

Certificate of Analysis

CERTIFIED REFERENCE MATERIAL Organic Standard Solution

This document is designed and the certified value(s) and uncertainty(ies) are determined in accordance with ISO Guide 31^[1], ISO Guide 35^[2], and Eurachem / CITAC Guides^[3]

Lot N: X89180

Barcode: 92151192

Certification Date: 07.02.2013

Description of the Reference Material (CRM):

Solution of: 16 components; 100ug/ml each of Aldrin [CAS:309-00-2] ; 2,4'-DDE [CAS:3424-82-6] ; 4,4'-DDE [CAS:72-55-9] ; 2,4'-DDD [CAS:53-19-0] ; 4,4'-DDD (TDE) [CAS:72-54-8] ; 2,4'-DDT [CAS:789-02-6] ; 4,4'-DDT [CAS:50-29-3] ; Dieldrin [CAS:60-57-1] ; Endosulfan-alpha [CAS:959-98-8] ; Endosulfan-beta [CAS:33213-65-9] ; Endrin [CAS:72-20-8] ; Alpha-HCH [CAS:319-84-6] ; Gamma-HCH (Lindane) [CAS:58-89-9] ; Heptachlor [CAS:76-44-8] ; Hexachlorobenzene [CAS:118-74-1] ; Trifluralin [CAS:1582-09-8] in Acetone

Ref N:

D668.K1.A.1

Certified value/ Uncertainty:	Component	Chem. Formula	CAS No.	Certified Value / Uncertainty (mg/l)*
	Aldrin	C ₁₂ H ₆ Cl ₆	309-00-2	99.83 ± 1.69
	2,4'-DDE	C ₁₄ H ₈ Cl ₄	3424-82-6	99.84 ± 1.75
	4,4'-DDE	C ₁₄ H ₈ Cl ₄	72-55-9	99.81 ± 2.01
	2,4'-DDD	C ₁₄ H ₁₀ Cl ₄	53-19-0	99.68 ± 1.55
	4,4'-DDD (TDE)	C ₁₄ H ₁₀ Cl ₄	72-54-8	100.08 ± 1.46
	2,4'-DDT	C ₁₄ H ₉ Cl ₅	789-02-6	99.92 ± 1.68
	4,4'-DDT	C ₁₄ H ₉ Cl ₅	50-29-3	100.33 ± 1.55
	Dieldrin	C ₁₂ H ₈ Cl ₆ O	60-57-1	99.85 ± 1.72
	Endosulfan-alpha	C ₈ H ₆ Cl ₆ O ₃ S	959-98-8	100.16 ± 1.57
	Endosulfan-beta	C ₈ H ₆ Cl ₆ O ₃ S	33213-65-9	100.78 ± 1.56
	Endrin	C ₁₂ H ₈ Cl ₆ O	72-20-8	100.41 ± 1.84
	Alpha-HCH	C ₆ H ₆ Cl ₆	319-84-6	99.91 ± 1.57
	Gamma-HCH (Lindane)	C ₆ H ₆ Cl ₆	58-89-9	99.95 ± 1.74
	Heptachlor	C ₁₀ H ₅ Cl ₇	76-44-8	100.51 ± 1.59
	Hexachlorobenzene	C ₆ Cl ₆	118-74-1	100.75 ± 1.50
	Trifluralin	C ₁₁ H ₇ F ₃ N ₃ O ₄	1582-09-8	99.90 ± 1.66

Concept of Certification and traceability statement:

This certified reference material is produced by gravimetric measurement and dissolving the individual substances in Acetone .

Method of certification:

CRM's calibration procedure (CP-002)

The certified value was obtained gravimetrically and confirmed experimentally by GC/MS or HPLC



The reported expanded uncertainty of measurement is stated as the standard uncertainty of measurement multiplied by the coverage factor $k = 2$, which for a normal distribution corresponds to a coverage probability of approximately 95%. The standard uncertainty of measurement has been determined in accordance with EA 4/02

Property of the result of a measurement whereby it can be related to stated references, usually national or international standards, through an unbroken chain of comparisons all having stated uncertainties (ISO VIM^[5])
The metrological traceability is assured through gravimetric measurement and dissolving the certified reference material from accredited according to ISO/IEC 17025^[6] and/or ISO Guide 34^[7] laboratories/producers and traceable to SI. All contributions in relation to the certification of standard solutions are considered when evaluating the uncertainty.

The measurement results are traceable to SI. All analytical balances used for the preparation of the solution are calibrated yearly under an in-house procedure with class E1 and class E2 analytical weights, traceable to SI (DKD) and are daily checked.

Class A laboratory glassware is used.

The results from temperature measurement are traceable to SI. The thermometers used for solution's calibration are calibrated from an ISO 17025 accredited laboratory. The ambient conditions are controlled with a hygrometer calibrated from an ISO 17025 accredited laboratory.

Both, purity of the starting materials and solvent were checked using appropriate analytical instrument.

