

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-417

ORGANOCHLORINE COMPOUNDS AND PETROLEUM HYDROCARBONS IN SEDIMENT SAMPLE

Recommended values (based on dry mass)

Pesticides and PCBs

Analyte	Recommended value* [ng g ⁻¹]	95% Confidence interval [ng g ⁻¹]
γ HCH (Lindane)	0.54	0.39 - 0.69
pp' DDE	14.0	12.1 – 15.9
pp' DDD	21.0	18.1 – 23.9
Trans nonachlor	0.32	0.08 – 0.56
PCB No 28	5.7	4.7 – 6.7
PCB No 31	4.1	3.2 – 5.1
PCB No 44	9.7	7.2 – 12.2
PCB No 49	7.8	5.4 – 10.2
PCB No 52	17.0	14.5 – 19.5
PCB No 74	5.1	2.5 – 7.7
PCB No 87	19.0	14.4 – 23.6
PCB No 99	19.0	11.8 – 26.2
PCB No 101	42.0	37.1 – 46.9

PCB No 105	22.0	19.8 – 24.2
PCB No 110	42.0	32.9 – 51.1
PCB No 128	12.0	9.6 – 14.4
PCB No 138	45.0	38.4 – 51.6
PCB No 149	25.0	18.4 – 31.6
PCB No 156	5.9	5.1 – 6.7
PCB No 170	8.1	5.9 – 10.3
PCB No 174	3.1	1.9 – 4.3
PCB No 177	1.8	1.0 – 2.6
PCB No 183	3.4	2.4 – 4.4
PCB No 187	8.1	5.7 – 10.5
PCB No 194	2.7	1.9 – 3.5
PCB No 195	1.2	0.6 – 1.8
PCB No 206	1.8	0.9 – 2.7
PCB No 209	1.2	0.5 – 1.9

*Average values calculated after rejection of outliers expressed on a dry mass basis

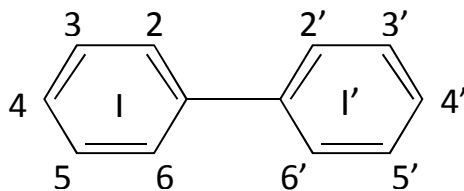
Petroleum Hydrocarbons

Analyte	Recommended value* [ng g ⁻¹]	95% Confidence interval [ng g ⁻¹]
<i>n</i> -C ₁₇	200	120 – 280
Phenanthrene	3900	3400 – 4400
1 Methyl Phenanthrene	320	230 – 420
Anthracene	630	520 – 740
Chrysene	3600	3100 – 4100
Fluorene	230	160 – 300
Fluoranthene	7700	6800 – 8600
Pyrene	6000	5300 – 6700
Benzo [b] Fluoranthene	4100	3100 – 5100
Benzo [k] Fluoranthene	2000	1800 – 2200
Benz [a] Anthracene	3200	2800 – 3600
Perylene	1200	470 – 1900
Benzo [e] Pyrene	3000	2500 – 3500
Benzo [a] Pyrene	2800	2400 – 3200
Benzo [g,h,i] Perylene	2300	1900 – 2700
Indeno [1,2,3-cd] Pyrene	2700	2500 – 2900
Acenaphthene	180	130 – 230
Dibenzothiophene	280	260 – 310

*Average values calculated after rejection of outliers expressed on a dry mass basis

