IARMA Limited
Naver House, Naver Road
Thurso, Caithness, KW14 7QA
United Kingdom
Email: office@iarma.co.uk
www.iarma.co.uk

The International Atomic Reference Material Agency

Certificate

CERTIFIED REFERENCE MATERIAL

IARMA-001

NATURAL AND ANTHROPOGENIC RADIONUCLIDES IN SOIL

Certified Values: Massic Activity (based on dry mass)

Radionuclide*	Certified value[1] [Bq kg^{-1}]	Expanded uncertainty[2] [Bq kg^{-1}]	Number of Accepted Results
K-40	440	30	12
Cs-137	52.0	2.6	12
Tl-208	12.8	1.2	12
Pb-210	60.5	6.8	12
Pb-212	41.3	6.0	12
Pb-214	42.9	4.6	12
Bi-214	42.7	2.0	12
Ra-226	44.1	3.4	12
Ac-228	35.9	1.8	12
Th-234[3]	40.0	6.2	12

[1] *The certified values are the median of all accepted results. The certified values and associated uncertainties are reported on dry mass basis and are traceable to the SI units.*
[2] *Combined expanded uncertainty with a coverage factor k = 2 consists of three components associated with the characterisation, heterogeneity and stability of the material.*
[3] *Information value.*
* *Reference date for decay correction: 01-November-2013*

Issue date of Certificate: 31-March-2014

J. P. Doherty
Chief Executive Officer

A. G. Shakhashiro
Chief Technical Officer

Origin and preparation of the material

The material was collected in a forestry area from the upper 2 cm layer of undisturbed soil.
The bulk material was first air-dried in an oven, then milled and homogenised in one batch using a 300 litre rolling–drum homogeniser in a clean laboratory environment. The homogeneity of the bulk material at all stages was checked prior to further processing or mixing.
Bottling of the material was done under normal laboratory conditions; 400 HDPE bottles were filled in one day taking all precautions to avoid material segregation. The bottles were labelled and sterilized using gamma-ray irradiation with a total dose of 25 kGy using a Co-60 source. The characteristics and quality of the HDPE bottles assures resistance to the radiation dose used during the sterilization process especially for long term storage.

Intended use

Based on well-defined metrological characteristics (metrological traceability of the certified values and associated measurement uncertainty) as well as good physical characteristics (homogeneity and small particle size) IARMA-001 is suitable for quality assurance and quality control purposes and for method development and all aspects of analytical method validation, including potential bias evaluation, and for training purposes. IARMA-001 CRM could also be used as a test item for inter-laboratory comparisons and/or proficiency tests.

Instructions for use

IARMA-001 Certified Reference Material is supplied in 200 g units and the material homogeneity is guaranteed if a minimum test portion of 25 g is used.
When the CRM is used for evaluation of method bias, the method can be considered free of significant bias at a confidence level of 99% if the following criterion is fulfilled:

$$A1 \leq A2 \qquad \text{where: } A1 = \left|\text{Value}_{Cer} - \text{Value}_{Meas}\right|; \qquad A2 = 2.58 \times \sqrt{u_{Cer}^2 + u_{Meas}^2}$$

Value_{Cer} , u_{Cer}: Certified value and its associated standard uncertainty of the studied analyte from the Certificate.

Value_{Meas}, u_{Meas}: Mean value of at least 7 independent measurement results and its associated standard uncertainty of the studied analyte.

Homogeneity test of the material

To assess the homogeneity of the IARMA-001 CRM, 10 bottles covering the whole bottling range were randomly selected. Gamma-ray spectrometry was used to analyse three test portions of approximately 25 g from each. The homogeneity study was performed under repeatability conditions to minimise variations.
The standard uncertainty associated with the between-bottle heterogeneity was calculated using the formulae stated in ISO Guide 35 [1].
The outcome of the homogeneity study, as shown in Table 1, demonstrates that the uncertainties due to between-bottle heterogeneity (u_{bb}) were within the acceptable limits and the material could be considered sufficiently homogeneous for the tested radionuclides at the range of mass used.

