### MASS FRACTIONS OF TRACE ELEMENTS IN MARINE SEDIMENT SAMPLE

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

<table>
<thead>
<tr>
<th>Analyte</th>
<th>Certified value(a) (mg kg(^{-1}))</th>
<th>Expanded uncertainty(b) (mg kg(^{-1}))</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ag</td>
<td>1.85</td>
<td>0.39</td>
</tr>
<tr>
<td>Al</td>
<td>82660</td>
<td>3430</td>
</tr>
<tr>
<td>As</td>
<td>10.2</td>
<td>1.0</td>
</tr>
<tr>
<td>Cd</td>
<td>1.09</td>
<td>0.08</td>
</tr>
<tr>
<td>Co</td>
<td>14.7</td>
<td>1.0</td>
</tr>
<tr>
<td>Cr</td>
<td>144</td>
<td>8</td>
</tr>
<tr>
<td>Cu</td>
<td>365</td>
<td>19</td>
</tr>
<tr>
<td>Fe</td>
<td>41450</td>
<td>2240</td>
</tr>
<tr>
<td>Hg</td>
<td>0.143</td>
<td>0.012</td>
</tr>
</tbody>
</table>

\(a\) Information on calculation of certified values  
Certified values are calculated from the accepted data sets, each set being obtained by a different laboratory and/or a different method of determination following ISO Guide 35 ‘Reference materials – General and statistical principles for certification’ \[1\].

\(b\) Information on method used for uncertainty estimation  
The uncertainty is expressed as a combined uncertainty with a coverage factor \(k = 2\) estimated in accordance with the JCGM 100:2008 ‘Evaluation of measurement data – Guide to the expression of uncertainty in measurement’ \[2\] and ISO Guide 35 \[1\].

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**REFERENCES**

1. ISO Guide 35  
2. JCGM 100:2008
Certified mass fraction values (Contd.)
(based on dry mass)

<table>
<thead>
<tr>
<th>Analyte</th>
<th>Certified value(^{(a)}) (mg kg(^{-1}))</th>
<th>Expanded uncertainty(^{(b)}) (mg kg(^{-1}))</th>
</tr>
</thead>
<tbody>
<tr>
<td>Li</td>
<td>64.2</td>
<td>5.5</td>
</tr>
<tr>
<td>Mn</td>
<td>427</td>
<td>30</td>
</tr>
<tr>
<td>Ni</td>
<td>53.1</td>
<td>2.7</td>
</tr>
<tr>
<td>Pb</td>
<td>105</td>
<td>7</td>
</tr>
<tr>
<td>Sn</td>
<td>27.40</td>
<td>1.4</td>
</tr>
<tr>
<td>Sr</td>
<td>137</td>
<td>10</td>
</tr>
<tr>
<td>V</td>
<td>87.4</td>
<td>8.1</td>
</tr>
<tr>
<td>Zn</td>
<td>425</td>
<td>26</td>
</tr>
</tbody>
</table>

\(^{(a)}\) Information on calculation of certified values.
Certified values are calculated from the accepted data sets, each set being obtained by a different laboratory and/or a different method of determination following ISO Guide 35 ‘Reference materials – General and statistical principles for certification’ [1].

\(^{(b)}\) Information on method used for uncertainty estimation.
The uncertainty is expressed as a combined uncertainty with a coverage factor \(k = 2\) estimated in accordance with the JCGM 100:2008 ‘Evaluation of measurement data – Guide to the expression of uncertainty in measurement’ [2] and ISO Guide 35 [1].

Origin and preparation of the material

A sample of 44 kg of sediment was delivered to the IAEA’s Marine Environmental Studies Laboratory (MESL) by the Korean Ocean Research and Development Institute. The freeze dried material was milled to a powder and then sieved through a set of sieves. The fraction of 26 μm was collected and further homogenized for 14 days in a clean atmosphere at a temperature of 20 (+/- 2)°C and relative humidity of 50%. After checking for the homogeneity of the sample material, aliquots of about 20 g were packed into pre-cleaned brown borosilicate glass bottles with polyethylene screw caps and then sealed in plastic bags. The sample material was labeled IAEA-457.

Homogeneity of the material

The between-bottle homogeneity was tested by the determination of the mass fractions of all certified elements (Ag, Al, As, Cd, Cr, Co, Cu, Fe, Hg, Li, Mn, Ni, Pb, Sn, Sr, V and Zn). In total, 10 bottles were selected using random stratified sampling of the whole batch. Three subsamples from each bottle were analyzed for their total element mass fractions. The within-bottle homogeneity was assessed by 15 replicate determinations of mass fractions of trace elements in one bottle. The final measurements were performed by Graphite Furnace Atomic Absorption Spectrometry after microwave acid digestion. The determination of the total mercury was done in solid subsamples with solid mercury analyzer. All analytical procedures used for homogeneity studies were previously validated by MESL, IAEA.

