NP1								
CGA 171683	C6H5F4N3O2	0.06	0.08	0.04	C		-	15+50	3
	Responses: OV-101: NI30 OV-17: NI10 OV-225: NI40								
CGA 189138	C13H8O3Cl2	1.39	1.89	1.54	-	-	-	-	-
	Responses: OV-101: NI1000 OV-17: NI1000 OV-225: NI1000								
CGA 205374	C16H11N3O2Cl2	12	8.9	6.1	-	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI50 OV-17: NI200 OV-225: NI500								
CGA 205375	C16H13N3O2Cl2	6.7	-	1.59	-	-	-	-	-
	Responses: OV-101: NI1000 OV-17: NI1000								
CGA 236431	C8H7F3N2O2	0.17	-	0.11	-	-	-	-	-
	Responses: OV-101: NP200 OV-17: NP20								
CGA 236432	C9H9F3N2O2	0.26	-	0.13	-	-	-	-	-
	Responses: OV-101: NP20 OV-17: NP8								
CGA 27092	C8H7F3N2O	-	-	0.62	-	-	-	-	-
	Responses: OV-17: NP50								
CGA 37734	C10H13NO2	0.4	-	0.47	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NP(V)20 OV-17: NP100								
CGA 51702	C9H9F3N2O	0.46	-	0.49	-	-	-	-	-
	Responses: OV-101: NP2 OV-17: NP3								
CGA 72903	C7H6F3N	0.22	-	0.14	-	-	-	-	-
	Responses: OV-101: NP100 OV-17: NP50								
CGA 91305	C10H8Cl2N3O	1.15	4.3	1.54	V	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI3								
CGA 94689A	C15H21NO5	1.53	6.5	2.41	V	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI5/NP150 OV-17: NI5/NP38 OV-225: NI5								

Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
CGA 94689B	C15H21NO5	1.54	6.6	2.45	S	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI12/NP150 OV-17: NI10/NP75 OV-225: NI8								
chloramben methyl ester	C8H7Cl2NO2	0.44	1.03	-	-	-	-	-	-
	Responses: OV-101: HX0.8/NI0.8								
chlorbenside	C13H10Cl2S	1.39	1.62	1.54	C	S	P	6	1
	Responses: OV-101: NI6 OV-17: HX3/NI1								
chlorbromuron	C9H10BrClN2O2	1.27	3.39	1.42	V	V	V	50	3
	Responses: OV-101: HX12/NI19								
chlorbufam	C11H10ClNO2	0.42	0.75	0.45	C		-	15	2+3
	Responses: OV-101: HX4 OV-17: HN(WB)0.4								
chlordane*	C10H6Cl8	0.45	0.16	-	C	C	C	6	1
		0.63	0.5	-					
		0.73	0.52	-					
		0.81	0.85	0.23					
		0.97	0.9	0.53					
		1.16	1.45	0.61					
		1.45	1.54	0.88					
		1.62	2.69	1.33					
		2.61	3.33	1.47					
	Responses: OV-101: NI(WB)5 OV-17: NI11 OV-225: NI4								
chlordane, cis-	C10H6Cl8	1.66	1.54	1.48	C	C	C	6	1
	Responses: OV-101: TR1 OV-17: NI0.8								
chlordane, trans-	C10H6Cl8	1.49	1.46	1.34	C	C	C	6	1
	Responses: OV-101: TR1 OV-17: NI0.6								
chlordecone	C10H8Cl10O5	2.75	1.67	2.38	-	S #	P #	15+50	1-2-3
	Responses: OV-101: NI(WB)2 OV-17: HX2/NI5 OV-225: NI6								
chlordene	C10H6Cl6	0.56	0.4	0.32	-	C	C	6	1
	Responses: OV-101: NI(WB)1 OV-17: NI0.4 OV-225: NI0.3								
chlordene epoxide	C10H6Cl6O	0.84	0.65	-	-	C	-	15	-
	Responses: OV-101: NI0.6								
chlordene, alpha-	C10H6Cl6	0.82	0.64	0.67	-	-	-	-	-
	Responses: OV-101: TR2 OV-17: NI0.6								
chlordene, beta-	C10H6Cl6	0.98	0.84	0.89	-	-	-	-	-
	Responses: OV-101: TR1 OV-17: NI1								

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Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
chlordene, gamma-	C10H6Cl6	0.98	0.89	0.88	-	-	-	-	-
	Responses: OV-101: TR2 OV-17: NI1 OV-225: NI1								
chlorethoxyfos	C6H11Cl4O3PS	0.33	0.23	0.24	V	C	-	6	1
	Responses: OV-101: FP0.5/HX0.3/NI0.5 OV-17: FP0.5/HX0.3/NI0.3 OV-225: FP0.5/NI0.3								
chlorfenapyr (prop)	C15H11BrClF3N2O	2.21	-	2.34	P	-	S	50	2
	Responses: OV-101: NI2/NP50 OV-17: NI2/NP50 DEGS: NI4/NP190								
chlorfenvinphos, alpha-	C12H14Cl3O4P	1.21	1.58	1.29	C	-	NR	6-15-50	-
	Responses: OV-101: FP10/FP(WB)1.7/NI(WB)2/TI4 OV-17: FP(WB)2/NI3 OV-225: NI5								
chlorfenvinphos, beta-	C12H14Cl3O4P	1.29	2	1.52	C	S #	-	50	1-2-3
	Responses: OV-101: FP2/FP(WB)1.8/HX3/NI(WB)2/TI4 OV-17: FP4/FP(WB)2/NI3 OV-225: FP4/NI5								
chlorflurecol methyl ester	C15H11ClO3	1.73	-	1.88	C	-	-	-	-
	Responses: OV-101: HX8/NI3								
chlorimuron ethyl ester	C15H15ClN4O6S	0.13	0.15	0.1	P	NR	-	-	-
	Responses: OV-101: NI14/NI(WB)24/NP35 OV-17: NI(WB)1.4/NP23								
chlormephos	C5H12ClO2PS2	-	-	0.11	C	-	-	-	-
	Responses: OV-17: FP0.4								
chlornitrofen	C12H6Cl3NO3	2.85	4.7	-	C	C	C	6+15	2
	Responses: OV-101: TR5								
chlorobenzilate	C16H14Cl2O3	2.31	3.26	2.61	C	C #	P #	15+50	3
	Responses: OV-101: TR70 OV-17: NI15								
chloroneb	C8H8Cl2O2	0.19	0.19	-	C	C	-	6	2
	Responses: OV-101: NI3.5								
chloropropylate	C17H16Cl2O3	2.33	2.9	2.41	P	C	C	15+50	3
	Responses: OV-101: TR80 OV-17: NI15								
chlorothalonil	C8Cl4N2	0.55	1.44	0.74	S	C #	C #	6-15-50	2+3
	Responses: OV-101: HX1/NI0.6 OV-17: HX1/NI2								
chlorothalonil trichloro impurity	C8HCl3N2	0.32	-	-	R	R #	NR	6-15-50	2+3
	Responses:								
chloroxuron	C15H15ClN2O2	0.81	0.85	-	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: HX16/NI300								
chlorpropham	C10H12ClNO2	0.32	0.43	0.25	C	C	C	15	2
	Responses: OV-101: HX2 OV-17: NI80								

Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
chlorpyrifos	C9H11Cl3NO3PS	1	1	1	C	C	P	6	2
	Responses: OV-101: NI1.5/TI3 OV-17: FP2/NI2								
chlorpyrifos oxygen analog	C9H11Cl3NO4P	0.95	1.51	1.08	C	NR	-	6-15-50	-
	Responses: OV-101: FP27/NI6/TI8								
chlorpyrifos-methyl	C7H7Cl3NO3PS	0.72	0.86	0.79	C	C	-	6	2
	Responses: OV-101: FP1/FP(WB)1.3/HX1.5/NI1/NP1 OV-17: FP(WB)1.5								
chlorsulfuron	C12H12ClN5O4S	1.3	8.9	-	-	NR	NR	6-15-50	-
	Responses: OV-101: NI50								
chlorthiamid	C7H5Cl2NS	0.69	-	-	-	-	-	-	-
	Responses:								
chlorthiophos oxygen analog	C11H15Cl2O4PS	2.22	4.1	2.99	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: HX9/NI6 OV-17: FP10/HX11 OV-225: FP6								
chlorthiophos sulfone	C11H15Cl2O5PS2	5.3	18.8	9.1	C	C	-	50	3
	Responses: OV-101: HX20/NI9 OV-17: FP100/HX22 OV-225: FP39								
chlorthiophos sulfoxide	C11H15Cl2O4PS2	4.7	10.3	6.9	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: HX20/NI6 OV-17: FP25/HX17 OV-225: FP15								
chlorthiophos*	C11H15Cl2O3PS2	2.24	-	2.58	C	C	C	6	2
		2.36	-	2.77					
		2.56	-	3.16					
	Responses: OV-101: FP8 OV-17: FP5								
CL 202,347	C13H19N3O5	2.96	11.5	4.1	-	-	-	-	-
	Responses: OV-101: NI15/NP50 OV-17: NI20/NP100 OV-225: NI60								
clodinafop-propargyl	C17H13ClFNO4	3.26	5.8	4.67	V	V	-	50	3
	Responses: OV-101:NP(WB)30 OV-17:NI(WB)20 OV-225:NI(WB)5								
clofentezine	C14H8Cl2N4	5.9	-	9.8	R	S	-	15	2
	Responses: OV-101: HN(WB)10.5/HX20/NI100 OV-17: NP165								
clomazone	C12H14ClNO2	0.45	0.59	0.46	C		-	50	3
	Responses: OV-101: HX1.5/HX2/NI110 OV-17: HX2/NP11 OV-225: NI150								
clopyralid methyl ester	C7H4Cl2NO2	0.18	-	-	-		-	50	-
	Responses: OV-101: NI0.25								
cloquintocet-mexyl	C18H22ClNO3	4.8	6.6	6.3	V	NR	-	6-15-50	1-2-3
	Responses: OV-101:NP(WB)40 OV-17:NI(WB)20 OV-225:NI(WB)5								

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Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
Compound K*	C10H6Cl8	0.83	-	-	-	C	-	-	1
		2.53	2.66	-					
	Responses: OV-101: TR5								
coumaphos	C14H16ClO5PS	9	40	18	C	NR	C #	6-15-50	3
	Responses: OV-101: NI39/NP38 OV-17: FP50/FP(WB)26/NI38/NP34 OV-225: NI100								
coumaphos oxygen analog	C14H16ClO6P	8	45	16	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI200/NP130 OV-17: FP75/NI50/NP40 OV-225: NI150								
CP 108064, methylated	C15H21NO4	0.73	-	0.67	-	-	-	-	-
	Responses: OV-101: NP6 OV-17: NP6								
CP 51214	C14H21NO3	0.7	-	0.58	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NP13 OV-17: NP24								
crotoxyphos	C14H19O6P	1.37	2.85	1.9	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI60/TI10 OV-17: FP10/FP(WB)3								
crufomate	C12H19ClNO3P	1.08	2.33	1.3	C	NR	NR	6-15-50	-
	Responses: OV-101: TI6 OV-17: FP2/NI3								
cyanazine	C9H13ClN6	0.89	4.9	1.48	C	NR	-	6-15-50	-
	Responses: OV-101: NI4/TI26 OV-17: HX6								
cyanofenphos	C15H14NO2PS	3.1	8.2	4.6	C	-	-	-	-
	Responses: OV-101: FP3.5/NI3/NP(WB)3								
cyanophos	C9H10O3NSP	0.47	-	0.59	C	-	-	-	-
	Responses: OV-101: FP(WB)0.7/NI(WB)2/NP1 OV-17: FP(WB)0.7/NP(WB)1								
cyclanilide methyl ester	C12H11Cl2NO3	1.57	1.84	1.64	-	-	-	-	-
	Responses: OV-101: NI5/NP30 OV-17: NI6/NP30 OV-225: NI6								
cycloate	C11H21NOS	0.3	-	-	C	V #	S	15+50	3
	Responses: OV-101: FS2/NP15								
cyfluthrin*	C22H18Cl2FNO3	11.7	-	-	C	P	-	15	-
		12.5	-	-					
		12.8	-	-					
	Responses: OV-101: HX30/NI30								
cymiazole	C12H14N2S	0.73	-	0.89	-	-	-	-	-
	Responses: OV-101: NP2 OV-17: NP(WB)2								
cymoxanil	C7H10N4O3	0.25	0.5	0.16	V	NR	NR	6-15-50	1-2-3
	Responses: OV-101: HN(WB)1/NI(WB)23/NP(WB)10 OV-17: HN(WB)3/NI(WB)120/NP(WB)7 OV-225: NI(WB)70								

Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
cypermethrin*	C22H19Cl2NO3	-	29	-	C	C	C	15	2
		14.1	33	23					
		15.1	36	25					
	Responses: OV-101: NI90								
cyprazine	C9H14ClN5	0.64	1.22	0.74	C	-	-	-	-
	Responses: OV-101: HX1.5/NI13								
cyproconazole	C15H18ClN3O	2.04	1.61	2.69	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: HN(WB)182/HX(WB)73/NI(WB)72/NP(WB)12								
cyprodinil	C14H15N3	1.18	-	1.39	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NP(WB)2 OV-17: NP(WB)10								
cyromazine	C6H10N6	0.58	-	0.68	S	-	-	-	-
	Responses: OV-101: NP10 OV-17: NP2								
dazomet	C5H10N2S2	0.4	-	0.71	S	NR	-	6-15-50	1-2-3
	Responses: OV-101: HX500/NI300 OV-17: FS(WB)80/HN(WB)0.4/HX500/NI300								
DCPA	C10H6Cl4O4	1.06	1.13	1	C	C	C	15	2
	Responses: OV-101: NI1 OV-17: NI1 OV-225: NI1								
DDE, o,p'-	C14H8Cl4	1.55	1.28	1.51	C	C	C	6	1
	Responses: OV-101: TR2 OV-17: NI1								
DDE, p,p'-	C14H8Cl4	1.92	1.59	1.86	C	C	C	6	1
	Responses: OV-101: NI1.5 OV-17: NI1								
DDM	C13H10Cl2	0.72	-	-	-	-	-	-	-
	Responses:								
DDMS	C14H11Cl3	1.65	-	1.65	-	R	-	6	-
	Responses:								
DDMU	C14H9Cl3	1.47	-	-	-	-	-	-	-
	Responses:								
DDNS	C14H12Cl2	0.83	-	-	-	-	-	-	-
	Responses:								
DDNU	C14H10Cl2	0.83	-	-	-	-	-	-	-
	Responses:								
DDT, o,p'-	C14H9Cl5	2.55	2.27	2.7	C	C	C	6	1
	Responses: OV-101: TR4 OV-17: NI2								

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Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
DDT, p,p'-	C14H9Cl5	3.13	3.6	3.5	C	C	C	6	1
Responses: OV-101: TR4 OV-17: NI2									
deltamethrin*	C22H19Br2NO3	17.1	-	21	C	S #	P	15	2
		27	-	35					
		29	19.9	38					
Responses: OV-101: NI1300									
deltamethrin, trans-*	C22H19Br2NO3	17	-	6.2	-	P #	NR	15	2
		29	-	20					
		31	19.7	38					
Responses: OV-101: NI200									
demeton-O oxygen analog	C8H19O4PS	0.22	0.32	0.21	-	-	-	-	-
Responses: OV-101: TI6 OV-17: FP25									
demeton-O sulfone*	C8H19O5PS2	-	-	0.28	C	-	-	-	-
		0.71	2.95	0.96					
Responses: OV-101: FP5/TI12 OV-17: FP3									
demeton-O sulfoxide	C8H15O4PS2	0.87	-	1.05	C	-	-	-	-
Responses: OV-101: FP4									
demeton-O*	C8H19O3PS2	-	-	0.2	C	NR	-	6-15	-
		0.28	-	0.36					
Responses: OV-101: FP(WB)2 OV-17: FP2									
demeton-S	C8H19O3PS2	0.41	0.56	0.41	C	NR	-	6-15-50	-
Responses: OV-101: FP(WB)0.8/TI2 OV-17: FP0.8/FP(WB)0.8									
demeton-S sulfone	C8H19O5PS2	1.15	5.8	1.75	C	-	-	-	-
Responses: OV-101: FP40/TI20 OV-17: FP5 OV-225: FP60									
demeton-S sulfoxide	C8H19O4PS2	-	-	-	C	-	-	-	-
Responses: DEGS: FP30									
des N-isopropyl isofenphos	C12H18NO4PS	1.21	2.73	1.5	C	S	-	50	-
Responses: OV-101: FP2 OV-17: FP3									
des N-isopropyl isofenphos oxygen analog	C12H18NO5P	0.93	-	1.43	-	-	-	-	-
Responses: OV-101: FP(WB)5 OV-17: FP(WB)12									
desdiethyl simazine	C3H4ClN5	0.2	0.86	0.61	-	NR	NR	6-15-50	1-2-3
Responses: OV-101: HX25/NI20 OV-17: HN(WB)0.1/HX25 OV-225: NI20									
desethyl simazine	C5H8ClN5	0.3	0.8	0.53	-	NR	NR	50	1-2-3
Responses: OV-101: HX12/NI20 OV-17: HN(WB)0.1/HX12 OV-225: NI80									

Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
desisopropyl iprodione	C10H6Cl2N3O3	2.31	15	3.93	P		-	50	1-2-3
	Responses: OV-101: NI20/NP50								
desmedipham	C16H16N2O4	0.44	0.29	0.45	-	-	-	-	-
	Responses: OV-101: NI1000/NP300 OV-17: NI1000/NP218 OV-225: NI1000								
desmethyl diphenamid	C15H15NO	0.98	-	-	-	-	-	-	-
	Responses: OV-101: TI340								
desmethyl norflurazon	C11H7ClF3N3O	3.38	1.41	4.9	V	NR	NR	6-15-50	1-2-3
	Responses: OV-101: HX(WB)3/NI27 OV-17: HX(WB)3 OV-225: NI200								
di-allate	C10H17ClNOS	0.42	0.26	0.33	C	C	-	6	-
	Responses:								
di-n-octyl phthalate	C24H38O4	12	-	-	-	C	C	15+50	-
	Responses: OV-101: NI(V)330								
dialifor	C14H17ClNO4PS2	6.5	-	14.3	C	C	P	15	2
	Responses: OV-101: TI30/TR28 OV-17: FP25/FP(WB)31								
diazinon	C12H21N2O3PS	0.51	0.4	0.44	C	C	C	15	3
	Responses: OV-101: FP(WB)1/NI3/NP0.4 OV-17: FP0.7/FP(WB)0.9/NI4/NP0.25 OV-225: FP6/NI4.5								
diazinon oxygen analog	C12H21N2O4P	0.5	0.53	0.47	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI18/NP0.6 OV-17: NI30/NP0.6 OV-225: NI60								
dibromochloropropane	C3H5Br2Cl	0.04	-	0.03	-	-	-	-	-
	Responses: OV-101: TR0.6 OV-17: NI0.2								
dibutyl phthalate	C16H22O4	0.88	0.92	0.84	-	C	C	15+50	-
	Responses: OV-101: NI30								
dicamba methyl ester	C8H6Cl2O3	0.19	0.18	-	-	-	-	-	-
	Responses: OV-101: HX(WB)1.6/NI0.6								
dichlobenil	C7H3Cl2N	0.11	-	0.1	C	P	C	15	2
	Responses: OV-101: TR0.5 OV-17: NI0.6								
dichlofenthion	C10H13Cl2O3PS	0.67	0.64	0.56	C	C	V	6	2
	Responses: OV-101: FP1/FP(WB)3.5/NI1.9/TI2 OV-17: FP0.8/HX(WB)2								
dichlofluanid	C9H11Cl2FN2O2S2	0.9	1.71	1.01	C	C #	-	15+50	2+3
	Responses: OV-101: NI1/NP44								
dichlone	C10H4Cl2O2	0.55	0.92	-	P	S #	S #	6-15-50	2+3
	Responses: OV-101: NI2								

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Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
dichlorobenzene, p-	C6H4Cl2	0.03	-	0.02	-	C	C	6	1
	Responses: OV-101: TR37 OV-17: NI4								
dichlorobenzophenone, o,p'-	C13H8Cl2O	0.82	1.07	0.92	-	C	C	15	2
	Responses: OV-101: TR4 OV-17: NI2								
dichlorobenzophenone, p,p'-	C13H8Cl2O	0.99	1.25	1.08	-	C	C	15	2
	Responses: OV-101: TR3 OV-17: NI2								
dichlorprop methyl ester	C10H10Cl2O3	0.28	-	-	-	-	-	-	-
	Responses: OV-101: HX(WB)1.6/NI2								
dichlorvos	C4H7Cl2O4P	0.07	0.08	0.08	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: FP9/NI1/TI0.5 OV-17: FP2.5 OV-225: FP(WB)0.7								
diclobutrazol	C15H19Cl2N3O	2.02	3.4	2.03	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: HX7/NI7/NP(WB)8 OV-17: HN(WB)1.3/HX7/HX4/HX(WB)2/NI(WB)4/NP(WB)8 OV-225: NI7								
diclofop-methyl	C16H14Cl2O4	3.57	4.9	4.7	C	C	C	15	2
	Responses: OV-101: HX8/NI10 OV-17: HX10 OV-225: NI12								
dicloran	C6H4Cl2N2O2	0.42	0.96	0.45	C	S	P	15+50	2+3
	Responses: OV-101: TR0.5 OV-17: NI0.4								
dicofol, o,p'-*	C14H9Cl5O	0.86	-	-	C	V	S	6+15	2
		4.1	1.08	0.91					
	Responses: OV-101: NI5 OV-17: HX2								
dicofol, p,p'-*	C14H9Cl5O	1.04	-	-	C	V	P #	6+15	1+2
		4.4	1.28	1.08					
	Responses: OV-101: NI5 OV-17: HX3								
dicrotophos	C8H16NO5P	0.31	0.96	0.43	C	NR	-	6-15-50	-
	Responses: OV-101: FP(WB)0.6/TI10 OV-17: FP1/FP(WB)0.8								
dieldrin	C12H8Cl6O	1.91	1.87	1.84	C	C	C	15	2
	Responses: OV-101: HX1/NI1.5 OV-17: HX1.5/NI1								
diethatyl-ethyl	C16H22ClNO3	1.78	3.14	2	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: HX11/NI10/NP180 OV-17: NI11/NP200 OV-225: NI14								
diethyl phthalate	C12H14O4	0.26	-	-	-	P	P	15+50	-
	Responses: OV-101: NI3500								
difenoxuron	C16H18N2O3	0.97	-	0.96	-	-	-	-	-
	Responses: OV-101: HN(WB)5 OV-17: NP16								

Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
diisobutyl phthalate	C16H2204	0.65	0.61	0.56	-	P	-	15+50	-
	Responses: OV-101: NI20								
diisohexyl phthalate*	C20H30O4	2.45	-	-	-	C	-	15+50	-
		2.66	-	-					
		2.9	-	-					
		3.27	-	-					
	Responses: OV-101: TR340								
diisooctyl phthalate*	C24H38O4	0.91	-	-	-	C	C	15+50	-
		5.5	-	-					
		6.2	-	-					
		6.7	-	-					
		7.5	-	-					
		9	-	-					
		10.5	-	-					
	Responses: OV-101: TR850								
Dilan*	C15.5H14Cl2NO2	-	5.3	-	-	P	P	15	-
		-	4.8	-					
		2.33	5.8	-					
		2.81	7.5	-					
		3.39	8.2	-					
	Responses: OV-101: TR8								
dimethachlor	C13H18ClNO2	0.71	1.11	0.71	C	-	-	-	-
	Responses: OV-101: NI30 OV-17: NI10 OV-225: NI20								
dimethametryn	C11H21N5S	-	-	-	C	-	-	-	-
	Responses:								
dimethenamid	C12H18ClNO2S	0.72	0.98	-	-	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI(WB)19/NP(WB)14								
dimethipin	C6H10O4S2	0.41	2.71	0.81	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: FS0.5 OV-17: FS1.5 OV-225: FS2								
dimethoate	C5H12NO3PS2	0.4	1.6	0.62	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: FP(WB)0.7/NI(WB)6/NP1 OV-17: FP0.8/FP(WB)0.8/NI5								
dimethyl phthalate	C10H10O4	0.15	0.15	0.14	-	P	-	6+15+50	-
	Responses: OV-101: NI300								
dinitramine	C11H13F3N4O4	0.52	0.93	0.44	C	-	P	15	-
	Responses: OV-101: NI(WB)1/TI166 OV-17: NI1 OV-225: NI1								

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Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
dinobuton	C14H18N2O7	1.4	-	1.32	C	-	-	-	-
	Responses: OV-101: HN(WB)1.5 OV-17: HN(WB)1/NP(WB)50								
dinocap*	C18H24N2O6	-	-	3.5	C	P	P	15	2
		4	-	3.9					
		4.3	6.9	4.4					
		4.8	7.7	4.8					
		5.1	9.5	5.6					
	Responses: OV-101: NI(WB)6 OV-17: NI150								
dinoseb methyl ether	C11H14N2O5	0.63	-	-	-	-	-	-	-
	Responses: OV-101: HN1/NI1.2								
dioxabenzofos	C8H9O3PS	0.34	-	0.36	C	P	-	15	-
	Responses: OV-17: FP0.7								
dioxacarb	C11H13NO4	-	-	-	C	-	-	-	-
	Responses:								
dioxathion	C12H26O6P2S4	0.47	-	0.5	V	NR	-	6-15-50	2
	Responses: OV-101: NI100/TI10 OV-17: FP7/FP(WB)13								
diphenamid	C16H17NO	1.1	-	1.55	V	NR	-	6-15	-
	Responses: OV-17: NP(WB)25								
diphenylamine	C12H11N	0.29	-	0.25	C	S	-	6+15	-
	Responses:								
disul-Na	C8H7Cl2O5S•Na	0.23	-	-	-	-	-	-	-
	Responses: OV-101: NI3								
disulfoton	C8H19O2PS3	0.54	0.6	0.46	C	P #	NR	6	1-2-3
	Responses: OV-101: TI2 OV-17: FP1								
disulfoton sulfone	C8H19O4PS3	1.5	6.7	2.39	C	NR	-	6-15-50	-
	Responses: OV-101: TI7 OV-17: FP7								
disulfoton sulfoxide	C8H1903PS3	-	-	-	C	-	-	-	-
	Responses:								
dithianon	C14H4O2N2S2	4.7	53	11.3	NR	-	-	-	-
	Responses: OV-101: NI(WB)12 OV-17: NP(WB)120								
diuron	C9H10Cl2N2O	0.11	0.09	0.11	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI(WB)9 OV-17: NI12 OV-225: NI27								
DNOC methyl ether	C8H8N2O5	0.35	-	-	-	-	-	-	-
	Responses: OV-101: HN(WB)30								

Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
edifenphos	C14H15O2PS2	2.87	6.3	5.3	C	-	-	-	-
	Responses: OV-101: FP(WB)4/NI(WB)4 OV-17: FP(WB)8								
endosulfan I	C9H6Cl6O3S	1.64	1.38	1.47	C	C	C	15	2
	Responses: OV-101: HX1/NI1.3 OV-17: HX1/NI2								
endosulfan II	C9H6Cl6O3S	2.21	3.9	2.77	C	C	C	15+50	2
	Responses: OV-101: HX2/NI2 OV-17: HX3/NI2								
endosulfan sulfate	C9H6Cl6O4S	2.83	8.3	4	C	C	C	50	2
	Responses: OV-101: HX4/TR5 OV-17: HX6/NI6								
endrin	C12H8Cl6O	2.13	2.22	2.29	C	C #	C #	15	2
	Responses: OV-101: TR2 OV-17: NI2								
endrin alcohol	C12H8Cl6O	2.55	-	-	-	P	C	15+50	2+3
	Responses: OV-101: TR4								
endrin aldehyde	C12H8Cl6O	2.35	-	-	C	P	C	15+50	-
	Responses: OV-101: TR4								
endrin ketone	C12H8Cl6O	3.6	10.3	-	-	C	C	50	2
	Responses: OV-101: TR5								
EPN	C14H14NO4PS	4.5	10.6	6.9	C	C	C	15	2
	Responses: OV-101: NI0.5/TI16 OV-17: FP50/NI9								
epoxyhexachloronorbornene	C7H2Cl6O	-	-	0.2	-	-	-	-	-
	Responses:								
EPTC	C9H19NOS	0.12	-	-	-	P	-	15	-
	Responses: OV-101: TI30								
esfenvalerate	C25H22ClNO3	22.5	-	-	C	C	C	15	2
	Responses: OV-101: NI90								
etaconazole*	C14H15Cl2N3O2	2.36	-	-	C	-	-	-	-
		2.43	-	3.17					
	Responses: OV-101: NP(WB)12 OV-17: HX7								
ethalfluralin	C13H14F3N3O4	0.34	0.27	0.19	C	C	C	6	2
	Responses: OV-101: HX4/HX(WB)7/NI1 OV-17: HX6 OV-225: NI0.4								
ethametsulfuron methyl ester*	C15H18N6O6S	0.35	2.85	0.4	-	NR	NR	6-15-50	1-2-3
		0.55	3.6	0.95					
	Responses: OV-101: HN(WB)17/NI(WB)34/NP(WB)600 OV-17: HN(WB)30 OV-225: NI(WB)780								

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Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
ethephon	C2H6ClO3P	3.03	2.74	2.88	NR		-	6+15+50	1+2+3
	Responses: OV-101: NI170								
ethiofencarb	C10H15NO2S	0.6	1.4	0.78	C	NR	NR	6-15-50	-
	Responses: OV-101: FP20/FP400/FS1.5/HN(WB)3.5/NI950/NP(WB)4 OV-17: NP133								
ethiolate	C7H15NOS	0.06	-	-	C	-	-	-	-
	Responses: OV-101: FS9								
ethion	C9H22O4P2S4	2.56	3.93	3.36	C	C	C	6	2
	Responses: OV-101: FP(WB)1.9/NI(WB)3/NP2 OV-17: FP4/FP(WB)2.3/NI8 OV-225: FP2/NI8								
ethion oxygen analog	C9H22O5P2S3	1.88	4.1	-	C	-	-	-	-
	Responses: OV-101: NP1								
ethofumesate	C13H18O5S	0.86	1.93	1.02	C	-	-	-	-
	Responses: OV-101: FS32/NI315 OV-17: FS65/NI333 OV-225: FS56/NI638								
ethoprop	C8H19O2PS2	0.33	0.31	0.25	C	P #	S #	50	1-2-3
	Responses: OV-101: FP0.7/TI0.8 OV-17: FP1								
ethoxyquin	C14H19N0	0.6	1.64	0.7	C	NR	NR	6-15-50	-
	Responses: OV-101: NI20/NP15 OV-17: NI12/NP15 OV-225: NI30								
ethyl p-toluene sulfonamide	C9H13NO2S	-	-	-	C	-	-	-	-
	Responses:								
ethylenethiourea	C3H6N2S	0.5	2.33	0.66	S	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI2200/NP23 OV-17: NI4500/NP64 OV-225: NI6250								
etridiazole	C5H5Cl3N2OS	0.18	0.12	0.21	C	C	P	6	2
	Responses: OV-101: NI0.3/NP0.6 OV-17: HX(WB)0.8/NI0.4/NP0.5 OV-225: NI0.2								
etrimfos	C10H17N2O4PS	0.58	0.59	0.51	C	C	C	15	2+3
	Responses: OV-101: FP2/NI50 OV-17: NI50 OV-225: NI30								
etrimfos oxygen analog	C10H17N2O5P	0.51	0.8	0.63	C	-	-	-	-
	Responses: OV-101: FP7/NI1000/NP(WB)3.5								
famphur	C10H16NO5PS2	2.65	14	5	C	NR	-	6-15-50	-
	Responses: OV-101: FP8/TI40 OV-17: FP50/FP(WB)7								
famphur oxygen analog	C10H16NO6PS	2.26	-	4.4	C	-	-	-	-
	Responses: OV-101: FP54 OV-17: FP(WB)12								
fenac	C8H5Cl3O2	1.42	3.7	-	-	NR	NR	6-15-50	-
	Responses: OV-101: NI2800								

Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
fenac methyl ester	C9H7Cl3O2	0.32	-	-	-	-	-	-	-
	Responses: OV-101: NI0.4								
fenamiphos	C13H22NO3PS	1.66	3.7	2.41	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: FP8/NP8 OV-17: FP10/NP3 OV-225: NP6								
fenamiphos sulfone	C13H22NO5PS	4.5	-	8.4	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: FP50/NP20 OV-17: FP(WB)20/NP60								
fenamiphos sulfoxide	C13H22N04PS	5.2	-	8.1	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: FP50/NP55 OV-17: FP(WB)28/NP45								
fenarimol	C17H12Cl2N2O	6.6	-	10.1	C	P #	C #	50	3
	Responses: OV-101: HX10/NI5								
fenarimol metabolite B	C17H14N2OCl2	4.6	-	-	NR	NR	NR	6-15-50	-
	Responses: OV-101: HX19								
fenarimol metabolite C	C17H14N2OCl2	4.6	-	-	S		-	6	-
	Responses: OV-101: HX8								
fenbuconazole	C19H17ClN4	9.8	-	-	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI1000/NP70								
fenfuram	C12H11NO2	0.54	1.47	0.62	C	-	-	-	-
	Responses: OV-101: NP5/NP(WB)6								
fenhexamid	C14H17Cl2NO2	3.1	-	3.7	NR	NR	NR	6-15-50	1-2-3
	Responses:OV-101:NI(WB)2/NP(WB)220 OV-17:NI(WB)5								
fenitrothion	C9H12NO5PS	0.84	1.82	1.05	C	C	C	15	2
	Responses: OV-101: FP(WB)1/NP1 OV-17: FP3/FP(WB)1.1								
fenitrothion oxygen analog	C9H12NO6P	0.72	-	0.83	C	-	-	-	-
	Responses: OV-101: FP3 OV-17: FP10								
fenoxaprop ethyl ester	C18H16NO5Cl	8.1	11.3	10.5	S	V	V	50	3
	Responses: OV-101: NI250								
fenoxycarb	C17H19NO4	5	-	7.3	C	-	-	-	-
	Responses: OV-101: NP50 OV-17: NP50								
fenpropathrin	C22H23NO3	4.8	7	5.7	-	V #	V	15	2
	Responses: OV-101: NI7/NI(WB)0.2/NP30 OV-17: NI13 OV-225: NI10								
fenpropimorph	C20H33NO	1.09	-	0.62	C		-	50	1-2-3
	Responses: OV-101: NP33 OV-17: NP14								

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Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
fenson	C12H10O3ClS	-	2.03	-	-	-	-	-	-
	Responses: OV-225: NI1.5								
fensulfothion	C11H17O4PS2	2.4	-	3.8	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: TI12 OV-17: FP6/FP(WB)7								
fensulfothion oxygen analog	C11H17O5PS	2.49	-	5	C	NR	-	6-15-50	-
	Responses: OV-101: TI10 OV-17: FP5								
fensulfothion oxygen analog sulfone	C11H17O7PS2	1.99	-	3.8	-	-	-	-	-
	Responses: OV-101: TI45 OV-17: FP6								
fensulfothion sulfone	C11H17O5PS2	2.8	-	3.6	C	NR	-	6-15-50	-
	Responses: OV-101: NI9/NP7 OV-17: FP100								
fenthion	C10H15O3PS2	0.96	1.46	1.18	C	S #	NR	6+15	1-2-3
	Responses: OV-101: FP2/TI4 OV-17: FP3								
fenthion oxygen analog	C10H15O4PS	0.78	-	1.12	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: FP7 OV-17: FP9								
fenthion oxygen analog sulfone*	C10H15O6PS2	1.77	-	-	-	-	-	-	-
		2.29	-	4.1					
	Responses: OV-101: FP(WB)4 OV-17: FP(WB)8								
fenthion oxygen analog sulfoxide	C10H15O5PS	0.43	-	0.62	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: FP100 OV-17: FP135								
fenthion sulfone	C10H15O5PS2	2.39	-	4.7	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: FP22 OV-17: FP20								
fenvalerate*	C25H22ClNO3	20.3	44	35	C	C	C	15	2
		22.5	51	40					
	Responses: OV-101: NI90								
fipronil	C12H4Cl2F6N4OS	1.35	8.7	1.16	S	S	V	50	3
	Responses: OV-101: NI2/NP10 OV-17: NI1/NP5 OV-225: NI10								
flamprop-M-isopropyl	C19H19ClFNO3	2.46	-	2.81	C	-	-	-	-
	Responses: OV-101: HX9/NI9								
flamprop-methyl	C17H15ClFNO3	1.94	-	2.45	C	-	-	-	-
	Responses: OV-101: HX8/NI7								
fluazifop butyl ester	C19H20F3NO4	2.3	2.36	2.31	C	C	V	15	3
	Responses: OV-101: HX19/HX(WB)18/NI125 OV-17: HX40 OV-225: NI3000								
fluchloralin	C12H13ClF3N3O4	0.53	0.76	0.37	C	C	-	6	2
	Responses: OV-101: HX3/NI1.5 OV-17: HX3/NI0.5								

Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
flucythrinate*	C26H23F2NO4	14.7 16.1	36.9 42	21.4 24	C	C	-	15	2+3
	Responses: OV-101: NI40/NI(WB)15								
flumetsulam, methylated	C13H11F2N5O2S	12.4	-	-	-	-	-	-	-
	Responses: OV-101: NP(WB)14								
fluometuron	C10H11F3N2O	-	-	0.14	-	-	-	-	-
	Responses: OV-17: NP2								
fluridone	C19H14F3NO	16.3	24	-	-	NR	NR	6-15-50	-
	Responses: OV-101: HX400/NI1500								
fluroxypyr, methylated*	C8H7O3N2Cl2F	0.61 0.79	- -	- -	-	-	-	-	-
	Responses: OV-101: NI2								
flusilazole	C16H15F2N3Si	1.97	-	2.33	C	-	-	-	-
	Responses: OV-101: NP(WB)5 OV-17: HX24/HX(WB)5/NP(WB)6								
fluvalinate*	C26H22ClF3N2O3	- 25	56 59	35 38	C	C	-	15	2
	Responses:								
FMTU	C10H9F3N2O2	-	-	-	-	-	-	-	-
	Responses:								
folpet	C9H4Cl3O2NS	1.23	3.01	1.94	C	C	P	15+50	2+3
	Responses: OV-101: NI(WB)1 OV-17: NI9								
fonofos	C10H15OPS2	0.52	0.56	0.44	C	C	C	6	2+3
	Responses: OV-101: TI2 OV-17: FP0.7								
fonofos oxygen analog	C10H15O2PS	0.39	0.53	0.38	V	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NP1 OV-17: FP4/FP(WB)1								
formetanate hydrochloride	C11H16ClN3O2	0.9	-	0.45	-	-	-	-	-
	Responses: OV-101: NP400 OV-17: NP430								
formothion	C6H12NO4PS2	-	-	0.91	C	NR	NR	6-15-50	1-2-3
	Responses: OV-17: FP(WB)10								
fosthiazate	C9H18NO3PS2	1.08	3	1.66	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: FP100/NI1000/NP40 OV-17: FP10/NI120/NP7 OV-225: FP15/NI300								
fuberidazole	C11H8N2O	0.71	-	0.95	C	-	-	-	-
	Responses: OV-101: HN(WB)0.5/NP(WB)3 OV-17: NP5.5								

Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
furilazole	C11H13Cl2NO3	0.46	0.6	0.39	C	S	-	50	3
	Responses: OV-101: NI1.5/NP50 OV-17: NI1/NP1.8 OV-225: NI1.6								
G-27550	C8H12N2O	0.28	-	0.29	C	-	-	-	-
	Responses: OV-101: NP3 OV-17: NP3								
Gardona	C10H9Cl4O4P	1.58	2.72	1.97	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: FP9/TI8 OV-17: FP10/FP(WB)3								
GS-31144	C8H12N2O2	-	-	-	-	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI220/NP100 OV-17: NI750/NP100								
haloxyfop methyl ester	C16H13ClF3NO4	1.55	-	1.4	-	-	-	-	-
	Responses: OV-101: HX(WB)9/NI5 OV-17: HX(WB)6/NI4.5								
heptachlor	C10H5Cl7	0.83	0.52	0.6	C	C	C	6	1
	Responses: OV-101: NI0.6 OV-17: NI0.4								
heptachlor epoxide	C10H5Cl7O	1.29	1.22	1.15	C	C	C	6	2
	Responses: OV-101: HX0.7/TR1 OV-17: HX0.9/NI2								
heptachloronorbornene	C7H3Cl7	-	-	0.23	-	-	-	-	-
	Responses: OV-17: NI0.2								
heptenophos	C9H12ClO4P	-	-	-	C	-	-	-	-
	Responses:								
hexachlorobenzene	C6Cl6	0.45	0.25	0.33	C	C	P	6	1
	Responses: OV-101: HX0.5/NI0.25 OV-17: HX0.3/NI0.3 OV-225: NI0.3								
hexachlorobutadiene	C4Cl6	-	-	0.04	-	V #	P	6	1
	Responses: OV-17: NI0.1								
hexachlorocyclopentadiene	C5Cl6	0.12	-	0.06	-	-	-	-	-
	Responses: OV-101: TR0.4 OV-17: NI0.8								
hexachloroethane	C2Cl6	-	-	0.02	-	-	-	-	-
	Responses: OV-17: NI0.1								
hexachloronorbornadiene	C7H2Cl6	-	-	0.12	-	-	-	-	-
	Responses: OV-17: NI0.2								
hexachlorophene	C13H6Cl6O2	13	13	16	-	NR	NR	6-15-50	-
	Responses: OV-101: NI(WB)2 OV-17: NI400 OV-225: NI1200								
hexachlorophene dimethyl ether	C15H10Cl6O2	9.7	-	-	-	NR	NR	6-15	-
	Responses: OV-101: TR7								

Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
hexaconazole	C14H17Cl2N3O	1.86	2.91	1.79	C	-	-	-	-
	Responses: OV-101: HX6/NI3 OV-17: NP(WB)23								
hexazinone	C12H20N4O2	2.91	-	-	P	NR	NR	6-15-50	1-2-3
	Responses: OV-101: HN(WB)3/NP(WB)15								
hexythiazox	C17H21ClN2O2S	1.28	-	2.41	-	S #	NR	50	2+3
	Responses: OV-101: NI(WB)70/NP(WB)90 OV-17: FS(WB)90/NI(WB)80/NP(WB)80								
HOE-030291	C17H16Cl2O5	7.9	10.8	12.6	-	-	-	-	-
	Responses: OV-101: NI20 OV-17: NI70 OV-225: NI20								
hydramethylnon*	C25H24F6N4	2.55	-	-	-	-	-	-	-
		32	4.5	-					
		44	53	-					
	Responses: OV-101: NI(V)250								
hydroxy chloroneb	C7H6Cl2O2	0.15	0.24	-	-	NR	-	6-15	-
	Responses: OV-101: TR7								
imazalil	C14H14Cl2N2O	1.76	4	2.08	C	NR	NR	6-15-50	-
	Responses: OV-101: NI30/NP(WB)12 OV-17: HX19/HX(WB)2/NI(WB)5/NP(WB)10								
imazamethabenz methyl ester*	C16H20N2O3	-	-	2.44	C	-	-	-	-
		1.79	3.5	2.76					
	Responses: OV-101: HX540/HX(WB)50/NI45/NP(WB)40 OV-17: HX60								
imazethapyr ammonium salt methyl ester	C16H21N3O3	2.4	4.3	3	-	-	-	-	-
	Responses: OV-101: NI120/NP50 OV-17: NI120/NP70 OV-225: NI160								
imidacloprid	C9H10ClN5O2	1.84	-	-	-	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI200/NP50								
IN-A3928	C11H18N4O2	3.06	-	-	S	NR	NR	6-15-50	1-2-3
	Responses: OV-101: HN(WB)1.4/NP(WB)29								
IN-B2838	C10H15N3O3	0.7	-	0.8	P	NR	NR	6-15-50	1-2-3
	Responses: OV-101: HN(WB)0.6/NP(WB)23 OV-17: HN(WB)0.2/NP(WB)13								
IN-T3935	C11H18N4O3	4.7	-	-	S	-	-	-	-
	Responses: OV-101: HN(WB)73/NI(WB)230								
IN-T3936	C10H15N3O4	1.41	-	2.55	S	NR	NR	6-15-50	1-2-3
	Responses: OV-101: HN(WB)1.6/NP(WB)51 OV-17: HN(WB)1.2/NP(WB)53								
IN-T3937	C12H20N4O3	4.7	-	-	S	-	-	-	-
	Responses: OV-101: HN(WB)390/NI(WB)400								

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Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
ioxynil methyl ether	C8H5I2NO	0.71	-	-	-	-	-	-	-
	Responses: OV-101: HN(WB)5/NI1								
iprobenfos	C13H21O3PS	0.6	-	0.54	C	-	-	-	-
	Responses: OV-101: FP(WB)1 OV-17: FP2								
iprodione metabolite isomer	C13H13Cl2N3O3	5.3	-	7.5	C	S	-	50	-
	Responses: OV-101: HX(WB)20/NI1000 OV-17: HX80								
iprodione*	C13H13Cl2N3O3	-	-	5.2	C	S #	NR	50	1-2-3
		4.2	18	6.3					
	Responses: OV-101: HX13/NI15/NP15 OV-17: HX20/NI35/NP200 OV-225: NI75								
isazofos	C9H17ClN3O3PS	0.55	0.8	0.63	C	C #	-	50	2+3
	Responses: OV-101: NI30/NP0.4 OV-17: FP(WB)1/NI10/NP0.2 OV-225: NI20								
isocarbamid	C8H15N3O2	0.44	-	0.82	C	-	-	-	-
	Responses: OV-101: HN(WB)0.2/NP(WB)0.5 OV-17: NP7								
isofenphos	C15H24NO4PS	1.36	1.73	1.38	C	C	-	15+50	-
	Responses: OV-101: FP2/NI20 OV-17: FP2								
isofenphos oxygen analog	C15H24NO5P	1.17	1.74	1.24	C	-	-	-	-
	Responses: OV-101: FP10 OV-17: FP7 OV-225: FP5								
isopropalin	C15H23N3O4	1.14	1.24	1.01	C	C	-	6	-
	Responses: OV-101: NI2/NP3 OV-17: NI3/NP15 OV-225: NI2								
isoprothiolane	C12H18O4S2	1.6	4.1	3.17	C	-	-	-	-
	Responses: OV-101: FS(WB)10/NI(WB)3								
isoproturon	C12H18N2O	-	-	0.89	S	-	-	-	-
	Responses: OV-17: NP(WB)10								
isoxaflutole (prop)	C15H12SNO4F3	1.11	4.7	1.33	NR	V #	S #	50	3
	Responses: OV-101: NI40/NP100 OV-17: NI40/NP200 OV-225: NI40								
jodfenphos	C8H8Cl2IO3PS	-	-	2.11	C	-	-	-	-
	Responses: OV-17: FP7								
kresoxim-methyl	C18H19NO4	2	3.02	3.38	P	C	C	15+50	3
	Responses: OV-101: NI(WB)10/NP(WB)50 OV-17: NI(WB)15/NP(WB)40 OV-225: NI(WB)10								
KWG 1323	C14H16ClN3O3	0.99	1.91	0.96	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI3/NP35 OV-17: NI1/NP5 OV-225: NI3/NP65								
KWG 1342	C14H18ClN3O3	4	15	4.2	-	-	-	-	-
	Responses: OV-101: NI1000/NP1000 OV-17: NI200/NP50 OV-225: NP1000								

Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
lactofen	C19H15ClF3NO7	7.3	-	-	-	-	C	50	2+3
	Responses: OV-101: NI10								
lambda-cyhalothrin	C23H19ClF3NO3	7.4	-	8	C	-	-	-	-
	Responses: OV-101: NI10 OV-17: HX30								
leptophos	C13H10BrCl2O2PS	5.8	7.7	8.5	C	C	C	6	2
	Responses: OV-101: FP14/TI20/TR11 OV-17: FP(WB)15/NI12								
leptophos oxygen analog	C13H10BrCl2O3P	4.2	7.6	6.5	C	-	-	-	-
	Responses: OV-101: FP60/TI65								
leptophos photoproduct	C13H11Cl2O2PS	2.38	3.24	3.14	C	-	-	-	-
	Responses: OV-101: NI(WB)2/TI5 OV-17: NI5 OV-225: NI5								
lindane	C6H6Cl6	0.48	0.69	0.47	C	C	C	6	1
	Responses: OV-101: HX0.6/TR0.5 OV-17: NI0.4								
linuron	C9H10Cl2N2O2	0.85	2.13	0.95	V	V #	V	50	3
	Responses: OV-101: TR28								
malathion	C10H19O6PS2	0.91	1.49	1.05	C	C	C	15+50	3
	Responses: OV-101: NI(WB)7/NP1 OV-17: FP3/NI7 OV-225: NI44								
malathion oxygen analog	C10H19O7PS	0.68	1.55	0.87	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: TI7 OV-17: FP5								
MB45950	C12H4SN4F6Cl2	1.25	8	1.09	S	P	V	15+50	2+3
	Responses: OV-101: NI2/NP8 OV-17: NI1/NP6 OV-225: NI6								
MB46136	C12H4SO2N4F6Cl2	2.06	31	1.98	S	S	V	50	2+3
	Responses: OV-101: NI2/NP30 OV-17: NI2/NP10 OV-225: NI30								
MCPA methyl ester	C10H11ClO3	0.19	-	-	-	-	-	-	-
	Responses: OV-101: HX(WB)2/NI600								
mecarbam	C10H20NO5PS2	1.28	2.67	1.58	C		-	50	-
	Responses: OV-101: FP(WB)1.7/NI5 OV-17: FP3/FP(WB)1.9 OV-225: HN4								
mecoprop methyl ester	C11H13ClO3	0.19	-	-	-	-	-	-	-
	Responses: OV-101: HX(WB)2/NI30								
melamine	C3H6N6	0.42	-	0.42	NR	-	-	-	-
	Responses: OV-101: NP100 OV-17: NP9								
mephosfolan	C8H16NO3PS2	-	-	2.51	C	-	-	-	-
	Responses: OV-17: FP3								

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Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
merphos*	C12H27PS3	1.34 1.95	0.65 1.64	1.43 1.88	-	C	C	6+15+50	3
	Responses: OV-101: FP3/TI5 OV-17: FP25								
metalaxyl	C15H21NO4	0.81	-	0.9	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: HX1000/NI1000/NP7 OV-17: NI1000/NP50								
metamitron	C10H10N4O	2.24	-	-	-	-	-	-	-
	Responses: OV-101: NI20								
metasystox thiol	C6H15O3PS2	0.28	0.49	0.32	C	-	-	-	-
	Responses: OV-101: FP(WB)8 OV-17: FP(WB)0.8								
metasystox thiono*	C6H15O3PS2	- 0.31	- 0.49	0.18 0.32	-	-	-	-	-
	Responses: OV-101: FP6 OV-17: FP(WB)2								
metazachlor	C14H16ClN3O	1.5	-	1.46	C	-	-	-	-
	Responses: OV-101: NI34 OV-17: HX5/NP4								
methabenzthiazuron	C10H11N3OS	0.35	0.7	0.41	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: HN(WB)0.5/NI320/NP15 OV-17: NP24 OV-225: NI2550								
methamidophos	C2H8NO2PS	0.07	0.25	0.09	V	-	-	-	-
	Responses: OV-101: FP(WB)0.7 OV-17: FP1/FP(WB)0.6 OV-225: FP4								
methazole	C9H6Cl2N2O3	0.97	1.46	-	-	-	-	-	-
	Responses: OV-101: HX13/NI40								
methidathion	C6H11N2O4PS3	1.4	3.33	2.28	C	S	P #	50	3
	Responses: OV-101: FP5/FP(WB)1.6/NP3 OV-17: FP(WB)2.4/NI10 OV-225: NI50								
methidathion oxygen analog	C6H11N2O5PS2	1.07	-	1.8	-	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI100/NP40 OV-17: FP125/NI200/NP40								
methidathion sulfone	C5H8N2O3S2	0.56	2.29	0.82	-	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI150/NP100 OV-17: NI200/NP35 OV-225: NI400								
methidathion sulfoxide	C5H8N2O4S2	0.45	2.25	0.71	-	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI8/NP35 OV-17: NI8/NP10 OV-225: NI30								
methiocarb	C11H15NO2S	0.88	-	-	C	-	-	-	-
	Responses:								
methiocarb sulfone	C11H15NO4S	0.8	-	1.17	S	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI200/NP200								
methomyl	C5H10N2O2S	0.1	-	-	-	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI220								

Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
methoprotryne	C11H21N5OS	2.07	-	2.92	C	-	-	-	-
	Responses: OV-101: NP189 OV-17: NP(WB)8								
methoxychlor olefin	C16H14Cl2O2	2.97	3.7	4.2	C	C	C	6	2
	Responses: OV-101: NI(WB)8 OV-17: NI10 OV-225: NI9								
methoxychlor, o, p'-	C16H15Cl3O2	3.3	4.5	5	-	C	-	6	-
	Responses: OV-101: NI(WB)2 OV-17: NI9 OV-225: NI23								
methoxychlor, p, p'-	C16H15Cl3O2	4.7	7.2	7.2	C	C	C	6	2
	Responses: OV-101: TR9 OV-17: NI7								
methyl 2,3,5-triiodobenzoate	C8H5I3O2	2.28	-	-	-	-	-	-	-
	Responses: OV-101: NI1.5								
methyl 2,3,6-trichlorobenzoate	C8H5Cl3O2	0.23	0.21	0.18	-	-	-	-	-
	Responses: OV-101: TR0.6 OV-17: NI0.3								
methyl 3,5-dibromo-4-methoxy= benzoate	C9H8Br2O3	0.56	0.49	0.47	-	-	-	-	-
	Responses: OV-101: NI(WB)2 OV-17: NI(WB)1 OV-225: NI(WB)1.5								
methyl 4-chloro-1H-indole-3-acetate	C11H10ClNO2	1.17	-	-	R	R #	NR	50	1-2-3
	Responses: OV-101: MC25/NI200								
metobromuron	C9H11BrN2O2	0.67	1.44	0.69	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: HX8/NI5 OV-17: HX10								
metolachlor	C15H22ClNO2	1.03	1.21	0.93	C	S #	NR	50	1-2-3
	Responses: OV-101: HX5/NI12 OV-17: HX7 OV-225: NI9								
metolcarb	C9H11NO2	0.17	-	-	C	-	-	-	-
	Responses:								
metoxuron	C10H13ClN2O2	-	-	0.18	V	NR	NR	6-15-50	1-2-3
	Responses: OV-17: HX25								
metribuzin	C8H14N4OS	0.57	1.47	0.91	V	NR	NR	50	1-2-3
	Responses: OV-101: NI0.5/NP2 OV-17: NI0.4/NP11 OV-225: NI0.7								
metribuzin, deaminated diketo metabolite*	C7H11N3O2	0.5 0.73	0.79 1.29	0.44 0.52	NR	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI75 OV-17: NI150 OV-225: NI160								
metribuzin, deaminated metabolite	C8H13N3OS	0.83	3.77	1.06	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI60/NP45 OV-17: NI10/NP125 OV-225: NI60								
metribuzin, diketo metabolite	C7H12N4O2	0.56	1.41	0.55	NR	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI35/NP40 OV-17: NI6/NP20 OV-225: NI15								

Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
mevinphos, (E)-	C7H13O6P	0.16	-	0.13	C	NR	NR	6-15-50	-
	Responses: OV-101: FP2 OV-17: FP2								
mevinphos, (Z)-	C7H13O6P	0.13	-	0.15	C	NR	-	6-15-50	-
	Responses: OV-101: FP2/FP(WB)1 OV-17: FP2/FP(WB)0.6								
MGK 264	C17H25NO2	1.6	-	-	-	-	-	-	-
	Responses:								
mirex	C10Cl12	5.8	2.95	5.6	P	C	P	6	1
	Responses: OV-101: NI7 OV-17: NI4								
mirex, 5,10-dihydro-*	C10H2Cl10	2.14	-	-	-	-	-	-	-
		2.47	-	-					
		3.21	-	-					
		4.3	-	-					
	Responses: OV-101: NI100								
molinate	C9H17NOS	-	0.19	-	-	-	-	-	-
	Responses:								
monocrotophos	C7H14NO5P	0.31	1.6	0.5	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: FP(WB)0.7/TI15 OV-17: FP2/FP(WB)0.8 OV-225: FP3								
monolinuron	C9H11ClN2O2	0.48	0.91	0.48	C	-	-	-	-
	Responses: OV-101: HX4 OV-17: HX18 OV-225: NI180								
monuron	C9H11ClN2O	0.1	-	-	-	NR	NR	6-15-50	1-2-3
	Responses: OV-101: TR175								
myclobutanil	C15H17ClN4	1.9	7.2	2.6	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: HN(WB)0.7/NI25/NP(WB)7 OV-17: HX10/HX(WB)4/NI(WB)21/NP75/NP(WB)6								
myclobutanil alcohol metabolite	C15H17ClN4O	3.6	37.1	7.5	S	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI60 OV-17: NP375								
myclobutanil dihydroxy metabolite	C15H17N4O2Cl	6.5	-	11.5	NR	NR	NR	6-15-50	1-2-3
	Responses: OV-17: NI1000/NP1000								
N, N-diallyl dichloroacetamide	C8H11Cl2NO	0.19	0.22	0.14	C	S	S	15+50	2+3
	Responses: OV-101: HX1/NI1/NP1 OV-17: NI1 OV-225: NI1								
N-(3,4-dichlorophenyl)-N'-methyl= urea	C8H8Cl2N2O	0.17	0.14	0.1	-	NR	NR	6-15-50	-
	Responses: OV-101: HX20/NI9 OV-17: NI5 OV-225: NI12								
n-acetyl nitrofen	C14H11Cl2NO2	6.6	-	-	-	-	-	-	-
	Responses: OV-101: TR500								

Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
naled	C4H7Br2Cl2O4P	0.34	-	0.32	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NP11 OV-17: FP20/NI8								
napropamide	C17H20NO2	1.7	-	2.12	C	-	-	-	-
	Responses: OV-101: NP40								
neburon	C12H16Cl2N2O	0.11	-	-	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: TR25								
nitralin	C13H19N3O6S	3.8	24	6.3	C	P	P	50	3
	Responses: OV-101: NI(WB)1 OV-17: NI7 OV-225: NI21								
nitrapyrin	C6H3Cl4N	0.2	0.18	0.2	C	C	V	6	2
	Responses: OV-101: HX2/NI(WB)0.8/NP(WB)4 OV-17: HX(WB)0.6 OV-225: NI0.9								
nitrofen	C12H7Cl2NO3	2.03	3.8	2.71	C	C	C	15	2
	Responses: OV-101: NI(WB)1 OV-17: NI3 OV-225: NI3								
nitrofluorfen	C13H7ClF3NO3	0.96	1.45	0.86	C	C	C	15	2
	Responses: OV-101: HX5/NI1 OV-17: HX3 OV-225: NI1.5								
nitrothal-isopropyl	C14H17O6N	1.1	-	0.68	C	-	-	-	-
	Responses: OV-101: NI(WB)2 OV-17: NP(WB)65								
nonachlor, cis-	C10H5Cl9	2.52	3.33	2.61	C	C	C	6	1
	Responses: OV-101: HX1/NI2 OV-17: NI2 OV-225: NI3								
nonachlor, trans-	C10H5Cl9	1.75	1.45	1.42	C	C	C	6	1
	Responses: OV-101: TR2 OV-17: HX0.4/NI0.8								
norea	C13H15N2O	-	-	1.05	C	-	-	-	-
	Responses: OV-17: NP(WB)8								
norflurazon	C12H9ClF3N3O	4.5	-	5.01	V	NR	NR	6-15-50	-
	Responses: OV-101: HX50 OV-17: NP(WB)10								
NTN33823	C9H11N4Cl	3	-	1.18	-	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NP300 OV-17: NP1000								
NTN35884*	C9H9N5O2Cl	1.59	-	-	-	NR	NR	6-15-50	1-2-3
		5	-	-					
	Responses: OV-101: NI550/NP210								
nuarimol	C17H12ClFN2O	3.36	7.3	4.8	C	NR	C #	50	1-2-3
	Responses: OV-101: NI5 OV-17: HX4								
octachlor epoxide	C10H4Cl8O	1.33	0.94	1.05	C	C	C	6	1
	Responses: OV-101: TR1 OV-17: NI0.6								

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Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
octachlorocyclopentane	C5Cl8	-	-	0.22	-	-	-	-	-
	Responses: OV-17: NI0.2								
octhilinone	C11H19NOS	0.66	-	0.64	C	-	-	-	-
	Responses: OV-101: FS4								
ofurace	C14H16NO3Cl	2.62	18.6	5.4	C	-	-	-	-
	Responses: OV-101: HX17 OV-17: HX51 OV-225: NI44								
omethoate	C5H12NO4PS	0.25	1.11	0.39	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: FP(WB)0.9/TI25 OV-17: FP5/FP(WB)1.1 OV-225: FP5								
oryzalin	C12H8N4O6S	4.7	-	-	-	NR	NR	6-15-50	-
	Responses: OV-101: NI8800								
ovex	C12H8Cl2O3S	1.61	3.04	2.2	C	C	C	15	2
	Responses: OV-101: HX4/TR3 OV-17: HX5 OV-225: NI3								
oxadiazon	C15H18Cl2N2O3	1.97	2.48	1.96	C	C	P	15	-
	Responses: OV-101: HX4/NI4 OV-17: NI2								
oxadixyl	C14H18N2O4	2.5	14	5	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI1700/NP8 OV-17: NP14 OV-225: NI4500								
oxamyl	C7H13N3O3S	-	-	-	-	-	-	-	-
	Responses:								
oxamyl oxime metabolite	C5H10N2O2S	0.25	0.92	0.28	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: HN(WB)0.5/NI(WB)4/NP(WB)4 OV-17: HN(WB)0.4/NI(WB)3/NP(WB)3 OV-225: NI(WB)13								
oxycarboxin	C12H13NO4S	3.28	-	9.4	R	-	-	-	-
	Responses: OV-101: FS(WB)17/HN(WB)3/NP(WB)130 OV-17: HN(WB)4								
oxydemeton-methyl	C6H15O4PS2	0.46	0.49	0.31	C	-	-	-	-
	Responses: OV-17: FP(WB)4								
oxydemeton-methyl sulfone	C6H15O5PS2	0.72	-	1.48	C	-	-	-	-
	Responses: OV-101: FP(WB)2 OV-17: FP(WB)3								
oxydeprofos	C7H17O4SP	-	-	-	-	-	-	-	-
	Responses:								
oxyfluorfen	C15H11ClF3NO4	2	4	2.16	C	C	C	15	2
	Responses: OV-101: NI5 OV-17: HX9/NI2/NP350								
oxythioquinox	C10H6N2OS2	1.57	-	1.85	C	-	-	-	-
	Responses: OV-101: NI(WB)1								

Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
paclobutrazol	C15H20ClN3O	1.52	-	1.59	C	-	-	-	-
	Responses: OV-101: NP(WB)6 OV-17: HX7/HX(WB)3/NI(WB)85/NP(WB)5								
parathion	C10H14NO5PS	0.98	1.91	1.07	C	C	C	15	2
	Responses: OV-101: NI(WB)4/NP2 OV-17: FP2/NI4 OV-225: FP2/NI6								
parathion oxygen analog	C10H14NO6P	0.8	-	0.86	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI(WB)5/NP3 OV-17: FP4/NI15								
parathion-methyl	C8H10NO5PS	0.71	1.64	0.87	C	C	C	15	2
	Responses: OV-101: FP(WB)0.9/NI(WB)3/NP1.5 OV-17: FP2/FP(WB)1/NI3 OV-225: FP1/NI11								
parathion-methyl oxygen analog	C8H10NO6P	0.55	1.71	0.66	-	NR	NR	6-15-50	1-2-3
	Responses: OV-101: TI5/TR11 OV-17: FP10								
PB-7, methylated	C20H25ClN2O3S	23	57	43	-	-	-	-	-
	Responses: OV-101: NI150/NP300 OV-17: NI200/NP500 OV-225: NI300								
PB-9	C19H25ClN2O2S	25	87	46	V	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI300/NP500 OV-17: NI250/NP750 OV-225: NI500								
pebulate	C10H21NOS	0.17	-	0.1	C	P	-	15	-
	Responses: OV-101: HN(WB)0.4/NP7 OV-17: NP6								
penconazole	C13H15Cl2N3	1.24	-	1.32	C	-	-	-	-
	Responses: OV-101: NP(WB)3 OV-17: HX3/HX(WB)2/NI(WB)2/NP(WB)3								
pendimethalin	C13H19N3O4	1.22	1.48	1.21	C	C	P	15	2
	Responses: OV-101: NI3/NP5 OV-17: NI1.5/NP6 OV-225: NI3								
pentachloroaniline	C6H2Cl5N	0.67	0.79	0.66	C	C	C	6	1
	Responses: OV-101: HX0.5/NI0.4/NP10 OV-17: NI0.6/NP10 OV-225: NI0.5								
pentachlorobenzene	C6HCl5	0.24	0.13	0.16	C	C	C	6	1
	Responses: OV-101: HX0.3/NI0.25 OV-17: NI0.3 OV-225: NI0.25								
pentachlorobenzonitrile	C7Cl5N	0.5	0.59	0.45	C	C	P	15	2
	Responses: OV-101: NI0.5 OV-17: NI3								
pentachlorophenyl methyl ether	C7H3Cl5O	0.46	0.3	0.34	C	C	C	6	1
	Responses: OV-101: HX0.4/TR0.4 OV-17: NI0.3								
pentachlorophenyl methyl sulfide	C7H3Cl5S	0.94	0.69	0.87	C	C	C	6	1
	Responses: OV-101: HX0.7/NI0.4 OV-17: NI3 OV-225: NI0.5								
permethrin, cis-	C21H20Cl2O3	9.4	11.1	13.8	C	V #	C	6+15	2
	Responses: OV-101: NI75								

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Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
permethrin, trans-	C21H20Cl2O3	10.2	13	15	C	V #	C	6+15	2
	Responses: OV-101: NI100								
Perthane	C18H2OCl2	2.23	2.01	2.42	C	C	C	6	1
	Responses: OV-101: TR150 OV-17: NI25								
Perthane olefin	C18H19Cl	1.53	0.95	-	-	C	C	6	1
	Responses: OV-101: TR40								
phenmedipham	C16H16N2O4	0.32	-	0.41	-	-	-	-	-
	Responses: OV-101: HN(WB)1.5 OV-17: NP40								
phenothiazine	C12H9NS	1.16	-	1.56	-	-	-	-	-
	Responses: OV-101: HN(WB)1.4 OV-17: NP4								
phenothrin*	C23H26O3	5.4 11.5	4.8 10.9	6.5 15	-	-	-	-	-
	Responses: OV-101: NI500/NI(WB)40								
phenthoate	C12H17O4PS2	1.31	2.05	1.83	C	C	-	15+50	-
	Responses: OV-101: FP(WB)2/NI5/TI3 OV-17: FP2/FP(WB)2.6/NI5								
phorate	C7H17O2PS3	0.37	0.38	0.32	C	V #	V #	6	1
	Responses: OV-101: FP1/NI(WB)24/TI1 OV-17: FP0.5/NI14 OV-225: FP0.5/NI17								
phorate oxygen analog	C7H17O3PS2	0.3	0.37	0.29	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI400/TI2 OV-17: FP1 OV-225: FP0.5								
phorate oxygen analog sulfone	C7H17O5PS2	0.66	-	1.02	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: FP1/TI10 OV-17: FP6								
phorate oxygen analog sulfoxide	C7H17O4PS2	0.78	-	1.06	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI300 OV-17: FP40								
phorate sulfone	C7H17O4PS3	0.97	3.26	1.3	C	S #	S #	6-15-50	3
	Responses: OV-101: NI4/TI4 OV-17: FP2								
phorate sulfoxide	C7H17O3PS3	0.89	2.55	1.26	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: FP5/NI8/NP4 OV-17: FP6								
phosalone	C12H15ClNO4PS2	5.5	5.5	9.1	C	C	C	50	2+3
	Responses: OV-101: NP10/TR15 OV-17: NI12								
phosalone oxygen analog	C12H15ClNO5PS	3.8	-	6.2	C	-	-	-	-
	Responses: OV-101: FP600/FP(WB)8/NI160 OV-17: FP(WB)12								
phosfolan	C7H14NO3PS2	-	-	2.69	C	-	-	-	-
	Responses: OV-17: FP5								

Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
phosmet	C11H12O4NPS2	4	14.9	8.4	C	NR	-	6-15-50	3
	Responses: OV-101: NI(WB)2/NP19 OV-17: FP50/NI78 OV-225: FP50								
phosmet oxygen analog*	C11H12NO5PS	0.5	0.53	0.24	-	NR	NR	6-15-50	-
		2	0.93	0.44					
		3.1	14.8	7.1					
	Responses: OV-101: NI900/NP100 OV-17: FP150 OV-225: NI600								
phosphamidon*	C10H19ClNO5P	0.53	-	0.57	C	NR	NR	6-15-50	1-2-3
		0.67	-	0.76					
	Responses: OV-101: FP10 OV-17: FP2								
photodieldrin	C12H8Cl6O	4.4	15.5	8.5	-	C	C	15+50	2
	Responses: OV-101: TR6 OV-17: NI5								
photodieldrin B	C13H9Cl5O	1.43	-	-	-	-	-	-	-
	Responses: OV-101: TR2								
phoxim oxygen analog	C12H15N204P	0.86	-	-	C	-	-	-	-
	Responses: OV-101: FP5								
picloram methyl ester	C7H5Cl3N2O2	0.75	2.67	-	-	-	-	-	-
	Responses: OV-101: HX(WB)1.2/NI1.5/TR1								
picloram*	C6H3Cl3N2O2	0.25	-	-	-	-	-	-	-
		0.67	-	-					
	Responses: OV-101: NI100								
piperophos	C14H28NO3PS2	4.8	9.7	6.8	C	-	-	-	-
	Responses: OV-101: FP15								
pirimicarb	C11H18N4O2	0.61	-	0.73	C	-	-	-	-
	Responses: OV-17: FP(WB)2								
pirimiphos-ethyl	C13H24N3O3PS	1.14	1.03	1.14	C	C	C	15+50	3
	Responses: OV-101: FP2/TI4/TR150 OV-17: FP3/NI4								
pirimiphos-ethyl oxygen analog	C13H24N3O4P	1.01	-	1.14	C	-	-	-	-
	Responses: OV-101: FP4/FP(WB)1.6 OV-17: FP(WB)2.4								
pirimiphos-methyl	C11H20N3O3PS	0.87	-	0.92	C	C	C	15	3
	Responses: OV-101: FP2/FP(WB)1.2/NI100/NP2 OV-17: FP2/FP(WB)1.2								
PP 890	C9H10O2ClF3	1.05	1	-	-	-	-	-	-
	Responses: OV-101: NI220 OV-225: NI150								
PPG-1576	C19H17ClF3NO5	6.7	-	-	-	-	P	50	2+3
	Responses: OV-101: NI13								

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Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
PPG-2597	C20H17ClF3NO6	1.9	3.16	1.86	-	NR	NR	6-15-50	1-2-3
	Responses: OV-101: HN(WB)58/HX(WB)110/NI(WB)85/NP(WB)150 OV-17: HN(WB)120/HX(WB)66/NI(WB)78 OV-225: NI(WB)120								
PPG-847, methylated	C15H9ClF3NO3	2.15	5	2.4	-	-	-	-	-
	Responses: OV-101: HN(WB)3/HX(WB)10/NI(WB)3/NP(WB)34 OV-17: HN(WB)4/HX(WB)6.5/NI(WB)3/NP(WB)210 OV-225: NI(WB)4								
PPG-947	C17H11ClF3NO7	1.04	-	1.13	-	NR	NR	6-15-50	1-2-3
	Responses: OV-101: HN(WB)620/NI(WB)1000 OV-17: NI(WB)600								
PPG-947, methylated*	C18H13ClF3NO7	0.42 2.14	0.97 5	0.49 2.4	-	-	-	-	-
	Responses: OV-101: HN(WB)5/NI(WB)5/NP(WB)65 OV-17: HN(WB)5 OV-225: NI(WB)6								
pretilachlor	C17H26ClNO2	1.88	-	1.99	C	-	-	-	-
	Responses: OV-101: NI25 OV-17: HX10								
probenazole	C10H9NO3S	-	-	-	C	-	-	-	-
	Responses:								
prochloraz	C15H16Cl3N3O2	10.4	-	15.4	C	-	-	-	-
	Responses: OV-101: HX50/NI12								
procyazine	C10H13ClN6	1.5	7.9	-	C	-	-	-	-
	Responses:								
procymidone	C13H11Cl2NO2	1.37	3.04	1.49	C	C	P	15	-
	Responses: OV-101: NI12 OV-17: HX1 OV-225: NI7								
prodiamine	C13H17F3N4O4	0.94	0.97	0.66	C	-	-	-	-
	Responses: OV-17: NP(WB)50 OV-225: NP50								
profenofos	C11H15BrClO3PS	1.8	2.34	2.13	C	P	P	50	3
	Responses: OV-101: FP7/FP(WB)2.6/NI3 OV-17: FP5/FP(WB)2.9								
profluralin	C14H16F3N3O4	0.53	0.46	0.3	V	V	-	6	-
	Responses: OV-101: HX5/NI1/NP1 OV-17: NI0.8/NP9 OV-225: NI1								
Prolan	C15H13Cl2NO2	2.81	7.5	3.9	P	S	S	15	2
	Responses: OV-101: NI(WB)1 OV-17: NI6 OV-225: NI10								
promecarb	C12H17NO2	1.58	-	-	V	-	-	-	-
	Responses: OV-101: TI400								
prometryn	C10H19N5S	0.77	-	0.74	C	P #	P #	50	1-2-3
	Responses: OV-101: FP200/NI40/TI50 OV-17: FP20								

Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
pronamide	C12H11Cl2NO	0.51	0.84	0.4	C	P	-	15+50	-
	Responses: OV-101: HX1/NI2/NP7 OV-17: NI1/NP85 OV-225: NI3								
propachlor	C11H14ClNO	0.34	0.37	0.26	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI(WB)16/NP(WB)13 OV-17: NI5/NI(WB)5 OV-225: NI9/NI(WB)5								
propanil	C9H9Cl2NO	0.66	2.82	0.78	C	NR	NR	6-15	3
	Responses: OV-101: HX6/NI3/NI(WB)5 OV-17: NI4 OV-225: NI8								
propargite	C19H26O4S	3.8	4.8	4.3	C	C	-	15	2
	Responses: OV-101: FS45/NI(WB)230 OV-17: NI2600 OV-225: NI1300								
propazine	C9H16ClN5	0.53	0.65	0.41	C	S	NR	15+50	3
	Responses: OV-101: NI(WB)110/TI30 OV-17: NI43 OV-225: NI37								
propetamphos	C10H20NO4PS	0.48	-	0.42	C	C #	-	15+50	2+3
	Responses: OV-101: FP1.5 OV-17: FP0.5								
propham	C10H13NO2	0.13	-	0.12	C	P	P	15	-
	Responses: OV-101: NP(WB)2 OV-17: NP16								
propiconazole*	C15H17Cl2N3O2	3.06 3.21	- 5.6	- 4	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI10/NI(WB)17 OV-17: HX9								
propoxur	C11H15NO3	-	-	-	C	-	-	-	-
	Responses:								
prosulfuron*	C15H16F3N5O4S	0.18 0.44	- 2.49	- 0.64	-	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NP8 OV-17: NI120 OV-225: NI20								
prothiofos	C11H15Cl2PO2S2	1.85	1.74	1.82	C	C	C	6	2
	Responses: OV-101: FP4/NI3/NP3 OV-17: NI3 OV-225: NI1								
prothoate	C9H20NO3PS2	0.75	1.55	0.79	C	-	-	-	-
	Responses: OV-101: FP1/NI5								
pyracarbolid	C13H15NO2	1.05	-	1.43	-	-	-	-	-
	Responses: OV-101: HN(WB)1.4 OV-17: NP6								
pyrazon	C10H8ClN3O	2.67	13	8	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: HN(WB)8/HX(WB)30/NI(WB)26/NP(WB)56 OV-17: HX(WB)50 OV-225: NI3700								
pyrazon metabolite A	C16H18ClN3O6	2.62	-	-	-	-	-	-	-
	Responses: OV-101: HX(WB)260								
pyrazon metabolite B	C6H4ClN3O	0.42	-	0.9	-	NR	NR	6-15-50	1-2-3
	Responses: OV-101: HN(WB)3/HX(WB)42/NI(WB)35/NP(WB)14 OV-17: HN(WB)3/HX(WB)23/NI(WB)23/NP(WB)44								

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Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
pyrazophos	C14H20N3O5PS	8.1	-	13	C	-	-	-	-
	Responses: OV-101: FP(WB)12 OV-17: FP25								
pyrethrins*	C21H27O4	2.12	1.76	1.76	-	C	C	50	-
		2.95	2.84	2.7					
	Responses: OV-101: NI25								
pyridaphenthion	C14H17O4N2SP	4.2	14	8.7	C	-	-	-	-
	Responses: OV-101: NI(WB)16								
pyrimethanil	C12H13N3	0.67	-	-	C	S	S #	50	3
	Responses: OV-101: NP(WB)0.5								
pyrithiobac-sodium methyl ester	C14H13ClN2O4	2.51	4	4.2	-	-	-	-	-
	Responses: OV-101: HN(WB)1/HX(WB)12/NI(WB)9/NP(WB)90 OV-17: HN(WB)2/HX(WB)13/NI(WB)18/NP(WB)85 OV-225: NI(WB)13								
quinalphos	C12H15N2O3PS	1.32	2	1.64	C	C	-	15	-
	Responses: OV-101: FP5/FP(WB)2.7 OV-17: FP3/FP(WB)3								
quintozene	C6Cl5NO2	0.51	0.46	0.46	C	C	C	6	1
	Responses: OV-101: TR0.3 OV-17: NI0.5								
quizalofop ethyl ester	C19H17ClN2O4	13.6	-	25	C	-	-	-	-
	Responses: OV-101: HX70/NI80								
RH-6467*	C19H15N4ClO	7.9	-	-	S	NR	NR	6-15-50	1-2-3
		10.4	-	-					
		15	-	-					
	Responses: OV-101: NI90/NP300								
RH-9129	C19H16N3ClO2	14	-	-	V	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI40/NP190								
RH-9130	C19H16N3ClO2	12	-	-	P	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI50/NP170								
ronnel	C8H8Cl3O3PS	0.81	0.86	0.76	C	C	C	6	2
	Responses: OV-101: NI(WB)1/TI3 OV-17: FP1/NI1 OV-225: NI2								
ronnel oxygen analog	C8H8Cl3O4P	0.64	1.02	0.62	C	NR	-	6-15-50	-
	Responses: OV-101: NP3 OV-17: FP5/FP(WB)1								
RPA 203328, methylated	C10H9F3O4S	0.26	0.51	0.23	-	-	-	-	-
	Responses: OV-101: NI0.5 OV-17: NI0.4 OV-225: NI0.5 5								
RPA202248	C15H12SNO4F3	1.13	4.7	1.38	NR	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI40/NP120 OV-17: NI40/NP250 OV-225: NI60								

Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
S-bioallethrin	C19H26O3	1.37	1.22	1.12	-	C	-	50	-
	Responses: OV-101: NI2 OV-17: NI2 OV-225: NI2								
schradan	C8H24N4O3P2	0.58	-	0.52	C	NR	-	6-15-50	-
	Responses: OV-101: FP4/TI5 OV-17: FP1								
sethoxydim	C17H29NO3S	3.34	-	-	-	NR	NR	6-15-50	3
	Responses: OV-101: NI50								
sethoxydim sulfoxide	C17H29NO4S	0.84	-	-	-	NR	NR	6-15-50	3
	Responses: OV-101: NI50								
silvex	C9H7Cl3O3	0.48	-	-	-	-	-	-	-
	Responses: OV-101: NI40								
silvex methyl ester	C10H9Cl3O3	0.45	0.44	-	-	-	-	-	-
	Responses: OV-101: HX(WB)0.8/NI0.6								
simazine	C7H12ClN5	0.41	0.83	0.5	C	NR	NR	50	1-2-3
	Responses: OV-101: NI(WB)90 OV-17: NI56/NP(WB)1.5 OV-225: NI130								
simetryn	C8H15N5S	2.02	1.21	-	C	-	-	-	-
	Responses: OV-101: NP7								
Strobane*	C10H11Cl7	1.09	-	-	-	C	C	6	1
		1.32	-	-					
		1.53	-	-					
		1.8	-	-					
		1.94	-	-					
		2.09	-	-					
		2.33	-	-					
		2.69	-	-					
		3.1	-	-					
		3.7	-	-					
	Responses: OV-101: TR40								
sulfallate	C8H14ClNS2	0.38	0.44	0.36	C	C	C	6+15	2
	Responses: OV-101: NI(WB)1 OV-17: NI1 OV-225: NI1								
sulfanilamide	C6H8O2N2S	-	-	2.11	NR	NR	NR	6-15-50	1-2-3
	Responses: OV-17: NP200								
sulfotep	C8H20O5P2S2	0.34	-	0.29	C	C	P	6+15	2
	Responses: OV-101: TI0.8 OV-17: FP0.5								
Sulphenone	C12H9ClO2S	1.26	3.5	1.92	C		-	50	3
	Responses: OV-101: TR4								

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Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
sulprofos	C12H19O2PS3	2.79	-	3.5	C	-	-	-	-
	Responses: OV-101: FP(WB)47 OV-17: FP3								
sulprofos oxygen analog sulfone	C12H19O5PS2	5.1	-	10.6	C	-	-	-	-
	Responses: OV-101: FP(WB)80 OV-17: FP40/FP(WB)28/NI20/NP40								
sulprofos sulfone	C12H19O4PS3	7.2	-	13.1	C	-	-	-	-
	Responses: OV-101: FP(WB)16 OV-17: FP(WB)26								
sulprofos sulfoxide*	C12H19O3PS3	2.78	-	3.6	C	-	-	-	-
		6.1	-	11.7					
	Responses: OV-101: FP(WB)14 OV-17: FP(WB)29								
TCMTB	C9H6N2S3	1.5	4.3	2.67	C	P	P	15	-
	Responses: OV-101: NI3/NP22 OV-17: NP36 OV-225: NI12								
TDE, o,p'-	C14H10Cl4	1.9	2.46	2.19	-	C	C	6	1
	Responses: OV-101: TR2 OV-17: NI2								
TDE, o,p'-, olefin	C14H9Cl3	1.19	1.15	1.2	-	-	-	-	-
	Responses: OV-101: TR12 OV-17: NI2								
TDE, p,p'-	C14H10Cl4	2.41	3.8	2.87	C	C	C	6	1
	Responses: OV-101: TR4 OV-17: NI2								
TDE, p,p'-, olefin	C14H9Cl3	1.45	1.36	1.45	C	C	C	6	1
	Responses: OV-101: NI(WB)4 OV-17: NI4 OV-225: NI3								
tebuconazole	C16H22ClN3O	3.38	-	4.2	C	-	-	-	-
	Responses: OV-101: HX(WB)1 OV-17: HX(WB)3								
tebufenozide	C22H28N2O2	7	-	11	-	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI30/NP4000								
tebupirimfos	C13H23N2O3PS	0.63	-	-	-	V	V	6+15	2+3
	Responses: OV-101: NI16/NP1								
tebupirimfos oxygen analog	C13H23O4N2P	0.51	-	-	-	NR	NR	6-15-50	1-2-3
	Responses: OV-101: NI100/NP1								
tebuthiuron	C9H16N4OS	0.26	-	0.21	-	-	-	-	-
	Responses: OV-101: NI1000/NP(WB)0.5 OV-17: FP20								
tecnazene	C6HCl4NO2	0.29	0.26	0.24	C	C	C	6	1
	Responses: OV-101: TR0.5 OV-17: NI0.3								
teflubenzuron*	C14H6Cl2F4N2O2	-	0.11	0.09	-	NR	NR	6-15-50	1-2-3
		0.14	0.23	0.14					
	Responses: OV-101: NI(WB)1.5 OV-17: NP(WB)5								

Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
TEPP	C8H20O7P2	0.21	0.27	0.24	C	-	-	-	-
	Responses: OV-101: FP(WB)46/NI(WB)4000 OV-17: FP(WB)70/NI10000/NP(WB)70 OV-225: NI13700								
terbacil	C9H13ClN2O2	0.54	2.1	0.72	C	NR	NR	6-15	2+3
	Responses: OV-101: HN(WB)1/HX5/NI(WB)6/NP(WB)8 OV-17: HN(WB)0.6/HX(WB)5/NI(WB)2/NP(WB)8 OV-225: NI(WB)7								
terbufos	C9H21O2PS3	0.5	0.44	0.41	C	P	S	6	-
	Responses: OV-101: FP2/NI40/TI2 OV-17: FP0.5/FP(WB)1.6/NI20								
terbufos oxygen analog	C9H21O3PS2	0.42	-	0.39	C	-	NR	6-15-50	1-2-3
	Responses: OV-101: FP0.5/NI1000 OV-17: FP1/NI2500								
terbufos oxygen analog sulfone	C9H21O5PS2	0.92	2.9	1.28	C	NR	NR	6-15-50	1-2-3
	Responses: OV-101: FP7/NI40 OV-17: FP2/NI60 OV-225: FP5/NI1500								
terbufos sulfone	C9H21O4PS3	1.2	-	1.58	C	C #	C #	6-15-50	2+3
	Responses: OV-101: FP2/NI5 OV-17: FP2/NI10								
terbumeton	C10H19N5O	0.47	0.42	0.53	C	-	-	-	-
	Responses: OV-17: NP(WB)25 OV-225: NP20								
terbuthylazine	C9H16N5Cl	0.47	0.71	0.48	C	P	-	15+50	-
	Responses: OV-101: NI(WB)87/NP(WB)6 OV-17: NI250 OV-225: NI43								
terbutryn	C10H19N5S	0.84	1.08	-	C	-	-	-	-
	Responses:								
tetradifon	C12H6Cl4O2S	5.2	-	8.3	C	C	C	15	2
	Responses: OV-101: TR6 OV-17: NI5								
tetraiodoethylene	C2I4	0.55	0.86	1.04	-	P	P	6	-
	Responses: OV-101: NI(WB)3 OV-17: NI4								
tetramethrin*	C19H25NO4	4.3	-	-	C	NR	NR	6-15-50	1-2-3
		4.5	8.5	7.2					
	Responses: OV-101: NI50								
tetrasul	C12H6Cl4S	2.64	2.33	2.8	C	C	C	6	1
	Responses: OV-101: NI(WB)1 OV-17: NI5 OV-225: NI3								
tetrasul sulfoxide	C12H6Cl4OS	4.7	8.6	7.2	-	-	-	-	-
	Responses: OV-101: NI(WB)1 OV-17: NI8 OV-225: NI7								
thiabendazole	C10H7N3S	1.48	-	2.04	C	NR	-	6-15-50	-
	Responses: OV-101: NP90								

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Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
thiobencarb	C12H16ClNOS	0.94	1	0.98	C		V	15	2+3
	Responses: OV-101: NI73 OV-17: HX3								
thiometon	C6H15O2PS3	0.41	0.51	0.4	C	NR	NR	6-15-50	-
	Responses: OV-101: NI10/NI(WB)3/TI1 OV-17: FP0.4/NP(WB)1								
thionazin	C8H13N2O3PS	0.26	-	0.26	C	P	NR	15+50	-
	Responses: OV-101: FP0.5/TI1 OV-17: FP0.5								
thionazin oxygen analog	C8H13N2O4P	-	-	-	-	-	-	-	-
	Responses:								
thiophanate-methyl	C12H14N4O4S2	-	-	-	-	-	-	-	-
	Responses:								
THPI	C8H9NO2	0.21	-	-	C	NR	NR	6-15-50	-
	Responses:								
tolylfluanid	C10H13ClFNOS	1.25	-	1.41	C	-	-	-	-
	Responses: OV-101: NI3/NP70								
toxaphene*	C10H10Cl8	-	2.6	-	C	C	C	6	1
		-	2.35	-					
		-	2.14	-					
		-	1.75	-					
		1.2	2.74	-					
		1.54	3.05	-					
		1.8	3.9	-					
		2.39	4.3	-					
		2.68	4.5	-					
		3.12	5.2	-					
		3.7	5.6	-					
		4.4	6	-					
		4.6	6.4	-					
		5.1	7	-					
	Responses: OV-101: TR30								
tralkoxydim*	C20H27NO3	3.32	-	-	V	NR	NR	50	1-2-3
		6.1	1.48	4.5					
	Responses: OV-101: NI100/NP10 OV-17: NI100 OV-225: NI100								
tralomethrin	C22H19Br4NO3	27	64	44	C	V	S	15	2
	Responses: OV-101: NI30/NI(WB)1								
tri-allate	C10H16Cl3NOS	0.6	-	0.45	C	C	C	6	2
	Responses: OV-101: HX1/NI1.5								

Appendix I: PESTDATA Chemicals in Order by Chemical Name (continued)

Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
triadimefon	C14H16ClN3O2	1.05	1.64	1	C	S #	S #	50	1-2-3
Responses: OV-101: HX3/NI2/NP20/NP(WB)2 OV-17: HX5/HX5/HX(WB)3/NI(WB)2/NP2/NP(WB)3 OV-225: NI3/NP20									
triadimenol	C14H18ClN3O2	1.36	-	1.44	C	NR	NR	6-15-50	-
Responses: OV-101: HX10/NI90/NP30/NP(WB)5 OV-17: HX7/HX(WB)4/NI(WB)50/NP(WB)6									
triazamate	C13H22N4O3S	1.58	2.46	2.1	C	NR	NR	6-15-50	1-2-3
Responses: OV-101: NI15/NP25									
triazophos	C12H16N3O3PS	2.62	-	5.2	C	-	-	-	-
Responses: OV-101: FP(WB)3 OV-17: FP5/FP(WB)5									
tribufos	C12H27OPS3	1.95	1.65	1.88	C	C	P	15+50	3
Responses: OV-101: FP3/NP3/TR4 OV-17: FP3/NI3									
tributyl phosphate	C12H27O4P	0.3	-	0.23	-	R	-	50	-
Responses:									
trichlorfon	C4H8Cl3O4P	0.16	-	0.13	C	NR	NR	6-15-50	1-2-3
Responses: OV-101: NP3 OV-17: FP4									
trichloronat	C10H12Cl3O2PS	1.13	-	0.97	C	C	-	6	-
Responses: OV-101: FP3/HX2/NI2/NP3									
triclopyr methyl ester	C8H6Cl3NO3	0.36	0.36	0.39	-	-	-	-	-
Responses: OV-101: NI0.5/NI(WB)0.7/NP(WB)7									
tricyclazole	C9H7N3S	1.59	-	3.9	C	-	-	-	-
Responses: OV-101: NP500/NP(WB)8 OV-17: NP500/NP(WB)8									
tridiphane	C10H7Cl5O	0.81	0.85	0.75	C	C	-	6	1+2
Responses: OV-101: HX(WB)1/NI0.8 OV-17: HX0.8/HX(WB)0.4 OV-225: NI1									
triflumizole	C15H15ClF3N3O	1.44	2.23	1.19	C	-	-	-	-
Responses: OV-101: NP(WB)4 OV-17: HX13/HX(WB)2/NI(WB)3/NP(WB)3									
trifluralin	C13H16F3N3O4	0.34	0.27	0.17	C	C	C	6	2
Responses: OV-101: TR1 OV-17: NI0.7									
triflusulfuron methyl ester	C17H19F3N6O6S	0.3	-	0.2	V	NR	NR	6-15-50	1-2-3
Responses: OV-101: HN(WB)1/HX(WB)20/NI(WB)500/NP(WB)13 OV-17: HN(WB)1/HX(WB)35/NP(WB)11									
tris(beta-chloroethyl) phosphate	C6H12Cl3O4P	0.45	-	-	C	-	-	-	-
Responses: OV-101: FP1									
tris(chloropropyl) phosphate	C9H18Cl3O4P	0.5	2.02	0.5	C	NR	NR	6-15-50	1-2-3
Responses: OV-101: FP1									

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Name	Molecular Formula	RRT/c OV-101	RRT/c OV-225	RRT/c OV-17	Recoveries 302	303	304	Ethers	CH_2Cl_2
Tycor	C9H16N4OS	0.77	1.62	0.99	C	S	S	50	3
Responses: OV-101: NI1/NP2 OV-17: NI1/NP9 OV-225: NI1									
vamidothion sulfone	C8H18NO6PS2	2.19	-	-	C	-	-	-	-
Responses:									
vernolate	C10H21NOS	0.15	-	0.09	-	P	-	15	-
Responses: OV-101: TI50 OV-17: FP11									
vinclozolin	C12H9Cl2NO3	0.69	1.15	0.64	C	C	C	15	2
Responses: OV-101: HX1.5/NI1 OV-17: HX1/NI2									
vinclozolin metabolite B	C12H11Cl2NO4	0.74	1.2	0.66	C	P #	C	6+15	2
Responses: OV-101: HN(WB)5/HX(WB)18/NI(WB)5/NP(WB)84 OV-17: HN(WB)6/HX(WB)9/NI(WB)10/NP(WB)19 OV-225: NI(WB)8									
vinclozolin metabolite E	C11H11Cl2NO2	0.89	3.02	0.93	C	S	NR	15+50	-
Responses: OV-101: HN(WB)3/HX(WB)3/NI(WB)3/NP(WB)15 OV-17: HN(WB)3/HX(WB)3/NI(WB)4/NP(WB)7 OV-225: NI(WB)9									
vinclozolin metabolite F	C11H13Cl2NO4	2.87	-	4.6	R	NR	NR	6-15-50	1-2-3
Responses: OV-101: HX(WB)160/NI(WB)57/NP(WB)140 OV-17: HN(WB)100/HX(WB)14/NI(WB)85/NP(WB)210									
vinclozolin metabolite S	C10H7Cl2NO3	0.69	2.01	0.79	V	P	V #	15	2
Responses: OV-101: HN(WB)1/HX(WB)2/NI(WB)1/NP(WB)19 OV-17: HN(WB)1/HX(WB)2/NI(WB)1/NP(WB)7 OV-225: NI(WB)2									
zoxamide*	C14H16NO2Cl3	1.44 3.9	-	-	C	C	-	50	3
Responses: OV-101:NI(WB)0.8/NP(WB)18/HX(WB)5									

Appendix I: PESTDATA Chemicals in Order by OV-101 Relative Retention Time

RRT/c OV-101	Detector Responses with OV-101 Column	RRT/c OV-225	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
0.03	TR37	-	0.02	dichlorobenzene, p-	C6H4Cl2	-	C	C	6	1
0.04	TR0.6	-	0.03	dibromochloropropane	C3H5Br2Cl	-	-	-	-	-
0.06	NI30	0.08	0.04	CGA 171683	C6H5F4N3O2	C		-	15+50	3
0.06	FS9	-	-	ethiolate	C7H15NOS	C	-	-	-	-
0.07	FP(WB)0.7	0.25	0.09	methamidophos	C2H8NO2PS	V	-	-	-	-
0.07	FP9/NI1/TI0.5	0.08	0.08	dichlorvos	C4H7Cl2O4P	C	NR	NR	6-15-50	1-2-3
0.08	TR2	-	-	1,2,3-trichlorobenzene	C6H3Cl3	-	C	P	6	1
0.09	TR5	-	-	allidochlor	C8H12ClNO	C	NR	-	6-15	1-2-3
0.1	NI220	-	-	methomyl	C5H10N202S	-	NR	NR	6-15-50	1-2-3
0.1	TR175	-	-	monuron	C9H11ClN2O	-	NR	NR	6-15-50	1-2-3
0.11	TR0.5	-	0.1	dichlobenil	C7H3Cl2N	C	P	C	15	2
0.11	TR25	-	-	neburon	C12H16Cl2N2O	C	NR	NR	6-15-50	1-2-3
0.11	NI(WB)9	0.09	0.11	diuron	C9H10Cl2N2O	C	NR	NR	6-15-50	1-2-3
0.12	TI30	-	-	EPTC	C9H19NOS	-	P	-	15	-
0.12	TR0.4	-	0.06	hexachlorocyclopentadiene	C5Cl6	-	-	-	-	-
0.13	NI14/NI(WB)24/NP35	0.15	0.1	chlorimuron ethyl ester	C15H15ClN4O6S	P	NR	-	-	-
0.13	FS10	0.23	0.11	carboxin sulfoxide	C12H13NO3S	-	NR	NR	6-15-50	1-2-3
0.13	FP2/FP(WB)1	-	0.15	mevinphos, (Z)-	C7H13O6P	C	NR	-	6-15-50	-
0.13	NP(WB)2	-	0.12	propham	C10H13NO2	C	P	P	15	-
0.14	NI(WB)1.5	0.23	0.14	teflubenzuron*	C14H6Cl2F4N2O2	-	NR	NR	6-15-50	1-2-3
0.15	FP(WB)0.9/NP3	0.64	0.19	acephate	C4H10NO3PS	C	-	-	-	-
0.15	TR7	0.24	-	hydroxy chloroneb	C7H6Cl2O2	-	NR	-	6-15	-
0.15	TI50	-	0.09	vernolate	C10H21NOS	-	P	-	15	-
0.15	NI300	0.15	0.14	dimethyl phthalate	C10H10O4	-	P	-	6+15+50	-
0.16	TR35	0.14	-	2,4,5-T BEP ester*	C17H23Cl3O3	-	-	-	-	-
0.16	FP2	-	0.13	mevinphos, (E)-	C7H13O6P	C	NR	NR	6-15-50	-
0.16	NP3	-	0.13	trichlorfon	C4H8Cl3O4P	C	NR	NR	6-15-50	1-2-3
0.17	NP200	-	0.11	CGA 236431	C8H7F3N2O2	-	-	-	-	-
0.17	NI3/NP7	0.22	0.13	3-methyl-4-nitrophenol methyl ether	C8H9O3N	-	-	-	-	-

* Multipeak chemical.

Recovery may vary with choice of Florisil elution system; see Tables 303-a, 304-a.

Appendix I: PESTDATA Chemicals in Order by OV-101 Relative Retention Time (continued)

RRT/c OV-101	Detector Responses with OV-101 Column	RRT/c OV-225	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
0.17	HX20/NI9	0.14	0.1	N-(3,4-dichlorophenyl)-N'-methylurea	C8H8Cl2N2O	-	NR	NR	6-15-50	-
0.17		-	-	metolcarb	C9H11NO2	C	-	-	-	-
0.17	HN(WB)0.4/NP7	-	0.1	pebulate	C10H21NOS	C	P	-	15	-
0.18	NP8	-	-	prosulfuron*	C15H16F3N5O4S	-	NR	NR	6-15-50	1-2-3
0.18	NI200	-	0.22	4-hydroxymethyl-3,5-dimethylphenyl methylcarbamate*	C11H15NO3	-	NR	NR	15-50	1-2-3
0.18	NI0.25	-	-	clopyralid methyl ester	C7H4Cl2NO2	-		-	50	-
0.18	NI0.3/NP0.6	0.12	0.21	etridiazole	C5H5Cl3N2OS	C	C	P	6	2
0.18	HN(WB)1/HX0.5/NI9/ NI(WB)16/NP0.9/NP(WB)1	0.27	0.14	3,5-dichloroaniline	C6H5Cl2N	S	S	S	6+15	1+2
0.19	HX(WB)1.6/NI0.6	0.18	-	dicamba methyl ester	C8H6Cl2O3	-	-	-	-	-
0.19	HX(WB)2/NI600	-	-	MCPA methyl ester	C10H11ClO3	-	-	-	-	-
0.19	HX(WB)2/NI30	-	-	mecoprop methyl ester	C11H13ClO3	-	-	-	-	-
0.19	HX1/NI1/NP1	0.22	0.14	N, N-diallyl dichloroacetamide	C8H11Cl2NO	C	S	S	15+50	2+3
0.19	NI0.5/NI(WB)0.4/NP(WB)9	0.08	0.1	2-methoxy-3,5,6-trichloropyridine	C6H4Cl3NO	C	P#	C	6+15	1+2
0.19		-	-	bis(trichloromethyl)disulfide	C2Cl6S2	-	R	-	6	-
0.19	NI3.5	0.19	-	chloroneb	C8H8Cl2O2	C	C	-	6	2
0.2	HX25/NI20	0.86	0.61	desdiethyl simazine	C3H4ClN5	-	NR	NR	6-15-50	1-2-3
0.2	HX2/NI(WB)0.8/NP(WB)4	0.18	0.2	nitrapyrin	C6H3Cl4N	C	C	V	6	2
0.2		-	0.27	1,2,4-triazole	C2H3N3	V	NR	NR	6-15-50	1-2-3
0.2	HX0.6/NI16/NP1	0.32	0.16	3,4-dichloroaniline	C6H5Cl2N	V	S	-	15	-
0.21	HX9/NI25	-	1.36	3-(3,4-dichlorophenyl)-1-methoxyurea	C8H8Cl2N2O2	R	NR	NR	6-15-50	-
0.21		-	-	THPI	C8H9NO2	C	NR	NR	6-15-50	-
0.21	FP(WB)46/NI(WB)4000	0.27	0.24	TEPP	C8H20O7P2	C	-	-	-	-
0.22	NP100	-	0.14	CGA 72903	C7H6F3N	-	-	-	-	-
0.22	NI170/NP40	0.25	0.2	3-carboxy-5-ethoxy-1,2,4-thiadiazole	C3H2N2O3S	NR	-	-	-	-
0.22	TI6	0.32	0.21	demeton-O oxygen analog	C8H19O4PS	-	-	-	-	-
0.22	HX9/NI18/NP60	0.14	0.1	3,4-dichlorophenylurea	C7H6Cl2N2O	-	NR	NR	6-15-50	-
0.22		-	-	butylate	C11H23NOS	-	-	-	-	-
0.22	NP0.5	-	0.14	CGA 150829	C5H14N4O	V	-	-	-	-
0.23	TR0.6	0.21	0.18	methyl 2,3,6-trichlorobenzoate	C8H5Cl3O2	-	-	-	-	-
0.23	NI3	-	-	disul-Na	C8H7Cl2O5S•Na	-	-	-	-	-

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Appendix I: PESTDATA Chemicals in Order by OV-101 Relative Retention Time (continued)

RRT/c OV-101	Detector Responses with OV-101 Column	RRT/c OV-225	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
0.24	NI0.2	0.15	0.22	2,3,5,6-tetrachloroanisole	C7H4Cl4O	-	C	-	6	1
0.24	HX0.3/NI0.25	0.13	0.16	pentachlorobenzene	C6HCl5	C	C	C	6	1
0.25	NI100	-	-	picloram*	C6H3Cl3N2O2	-	-	-	-	-
0.25	HN(WB)1/NI(WB)23/ NP(WB)10	0.5	0.16	cymoxanil	C7H10N4O3	V	NR	NR	6-15-50	1-2-3
0.25	HN(WB)0.5/NI(WB)4/ NP(WB)4	0.92	0.28	oxamyl oxime metabolite	C5H10N2O2S	C	NR	NR	6-15-50	1-2-3
0.25	FP(WB)0.9/TI25	1.11	0.39	omethoate	C5H12NO4PS	C	NR	NR	6-15-50	1-2-3
0.26	NP20	-	0.13	CGA 236432	C9H9F3N2O2	-	-	-	-	-
0.26	NI0.5	0.51	0.23	RPA 203328, methylated	C10H9F3O4S	-	-	-	-	-
0.26	NI1000/NP(WB)0.5	-	0.21	tebuthiuron	C9H16N4OS	-	-	-	-	-
0.26	FP0.5/TI1	-	0.26	thionazin	C8H13N2O3PS	C	P	NR	15+50	-
0.26	NI3500	-	-	diethyl phthalate	C12H14O4	-	P	P	15+50	-
0.27	NI200	-	0.31	4-hydroxymethyl-3,5-dimethylphenyl methylcarbamate*	C11H15NO3	-	NR	NR	15-50	1-2-3
0.28	FP(WB)2	-	0.36	demeton-O*	C8H19O3PS2	C	NR	-	6-15	-
0.28	HX(WB)1.6/NI2	-	-	dichlorprop methyl ester	C10H10Cl2O3	-	-	-	-	-
0.28	NP3	-	0.29	G-27550	C8H12N2O	C	-	-	-	-
0.28	FP(WB)8	0.49	0.32	metasystox thiol	C6H15O3PS2	C	-	-	-	-
0.29		-	0.25	diphenylamine	C12H11N	C	S	-	6+15	-
0.29	TR0.5	0.26	0.24	tecnazene	C6HCl4NO2	C	C	C	6	1
0.3	TR6	0.38	0.25	2,4-D methyl ester	C9H8Cl2O3	-	-	-	-	-
0.3	NI0.4	-	-	bromoxynil methyl ether	C8H5BR2ON	-	-	-	-	-
0.3	HN(WB)6/NI1	-	-	bromofenoxim methyl ether	C14H9Br2O6N3	-	-	-	-	-
0.3	HN(WB)1/HX(WB)20/ NI(WB)500/NP(WB)13	-	0.2	triflusulfuron methyl ester	C17H19F3N6O6S	V	NR	NR	6-15-50	1-2-3
0.3	HX2/NI0.4/NP8	-	-	2,4-dichloro-6-nitrobenzenamine	C6H4Cl2N2O2	-	R	-	15	2
0.3	NI400/TI2	0.37	0.29	phorate oxygen analog	C7H17O3PS2	C	NR	NR	6-15-50	1-2-3
0.3	FS2/NP15	-	-	cycloate	C11H21NOS	C	V #	S	15+50	3
0.3	HX12/NI20	0.8	0.53	desethyl simazine	C5H8ClN5	-	NR	NR	50	1-2-3
0.3		-	0.23	tributyl phosphate	C12H27O4P	-	R	-	50	-
0.31	FP6	0.49	0.32	metasystox thiono*	C6H15O3PS2	-	-	-	-	-
0.31	FP(WB)0.7/TI15	1.6	0.5	monocrotophos	C7H14NO5P	C	NR	NR	6-15-50	1-2-3

Appendix I: PESTDATA Chemicals in Order by OV-101 Relative Retention Time (continued)

RRT/c OV-101	Detector Responses with OV-101 Column	RRT/c OV-225	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
0.31	FP(WB)0.6/TI10	0.96	0.43	dicrotophos	C8H16NO5P	C	NR	-	6-15-50	-
0.32	NI(WB)0.6/NP(WB)5	0.44	0.36	3, 5, 6-trichloro-2-pyridinol methyl ester	C6H4Cl3NO	-	-	-	-	-
0.32	NI0.4	-	-	fenac methyl ester	C9H7Cl3O2	-	-	-	-	-
0.32		-	-	chlorothalonil trichloro impurity	C8HCl3N2	R	R #	NR	6-15-50	2+3
0.32		-	-	bendiocarb	C11H13NO4	C	-	-	-	-
0.32	HX2	-	-	2-chloroethyl caprate	C8H15ClO2	-	C	C	15	2
0.32	HN(WB)1.5	-	0.41	phenmedipham	C16H16N2O4	-	-	-	-	-
0.32	HX2	0.43	0.25	chlorpropham	C10H12ClNO2	C	C	C	15	2
0.33	FP0.5/HX0.3/NI0.5	0.23	0.24	chlorethoxyfos	C6H11Cl4O3PS	V	C	-	6	1
0.33	FP0.7/TI0.8	0.31	0.25	ethoprop	C8H19O2PS2	C	P #	S #	50	1-2-3
0.34	HX4/HX(WB)7/NI1	0.27	0.19	ethalfluralin	C13H14F3N3O4	C	C	C	6	2
0.34		-	0.25	2,4,5-trichloro-alpha-methylbenzene methanol	C8H7OCl3	R	R	-	15	-
0.34		-	0.36	dioxabenzofos	C8H9O3PS	C	P	-	15	-
0.34	TI0.8	-	0.29	sulfotep	C8H20O5P2S2	C	C	P	6+15	2
0.34	NI(WB)16/NP(WB)13	0.37	0.26	propachlor	C11H14ClNO	C	NR	NR	6-15-50	1-2-3
0.34	TR1	0.27	0.17	trifluralin	C13H16F3N3O4	C	C	C	6	2
0.34	NP11	-	0.32	naled	C4H7Br2Cl2O4P	C	NR	NR	6-15-50	1-2-3
0.35	HN(WB)17/NI(WB)34/ NP(WB)600	2.85	0.4	ethametsulfuron methyl ester*	C15H18N6O6S	-	NR	NR	6-15-50	1-2-3
0.35	HN(WB)30	-	-	DNOC methyl ether	C8H8N2O5	-	-	-	-	-
0.35	HN(WB)0.5/NI320/NP15	0.7	0.41	methabenzthiazuron	C10H11N3OS	C	NR	NR	6-15-50	1-2-3
0.35	NP8	0.6	0.38	2,3,5-trimethacarb	C11H15NO2	C	S #	NR	50	1-2-3
0.36	NI0.5/NI(WB)0.7/NP(WB)7	0.36	0.39	triclopyr methyl ester	C8H6Cl3NO3	-	-	-	-	-
0.37	FP(WB)0.5	0.27	0.29	cadusafos	C10H23O2PS2	C	NR	NR	6-15-50	1-2-3
0.37	HX(WB)1.5/NI(WB)2	0.28	0.18	benfluralin	C13H16F3N3O4	C	C	C	6	2
0.37	FP1/NI(WB)24/TI1	0.38	0.32	phorate	C7H17O2PS3	C	V #	V #	6	1
0.38	NI13/NP50	0.63	0.26	3-methyl-4-nitrophenol	C7H7O3N	V	NR	NR	6-15-50	1-2-3
0.38	NI(WB)1	0.44	0.36	sulfallate	C8H14ClNS2	C	C	C	6+15	2
0.39	NI(WB)20	1.16	0.54	4-chlorobenzylmethyl sulfoxide	C8H9ClOS	-	NR	NR	6-15-50	1-2-3
0.39		1.3	0.52	2,6-dichlorobenzamide	C7H5NOCl2	C	NR	NR	6-15-50	1-2-3

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Appendix I: PESTDATA Chemicals in Order by OV-101 Relative Retention Time (continued)

RRT/c OV-101	Detector Responses with OV-101 Column	RRT/c OV-225	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
0.39		-	-	carbofuran	C12H15NO3	C	-	-	-	-
0.39	NP1	0.53	0.38	fonofos oxygen analog	C10H15O2PS	V	NR	NR	6-15-50	1-2-3
0.4	NP(V)20	-	0.47	CGA 37734	C10H13NO2	C	NR	NR	6-15-50	1-2-3
0.4	HX500/NI300	-	0.71	dazomet	C5H10N2S2	S	NR	-	6-15-50	1-2-3
0.4	TR0.4	0.48	0.35	BHC, alpha-	C6H6Cl6	C	C	C	6	1
0.4	FP(WB)0.7/NI(WB)6/NP1	1.6	0.62	dimethoate	C5H12NO3PS2	C	NR	NR	6-15-50	1-2-3
0.41	FS0.5	2.71	0.81	dimethipin	C6H10O4S2	C	NR	NR	6-15-50	1-2-3
0.41	NI(WB)0.4	1.91	0.66	4-chlorobenzylmethyl sulfone	C8H9ClO2S	-	NR	NR	6-15-50	1-2-3
0.41	NI10/NI(WB)3/TI1	0.51	0.4	thiometon	C6H15O2PS3	C	NR	NR	6-15-50	-
0.41	FP(WB)0.8/TI2	0.56	0.41	demeton-S	C8H19O3PS2	C	NR	-	6-15-50	-
0.41	NI(WB)90	0.83	0.5	simazine	C7H12ClN5	C	NR	NR	50	1-2-3
0.42	HN(WB)5/NI(WB)5/ NP(WB)65	0.97	0.49	PPG-947, methylated*	C18H13ClF3NO7	-	-	-	-	-
0.42	TR10	0.74	0.33	2,4-D isopropyl ester*	C11H12Cl2O3	-	-	-	-	-
0.42	HN(WB)3/HX(WB)42/ NI(WB)35/NP(WB)14	-	0.9	pyrazon metabolite B	C6H4ClN3O	-	NR	NR	6-15-50	1-2-3
0.42	FP0.5/NI1000	-	0.39	terbufos oxygen analog	C9H21O3PS2	C	-	NR	6-15-50	1-2-3
0.42		0.26	0.33	di-allate	C10H17ClNOS	C	C	-	6	-
0.42	HX4	0.75	0.45	chlorbufam	C11H10ClNO2	C		-	15	2+3
0.42	NP100	-	0.42	melamine	C3H6N6	NR	-	-	-	-
0.42	TR0.5	0.96	0.45	dicloran	C6H4Cl2N2O2	C	S	P	15+50	2+3
0.43	HN(WB)0.4/NI(WB)26/ NP(WB)3	1.34	0.6	6-chloro-2,3-dihydro-3,3,7-methyl-5H-oxazolo(3,2-a)pyrimidin-5-one	C9H13ClN2O2	-	NR	NR	6-15-50	1-2-3
0.43	FP100	-	0.62	fenthion oxygen analog sulfoxide	C10H15O5PS	C	NR	NR	6-15-50	1-2-3
0.43	TI58/TR200	0.74	0.44	atrazine	C8H14ClN5	C	S #	NR	50	1-2-3
0.43	TR2	1.62	0.56	BHC, beta-	C6H6Cl6	C	C	C	6	1
0.44	NP8	2.49	0.64	prosulfuron*	C15H16F3N5O4S	-	NR	NR	6-15-50	1-2-3
0.44	HN(WB)0.2/NP(WB)0.5	-	0.82	isocarbamid	C8H15N3O2	C	-	-	-	-
0.44	NI1000/NP300	0.29	0.45	desmedipham	C16H16N2O4	-	-	-	-	-
0.44	HX0.8/NI0.8	1.03	-	chloramben methyl ester	C8H7Cl2NO2	-	-	-	-	-
0.45	NI8/NP35	2.25	0.71	methidathion sulfoxide	C5H8N2O4S2	-	NR	NR	6-15-50	1-2-3
0.45	HX(WB)0.8/NI0.6	0.44	-	silvex methyl ester	C10H9Cl3O3	-	-	-	-	-

Appendix I: PESTDATA Chemicals in Order by OV-101 Relative Retention Time (continued)

RRT/c OV-101	Detector Responses with OV-101 Column	RRT/c OV-225	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
0.45	HX1.5/HX2/NI110	0.59	0.46	clomazone	C12H14ClNO2	C		-	50	3
0.45	NP25	0.78	0.5	3,4,5-trimethacarb	C11H15NO2	C	NR	NR	50	1-2-3
0.45	HX0.5/NI0.25	0.25	0.33	hexachlorobenzene	C6Cl6	C	C	P	6	1
0.45	FP1	-	-	tris(beta-chloroethyl) phosphate	C6H12Cl3O4P	C	-	-	-	-
0.46	NP2	-	0.49	CGA 51702	C9H9F3N2O	-	-	-	-	-
0.46	NI1.5/NP50	0.6	0.39	furilazole	C11H13Cl2NO3	C	S	-	50	3
0.46	HX0.4/TR0.4	0.3	0.34	pentachlorophenyl methyl ether	C7H3Cl5O	C	C	C	6	1
0.46		0.49	0.31	oxydemeton-methyl	C6H15O4PS2	C	-	-	-	-
0.47		0.42	0.53	terbumeton	C10H19N5O	C	-	-	-	-
0.47	NI(WB)87/NP(WB)6	0.71	0.48	terbuthylazine	C9H16N5Cl	C	P	-	15+50	-
0.47	FP(WB)0.7/NI(WB)2/NP1	-	0.59	cyanophos	C9H10O3NSP	C	-	-	-	-
0.47	NI100/TI10	-	0.5	dioxathion	C12H26O6P2S4	V	NR	-	6-15-50	2
0.48	FP1.5	-	0.42	propetamphos	C10H20NO4PS	C	C #	-	15+50	2+3
0.48	HX4	0.91	0.48	monolinuron	C9H11ClN2O2	C	-	-	-	-
0.48	NI40	-	-	silvex	C9H7Cl3O3	-	-	-	-	-
0.48	HX0.6/TR0.5	0.69	0.47	lindane	C6H6Cl6	C	C	C	6	1
0.49	TR1	0.63	0.47	2,4,5-T methyl ester	C9H7Cl3O3	-	-	-	-	-
0.49	NI0.3	0.35	0.48	1,2,4,5-tetrachloro-3-(methylthio)= benzene	C7H4Cl4S	R	C	-	6	1
0.5	NI75	0.79	0.44	metribuzin, deaminated diketo metabolite*	C7H11N3O2	NR	NR	NR	6-15-50	1-2-3
0.5	NI900/NP100	0.53	0.24	phosmet oxygen analog*	C11H12NO5PS	-	NR	NR	6-15-50	-
0.5	NI1.4/NP34	0.69	0.48	4-(dichloroacetyl)-1-oxa-4-azapiro= [4.5]decane	C10H15Cl2NO2	C	P	-	50	3
0.5	FP1	2.02	0.5	tris(chloropropyl) phosphate	C9H18Cl3O4P	C	NR	NR	6-15-50	1-2-3
0.5	NI0.5	0.59	0.45	pentachlorobenzonitrile	C7Cl5N	C	C	P	15	2
0.5	FP2/NI40/TI2	0.44	0.41	terbufos	C9H21O2PS3	C	P	S	6	-
0.5	NI18/NP0.6	0.53	0.47	diazinon oxygen analog	C12H21N2O4P	C	NR	NR	6-15-50	1-2-3
0.5	HX0.5/TR0.4	1.71	0.67	BHC, delta-	C6H6Cl6	C	C	C	6+15	1
0.5	NI2200/NP23	2.33	0.66	ethylenethiourea	C3H6N2S	S	NR	NR	6-15-50	1-2-3
0.51	NI100/NP1	-	-	tebupirimfos oxygen analog	C13H23O4N2P	-	NR	NR	6-15-50	1-2-3
0.51	FP7/NI1000/NP(WB)3.5	0.8	0.63	etrimfos oxygen analog	C10H17N2O5P	C	-	-	-	-

