IAEA/AQ/30

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Certified Reference Material IAEA-448: Soil from Oil Field Contaminated with Technically Enhanced Radium-226

IAEA

International Atomic Energy Agency

CERTIFIED REFERENCE MATERIAL IAEA-448: SOIL FROM OIL FIELD CONTAMINATED WITH TECHNICALLY ENHANCED RADIUM-226

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Marketing and Sales Unit, Publishing Section
International Atomic Energy Agency
Vienna International Centre
PO Box 100
1400 Vienna, Austria
fax: +43 1 2600 29302
tel.: +43 1 2600 22417
email: sales.publications@iaea.org
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For further information on this publication, please contact:

Terrestrial Environment Laboratory, Seibersdorf
International Atomic Energy Agency
2444 Seibersdorf
Austria

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FOREWORD

To ensure reliable evaluation of potential radiological hazards and proper decision making related to radiation protection measures, the IAEA, through the IAEA Environment Laboratories, supports Member State laboratories in their efforts to maintain readiness and to improve the quality of analytical results. It does so by producing reference materials, by developing standardized methods for sample collection and analysis, and by conducting interlaboratory comparisons and proficiency tests as tools for external quality control of analytical results.

The problem of naturally occurring radioactive material (NORM) contamination is known to be widespread, occurring in oil and gas production facilities throughout the world. It has become a subject of attention in many IAEA Member States. In response to this radiological concern, facilities in many Member States have been characterizing the nature and extent of NORM in oil and gas installations and in the surrounding environment, evaluating the potential for exposure to workers and the public, and developing methods for properly managing these relatively high massic activity residues.

Within this context, the IAEA Environment Laboratories, in cooperation with the Atomic Energy Commission of Syria, an IAEA Collaborating Centre, have prepared a new certified reference material of soil contaminated with NORM, identified as IAEA-448, certified for the massic activity of ^{226}Ra. This report presents the methodologies used for the production and certification of IAEA-448.

The IAEA officer responsible for this publication was A. Pitois of the IAEA Environment Laboratories.

EDITORIAL NOTE

CONTENTS

1. INTRODUCTION

It is well known that radioactivity contamination in oil and gas production and processing equipment is of natural origin and widespread, occurring throughout the world. This problem is known as Naturally Occurring Radioactive Materials (NORM) contamination, and has become a subject of interest in many IAEA Member States.

The gravity of the problem is not only due to potential serious radiological exposure of workers, but also due to the fact that the NORM could contaminate the environment surrounding oil and gas fields.

Uranium and thorium compounds are mostly insoluble; as oil and gas are brought to the surface, these compounds remain in the underground reservoir. As the natural pressure within the bearing formation falls, formation water present in the reservoir will also be extracted with the oil and gas. Some radium and radium daughter compounds are slightly soluble in water and may become mobilized when this production water is brought to the surface.

The oil and gas production stream passes through a separator where the oil, gas, and water are divided into separate streams based on their different fluid densities. Most of the solids removed in the separator accumulate there. The produced water flows from the separators into storage tanks and is often injected into disposal or recovery wells. ^{226}Ra is generally present in produced water in higher concentrations than ^{228}Ra [1].

In many cases, the produced water is evaporated in lagoons and this leads to soil contamination with Ra isotopes. In addition, produced water leakage also leads to soil contamination in oil fields. ^{226}Ra concentrations in contaminated soil in oil fields can range up to tens of thousands of Bq kg^{-1}.

To meet the IAEA Member States' needs for a certified reference material of soil contaminated with ^{226}Ra from oil fields, the Terrestrial Environment Laboratory prepared IAEA-448 in cooperation with the Atomic Energy Commission of Syria, an IAEA collaborating center.

This report presents the sample preparation methodology, materials, evaluation of certification campaign results, and assignment of property values and their associated uncertainty. The reference value and associated uncertainty for ^{226}Ra in the IAEA-448 soil certified reference material (CRM) was established.

The new IAEA-448 CRM can be used for the development and validation of analytical methods for determination of ^{226}Ra in soil and to ensure the reliability and validity of ^{226}Ra analytical results.

