IAEA/AQ/21

IAEA Analytical Quality in Nuclear Applications Series No. 21

Certified Reference Materials for Radioactivity Measurements in Environmental Samples of Soil and Water: IAEA-444 and IAEA-445

IAEA

International Atomic Energy Agency

Certified Reference Materials for Radioactivity Measurements in Environmental Samples of Soil and Water: IAEA-444 and IAEA-445

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Certified Reference Materials for Radioactivity Measurements in Environmental Samples of Soil and Water: IAEA-444 and IAEA-445

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FOREWORD

Reference Materials are an important requirement for any sort of quantitative chemical and radiochemical analysis. Laboratories need them for calibration and quality control throughout their analytical work.

The IAEA started to produce reference materials in the early 1960's to meet the needs of the analytical laboratories in its Member States that required reference materials for quality control of their measurements. The initial efforts were focused on the preparation of environmental reference materials containing anthropogenic radionuclides for use by those laboratories employing nuclear analytical techniques. These reference materials were characterized for their radionuclide content through interlaboratory comparison involving a core group of some 10 to 20 specialist laboratories. The success of these early exercises led the IAEA to extend its activities to encompass both terrestrial and marine reference materials containing primordial radionuclides and trace elements.

Within the frame of IAEA activities in production and certification of reference materials, this report describes the certification of the IAEA-444 and IAEA-445: soil and water spiked with gamma emitting radionuclides respectively. Details are given on methodologies and data evaluation.

The IAEA officer responsible for this publication was A. Shakhashiro of the IAEA Environment Laboratories, Department of Nuclear Sciences and Applications.

EDITORIAL NOTE

CONTENTS

1. INTRODUCTION

The IAEA Environment Laboratories in Seibersdorf (Austria) has the programmatic responsibility to provide assistance to the IAEA Member State laboratories in maintaining and improving the reliability of analytical measurement results, both in trace element and radionuclide determinations. This is accomplished through the provision of reference materials of terrestrial origin, validated analytical procedures, training, and through the evaluation of measurement performance by organization of worldwide and regional proficiency tests.

Environmental matrices, such as soil and water, are examples of environmental samples that have been widely analyzed by laboratories and regulatory bodies to monitor and evaluate radiological contamination. The second pillar supporting reliability of radiological measurements is the availability of appropriate reference materials for calibration, analytical quality control and method validation. The IAEA-444 and IAEA-445 reference materials (RMs) were prepared for this aim.

Certified values for the massic activities and associated standard uncertainties could be established for: Mn-54, Co-60, Zn-65, Cd-109, Cs-134, Cs-137, Pb-210 and Am-241.

During production and certification of the IAEA-444 and IAEA-445, the requirements for reference materials production and certification as stated in ISO guides 34 and 35 [1, 2] were taken into account. This report summarizes the preparation and certification process.

The RMs were prepared by spiking soil and water with known quantity of standards solutions of gamma emitting radionuclides. Full details of the spiking solutions and procedure used are discussed later.

The property values of the spiked RMs were verified by analysing representative samples in the gamma spectroscopy laboratory of the IAEA Environment Laboratories in Seibersdorf, Austria. In addition, the IAEA-444 and IAEA-445 CRMs were used as test items in the world wide proficiency test organised in 2007-2008 by the IAEA [3].

2. MATERIALS AND METHODS

2.1. Preparation of the IAEA-444: Soil spiked with gamma emitting radionuclides

A soil from China was used to prepare the IAEA-444 soil certified reference material (CRM) spiked with gamma emitting radionuclides.

Before using the soil for spiking, it was milled and sieved to collect the appropriate fraction at mesh size less than 0.3 mm, and then homogenised.

The matrix of Chinese soil was characterized and a number of samples were pre-screened for the radionuclides of interest prior to spiking. The results have shown that the material is free from man-made radionuclides, except for Cs-137, which was present at 2.6±0.2 Bq kg^{-1} based on dry mass. (Ref. date: 2006-01-01). The moisture content found to be 2.3±0.2 %.

The preparation of the spiked soil CRM was performed according to a validated procedure [4]. According to this procedure a mixed gamma-emitting radionuclide solution was prepared by taking a precise amount of a calibrated standard solution of Mn-54, Co-60, Zn-65, Cd-109, Cs-134, Cs-137, Pb-210 and Am-241 as listed in Table 1, with the appropriate carriers, and diluting to furnish target radionuclide activities in the soil. An aliquot of 0.502±0.003 g mixed gamma-emitting radionuclide solution was mixed with 100 mL methanol and added to processed 200.00± 0.01 g soil portions previously weighed in polypropylene bottles. The slurry formed was thoroughly mixed, the mixer was washed with methanol inside the bottle, and the soil portions were then dried in an oven at 40 °C overnight. The samples were left to cool to room temperature and homogenized in a Turbula mixer for 30 min. Each bottle was spiked separately using a calibrated pipette with known uncertainty. After spiking, the material was tested for homogeneity. In total, 100 bottles of 200 grams each were prepared.

It should be noted that the IAEA-444 soil reference material was not spiked with Pb-210, the Pb-210 is naturally present in the IAEA-444.

The reference values of the massic activities of each nuclide and its associated standard uncertainty were calculated from the certified activity value of each standard solution used for spiking the soil, taking into account the successive dilution steps, the mass of spiking solutions and the dry mass of the soil being spiked. Table 1 lists the standard sources used for spiking. The components of uncertainties taken into calculation of the combined standard uncertainty of the certified massic activities are shown in Table 2. It can be seen that the uncertainty of the spike amount and the uncertainty associated with between bottles heterogeneity are the dominant two factors.