Starting material, purity (Lot N):

Aldrin	98.8% (41113585)
2,4'-DDE	99% (41107690)
4,4'-DDE	98.5% (41104309)
2,4'-DDD	98% (41104675)
4,4'-DDD (TDE)	99% (41117156)
2,4'-DDT	98.2% (41109915)
4,4'-DDT	99.5% (41107256)
Dieldrin	97.5% (41108918)
Endosulfan-alpha	97% (41108970)
Endosulfan-beta	99.5% (41106402)
Endrin	99% (41108857)
Alpha-HCH	98% (41109113)
Gamma-HCH (Lindane)	98.5% (41113684)
Heptachlor	99.5% (41104682)
Hexachlorobenzene	99.8% (41105511)
Trifluralin	99.5% (41093658)

Density: 0.7900 g/cm³ at 21.3 °C

Expiry date: until 03.2015

Intended use:

For Laboratory Use Only

This CRM is intended for:

- Calibration of TLC, GC/FID, GC/TCD, GC/ECD, GC/MS, HPLC/UV and HPLC/MS
- Validation of analytical methods
- Preparation of "working reference samples"
- Detection limit and linearity studies

This statement is not intended to restrict the use for other purposes.

Instructions for the correct use of this reference material:

This certified reference material can be used directly or can be diluted in an appropriate solvent. Only a clean class A glassware should be used. Do not pipet from container. Obtained concentration (in mg/l) after dilution is a result from the multiplication of certified value of CRM concentration and the CRM's volume used for dilution and divided into the flask's volume used for dilution.

Stability and storage:

This CRM is with a guaranteed stability until $\pm 0.5\%$ of the certified concentration for a period of 24 months. Stability is guaranteed provided that the solution is kept in its original packaging, tightly closed under normal laboratory conditions.

Hazardous situation:

The normal laboratory safety precautions should be observed when working with this RM. Further details for the handling of this RM are available as safety data sheet.

Level of homogeneity

This solution was mixed according to an in-house procedure (OQP 5.13.1) and is guaranteed to be homogeneous.

To ensure sufficient homogeneity of the sample prior to use thoroughly mix by inversion.

This Certified Reference Material was produced under ISO 9001 Quality Control System.
The instructions of the ISO Guide 34 [7] were considered for the preparation of this solution.

Names and signatures of certifying officers:

Laboratory:  Tihomir Stoyanov

Manager:  Krassimira Taralova

- [1] ISO Guide 31: Reference materials - Contents of certificates and labels
- [2] ISO Guide 35: Reference materials - General and statistical principles for certification
- [3] EURACHEM/CITAC Guide: Quantifying Uncertainty in Analytical Measurement
- [4] EA 4/02: Expression of the Uncertainty of Measurement in Calibration
- [5] ISO/IEC Guide 99: International Vocabulary of Metrology-Basic and general concepts and associated terms (VIM)
- [6] ISO/IEC 17025: General requirements for the competence of testing and calibration laboratories
- [7] ISO Guide 34: General Requirements for the Competence of Reference Material Producers

This certificate relates solely to the lot number given above.

All processes (including generating of this certificate) are completely controlled by the specialized Computer-Aided-Manufacturing (CAM) software.

Additional Information Gravimetric Data

Component	Purity %	Source Lot No	Weighed quantity, g	Final quantity, kg.10 ³	Bulk/ Standard Solution lot No	Concen- tration mg/kg	Chemist ID
Aldrin	98.8	41113585	0.01672	3.1076	91161505	5315.8	AS
		91161505	0.1878	7.9000	92151192	126.368	KR
2,4'-DDE	99	41107690	0.01449	2.7172	91161512	5279.5	AS
		91161512	0.1891	7.9000	92151192	126.373	KR
4,4'-DDE	98.5	41104309	0.01308	2.4886	91159519	5177.1	AS
		91159519	0.1928	7.9000	92151192	126.347	KR
2,4'-DDD	98	41104675	0.02909	2.9573	91154118	9639.9	AS
		91154118	0.1034	7.9000	92151192	126.173	KR
4,4'-DDD (TDE)	99	41117156	0.03184	3.0019	91162168	10500.5	AS
		91162168	0.0953	7.9000	92151192	126.677	KR
2,4'-DDT	98.2	41109915	0.01724	2.9922	91154095	5657.9	AS
		91154095	0.1766	7.9000	92151192	126.480	KR
4,4'-DDT	99.5	41107256	0.02955	2.9805	91162397	9864.9	AS
		91162397	0.1017	7.9000	92151192	126.995	KR
Dieldrin	97.5	41108918	0.01544	3.0952	91162670	4863.7	AS
		91162670	0.2053	7.9000	92151192	126.394	KR
Endosulfan-alpha	97	41108970	0.02651	2.5698	91162328	10006.5	AS
		91162328	0.1001	7.9000	92151192	126.785	KR
Endosulfan-beta	99.5	41106402	0.02826	3.4569	91163264	8134.1	AS
		91163264	0.1239	7.9000	92151192	127.571	KR
Endrin	99	41108857	0.01283	2.5806	91154316	4922.1	AS
		91154316	0.204	7.9000	92151192	127.101	KR
Alpha-HCH	98	41109113	0.02558	2.6045	91162465	9625.0	AS
		91162465	0.1038	7.9000	92151192	126.466	KR
Gamma-HCH (Lindane)	98.5	41113684	0.01501	2.9348	91162434	5037.8	AS
		91162434	0.1984	7.9000	92151192	126.519	KR
Heptachlor	99.5	41104682	0.02373	2.4056	91162946	9815.2	AS
		91162946	0.1024	7.9000	92151192	127.225	KR
Hexachlorobenzene	99.8	41105511	0.02746	2.7147	91162144	10095.1	AS
		91162144	0.0998	7.9000	92151192	127.529	KR
Trifluralin	99.5	41093658	0.0178	3.1346	91159168	5650.2	AS
		91159168	0.1768	7.9000	92151192	126.451	KR