REFERENCE SHEET

CERTIFIED REFERENCE MATERIAL

IAEA-451

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

Certified mass fraction values (based on dry mass)

<table>
<thead>
<tr>
<th>Analyte</th>
<th>Unit</th>
<th>Certified value$^{(1)}$</th>
<th>Expanded uncertainty$^{(2)}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>EOM</td>
<td>mg g$^{-1}$</td>
<td>42.2</td>
<td>4.4</td>
</tr>
<tr>
<td>HCB</td>
<td>ng g$^{-1}$</td>
<td>0.39</td>
<td>0.04</td>
</tr>
<tr>
<td>Dieldrin</td>
<td>ng g$^{-1}$</td>
<td>1.88</td>
<td>0.16</td>
</tr>
<tr>
<td>α-Chlordane</td>
<td>ng g$^{-1}$</td>
<td>0.56</td>
<td>0.04</td>
</tr>
<tr>
<td>γ-Chlordane</td>
<td>ng g$^{-1}$</td>
<td>0.46</td>
<td>0.13</td>
</tr>
<tr>
<td>Aroclor 1260</td>
<td>ng g$^{-1}$</td>
<td>53.2</td>
<td>4.0</td>
</tr>
</tbody>
</table>
### Certified mass fraction values (based on dry mass)

**PCB congeners**

<table>
<thead>
<tr>
<th>Analyte</th>
<th>Certified value&lt;sup&gt;(1)&lt;/sup&gt; [ng g&lt;sup&gt;-1&lt;/sup&gt;]</th>
<th>Expanded uncertainty&lt;sup&gt;(2)&lt;/sup&gt; [ng g&lt;sup&gt;-1&lt;/sup&gt;]</th>
</tr>
</thead>
<tbody>
<tr>
<td>PCB 28</td>
<td>0.85</td>
<td>0.09</td>
</tr>
<tr>
<td>PCB 95</td>
<td>0.58</td>
<td>0.10</td>
</tr>
<tr>
<td>PCB 101</td>
<td>1.74</td>
<td>0.14</td>
</tr>
<tr>
<td>PCB 105</td>
<td>0.49</td>
<td>0.12</td>
</tr>
<tr>
<td>PCB 110</td>
<td>0.88</td>
<td>0.13</td>
</tr>
<tr>
<td>PCB 118</td>
<td>1.01</td>
<td>0.08</td>
</tr>
<tr>
<td>PCB 128</td>
<td>0.49</td>
<td>0.04</td>
</tr>
<tr>
<td>PCB 138</td>
<td>5.30</td>
<td>0.58</td>
</tr>
<tr>
<td>PCB 149</td>
<td>3.33</td>
<td>0.42</td>
</tr>
<tr>
<td>PCB 153</td>
<td>8.59</td>
<td>0.78</td>
</tr>
<tr>
<td>PCB 170</td>
<td>3.05</td>
<td>0.40</td>
</tr>
<tr>
<td>PCB 174</td>
<td>1.32</td>
<td>0.07</td>
</tr>
<tr>
<td>PCB 177</td>
<td>0.94</td>
<td>0.10</td>
</tr>
<tr>
<td>PCB 180</td>
<td>6.56</td>
<td>1.20</td>
</tr>
<tr>
<td>PCB 183</td>
<td>1.82</td>
<td>0.22</td>
</tr>
<tr>
<td>PCB 187</td>
<td>3.97</td>
<td>0.26</td>
</tr>
<tr>
<td>PCB 194</td>
<td>1.45</td>
<td>0.09</td>
</tr>
<tr>
<td>PCB 206</td>
<td>0.24</td>
<td>0.03</td>
</tr>
</tbody>
</table>

**PBDE**

<table>
<thead>
<tr>
<th>Analyte</th>
<th>Certified value&lt;sup&gt;(1)&lt;/sup&gt; [ng g&lt;sup&gt;-1&lt;/sup&gt;]</th>
<th>Expanded uncertainty&lt;sup&gt;(2)&lt;/sup&gt; [ng g&lt;sup&gt;-1&lt;/sup&gt;]</th>
</tr>
</thead>
<tbody>
<tr>
<td>PBDE 100</td>
<td>0.23</td>
<td>0.04</td>
</tr>
</tbody>
</table>

<sup>(1)</sup> Robust mean of the accepted data sets, each set being obtained by a different laboratory and/or a different method of determination.