TABLE 1: IDENTIFICATION OF THE CERTIFIED SOURCES OF EACH RADIONUCLIDE USED IN SPIKING THE SOIL AND WATER CRMs

Nuclide	Source manufacturer and batch number
Mn-54	AMERSHAM: MFZ64; NO S3/28/12
Co-60	CERCA-LEA FRAMATOME: CO60-ELSB50; NO 72452
Zn-65	CERCA-LEA FRAMATOME: ZN65-ELSB50; NO 7020
Cd-109	AMERSHAM: CUZ64;NO S3/36/23
Cs-134	CERCA-LEA FRAMATOME: CS134-ELSB50; NO 70823
Cs-137	AMERSHAM: CDZ64; NO S4/14/70
Pb-210	SRM 4337, NIST, Reference date 15-06-2007.
Am-241	CERCA-LEA FRAMATOME: AM241-ELSB30; NO 5104

TABLE 2: COMPONENTS OF UNCERTAINTIES OF PREPARATION, CERTIFIED REFERENCE MASSIC ACTIVITIES AND STANDARD UNCERTAINTIES OF IAEA-444

Radio-nuclide	Uncertainty of the spike amount	Uncertainty associated with between bottles heterogeneity	Uncertainty associated with dry mass determination	Uncertainty associated with the mass	Certified Reference massic activities	Combined standard uncertainty
	%	%	%	%	**Bq kg^{-1} dry mass**	
Mn-54	0.34	2.00	0.2	0.035	**61.0**	**1.3**
Co-60	0.77	2.30	0.2	0.035	**82.6**	**2.0**
Zn-65	1.04	3.10	0.2	0.035	**29.9**	**1.0**
Cd-109	0.53	4.70	0.2	0.035	**248.7**	**5.2**
Cs-134	0.78	2.80	0.2	0.035	**59.4**	**1.7**
Cs-137	0.99	1.80	0.2	0.035	**68.5**	**1.4**
*Pb-210		3.40			**48.0**	**1.6**
Am-241	0.63	2.80	0.2	0.035	**55.6**	**1.6**

The combined uncertainty is expressed as 1σ (k=1)
The reference date for all certified massic activities is 15 October 2007.
** Information value, naturally present and not added during the spiking procedure.*

The information property value of Pb-210 was established based on a consensus value derived from measurement results of 10 test portions measured using a broad energy type Ge detector model BE2825 (Canberra); energy resolutions were 0.670 and 1.78 keV at 122 keV and 1332 keV gamma-rays, respectively. The obtained consensus value from measurement results was comparable to the median and median of absolute differences (MAD) (48.9 ± 1.7) calculated from 172 measurement results reported in the IAEA-CU-03-2007 world wide proficiency test

[3]. This value of Pb-210 should not be used for calibration purposes, it could only be used for quality control purposes and method validation.

2.1.1. Homogeneity test of the IAEA 444

To test the homogeneity and to verify the reference values of the measurands of the spiked soil IAEA-444 CRM, 4 bottles were randomly selected and three test portions at 50 g from each bottle were measured by three different gamma-ray spectrometers.

The first detector was a n-type coaxial Ge detector (OX) model GR3019 (Canberra); of 35% efficiency relative to 3”x3” of NaI(Tl) scintillation detector and resolution of 1.85 keV at 1.332 keV.

The second detector was a broad energy type Ge detector (BE) model BE2825 (Canberra); energy resolutions were 0.670 and 1.78 keV at 122 keV and 1332 keV gamma-rays, respectively.

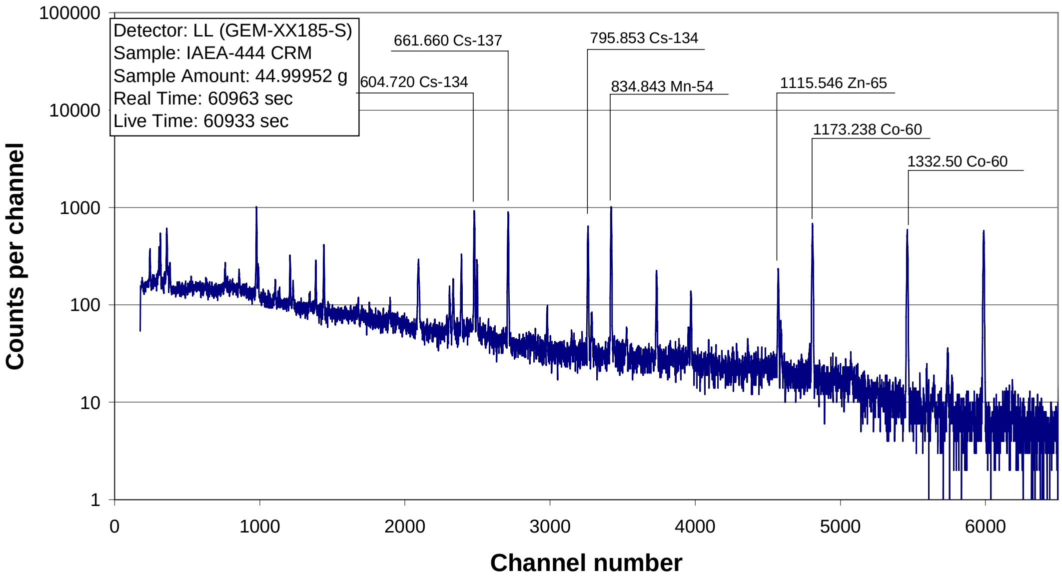
The third one was a p-type coaxial Ge detector (LL) model GEM-XX185-S (Ortec); of 60% efficiency relative to 3”x3” of NaI(Tl) scintillation detector and resolution of 1.81 keV at 1.332 keV.

