IAEA
International Atomic Energy Agency
Atoms for Peace and Development

Certified Reference Material

CERTIFICATE

IAEA-478

RADIONUCLIDES IN AGRICULTURAL SOIL

Certified values for activity concentration

(based on dry mass)

Radionuclide	Certified value [Bq kg^{-1}]	Uncertainty[(a)] [Bq kg^{-1}]	Half-life [1]
^{40}K	374	15	1.2504 (30) ×10^{9} years
^{60}Co	142	2	5.2711 (8) years
^{133}Ba	56.8	0.7	10.539 (6) years
^{134}Cs	112.2	1.4	2.0644 (14) years
^{137}Cs	65.0	0.9	30.05 (8) years
^{210}Pb	485	10	22.23 (12) years
^{241}Am	53.1	0.7	432.6 (6) years

(a) The uncertainty is expressed as a combined standard uncertainty (coverage factor k = 1).

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Information values for activity concentration

(based on dry mass)

Radionuclide	Information value [Bq kg^{-1}]	Uncertainty[(a)] [Bq kg^{-1}]	Half-life [1]
Th-series			
^{232}Th	32.6	0.8	14.02 (6) × 10^9 years
^{228}Ra	32.6	0.8	5.75 (4) years
^{228}Ac	32.6	0.8	6.15 (3) hours
^{228}Th	32.6	0.8	1.9126 (9) years
U-series			
^{238}U	25.0	1.7	4.468 (5) × 10^9 years
^{234}U	25.0	1.7	2.455 (6) × 10^5 years
^{226}Ra	31.2	1.5	1600 (6) years

(a) The uncertainty is expressed as a combined standard uncertainty (coverage factor k = 1)

Reference date for all specified radionuclide decay corrections: 01 January 2018

Origin and preparation of the material

The raw material was collected by Radioanalytical Reference Laboratory (RRL) staff of the Hungarian Food Chain Safety Office on 10 October 2016. The sampling area near Gyömrő, Hungary (E19.22625 N47.23724) is the subject of a long-term monitoring program conducted by the RRL and the soil has one of the lowest radionuclide contents in the region.
The raw material (449 kg) was firstly dried in open air in the laboratory and the macroscopic parts of the vegetation, stones and other non-soil type materials were removed (399.7 kg). After drying (60 °C), the remaining soil mass was 393.8 kg.

The treated soil was milled in a large volume ceramic drum mill using 1:1 soil:milling body ratio for 2 days. The material was then separated into different grain size fractions using a multi-screen vibro-separator with 250 µm, 150 µm and 90 µm sieves (Table 1).

TABLE 1: RESULTS OF SIEVING PROCESS

Grain Size	Mass fraction	
	kg	%
Over 250 µm	-	-
150-250 µm	**201.8**	**51.24**
90-150 µm	146.2	37.13
Less than 90 µm	44.0	11.17
Losses	1.8	0.46

The 150-250 µm grain size fraction (201.8 kg) was selected for reference material and proficiency test purposes. This fraction was homogenised (5 days, large volume/slow speed) and the natural radionuclide content was analysed. The bulk material was transferred to IAEA, where it was spiked with radioisotopes.

The certified reference material (CRM) was designed to be suitable for checking the efficiency function of gamma-ray spectrometers across a wide energy range. The radioisotopes used for spiking (^{60}Co, ^{133}Ba, ^{134}Cs, ^{137}Cs, ^{210}Pb, ^{241}Am) were selected to have reasonably long half-lives to increase the longevity of the CRM for this function. The parameters of radionuclides used for spiking including certificate IDs for traceability are listed in Table 2.

TABLE 2. RADIONUCLIDES USED FOR SPIKING

Nuclide	Main energy, keV	Certificate ID
^{60}Co	1173.24; 1332.51	Co60-ELSB50, CERCA
^{133}Ba	356.01	PTB-6.11-2007-1525
^{134}Cs	604.72; 795.86	PTB-6.11-2008-1119
^{137}Cs	661.67	PTB-6.11-2008-1367
^{210}Pb	46.54	PTB-6.11-2014-1341
^{241}Am	59.54	ER 25 179-19, CMI

One combined master spike solution (MSS) was prepared containing all six (6) radioisotopes, which was added to the raw material. The MSS was measured by gamma-ray spectrometry prior to spiking, confirming the activity of the radionuclides. In addition, all processes were controlled gravimetrically using a high precision (Mettler Toledo XP205) analytical balance. The spiked portion of soil was homogenised, then this was further homogenised with the remaining part of raw material.

Homogeneity of the material

The final homogeneity study was performed using 3 subsamples from each of 11 bottles randomly selected throughout the production batch, which were accurately weighed and measured by gamma-ray spectrometry. The results for all spiked radionuclides were evaluated to determine the contribution of the heterogeneity between and within bottles to the final uncertainty budget and the minimum sample amount for use. The results were satisfactory; there was no production trend indicated, and the between-bottle heterogeneity (1.13%) was below the maximum target value (5%).

Stability

The short-term stability of the material was tested by keeping two bottles at -25 °C and two bottles at +70 °C for a period of two weeks. After the stability test period, 3 sub-samples from each bottle at both conditions were analysed in the same way as described for the final homogeneity study. The results were evaluated against the reference values using Zeta-scores, and demonstrate that the material is stable under the transport conditions listed above.

After 2 years storage, the long-term stability analysis on two identical samples showed the maximum discrepancies between results evaluated by Zeta-scores against the reference values were randomly distributed in range of -1.79 to +2.21 [4]. The long-term stability is assured during the validity period of this certificate (as indicated below), assuming the material is stored as described in the handling and storage section.