<sup>(2)</sup> 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**

<table>
<thead>
<tr>
<th>Analyte</th>
<th>Unit</th>
<th>Certified value&lt;sup&gt;(1)&lt;/sup&gt;</th>
<th>Expanded uncertainty&lt;sup&gt;(2)&lt;/sup&gt;</th>
</tr>
</thead>
<tbody>
<tr>
<td>EOM</td>
<td>mg g&lt;sup&gt;-1&lt;/sup&gt;</td>
<td>36.7</td>
<td>6.4</td>
</tr>
<tr>
<td>Total aliphatics</td>
<td>µg g&lt;sup&gt;-1&lt;/sup&gt;</td>
<td>244</td>
<td>34</td>
</tr>
<tr>
<td>n-C&lt;sub&gt;17&lt;/sub&gt;</td>
<td>ng g&lt;sup&gt;-1&lt;/sup&gt;</td>
<td>373</td>
<td>44</td>
</tr>
<tr>
<td>Naphthalene</td>
<td>ng g&lt;sup&gt;-1&lt;/sup&gt;</td>
<td>14.8</td>
<td>1.2</td>
</tr>
<tr>
<td>Phenanthrene</td>
<td>ng g&lt;sup&gt;-1&lt;/sup&gt;</td>
<td>15.8</td>
<td>5.6</td>
</tr>
<tr>
<td>Chrysene</td>
<td>ng g&lt;sup&gt;-1&lt;/sup&gt;</td>
<td>26.9</td>
<td>2.0</td>
</tr>
<tr>
<td>Fluoranthene</td>
<td>ng g&lt;sup&gt;-1&lt;/sup&gt;</td>
<td>49.3</td>
<td>3.2</td>
</tr>
<tr>
<td>Pyrene</td>
<td>ng g&lt;sup&gt;-1&lt;/sup&gt;</td>
<td>40.0</td>
<td>4.6</td>
</tr>
<tr>
<td>Benzo[b]fluoranthene</td>
<td>ng g&lt;sup&gt;-1&lt;/sup&gt;</td>
<td>35.8</td>
<td>6.2</td>
</tr>
<tr>
<td>Benzo[k]fluoranthene</td>
<td>ng g&lt;sup&gt;-1&lt;/sup&gt;</td>
<td>14.7</td>
<td>3.2</td>
</tr>
<tr>
<td>Benz[a]anthracene</td>
<td>ng g&lt;sup&gt;-1&lt;/sup&gt;</td>
<td>19.2</td>
<td>1.3</td>
</tr>
<tr>
<td>Benzo[a]pyrene</td>
<td>ng g&lt;sup&gt;-1&lt;/sup&gt;</td>
<td>18.2</td>
<td>2.4</td>
</tr>
<tr>
<td>Benzo[g,h,i]perylene</td>
<td>ng g&lt;sup&gt;-1&lt;/sup&gt;</td>
<td>19.5</td>
<td>2.4</td>
</tr>
<tr>
<td>Dibenz[a,h]anthracene</td>
<td>ng g&lt;sup&gt;-1&lt;/sup&gt;</td>
<td>5.32</td>
<td>1.36</td>
</tr>
</tbody>
</table>