The detectors were mounted in a 10 cm thick lead shield. Canberra digital electronics and GENIE-2000 software were used. The measurements were performed at the Chemistry Unit of Seibersdorf laboratories. The OX system was used for measurement of whole energy region of gamma-rays, and BE and LL systems were used for low and high energy regions of gamma-rays, respectively. Figures 1 to 4 present examples of the obtained spectra.

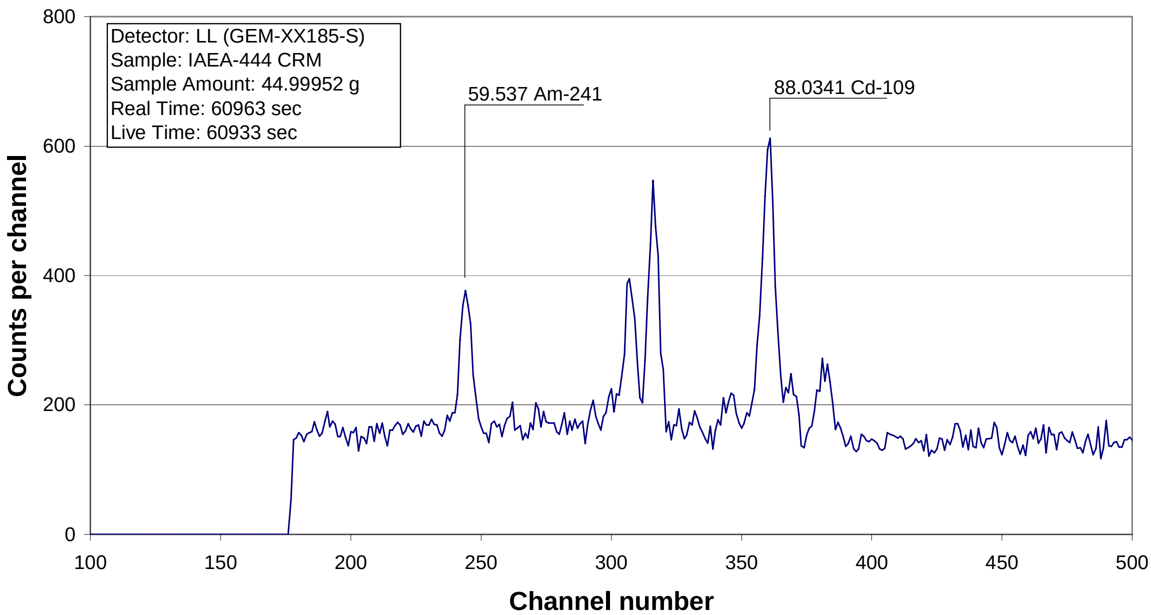
The measurement results of the homogeneity test (Figures 5–12) showed that there is no significant difference between the method relative standard deviation and the between bottles relative standard deviation for the 12 measured test portions, which indicates satisfactory homogeneity of the material.

To control the quality of the spiked soil, all prepared bottles were measured for 900 seconds and the spectra were measured at the IAEA Terrestrial Environment Laboratory using a high resolution gamma spectrometer to check for any inconsistency or an outlier. Figures 13 and 14 show an example of the obtained quality control charts of the measurement results.

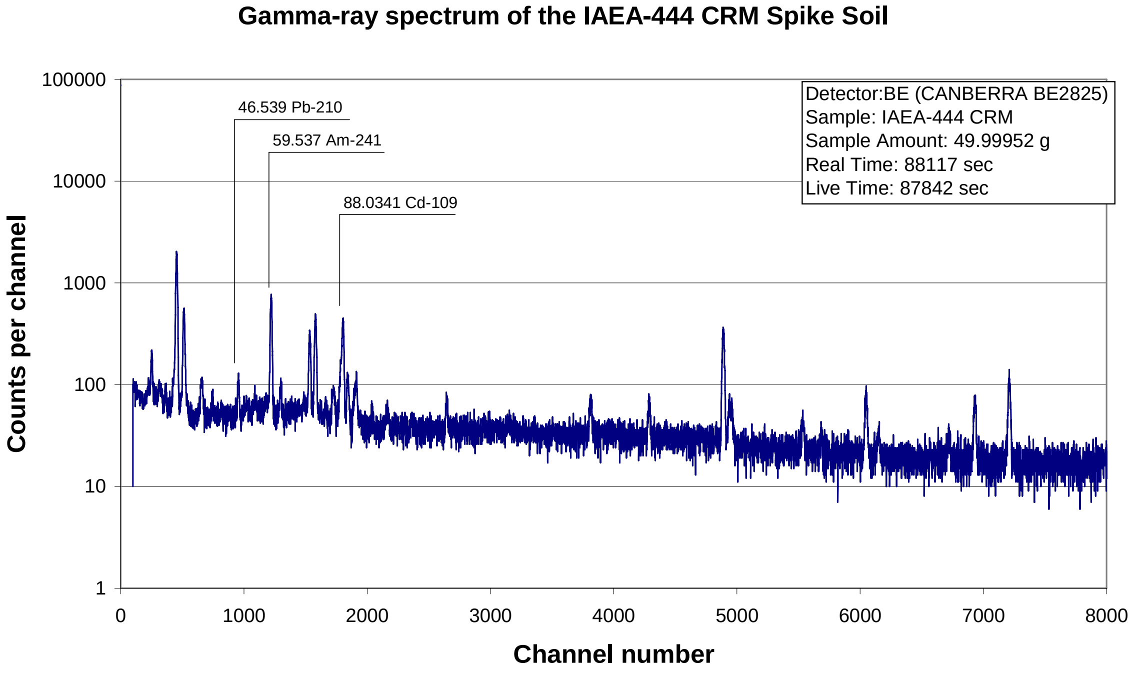
Gamma-ray spectrum of the IAEA-444 CRM Spiked Soil