2. METHODOLOGY

2.1. SAMPLING 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 from the Syrian Atomic Energy Commission. Stones and undesired parts were manually removed from the bulk material. The soil was evenly distributed on stainless trays in a laboratory environment for primary drying at room temperature. The soil was further dried in a closed oven at 105 °C for 48 hours and the moisture content was determined to be (3.4 ± 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 as shown in Fig. 1, 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 the 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 ^{60}Co source.

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

The average moisture content of the soil after bottling was determined by drying 5 g overnight at 105 °C.

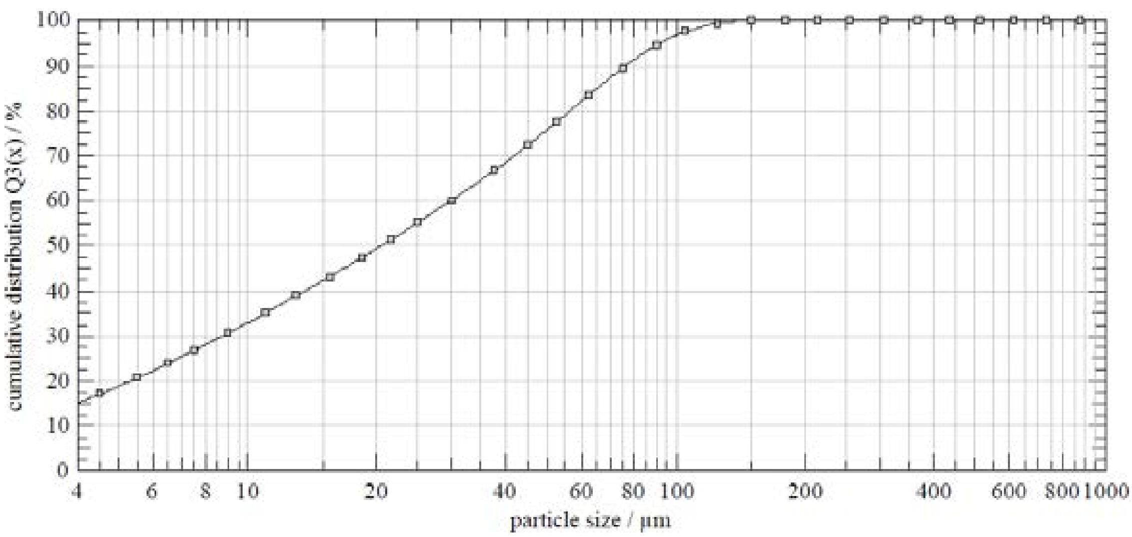

FIG. 1. Cumulative particle size distribution of the IAEA-448 soil CRM.

2.2. HOMOGENEITY STUDY

A comprehensive homogeneity study was carried out on this soil CRM to estimate the uncertainty associated with its heterogeneity. Between-bottle homogeneity was tested by the determination of the massic activity of ^{226}Ra using a validated analytical procedure engaging high resolution gamma ray spectrometry. In total, 10 bottles were randomly selected to cover the whole range of the bottles. Five independent sample portions of 5 g from each bottle were analyzed. 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 collected set of data of ^{226}Ra was subjected to several statistical tests. Beside general descriptive statistics, the following tests were performed:

– Outlier tests (Dixon, Grubbs, Skewness, Kurtosis);
– Directional tests (Skewness, Kurtosis);
– Normality tests (Kolmogorov-Smirnov, Kolmogorov-Smirnov-Lilliefors).

No statistically detected outlying data was observed within the set of reported results. The directional tests pass the acceptance criteria, the Kolmogorov-Smirnov and Kolmogorov-Smirnov-Lilliefors normality tests showed normal distributions of the data set.