Transmittal No. 2000-1 (10/1999)
Form FDA 2905a (6/92)

Appendix I: PESTDATA Chemicals in Order by OV-101 Relative Retention Time (continued)

RRT/c OV-101	Detector Responses with OV-101 Column	RRT/c OV-225	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
0.51	HX1/NI2/NP7	0.84	0.4	pronamide	C12H11Cl2NO	C	P	-	15+50	-
0.51	FP(WB)1/NI3/NP0.4	0.4	0.44	diazinon	C12H21N2O3PS	C	C	C	15	3
0.51	TR0.3	0.46	0.46	quintozene	C6Cl5NO2	C	C	C	6	1
0.52	NI(WB)1/TI166	0.93	0.44	dinitramine	C11H13F3N4O4	C	-	P	15	-
0.52	TI2	0.56	0.44	fonofos	C10H15OPS2	C	C	C	6	2+3
0.53	FP10	-	0.57	phosphamidon*	C10H19ClNO5P	C	NR	NR	6-15-50	1-2-3
0.53	HX3/NI1.5	0.76	0.37	fluchloralin	C12H13ClF3N3O4	C	C	-	6	2
0.53	HX5/NI1/NP1	0.46	0.3	profluralin	C14H16F3N3O4	V	V	-	6	-
0.53	NI(WB)110/TI30	0.65	0.41	propazine	C9H16ClN5	C	S	NR	15+50	3
0.54	HX2.5/NI1000	-	0.66	4-chloro-6-methoxyindole	C9H8NOCl	-	R	-	15	-
0.54	NP5/NP(WB)6	1.47	0.62	fenfuram	C12H11NO2	C	-	-	-	-
0.54	HN(WB)1/HX5/NI(WB)6/ NP(WB)8	2.1	0.72	terbacil	C9H13ClN2O2	C	NR	NR	6-15	2+3
0.54	NI(WB)15	-	1.07	4-chlorophenylurea	C7H7ClN2O	NR	NR	NR	6-15-50	1-2-3
0.54	TI2	0.6	0.46	disulfoton	C8H19O2PS3	C	P #	NR	6	1-2-3
0.55	HN(WB)17/NI(WB)34/ NP(WB)600	3.6	0.95	ethametsulfuron methyl ester*	C15H18N6O6S	-	NR	NR	6-15-50	1-2-3
0.55	NI30/NP0.4	0.8	0.63	isazofos	C9H17ClN3O3PS	C	C #	-	50	2+3
0.55	HN(WB)17/NI(WB)15/ NP(WB)9	1.41	0.9	3-ketocarbofuran	C12H12NO4	S	NR	NR	6	1
0.55	HX1/NI0.6	1.44	0.74	chlorothalonil	C8Cl4N2	S	C #	C #	6-15-50	2+3
0.55	TI5/TR11	1.71	0.66	parathion-methyl oxygen analog	C8H10NO6P	-	NR	NR	6-15-50	1-2-3
0.55	NI(WB)3	0.86	1.04	tetraiodoethylene	C2I4	-	P	P	6	-
0.55	NI2	0.92	-	dichlone	C10H4Cl2O2	P	S #	S #	6-15-50	2+3
0.56	NI(WB)2	0.49	0.47	methyl 3,5-dibromo-4-methoxy= benzoate	C9H8Br2O3	-	-	-	-	-
0.56	NI150/NP100	2.29	0.82	methidathion sulfone	C5H8N2O3S2	-	NR	NR	6-15-50	1-2-3
0.56	NI35/NP40	1.41	0.55	metribuzin, diketo metabolite	C7H12N4O2	NR	NR	NR	6-15-50	1-2-3
0.56	NI(WB)1	0.4	0.32	chlordene	C10H6Cl6	-	C	C	6	1
0.56	NI0.4	0.63	0.56	2,3,5,6-tetrachloronitroanisole	C7H3Cl4NO3	-	C	-	6	1+2
0.56	NP10	-	-	aminocarb	C11H16N2O2	C	-	-	-	-
0.57	NI0.5/NP2	1.47	0.91	metribuzin	C8H14N4OS	V	NR	NR	50	1-2-3
0.58	NP10	-	0.68	cyromazine	C6H10N6	S	-	-	-	-

Appendix I: PESTDATA Chemicals in Order by OV-101 Relative Retention Time (continued)

RRT/c OV-101	Detector Responses with OV-101 Column	RRT/c OV-225	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
0.58	FP2/NI50	0.59	0.51	etrimfos	C10H17N2O4PS	C	C	C	15	2+3
0.58	FP4/TI5	-	0.52	schradan	C8H24N4O3P2	C	NR	-	6-15-50	-
0.59	NI0.5	0.73	0.66	2,3,5,6-tetrachloroanisidine	C7H5Cl4NO	-	C	-	6	2
0.59	HX2	-	-	2-chloroethyl laurate	C14H27ClO2	-	C	C	15	2
0.6	NI200/NP200	2.65	0.9	CGA 120844	C8H9NSO3	-	NR	NR	6-15-50	1-2-3
0.6	FP20/FP400/FS1.5/ HN(WB)3.5/NI950/NP(WB)4	1.4	0.78	ethiofencarb	C10H15NO2S	C	NR	NR	6-15-50	-
0.6	FP(WB)1	-	0.54	iprobenfos	C13H21O3PS	C	-	-	-	-
0.6	HX1/NI1.5	-	0.45	tri-allate	C10H16Cl3NOS	C	C	C	6	2
0.6	NI20/NP15	1.64	0.7	ethoxyquin	C14H19N0	C	NR	NR	6-15-50	-
0.61	NI2	-	-	fluroxypyr, methylated*	C8H7O3N2Cl2F	-	-	-	-	-
0.61		-	0.73	pirimicarb	C11H18N4O2	C	-	-	-	-
0.62	TR28	0.72	-	2,4-DB methyl ester	C11H12Cl2O3	-	-	-	-	-
0.62	TR5	0.62	0.49	2,4-D isobutyl ester	C12H14Cl2O3	-	-	-	-	-
0.63	HN1/NI1.2	-	-	dinoseb methyl ether	C11H14N2O5	-	-	-	-	-
0.63	NI16/NP1	-	-	tebupirimfos	C13H23N2O3PS	-	V	V	6+15	2+3
0.64	NI1/NP6	1.06	0.7	benoxacor	C11H11Cl2NO2	C	P	C	15+50	2+3
0.64	HX1.5/NI13	1.22	0.74	cyprazine	C9H14ClN5	C	-	-	-	-
0.64	NP3	1.02	0.62	ronnel oxygen analog	C8H8Cl3O4P	C	NR	-	6-15-50	-
0.65	NI20	0.61	0.56	diisobutyl phthalate	C16H2204	-	P	-	15+50	-
0.66	FS4	-	0.64	octhilinone	C11H19NOS	C	-	-	-	-
0.66	FP1/TI10	-	1.02	phorate oxygen analog sulfone	C7H17O5PS2	C	NR	NR	6-15-50	1-2-3
0.66	HX6/NI3/NI(WB)5	2.82	0.78	propanil	C9H9Cl2NO	C	NR	NR	6-15	3
0.67	FP10	-	0.76	phosphamidon*	C10H19ClNO5P	C	NR	NR	6-15-50	1-2-3
0.67	NI100	-	-	picloram*	C6H3Cl3N2O2	-	-	-	-	-
0.67	NP(WB)0.5	-	-	pyrimethanil	C12H13N3	C	S	S #	50	3
0.67	HX8/NI5	1.44	0.69	metobromuron	C9H11BrN2O2	C	NR	NR	6-15-50	1-2-3
0.67	HX0.5/NI0.4/NP10	0.79	0.66	pentachloroaniline	C6H2Cl5N	C	C	C	6	1
0.67	FP1/FP(WB)3.5/NI1.9/TI2	0.64	0.56	dichlofenthion	C10H13Cl2O3PS	C	C	V	6	2
0.67	TR2	0.65	-	2,4,5-T isopropyl ester	C11H11Cl3O3	-	-	-	-	-
0.68	TR35	0.66	-	2,4,5-T BEP ester*	C17H23Cl3O3	-	-	-	-	-
0.68	FS25/NI4	2.89	0.93	2,3-dihydro-3,3-methyl-2-oxo-5-	C11H12O5S	-	-	-	-	-

Appendix I: PESTDATA Chemicals in Order by OV-101 Relative Retention Time (continued)

RRT/c OV-101	Detector Responses with OV-101 Column	RRT/c OV-225	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
				benzofuranyl methyl sulfonate						
0.68	TI7	1.55	0.87	malathion oxygen analog	C10H19O7PS	C	NR	NR	6-15-50	1-2-3
0.69	TR60	0.74	-	2,4-D BEP ester*	C17H24Cl2O4	-	-	-	-	-
0.69	HN(WB)1/HX(WB)2/ NI(WB)1/NP(WB)19	2.01	0.79	vinclozolin metabolite S	C10H7Cl2NO3	V	P	V #	15	2
0.69	HX1.5/NI1	1.15	0.64	vinclozolin	C12H9Cl2NO3	C	C	C	15	2
0.69		-	-	chlorthiamid	C7H5Cl2NS	-	-	-	-	-
0.7	NP13	-	0.58	CP 51214	C14H21NO3	C	NR	NR	6-15-50	1-2-3
0.7	HN(WB)0.6/NP(WB)23	-	0.8	IN-B2838	C10H15N3O3	P	NR	NR	6-15-50	1-2-3
0.71	FP5/TI12	2.95	0.96	demeton-O sulfone*	C8H19O5PS2	C	-	-	-	-
0.71	HN(WB)5/NI1	-	-	ioxynil methyl ether	C8H5I2NO	-	-	-	-	-
0.71	NI30	1.11	0.71	dimethachlor	C13H18ClNO2	C	-	-	-	-
0.71	HN(WB)0.5/NP(WB)3	-	0.95	fuberidazole	C11H8N2O	C	-	-	-	-
0.71	FP(WB)0.9/NI(WB)3/NP1.5	1.64	0.87	parathion-methyl	C8H10NO5PS	C	C	C	15	2
0.72		-	-	DDM	C13H10Cl2	-	-	-	-	-
0.72	NI(WB)19/NP(WB)14	0.98	-	dimethenamid	C12H18ClNO2S	-	NR	NR	6-15-50	1-2-3
0.72	FP(WB)2	-	1.48	oxydemeton-methyl sulfone	C6H15O5PS2	C	-	-	-	-
0.72	FP1/FP(WB)1.3/HX1.5/ NI1/NP1	0.86	0.79	chlorpyrifos-methyl	C7H7Cl3NO3PS	C	C	-	6	2
0.72	FP3	-	0.83	fenitrothion oxygen analog	C9H12NO6P	C	-	-	-	-
0.72	TR40	-	-	2,4-D n-butyl ester	C12H14Cl2O3	-	-	-	-	-
0.73	NI75	1.29	0.52	metribuzin, deaminated diketo metabolite*	C7H11N3O2	NR	NR	NR	6-15-50	1-2-3
0.73	NP6	-	0.67	CP 108064, methylated	C15H21NO4	-	-	-	-	-
0.73	NP2	-	0.89	cymiazole	C12H14N2S	-	-	-	-	-
0.74	HN(WB)5/HX(WB)18/ NI(WB)5/NP(WB)84	1.2	0.66	vinclozolin metabolite B	C12H11Cl2NO4	C	P #	C	6+15	2
0.75	HX(WB)1.2/NI1.5/TR1	2.67	-	picloram methyl ester	C7H5Cl3N2O2	-	-	-	-	-
0.75	HX5/NI9/NP5	0.88	0.67	acetochlor	C14H20NO2Cl	C	C #	P	50	3
0.75	NI2/NP2	0.8	0.68	CGA 14128	C12H21N2O4PS	C		-	50	1-2-3
0.75	FP1/NI5	1.55	0.79	prothoate	C9H20NO3PS2	C	-	-	-	-
0.75	NP60	-	1.05	carbaryl	C12H11NO2	C	-	-	-	-
0.77	NI1/NP2	1.62	0.99	Tycor	C9H16N4OS	C	S	S	50	3

Appendix I: PESTDATA Chemicals in Order by OV-101 Relative Retention Time (continued)

RRT/c OV-101	Detector Responses with OV-101 Column	RRT/c OV-225	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
0.77	FP200/NI40/TI50	-	0.74	prometryn	C10H19N5S	C	P #	P #	50	1-2-3
0.77		1.1	-	ametryn	C9H17N5S	C	-	-	-	-
0.78	NI300	-	1.06	phorate oxygen analog sulfoxide	C7H17O4PS2	C	NR	NR	6-15-50	1-2-3
0.78	FP7	-	1.12	fenthion oxygen analog	C10H15O4PS	C	NR	NR	6-15-50	1-2-3
0.78	NI0.5/NP7.5	-	-	bromoxynil butyrate	C11H9Br2NO2	-	V	-	15+50	2
0.79	NI2	-	-	fluroxypyr, methylated*	C8H7O3N2Cl2F	-	-	-	-	-
0.8	HN(WB)1.2/HX(WB)90/ NI(WB)1.5/NP(WB)10	2.1	-	bromacil methyl ether	C10H16BrN2O2	-	-	-	-	-
0.8	NP200	-	1.03	3-hydroxymethyl-2,5-dimethylphenyl methylcarbamate	C11H15NO3	-	NR	NR	6-15-50	1-2-3
0.8	NI(WB)7	1	0.72	alachlor	C14H2OClNO2	C	C	C #	50	3
0.8	NI200/NP200	-	1.17	methiocarb sulfone	C11H15NO4S	S	NR	NR	6-15-50	1-2-3
0.8	HN(WB)2/NI(WB)2/ NP(WB)17	4.8	1.36	bromacil	C9H13BrN2O2	C	NR	NR	6-15-50	1-2-3
0.8	NI(WB)5/NP3	-	0.86	parathion oxygen analog	C10H14NO6P	C	NR	NR	6-15-50	1-2-3
0.81	HX(WB)1/NI0.8	0.85	0.75	tridiphane	C10H7Cl5O	C	C	-	6	1+2
0.81	HX1000/NI1000/NP7	-	0.9	metalaxyl	C15H21NO4	C	NR	NR	6-15-50	1-2-3
0.81	HX16/NI300	0.85	-	chloroxuron	C15H15ClN2O2	C	NR	NR	6-15-50	1-2-3
0.81	NI(WB)1/TI3	0.86	0.76	ronnel	C8H8Cl3O3PS	C	C	C	6	2
0.82	TR2	0.64	0.67	chlordene, alpha-	C10H6Cl6	-	-	-	-	-
0.82	TR4	1.07	0.92	dichlorobenzophenone, o,p'-	C13H8Cl2O	-	C	C	15	2
0.83	TR5	-	-	Compound K*	C10H6Cl8	-	C	-	-	1
0.83		-	-	DDNU	C14H10Cl2	-	-	-	-	-
0.83		-	-	DDNS	C14H12Cl2	-	-	-	-	-
0.83	NI60/NP45	3.77	1.06	metribuzin, deaminated metabolite	C8H13N3OS	C	NR	NR	6-15-50	1-2-3
0.83	NI0.6	0.52	0.6	heptachlor	C10H5Cl7	C	C	C	6	1
0.84	NI50	-	-	sethoxydim sulfoxide	C17H29NO4S	-	NR	NR	6-15-50	3
0.84	NI0.6	0.65	-	chlordene epoxide	C10H6Cl6O	-	C	-	15	-
0.84		1.08	-	terbutryn	C10H19N5S	C	-	-	-	-
0.84	FP(WB)1/NP1	1.82	1.05	fenitrothion	C9H12NO5PS	C	C	C	15	2
0.85	TR28	2.13	0.95	linuron	C9H10Cl2N2O2	V	V #	V	50	3
0.86	NI5	-	-	dicofol, o,p'-*	C14H9Cl5O	C	V	S	6+15	2

Appendix I: PESTDATA Chemicals in Order by OV-101 Relative Retention Time (continued)

RRT/c OV-101	Detector Responses with OV-101 Column	RRT/c OV-225	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
0.86	HN(WB)4/HX(WB)11/ NI(WB)28/NP(WB)17	-	1.55	6-chloro-2,3-dihydro-7-hydroxy= methyl-3,3-methyl-5H-oxazolo= (3,2-a)pyrimidin-5-one	C9H13ClN2O3	-	NR	NR	6-15-50	1-2-3
0.86	FS32/NI315	1.93	1.02	ethofumesate	C13H18O5S	C	-	-	-	-
0.86	FP5	-	-	phoxim oxygen analog	C12H15N204P	C	-	-	-	-
0.87	HX7/NI1100	-	1.07	4-chlorophenoxyaniline*	C12H10ClNO	S	-	-	-	-
0.87	FP4	-	1.05	demeton-O sulfoxide	C8H15O4PS2	C	-	-	-	-
0.87	FP2/FP(WB)1.2/NI100/NP2	-	0.92	pirimiphos-methyl	C11H20N3O3PS	C	C	C	15	3
0.88		-	-	methiocarb	C11H15NO2S	C	-	-	-	-
0.88	NI30	0.92	0.84	dibutyl phthalate	C16H22O4	-	C	C	15+50	-
0.89	HN(WB)3/HX(WB)3/ NI(WB)3/NP(WB)15	3.02	0.93	vinclozolin metabolite E	C11H11Cl2NO2	C	S	NR	15+50	-
0.89	NI4/TI26	4.9	1.48	cyanazine	C9H13ClN6	C	NR	-	6-15-50	-
0.89	FP5/NI8/NP4	2.55	1.26	phorate sulfoxide	C7H17O3PS3	C	NR	NR	6-15-50	1-2-3
0.9	NP400	-	0.45	formetanate hydrochloride	C11H16ClN3O2	-	-	-	-	-
0.9	NI1/NP44	1.71	1.01	dichlofluanid	C9H11Cl2FN2O2S2	C	C #	-	15+50	2+3
0.91	TR850	-	-	diisooctyl phthalate*	C24H38O4	-	C	C	15+50	-
0.91	NI(WB)7/NP1	1.49	1.05	malathion	C10H19O6PS2	C	C	C	15+50	3
0.92	FP7/NI40	2.9	1.28	terbufos oxygen analog sulfone	C9H21O5PS2	C	NR	NR	6-15-50	1-2-3
0.93	FP(WB)5	-	1.43	des N-isopropyl isofenphos oxygen analog	C12H18NO5P	-	-	-	-	-
0.94	TR1	-	-	2,4,5-T isobutyl ester	C12H13Cl3O3	-	-	-	-	-
0.94		0.97	0.66	prodiamine	C13H17F3N4O4	C	-	-	-	-
0.94	NI73	1	0.98	thiobencarb	C12H16ClNOS	C		V	15	2+3
0.94	HX0.7/NI0.4	0.69	0.87	pentachlorophenyl methyl sulfide	C7H3Cl5S	C	C	C	6	1
0.95	FP27/NI6/TI8	1.51	1.08	chlorpyrifos oxygen analog	C9H11Cl3NO4P	C	NR	-	6-15-50	-
0.96	HX5/NI1	1.45	0.86	nitrofluorfen	C13H7ClF3NO3	C	C	C	15	2
0.96		-	1.32	carbetamide	C12H16N2O3	-	-	-	-	-
0.96	FP2/TI4	1.46	1.18	fenthion	C10H15O3PS2	C	S #	NR	6+15	1-2-3
0.97	HX13/NI40	1.46	-	methazole	C9H6Cl2N2O3	-	-	-	-	-
0.97	HN(WB)5	-	0.96	difenoxuron	C16H18N2O3	-	-	-	-	-
0.97	NI4/TI4	3.26	1.3	phorate sulfone	C7H17O4PS3	C	S #	S #	6-15-50	3
0.98	TR1	0.84	0.89	chlordene, beta-	C10H6Cl6	-	-	-	-	-

Appendix I: PESTDATA Chemicals in Order by OV-101 Relative Retention Time (continued)

RRT/c OV-101	Detector Responses with OV-101 Column	RRT/c OV-225	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
0.98	TR2	0.89	0.88	chlordene, gamma-	C10H6Cl6	-	-	-	-	-
0.98	TI340	-	-	desmethyl diphenamid	C15H15NO	-	-	-	-	-
0.98	NI(WB)4/NP2	1.91	1.07	parathion	C10H14NO5PS	C	C	C	15	2
0.99	NI1	-	-	benazolin methyl ester	C9H6O3SNCl	-	-	-	-	-
0.99	NI3/NP35	1.91	0.96	KWG 1323	C14H16ClN3O3	C	NR	NR	6-15-50	1-2-3
0.99	NI(WB)7	1.63	1.07	1-hydroxychlordene	C10H6Cl6O	-	R	-	15	-
0.99	TR3	1.25	1.08	dichlorobenzophenone, p,p'-	C13H8Cl2O	-	C	C	15	2
1	FS48/NI135	6.6	1.46	2-hydroxy-2,3-dihydro-3,3-methyl-5-benzofuranyl methyl sulfonate	C11H14O5S	-	-	-	-	-
1	NI1.5/TI3	1	1	chlorpyrifos	C9H11Cl3NO3PS	C	C	P	6	2
1.01	FP4/FP(WB)1.6	-	1.14	pirimiphos-ethyl oxygen analog	C13H24N3O4P	C	-	-	-	-
1.03	HX5/NI12	1.21	0.93	metolachlor	C15H22ClNO2	C	S #	NR	50	1-2-3
1.04	NI5	-	-	dicofol, p,p'-*	C14H9Cl5O	C	V	P #	6+15	1+2
1.04	HN(WB)620/NI(WB)1000	-	1.13	PPG-947	C17H11ClF3NO7	-	NR	NR	6-15-50	1-2-3
1.05	NI220	1	-	PP 890	C9H10O2ClF3	-	-	-	-	-
1.05	HN(WB)170/HX(WB)980/NI(WB)40/NP(WB)270	1.47	0.88	acifluorfen	C14H7ClF3NO3	-	NR	NR	6-15-50	1-2-3
1.05	HX3/NI2/NP20/NP(WB)2	1.64	1	triadimefon	C14H16ClN3O2	C	S #	S #	50	1-2-3
1.05	HN(WB)1.4	-	1.43	pyracarbolid	C13H15NO2	-	-	-	-	-
1.05	TR0.8	0.58	0.76	aldrin	C12H8Cl6	C	C	C	6	1
1.06	NI1	1.13	1	DCPA	C10H6Cl4O4	C	C	C	15	2
1.07	NI100/NP40	-	1.8	methidathion oxygen analog	C6H11N2O5PS2	-	NR	NR	6-15-50	1-2-3
1.07		-	-	3-chloro-5-methyl-4-nitro-1H-pyrazole	C4H4ClN3O2	C	-	-	-	-
1.08	TR35	0.91	-	2,4,5-T BEP ester*	C17H23Cl3O3	-	-	-	-	-
1.08	FP100/NI1000/NP40	3	1.66	fosthiazate	C9H18NO3PS2	C	NR	NR	6-15-50	1-2-3
1.08	TI6	2.33	1.3	crufomate	C12H19ClNO3P	C	NR	NR	6-15-50	-
1.09	NP33	-	0.62	fenpropimorph	C20H33NO	C		-	50	1-2-3
1.1	NI(WB)2	-	0.68	nitrothal-isopropyl	C14H17O6N	C	-	-	-	-
1.1		-	1.55	diphenamid	C16H17NO	V	NR	-	6-15	-
1.1	TR1	-	-	2,4,5-T n-butyl ester	C12H13Cl3O3	-	-	-	-	-
1.11	NI40/NP100	4.7	1.33	isoxaflutole (prop)	C15H12SNO4F3	NR	V #	S #	50	3
1.11	FP3/NI(WB)1/TI3	1.29	1.16	bromophos	C8H8BrCl2O3PS	C	C	C	6	-

Appendix I: PESTDATA Chemicals in Order by OV-101 Relative Retention Time (continued)

RRT/c OV-101	Detector Responses with OV-101 Column	RRT/c OV-225	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
1.13	NI40/NP120	4.7	1.38	RPA202248	C15H12SNO4F3	NR	NR	NR	6-15-50	1-2-3
1.13	FP3/HX2/NI2/NP3	-	0.97	trichloronat	C10H12Cl3O2PS	C	C	-	6	-
1.14	NI2/NP3	1.24	1.01	isopropalin	C15H23N3O4	C	C	-	6	-
1.14	FP2/TI4/TR150	1.03	1.14	pirimiphos-ethyl	C13H24N3O3PS	C	C	C	15+50	3
1.15	NI3	4.3	1.54	CGA 91305	C10H8Cl2N3O	V	NR	NR	6-15-50	1-2-3
1.15	NI7/NP3	1.22	0.93	butralin	C14H21N3O4	V	C	-	6+15+50	-
1.15	FP40/TI20	5.8	1.75	demeton-S sulfone	C8H19O5PS2	C	-	-	-	-
1.16	HN(WB)1.4	-	1.56	phenothiazine	C12H9NS	-	-	-	-	-
1.17	HX4	-	-	2-chloroethyl myristate	C16H31ClO2	C	V	V	15	2
1.17	FP10	1.74	1.24	isofenphos oxygen analog	C15H24NO5P	C	-	-	-	-
1.17	MC25/NI200	-	-	methyl 4-chloro-1H-indole-3-acetate	C11H10ClNO2	R	R #	NR	50	1-2-3
1.18	NP(WB)2	-	1.39	cyprodinil	C14H15N3	C	NR	NR	6-15-50	1-2-3
1.19	TR12	1.15	1.2	TDE, o,p'-, olefin	C14H9Cl3	-	-	-	-	-
1.2	FP2/NI5	-	1.58	terbufos sulfone	C9H21O4PS3	C	C #	C #	6-15-50	2+3
1.2	TR2	3.49	1.85	captan	C9H8Cl3NO2S	C	P	C	50	3
1.21	FP2	2.73	1.5	des N-isopropyl isofenphos	C12H18NO4PS	C	S	-	50	-
1.21	FP10/FP(WB)1.7/ NI(WB)2/TI4	1.58	1.29	chlorfenvinphos, alpha-	C12H14Cl3O4P	C	-	NR	6-15-50	-
1.22	NI3/NP5	1.48	1.21	pendimethalin	C13H19N3O4	C	C	P	15	2
1.23	NI(WB)1	3.01	1.94	folpet	C9H4Cl3O2NS	C	C	P	15+50	2+3
1.24	NP(WB)3	-	1.32	penconazole	C13H15Cl2N3	C	-	-	-	-
1.24	HX(WB)8/NI4	1.88	1.47	anilazine	C9H5Cl3N4	V	S	P	15+50	2+3
1.25	NI2/NP8	8	1.09	MB45950	C12H4SN4F6Cl2	S	P	V	15+50	2+3
1.25	NI3/NP70	-	1.41	tolylfluanid	C10H13ClFNOS	C	-	-	-	-
1.26	TR4	3.5	1.92	Sulphenone	C12H9ClO2S	C		-	50	3
1.27	HX12/NI19	3.39	1.42	chlorbromuron	C9H10BrClN2O2	V	V	V	50	3
1.28	HX7/NI1100	-	1.31	4-chlorophenoxyaniline*	C12H10ClNO	S	-	-	-	-
1.28	NI(WB)70/NP(WB)90	-	2.41	hexythiazox	C17H21ClN2O2S	-	S #	NR	50	2+3
1.28	NI1000	-	1.6	3-phenoxybenzenemethanol	C13H12O2	-	-	-	-	-
1.28	FP(WB)1.7/NI5	2.67	1.58	mecarbam	C10H20NO5PS2	C		-	50	-
1.29	FP2/FP(WB)1.8/HX3/ NI(WB)2/TI4	2	1.52	chlorfenvinphos, beta-	C12H14Cl3O4P	C	S #	-	50	1-2-3

Appendix I: PESTDATA Chemicals in Order by OV-101 Relative Retention Time (continued)

RRT/c OV-101	Detector Responses with OV-101 Column	RRT/c OV-225	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
1.29	HX0.7/TR1	1.22	1.15	heptachlor epoxide	C10H5Cl7O	C	C	C	6	2
1.3	NI50	8.9	-	chlorsulfuron	C12H12ClN5O4S	-	NR	NR	6-15-50	-
1.31	FP(WB)2/NI5/TI3	2.05	1.83	phenthoate	C12H17O4PS2	C	C	-	15+50	-
1.32	FP5/FP(WB)2.7	2	1.64	quinalphos	C12H15N2O3PS	C	C	-	15	-
1.33	TR1	0.94	1.05	octachlor epoxide	C10H4Cl8O	C	C	C	6	1
1.34	FP3/TI5	0.65	1.43	merphos*	C12H27PS3	-	C	C	6+15+50	3
1.35	HN(WB)4/HX(WB)40/ NI(WB)67/NP(WB)39	2.27	2.55	3-tert-butyl-5-chloro-6-hydroxy= methyluracil	C9H13ClN2O3	-	NR	NR	6-15-50	1-2-3
1.35	NI2/NP10	8.7	1.16	fipronil	C12H4Cl2F6N4OS	S	S	V	50	3
1.36	HX10/NI90/NP30/NP(WB)5	-	1.44	triadimenol	C14H18ClN3O2	C	NR	NR	6-15-50	-
1.36	FP2/NI20	1.73	1.38	isofenphos	C15H24NO4PS	C	C	-	15+50	-
1.36	NI8	1.22	-	allethrin	C19H26O3	-	C	C #	50	3
1.37	NI12	3.04	1.49	procymidone	C13H11Cl2NO2	C	C	P	15	-
1.37	NI2	1.22	1.12	S-bioallethrin	C19H26O3	-	C	-	50	-
1.37	NI60/TI10	2.85	1.9	crotoxyphos	C14H19O6P	C	NR	NR	6-15-50	1-2-3
1.39	NI1000	1.89	1.54	CGA 189138	C13H8O3Cl2	-	-	-	-	-
1.39	NI6	1.62	1.54	chlorbenside	C13H10Cl2S	C	S	P	6	1
1.4	NI500/NP500	-	-	WAK4103*	C9H9N5O3Cl	-	NR	NR	6-15-50	1-2-3
1.4	HN(WB)1.5	-	1.32	dinobuton	C14H18N2O7	C	-	-	-	-
1.4	FP5/FP(WB)1.6/NP3	3.33	2.28	methidathion	C6H11N2O4PS3	C	S	P #	50	3
1.41	HN(WB)1.6/NP(WB)51	-	2.55	IN-T3936	C10H15N3O4	S	NR	NR	6-15-50	1-2-3
1.42	NI2800	3.7	-	fenac	C8H5Cl3O2	-	NR	NR	6-15-50	-
1.43	TR2	-	-	photodieldrin B	C13H9Cl5O	-	-	-	-	-
1.44	NP(WB)4	2.23	1.19	triflumizole	C15H15ClF3N3O	C	-	-	-	-
1.44	TR60	-	-	4-(2,4-dichlorophenoxy)benzenamine	C12H9Cl2NO	-	-	-	-	-
1.44	NI(WB)0.8/NP(WB)18/ HX(WB)5	-	-	zoxamide*	C14H16NO2Cl3	C	C	-	50	3
1.45	NI(WB)4	1.36	1.45	TDE, p,p'-, olefin	C14H9Cl3	C	C	C	6	1
1.47		-	-	DDMU	C14H9Cl3	-	-	-	-	-
1.48	NP90	-	2.04	thiabendazole	C10H7N3S	C	NR	-	6-15-50	-
1.49	TR1	1.46	1.34	chlordane, trans-	C10H6Cl8	C	C	C	6	1
1.5	NI34	-	1.46	metazachlor	C14H16ClN3O	C	-	-	-	-

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Appendix I: PESTDATA Chemicals in Order by OV-101 Relative Retention Time (continued)

RRT/c OV-101	Detector Responses with OV-101 Column	RRT/c OV-225	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
1.5		7.9	-	procyazine	C10H13ClN6	C	-	-	-	-
1.5	NI3/NP22	4.3	2.67	TCMTB	C9H6N2S3	C	P	P	15	-
1.5	TI7	6.7	2.39	disulfoton sulfone	C8H19O4PS3	C	NR	-	6-15-50	-
1.51	FP3/NI3/TI4	1.42	1.45	bromophos-ethyl	C10H12BrCl2O3PS	C	C	P	6	-
1.52	NP(WB)6	-	1.59	paclobutrazol	C15H20ClN3O	C	-	-	-	-
1.53	NI5/NP150	6.5	2.41	CGA 94689A	C15H21NO5	V	NR	NR	6-15-50	1-2-3
1.53	TR40	0.95	-	Perthane olefin	C18H19Cl	-	C	C	6	1
1.54	TR20	3.6	-	2,4-D propylene glycol butyl ether ester*	C15H2OCl2O4	-	-	-	-	-
1.54	NI12/NP150	6.6	2.45	CGA 94689B	C15H21NO5	S	NR	NR	6-15-50	1-2-3
1.55	HX(WB)9/NI5	-	1.4	haloxyfop methyl ester	C16H13ClF3NO4	-	-	-	-	-
1.55	TR2	1.28	1.51	DDE, o,p'-	C14H8Cl4	C	C	C	6	1
1.57	NI5/NP30	1.84	1.64	cyclanilide methyl ester	C12H11Cl2NO3	-	-	-	-	-
1.57	NI(WB)1	-	1.85	oxythioquinox	C10H6N2OS2	C	-	-	-	-
1.58	NI15/NP25	2.46	2.1	triazamate	C13H22N4O3S	C	NR	NR	6-15-50	1-2-3
1.58	FP9/TI8	2.72	1.97	Gardona	C10H9Cl4O4P	C	NR	NR	6-15-50	1-2-3
1.58	TI400	-	-	promecarb	C12H17NO2	V	-	-	-	-
1.59	NI550/NP210	-	-	NTN35884*	C9H9N5O2Cl	-	NR	NR	6-15-50	1-2-3
1.59	NP500/NP(WB)8	-	3.9	tricyclazole	C9H7N3S	C	-	-	-	-
1.6	FS(WB)10/NI(WB)3	4.1	3.17	isoprothiolane	C12H18O4S2	C	-	-	-	-
1.6		-	-	MGK 264	C17H25NO2	-	-	-	-	-
1.61	HX4/TR3	3.04	2.2	ovex	C12H8Cl2O3S	C	C	C	15	2
1.64	HX1/NI1.3	1.38	1.47	endosulfan I	C9H6Cl6O3S	C	C	C	15	2
1.65		-	1.65	DDMS	C14H11Cl3	-	R	-	6	-
1.66	TR60	1.18	-	2,4-D BEP ester*	C17H24Cl2O4	-	-	-	-	-
1.66	FP8/NP8	3.7	2.41	fenamiphos	C13H22NO3PS	C	NR	NR	6-15-50	1-2-3
1.66	TR1	1.54	1.48	chlordane, cis-	C10H6Cl8	C	C	C	6	1
1.7	NP40	-	2.12	napropamide	C17H20NO2	C	-	-	-	-
1.73	HX9	1.83	1.46	butachlor	C17H26ClNO2	C	C	-	50	-
1.73	HX8/NI3	-	1.88	chlorflurecol methyl ester	C15H11ClO3	C	-	-	-	-
1.75	TR2	1.45	1.42	nonachlor, trans-	C10H5Cl9	C	C	C	6	1
1.76	NI30/NP(WB)12	4	2.08	imazalil	C14H14Cl2N2O	C	NR	NR	6-15-50	-

Appendix I: PESTDATA Chemicals in Order by OV-101 Relative Retention Time (continued)

RRT/c OV-101	Detector Responses with OV-101 Column	RRT/c OV-225	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
1.77	FP(WB)4	-	-	fenthion oxygen analog sulfone*	C10H15O6PS2	-	-	-	-	-
1.78	HX11/NI10/NP180	3.14	2	diethatyl-ethyl	C16H22ClNO3	C	NR	NR	6-15-50	1-2-3
1.79	HX540/HX(WB)50/NI45/ NP(WB)40	3.5	2.76	imazamethabenz methyl ester*	C16H20N2O3	C	-	-	-	-
1.8	NP100	-	2.96	CGA 100255	C15H12NO5	S	-	-	-	-
1.8	FP7/FP(WB)2.6/NI3	2.34	2.13	profenofos	C11H15BrClO3PS	C	P	P	50	3
1.82	TR12	2.08	1.79	2,4-D butoxyethyl ester*	C14H18Cl2O4	-	-	-	-	-
1.84	NI200/NP50	-	-	imidacloprid	C9H10ClN5O2	-	NR	NR	6-15-50	1-2-3
1.85	FP4/NI3/NP3	1.74	1.82	prothiofos	C11H15Cl2PO2S2	C	C	C	6	2
1.86	HX6/NI3	2.91	1.79	hexaconazole	C14H17Cl2N3O	C	-	-	-	-
1.87	FS50	-	-	carboxin	C12H13NO2S	C	NR	NR	6-15-50	-
1.88	NI25	-	1.99	pretilachlor	C17H26ClNO2	C	-	-	-	-
1.88	NP1	4.1	-	ethion oxygen analog	C9H22O5P2S3	C	-	-	-	-
1.9	HN(WB)58/HX(WB)110/ NI(WB)85/NP(WB)150	3.16	1.86	PPG-2597	C20H17ClF3NO6	-	NR	NR	6-15-50	1-2-3
1.9	HN(WB)0.7/NI25/NP(WB)7	7.2	2.6	myclobutanil	C15H17ClN4	C	NR	NR	6-15-50	1-2-3
1.9	TR2	2.46	2.19	TDE, o,p'-	C14H10Cl4	-	C	C	6	1
1.91	HX1/NI1.5	1.87	1.84	dieldrin	C12H8Cl6O	C	C	C	15	2
1.92	NI1.5	1.59	1.86	DDE, p,p'-	C14H8Cl4	C	C	C	6	1
1.94	HX8/NI7	-	2.45	flamprop-methyl	C17H15ClFNO3	C	-	-	-	-
1.95	FP3/TI5	1.64	1.88	merphos*	C12H27PS3	-	C	C	6+15+50	3
1.95	FP3/NP3/TR4	1.65	1.88	tribufos	C12H27OPS3	C	C	P	15+50	3
1.97	NP(WB)5	-	2.33	flusilazole	C16H15F2N3Si	C	-	-	-	-
1.97	HX4/NI4	2.48	1.96	oxadiazon	C15H18Cl2N2O3	C	C	P	15	-
1.99	TI45	-	3.8	fensulfothion oxygen analog sulfone	C11H17O7PS2	-	-	-	-	-
2	TR60	1.79	-	2,4-D BEP ester*	C17H24Cl2O4	-	-	-	-	-
2	NI900/NP100	0.93	0.44	phosmet oxygen analog*	C11H12NO5PS	-	NR	NR	6-15-50	-
2	FP600/TR10000	2.77	-	aramite*	C15H23ClO4S	C	P	NR	15	-
2	NI5	4	2.16	oxyfluorfen	C15H11ClF3NO4	C	C	C	15	2
2	FS(WB)20/NI(WB)8	3.7	2.6	bupirimate	C13H24N4SO3	C	-	-	-	-
2	NI(WB)10/NP(WB)50	3.02	3.38	kresoxim-methyl	C18H19NO4	P	C	C	15+50	3
2.02	HX7/NI7/NP(WB)8	3.4	2.03	diclobutrazol	C15H19Cl2N3O	C	NR	NR	6-15-50	1-2-3

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Appendix I: PESTDATA Chemicals in Order by OV-101 Relative Retention Time (continued)