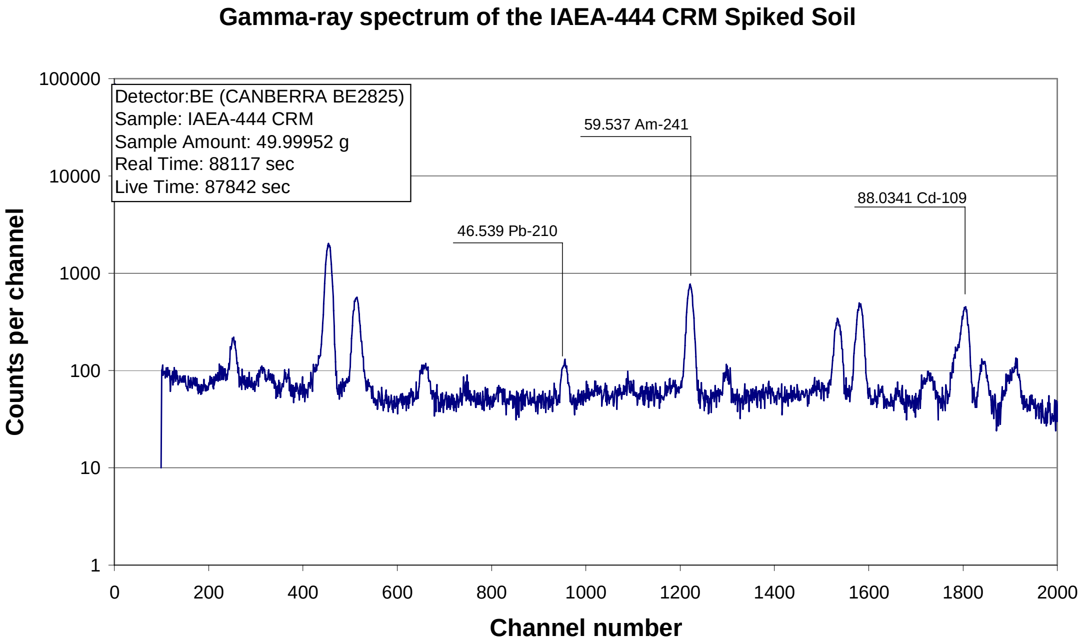


Gamma-ray spectrum of the IAEA-444 CRM Spiked Soil

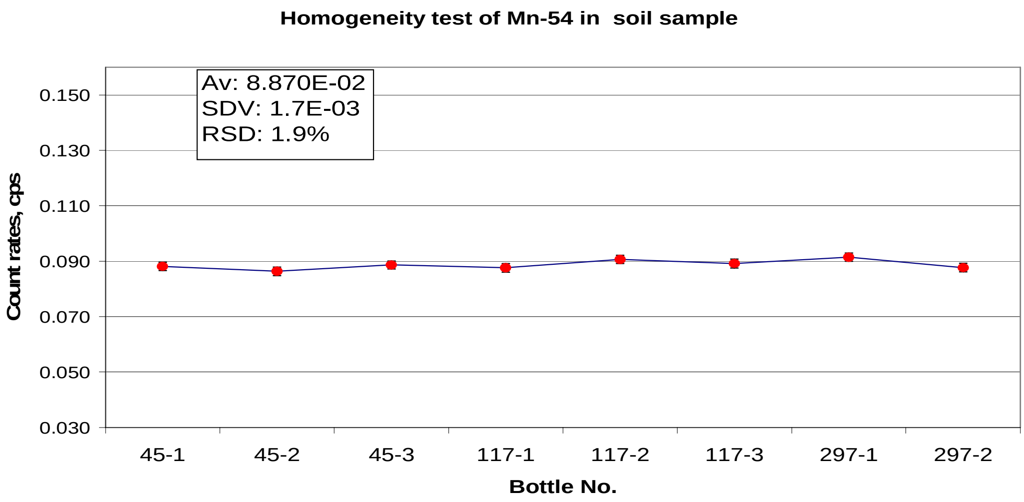


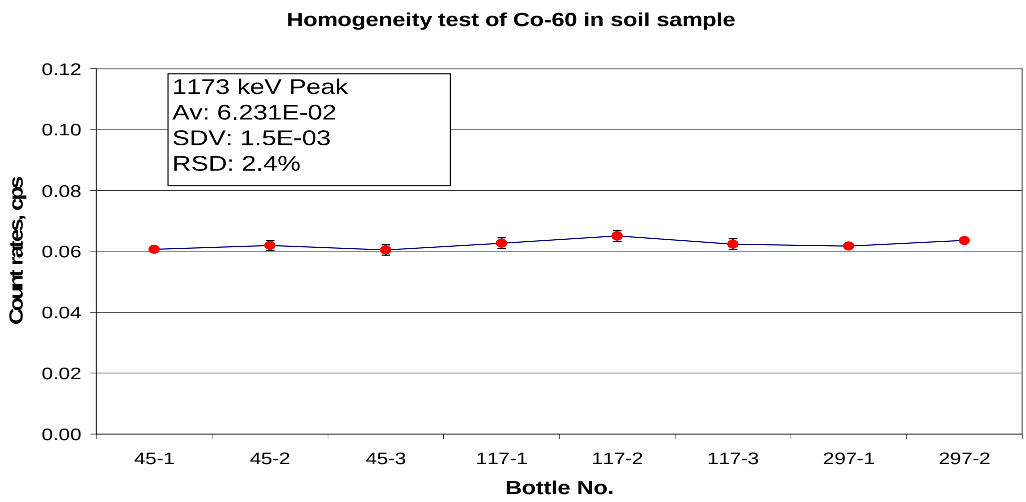
Figures 1 and 2: Gamma-ray spectrum of the low and high energy region of the IAEA-444