The standard uncertainty associated with the material heterogeneity was calculated using the formulae stated in ISO Guide 35 [2]. Single way ANOVA results were used to apply formulae 1 to 3. Table 1 shows the results of the homogeneity study results. The outcome of the homogeneity study demonstrated that the uncertainty due to between-bottle heterogeneity u_{bb} is generally very small and the material can be considered sufficiently homogeneous for the tested radionuclide at the range of mass used.

$$s_{wb}^2 = MS_{within} \tag{1}$$

$$s_{bb}^2 = \frac{MS_{among} - MS_{within}}{n_0} \tag{2}$$

$$u_{bb} = \sqrt{\frac{s_{wb}^2}{n_{bot} \cdot n} + \frac{s_{bb}^2}{n_{bot}}} \tag{3}$$

Where:

MS_{among}	Mean square (ANOVA) between bottles
MS_{within}	Mean square (ANOVA) within bottles
n	Number of observations
n_{bot}	Number of bottles
n_0	(Effective) number of (sub) group members
s_{bb}^2	Variance between bottles

s_{wb}^2	Variance within bottles
u_{bb}	Uncertainty associated with the between bottle heterogeneity

TABLE 1. HETEROGENEITY UNCERTAINTY EXPRESSED IN Bq kg^{-1} AND IN PERCENTAGE OF THE REFERENCE VALUE

Heterogeneity standard uncertainty	(Bq kg^{-1})	(%)
u_{bb}	190	1.0

2.3. CHARACTERIZATION CAMPAIGN

Six expert laboratories participated in the characterization campaign of the IAEA-448. The list of participating laboratories is reported in Appendix.

The selection of expert laboratories was based on their demonstrated analytical performances through application of a quality assurance system including method validation and established uncertainty budget, and on good analytical performances in proficiency tests.

Each laboratory received one bottle of the IAEA-448 and one ampoule of a calibrated standard solution of ^{226}Ra. Laboratories using gamma ray spectrometry were requested to measure ^{226}Ra (186.2 keV line) and its short-lived daughters at secular equilibrium, namely ^{214}Pb (351.9 keV line) and ^{214}Bi (609.3 keV line). In addition, the participating 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 applied quality control mechanism. The reported ^{226}Ra measurement results of the standard solution were used to validate the calibration of the system and the observed variations between the reported results were found to be within the reported measurement results uncertainty.

The participating laboratories reported analytical measurement results of ^{226}Ra accompanied with the combined standard uncertainty calculated in compliance with the Evaluation of measurement data - Guide to the expression of uncertainty in measurement, JGCM 100:2008 [3]. The measurement result uncertainty budget using gamma ray spectrometry contained the following components: sample and background counts, detector efficiency, dry mass determination, self-absorption and other corrections, nuclear data and method reproducibility.

The reported analytical results and associated uncertainties in addition to a summary of the analytical procedure applied are reported in Table 2.

All participating laboratories claimed metrological traceability of provided results to the International System of Units (SI) through standard calibration sources. They used in addition certified reference materials as part of their quality control programme.

TABLE 2. RADIUM-226: MEASUREMENT RESULTS IN Bq kg^{-1} AS REPORTED BY PARTICIPANTS IN THE IAEA-448 CHARACTERIZATION

Laboratory	Energy (keV)	Massic activity (Bq kg^{-1})	Combined standard uncertainty (u) (Bq kg^{-1})	Expanded uncertainty (U) k = 2 (Bq kg^{-1})	Analytical method
01	186.2	21450	316	632	A steel radon tight sample container was used to count the soil in cylindrical geometry. Gamma ray spectrometry system with HPGe detector P type, 80% efficiency was used. Calibration carried out using ^{226}Ra standard solution NIST SRM 4966A traceable to SI units.
	351.9	19783	207	414	
	609.3	20075	207	414	
02	186.2	19100	950	1900	Aluminum radon tight sample container was used. Gamma ray spectrometry system: HPGe detector P type with 40% relative efficiency. Efficiency calibration was performed using diluted ^{226}Ra standard solution.
	351.9	17300	690	1380	
	609.3	16600	830	1660	
03	186.2	18634	428	856	A steel radon tight sample container was used to count the soil, with HPGe detector, P-type, 30% Relative Efficiency. Calibration carried out using ^{226}Ra standard solution traceable to SI units through ENEA primary ^{226}Ra standard. Peak area evaluated by Canberra Genie 2K.
	351.9	19485	1222	2444	
	609.3	18994	713	1426	
04	186.2	19349	618	1236	Sample container made of Teflon 2.2 g/cm^{-3}. Geometry: cylindrical. HPGe detector, P-type, 40 % Relative Efficiency, Coaxial was used. Calibration carried out using ^{226}Ra standard solution traceable to SI units. Peak area evaluated by Ortec γ−Vision.
	351.9	20253	717	1434	
	609.3	19779	595	1190	

