

International Atomic Energy Agency
Department of Nuclear Sciences and Applications
IAEA Environment Laboratories

Vienna International Centre, P.O. Box 100, 1400 Vienna, Austria

REFERENCE SHEET

REFERENCE MATERIAL

IAEA-408

ORGANOCHLORINE COMPOUNDS, PETROLEUM HYDROCARBONS AND STEROLS IN SEDIMENT SAMPLE

Recommended values (based on dry mass)

Pesticides and PCBs

Analyte	Recommended value* [ng g ⁻¹]	95% Confidence interval** [ng g ⁻¹]
HCB	0.41	0.30-0.57
γ-HCH (Lindane)	0.19	0.11-0.21
pp' DDE	1.4	0.9-2.0
pp' DDT	0.67	0.48-0.98
Dieldrin	0.30	0.30-0.48
PCB 28	0.79	0.35-0.98
PCB 52	0.60	0.38-0.93
PCB 101	1.2	0.8-1.7
PCB 105	0.57	0.44-0.67
PCB 110	0.83	0.50-0.90
PCB 118	1.2	0.9-1.6
PCB 128	0.33	0.27-0.53
PCB 138	1.6	1.1-2.1
PCB 149	1.4	1.3-1.6
PCB 153	1.9	1.0-2.1
PCB 170	0.47	0.34-0.59
PCB 180	1.1	0.9-1.2

PCB 187	0.68	0.49-0.94
PCB 194	0.20	0.18-0.23

*Median values expressed on a dry mass basis

**95% confidence intervals of the median

Petroleum hydrocarbons

Analyte	Unit	Recommended value*	95% Confidence interval**
Resolved Aliphatics	µg/g	11	6-17
<i>n</i> -C ₁₇	ng/g	74	56-140
Pristane	ng/g	69	50-130
Phytane	ng/g	78	63-130
Σ <i>n</i> -Alkanes (C ₁₄ -C ₃₄)	µg/g	8.1	4.3-12.0
Phenanthrene	ng/g	35	21-43
Anthracene	ng/g	9.8	8.0-13.0
Chrysene	ng/g	35	25-56
Fluoranthene	ng/g	84	53-110
Pyrene	ng/g	77	57-93
Benzo[b]fluoranthene	ng/g	46	32-69
Benzo[k]fluoranthene	ng/g	46	26-61
Benz[a]anthracene	ng/g	53	35-60
Perylene	ng/g	320	140-420
Benzo[e]pyrene	ng/g	55	51-81
Benzo[a]pyrene	ng/g	48	30-63
Naphthalene	ng/g	27	16-47
Benzo[g,h,i]perylene	ng/g	38	20-52

*Median values expressed on a dry mass basis

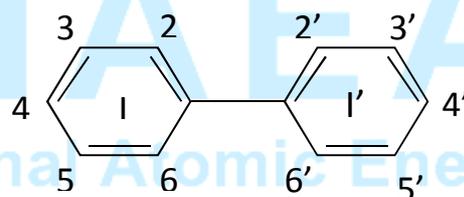
**95% confidence intervals of the median

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Systematic numbering of PCB congeners

IUPAC No		IUPAC No	
8	Dichlorobiphenyl 2,4'	128	Hexachlorobiphenyl 2,2',3,3',4,4'
		138	2,2',3,4,4',5'
18	Trichlorobiphenyl 2,2',5	149	2,2',3,4',5',6
28	2,4,4'	153	2,2',4,4',5,5'
31	2,4',5	156	2,3,3',4,4',5
			Heptachlorobiphenyl
44	Tetrachlorobiphenyl 2,2',3,5'	170	2,2',3,3',4,4',5
49	2,2',4,5'	174	2,2',3,3',4,5,6'
52	2,2',5,5'	177	2,2',3,3',4',5,6
		180	2,2',3,4,4',5,5'
87	Pentachlorobiphenyl 2,2',3,4,5'	183	2,2',3,4,4',5',6
101	2,2',4,5,5'	187	2,2',3,4',5,5',6
105	2,3,3',4,4'		Octachlorobiphenyl
110	2,3,3',4',6	194	2,2',3,3',4,4',5,5'
118	2,3',4,4',5	195	2,2',3,3',4,4',5,6
		196	2,2',3,3',4,4',5',6



Information Values (based on dry mass)