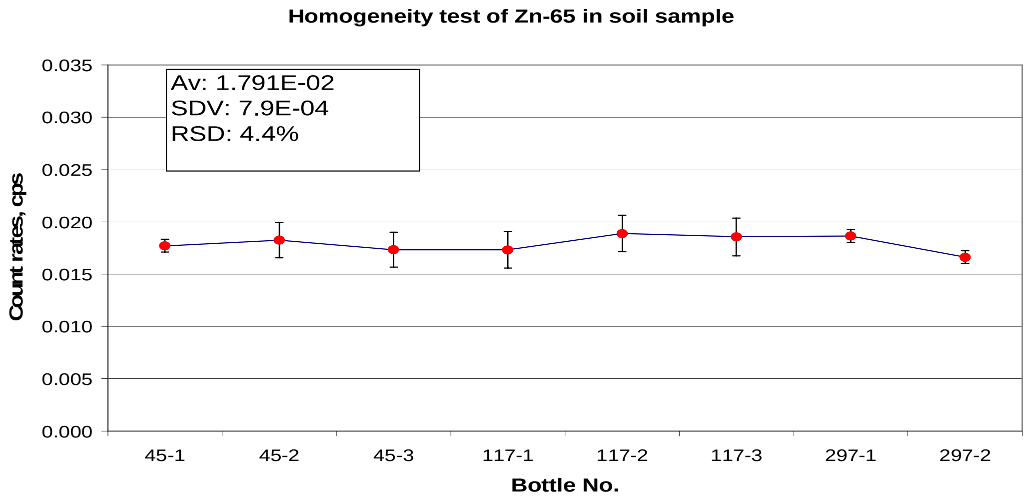




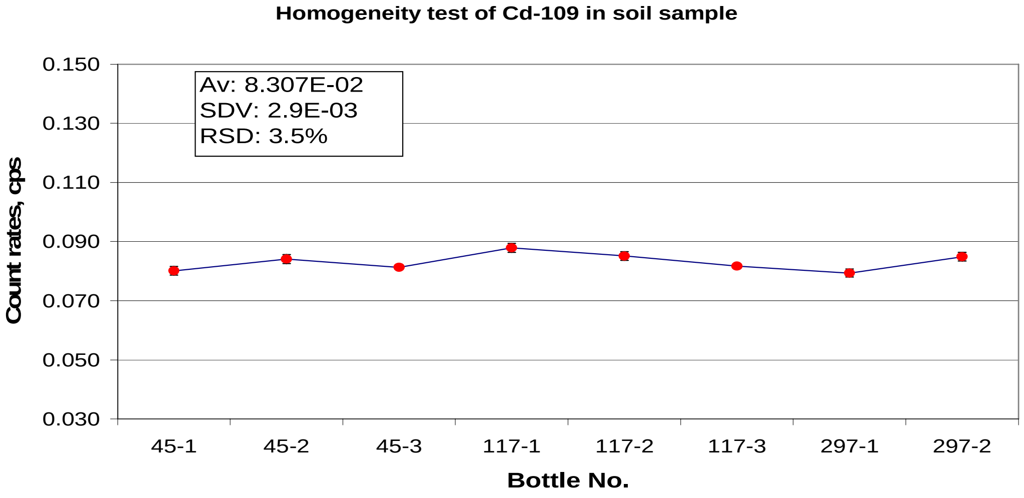
Figures 3 and 4: Low energy region of gamma spectrum of the IAEA-444 using broad energy range detector.