Laboratory	Energy (keV)	Massic activity (Bq kg^{-1})	Combined standard uncertainty (u) (Bq kg^{-1})	Expanded uncertainty (U) k = 2 (Bq kg^{-1})	Analytical method
05	186.2	19000	400	800	The sample was pressed into a polyethylene canister with diameter of 90 mm, up to the height of 13 mm, covered by a plastic plate, which was glued to the canister walls to stabilize the sample geometry. Efficiency calibration was done using a standard solution 9ML01ELMH05 from CERCA.
	351.9	17800	500	1000	
	609.3	17700	300	600	
06	186.2	18900	700	1400	Aluminum radon tight sample container was used. Gamma system: HPGe detector P type (80% relative efficiency, efficiency calibration was performed using diluted QCY48 CRM. Gamma analysis software: Interwinner V. 4.1.
	351.9	18600	500	1000	
	609.3	20172	600	1200	

2.4. ASSIGNMENT OF CERTIFIED PROPERTY VALUE AND ASSOCIATED UNCERTAINTY

The collected set of data of ^{226}Ra in the characterization campaign reported in Table 2 was subjected to several statistical tests as applied in 2.2. The statistical tests showed that there is no outlying data within the set of reported results. The directional tests passed the acceptance criteria, the Kolmogorov-Smirnov and Kolmogorov-Smirnov-Lilliefors normality tests showed normal distributions of the data set.

The reported measurement results were first checked for compliance with the characterization requirements (reported measurement result uncertainty, technical information, and demonstrated metrological traceability) and for correctness of the system calibration based on reported values of the standard solution provided to the participants. All reported results for the standard solution were within ± 3 %, therefore it could be concluded that the participating laboratories demonstrated a good agreement regarding cross-calibration.

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 13528 [4]. The arithmetic mean, Algorithm A mean and Hampel mean as described in ISO/TS 20612 [5] were also calculated and compared with the median. No significant difference was observed as shown in Table 3, therefore, the reference value obtained using the median estimator was adopted. This value is a reliable estimation of the property value.

TABLE 3. ESTIMATED CONSENSUS VALUE OF ^{226}Ra USING DIFFERENT STATISTICAL ESTIMATORS

Statistical estimator	Estimated mean value of ^{226}Ra (Bq kg^{-1})
Arithmetic mean	19054
Median (assigned reference value)	19050
Algorithm A mean	19057
Hampel mean	19064

The derived reference value was confirmed using a radiochemical procedure with a test portion of 0.5 g. In this procedure ^{226}Ra was determined by isotope dilution alpha spectrometry. The samples were spiked with ^{229}Th tracer in equilibrium with its ^{225}Ra daughter. The samples were melted and dissolved using Li-methaborate fusion. Then Ra was pre-concentrated by co-precipitation with $PbSO_4$. The precipitates were dissolved and alpha spectrometry sources were made by micro co-precipitation with $BaSO_4$. The sources were measured when the ^{217}At decay product of ^{225}Ra reached its maximum activity. The average massic activity of four independent test portions taken from four different bottles was (20080 ± 600) Bq kg^{-1} which is comparable within 2 σ with the reference value.

According to the ISO Guide 35 [2] the combined uncertainty associated with the reference value consists of uncertainties related to characterization u_{char}, between-bottle heterogeneity

(u_{bb}) and long–term stability (u_{stab}). These different uncertainty components were estimated and propagated to estimate the combined standard uncertainty of the reference value of ^{226}Ra.

$$u^2_{CRM} = u^2_{char} + u^2_{bb} + u^2_{stab} \tag{5}$$

The uncertainty component associated with the characterization (u_{char}) was estimated using the approach described by Pauwels et al. [6]. In this approach, the uncertainty of characterization is separated into laboratory dependent uncertainty u(I), uncertainty common to all laboratories u(II) and uncertainty common to groups of laboratories u(III).

$$u(I) = \frac{\sqrt{\sum_1^n u_i^2}}{n} \tag{6}$$

where u_i is the combined measurement result standard uncertainty reported by each laboratory and n is the number of reported results. For this case u (II) and u (III) were set to zero.