Systematic numbering of PCB congeners

IUPAC No		IUPAC No	
	Dichlorobiphenyl		Hexachlorobiphenyl
8	2,4'	128	2,2',3,3',4,4'
		138	2,2',3,4,4',5'
	Trichlorobiphenyl	141	2,2',3,4,5,5'
18	2,2',5	149	2,2',3,4',5',6
22	2,3,4'	151	2,2',3,5,5',6
28	2,4,4'	153	2,2',4,4',5,5'
31	2,4',5	156	2,3,3',4,4',5
		157	2,3,3',4,4',5'
	Tetrachlorobiphenyl	158	2,3,3',4,4',6
40	2,2',3,3'	167	2,3',4,4',5,5'
44	2,2',3,5'		
45	2,2',3,6		Heptachlorobiphenyl
49	2,2',4,5'	170	2,2',3,3',4,4',5
52	2,2',5,5'	174	2,2',3,3',4,5,6'
66	2,3',4,4'	177	2,2',3,3',4',5,6
70	2,3',4',5	180	2,2',3,4,4',5,5'
74	2,4,4',5	183	2,2',3,4,4',5',6
77	3,3',4,4'	185	2,2',3,4,5,5',6
		187	2,2',3,4',5,5',6
	Pentachlorobiphenyl	189	2,3,3',4,4',5,5'
87	2,2',3,4,5'		
91	2,2',3,4',6		Octachlorobiphenyl
95	2,2',3,5',6	194	2,2',3,3',4,4',5,5'
97	2,2',3',4,5	195	2,2',3,3',4,4',5,6
99	2,2',4,4',5	199	2,2',3,3',4,5,5',6'
101	2,2',4,5,5'	200	2,2',3,3',4,5,6,6'
105	2,3,3',4,4'	201	2,2',3,3',4,5',6,6'
110	2,3,3',4',6		
114	2,3,4,4',5		Nonachlorobiphenyl
118	2,3',4,4',5	206	2,2',3,3',4,4',5,5',6
126	3,3',4,4',5		
			Decachlorobiphenyl
		209	2,2',3,3',4,4',5,5',6,6'



Information Values (based on dry mass)

Chlorinated Pesticides

Analyte	Information value* [ng g ⁻¹]	95% Confidence interval [ng g ⁻¹]
HCB	1.2	0.9 – 1.5
pp' DDT	19.0	15.8 – 22.2
op DDD	11.0	4.8 – 17.2
Heptachlor	2.0	1.1 – 2.9
Heptachlor Epoxide	4.8	1.9 – 7.7
Aldrin	1.6	1.0 – 2.2
Dieldrin	2.3	1.2 – 3.4
Endrin	7.1	4.3 – 9.9
α Endosulfan	14.0	3.5 – 24.5
β Endosulfan	5.1	2.3 – 7.9
α Chlordane	1.8	0.7 – 2.9
γ Chlordane	1.2	0.7 – 1.8
Aroclor 1254	410	310 – 520
Aroclor 1260	170	110 – 230

*Average values calculated after rejection of outliers expressed on a dry mass basis

PCB Congeners

Analyte	Information value * [ng g ⁻¹]	95% Confidence interval [ng g ⁻¹]
PCB No 8	1.8	0.3 – 3.3
PCB No 18	3.7	2.0 – 5.4
PCB No 22	1.0	0.1 – 1.9
PCB No 45	0.46	0.32 – 0.60
PCB No 66	23.0	9.6 – 36.4
PCB No 70	20.0	10.8 – 29.2
PCB No 95	20.0	17.6 – 22.4
PCB No 97	9.1	5.9 – 12.3
PCB No 118	43.0	37.4 – 48.6
PCB No 151	3.9	3.5 – 4.4
PCB No 153	39.0	33.2 – 44.8
PCB No 158	6.0	1.5 – 10.5
PCB No 180	16.0	13.8 – 18.2
PCB No 199	1.2	0.2 – 2.2

*Average values calculated after rejection of outliers expressed on a dry mass basis

Petroleum Hydrocarbons

Analyte	Information value * [ng g ⁻¹]	95% Confidence interval [ng g ⁻¹]
Total Aliphatics	230000	115000 – 345000
Resolved Aliphatics	14000	10000 – 18000
Unresolved Aliphatics	230000	123000 – 337000
Pristane	420	56 – 780
<i>n</i> - C ₁₈	230	120 – 350
Phytane	370	190 – 550
Σ <i>n</i> -Alkanes [C ₁₄ -C ₃₄]	9500	5700 – 13000
Resolved Aromatics	48000	32000 – 64000
Biphenyl	42.0	32.1 – 51.9
Naphthalene	150	100 – 200
1 Methyl Naphthalene	53	31 – 75
2 Methyl Naphthalene	81	11 – 150
2 Methyl Phenanthrene	580	210 – 950
Dibenz [a,h] Anthracene	1100	510 – 1700