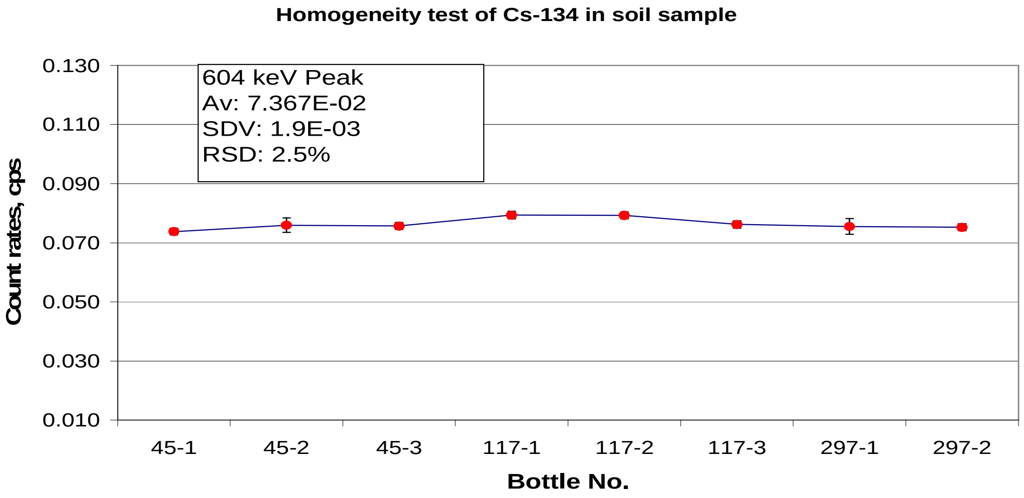


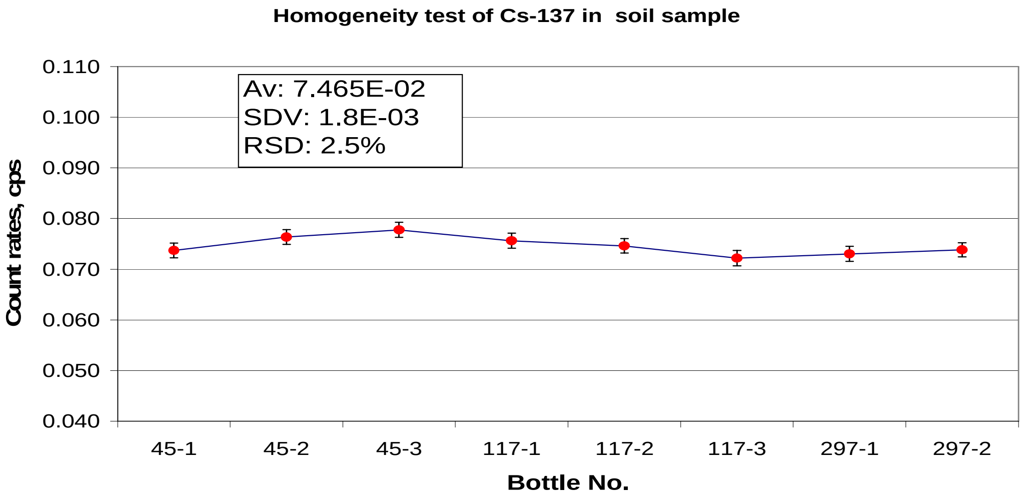




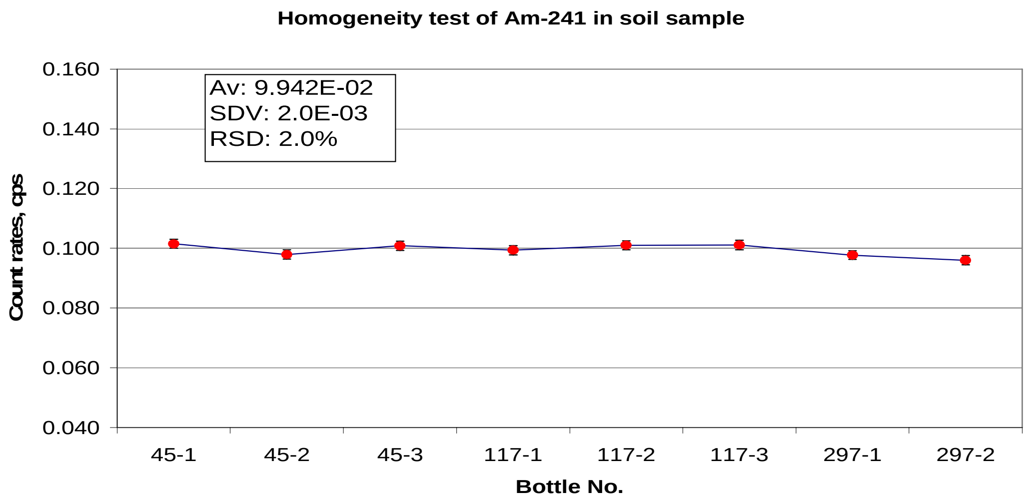
Figures 5, 6 and 7: Graphical presentation of homogeneity test results.