The calculated uncertainty component associated with the characterization (u_{char}) according to this approach was 150 Bq kg^{-1}.

Based on the technical expertise of such a material and analyte and known information about the material history, the u_{stab} uncertainty component associated stability during storage period was chosen as 0.5 % of the reference value, which guarantees the validity of the certificate for at least 10 years. In addition, the material stability will be monitored and if any significant variations are observed, the customers will be informed.

Figure 2 presents the reported results by the six expert laboratories for ^{226}Ra massic activity (Bq kg^{-1}) in the IAEA-448 soil sample. The reference value expressed as a median (red solid line) and corresponding expanded uncertainty ($k=2$) (blue dashed line) are shown. The error bars correspond to the expanded uncertainty ($k=2$) as reported by the participating laboratories.

An example of a typical gamma ray spectrum of IAEA-448 is shown in Fig. 3.

The assigned reference value of ^{226}Ra and associated standard uncertainty are shown in Table 4.

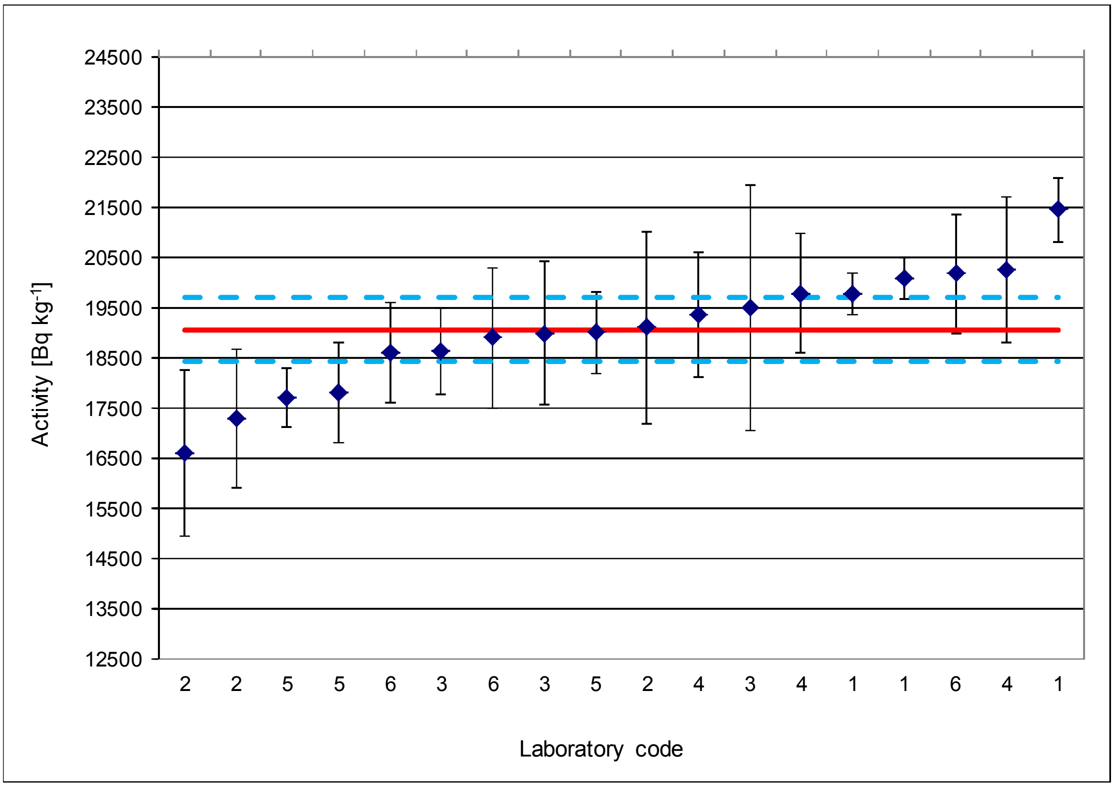

FIG. 2. Reported results for ^{226}Ra *massic activity (Bq kg^{-1}) in IAEA-448 soil sample plotted against the certified reference value expressed as a median (red solid line) and corresponding expanded uncertainty (k=2) (blue dashed line). The vertical error bars correspond to the expanded uncertainty reported by each laboratory.*