RRT/c OV-101	Detector Responses with OV-101 Column	RRT/c OV-225	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
2.02	NP7	1.21	-	simetryn	C8H15N5S	C	-	-	-	-
2.03	NI(WB)1	3.8	2.71	nitrofen	C12H7Cl2NO3	C	C	C	15	2
2.04	TR50	1.78	1.78	2,4-D isooctyl ester*	C16H22Cl2O3	-	-	-	-	-
2.04	HN(WB)182/HX(WB)73/ NI(WB)72/NP(WB)12	1.61	2.69	cyproconazole	C15H18ClN3O	C	NR	NR	6-15-50	1-2-3
2.06	NI2/NP30	31	1.98	MB46136	C12H4SO2N4F6Cl2	S	S	V	50	2+3
2.07	NP189	-	2.92	methoprotryne	C11H21N5OS	C	-	-	-	-
2.1	NI5	1.78	1.68	2,4-D ethyl hexyl ester*	C16H22Cl2O3	-	-	-	-	-
2.12	NI25	1.76	1.76	pyrethrins*	C21H27O4	-	C	C	50	-
2.13	TR2	2.22	2.29	endrin	C12H8Cl6O	C	C #	C #	15	2
2.14	HN(WB)5/NI(WB)5/ NP(WB)65	5	2.4	PPG-947, methylated*	C18H13ClF3NO7	-	-	-	-	-
2.14	NI100	-	-	mirex, 5,10-dihydro-*	C10H2Cl10	-	-	-	-	-
2.14	FP600/TR10000	3.05	-	aramite*	C15H23ClO4S	C	P	NR	15	-
2.15	HN(WB)3/HX(WB)10/ NI(WB)3/NP(WB)34	5	2.4	PPG-847, methylated	C15H9ClF3NO3	-	-	-	-	-
2.17	NI6/TI15	4.2	3.06	carbophenothion oxygen analog	C11H16ClO3PS2	C	NR	NR	6-15-50	1-2-3
2.19		-	-	vamidothion sulfone	C8H18NO6PS2	C	-	-	-	-
2.19	NI(WB)1	4.2	2.38	binapacryl	C15H18N2O6	C	P	P	15	-
2.21	NI2/NP50	-	2.34	chlorfenapyr (prop)	C15H11BrClF3N2O	P	-	S	50	2
2.21	HX2/NI2	3.9	2.77	endosulfan II	C9H6Cl6O3S	C	C	C	15+50	2
2.22	HX9/NI6	4.1	2.99	chlorthiophos oxygen analog	C11H15Cl2O4PS	C	NR	NR	6-15-50	1-2-3
2.23	TR150	2.01	2.42	Perthane	C18H2OCl2	C	C	C	6	1
2.24	FP8	-	2.58	chlorthiophos*	C11H15Cl2O3PS2	C	C	C	6	2
2.24	NI20	-	-	metamitron	C10H10N4O	-	-	-	-	-
2.26	FP54	-	4.4	famphur oxygen analog	C10H16NO6PS	C	-	-	-	-
2.28	NI1.5	-	-	methyl 2,3,5-triiodobenzoate	C8H5I3O2	-	-	-	-	-
2.29	FP(WB)4	-	4.1	fenthion oxygen analog sulfone*	C10H15O6PS2	-	-	-	-	-
2.3	HX19/HX(WB)18/NI125	2.36	2.31	fluazifop butyl ester	C19H20F3NO4	C	C	V	15	3
2.31	NI20/NP50	15	3.93	desisopropyl iprodione	C10H6Cl2N3O3	P		-	50	1-2-3
2.31	TR70	3.26	2.61	chlorobenzilate	C16H14Cl2O3	C	C #	P #	15+50	3
2.33	TR8	5.8	-	Dilan*	C15.5H14Cl2NO2	-	P	P	15	-

Appendix I: PESTDATA Chemicals in Order by OV-101 Relative Retention Time (continued)

RRT/c OV-101	Detector Responses with OV-101 Column	RRT/c OV-225	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
2.33	TR80	2.9	2.41	chloropropylate	C17H16Cl2O3	P	C	C	15+50	3
2.35	TR4	-	-	endrin aldehyde	C12H8Cl6O	C	P	C	15+50	-
2.35	HX10	-	-	2-chloroethyl palmitate	C18H35ClO2	-	V	P	15	2
2.36	FP8	-	2.77	chlorthiophos*	C11H15Cl2O3PS2	C	C	C	6	2
2.36	NP(WB)12	-	-	etaconazole*	C14H15Cl2N3O2	C	-	-	-	-
2.37		-	-	2,4,5-T propylene glycol butyl ether esters	C15H19Cl3O4	-	-	-	-	-
2.38	NI(WB)2/TI5	3.24	3.14	leptophos photoproduct	C13H11Cl2O2PS	C	-	-	-	-
2.39	FP22	-	4.7	fenthion sulfone	C10H15O5PS2	C	NR	NR	6-15-50	1-2-3
2.4	NI120/NP50	4.3	3	imazethapyr ammonium salt methyl ester	C16H21N3O3	-	-	-	-	-
2.4	TI12	-	3.8	fensulfothion	C11H17O4PS2	C	NR	NR	6-15-50	1-2-3
2.41	NI4	-	-	2,8-dihydromirex	C10H2Cl10	-	C	-	6	-
2.41	TR4	3.8	2.87	TDE, p,p'-	C14H10Cl4	C	C	C	6	1
2.43	NP(WB)12	-	3.17	etaconazole*	C14H15Cl2N3O2	C	-	-	-	-
2.43	NP60	-	4.5	benodanil	C13H10INO	C	-	-	-	-
2.45	TR340	-	-	diisohexyl phthalate*	C20H30O4	-	C	-	15+50	-
2.46	HX9/NI9	-	2.81	flamprop-M-isopropyl	C19H19ClFNO3	C	-	-	-	-
2.47	NI100	-	-	mirex, 5,10-dihydro-*	C10H2Cl10	-	-	-	-	-
2.49	TI10	-	5	fensulfothion oxygen analog	C11H17O5PS	C	NR	-	6-15-50	-
2.5	NI1700/NP8	14	5	oxadixyl	C14H18N2O4	C	NR	NR	6-15-50	1-2-3
2.51	HN(WB)1/HX(WB)12/ NI(WB)9/NP(WB)90	4	4.2	pyrithiobac-sodium methyl ester	C14H13ClN2O4	-	-	-	-	-
2.52	HX1/NI2	3.33	2.61	nonachlor, cis-	C10H5Cl9	C	C	C	6	1
2.53	TR5	2.66	-	Compound K*	C10H6Cl8	-	C	-	-	1
2.55	NI(V)250	-	-	hydramethylnon*	C25H24F6N4	-	-	-	-	-
2.55	TR4	-	-	endrin alcohol	C12H8Cl6O	-	P	C	15+50	2+3
2.55	TR4	2.27	2.7	DDT, o,p'-	C14H9Cl5	C	C	C	6	1
2.56	FP8	-	3.16	chlorthiophos*	C11H15Cl2O3PS2	C	C	C	6	2
2.56	TR20	3.1	-	2,4,5-T isooctyl ester*	C16H21Cl3O3	-	-	-	-	-
2.56	FP(WB)1.9/NI(WB)3/NP2	3.93	3.36	ethion	C9H22O4P2S4	C	C	C	6	2
2.62	HX(WB)260	-	-	pyrazon metabolite A	C16H18ClN3O6	-	-	-	-	-

Appendix I: PESTDATA Chemicals in Order by OV-101 Relative Retention Time (continued)

RRT/c OV-101	Detector Responses with OV-101 Column	RRT/c OV-225	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
2.62	HX17	18.6	5.4	ofurace	C14H16NO3Cl	C	-	-	-	-
2.62	FP(WB)3	-	5.2	triazophos	C12H16N3O3PS	C	-	-	-	-
2.64	NI(WB)1	2.33	2.8	tetrasul	C12H6Cl4S	C	C	C	6	1
2.65	FP8/TI40	14	5	famphur	C10H16NO5PS2	C	NR	-	6-15-50	-
2.66	TR340	-	-	diisohexyl phthalate*	C20H30O4	-	C	-	15+50	-
2.67	NI7	-	-	10,10-dihydromirex	C10H2Cl10	-	C	-	6	-
2.67	HN(WB)8/HX(WB)30/ NI(WB)26/NP(WB)56	13	8	pyrazon	C10H8ClN3O	C	NR	NR	6-15-50	1-2-3
2.72	NI500/NP500	-	-	WAK4103*	C9H9N5O3Cl	-	NR	NR	6-15-50	1-2-3
2.75	NI(WB)2	1.67	2.38	chlordecone	C10H8Cl10O5	-	S #	P #	15+50	1-2-3
2.78	FP(WB)14	-	3.6	sulprofos sulfoxide*	C12H19O3PS3	C	-	-	-	-
2.79	FP(WB)47	-	3.5	sulprofos	C12H19O2PS3	C	-	-	-	-
2.8	NI9/NP7	-	3.6	fensulfothion sulfone	C11H17O5PS2	C	NR	-	6-15-50	-
2.81	TR8	7.5	-	Dilan*	C15.5H14Cl2NO2	-	P	P	15	-
2.81	NI(WB)1	7.5	3.9	Prolan	C15H13Cl2NO2	P	S	S	15	2
2.83	HX4/TR5	8.3	4	endosulfan sulfate	C9H6Cl6O4S	C	C	C	50	2
2.85	TR35	1.19	-	2,4,5-T BEP ester*	C17H23Cl3O3	-	-	-	-	-
2.85	TR5	4.7	-	chlornitrofen	C12H6Cl3NO3	C	C	C	6+15	2
2.87	HX(WB)160/NI(WB)57/ NP(WB)140	-	4.6	vinclozolin metabolite F	C11H13Cl2NO4	R	NR	NR	6-15-50	1-2-3
2.87	FP(WB)4/NI(WB)4	6.3	5.3	edifenphos	C14H15O2PS2	C	-	-	-	-
2.9	TR340	-	-	diisohexyl phthalate*	C20H30O4	-	C	-	15+50	-
2.9		-	3.7	1,1'-(2,2-dichloroethylidene)bis(2-methoxybenzene)	C16H16Cl2O2	-	R	-	-	-
2.91	TR4	3.3	-	2,4,5-T butoxyethyl ester*	C14H17Cl3O4	-	-	-	-	-
2.91	HN(WB)3/NP(WB)15	-	-	hexazinone	C12H20N4O2	P	NR	NR	6-15-50	1-2-3
2.94	TI15/TR4	4.2	3.7	carbophenothion	C11H16ClO2PS3	C	C	P	6	2
2.95	NI25	2.84	2.7	pyrethrins*	C21H27O4	-	C	C	50	-
2.96	TR20	3.4	-	2,4,5-T isooctyl ester*	C16H21Cl3O3	-	-	-	-	-
2.96	NI15/NP50	11.5	4.1	CL 202,347	C13H19N3O5	-	-	-	-	-
2.97	NI(WB)8	3.7	4.2	methoxychlor olefin	C16H14Cl2O2	C	C	C	6	2
3	NP300	-	1.18	NTN33823	C9H11N4Cl	-	NR	NR	6-15-50	1-2-3

Appendix I: PESTDATA Chemicals in Order by OV-101 Relative Retention Time (continued)

RRT/c OV-101	Detector Responses with OV-101 Column	RRT/c OV-225	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
3.03	NI170	2.74	2.88	ethephon	C2H6ClO3P	NR		-	6+15+50	1+2+3
3.06	NI10/NI(WB)17	-	-	propiconazole*	C15H17Cl2N3O2	C	NR	NR	6-15-50	1-2-3
3.06	HN(WB)1.4/NP(WB)29	-	-	IN-A3928	C11H18N4O2	S	NR	NR	6-15-50	1-2-3
3.06	NI(WB)1	7.5	4.4	Bulan	C16H15Cl2NO2	C	P	P	15	2
3.06	NI35	5.1	4.5	butyl benzyl phthalate	C19H20O4	-	C	P	15+50	-
3.1	NI900/NP100	14.8	7.1	phosmet oxygen analog*	C11H12NO5PS	-	NR	NR	6-15-50	-
3.1	FP3.5/NI3/NP(WB)3	8.2	4.6	cyanofenphos	C15H14NO2PS	C	-	-	-	-
3.1	NI(WB)2/NP(WB)220	-	3.7	fenhexamid	C14H17Cl2NO2	NR	NR	NR	6-15-50	1-2-3
3.11	NI3	-	5.4	captafol	C10H9Cl4NO2S	C	P	-	50	3
3.13	TR4	3.6	3.5	DDT, p,p'-	C14H9Cl5	C	C	C	6	1
3.14	NI4/NP40	-	-	bromoxynil octanoate	C15H17Br2NO2	-	V #	-	15+50	2
3.21	NI100	-	-	mirex, 5,10-dihydro-*	C10H2Cl10	-	-	-	-	-
3.21	NI10/NI(WB)17	5.6	4	propiconazole*	C15H17Cl2N3O2	C	NR	NR	6-15-50	1-2-3
3.22	TR60	2.09	-	2,4-D BEP ester*	C17H24Cl2O4	-	-	-	-	-
3.25	TR20	3.8	-	2,4,5-T isooctyl ester*	C16H21Cl3O3	-	-	-	-	-
3.26	NP(WB)30	5.8	4.67	clodinafop-propargyl	C17H13ClFNO4	V	V	-	50	3
3.27	TR340	-	-	diisohexyl phthalate*	C20H30O4	-	C	-	15+50	-
3.28	FS(WB)17/HN(WB)3/ NP(WB)130	-	9.4	oxycarboxin	C12H13NO4S	R	-	-	-	-
3.3	TR35	2.78	-	2,4,5-T BEP ester*	C17H23Cl3O3	-	-	-	-	-
3.3	NI(WB)0.1/NP(WB)37	-	7.5	3-desmethyl sulfentrazone	C10H8Cl2F2N4O3S	-	NR	NR	6-15-50	1-2-3
3.3	NI(WB)2	4.5	5	methoxychlor, o, p'-	C16H15Cl3O2	-	C	-	6	-
3.32	NI100/NP10	-	-	tralkoxydim*	C20H27NO3	V	NR	NR	50	1-2-3
3.34	NI50	-	-	sethoxydim	C17H29NO3S	-	NR	NR	6-15-50	3
3.36	NI5	7.3	4.8	nuarimol	C17H12ClFN2O	C	NR	C #	50	1-2-3
3.38	HX(WB)3/NI27	1.41	4.9	desmethyl norflurazon	C11H7ClF3N3O	V	NR	NR	6-15-50	1-2-3
3.38	HX(WB)1	-	4.2	tebuconazole	C16H22ClN3O	C	-	-	-	-
3.38	NI5	-	2.62	2,4,5-T ethylhexyl ester	C16H21Cl3O3	-	-	-	-	-
3.39	TR8	8.2	-	Dilan*	C15.5H14Cl2NO2	-	P	P	15	-
3.57	HX8/NI10	4.9	4.7	diclofop-methyl	C16H14Cl2O4	C	C	C	15	2
3.6	NI60	37.1	7.5	myclobutanil alcohol metabolite	C15H17ClN4O	S	NR	NR	6-15-50	1-2-3
3.6	TR5	10.3	-	endrin ketone	C12H8Cl6O	-	C	C	50	2

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Appendix I: PESTDATA Chemicals in Order by OV-101 Relative Retention Time (continued)

RRT/c OV-101	Detector Responses with OV-101 Column	RRT/c OV-225	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
3.7	FP20	-	10.1	azinphos-methyl oxygen analog	C10H12N3O4PS	C	-	-	-	-
3.74	NI5	-	-	8-monohydromirex	C10HCl11	-	C	-	6	-
3.8	FP600/FP(WB)8/NI160	-	6.2	phosalone oxygen analog	C12H15ClNO5PS	C	-	-	-	-
3.8	NI36/TI35	-	7.1	carbophenothion oxygen analog sulfone	C11H16ClO5PS2	-	-	-	-	-
3.8	NI(WB)1	24	6.3	nitralin	C13H19N3O6S	C	P	P	50	3
3.8	FS45/NI(WB)230	4.8	4.3	propargite	C19H26O4S	C	C	-	15	2
3.9	NI(WB)0.8/NP(WB)18/ HX(WB)5	-	-	zoxamide*	C14H16NO2Cl3	C	C	-	50	3
4	NI(WB)6	-	3.9	dinocap*	C18H24N2O6	C	P	P	15	2
4	NI1000/NP1000	15	4.2	KWG 1342	C14H18ClN3O3	-	-	-	-	-
4	NI(WB)2/NP19	14.9	8.4	phosmet	C11H12O4NPS2	C	NR	-	6-15-50	3
4.1	TR60	5.1	-	2,4-D BEP ester*	C17H24Cl2O4	-	-	-	-	-
4.1	NI5	1.08	0.91	dicofol, o,p'-*	C14H9Cl5O	C	V	S	6+15	2
4.1	HX15	-	-	2-chloroethyl linoleate	C20H35ClO2	-	V	P	15	2
4.2	HX13/NI15/NP15	18	6.3	iprodione*	C13H13Cl2N3O3	C	S #	NR	50	1-2-3
4.2	FP60/TI65	7.6	6.5	leptophos oxygen analog	C13H10BrCl2O3P	C	-	-	-	-
4.2	TI250	-	2.87	carbophenothion oxygen analog sulfoxide	C11H16ClO4PS2	-	-	-	-	-
4.2	NI(WB)16	14	8.7	pyridaphenthion	C14H17O4N2SP	C	-	-	-	-
4.26	NI7	-	-	10-monohydromirex	C10HCl11	-	C	-	6	-
4.3	NI100	-	-	mirex, 5,10-dihydro-*	C10H2Cl10	-	-	-	-	-
4.3	NI50	-	-	tetramethrin*	C19H25NO4	C	NR	NR	6-15-50	1-2-3
4.3	NI(WB)6	6.9	4.4	dinocap*	C18H24N2O6	C	P	P	15	2
4.3	NI(WB)3	8.4	6	benzoylprop-ethyl	C18H17Cl2NO3	P	NR	NR	6-15-50	1-2-3
4.4	NI5	1.28	1.08	dicofol, p,p'-*	C14H9Cl5O	C	V	P #	6+15	1+2
4.4	TR12	6.5	-	bromopropylate	C17H16Br2O3	C	C #	C #	15+50	1-2-3
4.4	TR6	15.5	8.5	photodieldrin	C12H8Cl6O	-	C	C	15+50	2
4.5	NI50	8.5	7.2	tetramethrin*	C19H25NO4	C	NR	NR	6-15-50	1-2-3
4.5	FP50/NP20	-	8.4	fenamiphos sulfone	C13H22NO5PS	C	NR	NR	6-15-50	1-2-3
4.5	HX50	-	5.01	norflurazon	C12H9ClF3N3O	V	NR	NR	6-15-50	-
4.5	NI0.5/TI16	10.6	6.9	EPN	C14H14NO4PS	C	C	C	15	2

Appendix I: PESTDATA Chemicals in Order by OV-101 Relative Retention Time (continued)

RRT/c OV-101	Detector Responses with OV-101 Column	RRT/c OV-225	RRT/c OV-17		Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
4.6	HX8	-	-		fenarimol metabolite C	C17H14N2OCl2	S		-	6	-
4.6	HX19	-	-		fenarimol metabolite B	C17H14N2OCl2	NR	NR	NR	6-15-50	-
4.7	NI500/NP500	-	-		WAK4103*	C9H9N5O3Cl	-	NR	NR	6-15-50	1-2-3
4.7	HN(WB)390/NI(WB)400	-	-		IN-T3937	C12H20N4O3	S	-	-	-	-
4.7	HN(WB)73/NI(WB)230	-	-	-	IN-T3935	C11H18N4O3	S	-	-	-	-
4.7	HX20/NI6	10.3	6.9		chlorthiophos sulfoxide	C11H15Cl2O4PS2	C	NR	NR	6-15-50	1-2-3
4.7	NI(WB)1	8.6	7.2		tetrasul sulfoxide	C12H6Cl4OS	-	-	-	-	-
4.7	NI8800	-	-		oryzalin	C12H8N4O6S	-	NR	NR	6-15-50	-
4.7	NI(WB)12	53	11.3		dithianon	C14H4O2N2S2	NR	-	-	-	-
4.7	TR9	7.2	7.2		methoxychlor, p, p'-	C16H15Cl3O2	C	C	C	6	2
4.8	NI(WB)6	7.7	4.8		dinocap*	C18H24N2O6	C	P	P	15	2
4.8	NI7/NI(WB)0.2/NP30	7	5.7		fenpropathrin	C22H23NO3	-	V #	V	15	2
4.8	FP15	9.7	6.8		piperophos	C14H28NO3PS2	C	-	-	-	-
4.8	NP(WB)40	6.6	6.3		cloquintocet-mexyl	C18H22ClNO3	V	NR	-	6-15-50	1-2-3
4.9	NI8	3.8	4.5		bifenthrin	C23H22ClF3O2	V	C	-	6+15	2
5	NI550/NP210	-	-		NTN35884*	C9H9N5O2Cl	-	NR	NR	6-15-50	1-2-3
5	NP50	-	7.3		fenoxycarb	C17H19NO4	C	-	-	-	-
5	HX16/NI4	14.9	8.8		bifenox	C12H9Cl2NO5	C	C	P	15+50	2+3
5.1	NI(WB)6	9.5	5.6		dinocap*	C18H24N2O6	C	P	P	15	2
5.1	FP(WB)80	-	10.6		sulprofos oxygen analog sulfone	C12H19O5PS2	C	-	-	-	-
5.1	FP3/TI20	-	9.2		carbophenothion sulfone	C11H16ClO4PS3	C	C	P	6	1
5.2	FP50/NP55	-	8.1		fenamiphos sulfoxide	C13H22N04PS	C	NR	NR	6-15-50	1-2-3
5.2	TR6	-	8.3		tetradifon	C12H6Cl4O2S	C	C	C	15	2
5.2	TI30/TR50	-	11.8		azinphos-methyl	C10H12N3O3PS2	C	NR	NR	6-15-50	1-2-3
5.3	TR35	3.28	-		2,4,5-T BEP ester*	C17H23Cl3O3	-	-	-	-	-
5.3	HX20/NI9	18.8	9.1		chlorthiophos sulfone	C11H15Cl2O5PS2	C	C	-	50	3
5.3	HX(WB)20/NI1000	-	7.5		iprodione metabolite isomer	C13H13Cl2N3O3	C	S	-	50	-
5.4	NI500/NI(WB)40	4.8	6.5		phenothrin*	C23H26O3	-	-	-	-	-
5.4	NP20	-	5.3		carbosulfan	C20H32N2O3S	P	-	-	-	-
5.4	FP3/TI35	-	4		carbophenothion sulfoxide	C11H16ClO3PS3	-	-	-	-	-
5.5	TR850	-	-		diisooctyl phthalate*	C24H38O4	-	C	C	15+50	-
5.5	NP10/TR15	5.5	9.1		phosalone	C12H15ClNO4PS2	C	C	C	50	2+3

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Appendix I: PESTDATA Chemicals in Order by OV-101 Relative Retention Time (continued)

RRT/c OV-101	Detector Responses with OV-101 Column	RRT/c OV-225	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
5.8	FP14/TI20/TR11	7.7	8.5	leptophos	C13H10BrCl2O2PS	C	C	C	6	2
5.8	NI7	2.95	5.6	mirex	C10Cl12	P	C	P	6	1
5.9	HN(WB)10.5/HX20/NI100	-	9.8	clofentezine	C14H8Cl2N4	R	S	-	15	2
6.1	NI100/NP10	1.48	4.5	tralkoxydim*	C20H27NO3	V	NR	NR	50	1-2-3
6.1	FP(WB)14	-	11.7	sulprofos sulfoxide*	C12H19O3PS3	C	-	-	-	-
6.2	TR850	-	-	diisooctyl phthalate*	C24H38O4	-	C	C	15+50	-
6.4	NI(V)200	4.5	6.1	bis(2-ethylhexyl) phthalate	C24H38O4	-	C	C	15+50	-
6.5		-	11.5	myclobutanil dihydroxy metabolite	C15H17N4O2Cl	NR	NR	NR	6-15-50	1-2-3
6.5	TI30/TR28	-	14.3	dialifor	C14H17ClNO4PS2	C	C	P	15	2
6.6	HX10/NI5	-	10.1	fenarimol	C17H12Cl2N2O	C	P #	C #	50	3
6.6	TR500	-	-	n-acetyl nitrofen	C14H11Cl2NO2	-	-	-	-	-
6.7	TR850	-	-	diisooctyl phthalate*	C24H38O4	-	C	C	15+50	-
6.7	NI1000	-	1.59	CGA 205375	C16H13N3O2Cl2	-	-	-	-	-
6.7	NI13	-	-	PPG-1576	C19H17ClF3NO5	-	-	P	50	2+3
6.9	TI58/TR200	-	14.8	azinphos-ethyl	C12H16N3O3PS2	C	P	S	50	3
7	TR35	7.7	-	2,4,5-T BEP ester*	C17H23Cl3O3	-	-	-	-	-
7	NI30/NP4000	-	11	tebufenozide	C22H28N2O2	-	NR	NR	6-15-50	1-2-3
7	NI40	-	11.4	CGA 118244	C15H13Cl2N3O3	V	NR	NR	6-15-50	1-2-3
7.2	FP(WB)16	-	13.1	sulprofos sulfone	C12H19O4PS3	C	-	-	-	-
7.3	NI10	-	-	lactofen	C19H15ClF3NO7	-	-	C	50	2+3
7.4	NI10	-	8	lambda-cyhalothrin	C23H19ClF3NO3	C	-	-	-	-
7.5	TR850	-	-	diisooctyl phthalate*	C24H38O4	-	C	C	15+50	-
7.9	NI90/NP300	-	-	RH-6467*	C19H15N4ClO	S	NR	NR	6-15-50	1-2-3
7.9	NI20	10.8	12.6	HOE-030291	C17H16Cl2O5	-	-	-	-	-
8	NI200/NP130	45	16	coumaphos oxygen analog	C14H16ClO6P	C	NR	NR	6-15-50	1-2-3
8.1	NI250	11.3	10.5	fenoxaprop ethyl ester	C18H16NO5Cl	S	V	V	50	3
8.1	FP(WB)12	-	13	pyrazophos	C14H20N3O5PS	C	-	-	-	-
9	TR850	-	-	diisooctyl phthalate*	C24H38O4	-	C	C	15+50	-
9	NI39/NP38	40	18	coumaphos	C14H16ClO5PS	C	NR	C #	6-15-50	3
9.4		-	11.8	bitertanol*	C20H23N3O2	C	-	-	-	-
9.4	NI75	11.1	13.8	permethrin, cis-	C21H20Cl2O3	C	V #	C	6+15	2
9.5	FP100/NI(WB)9/TI190	-	20.2	bensulide	C14H24NO4PS3	C	P	C	50	3

Appendix I: PESTDATA Chemicals in Order by OV-101 Relative Retention Time (continued)

RRT/c OV-101	Detector Responses with OV-101 Column	RRT/c OV-225	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
9.7		-	12.5	bitertanol*	C20H23N3O2	C	-	-	-	-
9.7	TR7	-	-	hexachlorophene dimethyl ether	C15H10Cl6O2	-	NR	NR	6-15	-
9.8	NI1000/NP70	-	-	fenbuconazole	C19H17ClN4	C	NR	NR	6-15-50	1-2-3
10.2	TR60	13	-	2,4-D BEP ester*	C17H24Cl2O4	-	-	-	-	-
10.2	NI100	13	15	permethrin, trans-	C21H20Cl2O3	C	V #	C	6+15	2
10.4	NI90/NP300	-	-	RH-6467*	C19H15N4ClO	S	NR	NR	6-15-50	1-2-3
10.4	NI15/NP125	12.8	8.9	acrinathrin	C26H21F6NO5	V	V	V#	15	2
10.4	HX50/NI12	-	15.4	prochloraz	C15H16Cl3N3O2	C	-	-	-	-
10.5	TR850	-	-	diisooctyl phthalate*	C24H38O4	-	C	C	15+50	-
11.5	NI500/NI(WB)40	10.9	15	phenothrin*	C23H26O3	-	-	-	-	-
11.7	HX30/NI30	-	-	cyfluthrin*	C22H18Cl2FNO3	C	P	-	15	-
12	NI50	8.9	6.1	CGA 205374	C16H11N3O2Cl2	-	NR	NR	6-15-50	1-2-3
12	NI50/NP170	-	-	RH-9130	C19H16N3ClO2	P	NR	NR	6-15-50	1-2-3
12	NI(V)330	-	-	di-n-octyl phthalate	C24H38O4	-	C	C	15+50	-
12.4	NP(WB)14	-	-	flumetsulam, methylated	C13H11F2N5O2S	-	-	-	-	-
12.5	HX30/NI30	-	-	cyfluthrin*	C22H18Cl2FNO3	C	P	-	15	-
12.8	HX30/NI30	-	-	cyfluthrin*	C22H18Cl2FNO3	C	P	-	15	-
13	NI(WB)2	13	16	hexachlorophene	C13H6Cl6O2	-	NR	NR	6-15-50	-
13.6	HX70/NI80	-	25	quizalofop ethyl ester	C19H17ClN2O4	C	-	-	-	-
14	NI40/NP190	-	-	RH-9129	C19H16N3ClO2	V	NR	NR	6-15-50	1-2-3
14	HX9/NI22	-	-	alpha-cypermethrin	C22H19Cl2O3N	C	C	-	-	2
14	NI(WB)90	-	-	azafenidin	C15H13Cl2N3O2	V	-	-	-	-
14.1	NI90	33	23	cypermethrin*	C22H19Cl2NO3	C	C	C	15	2
14.7	NI40/NI(WB)15	36.9	21.4	flucythrinate*	C26H23F2NO4	C	C	-	15	2+3
15	NI90/NP300	-	-	RH-6467*	C19H15N4ClO	S	NR	NR	6-15-50	1-2-3
15.1	NI90	36	25	cypermethrin*	C22H19Cl2NO3	C	C	C	15	2
16.1	NI40/NI(WB)15	42	24	flucythrinate*	C26H23F2NO4	C	C	-	15	2+3
16.3	HX400/NI1500	24	-	fluridone	C19H14F3NO	-	NR	NR	6-15-50	-
17	NI200	-	6.2	deltamethrin, trans-*	C22H19Br2NO3	-	P #	NR	15	2
17.1	NI1300	-	21	deltamethrin*	C22H19Br2NO3	C	S #	P	15	2
20.3	NI90	44	35	fenvalerate*	C25H22ClNO3	C	C	C	15	2
20.7	NI500/NP500	-	-	WAK4103*	C9H9N5O3Cl	-	NR	NR	6-15-50	1-2-3

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Appendix I: PESTDATA Chemicals in Order by OV-101 Relative Retention Time (continued)

RRT/c OV-101	Detector Responses with OV-101 Column	RRT/c OV-225	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
22.5	NI90	51	40	fenvalerate*	C25H22ClNO3	C	C	C	15	2
22.5	NI90	-	-	esfenvalerate	C25H22ClNO3	C	C	C	15	2
23	NI150/NP300	57	43	PB-7, methylated	C20H25ClN2O3S	-	-	-	-	-
25		59	38	fluvalinate*	C26H22ClF3N2O3	C	C	-	15	2
25	NI300/NP500	87	46	PB-9	C19H25ClN2O2S	V	NR	NR	6-15-50	1-2-3
27	NI1300	-	35	deltamethrin*	C22H19Br2NO3	C	S #	P	15	2
27	NI30/NI(WB)1	64	44	tralomethrin	C22H19Br4NO3	C	V	S	15	2
29	NI200	-	20	deltamethrin, trans-*	C22H19Br2NO3	-	P #	NR	15	2
29	NI1300	19.9	38	deltamethrin*	C22H19Br2NO3	C	S #	P	15	2
31	NI200	19.7	38	deltamethrin, trans-*	C22H19Br2NO3	-	P #	NR	15	2
32	NI(V)250	4.5	-	hydramethylnon*	C25H24F6N4	-	-	-	-	-
44	NI(V)250	53	-	hydramethylnon*	C25H24F6N4	-	-	-	-	-

Appendix I: PESTDATA Chemicals in Order by OV-225 Relative Retention Time

RRT/c OV-225	Detector Responses with OV-225 Column	RRT/c OV-101	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
0.08		-	-	2,4-D BEP ester*	C17H24Cl2O4	-	-	-	-	-
0.08	NI40	0.06	0.04	CGA 171683	C6H5F4N3O2	C		-	15+50	3
0.08		0.19	0.1	2-methoxy-3,5,6-trichloropyridine	C6H4Cl3NO	C	P#	C	6+15	1+2
0.08	FP(WB)0.7	0.07	0.08	dichlorvos	C4H7Cl2O4P	C	NR	NR	6-15-50	1-2-3
0.09	NI27	0.11	0.11	diuron	C9H10Cl2N2O	C	NR	NR	6-15-50	1-2-3
0.11		-	0.09	teflubenzuron*	C14H6Cl2F4N2O2	-	NR	NR	6-15-50	1-2-3
0.12	NI0.2	0.18	0.21	etridiazole	C5H5Cl3N2OS	C	C	P	6	2
0.13	NI0.25	0.24	0.16	pentachlorobenzene	C6HCl5	C	C	C	6	1
0.14		0.16	-	2,4,5-T BEP ester*	C17H23Cl3O3	-	-	-	-	-
0.14	NI12	0.17	0.1	N-(3,4-dichlorophenyl)-N'-methylurea	C8H8Cl2N2O	-	NR	NR	6-15-50	-
0.14	NI6	0.22	0.1	3,4-dichlorophenylurea	C7H6Cl2N2O	-	NR	NR	6-15-50	-
0.15		0.13	0.1	chlorimuron ethyl ester	C15H15ClN4O6S	P	NR	-	-	-
0.15	NI0.2	0.24	0.22	2,3,5,6-tetrachloroanisole	C7H4Cl4O	-	C	-	6	1
0.15		0.15	0.14	dimethyl phthalate	C10H10O4	-	P	-	6+15+50	-
0.18		0.19	-	dicamba methyl ester	C8H6Cl2O3	-	-	-	-	-
0.18	NI0.9	0.2	0.2	nitrapyrin	C6H3Cl4N	C	C	V	6	2
0.19		0.19	-	chloroneb	C8H8Cl2O2	C	C	-	6	2
0.19		-	-	molinate	C9H17NOS	-	-	-	-	-
0.21		0.23	0.18	methyl 2,3,6-trichlorobenzoate	C8H5Cl3O2	-	-	-	-	-
0.22	NI2	0.17	0.13	3-methyl-4-nitrophenol methyl ether	C8H9O3N	-	-	-	-	-
0.22	NI1	0.19	0.14	N, N-diallyl dichloroacetamide	C8H11Cl2NO	C	S	S	15+50	2+3
0.23		0.14	0.14	teflubenzuron*	C14H6Cl2F4N2O2	-	NR	NR	6-15-50	1-2-3
0.23	FP0.5/NI0.3	0.33	0.24	chlorethoxyfos	C6H11Cl4O3PS	V	C	-	6	1
0.23	FS25	0.13	0.11	carboxin sulfoxide	C12H13NO3S	-	NR	NR	6-15-50	1-2-3
0.24		0.15	-	hydroxy chloroneb	C7H6Cl2O2	-	NR	-	6-15	-
0.24		-	-	carbofuran-3-keto-7-phenol	C10H10O3	-	-	-	-	-
0.25	NI100	0.22	0.2	3-carboxy-5-ethoxy-1,2,4-thiadiazole	C3H2N2O3S	NR	-	-	-	-
0.25	FP4	0.07	0.09	methamidophos	C2H8NO2PS	V	-	-	-	-
0.25	NI0.3	0.45	0.33	hexachlorobenzene	C6Cl6	C	C	P	6	1

* Multipeak chemical.

\# Recovery may vary with choice of Florisil elution system; see Tables 303-a, 304-a.