Table 1: Results of the Homogeneity Study

Radionuclides	u_{bb} [Bq/kg]	u_{bb} [%]
K-40	4.8	1.1%
Cs-137	0.52	1.0%
Tl-208	0.20	1.6%
Pb-210	2.5	4.1%
Pb-212	0.38	0.9%
Pb-214	0.64	1.5%
Bi-214	0.52	1.2%
Ra-226	1.2	2.7%
Ac-228	0.47	1.3%
Th-234	1.0	2.2%

Dry mass determination

The average moisture content of the material at the time of bottling was determined by drying several test portions of 0.5 g in an oven at 105°C for 24 hours, and was found to be approximately 3.4± 0.3 %. Since the moisture content can vary with ambient humidity and temperature, it is recommended that it should be checked prior to analysis and that all results be reported on a dry mass basis.

Characterisation study

Four expert laboratories participated in the characterisation campaign of the material. The selection of expert laboratories was based on their demonstrated analytical performances through the application of a quality assurance system including method validation and well-established uncertainty budget and on good analytical performance in proficiency tests.

Each laboratory received three bottles of the IARMA-001 along with a technical protocol and a reporting form. The laboratories were requested to perform three independent determinations of the massic activity of the studied radionuclides from each bottle using using a validated analytical method of high resolution gamma spectrometry. In addition, the laboratories were asked to report the measurement result standard uncertainty along with the technical information about the analytical method, calibration procedure, measurement results metrological traceability and the applied quality control mechanism.

The gamma-emitting radionuclides were measured using a radon-tight sample container. The spectrum was collected after 30 days when the Ra-226:Rn-222 equilibrium was reached. A special low background n-type HPGe detector of 30% relative efficiency with a CARBON-EPOXI window was used for the spectrum collection. The shielding was made of 7 cm low background lead and 3 mm of copper.

Assignment of certified values - certification procedure

The certified values of all radionuclides of IARMA-001 were established on the basis of a consensus of all reported results estimated using the median as described in ISO 13528 [2], rounded off to the most significant number of the uncertainty. The arithmetic mean and Algorithm A mean as described in ISO/TS 20612 [3] were also calculated and compared with the median. No significant difference was observed between these location estimators, therefore, the reference value obtained using the median estimator was adopted.

The uncertainty component associated with the characterisation (u_{char}) was estimated using the normalised interquartile range (IQR) as described in ISO 13528 [2]. The normalised IQR is a measure of the variability of the results. It is equal to the interquartile range (IQR) multiplied by a factor (0.7413), which makes it comparable to a standard deviation.

Statement on metrological traceability and uncertainty of assigned values

The certified value assigned to the measurands of IARMA-001 is massic activity, expressed in the derived SI unit Bq kg^{-1} . The certified values were derived from individual results reported by the expert laboratories. Evidence on metrological traceability to the SI Units was provided for all results taken into account for the calculation of the assigned value.

Commutability of the IARMA-001 Certified Reference Material

The user of the IARMA-001 CRM should be aware of the aspect of commutability when using an analytical method other than gamma-ray spectrometry, as applied during the characterisation campaign.

Handling and storage

It is recommended that the original unopened bottle 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 Certificate is 31-March-2014. Based on experience with similar materials and the performed stability study, the certified values for the studied measurands are valid until 31-March-2024, provided the original bottle is handled and stored in accordance with the provided instructions.

Legal disclaimer

IARMA Limited makes no warranties, expressed or implied, with respect to the data contained in this Certificate 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 Certificate is in compliance with the ISO Guide 31:2000, Reference materials - Content of certificates and labels [4].

Note

Certified values as stated in this Certificate may be updated if more information becomes available. Users of the CRM should ensure that the Certificate in their possession is current. The current version can be found on the IARMA Limited website (http://www.iarma.co.uk).
For further information regarding this material, please contact office@iarma.co.uk

REFERENCES

[1] International Organization for Standardization, ISO Guide 35:2006(E), Reference materials – General and statistical principals for certification

[2] International Organization for Standardization, ISO Guide 13528:2005(E), Statistics methods for use in proficiency testing by interlaboratory comparisons.

[3] International Organization for Standardization, ISO/TS 20612:2007, Water quality -- Interlaboratory comparisons for proficiency testing of analytical chemistry laboratories.

[4] International Organization for Standardization, ISO Guide 31: 2000(E), Reference materials - Content of certificates and labels.