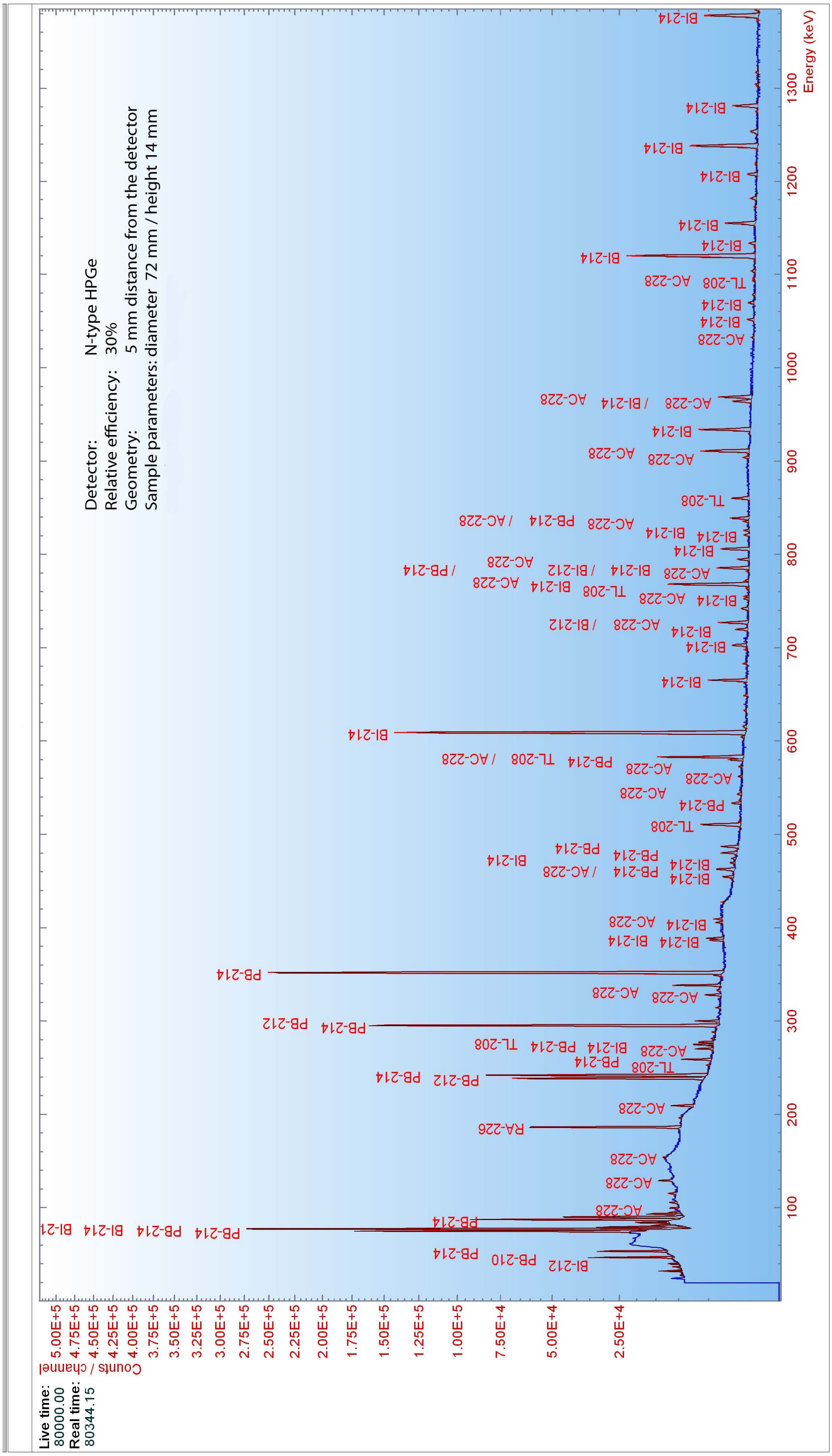

FIG. 3. Gamma ray spectrum of the IAEA-448 CRM

TABLE 4. DERIVED CERTIFIED REFERENCE VALUE OF ^{226}Ra AND ASSOCIATED COMBINED STANDARD UNCERTAINTY

Radionuclide	Standard uncertainty associated with characterization u_{char}	Standard uncertainty associated with material heterogeneity u_{bb}	Standard uncertainty associated with material stability u_{stab}	Certified reference value	Combined standard uncertainty u
			Bq kg^{-1}		
^{226}Ra	150	190	95	19050	260

The reference date for decay correction is 01 January 2009. The certified value is reported on dry mass basis.

2.5. MOISTURE CONTENT DETERMINATION

The average moisture content of the material was determined by drying several test portions of 2 g in an oven at 105 ºC 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.

2.6. METROLOGICAL TRACEABILITY AND COMMUTABILITY

The quantity value assigned to the certified reference material is 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. Evidence on metrological traceability to the SI units was provided for all results taken into account for the calculation of the assigned value.

Pure standard solutions with stated purity and uncertainty were employed 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 through this unbroken chain of comparisons with defined uncertainties is metrologically traceable to SI units.

2.7. INTENDED USE

Based on well-defined metrological characteristics — metrological traceability of assigned reference value and associated measurement uncertainty — as well as good physical characteristics — homogeneity and small particle size — this certified reference material is suitable for quality assurance and quality control purposes in determination of massic activity of ^{226}Ra in soil. The IAEA-448 CRM is also suitable for method development and all aspects of analytical method validation, including potential bias evaluation, and for training purposes.

2.8. 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.

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.