The analysis of homogeneity study was performed under repeatability conditions and in a randomized way in order to separate a potential analytical drift from a trend in the filling sequence and minimize variations. The homogeneity test results provided experimental evidence that satisfactory levels of
between- and within-bottles homogeneity have been attained and the uncertainty due to the between- and within-bottles heterogeneity were within acceptable limits.

**Characterization study**

The values above were established on the basis of results reported to the IAEA-MESL, Monaco, by 21 laboratories from Brazil, Canada, Cuba, Chile, China, Denmark, France, Monaco, Mexico, Peru, Portugal, Romania, Russia, Spain, U.K. and U.S.A.

Laboratories were requested to determine 17 trace elements with one of the following analytical techniques: Atomic-Absorption Spectrometry (Flame or Graphite Furnace), Inductively-Coupled Optical Emission Spectrometry, Inductively-Coupled Mass Spectrometry, Neutron-Activation Analysis, Atomic Fluorescence Spectrometry, X-ray Fluorescence.

**Assignment of values – Certification procedure**

The assigned property values were established on the basis of results reported by participating laboratories to the IAEA-MESL, Monaco. The robust mean concentrations for the sets of individual data were chosen as the best estimate of the property values [3]. Certified values were assigned when at least 5 laboratory means were available, and at least 3 different analytical methods applied.

The details concerning all reported results as well as the criteria for certification can be found in [3]
The report may be downloaded free of charge from:  

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 as presented in the Table above.

**Statement on metrological traceability, commutability and uncertainty of assigned values**

The property values assigned to the IAEA-457 reference material are calculated as mass fractions of specified trace elements, expressed in the derived SI unit mg kg⁻¹. Certified values are SI traceable and evidence on their metrological traceability to the SI units may be found in reference [3].

Expanded uncertainties with a coverage factor of \( k = 2 \), corresponding to a level of confidence of about 95 %, were calculated according to the JCGM 100:2008 Evaluation of measurement data – Guide to the expression of uncertainty in measurement [1]. The measurement uncertainty associated with the certified value is expressed as mg kg⁻¹.

The agreement between results obtained with different analytical methods selected for the IAEA-457 characterization study confirms the absence of any significant method bias and demonstrates commutability of the material for all certified trace elements. More information on commutability of certified values may be found in reference [3].
**Intended use**

This Certified Reference Material is intended to be used for quality assurance and quality control purposes. The IAEA-457 CRM is also suitable for method development and validation of analytical procedures, including potential bias evaluation, and for training purposes.

**Instructions for use**

The IAEA-457 Certified Reference Material is supplied in 20 g units. The material homogeneity is guaranteed if a minimum test portion of 0.2 g is used. To overcome segregation effects due to storage or transportation, the material should be mixed before opening the bottle. All necessary precautions should be taken when opening the bottle to prevent any spread of the fine powder in the laboratory.

**Dry mass determination**

The average moisture content of the material was determined by drying several test portions of 1 g in an oven at 105°C until constant mass (usually 24 hours), and was found to be $(2.5 \pm 0.5)\%$ $(k=2)$. Since the moisture content may vary with ambient humidity and temperature, it is recommended to check it prior to analysis and to report all results on a dry mass basis.

**Handling and storage**

The original unopened bottle should be stored securely at ambient temperature in a dark and dry place. It is recommended to avoid direct exposure to sunlight or to a source of heat.

**Issue and expiry date**

The issue date of this Certified Reference Material is **September 2012**. The material was re-assessed in August 2019 by long-term stability test, confirming that there are no changes in the assigned property values. The expiry date has therefore been extended to **August 2024**. 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 [4].

**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-457,
Mass fractions of trace elements in marine sediment sample, IAEA, Vienna, 5 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-457.aspx

Further information:

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REFERENCES


Revision Information:
- Original reference sheet issue date: 2012-12-11
- Rev.01 (2015-11-19): added statement on commutability; change of expiry date confirmed by long term stability test; references were updated (Certification report IAEA/AQ/26, 2013; ISO Guide31:2015)
- Rev.02 (2019-11-26): change of expiry date confirmed by long term stability test; revision of uncertainty of Sn
- Rev.02.01 (2020-04-27): update of web link to the online catalogue in section ‘Note’