

**International Atomic Energy Agency
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IAEA Environment Laboratories**

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REFERENCE SHEET

CERTIFIED REFERENCE MATERIAL

IAEA-448

RADIUM-226 IN SOIL FROM OIL FIELD

Certified values for massic activity

(based on dry mass)

Radionuclide	Certified value [Bq kg ⁻¹]	Uncertainty* [Bq kg ⁻¹]
²²⁶ Ra	19.05 x 10 ³	0.26 x 10 ³

*The uncertainty is expressed as a combined standard uncertainty with a coverage factor $k = 1$ estimated in accordance with the JCGM 100:2008 Evaluation of measurement data – Guide to the expression of uncertainty in measurement [1].

Reference date for decay correction: 01 January 2009

Information values for massic activities

(Based on dry mass)

Radionuclide	Information value* [Bq kg ⁻¹]	Uncertainty** [Bq kg ⁻¹]
⁴⁰ K	234	12
²⁰⁸ Tl	555	26
²¹⁰ Pb	6653	283

²¹² Pb	1623	69
²²⁸ Ac	1166	55
²³² Th	13.4	1.1
²³⁵ U	2.3	0.1
²³⁸ U	49.2	0.9

*As clearly shown from the information values, reported radionuclides part of natural decay series are not in secular equilibrium and therefore decay corrections should be applied using the appropriate Bateman equations. The information values are given at the following date: **08 October 2012**.

**The uncertainty is expressed as a combined standard uncertainty with a coverage factor $k = 1$ estimated in accordance with the JCGM 100:2008 Evaluation of measurement data – Guide to the expression of uncertainty in measurement [1].

Origin and preparation of the material

A sample of two hundred kg of contaminated bulk soil was collected in September 2007 from a Syrian oil field by staff of the Atomic Energy Commission of Syria, an IAEA collaborating centre. Stones and undesired parts were manually removed; the soil was evenly distributed on stainless steel plates in laboratory environment for primary drying at room temperature. Then it was dried in a closed oven at 105 °C for 48 hours and the content of moisture was determined and found to be $(3.4 \pm 0.3) \%$ with a coverage factor $k = 1$.

The material was milled to a powder in a rotary ball mill. The powder was then sieved through a 100 µm sieve. The sieved material, with a particle size of less than 100 µm, was further homogenized by using a plastic rotating drum for 7 days in a clean atmosphere at approximate room temperature of 24 °C and relative humidity of 50 %. To check the level of bulk material homogeneity, three samples from bottom, middle and top of the homogenizer were analyzed using gamma-ray spectrometry. The material mixing was stopped when a relative standard deviation of the three samples less than 4 % was attained.

The IAEA-448 soil CRM was bottled under normal laboratory conditions; 500 bottles were filled in one day. The bottles were labeled, arranged into plastic boxes and sterilized using gamma-ray irradiation with a total dose of 25 kGy using a ⁶⁰Co source.

The bottle is provided with a wide secure-sealed cover to preserve the integrity of the reference material in the bottle.

Homogeneity of the material

The homogeneity of the material was tested for the determination of the massic activity of ²²⁶Ra by analyzing 50 test portions using gamma-ray spectrometry; five test portions of ca. 5 g each were taken from each of 10 bottles randomly selected from the whole bottling range.

The analysis for the homogeneity study was performed under repeatability conditions and in a randomized way to separate potential analytical drifts from a trend in the filling sequence and to minimize variations. The homogeneity test results provided experimental evidence that satisfactory levels of between and within bottle homogeneity were attained, and that the uncertainties due to between and within bottle heterogeneity were within acceptable limits. Thus the material was considered sufficiently homogeneous for the tested radionuclide [2].

Characterization study

The derived certified massic activity of ²²⁶Ra is based on measurement results obtained by six expert laboratories using the gamma-ray spectrometry technique and validated methods with known accuracy. The participating laboratories reported 18 analytical measurement results of ²²⁶Ra accompanied with the combined standard uncertainty calculated in compliance with the JCGM 100:2008 Evaluation of measurement data – Guide to the expression of uncertainty in measurement [1]. The massic activities of ²³²Th, ²³⁵U and ²³⁸U

are based on measurement results obtained by inductively coupled plasma mass spectrometry at the IAEA Terrestrial Environment Laboratory. The massic activities of ^{40}K , ^{208}Tl , ^{210}Pb , ^{212}Pb and ^{228}Ac are based on measurement results obtained by gamma-ray spectrometry at the IAEA Terrestrial Environment Laboratory.

Assignment of values - Certification procedure

The reference value of ^{226}Ra in the IAEA-448 CRM was derived as a consensus of all reported results estimated using the median as described in ISO Guide 35 [3] and ISO 13528 [4]. The arithmetic mean, Algorithm A mean and Hampel mean [5] were also calculated and compared with the median. No significant difference was observed between the median and other mean value estimators.

The reference value obtained using the median estimator was adopted. This value is a reliable estimation of the property value.

In addition, the derived reference value of ^{226}Ra was confirmed by the IAEA Terrestrial Environment Laboratory using alpha spectrometry after radiochemical separation.

The Certification Committee decided to accept the assigned value(s) as certified or information as presented in the Tables above.

The details concerning all reported results as well as the criteria for certification may be found in [2]. The Certification report "Certified reference material IAEA-448: Soil from oil field contaminated with technically enhanced Radium-226", IAEA/AQ/30, IAEA, Vienna, 2013 may be downloaded free of charge from: http://www-pub.iaea.org/MTCD/Publications/PDF/IAEA-AQ-30_web.pdf.

The information values were established by measurements at a single laboratory, namely the IAEA Terrestrial Environment Laboratory in Seibersdorf, Austria.

Statement on metrological traceability and uncertainty of assigned values

The property value of ^{226}Ra assigned to the IAEA-448 Certified Reference Material is calculated as massic activity of ^{226}Ra , expressed in the derived SI unit Bq kg^{-1} . This value was derived from individual results reported by the participating expert laboratories.

The measurement uncertainty associated with the assigned value is expressed as a combined standard uncertainty with a coverage factor $k = 1$ estimated in accordance with the JCGM 100:2008 Evaluation of measurement data – Guide to the expression of uncertainty in measurement [1].

Evidence on metrological traceability to the SI Units was provided for all results considered for the calculation of the assigned value. Pure standard solutions with stated purity and uncertainty were used for calibration in the characterization campaign. In addition, the cross calibration amongst participating laboratories was checked using a standard solution of ^{226}Ra provided to all participants. Consequently the ^{226}Ra certified value derived by this unbroken chain of comparisons is metrologically traceable to SI units.

Intended use

This Certified Reference Material is intended to be used for quality assurance and quality control purposes in the determination of massic activity of ^{226}Ra in soil. The IAEA-448 CRM is also suitable for method development and validation of analytical procedures, including potential bias evaluation, and for training purposes. This material is not to be used as a calibrator.

Instructions for use

The IAEA-448 Certified Reference Material is supplied in 100 g units. The material homogeneity is guaranteed if a minimum test portion of 1 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 soil powder in the laboratory.

Dry mass determination

The average moisture content of the material was determined by drying several test portions of 2 g in an oven at 105 °C for 12 hours, and was found to be (3.7 ± 0.2) % with a coverage factor $k = 1$. Since the 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 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 **March 2013**. Based on experience with similar materials, the reference values for studied elements are valid until **March 2023**. 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 — Content of certificates and labels [6].

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-448, 'Radium-226 in soil from oil field'. 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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