International Atomic Energy Agency  
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IAEA Environment Laboratories  
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REFERENCE SHEET

CERTIFIED REFERENCE MATERIAL  
IAEA-414  
RADIONUCLIDES IN MIXED FISH FROM THE IRISH SEA AND NORTH SEA

<table>
<thead>
<tr>
<th>Radionuclide</th>
<th>Certified value [Bq kg(^{-1})]</th>
<th>95% Confidence interval [Bq kg(^{-1})]</th>
</tr>
</thead>
<tbody>
<tr>
<td>(^{40})K</td>
<td>481</td>
<td>470-486</td>
</tr>
<tr>
<td>(^{137})Cs</td>
<td>5.18</td>
<td>5.12-5.22</td>
</tr>
<tr>
<td>(^{232})Th</td>
<td>0.028</td>
<td>0.025-0.031</td>
</tr>
<tr>
<td>(^{234})U</td>
<td>1.22</td>
<td>1.15-1.26</td>
</tr>
<tr>
<td>(^{235})U</td>
<td>0.050</td>
<td>0.045-0.055</td>
</tr>
<tr>
<td>(^{238})U</td>
<td>1.11</td>
<td>1.07-1.15</td>
</tr>
<tr>
<td>(^{239-240})Pu</td>
<td>0.0230</td>
<td>0.0221-0.0250</td>
</tr>
<tr>
<td>(^{241})Am(^\text{a})</td>
<td>0.120</td>
<td>0.116-0.123</td>
</tr>
<tr>
<td></td>
<td>0.197</td>
<td>0.193-0.204</td>
</tr>
</tbody>
</table>

\(^\text{a}\) The values should be corrected for in-growth from \(^{241}\)Pu

Reference date for decay correction: 1 January 1997
Information values for massic activities

(Based on dry mass)

<table>
<thead>
<tr>
<th>Radionuclide</th>
<th>Information value [Bq kg(^{-1})]</th>
<th>95% Confidence interval [Bq kg(^{-1})]</th>
</tr>
</thead>
<tbody>
<tr>
<td>(^{90})Sr</td>
<td>0.28</td>
<td>0.10-0.54</td>
</tr>
<tr>
<td>(^{210})Pb (^{210})Po(^{\gamma})</td>
<td>2.1</td>
<td>1.8-2.5</td>
</tr>
<tr>
<td>(^{226})Ra</td>
<td>1.4</td>
<td>0.6-1.6</td>
</tr>
<tr>
<td>(^{239})Pu</td>
<td>0.066</td>
<td>0.055-0.073</td>
</tr>
<tr>
<td>(^{240})Pu</td>
<td>0.047</td>
<td>0.046-0.051</td>
</tr>
<tr>
<td>(^{241})Pu</td>
<td>2.0</td>
<td>1.8-2.3</td>
</tr>
</tbody>
</table>

\(^{210}\)Pb and \(^{210}\)Po are considered to be in equilibrium

Reference date for decay correction: 1 January 1997

Origin and preparation of the material

A sample of about 350 kg of mixed fish species was collected in the eastern Irish Sea by the Center for Environment, Fisheries and Aquaculture Science (CEFAS), Lowestoft, U.K., in 1996. The skin was removed, the fish filleted, freeze-dried and subsequently sent to IAEA-MEL for processing. The sample, which was reduced by freeze-drying to about 69 kg, was ground to powder, sieved through a 0.5 mm mesh and homogenized by mixing in a nitrogen atmosphere. The sample was freeze-dried once more, ground and sieved at 250 μm, and a final amount of 10 kg obtained. The sample was then mixed with 100 kg of North Sea fish powder (fraction below 250 μm). To ensure homogenization of the final sample, both samples were mixed by jet pulverizing in a nitrogen atmosphere. The samples were packed into bottles under nitrogen gas, sealed with polyethylene caps and labeled with the code IAEA-414. The bottles containing 100 g of fish powder each were sterilized by gamma ray irradiation with a total dose of 10 kGy using a \(^{60}\)Co source according to ISO 13485:2003 to ensure long-term stability of the material by inhibiting microbial action.