Appendix I: PESTDATA Chemicals in Order by OV-225 Relative Retention Time (continued)

RRT/c OV-225	Detector Responses with OV-225 Column	RRT/c OV-101	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
0.26		0.42	0.33	di-allate	C10H17ClNOS	C	C	-	6	-
0.26		0.29	0.24	tecnazene	C6HCl4NO2	C	C	C	6	1
0.27	FP(WB)1	0.37	0.29	cadusafos	C10H23O2PS2	C	NR	NR	6-15-50	1-2-3
0.27	NI0.4	0.34	0.19	ethalfluralin	C13H14F3N3O4	C	C	C	6	2
0.27		0.34	0.17	trifluralin	C13H16F3N3O4	C	C	C	6	2
0.27	NI20/NI(WB)25	0.18	0.14	3,5-dichloroaniline	C6H5Cl2N	S	S	S	6+15	1+2
0.27	NI13700	0.21	0.24	TEPP	C8H20O7P2	C	-	-	-	-
0.28	NI2	0.37	0.18	benfluralin	C13H16F3N3O4	C	C	C	6	2
0.29	NI1000	0.44	0.45	desmedipham	C16H16N2O4	-	-	-	-	-
0.3		0.46	0.34	pentachlorophenyl methyl ether	C7H3Cl5O	C	C	C	6	1
0.31		0.33	0.25	ethoprop	C8H19O2PS2	C	P #	S #	50	1-2-3
0.32		0.22	0.21	demeton-O oxygen analog	C8H19O4PS	-	-	-	-	-
0.32	NI30	0.2	0.16	3,4-dichloroaniline	C6H5Cl2N	V	S	-	15	-
0.35	NI0.3	0.49	0.48	1,2,4,5-tetrachloro-3-(methylthio)= benzene	C7H4Cl4S	R	C	-	6	1
0.36		0.36	0.39	triclopyr methyl ester	C8H6Cl3NO3	-	-	-	-	-
0.37	FP0.5	0.3	0.29	phorate oxygen analog	C7H17O3PS2	C	NR	NR	6-15-50	1-2-3
0.37	NI9/NI(WB)5	0.34	0.26	propachlor	C11H14ClNO	C	NR	NR	6-15-50	1-2-3
0.38		0.3	0.25	2,4-D methyl ester	C9H8Cl2O3	-	-	-	-	-
0.38	FP0.5/NI17	0.37	0.32	phorate	C7H17O2PS3	C	V #	V #	6	1
0.4	NI0.3	0.56	0.32	chlordene	C10H6Cl6	-	C	C	6	1
0.4	FP6/NI4.5	0.51	0.44	diazinon	C12H21N2O3PS	C	C	C	15	3
0.42	NP20	0.47	0.53	terbumeton	C10H19N5O	C	-	-	-	-
0.43		0.32	0.25	chlorpropham	C10H12ClNO2	C	C	C	15	2
0.44		0.32	0.36	3, 5, 6-trichloro-2-pyridinol methyl ester	C6H4Cl3NO	-	-	-	-	-
0.44		0.45	-	silvex methyl ester	C10H9Cl3O3	-	-	-	-	-
0.44		0.5	0.41	terbufos	C9H21O2PS3	C	P	S	6	-
0.44	NI1	0.38	0.36	sulfallate	C8H14ClNS2	C	C	C	6+15	2
0.46	NI1	0.53	0.3	profluralin	C14H16F3N3O4	V	V	-	6	-
0.46		0.51	0.46	quintozene	C6Cl5NO2	C	C	C	6	1
0.48		0.4	0.35	BHC, alpha-	C6H6Cl6	C	C	C	6	1

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Appendix I: PESTDATA Chemicals in Order by OV-225 Relative Retention Time (continued)

RRT/c OV-225	Detector Responses with OV-225 Column	RRT/c OV-101	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
0.49		0.31	0.32	metasystox thiono*	C6H15O3PS2	-	-	-	-	-
0.49	NI(WB)1.5	0.56	0.47	methyl 3,5-dibromo-4-methoxy= benzoate	C9H8Br2O3	-	-	-	-	-
0.49		0.28	0.32	metasystox thiol	C6H15O3PS2	C	-	-	-	-
0.49		0.46	0.31	oxydemeton-methyl	C6H15O4PS2	C	-	-	-	-
0.5	NI(WB)70	0.25	0.16	cymoxanil	C7H10N4O3	V	NR	NR	6-15-50	1-2-3
0.51	NI0.5	0.26	0.23	RPA 203328, methylated	C10H9F3O4S	-	-	-	-	-
0.51		0.41	0.4	thiometon	C6H15O2PS3	C	NR	NR	6-15-50	-
0.52		0.83	0.6	heptachlor	C10H5Cl7	C	C	C	6	1
0.53	NI600	0.5	0.24	phosmet oxygen analog*	C11H12NO5PS	-	NR	NR	6-15-50	-
0.53	NI60	0.5	0.47	diazinon oxygen analog	C12H21N2O4P	C	NR	NR	6-15-50	1-2-3
0.53		0.39	0.38	fonofos oxygen analog	C10H15O2PS	V	NR	NR	6-15-50	1-2-3
0.56		0.52	0.44	fonofos	C10H15OPS2	C	C	C	6	2+3
0.56		0.41	0.41	demeton-S	C8H19O3PS2	C	NR	-	6-15-50	-
0.58		1.05	0.76	aldrin	C12H8Cl6	C	C	C	6	1
0.59	NI150	0.45	0.46	clomazone	C12H14ClNO2	C		-	50	3
0.59	NI30	0.58	0.51	etrimfos	C10H17N2O4PS	C	C	C	15	2+3
0.59		0.5	0.45	pentachlorobenzonitrile	C7Cl5N	C	C	P	15	2
0.6	NI1.6	0.46	0.39	furilazole	C11H13Cl2NO3	C	S	-	50	3
0.6	NP10	0.35	0.38	2,3,5-trimethacarb	C11H15NO2	C	S #	NR	50	1-2-3
0.6		0.54	0.46	disulfoton	C8H19O2PS3	C	P #	NR	6	1-2-3
0.61		0.65	0.56	diisobutyl phthalate	C16H2204	-	P	-	15+50	-
0.62		-	-	2,4-D isopropyl ester*	C11H12Cl2O3	-	-	-	-	-
0.62		0.62	0.49	2,4-D isobutyl ester	C12H14Cl2O3	-	-	-	-	-
0.63		0.49	0.47	2,4,5-T methyl ester	C9H7Cl3O3	-	-	-	-	-
0.63	NI250	0.38	0.26	3-methyl-4-nitrophenol	C7H7O3N	V	NR	NR	6-15-50	1-2-3
0.63	NI0.4	0.56	0.56	2,3,5,6-tetrachloronitroanisole	C7H3Cl4NO3	-	C	-	6	1+2
0.64		0.82	0.67	chlordene, alpha-	C10H6Cl6	-	-	-	-	-
0.64	FP5	0.15	0.19	acephate	C4H10NO3PS	C	-	-	-	-
0.64		0.67	0.56	dichlofenthion	C10H13Cl2O3PS	C	C	V	6	2
0.65		1.34	1.43	merphos*	C12H27PS3	-	C	C	6+15+50	3
0.65		0.84	-	chlordene epoxide	C10H6Cl6O	-	C	-	15	-

Appendix I: PESTDATA Chemicals in Order by OV-225 Relative Retention Time (continued)

RRT/c OV-225	Detector Responses with OV-225 Column	RRT/c OV-101	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
0.65	NI37	0.53	0.41	propazine	C9H16ClN5	C	S	NR	15+50	3
0.65		0.67	-	2,4,5-T isopropyl ester	C11H11Cl3O3	-	-	-	-	-
0.66		0.68	-	2,4,5-T BEP ester*	C17H23Cl3O3	-	-	-	-	-
0.69	NI1.6	0.5	0.48	4-(dichloroacetyl)-1-oxa-4-azapiro=[4.5]decane	C10H15Cl2NO2	C	P	-	50	3
0.69	NI0.5	0.94	0.87	pentachlorophenyl methyl sulfide	C7H3Cl5S	C	C	C	6	1
0.69		0.48	0.47	lindane	C6H6Cl6	C	C	C	6	1
0.7	NI2550	0.35	0.41	methabenzthiazuron	C10H11N3OS	C	NR	NR	6-15-50	1-2-3
0.71	NI43	0.47	0.48	terbuthylazine	C9H16N5Cl	C	P	-	15+50	-
0.72		0.62	-	2,4-DB methyl ester	C11H12Cl2O3	-	-	-	-	-
0.73	NI0.5	0.59	0.66	2,3,5,6-tetrachloroanisidine	C7H5Cl4NO	-	C	-	6	2
0.74		0.69	-	2,4-D BEP ester*	C17H24Cl2O4	-	-	-	-	-
0.74		0.42	0.33	2,4-D isopropyl ester*	C11H12Cl2O3	-	-	-	-	-
0.74		0.43	0.44	atrazine	C8H14ClN5	C	S #	NR	50	1-2-3
0.75		0.42	0.45	chlorbufam	C11H10ClNO2	C		-	15	2+3
0.76		0.53	0.37	fluchloralin	C12H13ClF3N3O4	C	C	-	6	2
0.78	NP200	0.45	0.5	3,4,5-trimethacarb	C11H15NO2	C	NR	NR	50	1-2-3
0.79	NI160	0.5	0.44	metribuzin, deaminated diketo metabolite*	C7H11N3O2	NR	NR	NR	6-15-50	1-2-3
0.79	NI0.5	0.67	0.66	pentachloroaniline	C6H2Cl5N	C	C	C	6	1
0.8		0.51	0.63	etrimfos oxygen analog	C10H17N2O5P	C	-	-	-	-
0.8	NI20	0.55	0.63	isazofos	C9H17ClN3O3PS	C	C #	-	50	2+3
0.8	NI2	0.75	0.68	CGA 14128	C12H21N2O4PS	C		-	50	1-2-3
0.8	NI80	0.3	0.53	desethyl simazine	C5H8ClN5	-	NR	NR	50	1-2-3
0.83	NI130	0.41	0.5	simazine	C7H12ClN5	C	NR	NR	50	1-2-3
0.84		0.98	0.89	chlordene, beta-	C10H6Cl6	-	-	-	-	-
0.84	NI3	0.51	0.4	pronamide	C12H11Cl2NO	C	P	-	15+50	-
0.85	NI1	0.81	0.75	tridiphane	C10H7Cl5O	C	C	-	6	1+2
0.85		0.81	-	chloroxuron	C15H15ClN2O2	C	NR	NR	6-15-50	1-2-3
0.86		0.72	0.79	chlorpyrifos-methyl	C7H7Cl3NO3PS	C	C	-	6	2
0.86	NI20	0.2	0.61	desdiethyl simazine	C3H4ClN5	-	NR	NR	6-15-50	1-2-3
0.86		0.55	1.04	tetraiodoethylene	C2I4	-	P	P	6	-

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Appendix I: PESTDATA Chemicals in Order by OV-225 Relative Retention Time (continued)

RRT/c OV-225	Detector Responses with OV-225 Column	RRT/c OV-101	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
0.86	NI2	0.81	0.76	ronnel	C8H8Cl3O3PS	C	C	C	6	2
0.88	NI5	0.75	0.67	acetochlor	C14H20NO2Cl	C	C #	P	50	3
0.89	NI1	0.98	0.88	chlordene, gamma-	C10H6Cl6	-	-	-	-	-
0.91		1.08	-	2,4,5-T BEP ester*	C17H23Cl3O3	-	-	-	-	-
0.91	NI180	0.48	0.48	monolinuron	C9H11ClN2O2	C	-	-	-	-
0.92	NI(WB)13	0.25	0.28	oxamyl oxime metabolite	C5H10N2O2S	C	NR	NR	6-15-50	1-2-3
0.92		0.55	-	dichlone	C10H4Cl2O2	P	S #	S #	6-15-50	2+3
0.92		0.88	0.84	dibutyl phthalate	C16H22O4	-	C	C	15+50	-
0.93	NI600	2	0.44	phosmet oxygen analog*	C11H12NO5PS	-	NR	NR	6-15-50	-
0.93	NI1	0.52	0.44	dinitramine	C11H13F3N4O4	C	-	P	15	-
0.94		1.33	1.05	octachlor epoxide	C10H4Cl8O	C	C	C	6	1
0.95		1.53	-	Perthane olefin	C18H19Cl	-	C	C	6	1
0.96		0.31	0.43	dicrotophos	C8H16NO5P	C	NR	-	6-15-50	-
0.96		0.42	0.45	dicloran	C6H4Cl2N2O2	C	S	P	15+50	2+3
0.97	NI(WB)6	0.42	0.49	PPG-947, methylated*	C18H13ClF3NO7	-	-	-	-	-
0.97	NP50	0.94	0.66	prodiamine	C13H17F3N4O4	C	-	-	-	-
0.98		0.72	-	dimethenamid	C12H18ClNO2S	-	NR	NR	6-15-50	1-2-3
1	NI150	1.05	-	PP 890	C9H10O2ClF3	-	-	-	-	-
1		0.94	0.98	thiobencarb	C12H16ClNOS	C		V	15	2+3
1	NI6	0.8	0.72	alachlor	C14H2OClNO2	C	C	C #	50	3
1		1	1	chlorpyrifos	C9H11Cl3NO3PS	C	C	P	6	2
1.02		0.64	0.62	ronnel oxygen analog	C8H8Cl3O4P	C	NR	-	6-15-50	-
1.03		1.14	1.14	pirimiphos-ethyl	C13H24N3O3PS	C	C	C	15+50	3
1.03		0.44	-	chloramben methyl ester	C8H7Cl2NO2	-	-	-	-	-
1.06	NI2	0.64	0.7	benoxacor	C11H11Cl2NO2	C	P	C	15+50	2+3
1.07		0.82	0.92	dichlorobenzophenone, o,p'-	C13H8Cl2O	-	C	C	15	2
1.08		4.1	0.91	dicofol, o,p'-*	C14H9Cl5O	C	V	S	6+15	2
1.08		0.84	-	terbutryn	C10H19N5S	C	-	-	-	-
1.1		0.77	-	ametryn	C9H17N5S	C	-	-	-	-
1.11	NI20	0.71	0.71	dimethachlor	C13H18ClNO2	C	-	-	-	-
1.11	FP5	0.25	0.39	omethoate	C5H12NO4PS	C	NR	NR	6-15-50	1-2-3
1.13	NI1	1.06	1	DCPA	C10H6Cl4O4	C	C	C	15	2

Appendix I: PESTDATA Chemicals in Order by OV-225 Relative Retention Time (continued)

RRT/c OV-225	Detector Responses with OV-225 Column	RRT/c OV-101	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
1.15		0.69	0.64	vinclozolin	C12H9Cl2NO3	C	C	C	15	2
1.15		1.19	1.2	TDE, o,p'-, olefin	C14H9Cl3	-	-	-	-	-
1.16	NI(WB)55	0.39	0.54	4-chlorobenzylmethyl sulfoxide	C8H9ClOS	-	NR	NR	6-15-50	1-2-3
1.18		1.66	-	2,4-D BEP ester*	C17H24Cl2O4	-	-	-	-	-
1.19		2.85	-	2,4,5-T BEP ester*	C17H23Cl3O3	-	-	-	-	-
1.2	NI(WB)8	0.74	0.66	vinclozolin metabolite B	C12H11Cl2NO4	C	P #	C	6+15	2
1.21	NI9	1.03	0.93	metolachlor	C15H22ClNO2	C	S #	NR	50	1-2-3
1.21		2.02	-	simetryn	C8H15N5S	C	-	-	-	-
1.22	NI8	1.15	0.93	butralin	C14H21N3O4	V	C	-	6+15+50	-
1.22	NI2	1.37	1.12	S-bioallethrin	C19H26O3	-	C	-	50	-
1.22		0.64	0.74	cyprazine	C9H14ClN5	C	-	-	-	-
1.22		1.29	1.15	heptachlor epoxide	C10H5Cl7O	C	C	C	6	2
1.22		1.36	-	allethrin	C19H26O3	-	C	C #	50	3
1.24	NI2	1.14	1.01	isopropalin	C15H23N3O4	C	C	-	6	-
1.25		0.99	1.08	dichlorobenzophenone, p,p'-	C13H8Cl2O	-	C	C	15	2
1.28		4.4	1.08	dicofol, p,p'-*	C14H9Cl5O	C	V	P #	6+15	1+2
1.28		1.55	1.51	DDE, o,p'-	C14H8Cl4	C	C	C	6	1
1.29	NI160	0.73	0.52	metribuzin, deaminated diketo metabolite*	C7H11N3O2	NR	NR	NR	6-15-50	1-2-3
1.29	NI6	1.11	1.16	bromophos	C8H8BrCl2O3PS	C	C	C	6	-
1.3		0.39	0.52	2,6-dichlorobenzamide	C7H5NOCl2	C	NR	NR	6-15-50	1-2-3
1.34	NI(WB)51	0.43	0.6	6-chloro-2,3-dihydro-3,3,7-methyl-5H-oxazolo(3,2-a)pyrimidin-5-one	C9H13ClN2O2	-	NR	NR	6-15-50	1-2-3
1.36	NI3	1.45	1.45	TDE, p,p'-, olefin	C14H9Cl3	C	C	C	6	1
1.38		1.64	1.47	endosulfan I	C9H6Cl6O3S	C	C	C	15	2
1.4		0.6	0.78	ethiofencarb	C10H15NO2S	C	NR	NR	6-15-50	-
1.41	NI200	3.38	4.9	desmethyl norflurazon	C11H7ClF3N3O	V	NR	NR	6-15-50	1-2-3
1.41	NI15	0.56	0.55	metribuzin, diketo metabolite	C7H12N4O2	NR	NR	NR	6-15-50	1-2-3
1.41	NI(WB)80	0.55	0.9	3-ketocarbofuran	C12H12NO4	S	NR	NR	6	1
1.42		-	-	2,4-D propylene glycol butyl ether ester*	C15H2OCl2O4	-	-	-	-	-
1.42		1.51	1.45	bromophos-ethyl	C10H12BrCl2O3PS	C	C	P	6	-

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Appendix I: PESTDATA Chemicals in Order by OV-225 Relative Retention Time (continued)

RRT/c OV-225	Detector Responses with OV-225 Column	RRT/c OV-101	RRT/c OV-17	Name	Molecular Formula	Recoveries				
						302	303	304	Ethers	CH_2Cl_2
1.44		0.67	0.69	metobromuron	C9H11BrN2O2	C	NR	NR	6-15-50	1-2-3
1.44		0.55	0.74	chlorothalonil	C8Cl4N2	S	C #	C #	6-15-50	2+3
1.45	NI1.5	0.96	0.86	nitrofluorfen	C13H7ClF3NO3	C	C	C	15	2
1.45		1.75	1.42	nonachlor, trans-	C10H5Cl9	C	C	C	6	1
1.46		0.97	-	methazole	C9H6Cl2N2O3	-	-	-	-	-
1.46		1.49	1.34	chlordane, trans-	C10H6Cl8	C	C	C	6	1
1.46		0.96	1.18	fenthion	C10H15O3PS2	C	S #	NR	6+15	1-2-3
1.47	NI(WB)300	1.05	0.88	acifluorfen	C14H7ClF3NO3	-	NR	NR	6-15-50	1-2-3
1.47		0.54	0.62	fenfuram	C12H11NO2	C	-	-	-	-
1.47	NI0.7	0.57	0.91	metribuzin	C8H14N4OS	V	NR	NR	50	1-2-3
1.48	NI100	6.1	4.5	tralkoxydim*	C20H27NO3	V	NR	NR	50	1-2-3
1.48	NI3	1.22	1.21	pendimethalin	C13H19N3O4	C	C	P	15	2
1.49	NI44	0.91	1.05	malathion	C10H19O6PS2	C	C	C	15+50	3
1.51		-	-	2,4-D ethyl hexyl ester*	C16H22Cl2O3	-	-	-	-	-
1.51		0.95	1.08	chlorpyrifos oxygen analog	C9H11Cl3NO4P	C	NR	-	6-15-50	-
1.54		1.66	1.48	chlordane, cis-	C10H6Cl8	C	C	C	6	1
1.55		0.75	0.79	prothoate	C9H20NO3PS2	C	-	-	-	-
1.55		0.68	0.87	malathion oxygen analog	C10H19O7PS	C	NR	NR	6-15-50	1-2-3
1.58	NI5	1.21	1.29	chlorfenvinphos, alpha-	C12H14Cl3O4P	C	-	NR	6-15-50	-
1.59		1.92	1.86	DDE, p,p'-	C14H8Cl4	C	C	C	6	1
1.6	FP3	0.31	0.5	monocrotophos	C7H14NO5P	C	NR	NR	6-15-50	1-2-3
1.6		0.4	0.62	dimethoate	C5H12NO3PS2	C	NR	NR	6-15-50	1-2-3
1.61		2.04	2.69	cyproconazole	C15H18ClN3O	C	NR	NR	6-15-50	1-2-3
1.62	NI1	0.77	0.99	Tycor	C9H16N4OS	C	S	S	50	3
1.62		0.43	0.56	BHC, beta-	C6H6Cl6	C	C	C	6	1
1.62		1.39	1.54	chlorbenside	C13H10Cl2S	C	S	P	6	1
1.63	NI1	0.99	1.07	1-hydroxychlordene	C10H6Cl6O	-	R	-	15	-
1.64		1.95	1.88	merphos*	C12H27PS3	-	C	C	6+15+50	3
1.64	NI3/NP20	1.05	1	triadimefon	C14H16ClN3O2	C	S #	S #	50	1-2-3
1.64	FP1/NI11	0.71	0.87	parathion-methyl	C8H10NO5PS	C	C	C	15	2
1.64	NI30	0.6	0.7	ethoxyquin	C14H19N0	C	NR	NR	6-15-50	-
1.65		1.95	1.88	tribufos	C12H27OPS3	C	C	P	15+50	3

Appendix I: PESTDATA Chemicals in Order by OV-225 Relative Retention Time (continued)

RRT/c OV-225	Detector Responses with OV-225 Column	RRT/c OV-101	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
1.67		-	1.44	2,4-D butoxyethyl ester*	C14H18Cl2O4	-	-	-	-	-
1.67	NI6	2.75	2.38	chlordecone	C10H8Cl10O5	-	S #	P #	15+50	1-2-3
1.71		0.9	1.01	dichlofluanid	C9H11Cl2FN2O2S2	C	C #	-	15+50	2+3
1.71		0.55	0.66	parathion-methyl oxygen analog	C8H10NO6P	-	NR	NR	6-15-50	1-2-3
1.71		0.5	0.67	BHC, delta-	C6H6Cl6	C	C	C	6+15	1
1.73		1.36	1.38	isofenphos	C15H24NO4PS	C	C	-	15+50	-
1.74	NI1	1.85	1.82	prothiofos	C11H15Cl2PO2S2	C	C	C	6	2
1.74	FP5	1.17	1.24	isofenphos oxygen analog	C15H24NO5P	C	-	-	-	-
1.76		2.12	1.76	pyrethrins*	C21H27O4	-	C	C	50	-
1.78		2.04	1.78	2,4-D isooctyl ester*	C16H22Cl2O3	-	-	-	-	-
1.78		2.1	1.68	2,4-D ethyl hexyl ester*	C16H22Cl2O3	-	-	-	-	-
1.79		2	-	2,4-D BEP ester*	C17H24Cl2O4	-	-	-	-	-
1.82		0.84	1.05	fenitrothion	C9H12NO5PS	C	C	C	15	2
1.83	NI14	1.73	1.46	butachlor	C17H26ClNO2	C	C	-	50	-
1.84	NI6	1.57	1.64	cyclanilide methyl ester	C12H11Cl2NO3	-	-	-	-	-
1.87		1.91	1.84	dieldrin	C12H8Cl6O	C	C	C	15	2
1.88		1.24	1.47	anilazine	C9H5Cl3N4	V	S	P	15+50	2+3
1.89	NI1000	1.39	1.54	CGA 189138	C13H8O3Cl2	-	-	-	-	-
1.91	NI3/NP65	0.99	0.96	KWG 1323	C14H16ClN3O3	C	NR	NR	6-15-50	1-2-3
1.91	NI(WB)2	0.41	0.66	4-chlorobenzylmethyl sulfone	C8H9ClO2S	-	NR	NR	6-15-50	1-2-3
1.91	FP2/NI6	0.98	1.07	parathion	C10H14NO5PS	C	C	C	15	2
1.93	FS56/NI638	0.86	1.02	ethofumesate	C13H18O5S	C	-	-	-	-
2	FP4/NI5	1.29	1.52	chlorfenvinphos, beta-	C12H14Cl3O4P	C	S #	-	50	1-2-3
2		1.32	1.64	quinalphos	C12H15N2O3PS	C	C	-	15	-
2.01	NI(WB)2	0.69	0.79	vinclozolin metabolite S	C10H7Cl2NO3	V	P	V #	15	2
2.01		2.23	2.42	Perthane	C18H2OCl2	C	C	C	6	1
2.02		0.5	0.5	tris(chloropropyl) phosphate	C9H18Cl3O4P	C	NR	NR	6-15-50	1-2-3
2.03	NI1.5	-	-	fenson	C12H10O3ClS	-	-	-	-	-
2.05		1.31	1.83	phenthoate	C12H17O4PS2	C	C	-	15+50	-
2.08		1.82	1.79	2,4-D butoxyethyl ester*	C14H18Cl2O4	-	-	-	-	-
2.09		3.22	-	2,4-D BEP ester*	C17H24Cl2O4	-	-	-	-	-
2.1	NI(WB)3.8	0.8	-	bromacil methyl ether	C10H16BrN2O2	-	-	-	-	-

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RRT/c OV-225	Detector Responses with OV-225 Column	RRT/c OV-101	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
2.1	NI(WB)7	0.54	0.72	terbacil	C9H13ClN2O2	C	NR	NR	6-15	2+3
2.13		0.85	0.95	linuron	C9H10Cl2N2O2	V	V #	V	50	3
2.22		2.13	2.29	endrin	C12H8Cl6O	C	C #	C #	15	2
2.23		1.44	1.19	triflumizole	C15H15ClF3N3O	C	-	-	-	-
2.25	NI30	0.45	0.71	methidathion sulfoxide	C5H8N2O4S2	-	NR	NR	6-15-50	1-2-3
2.27	NI(WB)630	1.35	2.55	3-tert-butyl-5-chloro-6-hydroxy= methyluracil	C9H13ClN2O3	-	NR	NR	6-15-50	1-2-3
2.27		2.55	2.7	DDT, o,p'-	C14H9Cl5	C	C	C	6	1
2.29	NI400	0.56	0.82	methidathion sulfone	C5H8N2O3S2	-	NR	NR	6-15-50	1-2-3
2.33	NI3	2.64	2.8	tetrasul	C12H6Cl4S	C	C	C	6	1
2.33		1.08	1.3	crufomate	C12H19ClNO3P	C	NR	NR	6-15-50	-
2.33	NI6250	0.5	0.66	ethylenethiourea	C3H6N2S	S	NR	NR	6-15-50	1-2-3
2.34		1.8	2.13	profenofos	C11H15BrClO3PS	C	P	P	50	3
2.36	NI3000	2.3	2.31	fluazifop butyl ester	C19H20F3NO4	C	C	V	15	3
2.46		1.58	2.1	triazamate	C13H22N4O3S	C	NR	NR	6-15-50	1-2-3
2.46		1.9	2.19	TDE, o,p'-	C14H10Cl4	-	C	C	6	1
2.48		1.97	1.96	oxadiazon	C15H18Cl2N2O3	C	C	P	15	-
2.49	NI20	0.44	0.64	prosulfuron*	C15H16F3N5O4S	-	NR	NR	6-15-50	1-2-3
2.55		0.89	1.26	phorate sulfoxide	C7H17O3PS3	C	NR	NR	6-15-50	1-2-3
2.65	NI300	0.6	0.9	CGA 120844	C8H9NSO3	-	NR	NR	6-15-50	1-2-3
2.66		-	-	2,4,5-T butoxyethyl ester*	C14H17Cl3O4	-	-	-	-	-
2.66		2.53	-	Compound K*	C10H6Cl8	-	C	-	-	1
2.67		0.75	-	picloram methyl ester	C7H5Cl3N2O2	-	-	-	-	-
2.67	HN4	1.28	1.58	mecarbam	C10H20NO5PS2	C		-	50	-
2.69		-	-	2,4,5-T isooctyl ester*	C16H21Cl3O3	-	-	-	-	-
2.71	FS2	0.41	0.81	dimethipin	C6H10O4S2	C	NR	NR	6-15-50	1-2-3
2.72		1.58	1.97	Gardona	C10H9Cl4O4P	C	NR	NR	6-15-50	1-2-3
2.73		1.21	1.5	des N-isopropyl isofenphos	C12H18NO4PS	C	S	-	50	-
2.74		3.03	2.88	ethephon	C2H6ClO3P	NR		-	6+15+50	1+2+3
2.77		2	-	aramite*	C15H23ClO4S	C	P	NR	15	-
2.78		3.3	-	2,4,5-T BEP ester*	C17H23Cl3O3	-	-	-	-	-
2.82	NI8	0.66	0.78	propanil	C9H9Cl2NO	C	NR	NR	6-15	3

Appendix I: PESTDATA Chemicals in Order by OV-225 Relative Retention Time (continued)

RRT/c OV-225	Detector Responses with OV-225 Column	RRT/c OV-101	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
2.84		2.95	2.7	pyrethrins*	C21H27O4	-	C	C	50	-
2.85	NI(WB)780	0.35	0.4	ethametsulfuron methyl ester*	C15H18N6O6S	-	NR	NR	6-15-50	1-2-3
2.85		1.37	1.9	crotoxyphos	C14H19O6P	C	NR	NR	6-15-50	1-2-3
2.89	FS63/NI10	0.68	0.93	2,3-dihydro-3,3-methyl-2-oxo-5-benzofuranyl methyl sulfonate	C11H12O5S	-	-	-	-	-
2.9	FP5/NI1500	0.92	1.28	terbufos oxygen analog sulfone	C9H21O5PS2	C	NR	NR	6-15-50	1-2-3
2.9		2.33	2.41	chloropropylate	C17H16Cl2O3	P	C	C	15+50	3
2.91		1.86	1.79	hexaconazole	C14H17Cl2N3O	C	-	-	-	-
2.95		0.71	0.96	demeton-O sulfone*	C8H19O5PS2	C	-	-	-	-
2.95		5.8	5.6	mirex	C10Cl12	P	C	P	6	1
3	FP15/NI300	1.08	1.66	fosthiazate	C9H18NO3PS2	C	NR	NR	6-15-50	1-2-3
3.01		1.23	1.94	folpet	C9H4Cl3O2NS	C	C	P	15+50	2+3
3.02	NI(WB)9	0.89	0.93	vinclozolin metabolite E	C11H11Cl2NO2	C	S	NR	15+50	-
3.02	NI(WB)10	2	3.38	kresoxim-methyl	C18H19NO4	P	C	C	15+50	3
3.04	NI7	1.37	1.49	procymidone	C13H11Cl2NO2	C	C	P	15	-
3.04	NI3	1.61	2.2	ovex	C12H8Cl2O3S	C	C	C	15	2
3.05		2.14	-	aramite*	C15H23ClO4S	C	P	NR	15	-
3.1		2.56	-	2,4,5-T isooctyl ester*	C16H21Cl3O3	-	-	-	-	-
3.14	NI14	1.78	2	diethatyl-ethyl	C16H22ClNO3	C	NR	NR	6-15-50	1-2-3
3.16	NI(WB)120	1.9	1.86	PPG-2597	C20H17ClF3NO6	-	NR	NR	6-15-50	1-2-3
3.24	NI5	2.38	3.14	leptophos photoproduct	C13H11Cl2O2PS	C	-	-	-	-
3.26		0.97	1.3	phorate sulfone	C7H17O4PS3	C	S #	S #	6-15-50	3
3.26		2.31	2.61	chlorobenzilate	C16H14Cl2O3	C	C #	P #	15+50	3
3.28		5.3	-	2,4,5-T BEP ester*	C17H23Cl3O3	-	-	-	-	-
3.3		2.91	-	2,4,5-T butoxyethyl ester*	C14H17Cl3O4	-	-	-	-	-
3.33	NI3	2.52	2.61	nonachlor, cis-	C10H5Cl9	C	C	C	6	1
3.33	NI50	1.4	2.28	methidathion	C6H11N2O4PS3	C	S	P #	50	3
3.39		1.27	1.42	chlorbromuron	C9H10BrClN2O2	V	V	V	50	3
3.4		2.96	-	2,4,5-T isooctyl ester*	C16H21Cl3O3	-	-	-	-	-
3.4	NI7	2.02	2.03	diclobutrazol	C15H19Cl2N3O	C	NR	NR	6-15-50	1-2-3
3.49		1.2	1.85	captan	C9H8Cl3NO2S	C	P	C	50	3
3.5		1.79	2.76	imazamethabenz methyl ester*	C16H20N2O3	C	-	-	-	-

Appendix I: PESTDATA Chemicals in Order by OV-225 Relative Retention Time (continued)

RRT/c OV-225	Detector Responses with OV-225 Column	RRT/c OV-101	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
3.5		1.26	1.92	Sulphenone	C12H9ClO2S	C		-	50	3
3.6	NI(WB)780	0.55	0.95	ethametsulfuron methyl ester*	C15H18N6O6S	-	NR	NR	6-15-50	1-2-3
3.6		1.54	-	2,4-D propylene glycol butyl ether ester*	C15H2OCl2O4	-	-	-	-	-
3.6		3.13	3.5	DDT, p,p'-	C14H9Cl5	C	C	C	6	1
3.7		2	2.6	bupirimate	C13H24N4SO3	C	-	-	-	-
3.7	NP6	1.66	2.41	fenamiphos	C13H22NO3PS	C	NR	NR	6-15-50	1-2-3
3.7	NI9	2.97	4.2	methoxychlor olefin	C16H14Cl2O2	C	C	C	6	2
3.7		1.42	-	fenac	C8H5Cl3O2	-	NR	NR	6-15-50	-
3.77	NI60	0.83	1.06	metribuzin, deaminated metabolite	C8H13N3OS	C	NR	NR	6-15-50	1-2-3
3.8		3.25	-	2,4,5-T isooctyl ester*	C16H21Cl3O3	-	-	-	-	-
3.8		4.9	4.5	bifenthrin	C23H22ClF3O2	V	C	-	6+15	2
3.8	NI3	2.03	2.71	nitrofen	C12H7Cl2NO3	C	C	C	15	2
3.8		2.41	2.87	TDE, p,p'-	C14H10Cl4	C	C	C	6	1
3.9		2.21	2.77	endosulfan II	C9H6Cl6O3S	C	C	C	15+50	2
3.93	FP2/NI8	2.56	3.36	ethion	C9H22O4P2S4	C	C	C	6	2
4	NI(WB)13	2.51	4.2	pyrithiobac-sodium methyl ester	C14H13ClN2O4	-	-	-	-	-
4		2	2.16	oxyfluorfen	C15H11ClF3NO4	C	C	C	15	2
4		1.76	2.08	imazalil	C14H14Cl2N2O	C	NR	NR	6-15-50	-
4.1	FP6	2.22	2.99	chlorthiophos oxygen analog	C11H15Cl2O4PS	C	NR	NR	6-15-50	1-2-3
4.1		1.6	3.17	isoprothiolane	C12H18O4S2	C	-	-	-	-
4.1		1.88	-	ethion oxygen analog	C9H22O5P2S3	C	-	-	-	-
4.2		2.17	3.06	carbophenothion oxygen analog	C11H16ClO3PS2	C	NR	NR	6-15-50	1-2-3
4.2		2.94	3.7	carbophenothion	C11H16ClO2PS3	C	C	P	6	2
4.2		2.19	2.38	binapacryl	C15H18N2O6	C	P	P	15	-
4.3	NI160	2.4	3	imazethapyr ammonium salt methyl ester	C16H21N3O3	-	-	-	-	-
4.3		1.15	1.54	CGA 91305	C10H8Cl2N3O	V	NR	NR	6-15-50	1-2-3
4.3	NI12	1.5	2.67	TCMTB	C9H6N2S3	C	P	P	15	-
4.5		32	-	hydramethylnon*	C25H24F6N4	-	-	-	-	-
4.5	NI23	3.3	5	methoxychlor, o, p'-	C16H15Cl3O2	-	C	-	6	-
4.5		6.4	6.1	bis(2-ethylhexyl) phthalate	C24H38O4	-	C	C	15+50	-

Appendix I: PESTDATA Chemicals in Order by OV-225 Relative Retention Time (continued)

RRT/c OV-225	Detector Responses with OV-225 Column	RRT/c OV-101	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
4.7	NI60	1.13	1.38	RPA202248	C15H12SNO4F3	NR	NR	NR	6-15-50	1-2-3
4.7	NI40	1.11	1.33	isoxaflutole (prop)	C15H12SNO4F3	NR	V #	S #	50	3
4.7		2.85	-	chlornitrofen	C12H6Cl3NO3	C	C	C	6+15	2
4.8		5.4	6.5	phenothrin*	C23H26O3	-	-	-	-	-
4.8		-	-	Dilan*	C15.5H14Cl2NO2	-	P	P	15	-
4.8	NI1300	3.8	4.3	propargite	C19H26O4S	C	C	-	15	2
4.8	NI(WB)12	0.8	1.36	bromacil	C9H13BrN2O2	C	NR	NR	6-15-50	1-2-3
4.9	NI12	3.57	4.7	diclofop-methyl	C16H14Cl2O4	C	C	C	15	2
4.9		0.89	1.48	cyanazine	C9H13ClN6	C	NR	-	6-15-50	-
5	NI(WB)6	2.14	2.4	PPG-947, methylated*	C18H13ClF3NO7	-	-	-	-	-
5	NI(WB)4	2.15	2.4	PPG-847, methylated	C15H9ClF3NO3	-	-	-	-	-
5.1		4.1	-	2,4-D BEP ester*	C17H24Cl2O4	-	-	-	-	-
5.1		3.06	4.5	butyl benzyl phthalate	C19H20O4	-	C	P	15+50	-
5.3		-	-	Dilan*	C15.5H14Cl2NO2	-	P	P	15	-
5.5		5.5	9.1	phosalone	C12H15ClNO4PS2	C	C	C	50	2+3
5.6		3.21	4	propiconazole*	C15H17Cl2N3O2	C	NR	NR	6-15-50	1-2-3
5.8		2.33	-	Dilan*	C15.5H14Cl2NO2	-	P	P	15	-
5.8	FP60	1.15	1.75	demeton-S sulfone	C8H19O5PS2	C	-	-	-	-
5.8	NI(WB)5	3.26	4.67	clodinafop-propargyl	C17H13ClFNO4	V	V	-	50	3
6.3		2.87	5.3	edifenphos	C14H15O2PS2	C	-	-	-	-
6.5	NI5	1.53	2.41	CGA 94689A	C15H21NO5	V	NR	NR	6-15-50	1-2-3
6.5		4.4	-	bromopropylate	C17H16Br2O3	C	C #	C #	15+50	1-2-3
6.6	FS175/NI400	1	1.46	2-hydroxy-2,3-dihydro-3,3-methyl-5-benzofuranyl methyl sulfonate	C11H14O5S	-	-	-	-	-
6.6	NI8	1.54	2.45	CGA 94689B	C15H21NO5	S	NR	NR	6-15-50	1-2-3
6.6	NI(WB)5	4.8	6.3	cloquintocet-mexyl	C18H22ClNO3	V	NR	-	6-15-50	1-2-3
6.7		1.5	2.39	disulfoton sulfone	C8H19O4PS3	C	NR	-	6-15-50	-
6.9		4.3	4.4	dinocap*	C18H24N2O6	C	P	P	15	2
7	NI10	4.8	5.7	fenpropathrin	C22H23NO3	-	V #	V	15	2
7.2		1.9	2.6	myclobutanil	C15H17ClN4	C	NR	NR	6-15-50	1-2-3
7.2		4.7	7.2	methoxychlor, p, p'-	C16H15Cl3O2	C	C	C	6	2
7.3		3.36	4.8	nuarimol	C17H12ClFN2O	C	NR	C #	50	1-2-3

Appendix I: PESTDATA Chemicals in Order by OV-225 Relative Retention Time (continued)

RRT/c OV-225	Detector Responses with OV-225 Column	RRT/c OV-101	RRT/c OV-17		Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
7.5		2.81	-		Dilan*	C15.5H14Cl2NO2	-	P	P	15	-
7.5	NI10	2.81	3.9		Prolan	C15H13Cl2NO2	P	S	S	15	2
7.5	NI6	3.06	4.4		Bulan	C16H15Cl2NO2	C	P	P	15	2
7.6		4.2	6.5		leptophos oxygen analog	C13H10BrCl2O3P	C	-	-	-	-
7.7		7	-		2,4,5-T BEP ester*	C17H23Cl3O3	-	-	-	-	-
7.7		4.8	4.8		dinocap*	C18H24N2O6	C	P	P	15	2
7.7		5.8	8.5	1	leptophos	C13H10BrCl2O2PS	C	C	C	6	2
7.9		1.5	-		procyazine	C10H13ClN6	C	-	-	-	-
8	NI6	1.25	1.09		MB45950	C12H4SN4F6Cl2	S	P	V	15+50	2+3
8.2		3.39	-		Dilan*	C15.5H14Cl2NO2	-	P	P	15	-
8.2		3.1	4.6		cyanofenphos	C15H14NO2PS	C	-	-	-	-
8.3		2.83	4		endosulfan sulfate	C9H6Cl6O4S	C	C	C	50	2
8.4	NI6	4.3	6		benzoylprop-ethyl	C18H17Cl2NO3	P	NR	NR	6-15-50	1-2-3
8.5		4.5	7.2		tetramethrin*	C19H25NO4	C	NR	NR	6-15-50	1-2-3
8.6	NI7	4.7	7.2		tetrasul sulfoxide	C12H6Cl4OS	-	-	-	-	-
8.7	NI10	1.35	1.16		fipronil	C12H4Cl2F6N4OS	S	S	V	50	3
8.9	NI500	12	6.1		CGA 205374	C16H11N3O2Cl2	-	NR	NR	6-15-50	1-2-3
8.9		1.3	-		chlorsulfuron	C12H12ClN5O4S	-	NR	NR	6-15-50	-
9.5		5.1	5.6		dinocap*	C18H24N2O6	C	P	P	15	2
9.7		4.8	6.8		piperophos	C14H28NO3PS2	C	-	-	-	-
10.3	FP15	4.7	6.9		chlorthiophos sulfoxide	C11H15Cl2O4PS2	C	NR	NR	6-15-50	1-2-3
10.3		3.6	-		endrin ketone	C12H8Cl6O	-	C	C	50	2
10.6		4.5	6.9		EPN	C14H14NO4PS	C	C	C	15	2
10.8	NI20	7.9	12.6		HOE-030291	C17H16Cl2O5	-	-	-	-	-
10.9		11.5	15		phenothrin*	C23H26O3	-	-	-	-	-
11.1		9.4	13.8		permethrin, cis-	C21H20Cl2O3	C	V #	C	6+15	2
11.3		8.1	10.5		fenoxaprop ethyl ester	C18H16NO5Cl	S	V	V	50	3
11.5	NI60	2.96	4.1		CL 202,347	C13H19N3O5	-	-	-	-	-
12.8	NI40	10.4	8.9		acrinathrin	C26H21F6NO5	V	V	V #	15	2
13		10.2	-		2,4-D BEP ester*	C17H24Cl2O4	-	-	-	-	-
13		10.2	15		permethrin, trans-	C21H20Cl2O3	C	V #	C	6+15	2
13	NI3700	2.67	8		pyrazon	C10H8ClN3O	C	NR	NR	6-15-50	1-2-3