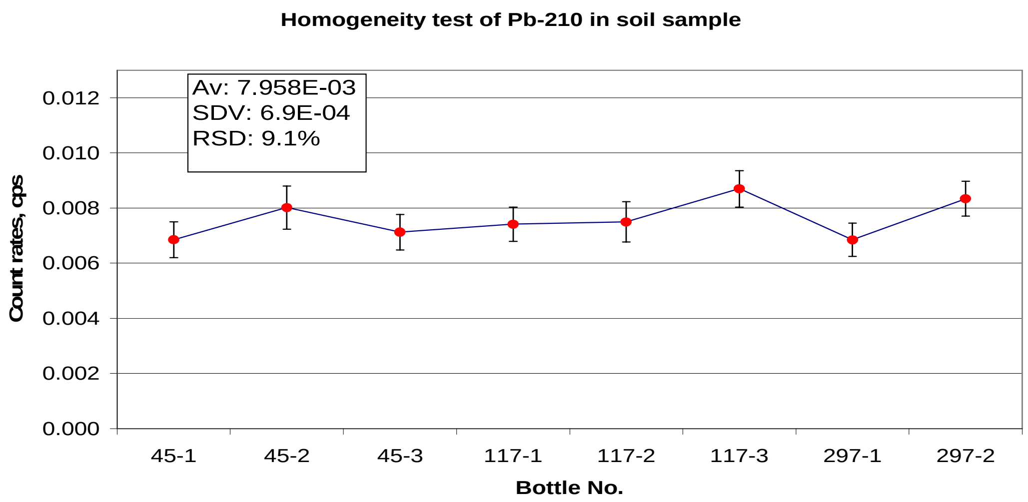






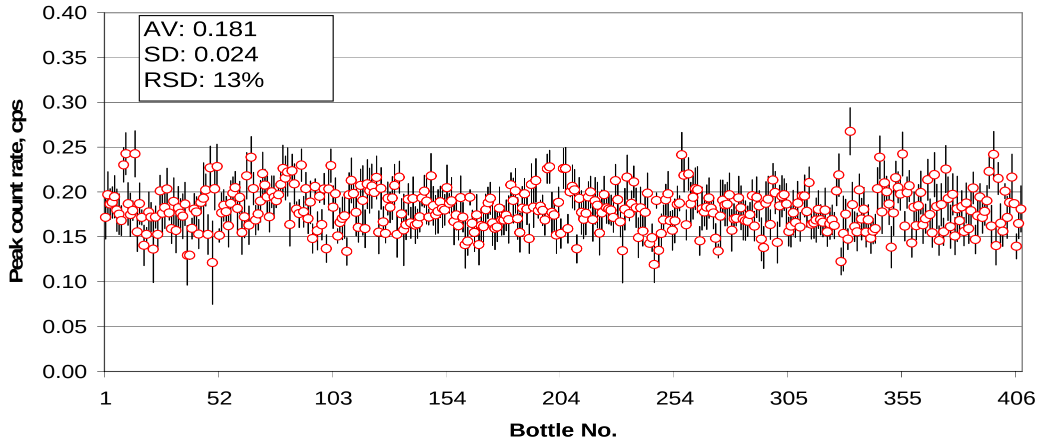
Figures 8, 9 and 10: Graphical presentation of homogeneity test results.



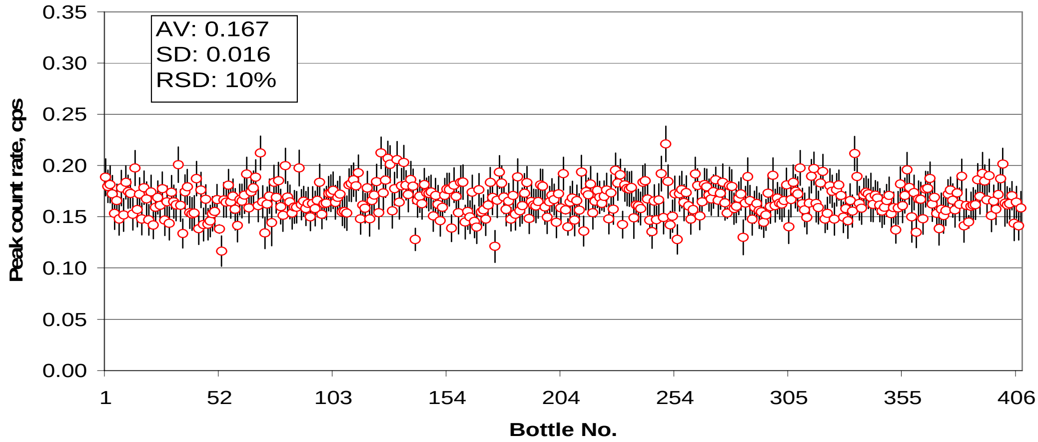


Figures 11 and 12: Graphical presentations of homogeneity test results.

Quality Control test of Am-241in the spiked soil samples



Quality Control test of Cs-137 in the spiked soil samples



Figures 13 and 14: Quality control measurement results of the IAEA-444, measuring time 900 seconds.

2.2. Preparation of the IAEA-445: water spiked with gamma emitting radionuclides

Demineralised tap water outsourced from Seibersdorf was used to prepare this sample. The water was screened using high resolution gamma spectrometry for artificial nuclides and Pb-210, and it was found that all nuclides were below the detection limit and far below the spiked values. The water sample was gravimetrically prepared in one batch. A portion of 200 kg demineralised water was acidified with nitric acid to 3% (mass/mass) and spiked with a mixture of certified single radionuclide solutions traceable to the international standard of radioactivity units. Table 1 lists the sources of the used standard solutions. A pump with multiple outlets was used to homogenise the bulk water sample in a tank of 600 L. The spiked water was dispensed in plastic bottles of 500 ml.

To test the homogeneity of the IAEA-445, three water test portions at 100 g each were analysed from three different bottles using high resolution gamma spectrometry at the IAEA Terrestrial Environment Laboratory. The relative standard deviation of each analyte was calculated. It was found that the relative standard deviations of all analytes were below the method repeatability relative standard deviation, which demonstrates satisfactory homogeneity of the water sample.

The final reference massic activity of each radionuclide, shown in Table 3, was established based on the certified activity concentration value of the standard solution used for spiking, taking into account the successive dilution steps, the mass of spiking mixture and the amount of water being spiked as determined from weighing. The combined standard uncertainty includes two major components: uncertainty of the activity concentration value of the certified standard solution and weighing uncertainty.