*Average values calculated after rejection of outliers expressed on a dry mass basis

Origin and preparation of the material

A bulk sediment sample was collected from the Venice Lagoon, Italy. This sediment was deep-frozen, dried, ground and sieved through a 250 µm stainless steel sieve. The sediment fraction of particle size less than 250 µm was homogenized by mixing it 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-417 candidate reference material was characterized in an interlaboratory comparison (ILC). 97 laboratories (including the IAEA-MEL) from 46 countries reported results.

Participants were requested to analyse 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

The assigned values were established on the basis of statistically valid results submitted by laboratories which had participated in an international interlaboratory comparison organized by the IAEA Marine Environment Laboratory, Monaco, in 2001. The details concerning the criteria for qualification as a reference or information value are reported in [1, 2]. The report "World-wide and regional intercomparison for the determination of organochlorine compounds and petroleum hydrocarbons in sediment sample IAEA-417", IAEA/AL/131; IAEA/MEL/71, IAEA, Monaco, 2002 may be downloaded free of charge from: http://nucleus.iaea.org/rpst/Documents/al_131.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 comparison; the Certification Committee confirmed these assigned values.

Statement on metrological traceability and uncertainty of assigned values

The property values assigned to the IAEA-417 Reference material are calculated as mass fraction of chlorinated pesticides, PCBs, aliphatic hydrocarbons and PAHs expressed in the derived SI unit ng 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 may be found in reference [1].

Intended use

This Reference Material is intended to be used as a quality control material for the assessment of a laboratory's analytical work, for the validation of analytical methods and for quality assurance within a laboratory in the determination of chlorinated compounds and petroleum hydrocarbons in sediment samples. 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 ten replicate analyses taken randomly in the bulk of the powder. A one-way variance analysis indicated that the material can be considered homogenous.

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 1.6%. Since the moisture content can change with the ambient humidity and temperature, it is recommended that it 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 40 g units. The recommended sample size for analysis is 2 g for petroleum hydrocarbons and 5 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 **May 2002**. The material was re-assessed in 2012 and corrections to the assigned values were introduced. The expiry date has therefore been extended to **May 2022**. 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-417, Organochlorine compounds and petroleum hydrocarbons in sediment sample. IAEA, Vienna, 8 pp. (The latest version published applies, see “Note” below).

Note

Certified values as stated in this reference sheet may be updated if more information becomes available. 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-417 RM, the values given in the current version of the reference sheet shall be considered.

Further information:

For further information regarding this material, please contact:

Head, Marine Environmental Studies Laboratory

International Atomic Energy Agency

Environment Laboratories

4, Quai Antoine 1er

MC 98000 MONACO

Tel.: 377 97 97 72 72

Fax: 377 97 97 72 73

E-mail: mel@iaea.org

REFERENCES

- [1] VILLENEUVE, J.-P., DE MORA, S. J., CATTINI, C., World-wide and regional intercomparison for the determination of organochlorine compounds and petroleum hydrocarbons in sediment sample IAEA-417, IAEA/AL/131; IAEA/MEL/71, IAEA, Monaco (2002).
- [2] VILLENEUVE, J.-P., DE MORA, S. J., CATTINI, C., Global intercomparison for the determination of chlorinated pesticides, PCBs and petroleum hydrocarbons in sediment sample IAEA-417, Environ. Technol. **23** (2002) 1203-1217.
- [3] INTERNATIONAL ORGANIZATION FOR STANDARDIZATION, Reference materials – Content of certificates and labels, ISO Guide 31: 2000, ISO, Geneva (2000).



Mr Ales Fajgelj
Chair,
RM Certification Committee



Ms Chantal Cattini
Project Officer,
Marine Environmental Studies
Laboratory

IAEA

International Atomic Energy Agency