

Separation of a Mixture of PCBs Using an Accucore C18 HPLC Column

Derek Hillbeck, Thermo Fisher Scientific, Runcorn, Cheshire, UK

Key Words

Accucore, Core Enhanced Technology, solid core, polychlorinated biphenyls, PCB, environment, HPLC

Abstract

Polychlorinated biphenyls (PCBs) are man-made organic compounds composed of two phenyl (benzene ring) groups with multiple chlorine atoms attached. The heat-resistant and non-conductive properties of PCBs led to their extensive use as coolants and lubricants in electrical equipment until the late 1970s when their toxicity was recognized and their usage restricted. However, PCBs are very persistent environmental contaminants. Improper disposal and storage of old electrical equipment continue to allow the chemicals to leach into the environment. Soil and water samples are routinely tested for the presence of PCBs, and a number of EPA methods exist for their analysis.^{1,2} This HPLC method shows the use of the Thermo Scientific Accucore C18 column for the separation of PCBs in a commercial mixture. The 20 minute analysis time provides good resolution between all components.

Introduction

Environmental monitoring of chemicals is becoming more widespread. Traditionally these analyses have been carried out using GC. In laboratories where access to GC equipment is not readily available, alternative approaches such as HPLC should be considered.

Accucore™ HPLC columns use Core Enhanced Technology™ to facilitate fast and highly efficient separations. The 2.6 µm diameter particles are not totally porous but have a solid core and a porous outer layer. The optimized phase bonding creates a series of high coverage, robust phases. The tightly controlled 2.6 µm diameter of the Accucore particles results in performance typically seen with sub-2 µm materials but at much lower backpressures.



Experimental Details

Consumables	Part Number
The PCB congener mixture of chlorinated biphenyls was obtained from a commercial supplier	
Fisher Scientific HPLC grade water	W/0106/17
Fisher Scientific HPLC grade acetonitrile	A/0626/17
Thermo Scientific Premium 2 mL vial convenience kit	60180-600

Sample Preparation

A 200 μ L aliquot of the PCB congener mixture was dissolved in 800 μ L methanol.

Separation Conditions

Instrumentation:	Thermo Scientific Accela UHPLC system		
Column:	Accucore C18 2.6 μ m, 150 x 2.1 mm	17126-152130	
Mobile phase A:	Water		
Mobile phase B:	Acetonitrile		
Gradient:	Time	%A	%B
	0	50	50
	8	30	70
	16	20	80
	19	10	90
	20	10	90
	21	50	50
Column temperature:	65 $^{\circ}$ C		
Injection details:	2 μ L partial loop		
Injection wash solvent:	Water / acetonitrile (80:20 v/v)		
UV detector wavelength:	205 nm		
Backpressure:	230 bar at 400 μ L/min		

Results

Sixteen components were successfully separated with good resolution except for the heptachloro isomers, which are peaks 13 and 14 in Figure 1. Peak class identities were established by correlation of compound hydrophobicity with LC-MS analysis of isotopic distributions and confirmatory mass ions. The isobaric components were not individually identified but treated as groups based on the number of chlorine atoms present in the compound (Table 1).