Neither the harsh conditions during transportation, nor the long-term storage shows any significant effect to the assigned values and associated uncertainties.

Assignment of values – Certification procedure

The Certified Reference Material was prepared by formulation and values were assigned by calculation, using the specified activity concentration values on the certificates of the high-precision radioactive reference solutions and gravimetrically traceable dilutions. In addition, the raw material contains natural radioactive isotopes (the progenies of U-series, Th-series and ^{40}K) and traces of ^{137}Cs, which were characterized by laboratory measurements, using two independent methods and calibrations.

The Reference Materials (RM) Certification Committee accepted these assigned values as certified or information values as presented in this certificate.

The criteria for certification and details on value assignment can be found in the certification report [4]. The report may be downloaded from:

https://nucleus.iaea.org/sites/ReferenceMaterials/Pages/Index-for-Radionuclides.aspx

Statement on metrological traceability, and uncertainty of assigned values

The property values assigned to the IAEA-478 Certified Reference Material were calculated as activity concentrations, expressed in the derived SI unit Bq kg^{-1}.

The assigned values are traceable to the International System of Units (SI) via the following chain of calibrations:

- Radioactive certified reference solutions were used in the preparation of the master spike solution. Certified activity concentration values are traceable to the SI units Bq and kg via the certificates provided by the metrological institutes.
- Calibrated balances and calibrated control weights were used in the gravimetric preparation of the CRM and for dry weight determination, ensuring an unbroken traceability chain to the SI unit kg.
- Losses during the preparation process were quantified by gamma-ray spectrometry measurements, using detectors calibrated with radioactive certified reference solutions.

The related references of metrological traceability are presented in the certification report of IAEA-478 [4].

The information values were determined by two independent radioanalytical methods in one laboratory, therefore they are traceable to the certificates of the calibration sources. In addition, these values were confirmed by measurements performed in the course of a proficiency test.

The measurement uncertainty associated with the assigned values is expressed as a combined standard uncertainty (coverage factor k = 1) estimated in accordance with ISO Guide 35 [2] and JCGM 100:2008 [3].

Intended use

This certified reference material is intended to be used for quality assurance and quality control purposes and is suitable for method development and validation of analytical procedures. IAEA-478 is not to be used for calibration.

Instructions for use

The IAEA-478 certified reference material is supplied in 250 g units. The material homogeneity is guaranteed if a minimum test portion of 45 g is used for gamma-ray spectrometry. To achieve acceptable counting statistics, it is recommended to measure 100 g in a sealed cylindrical geometry for one day. To analyse ^{222}Rn progenies, it is recommended the material is sealed in a Rn-tight sample container for at least 30 days before measurement to ensure equilibrium. The specified information values for U-isotopes were determined by total digestion or fusion techniques. Other preparation methods may not achieve these results due to the chemical matrix of the material.

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.

The IAEA does not guarantee the stability of the material for repeated sampling from the same RM unit, nor the stability of the material after opening and transfer to new containers.

Dry mass determination

The dry matter content of the soil shall be determined using a drying oven at 105 °C until the mass of the sample is constant (minimum 8 hours). The suggested sample amount for drying is 1-2 g. The average dry matter content of the material was determined by drying several test portions of 1 g in an oven at 105 °C for minimum 8 hours and was found to be 98-99 %. Since the moisture content can vary with ambient humidity and temperature, it is recommended to determine 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. The material should be handled by experienced persons and is for laboratory purposes only. Any remaining material in the opened bottle should be stored in the same conditions, however the stability of the material cannot be guaranteed after initial use.

Issue and expiry date

The issue date of this Certified Reference Material is **September 2023**. The revision number (if any) and issue date of this certificate are provided in the footer of this document. Based on experience with similar materials, the validity of this certificate expires in **December 2031**. 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 certificate and shall not be liable for any damage that may result from the use of such data.

Citation of this certificate

It is suggested to cite this certificate according to the following example, as appropriate to the citation format used: INTERNATIONAL ATOMIC ENERGY AGENCY, Certified Reference Material Certificate IAEA-478, IAEA, Vienna. (The latest version published applies, see “Note” below).

Note

Certified values as stated in this certificate may be updated if more information becomes available. Users of this material should ensure that the certificate in their possession is current. The current version is available on the IAEA's Reference Products website: https://nucleus.iaea.org/sites/ReferenceMaterials/Pages/Index-for-Radionuclides.aspx

Further information

For further information regarding this material, please contact the producer:

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References

[1] BNM – CEA/LABORATOIRE NATIONAL HENRI BECQUEREL, Table de Radionucleides, http://www.lnhb.fr/nuclear-data/nuclear-data-table/ (date accessed 2022-12-15).

[2] INTERNATIONAL ORGANIZATION FOR STANDARDIZATION, Reference materials — Guidance for characterization and assessment of homogeneity and stability, ISO Guide 35:2017, ISO, Geneva (2017).

[3] JOINT COMMITTEE FOR GUIDES IN METROLOGY (JCGM), Evaluation of Measurement data - Guide to the Expression of Uncertainty in Measurement, JCGM 100:2008 (GUM 1995 with minor corrections), (2008). http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (date accessed 2022-12-15).

[4] INTERNATIONAL ATOMIC ENERGY AGENCY, Certification of Activity Concentration of Radionuclides in IAEA-478 Agricultural Soil, IAEA Analytical Quality in Nuclear Applications Series, IAEA, Vienna (in preparation).

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The project officer is responsible for the content of this certificate.
The Chair of the RM Certification Committee approves this certificate and authorizes its release on behalf of the Committee.