Recommended mass fraction values *(based on dry mass)*

**Chlorinated Pesticides and PCBs Congeners**

<table>
<thead>
<tr>
<th>Analyte</th>
<th>Mass fraction&lt;sup&gt;(1)&lt;/sup&gt; [ng g&lt;sup&gt;-1&lt;/sup&gt;]</th>
<th>Expanded uncertainty&lt;sup&gt;(2)&lt;/sup&gt; [ng g&lt;sup&gt;-1&lt;/sup&gt;]</th>
</tr>
</thead>
<tbody>
<tr>
<td>α-HCH</td>
<td>0.78</td>
<td>0.14</td>
</tr>
<tr>
<td>γ-HCH (Lindane)</td>
<td>0.56</td>
<td>0.05</td>
</tr>
<tr>
<td>pp’ DDE</td>
<td>1.73</td>
<td>0.22</td>
</tr>
<tr>
<td>pp’ DDD</td>
<td>0.99</td>
<td>0.22</td>
</tr>
<tr>
<td>pp’ DDT</td>
<td>1.34</td>
<td>0.22</td>
</tr>
<tr>
<td>Heptachlor</td>
<td>2.07</td>
<td>0.22</td>
</tr>
<tr>
<td>Aldrin</td>
<td>0.87</td>
<td>0.10</td>
</tr>
<tr>
<td>α-Endosulfan</td>
<td>1.20</td>
<td>0.20</td>
</tr>
<tr>
<td>PCB 31</td>
<td>0.29</td>
<td>0.02</td>
</tr>
<tr>
<td>PCB 52</td>
<td>0.82</td>
<td>0.04</td>
</tr>
<tr>
<td>PCB 195</td>
<td>0.45</td>
<td>0.03</td>
</tr>
</tbody>
</table>
### PBDEs

<table>
<thead>
<tr>
<th>Analyte</th>
<th>Mass fraction(^{(1)}) [ng g(^{-1})]</th>
<th>Expanded uncertainty(^{(2)}) [ng g(^{-1})]</th>
</tr>
</thead>
<tbody>
<tr>
<td>PBDE 47</td>
<td>0.99</td>
<td>0.16</td>
</tr>
<tr>
<td>PBDE 154</td>
<td>0.17</td>
<td>0.03</td>
</tr>
<tr>
<td>PBDE 209</td>
<td>0.94</td>
<td>0.18</td>
</tr>
</tbody>
</table>

### Petroleum Hydrocarbons

<table>
<thead>
<tr>
<th>Analyte</th>
<th>Unit</th>
<th>Mass fraction(^{(1)}) [µg g(^{-1})]</th>
<th>Expanded uncertainty(^{(2)}) [µg g(^{-1})]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Unresolved Aliphatics</td>
<td>µg g(^{-1})</td>
<td>237</td>
<td>44</td>
</tr>
<tr>
<td>Σ (n)-Alkanes [C(<em>{14})-C(</em>{34})]</td>
<td>µg g(^{-1})</td>
<td>2.85</td>
<td>0.48</td>
</tr>
<tr>
<td>Anthracene</td>
<td>ng g(^{-1})</td>
<td>5.07</td>
<td>1.10</td>
</tr>
<tr>
<td>Benzo[e]pyrene</td>
<td>ng g(^{-1})</td>
<td>20.8</td>
<td>2.8</td>
</tr>
<tr>
<td>Indeno[1,2,3-cd]pyrene</td>
<td>ng g(^{-1})</td>
<td>23.8</td>
<td>1.2</td>
</tr>
<tr>
<td>Acenaphthylene</td>
<td>ng g(^{-1})</td>
<td>2.01</td>
<td>0.40</td>
</tr>
</tbody>
</table>

\(^{(1)}\) 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

<table>
<thead>
<tr>
<th>Analyte</th>
<th>Mass fraction(^{(1)}) [ng g(^{-1})]</th>
</tr>
</thead>
<tbody>
<tr>
<td>(op) DDE</td>
<td>4.34</td>
</tr>
<tr>
<td>(op) DDT</td>
<td>0.32</td>
</tr>
<tr>
<td>Heptachlor Epoxide</td>
<td>0.73</td>
</tr>
<tr>
<td>Endrin</td>
<td>4.60</td>
</tr>
<tr>
<td>(β)-Endosulfan</td>
<td>2.60</td>
</tr>
<tr>
<td>Endosulfan sulfate</td>
<td>1.95</td>
</tr>
<tr>
<td>(trans)-Nonachlor</td>
<td>0.15</td>
</tr>
</tbody>
</table>
### PCBs Congeners