Appendix I: PESTDATA Chemicals in Order by OV-225 Relative Retention Time (continued)

RRT/c OV-225	Detector Responses with OV-225 Column	RRT/c OV-101	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
13	NI1200	13	16	hexachlorophene	C13H6Cl6O2	-	NR	NR	6-15-50	-
14	NI4500	2.5	5	oxadixyl	C14H18N2O4	C	NR	NR	6-15-50	1-2-3
14		4.2	8.7	pyridaphenthion	C14H17O4N2SP	C	-	-	-	-
14		2.65	5	famphur	C10H16NO5PS2	C	NR	-	6-15-50	-
14.8	NI600	3.1	7.1	phosmet oxygen analog*	C11H12NO5PS	-	NR	NR	6-15-50	-
14.9		5	8.8	bifenox	C12H9Cl2NO5	C	C	P	15+50	2+3
14.9	FP50	4	8.4	phosmet	C11H12O4NPS2	C	NR	-	6-15-50	3
15		2.31	3.93	desisopropyl iprodione	C10H6Cl2N3O3	P		-	50	1-2-3
15	NP1000	4	4.2	KWG 1342	C14H18ClN3O3	-	-	-	-	-
15.5		4.4	8.5	photodieldrin	C12H8Cl6O	-	C	C	15+50	2
18	NI75	4.2	6.3	iprodione*	C13H13Cl2N3O3	C	S #	NR	50	1-2-3
18.1		-	-	carbofuran-7-phenol-DNP ether	C16H14N2O6	-	-	-	-	-
18.6	NI44	2.62	5.4	ofurace	C14H16NO3Cl	C	-	-	-	-
18.8	FP39	5.3	9.1	chlorthiophos sulfone	C11H15Cl2O5PS2	C	C	-	50	3
19.7		31	38	deltamethrin, trans-*	C22H19Br2NO3	-	P #	NR	15	2
19.9		29	38	deltamethrin*	C22H19Br2NO3	C	S #	P	15	2
24		16.3	-	fluridone	C19H14F3NO	-	NR	NR	6-15-50	-
24	NI21	3.8	6.3	nitralin	C13H19N3O6S	C	P	P	50	3
29		-	-	cypermethrin*	C22H19Cl2NO3	C	C	C	15	2
31	NI30	2.06	1.98	MB46136	C12H4SO2N4F6Cl2	S	S	V	50	2+3
33		14.1	23	cypermethrin*	C22H19Cl2NO3	C	C	C	15	2
36		15.1	25	cypermethrin*	C22H19Cl2NO3	C	C	C	15	2
36.9		14.7	21.4	flucythrinate*	C26H23F2NO4	C	C	-	15	2+3
37.1		3.6	7.5	myclobutanil alcohol metabolite	C15H17ClN40	S	NR	NR	6-15-50	1-2-3
40	NI100	9	18	coumaphos	C14H16ClO5PS	C	NR	C #	6-15-50	3
42		16.1	24	flucythrinate*	C26H23F2NO4	C	C	-	15	2+3
44		20.3	35	fenvalerate*	C25H22ClNO3	C	C	C	15	2
45	NI150	8	16	coumaphos oxygen analog	C14H16ClO6P	C	NR	NR	6-15-50	1-2-3
51		22.5	40	fenvalerate*	C25H22ClNO3	C	C	C	15	2
53		44	-	hydramethylnon*	C25H24F6N4	-	-	-	-	-
53		4.7	11.3	dithianon	C14H4O2N2S2	NR	-	-	-	-
56		-	35	fluvalinate*	C26H22ClF3N2O3	C	C	-	15	2

Appendix I: PESTDATA Chemicals in Order by OV-225 Relative Retention Time (continued)

RRT/c OV-225	Detector Responses with OV-225 Column	RRT/c OV-101	RRT/c OV-17	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
57	NI300	23	43	PB-7, methylated	C20H25ClN2O3S	-	-	-	-	-
59		25	38	fluvalinate*	C26H22ClF3N2O3	C	C	-	15	2
64		27	44	tralomethrin	C22H19Br4NO3	C	V	S	15	2
87	NI500	25	46	PB-9	C19H25ClN2O2S	V	NR	NR	6-15-50	1-2-3

Appendix I: PESTDATA Chemicals in Order by OV-17 Relative Retention Time

RRT/c OV-17	Detector Responses with OV-17 Column	RRT/c OV-101	RRT/c OV-225	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
0.02	NI4	0.03	-	dichlorobenzene, p-	C6H4Cl2	-	C	C	6	1
0.02	NI0.1	-	-	hexachloroethane	C2Cl6	-	-	-	-	-
0.03	NI0.2	0.04	-	dibromochloropropane	C3H5Br2Cl	-	-	-	-	-
0.04	NI10	0.06	0.08	CGA 171683	C6H5F4N3O2	C		-	15+50	3
0.04	NI0.1	-	-	hexachlorobutadiene	C4Cl6	-	V #	P	6	1
0.06	NI0.8	0.12	-	hexachlorocyclopentadiene	C5Cl6	-	-	-	-	-
0.07	NI0.1	-	-	1,2,3,5-tetrachlorobenzene	C6H2Cl4	-	P #	-	6	1
0.07	NI0.2	-	-	1,2,4,5-tetrachlorobenzene	C6H2Cl4	-	-	-	-	-
0.07	NP(WB)1.5	-	-	4-chlorobenzeneamine	C6H6ClN	S	NR	NR	6-15-50	1-2-3
0.08	FP2.5	0.07	0.08	dichlorvos	C4H7Cl2O4P	C	NR	NR	6-15-50	1-2-3
0.09	NP(WB)5	-	0.11	teflubenzuron*	C14H6Cl2F4N2O2	-	NR	NR	6-15-50	1-2-3
0.09	FP1/FP(WB)0.6	0.07	0.25	methamidophos	C2H8NO2PS	V	-	-	-	-
0.09	FP11	0.15	-	vernolate	C10H21NOS	-	P	-	15	-
0.09	NI0.2	-	-	1,2,3,4-tetrachlorobenzene	C6H2Cl4	-	-	-	-	-
0.1	NI(WB)1.4/NP23	0.13	0.15	chlorimuron ethyl ester	C15H15ClN4O6S	P	NR	-	-	-
0.1	HX1.5	0.19	0.08	2-methoxy-3,5,6-trichloropyridine	C6H4Cl3NO	C	P #	C	6+15	1+2
0.1	NI5	0.17	0.14	N-(3,4-dichlorophenyl)-N'-methylurea	C8H8Cl2N2O	-	NR	NR	6-15-50	-
0.1	NI4	0.22	0.14	3,4-dichlorophenylurea	C7H6Cl2N2O	-	NR	NR	6-15-50	-
0.1	NI0.6	0.11	-	dichlobenil	C7H3Cl2N	C	P	C	15	2
0.1	NP6	0.17	-	pebulate	C10H21NOS	C	P	-	15	-
0.11	NP20	0.17	-	CGA 236431	C8H7F3N2O2	-	-	-	-	-
0.11	FP0.4	-	-	chlormephos	C5H12ClO2PS2	C	-	-	-	-
0.11	FS30	0.13	0.23	carboxin sulfoxide	C12H13NO3S	-	NR	NR	6-15-50	1-2-3
0.11	NI12	0.11	0.09	diuron	C9H10Cl2N2O	C	NR	NR	6-15-50	1-2-3
0.12	NI0.2	-	-	hexachloronorbornadiene	C7H2Cl6	-	-	-	-	-
0.12	NP16	0.13	-	propham	C10H13NO2	C	P	P	15	-
0.13	NP8	0.26	-	CGA 236432	C9H9F3N2O2	-	-	-	-	-
0.13	NI1/NP2	0.17	0.22	3-methyl-4-nitrophenol methyl ether	C8H9O3N	-	-	-	-	-
0.13	FP2	0.16	-	mevinphos, (E)-	C7H13O6P	C	NR	NR	6-15-50	-

* Multipeak chemical.

Recovery may vary with choice of Florisil elution system; see Tables 303-a, 304-a.

Appendix I: PESTDATA Chemicals in Order by OV-17 Relative Retention Time (continued)

RRT/c OV-17	Detector Responses with OV-17 Column	RRT/c OV-101	RRT/c OV-225	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
0.13	FP4	0.16	-	trichlorfon	C4H8Cl3O4P	C	NR	NR	6-15-50	1-2-3
0.14	NP(WB)5	0.14	0.23	teflubenzuron*	C14H6Cl2F4N2O2	-	NR	NR	6-15-50	1-2-3
0.14	NP50	0.22	-	CGA 72903	C7H6F3N	-	-	-	-	-
0.14	NI1	0.19	0.22	N, N-diallyl dichloroacetamide	C8H11Cl2NO	C	S	S	15+50	2+3
0.14	NP2	-	-	fluometuron	C10H11F3N2O	-	-	-	-	-
0.14	NP1	0.22	-	CGA 150829	C5H14N4O	V	-	-	-	-
0.14	HN(WB)0.3/HX(WB)2/NI8/ NI(WB)14/NP8/NP(WB)0.4	0.18	0.27	3,5-dichloroaniline	C6H5Cl2N	S	S	S	6+15	1+2
0.14		0.15	0.15	dimethyl phthalate	C10H10O4	-	P	-	6+15+50	-
0.15	FP2/FP(WB)0.6	0.13	-	mevinphos, (Z)-	C7H13O6P	C	NR	-	6-15-50	-
0.16	HN(WB)3/NI(WB)120/ NP(WB)7	0.25	0.5	cymoxanil	C7H10N4O3	V	NR	NR	6-15-50	1-2-3
0.16	NI0.3	0.24	0.13	pentachlorobenzene	C6HCl5	C	C	C	6	1
0.16	NI13/NP8	0.2	0.32	3,4-dichloroaniline	C6H5Cl2N	V	S	-	15	-
0.17	NI0.7	0.34	0.27	trifluralin	C13H16F3N3O4	C	C	C	6	2
0.18	FP(WB)2	-	-	metasystox thiono*	C6H15O3PS2	-	-	-	-	-
0.18	HX25	-	-	metoxuron	C10H13ClN2O2	V	NR	NR	6-15-50	1-2-3
0.18	NI0.3	0.23	0.21	methyl 2,3,6-trichlorobenzoate	C8H5Cl3O2	-	-	-	-	-
0.18	HX(WB)1	0.37	0.28	benfluralin	C13H16F3N3O4	C	C	C	6	2
0.19	HX6	0.34	0.27	ethalfluralin	C13H14F3N3O4	C	C	C	6	2
0.19	FP(WB)0.6	0.15	0.64	acephate	C4H10NO3PS	C	-	-	-	-
0.2	FP2	-	-	demeton-O*	C8H19O3PS2	C	NR	-	6-15	-
0.2	NI160/NP30	0.22	0.25	3-carboxy-5-ethoxy-1,2,4-thiadiazole	C3H2N2O3S	NR	-	-	-	-
0.2		-	-	4-chlorobiphenyl	C12H9Cl	-	-	-	-	-
0.2	HN(WB)1/HX(WB)35/ NP(WB)11	0.3	-	triflusulfuron methyl ester	C17H19F3N6O6S	V	NR	NR	6-15-50	1-2-3
0.2		-	-	epoxyhexachloronorbornene	C7H2Cl6O	-	-	-	-	-
0.2	HX(WB)0.6	0.2	0.18	nitrapyrin	C6H3Cl4N	C	C	V	6	2
0.21	FP20	0.26	-	tebuthiuron	C9H16N4OS	-	-	-	-	-
0.21	FP25	0.22	0.32	demeton-O oxygen analog	C8H19O4PS	-	-	-	-	-
0.21	HX(WB)0.8/NI0.4/NP0.5	0.18	0.12	etridiazole	C5H5Cl3N2OS	C	C	P	6	2
0.22	NI150	0.18	-	4-hydroxymethyl-3,5-dimethylphenyl methylcarbamate*	C11H15NO3	-	NR	NR	15-50	1-2-3

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Appendix I: PESTDATA Chemicals in Order by OV-17 Relative Retention Time (continued)

RRT/c OV-17	Detector Responses with OV-17 Column	RRT/c OV-101	RRT/c OV-225	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
0.22	NI0.2	-	-	octachlorocyclopentane	C5Cl8	-	-	-	-	-
0.22	HX(WB)0.3	0.24	0.15	2,3,5,6-tetrachloroanisole	C7H4Cl4O	-	C	-	6	1
0.23	NI0.4	0.26	0.51	RPA 203328, methylated	C10H9F3O4S	-	-	-	-	-
0.23	NI0.2	-	-	heptachloronorbornene	C7H3Cl7	-	-	-	-	-
0.23		0.3	-	tributyl phosphate	C12H27O4P	-	R	-	50	-
0.24	FP150	0.5	0.53	phosmet oxygen analog*	C11H12NO5PS	-	NR	NR	6-15-50	-
0.24	FP0.5/HX0.3/NI0.3	0.33	0.23	chlorethoxyfos	C6H11Cl4O3PS	V	C	-	6	1
0.24	NI0.3	0.29	0.26	tecnazene	C6HCl4NO2	C	C	C	6	1
0.24	FP(WB)70/NI10000/ NP(WB)70	0.21	0.27	TEPP	C8H20O7P2	C	-	-	-	-
0.25		0.3	0.38	2,4-D methyl ester	C9H8Cl2O3	-	-	-	-	-
0.25		0.34	-	2,4,5-trichloro-alpha-methylbenzene methanol	C8H7OCl3	R	R	-	15	-
0.25	FP1	0.33	0.31	ethoprop	C8H19O2PS2	C	P #	S #	50	1-2-3
0.25		0.29	-	diphenylamine	C12H11N	C	S	-	6+15	-
0.25	NI80	0.32	0.43	chlorpropham	C10H12ClNO2	C	C	C	15	2
0.26	NI5/NP20	0.38	0.63	3-methyl-4-nitrophenol	C7H7O3N	V	NR	NR	6-15-50	1-2-3
0.26	NI5/NI(WB)5	0.34	0.37	propachlor	C11H14ClNO	C	NR	NR	6-15-50	1-2-3
0.26	FP0.5	0.26	-	thionazin	C8H13N2O3PS	C	P	NR	15+50	-
0.27		0.2	-	1,2,4-triazole	C2H3N3	V	NR	NR	6-15-50	1-2-3
0.28	FP3	-	-	demeton-O sulfone*	C8H19O5PS2	C	-	-	-	-
0.28	HN(WB)0.4/NI(WB)3/ NP(WB)3	0.25	0.92	oxamyl oxime metabolite	C5H10N2O2S	C	NR	NR	6-15-50	1-2-3
0.29	FP(WB)0.4/NI(WB)12/ NP(WB)0.5	0.37	0.27	cadusafos	C10H23O2PS2	C	NR	NR	6-15-50	1-2-3
0.29	FP0.5	0.34	-	sulfotep	C8H20O5P2S2	C	C	P	6+15	2
0.29	NP3	0.28	-	G-27550	C8H12N2O	C	-	-	-	-
0.29	FP1	0.3	0.37	phorate oxygen analog	C7H17O3PS2	C	NR	NR	6-15-50	1-2-3
0.3	NI0.8/NP9	0.53	0.46	profluralin	C14H16F3N3O4	V	V	-	6	-
0.31	NI150	0.27	-	4-hydroxymethyl-3,5-dimethylphenyl methylcarbamate*	C11H15NO3	-	NR	NR	15-50	1-2-3
0.31	FP(WB)4	0.46	0.49	oxydemeton-methyl	C6H15O4PS2	C	-	-	-	-
0.32	FP(WB)2	0.31	0.49	metasystox thiono*	C6H15O3PS2	-	-	-	-	-

Appendix I: PESTDATA Chemicals in Order by OV-17 Relative Retention Time (continued)

RRT/c OV-17	Detector Responses with OV-17 Column	RRT/c OV-101	RRT/c OV-225	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
0.32	NI0.4	0.56	0.4	chlordene	C10H6Cl6	-	C	C	6	1
0.32	FP(WB)0.8	0.28	0.49	metasystox thiol	C6H15O3PS2	C	-	-	-	-
0.32	FP20/NI8	0.34	-	naled	C4H7Br2Cl2O4P	C	NR	NR	6-15-50	1-2-3
0.32	FP0.5/NI14	0.37	0.38	phorate	C7H17O2PS3	C	V #	V #	6	1
0.33		0.42	0.74	2,4-D isopropyl ester*	C11H12Cl2O3	-	-	-	-	-
0.33		0.42	0.26	di-allate	C10H17ClNOS	C	C	-	6	-
0.33	HX0.3/NI0.3	0.45	0.25	hexachlorobenzene	C6Cl6	C	C	P	6	1
0.34	NI0.3	0.46	0.3	pentachlorophenyl methyl ether	C7H3Cl5O	C	C	C	6	1
0.35	NI0.3	0.4	0.48	BHC, alpha-	C6H6Cl6	C	C	C	6	1
0.36	FP2	0.28	-	demeton-O*	C8H19O3PS2	C	NR	-	6-15	-
0.36		0.32	0.44	3, 5, 6-trichloro-2-pyridinol methyl ester	C6H4Cl3NO	-	-	-	-	-
0.36	FP0.7	0.34	-	dioxabenzofos	C8H9O3PS	C	P	-	15	-
0.36	NI1	0.38	0.44	sulfallate	C8H14ClNS2	C	C	C	6+15	2
0.37	HX3/NI0.5	0.53	0.76	fluchloralin	C12H13ClF3N3O4	C	C	-	6	2
0.38	NP4	0.35	0.6	2,3,5-trimethacarb	C11H15NO2	C	S #	NR	50	1-2-3
0.38	FP4/FP(WB)1	0.39	0.53	fonofos oxygen analog	C10H15O2PS	V	NR	NR	6-15-50	1-2-3
0.39		0.36	0.36	triclopyr methyl ester	C8H6Cl3NO3	-	-	-	-	-
0.39	NI1/NP1.8	0.46	0.6	furilazole	C11H13Cl2NO3	C	S	-	50	3
0.39	FP1/NI2500	0.42	-	terbufos oxygen analog	C9H21O3PS2	C	-	NR	6-15-50	1-2-3
0.39	FP5/FP(WB)1.1	0.25	1.11	omethoate	C5H12NO4PS	C	NR	NR	6-15-50	1-2-3
0.4	HN(WB)30	0.35	2.85	ethametsulfuron methyl ester*	C15H18N6O6S	-	NR	NR	6-15-50	1-2-3
0.4	NI1/NP85	0.51	0.84	pronamide	C12H11Cl2NO	C	P	-	15+50	-
0.4	FP0.4/NP(WB)1	0.41	0.51	thiometon	C6H15O2PS3	C	NR	NR	6-15-50	-
0.41	NP24	0.35	0.7	methabenzthiazuron	C10H11N3OS	C	NR	NR	6-15-50	1-2-3
0.41	NP40	0.32	-	phenmedipham	C16H16N2O4	-	-	-	-	-
0.41	FP0.5/FP(WB)1.6/NI20	0.5	0.44	terbufos	C9H21O2PS3	C	P	S	6	-
0.41	NI43	0.53	0.65	propazine	C9H16ClN5	C	S	NR	15+50	3
0.41	FP0.8/FP(WB)0.8	0.41	0.56	demeton-S	C8H19O3PS2	C	NR	-	6-15-50	-
0.42	FP0.5	0.48	-	propetamphos	C10H20NO4PS	C	C #	-	15+50	2+3
0.42	NP9	0.42	-	melamine	C3H6N6	NR	-	-	-	-
0.43	FP1/FP(WB)0.8	0.31	0.96	dicrotophos	C8H16NO5P	C	NR	-	6-15-50	-

Appendix I: PESTDATA Chemicals in Order by OV-17 Relative Retention Time (continued)

RRT/c OV-17	Detector Responses with OV-17 Column	RRT/c OV-101	RRT/c OV-225	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
0.44	NI150	0.5	0.79	metribuzin, deaminated diketo metabolite*	C7H11N3O2	NR	NR	NR	6-15-50	1-2-3
0.44	FP150	2	0.93	phosmet oxygen analog*	C11H12NO5PS	-	NR	NR	6-15-50	-
0.44	NI1	0.52	0.93	dinitramine	C11H13F3N4O4	C	-	P	15	-
0.44	NI20	0.43	0.74	atrazine	C8H14ClN5	C	S #	NR	50	1-2-3
0.44	FP0.7	0.52	0.56	fonofos	C10H15OPS2	C	C	C	6	2+3
0.44	FP0.7/FP(WB)0.9/NI4/ NP0.25	0.51	0.4	diazinon	C12H21N2O3PS	C	C	C	15	3
0.45	NP430	0.9	-	formetanate hydrochloride	C11H16ClN3O2	-	-	-	-	-
0.45	NI3	0.5	0.59	pentachlorobenzonitrile	C7Cl5N	C	C	P	15	2
0.45	NI1000/NP218	0.44	0.29	desmedipham	C16H16N2O4	-	-	-	-	-
0.45		0.6	-	tri-allate	C10H16Cl3NOS	C	C	C	6	2
0.45	HN(WB)0.4	0.42	0.75	chlorbufam	C11H10ClNO2	C		-	15	2+3
0.45	NI0.4	0.42	0.96	dicloran	C6H4Cl2N2O2	C	S	P	15+50	2+3
0.46	HX2/NP11	0.45	0.59	clomazone	C12H14ClNO2	C		-	50	3
0.46	FP1	0.54	0.6	disulfoton	C8H19O2PS3	C	P #	NR	6	1-2-3
0.46	NI0.5	0.51	0.46	quintozene	C6Cl5NO2	C	C	C	6	1
0.47	NI(WB)1	0.56	0.49	methyl 3,5-dibromo-4-methoxy= benzoate	C9H8Br2O3	-	-	-	-	-
0.47		0.49	0.63	2,4,5-T methyl ester	C9H7Cl3O3	-	-	-	-	-
0.47	NP100	0.4	-	CGA 37734	C10H13NO2	C	NR	NR	6-15-50	1-2-3
0.47	NI30/NP0.6	0.5	0.53	diazinon oxygen analog	C12H21N2O4P	C	NR	NR	6-15-50	1-2-3
0.47	NI0.4	0.48	0.69	lindane	C6H6Cl6	C	C	C	6	1
0.48	NI1.1/NP1.2	0.5	0.69	4-(dichloroacetyl)-1-oxa-4-azapiro= [4.5]decane	C10H15Cl2NO2	C	P	-	50	3
0.48	HX(WB)0.3	0.49	0.35	1,2,4,5-tetrachloro-3-(methylthio)= benzene	C7H4Cl4S	R	C	-	6	1
0.48	NI250	0.47	0.71	terbuthylazine	C9H16N5Cl	C	P	-	15+50	-
0.48	HX18	0.48	0.91	monolinuron	C9H11ClN2O2	C	-	-	-	-
0.49	HN(WB)5	0.42	0.97	PPG-947, methylated*	C18H13ClF3NO7	-	-	-	-	-
0.49	NP3	0.46	-	CGA 51702	C9H9F3N2O	-	-	-	-	-
0.49		0.62	0.62	2,4-D isobutyl ester	C12H14Cl2O3	-	-	-	-	-
0.5		0.5	2.02	tris(chloropropyl) phosphate	C9H18Cl3O4P	C	NR	NR	6-15-50	1-2-3

Appendix I: PESTDATA Chemicals in Order by OV-17 Relative Retention Time (continued)

RRT/c OV-17	Detector Responses with OV-17 Column	RRT/c OV-101	RRT/c OV-225	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
0.5	FP2/FP(WB)0.8	0.31	1.6	monocrotophos	C7H14NO5P	C	NR	NR	6-15-50	1-2-3
0.5	NP10	0.45	0.78	3,4,5-trimethacarb	C11H15NO2	C	NR	NR	50	1-2-3
0.5	NI56/NP(WB)1.5	0.41	0.83	simazine	C7H12ClN5	C	NR	NR	50	1-2-3
0.5	FP7/FP(WB)13	0.47	-	dioxathion	C12H26O6P2S4	V	NR	-	6-15-50	2
0.51		-	-	4,4'-dichlorobiphenyl	C12H8Cl2	-	-	-	-	-
0.51	NI50	0.58	0.59	etrimfos	C10H17N2O4PS	C	C	C	15	2+3
0.52	NI150	0.73	1.29	metribuzin, deaminated diketo metabolite*	C7H11N3O2	NR	NR	NR	6-15-50	1-2-3
0.52		0.39	1.3	2,6-dichlorobenzamide	C7H5NOCl2	C	NR	NR	6-15-50	1-2-3
0.52	FP1	0.58	-	schradan	C8H24N4O3P2	C	NR	-	6-15-50	-
0.53	NP(WB)25	0.47	0.42	terbumeton	C10H19N5O	C	-	-	-	-
0.53	HN(WB)0.1/HX12	0.3	0.8	desethyl simazine	C5H8ClN5	-	NR	NR	50	1-2-3
0.54	FP2	0.6	-	iprobenfos	C13H21O3PS	C	-	-	-	-
0.54	NI(WB)16	0.39	1.16	4-chlorobenzylmethyl sulfoxide	C8H9ClOS	-	NR	NR	6-15-50	1-2-3
0.55	NI6/NP20	0.56	1.41	metribuzin, diketo metabolite	C7H12N4O2	NR	NR	NR	6-15-50	1-2-3
0.56	HX(WB)0.5	0.56	0.63	2,3,5,6-tetrachloronitroanisole	C7H3Cl4NO3	-	C	-	6	1+2
0.56	NI1	0.43	1.62	BHC, beta-	C6H6Cl6	C	C	C	6	1
0.56	FP0.8/HX(WB)2	0.67	0.64	dichlofenthion	C10H13Cl2O3PS	C	C	V	6	2
0.56		0.65	0.61	diisobutyl phthalate	C16H2204	-	P	-	15+50	-
0.57	FP2	0.53	-	phosphamidon*	C10H19ClNO5P	C	NR	NR	6-15-50	1-2-3
0.58	NP24	0.7	-	CP 51214	C14H21NO3	C	NR	NR	6-15-50	1-2-3
0.59	FP(WB)0.7/NP(WB)1	0.47	-	cyanophos	C9H10O3NSP	C	-	-	-	-
0.6	HN(WB)0.4/HX(WB)4/ NI(WB)36/NP(WB)2	0.43	1.34	6-chloro-2,3-dihydro-3,3,7-methyl-5H-oxazolo(3,2-a)pyrimidin-5-one	C9H13ClN2O2	-	NR	NR	6-15-50	1-2-3
0.6	NI0.4	0.83	0.52	heptachlor	C10H5Cl7	C	C	C	6	1
0.61	HN(WB)0.1/HX25	0.2	0.86	desdiethyl simazine	C3H4ClN5	-	NR	NR	6-15-50	1-2-3
0.62	NP50	-	-	CGA 27092	C8H7F3N2O	-	-	-	-	-
0.62	NP14	1.09	-	fenpropimorph	C20H33NO	C		-	50	1-2-3
0.62		0.54	1.47	fenfuram	C12H11NO2	C	-	-	-	-
0.62	FP135	0.43	-	fenthion oxygen analog sulfoxide	C10H15O5PS	C	NR	NR	6-15-50	1-2-3
0.62	FP5/FP(WB)1	0.64	1.02	ronnel oxygen analog	C8H8Cl3O4P	C	NR	-	6-15-50	-
0.62	FP0.8/FP(WB)0.8/NI5	0.4	1.6	dimethoate	C5H12NO3PS2	C	NR	NR	6-15-50	1-2-3

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Appendix I: PESTDATA Chemicals in Order by OV-17 Relative Retention Time (continued)

RRT/c OV-17	Detector Responses with OV-17 Column	RRT/c OV-101	RRT/c OV-225	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
0.63		0.51	0.8	etrimfos oxygen analog	C10H17N2O5P	C	-	-	-	-
0.63	FP(WB)1/NI10/NP0.2	0.55	0.8	isazofos	C9H17ClN3O3PS	C	C #	-	50	2+3
0.64	NI120	0.44	2.49	prosulfuron*	C15H16F3N5O4S	-	NR	NR	6-15-50	1-2-3
0.64	HX1/NI2	0.69	1.15	vinclozolin	C12H9Cl2NO3	C	C	C	15	2
0.64		0.66	-	octhilinone	C11H19NOS	C	-	-	-	-
0.66	HN(WB)6/HX(WB)9/ NI(WB)10/NP(WB)19	0.74	1.2	vinclozolin metabolite B	C12H11Cl2NO4	C	P #	C	6+15	2
0.66	HX0	0.54	-	4-chloro-6-methoxyindole	C9H8NOCl	-	R	-	15	-
0.66	HX(WB)0.6	0.59	0.73	2,3,5,6-tetrachloroanisidine	C7H5Cl4NO	-	C	-	6	2
0.66	NP(WB)50	0.94	0.97	prodiamine	C13H17F3N4O4	C	-	-	-	-
0.66	NI(WB)0.8	0.41	1.91	4-chlorobenzylmethyl sulfone	C8H9ClO2S	-	NR	NR	6-15-50	1-2-3
0.66	FP10	0.55	1.71	parathion-methyl oxygen analog	C8H10NO6P	-	NR	NR	6-15-50	1-2-3
0.66	NI0.6/NP10	0.67	0.79	pentachloroaniline	C6H2Cl5N	C	C	C	6	1
0.66	NI4500/NP64	0.5	2.33	ethylenethiourea	C3H6N2S	S	NR	NR	6-15-50	1-2-3
0.67	NP6	0.73	-	CP 108064, methylated	C15H21NO4	-	-	-	-	-
0.67	NI0.6	0.82	0.64	chlordene, alpha-	C10H6Cl6	-	-	-	-	-
0.67	NI5	0.75	0.88	acetochlor	C14H20NO2Cl	C	C #	P	50	3
0.67	NI0.5	0.5	1.71	BHC, delta-	C6H6Cl6	C	C	C	6+15	1
0.68	NP2	0.58	-	cyromazine	C6H10N6	S	-	-	-	-
0.68	NI0.6/NP2	0.75	0.8	CGA 14128	C12H21N2O4PS	C		-	50	1-2-3
0.68	NP(WB)65	1.1	-	nitrothal-isopropyl	C14H17O6N	C	-	-	-	-
0.69	HX10	0.67	1.44	metobromuron	C9H11BrN2O2	C	NR	NR	6-15-50	1-2-3
0.7	NI1/NP7	0.64	1.06	benoxacor	C11H11Cl2NO2	C	P	C	15+50	2+3
0.7	NI12/NP15	0.6	1.64	ethoxyquin	C14H19N0	C	NR	NR	6-15-50	-
0.71	NI8/NP10	0.45	2.25	methidathion sulfoxide	C5H8N2O4S2	-	NR	NR	6-15-50	1-2-3
0.71	NI10	0.71	1.11	dimethachlor	C13H18ClNO2	C	-	-	-	-
0.71	FS(WB)80/HN(WB)0.4/ HX500/NI300	0.4	-	dazomet	C5H10N2S2	S	NR	-	6-15-50	1-2-3
0.72	NP1000	-	-	1-methyl cyromazine	C7H13N6	-	-	-	-	-
0.72	NI6	0.8	1	alachlor	C14H2OClNO2	C	C	C #	50	3
0.72	HN(WB)0.6/HX(WB)5/ NI(WB)2/NP(WB)8	0.54	2.1	terbacil	C9H13ClN2O2	C	NR	NR	6-15	2+3

Appendix I: PESTDATA Chemicals in Order by OV-17 Relative Retention Time (continued)

RRT/c OV-17	Detector Responses with OV-17 Column	RRT/c OV-101	RRT/c OV-225	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
0.73	FP(WB)2	0.61	-	pirimicarb	C11H18N4O2	C	-	-	-	-
0.74		0.64	1.22	cyprazine	C9H14ClN5	C	-	-	-	-
0.74	FP20	0.77	-	prometryn	C10H19N5S	C	P #	P #	50	1-2-3
0.74	HX1/NI2	0.55	1.44	chlorothalonil	C8Cl4N2	S	C #	C #	6-15-50	2+3
0.75	HX0.8/HX(WB)0.4	0.81	0.85	tridiphane	C10H7Cl5O	C	C	-	6	1+2
0.76	FP2	0.67	-	phosphamidon*	C10H19ClNO5P	C	NR	NR	6-15-50	1-2-3
0.76	NI1	1.05	0.58	aldrin	C12H8Cl6	C	C	C	6	1
0.76	FP1/NI1	0.81	0.86	ronnel	C8H8Cl3O3PS	C	C	C	6	2
0.78	NP133	0.6	1.4	ethiofencarb	C10H15NO2S	C	NR	NR	6-15-50	-
0.78	NI4	0.66	2.82	propanil	C9H9Cl2NO	C	NR	NR	6-15	3
0.79	HN(WB)1/HX(WB)2/ NI(WB)1/NP(WB)7	0.69	2.01	vinclozolin metabolite S	C10H7Cl2NO3	V	P	V #	15	2
0.79	FP(WB)1.5	0.72	0.86	chlorpyrifos-methyl	C7H7Cl3NO3PS	C	C	-	6	2
0.79		0.75	1.55	prothoate	C9H20NO3PS2	C	-	-	-	-
0.8	HN(WB)0.2/NP(WB)13	0.7	-	IN-B2838	C10H15N3O3	P	NR	NR	6-15-50	1-2-3
0.81	FS1.5	0.41	2.71	dimethipin	C6H10O4S2	C	NR	NR	6-15-50	1-2-3
0.82	NI200/NP35	0.56	2.29	methidathion sulfone	C5H8N2O3S2	-	NR	NR	6-15-50	1-2-3
0.82		-	-	butylisodecyl phthalate	C22H34O4	-	-	-	-	-
0.82	NP7	0.44	-	isocarbamid	C8H15N3O2	C	-	-	-	-
0.83	FP10	0.72	-	fenitrothion oxygen analog	C9H12NO6P	C	-	-	-	-
0.84		0.88	0.92	dibutyl phthalate	C16H22O4	-	C	C	15+50	-
0.86		-	-	2,4,5-T butyl esters*	C12H13Cl3O3	-	-	-	-	-
0.86	HX3	0.96	1.45	nitrofluorfen	C13H7ClF3NO3	C	C	C	15	2
0.86	FP4/NI15	0.8	-	parathion oxygen analog	C10H14NO6P	C	NR	NR	6-15-50	1-2-3
0.87	NI3	0.94	0.69	pentachlorophenyl methyl sulfide	C7H3Cl5S	C	C	C	6	1
0.87	FP5	0.68	1.55	malathion oxygen analog	C10H19O7PS	C	NR	NR	6-15-50	1-2-3
0.87	FP2/FP(WB)1/NI3	0.71	1.64	parathion-methyl	C8H10NO5PS	C	C	C	15	2
0.88	NI1	0.98	0.89	chlordene, gamma-	C10H6Cl6	-	-	-	-	-
0.88	HN(WB)48/HX(WB)390/ NI(WB)27/NP(WB)1000	1.05	1.47	acifluorfen	C14H7ClF3NO3	-	NR	NR	6-15-50	1-2-3
0.89	NI1	0.98	0.84	chlordene, beta-	C10H6Cl6	-	-	-	-	-
0.89	NP(WB)2	0.73	-	cymiazole	C12H14N2S	-	-	-	-	-

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RRT/c OV-17	Detector Responses with OV-17 Column	RRT/c OV-101	RRT/c OV-225	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
0.89	NP(WB)10	-	-	isoproturon	C12H18N2O	S	-	-	-	-
0.9	HN(WB)3/HX(WB)23/ NI(WB)23/NP(WB)44	0.42	-	pyrazon metabolite B	C6H4ClN3O	-	NR	NR	6-15-50	1-2-3
0.9	NI100/NP300	0.6	2.65	CGA 120844	C8H9NSO3	-	NR	NR	6-15-50	1-2-3
0.9	NI1000/NP50	0.81	-	metalaxyl	C15H21NO4	C	NR	NR	6-15-50	1-2-3
0.9	HN(WB)15/NI(WB)80/ NP(WB)30	0.55	1.41	3-ketocarbofuran	C12H12NO4	S	NR	NR	6	1
0.91	HX2	4.1	1.08	dicofol, o,p'-*	C14H9Cl5O	C	V	S	6+15	2
0.91	NI0.4/NP11	0.57	1.47	metribuzin	C8H14N4OS	V	NR	NR	50	1-2-3
0.91	FP(WB)10	-	-	formothion	C6H12NO4PS2	C	NR	NR	6-15-50	1-2-3
0.92	FP2/FP(WB)1.2	0.87	-	pirimiphos-methyl	C11H20N3O3PS	C	C	C	15	3
0.92	NI2	0.82	1.07	dichlorobenzophenone, o,p'-	C13H8Cl2O	-	C	C	15	2
0.93	HN(WB)3/HX(WB)3/ NI(WB)4/NP(WB)7	0.89	3.02	vinclozolin metabolite E	C11H11Cl2NO2	C	S	NR	15+50	-
0.93	FS54/NI4.5	0.68	2.89	2,3-dihydro-3,3-methyl-2-oxo-5-benzofuranyl methyl sulfonate	C11H12O5S	-	-	-	-	-
0.93	HX7	1.03	1.21	metolachlor	C15H22ClNO2	C	S #	NR	50	1-2-3
0.93	NI6/NP15	1.15	1.22	butralin	C14H21N3O4	V	C	-	6+15+50	-
0.95	HN(WB)30	0.55	3.6	ethametsulfuron methyl ester*	C15H18N6O6S	-	NR	NR	6-15-50	1-2-3
0.95	NP5.5	0.71	-	fuberidazole	C11H8N2O	C	-	-	-	-
0.95		0.85	2.13	linuron	C9H10Cl2N2O2	V	V #	V	50	3
0.96	FP3	0.71	2.95	demeton-O sulfone*	C8H19O5PS2	C	-	-	-	-
0.96	NI1/NP5	0.99	1.91	KWG 1323	C14H16ClN3O3	C	NR	NR	6-15-50	1-2-3
0.96	NP16	0.97	-	difenoxuron	C16H18N2O3	-	-	-	-	-
0.97		1.13	-	trichloronat	C10H12Cl3O2PS	C	C	-	6	-
0.98	HX3	0.94	1	thiobencarb	C12H16ClNOS	C		V	15	2+3
0.99	NI1/NP9	0.77	1.62	Tycor	C9H16N4OS	C	S	S	50	3
1	HX5/HX5/HX(WB)3/ NI(WB)2/NP2/NP(WB)3	1.05	1.64	triadimefon	C14H16ClN3O2	C	S #	S #	50	1-2-3
1	FP2/NI2	1	1	chlorpyrifos	C9H11Cl3NO3PS	C	C	P	6	2
1	NI1	1.06	1.13	DCPA	C10H6Cl4O4	C	C	C	15	2
1.01	NI3/NP15	1.14	1.24	isopropalin	C15H23N3O4	C	C	-	6	-
1.01		0.9	1.71	dichlofluanid	C9H11Cl2FN2O2S2	C	C #	-	15+50	2+3

Appendix I: PESTDATA Chemicals in Order by OV-17 Relative Retention Time (continued)

