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

CERTIFIED REFERENCE MATERIAL

IAEA-451

MASS FRACTIONS OF ORGANOCHLORINE COMPOUNDS, POLYBROMINATED DIPENYL ETHERS AND PETROLEUM HYDROCARBONS IN CLAM (*Gafrarium tumidum*)

Certified mass fraction values
(based on dry mass)

Chlorinated pesticides

Analyte	Unit	Certified value[1]	Expanded uncertainty[2]
EOM	mg g^{-1}	42.2	4.4
HCB	ng g^{-1}	0.39	0.04
Dieldrin	ng g^{-1}	1.88	0.16
α-Chlordane	ng g^{-1}	0.56	0.04
γ-Chlordane	ng g^{-1}	0.46	0.13
Aroclor 1260	ng g^{-1}	53.2	4.0

Certified mass fraction values

(based on dry mass)

PCB congeners

Analyte	Certified value[1] [ng g^{-1}]	Expanded uncertainty[2] [ng g^{-1}]
PCB 28	0.85	0.09
PCB 95	0.58	0.10
PCB 101	1.74	0.14
PCB 105	0.49	0.12
PCB 110	0.88	0.13
PCB 118	1.01	0.08
PCB 128	0.49	0.04
PCB 138	5.30	0.58
PCB 149	3.33	0.42
PCB 153	8.59	0.78
PCB 170	3.05	0.40
PCB 174	1.32	0.07
PCB 177	0.94	0.10
PCB 180	6.56	1.20
PCB 183	1.82	0.22
PCB 187	3.97	0.26
PCB 194	1.45	0.09
PCB 206	0.24	0.03

PBDE

Analyte	Certified value[1] [ng g^{-1}]	Expanded uncertainty[2] [ng g^{-1}]
PBDE 100	0.23	0.04

(1) Robust mean of the accepted data sets, each set being obtained by a different laboratory and/or a different method of determination.
(2) Estimated expanded uncertainty with a coverage factor *k*=2, corresponding to a level of confidence of approximately 95%, as defined in the Evaluation of measurement data – Guide to the expression of uncertainty in measurement JCGM100:2008 [1].

Certified mass fraction values

(based on dry mass)

Petroleum hydrocarbons

Analyte	Unit	Certified value[1]	Expanded uncertainty[2]
EOM	mg g^{-1}	36.7	6.4
Total aliphatics	µg g^{-1}	244	34
n-C_{17}	ng g^{-1}	373	44
Naphthalene	ng g^{-1}	14.8	1.2
Phenanthrene	ng g^{-1}	15.8	5.6
Chrysene	ng g^{-1}	26.9	2.0
Fluoranthene	ng g^{-1}	49.3	3.2
Pyrene	ng g^{-1}	40.0	4.6
Benzo[b]fluoranthene	ng g^{-1}	35.8	6.2
Benzo[k]fluoranthene	ng g^{-1}	14.7	3.2
Benz[a]anthracene	ng g^{-1}	19.2	1.3
Benzo[a]pyrene	ng g^{-1}	18.2	2.4
Benzo[g,h,i]perylene	ng g^{-1}	19.5	2.4
Dibenz[a,h]anthracene	ng g^{-1}	5.32	1.36

Recommended mass fraction values

(based on dry mass)

Chlorinated pesticides and PCB congeners

Analyte	Mass fraction[1] [ng g^{-1}]	Expanded uncertainty[2] [ng g^{-1}]
α-HCH	0.78	0.14
γ-HCH (Lindane)	0.56	0.05
pp' DDE	1.73	0.22
pp' DDD	0.99	0.22
pp' DDT	1.34	0.22
Heptachlor	2.07	0.22
Aldrin	0.87	0.10
α-Endosulfan	1.20	0.20
PCB 31	0.29	0.02
PCB 52	0.82	0.04
PCB 195	0.45	0.03

PBDEs

Analyte	Mass fraction[(1)] [ng g^{-1}]	Expanded uncertainty[(2)] [ng g^{-1}]
PBDE 47	0.99	0.16
PBDE 154	0.17	0.03
PBDE 209	0.94	0.18

Petroleum hydrocarbons

Analyte	Unit	Mass fraction[(1)]	Expanded uncertainty[(2)]
Unresolved Aliphatics	µg g^{-1}	237	44
Σ *n*-Alkanes [C_{14}-C_{34}]	µg g^{-1}	2.85	0.48
Anthracene	ng g^{-1}	5.07	1.10
Benzo[e]pyrene	ng g^{-1}	20.8	2.8
Indeno[1,2,3-cd]pyrene	ng g^{-1}	23.8	1.2
Acenaphthylene	ng g^{-1}	2.01	0.40

(1) Robust mean of the accepted data sets, each set being obtained by a different laboratory and/or a different method of determination.
(2) Estimated expanded uncertainty with a coverage factor *k*=2, corresponding to a level of confidence of approximately 95%, as defined in the Evaluation of measurement data – Guide to the expression of uncertainty in measurement JCGM100:2008 [1].

Information mass fraction values

(based on dry mass)

Chlorinated pesticides and PCB congeners

Analyte	Mass fraction[(*)] [ng g^{-1}]
op DDE	4.34
op DDT	0.32
Heptachlor Epoxide	0.73
Endrin	4.60
β-Endosulfan	2.60
Endosulfan sulfate	1.95
trans-Nonachlor	0.15
Aroclor 1254	34.3
PCB 8	1.44
PCB 18	0.58
PCB 44	0.40
PCB 49	0.92
PCB 66	0.45

Chlorinated pesticides and PCB congeners (cont.)

