

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-385

NATURAL AND ARTFICIAL RADIONUCLIDES IN SEDIMENT **FROM THE IRISH SEA**

<u>Cer</u> i	<u>Certified values for massic activities</u> (Based on dry mass)		
Radionuclide	Certified value	95% Confidence interval	
ional	[Bq kg ⁻¹]	[Bq kg ⁻¹]	Adency
⁴⁰ K	607	604-612	
¹³⁷ Cs	33.0	32.7-33.6	
²²⁶ Ra	21.9	21.6-22.4	
²²⁸ Ra	32.0	31.3-32.5	
²³⁰ Th	30.6	30.0-33.6	
²³² Th	33.7	32.8-33.9	
²³⁴ U	27	26-28	
²³⁸ U	29	28-30	
²³⁸ Pu	0.44	0.42-0.48	
²³⁹⁺²⁴⁰ Pu	2.96	2.89-3.00	

3.84

3.78-4.01

The values should be corrected for in-growth from $^{\rm 241}{\rm Pu}$

²⁴¹Am[#]

Reference date for decay correction: 1 January 1996

Information values for massic activities

Radionuclide	Information value [Bq kg ⁻¹]	95% Confidence interval [Bq kg ⁻¹]	
⁹⁰ Sr	0.58	0.42-0.68	
²¹⁰ Pb (²¹⁰ Po) ^{\$}	32.9	31.2-35.3	
²³⁵ U	1.34	1.25-1.44	
²³⁹ Pu	1.92	1.30-2.07	
²⁴⁰ Pu	1.18	0.97-1.32	
²⁴¹ Pu	30	26-32	

(Based on dry mass)

\$ ²¹⁰Pb and ²¹⁰Po are considered to be in equilibrium

Reference date for decay correction: 1 January 1996

Origin and preparation of the material

A sample of about 250 kg of sediment was collected from the Irish Sea (54.3°N, 3.7°W) by the Centre for Environment, Fisheries and Aquaculture Science (CEFAS), Lowestoft, U.K., in 1995. The sediment, which was sent to IAEA-MEL for processing, was frozen for 2 hours at -40 °C, and then freeze-dried with a +5 °C/h temperature increase rate. Secondary drying was performed over 24 hours under 0.02 mbar pressure at a constant temperature of +40 °C. The sediment was then ground into powder, sieved through a 250 μ m mesh and homogenized by mixing in a nitrogen atmosphere. The samples were bottled in polyethylene flasks under nitrogen gas, sealed with polyethylene caps and labeled with the code IAEA-385. The bottles containing 100 g of sediment powder each were sterilized according to ISO standards at 25 kGy in an irradiation facility.

Characterization study

The IAEA-385 candidate reference material was characterized in an interlaboratory comparison (ILC) with participation of 99 laboratories, including expert laboratories, from Australia, Austria, Bulgaria, Finland, France, Germany, Greece, Hungary, Japan, Morocco, Lithuania, New Zealand, Netherlands, Poland, Portugal, Serbia, Slovenia, Slovakia, South Africa, Spain, Sweden, U.K., U.S.A, and the IAEA Laboratories in Monaco and Seibersdorf.

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. The medians for the sets of individual data were chosen as the best estimations of the property values [1, 2] and are reported as certified values when:

- (i) at least 5 laboratory means were available, reported from at least 3 different laboratories and
- (ii) the relative uncertainty of the median did not exceed $\pm 5\%$ for activities higher than 100 Bq kg⁻¹, $\pm 10\%$ for activities from 1 to 100 Bq kg⁻¹ and $\pm 20\%$ 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 [3, 4]. The report IAEA/AL/151, IAEA/MEL/76, "Report on the Worldwide Intercomparison Exercise IAEA-385: Radionuclides in Irish Sea Sediment", IAEA, Monaco, 2005 may be downloaded free of charge from: <u>http://nucleus.iaea.org/rpst/Documents/IAEA_AL_151.pdf</u>. All other documents are available upon request.

Evidence on metrological traceability to the higher level standards used for calibration (traceability to SI) was provided by all laboratories and is summarized in the final report [3].

Based on the evidence provided, quality control procedures applied by the participating laboratories and their generally high quality performance in the IAEA proficiency tests, 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-385 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 is intended to be used for quality assurance/quality control of the analysis of radionuclides in sediment samples, for the development and validation of analytical methods and for training purposes. Based on the above metrological traceability statement, this material is not to be used as calibrator.

Instructions for use

The reference material is supplied in 100 g units. The minimum sample mass laboratories should take when using the IAEA-385 is 0.1 g for alpha spectrometry, 5 g for mass spectrometry and 10 g for radiometric methods (gamma spectrometry, alpha spectrometry and beta counting), depending on the radionuclide analyzed.

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

Homogeneity of the material

Sample homogeneity was checked by measuring the activity of ⁴⁰K, ¹³⁷Cs, ²¹⁰Po, ²³⁵U, ²³⁸U, ²³⁸Pu, ²³⁹⁺²⁴⁰Pu and ²⁴¹Am in 44 bottles randomly chosen. Gamma spectrometry measurements (for ⁴⁰K, ¹³⁷Cs and ²⁴¹Am) were performed on 10 to 60 g aliquots. Massic activity of ²¹⁰Po, ²³⁵U, ²³⁸U, ²³⁸Pu, ²³⁹⁺²⁴⁰Pu and ²⁴¹Am was determined, prior to radiochemical purification, on 0.1 to 5 g aliquots by alpha or mass spectrometry. Homogeneity of these results was checked using one-way analysis of variance. The coefficient of variation was below 10% for radionuclides measured by gamma spectrometry and 15% for radionuclides measured by alpha or mass spectrometry. The "between samples" variances showed no significant differences from the "within sample" variances for all radionuclides tested.

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 1.14%. 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 a 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 **November 2008**. The expiry date is **November 2018**. 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 [5].

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-385, 'Natural and artificial radionuclides in sediment from the Irish Sea'. IAEA, Vienna, 5 pp. (The latest version published applies; see "Note" below).

<u>Note</u>

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:

Radiometrics Laboratory International Atomic Energy Agency Environment Laboratories 4, Quai Antoine 1er MC 98000 MONACO

ermational Atomic Energy Agency

Tel.: 377 97 97 72 72 Fax: 377 97 97 72 73 E-mail: <u>NAEL-RML.Contact-Point@iaea.org</u>

REFERENCES

[1] POVINEC, P.P., PHAM, M.K., IAEA reference materials for quality assurance of marine radioactivity measurements, J. Radioanal. Nucl. Chem. **248** 1 (2001) 211–216.

[2] SANCHEZ-CABEZA, J.A., PHAM, M.K., POVINEC, P.P., IAEA programme on the quality of marine radioactivity data, J. Env. Radioact. **99** (2008) 1680–1686.

[3] PHAM, M.K., SANCHEZ-CABEZA, J.A., POVINEC, P.P., Report on the Worldwide Intercomparison Exercise IAEA-385: Radionuclides in Irish Sea Sediment, IAEA/AL/151, IAEA/MEL/76, IAEA, Monaco (2005).

[4] PHAM, M.K., SANCHEZ-CABEZA, J.A., POVINEC, P.P., et al., A new Certified Reference Material for radionuclides in Irish Sea sediment (IAEA-385), Appl. Rad. Isot. 66 (2008) 1711–1717.
[5] 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

Wo Kliang

Ms Mai Khanh Pham Project Officer, Radiometrics Laboratory

International Atomic Energy Agency

Atoms for Peace