Homogeneity of the material

Sample homogeneity was checked by measuring the activity of \(^{40}\)K, \(^{137}\)Cs, \(^{210}\)Po, \(^{235}\)U, \(^{238}\)Pu, \(^{239}+^{240}\)Pu and \(^{241}\)Am of 16-30 bottles randomly chosen. Gamma spectrometry measurements were performed on 30 to 100 g aliquots. Massic activity of \(^{210}\)Po, \(^{238}\)U, \(^{239}+^{240}\)Pu and \(^{241}\)Am was determined by alpha spectrometry on 0.1 to 20 g aliquots. Homogeneity of these results was checked using one-way analysis of variance. The coefficient of variation was below 10% for radionuclides measured by gamma and alpha spectrometry. The "between samples" variances showed no significant differences from the "within sample" variances for all radionuclides tested.

Characterization study

The IAEA-414 candidate reference material was characterized in an international interlaboratory comparison (ILC) organized between 2000 and 2001. The data received from 90 laboratories were evaluated. Laboratories were requested to determine as many natural and anthropogenic radionuclides as possible by the analytical method of their choice. The following methods were used: gamma-spectrometry, low background gamma-spectrometry, alpha-spectrometry, and beta counting and/or mass spectrometry.

Assignment of values - Certification procedure

The assigned values were established on the basis of results reported by participating laboratories to the IAEA Marine Environment Laboratories in Monaco [1]. The medians for the sets of individual data were chosen as the best estimations of the property values and are reported as certified values when:
at least 5 laboratory means were available, reported from at least 3 different laboratories and the relative uncertainty of the median did not exceed ±5% for activities higher than 100 Bq kg⁻¹, ±10% for activities from 1 to 100 Bq kg⁻¹ and ±20% for activities lower than 1 Bq kg⁻¹.

An activity value was considered as an information value if at least 5 laboratory means calculated from the results of at least 2 different laboratories were available.

The details concerning all reported results as well as the criteria for certification may be found in [1, 2]. The report IAEA/AL/145, IAEA/MEL/73, "Report on the Worldwide Intercomparison IAEA-414 Radionuclides in Mixed Fish from Irish Sea and the North Sea", IAEA, Monaco, 2004 may be downloaded free of charge from: http://nucleus.iaea.org/rpst/Documents/al_145.pdf. All other documents are available upon request.

Evidence on metrological traceability to the SI Units was provided by all laboratories and is summarized in the final report [1].

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 decided to accept these assigned values as certified.

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

The property values assigned to the IAEA-414 Certified Reference Material are calculated as massic activities of each radionuclide, expressed in the derived SI unit Bq kg⁻¹. Measurement uncertainty associated with individual assigned values represents 95% confidence interval of the mean of means.

**Intended use**

This Certified Reference Material may be used for quality assurance/quality control of the analysis of radionuclides in fish samples, for the development and validation of analytical work and for training purposes with nine certified values (⁴⁰K, ¹³⁷Cs, ²³²Th, ²³⁴U, ²³⁵U, ²³⁸U, ²³⁸Pu, ²³⁹±²⁴⁰Pu and ²⁴¹Am).

Based on the above metrological traceability statement, this material is not to be used as a calibrator.

**Instructions for use**

The reference material is supplied in 100g units. The minimum sample mass laboratories should take when using the IAEA-414 is 0.1 g for alpha spectrometry, and 30 g for gamma spectrometry, depending on the radionuclide analyzed.

To overcome potential segregation effects due to storage, the material should be re-homogenized before use.

**Dry mass determination**

The average moisture content of the lyophilized sample after bottling, determined by drying several aliquots in an oven at 80 °C to constant mass (1-2 days), was found to be approximately 3%. Since moisture content can 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 dry place. Analysts are reminded to take appropriate precaution in order to avoid contamination of the material during handling.

**Issue and expiry date**

The issue date of this reference material is **October 2006**. The expiry date is **October 2016**. 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 – 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 IAEA-414, ‘Radionuclides in mixed fish from the Irish Sea and North Sea’. 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: http://nucleus.iaea.org/rpst/ReferenceProducts/ReferenceMaterials

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