Analyte	Mass fraction(*) [ng g^{-1}]
PCB 70	0.65
PCB 87	0.31
PCB 99	1.21
PCB 151	1.54
PCB 156	0.56
PCB 157	0.40
PCB 167	0.34
PCB 189	0.21
PCB 209	0.15

PBDEs

Analyte	Mass fraction(*) [ng g^{-1}]
PBDE 28	0.07
PBDE 66	0.05
PBDE 85	0.11
PBDE 99	0.81
PBDE 153	0.11
PBDE 183	0.09

Petroleum hydrocarbons

Analyte	Unit	Mass fraction(*)
UVF Chrysene eq.	µg g^{-1}	12.1
Resolved aliphatics	µg g^{-1}	20.0
n-C_{18}	ng g^{-1}	232
Pristane	ng g^{-1}	66.7
Phytane	ng g^{-1}	50.7
Total aromatics	µg g^{-1}	5.17
Resolved aromatics	µg g^{-1}	0.55
1-Methylnaphthalene	ng g^{-1}	4.98
1-Methylphenanthrene	ng g^{-1}	5.3
2-Methylphenanthrene	ng g^{-1}	17.6

Petroleum hydrocarbons (cont.)

Analyte	Unit	Mass fraction(*)
Fluorene	ng g^{-1}	2.62
Acenaphthene	ng g^{-1}	2.18

(*) Information values are robust means of the results from at least four laboratories participating in the interlaboratory comparison [2].

Origin and preparation of the material

60 kg of Tumid Venus clams (*Gafrarium tumidum*) were collected in Noumea, New Caledonia. The organisms were dissected and the soft tissues were deep-frozen, freeze dried, ground into powder and sieved through a 250 µm stainless steel sieve.
The sieved biota fraction with a particle size of less than 250 µm was homogenized by mixing it in a stainless steel rotating homogenizer for three weeks. Then, aliquots of about 20 g were packaged in amber glass bottles with aluminum screw caps, labeled IAEA-451 and sealed with Teflon tape.

Characterization study

The IAEA-451 candidate reference material was characterized in an interlaboratory comparison (ILC).
94 laboratories (including the IAEA's Marine Environmental Studies Laboratory, Monaco) from 51 countries reported results.
Participants were requested to analyse chlorinated pesticides, PCBs, PBDEs and petroleum hydrocarbons 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 Environment Laboratories, Monaco, in 2009. The details concerning all reported results as well as the criteria for qualification as a certified, recommended or information value are reported in "World-wide and regional laboratory comparison on the determination of organochlorine compounds, polybrominated diphenyl ethers and petroleum hydrocarbons in IAEA-451 clam (*Gafrarium tumidum*) sample", IAEA/AQ/28, IAEA, Monaco, 2013 [2]. The report may be downloaded free of charge from:
http://nucleus.iaea.org/rpst/ReferenceProducts/ReferenceMaterials/Organic_Contaminants_/index.htm

Based on the evidence on calibrators used, quality control procedures applied by the participating laboratories and their generally high quality performance in previous IAEA interlaboratory comparisons, the Certification Committee decided to accept these assigned values as certified, recommended or information as presented in the Tables above.

Statement on metrological traceability and uncertainty of assigned values

The property values assigned to the IAEA-451 reference material are calculated as mass fractions of chlorinated pesticides, PCBs, PBDEs, aliphatic hydrocarbons and PAHs expressed in the derived SI units µg g^{-1}, mg g^{-1} and ng g^{-1}. Evidence on metrological traceability to the SI Units 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 [2].

Expanded uncertainties with a coverage factor of *k*=2, corresponding to a level of confidence of approximately 95%, were calculated according to JCGM100:2008 Evaluation of measurement data – Guide to the expression of uncertainty in measurement [1].

Intended use

This Certified Reference Material is intended to be used as a quality control material for the assessment of a laboratory's analytical work, for the development and validation of analytical procedures, and for quality assurance within a laboratory in the determination of chlorinated pesticides, PCBs, polybrominated diphenyl ethers and petroleum hydrocarbons in biota samples.

Instructions for use

Homogeneity of the material

The homogeneity of the material was checked by determining the concentration of some representative analytes (chlorinated pesticides, PCBs, polybrominated diphenyl ethers and petroleum hydrocarbons) 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 (5.1 $\pm$ 0.3)%. 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 20 g units. The recommended sample size for analysis is 2 g for petroleum hydrocarbons and 3 g for organochlorine pesticides, PCBs and polybromodiphenyl ethers, 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 **January 2013**. The expiry date is **January 2023**. 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 this IAEA Reference Sheet is in compliance with the ISO Guide 31:2000: Reference materials — Contents 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 CRM IAEA-451, Mass fractions of organochlorine compounds, polybrominated diphenyl ethers and petroleum hydrocarbons in clam (*Gafrarium tumidum*). 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

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: NAEL-MESL.Contact-Point@iaea.org

REFERENCES

[1] JOINT COMMITTEE FOR GUIDES IN METROLOGY (JGCM), Evaluation of Measurement data - Guide to the Expression of Uncertainty in Measurement, JGCM 100:2008 (GUM with minor corrections), (2008). http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf

[2] INTERNATIONAL ATOMIC ENERGY AGENCY, World-wide and regional laboratory comparison on the determination of organochlorine compounds, polybrominated diphenyl ethers and petroleum hydrocarbons in IAEA-451 clam (*Gafrarium tumidum*) sample, IAEA Analytical Quality in Nuclear Applications Series No. 28 (IAEA/AQ/28), IAEA, Vienna (2013) *(in press).*

[3] INTERNATIONAL ORGANIZATION FOR STANDARDIZATION, Reference materials – Contents 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