Chlorinated Pesticides

Analyte	Information value* [ng g ⁻¹]	95% Confidence interval** [ng g ⁻¹]
α-HCH	0.61	0.21-1.50
β-HCH	0.55	0.38-1.70
pp' DDD	0.87	0.56-1.70
op DDD	0.19	0.18-0.33
op DDT	0.38	0.09-3.80
Heptachlor	0.42	0.23-0.70
Heptachlor epoxide	0.64	0.43-1.50
Aldrin	0.41	0.20-2.30
Endrin	0.57	0.14-1.20
α-Endosulfan	1.6	0.3-6.2
Endosulfan sulfate	1.6	0.5-8.2
α-Chlordane	0.12	0.10-0.34
γ-Chlordane	0.27	0.06-0.48
Aroclor 1254	19	14-40
Aroclor 1260	10	7-25

*Median values expressed on a dry mass basis

**95% confidence intervals of the median

PCB Congeners

Analyte	Information value* [ng g ⁻¹]	95% Confidence interval** [ng g ⁻¹]
PCB 8	0.24	0.22-4.60
PCB 18	0.74	0.13-1.90
PCB 31	0.43	0.09-1.50
PCB 44	0.47	0.23-3.00
PCB 49	0.35	0.22-0.38
PCB 87	0.87	0.26-1.10
PCB 156	0.36	0.31-0.39
PCB 174	0.34	0.20-0.44
PCB 177	0.35	0.23-0.43
PCB 183	0.32	0.26-0.33
PCB 195	0.12	0.06-0.16
PCB 196	0.22	0.17-0.40

*Median values expressed on a dry mass basis

**95% confidence intervals of the median

Petroleum Hydrocarbons

Analyte	Unit	Information value*	95% Confidence interval**
UVF equivalent Chrysene	µg/g	6.0	4.6-19.0
UVF equivalent ROPME oil	µg/g	49	35-120
Total aliphatics	µg/g	120	81-160
Unresolved aliphatics	µg/g	110	68-150
<i>n</i> -C ₁₈	ng/g	90	60-110
Total aromatics	µg/g	3.7	1.3-11.0
Resolved aromatics	µg/g	1.9	0.24-6.8
2 Methyl-phenanthrene	ng/g	12	12-14
1 Methyl-phenanthrene	ng/g	10	9-11
Fluorene	ng/g	6.7	4.6-24.0
1 Methyl-naphthalene	ng/g	7.5	4.7-12.0
2 Methyl-naphthalene	ng/g	14	13-33
Indeno[1,2,3,cd]pyrene	ng/g	51	45-53
Dibenz[ah]anthracene	ng/g	11	8-14
Acenaphthylene	ng/g	3.6	2.1-4.7
Acenaphthene	ng/g	3.3	2.0-17.0

*Median values expressed on a dry mass basis

**95% confidence intervals of the median

Sterols

Information values for sterol compounds (Coprostanol, Cholesterol, Cholestanol, β Sitosterol, Campesterol and Stigmasterol) are available in the report [1].

Origin and preparation of the material

A large sediment sample was collected from the mudflats of the Tagus estuary. This sediment was deep-frozen, freeze-dried, ground and sieved through a 150 µm stainless steel sieve.

The sediment fraction of particle size less than 150 µm was homogenized by mixing in a rotating drum for two weeks. Then, aliquots of about 40 g were packaged into glass bottles with aluminium screw caps and sealed with Teflon tape.

Characterization study

The IAEA-408 candidate reference material was characterized in an interlaboratory comparison (ILC).

48 laboratories (including the IAEA-MEL) from 36 countries reported results.

Participants were requested to analyze chlorinated compounds, petroleum hydrocarbons and sterols by the analytical technique of their choice. They were also requested to make at least one, but preferably three separate determinations for each compound and to report the results together with a short description of the method used.

Assignment of values – Certification procedure

An interlaboratory comparison for the determination of chlorinated compounds, petroleum hydrocarbons and sterols in IAEA-408 was carried out in 1998-1999 and the results returned by the participating laboratories were used to establish "recommended values" for the concentrations of some compounds in this material.

The details concerning the criteria for qualification as a recommended or information value are reported in [1, 2]; the report "World-wide and regional intercomparison for the determination of organochlorine compounds, petroleum hydrocarbons, and sterols in sediment sample IAEA-408", IAEA/AL/121; IAEA/MEL/67, IAEA, Monaco (1999) [1] may be downloaded free of charge from:

http://nucleus.iaea.org/rpst/Documents/al_121.pdf

All other documents are available upon request.