<table>
<thead>
<tr>
<th>Analyte</th>
<th>Mass fraction (ng g⁻¹)</th>
</tr>
</thead>
<tbody>
<tr>
<td>PCB 8</td>
<td>1.44</td>
</tr>
<tr>
<td>PCB 18</td>
<td>0.58</td>
</tr>
<tr>
<td>PCB 44</td>
<td>0.40</td>
</tr>
<tr>
<td>PCB 49</td>
<td>0.92</td>
</tr>
<tr>
<td>PCB 66</td>
<td>0.45</td>
</tr>
<tr>
<td>PCB 70</td>
<td>0.65</td>
</tr>
<tr>
<td>PCB 87</td>
<td>0.31</td>
</tr>
<tr>
<td>PCB 99</td>
<td>1.21</td>
</tr>
<tr>
<td>PCB 151</td>
<td>1.54</td>
</tr>
<tr>
<td>PCB 156</td>
<td>0.56</td>
</tr>
<tr>
<td>PCB 157</td>
<td>0.40</td>
</tr>
<tr>
<td>PCB 167</td>
<td>0.34</td>
</tr>
<tr>
<td>PCB 189</td>
<td>0.21</td>
</tr>
<tr>
<td>PCB 209</td>
<td>0.15</td>
</tr>
<tr>
<td>Aroclor 1254</td>
<td>34.3</td>
</tr>
</tbody>
</table>

### PBDEs

<table>
<thead>
<tr>
<th>Analyte</th>
<th>Mass fraction (ng g⁻¹)</th>
</tr>
</thead>
<tbody>
<tr>
<td>PBDE 28</td>
<td>0.07</td>
</tr>
<tr>
<td>PBDE 66</td>
<td>0.05</td>
</tr>
<tr>
<td>PBDE 85</td>
<td>0.11</td>
</tr>
<tr>
<td>PBDE 99</td>
<td>0.81</td>
</tr>
<tr>
<td>PBDE 153</td>
<td>0.11</td>
</tr>
<tr>
<td>PBDE 183</td>
<td>0.09</td>
</tr>
</tbody>
</table>
Petroleum hydrocarbons

<table>
<thead>
<tr>
<th>Analyte</th>
<th>Unit</th>
<th>Mass fraction(*) [ng g⁻¹]</th>
</tr>
</thead>
<tbody>
<tr>
<td>UVF Chrysene eq.</td>
<td>µg g⁻¹</td>
<td>12.1</td>
</tr>
<tr>
<td>Resolved aliphatics</td>
<td>µg g⁻¹</td>
<td>20.0</td>
</tr>
<tr>
<td>(n)-C₁₈</td>
<td>ng g⁻¹</td>
<td>232</td>
</tr>
<tr>
<td>Pristane</td>
<td>ng g⁻¹</td>
<td>66.7</td>
</tr>
<tr>
<td>Phytane</td>
<td>ng g⁻¹</td>
<td>50.7</td>
</tr>
<tr>
<td>Total aromatics</td>
<td>µg g⁻¹</td>
<td>5.17</td>
</tr>
<tr>
<td>Resolved aromatics</td>
<td>µg g⁻¹</td>
<td>0.55</td>
</tr>
<tr>
<td>1-Methylnaphthalene</td>
<td>ng g⁻¹</td>
<td>4.98</td>
</tr>
<tr>
<td>1-Methylphenanthrene</td>
<td>ng g⁻¹</td>
<td>5.3</td>
</tr>
<tr>
<td>2-Methylphenanthrene</td>
<td>ng g⁻¹</td>
<td>17.6</td>
</tr>
<tr>
<td>Fluorene</td>
<td>ng g⁻¹</td>
<td>2.62</td>
</tr>
<tr>
<td>Acenaphthene</td>
<td>ng g⁻¹</td>
<td>2.18</td>
</tr>
</tbody>
</table>

(*) 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”.

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**

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].

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].

**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±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 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: [https://nucleus.iaea.org/sites/ReferenceMaterials/Pages/IAEA-451.aspx](https://nucleus.iaea.org/sites/ReferenceMaterials/Pages/IAEA-451.aspx)

**Further information:**

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E-mail: mesl@iaea.org
REFERENCES


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- Rev.00.01 (2020-04-30): update of http link to the certificate