The issue date of this Certified Reference Material is March 2013. Based on experience with similar materials, the reference value for studied radionuclide is valid until March 2023, provided the original bottle is handled and stored in accordance with the provided instructions.

The IAEA makes no warranties, expressed or implied, with respect to the data contained in this report and shall not be liable for any damage that may result from the use of such data.

APPENDIX

List of participating laboratories in the characterization campaign of the IAEA-448 (in alphabetical order)

GERMANY

Möbius, S.	FTU, Research Center Karlsruhe Agricultural Office, Postfach 3640, D-76021 Karlsruhe, Germany

HUNGARY

Tarjan, S.	Radioanalytical Reference Laboratory, Food and Feed Safety Directorate of the Hungarian Agricultural Office, Fogoly u. 13-15, Budapest, Hungary

ITALY

Rusconi, R.	ARPA Lombardia, Dipartimento di Milano, Via Juvara 22, Milano - 20129, Italy

ITALY

De Felice, P.	INMRI, ENEA, Via Anguillarese 301, P.O. BOX 2400, Rome - 100100, Italy

SLOVENIA

Korun, M.	Laboratory for Radiation Measuring Systems and Radioactivity Measurements, Jozef Stefan Institute, Jamova, 39, Ljubljana 1000, Slovenia

SYRIAN ARAB REPUBLIC

Al-Masri, M.

Gamma Spectrometry Laboratory,
Environment Protection Division,
Atomic Energy Commission of Syria,
Kafar Sousah, 17 Nissan street,
Damascus, P.O.Box 6091000,
Syrian Arab Republic

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CONTRIBUTORS TO DRAFTING AND REVIEW

A. Ceccatelli	International Atomic Energy Agency
G. Kis-Benedek	International Atomic Energy Agency
A. Pitois	International Atomic Energy Agency
A. Shakhashiro	International Atomic Energy Agency

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Telephone: +358 9 121 41 • Fax: +358 9 121 4450
Email: akatilaus@akateeminen.com • Web site: http://www.akateeminen.com

FRANCE
Form-Edit, 5, rue Janssen, P.O. Box 25, F-75921 Paris Cedex 19
Telephone: +33 1 42 01 49 49 • Fax: +33 1 42 01 90 90
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