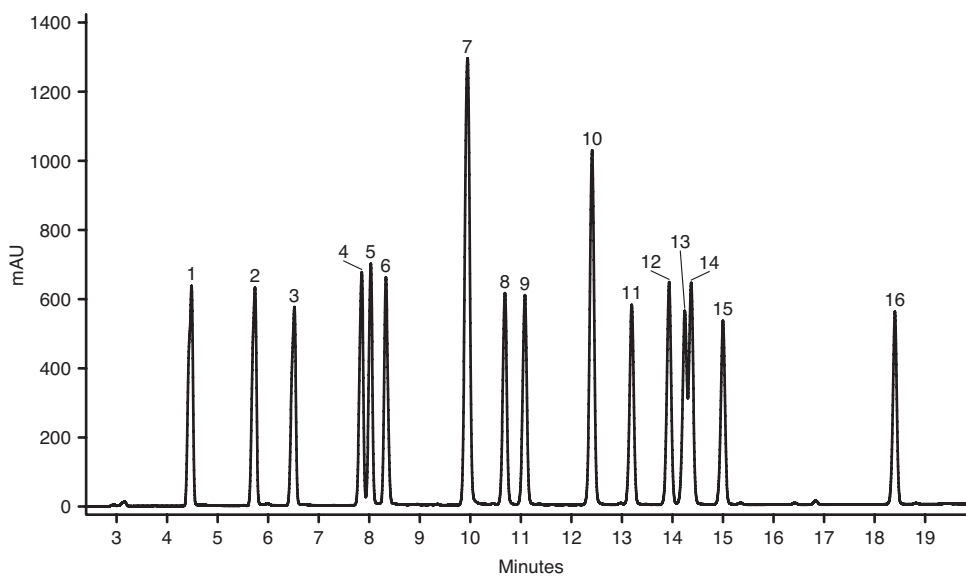


Figure 1: UV 205 nm chromatogram of the HPLC analysis of a commercial mixture of polychlorinated biphenyls

Peak #	Class	Retention Time		Peak Asymmetry		Peak Resolution	
		Mean	% CV	Mean	% CV	Mean	% CV
1	not determined	4.45	0.35	0.76	2.11		
2	Cl ₃	5.72	0.22	0.81	3.07	8.30	4.10
3	Cl ₃	6.49	0.15	0.80	2.78	5.13	4.34
4	Cl ₄	7.82	0.15	0.87	2.36	9.11	4.48
5	Cl ₄	8.00	0.14	0.90	2.46	1.36	4.00
6	Cl ₄	8.30	0.18	1.11	3.16	2.26	3.50
7	Cl ₅	9.89	0.30	0.96	2.29	10.44	3.06
8	Cl ₅	10.61	0.34	0.94	1.01	4.69	2.81
9	Cl ₆	11.01	0.37	0.93	1.19	2.68	2.89
10	Cl ₆	12.33	0.35	0.94	0.86	8.68	2.31
11	Cl ₆	13.11	0.31	0.96	2.18	5.05	2.41
12	Cl ₇	13.87	0.34	0.95	0.50	4.84	1.41
13	Cl ₇	14.18	0.34	Not calculated due to partial resolution		0.72	5.20
14	Cl ₇	14.31	0.31			1.88	2.21
15	Cl ₇	14.94	0.29	0.96	1.12	3.94	1.32
16	Cl ₉	18.34	0.20	0.93	0.94	22.99	0.74

Table 1: Retention time, peak asymmetry and resolution data from the HPLC analysis of eight replicate injections of a commercial mixture of polychlorinated biphenyls

Conclusion

The use of an Accucore C18 column allowed the separation of different PCB classes in less than 20 minutes at standard HPLC backpressures. Accucore C18 columns are therefore an excellent alternative choice for the analysis of PCBs where access to the more traditional GC instrumentation is not available.

References

1. EPA Method 1668: Chlorinated Biphenyl Congeners in Water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS, November 2008, EPA-821-R-08-020.
2. EPA SW 846 Method 8082A: Polychlorinated Biphenyls (PCBs) by Gas Chromatography, February 2007.

thermoscientific.com/chromatography

© 2012 Thermo Fisher Scientific Inc. All rights reserved. All trademarks are the property of Thermo Fisher Scientific Inc. and its subsidiaries. This information is presented as an example of the capabilities of Thermo Fisher Scientific Inc. products. It is not intended to encourage use of these products in any manners that might infringe the intellectual property rights of others. Specifications, terms and pricing are subject to change. Not all products are available in all countries. Please consult your local sales representative for details.

USA and Canada +1 800 332 3331
France +33 (0)1 60 92 48 34
Germany +49 (0) 2423 9431 20 or 21
United Kingdom +44 (0)1928 534110
Japan +81 3 5826 1615

China +86 21 68654588 +86 10 84193588
+86 20 83145199 800 810 5118
India +91 22 6742 9494 +91 27 1766 2352
Australia 1 300 735 292 (free call domestic)
New Zealand 0800 933 966 (free call domestic)
All Other Enquiries +44 (0) 1928 534 050

Technical Support
North America +1 800 332 3331
Outside North America +44 (0) 1928 534 440

Thermo
SCIENTIFIC

Part of Thermo Fisher Scientific