Based on the evidence on calibrators used, quality control procedures applied by the participating laboratories and their generally high quality performance in the IAEA interlaboratory comparisons, the Certification Committee confirmed these assigned values.

Statement on metrological traceability and uncertainty of assigned values

The property values assigned to the IAEA-408 Reference material are calculated as mass fraction of chlorinated pesticides, PCBs, aliphatic hydrocarbons, PAHs and sterols expressed in the respective derived SI unit (ng g^{-1} and $\mu\text{g g}^{-1}$).

Measurement uncertainty associated with individual assigned values represents 95% confidence interval of the mean of means.

Evidence on metrological traceability of reference materials and calibrators used in the characterization process was provided by all laboratories in their reports. More details can be found in reference [1, 2].

Intended use

IAEA-408 is intended to be used when evaluating the accuracy of analytical procedures for the determination of chlorinated compounds, petroleum hydrocarbons and sterols in sediment samples, in the elaboration of new analytical procedures for sediment samples, and for educational purposes. This material is not to be used as calibrator.

Instructions for use

Homogeneity of the material

The homogeneity of the material was checked by determining the concentration of some representative compounds (chlorinated pesticides, petroleum hydrocarbons and sterols) in 10 replicate analyses taken randomly in the bulk of the powder. A one-way variance analysis indicated that the material can be considered homogeneous.

Dry mass determination

The moisture content of the lyophilized sample, as determined by drying to a constant mass at 105 °C, was found to be 3.7%. Since the moisture content can change with the ambient humidity and temperature, it is recommended that the water content of this material always be determined in a separate sub-sample (not that taken for analysis) by drying to a constant mass (approximately 24 hours) at 105 °C. Results should always be reported on a dry mass basis.

Recommended minimum test portion

The reference material is supplied in 40g units. The recommended sample size for analysis is 5 g for petroleum hydrocarbons and sterols and 10 g for organochlorine pesticides and PCBs respectively.

Handling and storage

The material should be stored in the dark and kept in a refrigerator.

Analysts are reminded to take appropriate precautions in order to avoid contamination of the material during handling

Issue and expiry date

The original issue date of this reference material is **November 1999**. The material was re-assessed in 2012 and no changes in the assigned property values were found. The expiry date has therefore been extended to **November 2019**. The IAEA is monitoring the long term stability of the material and customers will be informed in case of any observed change.

Legal disclaimer

The IAEA makes no warranties, expressed or implied, with respect to the data contained in this reference sheet and shall not be liable for any damage that may result from the use of such data.

Compliance with ISO Guide 31:2000

The content of this IAEA Reference Sheet is in compliance with the ISO Guide 31:2000: Reference materials – Content of certificates and labels [3].

Citation of this reference sheet

It is suggested to cite this reference sheet according to the following example, as appropriate to the citation format used: INTERNATIONAL ATOMIC ENERGY AGENCY, Reference Sheet for RM IAEA-408, Organochlorine compounds, petroleum hydrocarbons and sterols in sediment sample. IAEA, Vienna, 8 pp. (The latest version published applies, see “Note” below).

Note

Users of this material should ensure that the reference sheet in their possession is current. The current version may be found in the IAEA’s Reference Materials online catalogue:

<http://nucleus.iaea.org/rpst/ReferenceProducts/ReferenceMaterials>

In case of any discrepancies with other published material related to the IAEA-408 RM, the values given in the current version of the reference sheet shall be considered.

Further information:

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REFERENCES

- [1] VILLENEUVE, J.-P., DE MORA, S.J., CATTINI, C., CARVALHO, F.P., World-wide and regional intercomparison for the determination of organochlorine compounds, petroleum hydrocarbons, and sterols in sediment sample IAEA-408, IAEA/AL/121; IAEA/MEL/67, International Atomic Energy Agency, Marine Environment Laboratory, Monaco (1999).
- [2] VILLENEUVE, J.-P., DE MORA, S. J., CATTINI, C., CARVALHO, F. P., Determination of organochlorinated compounds and petroleum hydrocarbons in sediment sample IAEA-408. Results from a world-wide intercalibration exercise, J. Environ. Monit. **2** (2000) 524-528.
- [3] INTERNATIONAL ORGANIZATION FOR STANDARDIZATION, Reference materials – Content of certificates and labels, ISO Guide 31: 2000, ISO, Geneva (2000).



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