RRT/c OV-17	Detector Responses with OV-17 Column	RRT/c OV-101	RRT/c OV-225	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
1.02	FS65/NI333	0.86	1.93	ethofumesate	C13H18O5S	C	-	-	-	-
1.02	FP6	0.66	-	phorate oxygen analog sulfone	C7H17O5PS2	C	NR	NR	6-15-50	1-2-3
1.03	NP600	0.8	-	3-hydroxymethyl-2,5-dimethylphenyl methylcarbamate	C11H15NO3	-	NR	NR	6-15-50	1-2-3
1.04	NI4	0.55	0.86	tetraiodoethylene	C2I4	-	P	P	6	-
1.05		-	-	2,4,5-T butyl esters*	C12H13Cl3O3	-	-	-	-	-
1.05		0.87	-	demeton-O sulfoxide	C8H15O4PS2	C	-	-	-	-
1.05	NI0.6	1.33	0.94	octachlor epoxide	C10H4Cl8O	C	C	C	6	1
1.05	NP(WB)8	-	-	norea	C13H15N2O	C	-	-	-	-
1.05	FP3/FP(WB)1.1	0.84	1.82	fenitrothion	C9H12NO5PS	C	C	C	15	2
1.05	FP3/NI7	0.91	1.49	malathion	C10H19O6PS2	C	C	C	15+50	3
1.05		0.75	-	carbaryl	C12H11NO2	C	-	-	-	-
1.06	FP40	0.78	-	phorate oxygen analog sulfoxide	C7H17O4PS2	C	NR	NR	6-15-50	1-2-3
1.06	NI10/NP125	0.83	3.77	metribuzin, deaminated metabolite	C8H13N3OS	C	NR	NR	6-15-50	1-2-3
1.07		0.87	-	4-chlorophenoxyaniline*	C12H10ClNO	S	-	-	-	-
1.07	NI1	0.99	1.63	1-hydroxychlordene	C10H6Cl6O	-	R	-	15	-
1.07	FP2/NI4	0.98	1.91	parathion	C10H14NO5PS	C	C	C	15	2
1.07	NI(WB)40	0.54	-	4-chlorophenylurea	C7H7ClN2O	NR	NR	NR	6-15-50	1-2-3
1.08	HX3	4.4	1.28	dicofol, p,p'-*	C14H9Cl5O	C	V	P #	6+15	1+2
1.08		0.95	1.51	chlorpyrifos oxygen analog	C9H11Cl3NO4P	C	NR	-	6-15-50	-
1.08	NI2	0.99	1.25	dichlorobenzophenone, p,p'-	C13H8Cl2O	-	C	C	15	2
1.09	NI1/NP6	1.25	8	MB45950	C12H4SN4F6Cl2	S	P	V	15+50	2+3
1.12	NI2	1.37	1.22	S-bioallethrin	C19H26O3	-	C	-	50	-
1.12	FP9	0.78	-	fenthion oxygen analog	C10H15O4PS	C	NR	NR	6-15-50	1-2-3
1.13	NI(WB)600	1.04	-	PPG-947	C17H11ClF3NO7	-	NR	NR	6-15-50	1-2-3
1.14	FP(WB)2.4	1.01	-	pirimiphos-ethyl oxygen analog	C13H24N3O4P	C	-	-	-	-
1.14	FP3/NI4	1.14	1.03	pirimiphos-ethyl	C13H24N3O3PS	C	C	C	15+50	3
1.15	HX0.9/NI2	1.29	1.22	heptachlor epoxide	C10H5Cl7O	C	C	C	6	2
1.16	NI1/NP5	1.35	8.7	fipronil	C12H4Cl2F6N4OS	S	S	V	50	3
1.16	FP3/NI2	1.11	1.29	bromophos	C8H8BrCl2O3PS	C	C	C	6	-
1.17		0.8	-	methiocarb sulfone	C11H15NO4S	S	NR	NR	6-15-50	1-2-3
1.18	NP1000	3	-	NTN33823	C9H11N4Cl	-	NR	NR	6-15-50	1-2-3

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RRT/c OV-17	Detector Responses with OV-17 Column	RRT/c OV-101	RRT/c OV-225	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
1.18	FP3	0.96	1.46	fenthion	C10H15O3PS2	C	S #	NR	6+15	1-2-3
1.19	HX13/HX(WB)2/NI(WB)3/NP(WB)3	1.44	2.23	triflumizole	C15H15ClF3N3O	C	-	-	-	-
1.2	NI2	1.19	1.15	TDE, o,p'-, olefin	C14H9Cl3	-	-	-	-	-
1.21	NI1.5/NP6	1.22	1.48	pendimethalin	C13H19N3O4	C	C	P	15	2
1.24	FP7	1.17	1.74	isofenphos oxygen analog	C15H24NO5P	C	-	-	-	-
1.26	FP6	0.89	2.55	phorate sulfoxide	C7H17O3PS3	C	NR	NR	6-15-50	1-2-3
1.28	FP2/NI60	0.92	2.9	terbufos oxygen analog sulfone	C9H21O5PS2	C	NR	NR	6-15-50	1-2-3
1.29	FP(WB)2/NI3	1.21	1.58	chlorfenvinphos, alpha-	C12H14Cl3O4P	C	-	NR	6-15-50	-
1.3	FP2	0.97	3.26	phorate sulfone	C7H17O4PS3	C	S #	S #	6-15-50	3
1.3	FP2/NI3	1.08	2.33	crufomate	C12H19ClNO3P	C	NR	NR	6-15-50	-
1.31		1.28	-	4-chlorophenoxyaniline*	C12H10ClNO	S	-	-	-	-
1.32	HX3/HX(WB)2/NI(WB)2/NP(WB)3	1.24	-	penconazole	C13H15Cl2N3	C	-	-	-	-
1.32		0.96	-	carbetamide	C12H16N2O3	-	-	-	-	-
1.32	HN(WB)1/NP(WB)50	1.4	-	dinobuton	C14H18N2O7	C	-	-	-	-
1.33	NI40/NP200	1.11	4.7	isoxaflutole (prop)	C15H12SNO4F3	NR	V #	S #	50	3
1.34	NI0.6	1.49	1.46	chlordane, trans-	C10H6Cl8	C	C	C	6	1
1.36	NP250	0.21	-	3-(3,4-dichlorophenyl)-1-methoxyurea	C8H8Cl2N2O2	R	NR	NR	6-15-50	
1.36	HN(WB)1/HX(WB)8/NI(WB)6/NP(WB)5	0.8	4.8	bromacil	C9H13BrN2O2	C	NR	NR	6-15-50	1-2-3
1.38	NI40/NP250	1.13	4.7	RPA202248	C15H12SNO4F3	NR	NR	NR	6-15-50	1-2-3
1.38	FP2	1.36	1.73	isofenphos	C15H24NO4PS	C	C	-	15+50	-
1.39	NP(WB)10	1.18	-	cyprodinil	C14H15N3	C	NR	NR	6-15-50	1-2-3
1.4	HX(WB)6/NI4.5	1.55	-	haloxyfop methyl ester	C16H13ClF3NO4	-	-	-	-	-
1.41		1.25	-	tolylfluanid	C10H13ClFNOS	C	-	-	-	-
1.42	HX0.4/NI0.8	1.75	1.45	nonachlor, trans-	C10H5Cl9	C	C	C	6	1
1.42		1.27	3.39	chlorbromuron	C9H10BrClN2O2	V	V	V	50	3
1.43	FP25	1.34	0.65	merphos*	C12H27PS3	-	C	C	6+15+50	3
1.43	FP(WB)12	0.93	-	des N-isopropyl isofenphos oxygen analog	C12H18NO5P	-	-	-	-	-
1.43	NP6	1.05	-	pyracarbolid	C13H15NO2	-	-	-	-	-
1.44	NI5	-	1.67	2,4-D butoxyethyl ester*	C14H18Cl2O4	-	-	-	-	-

Appendix I: PESTDATA Chemicals in Order by OV-17 Relative Retention Time (continued)

RRT/c OV-17	Detector Responses with OV-17 Column	RRT/c OV-101	RRT/c OV-225	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
1.44	HX7/HX(WB)4/NI(WB)50/NP(WB)6	1.36	-	triadimenol	C14H18ClN3O2	C	NR	NR	6-15-50	-
1.45	FP(WB)0.3	1.51	1.42	bromophos-ethyl	C10H12BrCl2O3PS	C	C	P	6	-
1.45	NI4	1.45	1.36	TDE, p,p'-, olefin	C14H9Cl3	C	C	C	6	1
1.46	FS88/NI96	1	6.6	2-hydroxy-2,3-dihydro-3,3-methyl-5-benzofuranyl methyl sulfonate	C11H14O5S	-	-	-	-	-
1.46	HX5/NP4	1.5	-	metazachlor	C14H16ClN3O	C	-	-	-	-
1.46	HX9	1.73	1.83	butachlor	C17H26ClNO2	C	C	-	50	-
1.47	HX1/NI2	1.64	1.38	endosulfan I	C9H6Cl6O3S	C	C	C	15	2
1.47	NP20	1.24	1.88	anilazine	C9H5Cl3N4	V	S	P	15+50	2+3
1.48	NI5	-	-	2,4-D isooctyl ester*	C16H22Cl2O3	-	-	-	-	-
1.48	HX6	0.89	4.9	cyanazine	C9H13ClN6	C	NR	-	6-15-50	-
1.48	FP(WB)3	0.72	-	oxydemeton-methyl sulfone	C6H15O5PS2	C	-	-	-	-
1.48	NI0.8	1.66	1.54	chlordane, cis-	C10H6Cl8	C	C	C	6	1
1.49	HX1	1.37	3.04	procymidone	C13H11Cl2NO2	C	C	P	15	-
1.5	FP3	1.21	2.73	des N-isopropyl isofenphos	C12H18NO4PS	C	S	-	50	-
1.51	NI1	1.55	1.28	DDE, o,p'-	C14H8Cl4	C	C	C	6	1
1.52	FP4/FP(WB)2/NI3	1.29	2	chlorfenvinphos, beta-	C12H14Cl3O4P	C	S #	-	50	1-2-3
1.54	NI1000	1.39	1.89	CGA 189138	C13H8O3Cl2	-	-	-	-	-
1.54		1.15	4.3	CGA 91305	C10H8Cl2N3O	V	NR	NR	6-15-50	1-2-3
1.54	HX3/NI1	1.39	1.62	chlorbenside	C13H10Cl2S	C	S	P	6	1
1.55	HN(WB)3/HX(WB)17/NI(WB)19/NP(WB)12	0.86	-	6-chloro-2,3-dihydro-7-hydroxy=methyl-3,3-methyl-5H-oxazolo=(3,2-a)pyrimidin-5-one	C9H13ClN2O3	-	NR	NR	6-15-50	1-2-3
1.55	NP(WB)25	1.1	-	diphenamid	C16H17NO	V	NR	-	6-15	-
1.56	NP4	1.16	-	phenothiazine	C12H9NS	-	-	-	-	-
1.58	FP2/NI10	1.2	-	terbufos sulfone	C9H21O4PS3	C	C #	C #	6-15-50	2+3
1.58	FP3/FP(WB)1.9	1.28	2.67	mecarbam	C10H20NO5PS2	C		-	50	-
1.59	NI1000	6.7	-	CGA 205375	C16H13N3O2Cl2	-	-	-	-	-
1.59	HX7/HX(WB)3/NI(WB)85/NP(WB)5	1.52	-	paclobutrazol	C15H20ClN3O	C	-	-	-	-
1.6	NI1000	1.28	-	3-phenoxybenzenemethanol	C13H12O2	-	-	-	-	-
1.64	NI6/NP30	1.57	1.84	cyclanilide methyl ester	C12H11Cl2NO3	-	-	-	-	-

Appendix I: PESTDATA Chemicals in Order by OV-17 Relative Retention Time (continued)

RRT/c OV-17	Detector Responses with OV-17 Column	RRT/c OV-101	RRT/c OV-225	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
1.64	FP3/FP(WB)3	1.32	2	quinalphos	C12H15N2O3PS	C	C	-	15	-
1.65		1.65	-	DDMS	C14H11Cl3	-	R	-	6	-
1.66	FP10/NI120/NP7	1.08	3	fosthiazate	C9H18NO3PS2	C	NR	NR	6-15-50	1-2-3
1.68		2.1	1.78	2,4-D ethyl hexyl ester*	C16H22Cl2O3	-	-	-	-	-
1.75	FP5	1.15	5.8	demeton-S sulfone	C8H19O5PS2	C	-	-	-	-
1.76		2.12	1.76	pyrethrins*	C21H27O4	-	C	C	50	-
1.78	NI5	2.04	1.78	2,4-D isooctyl ester*	C16H22Cl2O3	-	-	-	-	-
1.79	NI5	1.82	2.08	2,4-D butoxyethyl ester*	C14H18Cl2O4	-	-	-	-	-
1.79	NP(WB)23	1.86	2.91	hexaconazole	C14H17Cl2N3O	C	-	-	-	-
1.8	FP125/NI200/NP40	1.07	-	methidathion oxygen analog	C6H11N2O5PS2	-	NR	NR	6-15-50	1-2-3
1.82	NI3	1.85	1.74	prothiofos	C11H15Cl2PO2S2	C	C	C	6	2
1.83	FP2/FP(WB)2.6/NI5	1.31	2.05	phenthoate	C12H17O4PS2	C	C	-	15+50	-
1.84	HX1.5/NI1	1.91	1.87	dieldrin	C12H8Cl6O	C	C	C	15	2
1.85		1.57	-	oxythioquinox	C10H6N2OS2	C	-	-	-	-
1.85	NI2	1.2	3.49	captan	C9H8Cl3NO2S	C	P	C	50	3
1.86	HN(WB)120/HX(WB)66/ NI(WB)78	1.9	3.16	PPG-2597	C20H17ClF3NO6	-	NR	NR	6-15-50	1-2-3
1.86	NI1	1.92	1.59	DDE, p,p'-	C14H8Cl4	C	C	C	6	1
1.88	FP25	1.95	1.64	merphos*	C12H27PS3	-	C	C	6+15+50	3
1.88		1.73	-	chlorflurecol methyl ester	C15H11ClO3	C	-	-	-	-
1.88	FP3/NI3	1.95	1.65	tribufos	C12H27OPS3	C	C	P	15+50	3
1.9	FP10/FP(WB)3	1.37	2.85	crotoxyphos	C14H19O6P	C	NR	NR	6-15-50	1-2-3
1.92		1.26	3.5	Sulphenone	C12H9ClO2S	C		-	50	3
1.94	NI9	1.23	3.01	folpet	C9H4Cl3O2NS	C	C	P	15+50	2+3
1.96	NI2	1.97	2.48	oxadiazon	C15H18Cl2N2O3	C	C	P	15	-
1.97	FP10/FP(WB)3	1.58	2.72	Gardona	C10H9Cl4O4P	C	NR	NR	6-15-50	1-2-3
1.98	NI2/NP10	2.06	31	MB46136	C12H4SO2N4F6Cl2	S	S	V	50	2+3
1.99	HX10	1.88	-	pretilachlor	C17H26ClNO2	C	-	-	-	-
2	NI11/NP200	1.78	3.14	diethatyl-ethyl	C16H22ClNO3	C	NR	NR	6-15-50	1-2-3
2.03	HN(WB)1.3/HX7/HX4/ HX(WB)2/NI(WB)4/NP(WB)8	2.02	3.4	diclobutrazol	C15H19Cl2N3O	C	NR	NR	6-15-50	1-2-3
2.04		1.48	-	thiabendazole	C10H7N3S	C	NR	-	6-15-50	-

Appendix I: PESTDATA Chemicals in Order by OV-17 Relative Retention Time (continued)

RRT/c OV-17	Detector Responses with OV-17 Column	RRT/c OV-101	RRT/c OV-225	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
2.08	HX19/HX(WB)2/NI(WB)5/NP(WB)10	1.76	4	imazalil	C14H14Cl2N2O	C	NR	NR	6-15-50	-
2.1		1.58	2.46	triazamate	C13H22N4O3S	C	NR	NR	6-15-50	1-2-3
2.11	FP7	-	-	jodfenphos	C8H8Cl2IO3PS	C	-	-	-	-
2.11	NP200	-	-	sulfanilamide	C6H8O2N2S	NR	NR	NR	6-15-50	1-2-3
2.12		1.7	-	napropamide	C17H20NO2	C	-	-	-	-
2.13	FP5/FP(WB)2.9	1.8	2.34	profenofos	C11H15BrClO3PS	C	P	P	50	3
2.16	HX9/NI2/NP350	2	4	oxyfluorfen	C15H11ClF3NO4	C	C	C	15	2
2.19	NI2	1.9	2.46	TDE, o,p'-	C14H10Cl4	-	C	C	6	1
2.2	HX5	1.61	3.04	ovex	C12H8Cl2O3S	C	C	C	15	2
2.28	FP(WB)2.4/NI10	1.4	3.33	methidathion	C6H11N2O4PS3	C	S	P #	50	3
2.29	NI2	2.13	2.22	endrin	C12H8Cl6O	C	C #	C #	15	2
2.31	HX40	2.3	2.36	fluazifop butyl ester	C19H20F3NO4	C	C	V	15	3
2.33	HX24/HX(WB)5/NP(WB)6	1.97	-	flusilazole	C16H15F2N3Si	C	-	-	-	-
2.34	NI2/NP50	2.21	-	chlorfenapyr (prop)	C15H11BrClF3N2O	P	-	S	50	2
2.38	NI22/NP(WB)100	2.19	4.2	binapacryl	C15H18N2O6	C	P	P	15	-
2.38	HX2/NI5	2.75	1.67	chlordecone	C10H8Cl10O5	-	S #	P #	15+50	1-2-3
2.39	FP7	1.5	6.7	disulfoton sulfone	C8H19O4PS3	C	NR	-	6-15-50	-
2.4	HN(WB)5	2.14	5	PPG-947, methylated*	C18H13ClF3NO7	-	-	-	-	-
2.4	HN(WB)4/HX(WB)6.5/NI(WB)3/NP(WB)210	2.15	5	PPG-847, methylated	C15H9ClF3NO3	-	-	-	-	-
2.41	NI5/NP38	1.53	6.5	CGA 94689A	C15H21NO5	V	NR	NR	6-15-50	1-2-3
2.41	FS(WB)90/NI(WB)80/NP(WB)80	1.28	-	hexythiazox	C17H21ClN2O2S	-	S #	NR	50	2+3
2.41	FP10/NP3	1.66	3.7	fenamiphos	C13H22NO3PS	C	NR	NR	6-15-50	1-2-3
2.41	NI15	2.33	2.9	chloropropylate	C17H16Cl2O3	P	C	C	15+50	3
2.42	NI25	2.23	2.01	Perthane	C18H2OCl2	C	C	C	6	1
2.44	HX60	-	-	imazamethabenz methyl ester*	C16H20N2O3	C	-	-	-	-
2.45	NI10/NP75	1.54	6.6	CGA 94689B	C15H21NO5	S	NR	NR	6-15-50	1-2-3
2.45		1.94	-	flamprop-methyl	C17H15ClFNO3	C	-	-	-	-
2.51	FP3	-	-	mephosfolan	C8H16NO3PS2	C	-	-	-	-
2.55	HN(WB)13/HX(WB)99/NI(WB)35/NP(WB)290	1.35	2.27	3-tert-butyl-5-chloro-6-hydroxy= methyluracil	C9H13ClN2O3	-	NR	NR	6-15-50	1-2-3

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Appendix I: PESTDATA Chemicals in Order by OV-17 Relative Retention Time (continued)

RRT/c OV-17	Detector Responses with OV-17 Column	RRT/c OV-101	RRT/c OV-225		Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
2.55	HN(WB)1.2/NP(WB)53	1.41	-		IN-T3936	C10H15N3O4	S	NR	NR	6-15-50	1-2-3
2.58	FP5	2.24	-		chlorthiophos*	C11H15Cl2O3PS2	C	C	C	6	2
2.6	HX10/HX(WB)4/NI(WB)21/ NP75/NP(WB)6	1.9	7.2		myclobutanil	C15H17ClN4	C	NR	NR	6-15-50	1-2-3
2.6	NP(WB)300	2	3.7		bupirimate	C13H24N4SO3	C	-	-	-	-
2.61	NI2	2.52	3.33		nonachlor, cis-	C10H5Cl9	C	C	C	6	1
2.61	NI15	2.31	3.26	-	chlorobenzilate	C16H14Cl2O3	C	C #	P #	15+50	3
2.62		3.38	-		2,4,5-T ethylhexyl ester	C16H21Cl3O3	-	-	-	-	-
2.67	NP36	1.5	4.3		TCMTB	C9H6N2S3	C	P	P	15	-
2.69		2.04	1.61		cyproconazole	C15H18ClN3O	C	NR	NR	6-15-50	1-2-3
2.69	FP5	-	-		phosfolan	C7H14NO3PS2	C	-	-	-	-
2.7		2.95	2.84		pyrethrins*	C21H27O4	-	C	C	50	-
2.7	NI2	2.55	2.27		DDT, o,p'-	C14H9Cl5	C	C	C	6	1
2.71	NI3	2.03	3.8		nitrofen	C12H7Cl2NO3	C	C	C	15	2
2.76	HX60	1.79	3.5		imazamethabenz methyl ester*	C16H20N2O3	C	-	-	-	-
2.77	FP5	2.36	-		chlorthiophos*	C11H15Cl2O3PS2	C	C	C	6	2
2.77	HX3/NI2	2.21	3.9		endosulfan II	C9H6Cl6O3S	C	C	C	15+50	2
2.8	NI5	2.64	2.33		tetrasul	C12H6Cl4S	C	C	C	6	1
2.81		2.46	-		flamprop-M-isopropyl	C19H19ClFNO3	C	-	-	-	-
2.87	FP15	4.2	-		carbophenothion oxygen analog sulfoxide	C11H16ClO4PS2	-	-	-	-	-
2.87	NI2	2.41	3.8		TDE, p,p'-	C14H10Cl4	C	C	C	6	1
2.88		3.03	2.74		ethephon	C2H6ClO3P	NR		-	6+15+50	1+2+3
2.92	NP(WB)8	2.07	-		methoprotryne	C11H21N5OS	C	-	-	-	-
2.96	NI1000/NP150	1.8	-		CGA 100255	C15H12NO5	S	-	-	-	-
2.99	FP10/HX11	2.22	4.1		chlorthiophos oxygen analog	C11H15Cl2O4PS	C	NR	NR	6-15-50	1-2-3
3	NI120/NP70	2.4	4.3		imazethapyr ammonium salt methyl ester	C16H21N3O3	-	-	-	-	-
3.06	FP15	2.17	4.2		carbophenothion oxygen analog	C11H16ClO3PS2	C	NR	NR	6-15-50	1-2-3
3.14	NI5	2.38	3.24		leptophos photoproduct	C13H11Cl2O2PS	C	-	-	-	-
3.16	FP5	2.56	-		chlorthiophos*	C11H15Cl2O3PS2	C	C	C	6	2
3.17	HX7	2.43	-		etaconazole*	C14H15Cl2N3O2	C	-	-	-	-

Appendix I: PESTDATA Chemicals in Order by OV-17 Relative Retention Time (continued)

RRT/c OV-17	Detector Responses with OV-17 Column	RRT/c OV-101	RRT/c OV-225	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
3.17		1.6	4.1	isoprothiolane	C12H18O4S2	C	-	-	-	-
3.36	FP4/FP(WB)2.3/NI8	2.56	3.93	ethion	C9H22O4P2S4	C	C	C	6	2
3.38	NI(WB)15/NP(WB)40	2	3.02	kresoxim-methyl	C18H19NO4	P	C	C	15+50	3
3.5	NI150	-	-	dinocap*	C18H24N2O6	C	P	P	15	2
3.5	FP3	2.79	-	sulprofos	C12H19O2PS3	C	-	-	-	-
3.5	NI2	3.13	3.6	DDT, p,p'-	C14H9Cl5	C	C	C	6	1
3.6	FP(WB)29	2.78	-	sulprofos sulfoxide*	C12H19O3PS3	C	-	-	-	-
3.6	FP100	2.8	-	fensulfothion sulfone	C11H17O5PS2	C	NR	-	6-15-50	-
3.7		2.9	-	1,1'-(2,2-dichloroethylidene)bis(2-methoxybenzene)	C16H16Cl2O2	-	R	-	-	-
3.7	FP8	2.94	4.2	carbophenothion	C11H16ClO2PS3	C	C	P	6	2
3.7	NI(WB)5	3.1	-	fenhexamid	C14H17Cl2NO2	NR	NR	NR	6-15-50	1-2-3
3.8	FP6	1.99	-	fensulfothion oxygen analog sulfone	C11H17O7PS2	-	-	-	-	-
3.8	FP6/FP(WB)7	2.4	-	fensulfothion	C11H17O4PS2	C	NR	NR	6-15-50	1-2-3
3.9	NI150	4	-	dinocap*	C18H24N2O6	C	P	P	15	2
3.9	NP500/NP(WB)8	1.59	-	tricyclazole	C9H7N3S	C	-	-	-	-
3.9	NI6	2.81	7.5	Prolan	C15H13Cl2NO2	P	S	S	15	2
3.93		2.31	15	desisopropyl iprodione	C10H6Cl2N3O3	P		-	50	1-2-3
4	HX9	3.21	5.6	propiconazole*	C15H17Cl2N3O2	C	NR	NR	6-15-50	1-2-3
4	FP20	5.4	-	carbophenothion sulfoxide	C11H16ClO3PS3	-	-	-	-	-
4	HX6/NI6	2.83	8.3	endosulfan sulfate	C9H6Cl6O4S	C	C	C	50	2
4.1	FP(WB)8	2.29	-	fenthion oxygen analog sulfone*	C10H15O6PS2	-	-	-	-	-
4.1	NI20/NP100	2.96	11.5	CL 202,347	C13H19N3O5	-	-	-	-	-
4.2	HN(WB)2/HX(WB)13/ NI(WB)18/NP(WB)85	2.51	4	pyrithiobac-sodium methyl ester	C14H13ClN2O4	-	-	-	-	-
4.2	HX(WB)3	3.38	-	tebuconazole	C16H22ClN3O	C	-	-	-	-
4.2	NI200/NP50	4	15	KWG 1342	C14H18ClN3O3	-	-	-	-	-
4.2	NI10	2.97	3.7	methoxychlor olefin	C16H14Cl2O2	C	C	C	6	2
4.3	NI2600	3.8	4.8	propargite	C19H26O4S	C	C	-	15	2
4.4	NI150	4.3	6.9	dinocap*	C18H24N2O6	C	P	P	15	2
4.4	FP(WB)12	2.26	-	famphur oxygen analog	C10H16NO6PS	C	-	-	-	-
4.4	NI5	3.06	7.5	Bulan	C16H15Cl2NO2	C	P	P	15	2

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Appendix I: PESTDATA Chemicals in Order by OV-17 Relative Retention Time (continued)

RRT/c OV-17	Detector Responses with OV-17 Column	RRT/c OV-101	RRT/c OV-225	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
4.5	NI100	6.1	1.48	tralkoxydim*	C20H27NO3	V	NR	NR	50	1-2-3
4.5	HX5/HX(WB)20	4.9	3.8	bifenthrin	C23H22ClF3O2	V	C	-	6+15	2
4.5		2.43	-	benodanil	C13H10INO	C	-	-	-	-
4.5		3.06	5.1	butyl benzyl phthalate	C19H20O4	-	C	P	15+50	-
4.6	HN(WB)100/HX(WB)14/ NI(WB)85/NP(WB)210	2.87	-	vinclozolin metabolite F	C11H13Cl2NO4	R	NR	NR	6-15-50	1-2-3
4.6		3.1	8.2	cyanofenphos	C15H14NO2PS	C	-	-	-	-
4.67	NI(WB)20	3.26	5.8	clodinafop-propargyl	C17H13ClFNO4	V	V	-	50	3
4.7	HX10	3.57	4.9	diclofop-methyl	C16H14Cl2O4	C	C	C	15	2
4.7	FP20	2.39	-	fenthion sulfone	C10H15O5PS2	C	NR	NR	6-15-50	1-2-3
4.8	NI150	4.8	7.7	dinocap*	C18H24N2O6	C	P	P	15	2
4.8	HX4	3.36	7.3	nuarimol	C17H12ClFN2O	C	NR	C #	50	1-2-3
4.9	HX(WB)3	3.38	1.41	desmethyl norflurazon	C11H7ClF3N3O	V	NR	NR	6-15-50	1-2-3
5	NP14	2.5	14	oxadixyl	C14H18N2O4	C	NR	NR	6-15-50	1-2-3
5	NI9	3.3	4.5	methoxychlor, o, p'-	C16H15Cl3O2	-	C	-	6	-
5	FP5	2.49	-	fensulfothion oxygen analog	C11H17O5PS	C	NR	-	6-15-50	-
5	FP50/FP(WB)7	2.65	14	famphur	C10H16NO5PS2	C	NR	-	6-15-50	-
5.01	NP(WB)10	4.5	-	norflurazon	C12H9ClF3N3O	V	NR	NR	6-15-50	-
5.2	HX20/NI35/NP200	-	-	iprodione*	C13H13Cl2N3O3	C	S #	NR	50	1-2-3
5.2	FP5/FP(WB)5	2.62	-	triazophos	C12H16N3O3PS	C	-	-	-	-
5.3		5.4	-	carbosulfan	C20H32N2O3S	P	-	-	-	-
5.3	FP(WB)8	2.87	6.3	edifenphos	C14H15O2PS2	C	-	-	-	-
5.4	HX51	2.62	18.6	ofurace	C14H16NO3Cl	C	-	-	-	-
5.4	NI5	3.11	-	captafol	C10H9Cl4NO2S	C	P	-	50	3
5.6	NI150	5.1	9.5	dinocap*	C18H24N2O6	C	P	P	15	2
5.6	NI4	5.8	2.95	mirex	C10Cl12	P	C	P	6	1
5.7	NI13	4.8	7	fenpropathrin	C22H23NO3	-	V #	V	15	2
6	NI8	4.3	8.4	benzoylprop-ethyl	C18H17Cl2NO3	P	NR	NR	6-15-50	1-2-3
6.1	NI200	12	8.9	CGA 205374	C16H11N3O2Cl2	-	NR	NR	6-15-50	1-2-3
6.1		6.4	4.5	bis(2-ethylhexyl) phthalate	C24H38O4	-	C	C	15+50	-
6.2		17	-	deltamethrin, trans-*	C22H19Br2NO3	-	P #	NR	15	2
6.2	FP(WB)12	3.8	-	phosalone oxygen analog	C12H15ClNO5PS	C	-	-	-	-

Appendix I: PESTDATA Chemicals in Order by OV-17 Relative Retention Time (continued)

RRT/c OV-17	Detector Responses with OV-17 Column	RRT/c OV-101	RRT/c OV-225		Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
6.3	HX20/NI35/NP200	4.2	18		iprodione*	C13H13Cl2N3O3	C	S #	NR	50	1-2-3
6.3	NI7	3.8	24		nitralin	C13H19N3O6S	C	P	P	50	3
6.3	NI(WB)20	4.8	6.6		cloquintocet-mexyl	C18H22ClNO3	V	NR	-	6-15-50	1-2-3
6.5		5.4	4.8	-	phenothrin*	C23H26O3	-	-	-	-	-
6.5		4.2	7.6		leptophos oxygen analog	C13H10BrCl2O3P	C	-	-	-	-
6.8		4.8	9.7		piperophos	C14H28NO3PS2	C	-	-	-	-
6.9	FP25/HX17	4.7	10.3		chlorthiophos sulfoxide	C11H15Cl2O4PS2	C	NR	NR	6-15-50	1-2-3
6.9	FP50/NI9	4.5	10.6		EPN	C14H14NO4PS	C	C	C	15	2
7.1	FP150	3.1	14.8		phosmet oxygen analog*	C11H12NO5PS	-	NR	NR	6-15-50	-
7.1	FP(WB)24	3.8	-		carbophenothion oxygen analog sulfone	C11H16ClO5PS2	-	-	-	-	-
7.2		4.5	8.5		tetramethrin*	C19H25NO4	C	NR	NR	6-15-50	1-2-3
7.2	NI8	4.7	8.6		tetrasul sulfoxide	C12H6Cl4OS	-	-	-	-	-
7.2	NI7	4.7	7.2		methoxychlor, p, p'-	C16H15Cl3O2	C	C	C	6	2
7.3	NP50	5	-		fenoxycarb	C17H19NO4	C	-	-	-	-
7.5	FS(WB)1500/NI(WB)0.1/ NP(WB)73	3.3	-		3-desmethyl sulfentrazone	C10H8Cl2F2N4O3S	-	NR	NR	6-15-50	1-2-3
7.5	NP375	3.6	37.1		myclobutanil alcohol metabolite	C15H17ClN40	S	NR	NR	6-15-50	1-2-3
7.5	HX80	5.3	-		iprodione metabolite isomer	C13H13Cl2N3O3	C	S	-	50	-
8	HX30	7.4	-		lambda-cyhalothrin	C23H19ClF3NO3	C	-	-	-	-
8	HX(WB)50	2.67	13		pyrazon	C10H8ClN3O	C	NR	NR	6-15-50	1-2-3
8.1	FP(WB)28/NP45	5.2	-		fenamiphos sulfoxide	C13H22N04PS	C	NR	NR	6-15-50	1-2-3
8.3	NI5	5.2	-		tetradifon	C12H6Cl4O2S	C	C	C	15	2
8.4	FP(WB)20/NP60	4.5	-		fenamiphos sulfone	C13H22NO5PS	C	NR	NR	6-15-50	1-2-3
8.4	FP50/NI78	4	14.9		phosmet	C11H12O4NPS2	C	NR	-	6-15-50	3
8.5	FP(WB)15/NI12	5.8	7.7		leptophos	C13H10BrCl2O2PS	C	C	C	6	2
8.5	NI5	4.4	15.5		photodieldrin	C12H8Cl6O	-	C	C	15+50	2
8.7		4.2	14		pyridaphenthion	C14H17O4N2SP	C	-	-	-	-
8.8		5	14.9		bifenox	C12H9Cl2NO5	C	C	P	15+50	2+3
8.9	NI25/NP100	10.4	12.8		acrinathrin	C26H21F6NO5	V	V	V #	15	2
9.1	FP100/HX22	5.3	18.8		chlorthiophos sulfone	C11H15Cl2O5PS2	C	C	-	50	3
9.1	NI12	5.5	5.5		phosalone	C12H15ClNO4PS2	C	C	C	50	2+3

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Appendix I: PESTDATA Chemicals in Order by OV-17 Relative Retention Time (continued)

RRT/c OV-17	Detector Responses with OV-17 Column	RRT/c OV-101	RRT/c OV-225	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
9.2	FP30	5.1	-	carbophenothion sulfone	C11H16ClO4PS3	C	C	P	6	1
9.4	HN(WB)4	3.28	-	oxycarboxin	C12H13NO4S	R	-	-	-	-
9.8	NP165	5.9	-	clofentezine	C14H8Cl2N4	R	S	-	15	2
10.1		6.6	-	fenarimol	C17H12Cl2N2O	C	P #	C #	50	3
10.1	FP(WB)42	3.7	-	azinphos-methyl oxygen analog	C10H12N3O4PS	C	-	-	-	-
10.5		8.1	11.3	fenoxaprop ethyl ester	C18H16NO5Cl	S	V	V	50	3
10.6	FP40/FP(WB)28/NI20/NP40	5.1	-	sulprofos oxygen analog sulfone	C12H19O5PS2	C	-	-	-	-
11		7	-	tebufenozide	C22H28N2O2	-	NR	NR	6-15-50	1-2-3
11.3	NP(WB)120	4.7	53	dithianon	C14H4O2N2S2	NR	-	-	-	-
11.4		7	-	CGA 118244	C15H13Cl2N3O3	V	NR	NR	6-15-50	1-2-3
11.5	NI1000/NP1000	6.5	-	myclobutanil dihydroxy metabolite	C15H17N4O2Cl	NR	NR	NR	6-15-50	1-2-3
11.7	FP(WB)29	6.1	-	sulprofos sulfoxide*	C12H19O3PS3	C	-	-	-	-
11.8	NP(WB)200	9.4	-	bitertanol*	C20H23N3O2	C	-	-	-	-
11.8		5.2	-	azinphos-methyl	C10H12N3O3PS2	C	NR	NR	6-15-50	1-2-3
12.5	NP(WB)200	9.7	-	bitertanol*	C20H23N3O2	C	-	-	-	-
12.6	NI70	7.9	10.8	HOE-030291	C17H16Cl2O5	-	-	-	-	-
13	FP25	8.1	-	pyrazophos	C14H20N3O5PS	C	-	-	-	-
13.1	FP(WB)26	7.2	-	sulprofos sulfone	C12H19O4PS3	C	-	-	-	-
13.8		9.4	11.1	permethrin, cis-	C21H20Cl2O3	C	V #	C	6+15	2
14.3	FP25/FP(WB)31	6.5	-	dialifor	C14H17ClNO4PS2	C	C	P	15	2
14.8	FP(WB)26/NI20	6.9	-	azinphos-ethyl	C12H16N3O3PS2	C	P	S	50	3
15		11.5	10.9	phenothrin*	C23H26O3	-	-	-	-	-
15		10.2	13	permethrin, trans-	C21H20Cl2O3	C	V #	C	6+15	2
15.4		10.4	-	prochloraz	C15H16Cl3N3O2	C	-	-	-	-
16	FP75/NI50/NP40	8	45	coumaphos oxygen analog	C14H16ClO6P	C	NR	NR	6-15-50	1-2-3
16	NI400	13	13	hexachlorophene	C13H6Cl6O2	-	NR	NR	6-15-50	-
18	FP50/FP(WB)26/NI38/NP34	9	40	coumaphos	C14H16ClO5PS	C	NR	C #	6-15-50	3
20		29	-	deltamethrin, trans-*	C22H19Br2NO3	-	P #	NR	15	2
20.2	FP100	9.5	-	bensulide	C14H24NO4PS3	C	P	C	50	3
21		17.1	-	deltamethrin*	C22H19Br2NO3	C	S #	P	15	2
21.4		14.7	36.9	flucythrinate*	C26H23F2NO4	C	C	-	15	2+3
23		14.1	33	cypermethrin*	C22H19Cl2NO3	C	C	C	15	2

Appendix I: PESTDATA Chemicals in Order by OV-17 Relative Retention Time (continued)

RRT/c OV-17	Detector Responses with OV-17 Column	RRT/c OV-101	RRT/c OV-225	Name	Molecular Formula	Recoveries 302	303	304	Ethers	CH_2Cl_2
24		16.1	42	flucythrinate*	C26H23F2NO4	C	C	-	15	2+3
25		15.1	36	cypermethrin*	C22H19Cl2NO3	C	C	C	15	2
25		13.6	-	quizalofop ethyl ester	C19H17ClN2O4	C	-	-	-	-
35		-	56	fluvalinate*	C26H22ClF3N2O3	C	C	-	15	2
35		27	-	deltamethrin*	C22H19Br2NO3	C	S #	P	15	2
35		20.3	44	fenvalerate*	C25H22ClNO3	C	C	C	15	2
38		25	59	fluvalinate*	C26H22ClF3N2O3	C	C	-	15	2
38		31	19.7	deltamethrin, trans-*	C22H19Br2NO3	-	P #	NR	15	2
38		29	19.9	deltamethrin*	C22H19Br2NO3	C	S #	P	15	2
40		22.5	51	fenvalerate*	C25H22ClNO3	C	C	C	15	2
43	NI200/NP500	23	57	PB-7, methylated	C20H25ClN2O3S	-	-	-	-	-
44		27	64	tralomethrin	C22H19Br4NO3	C	V	S	15	2
46	NI250/NP750	25	87	PB-9	C19H25ClN2O2S	V	NR	NR	6-15-50	1-2-3

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