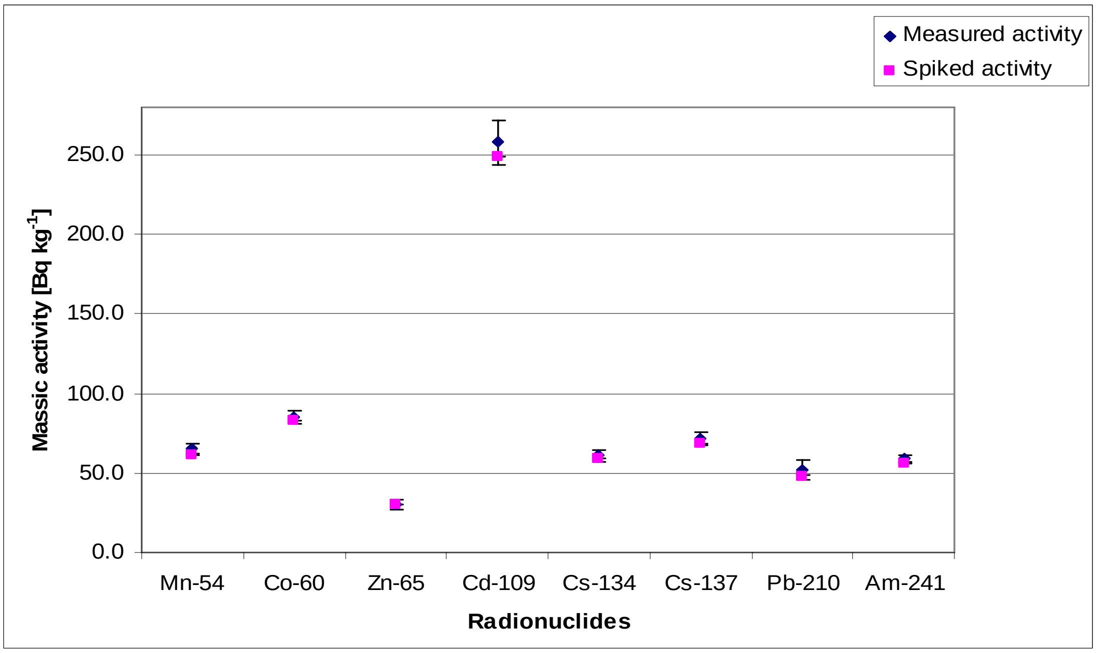
TABLE 3: CERTIFIED MASSIC ACTIVITY AND ASSOCIATED STANDARD UNCERTAINTIES OF THE IAEA-445 CRM

Radionuclide	Certified massic activity Bq kg^{1}	Combined standard uncertainty Bq kg^{-1}
Mn-54	4.74	0.05
Co-60	7.52	0.07
Zn-65	13.06	0.15
Cd-109	34.96	0.35
Cs-134	7.65	0.10
Cs-137	8.12	0.08
Pb-210	29.34	0.5
Am-241	7.11	0.07

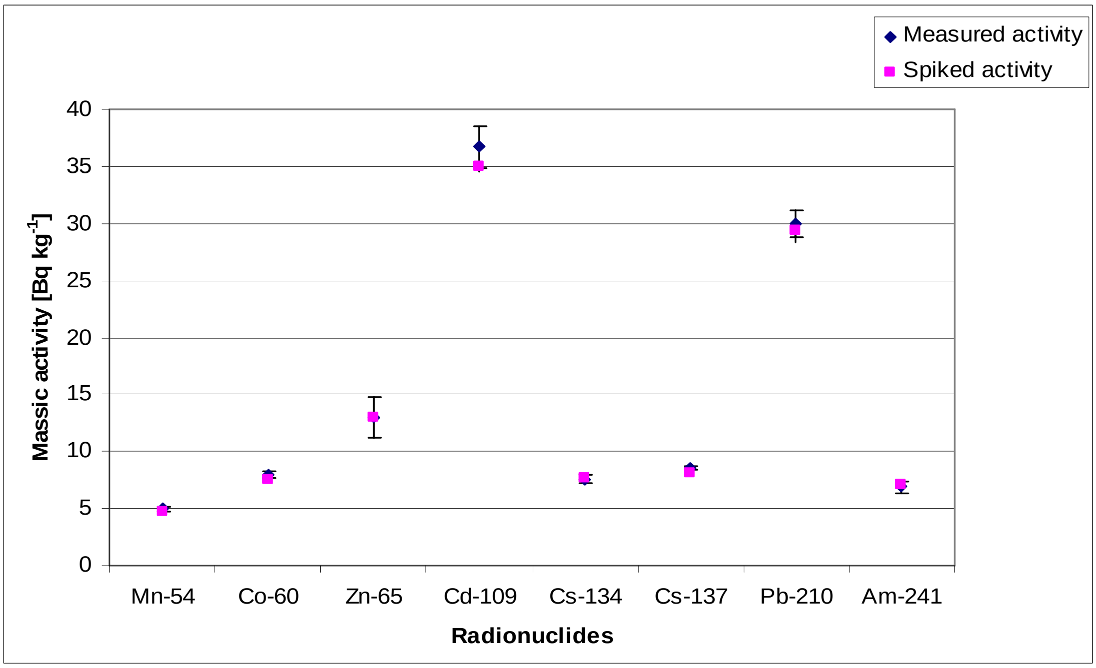
The combined uncertainty is expressed as 1σ (k=1)
The reference date for all certified massic activities is 15 October 2007.

2.3. Verification of the certified values

All certified values of the radionuclides of interest in both CRMs were verified by determining the activity of each radionuclide applying the procedure described in 2.1.1 by taking four test portions from four different bottles. The obtained measurement results were in good agreement with the spiked values. Figures 15 and 16 show the results of such comparison for the IAEA-444 and IAEA-445 respectively.



Figures 15: Graphical representation of the comparison between the spiked values and their measurement results in the IAEA-444.



Figures 16: Graphical representation of the comparison between the spiked values and their measurement results in the IAEA-445.

2.3.1. Equivalence statement

The degree of equivalence (and its uncertainty) of the measurement results of the spiked amounts for all measurands in the IAEA-444 and IAEA-445 compared to their values derived from the gravimetrical dilution of the standard solutions was calculated according to the following formulae 1 and 2:

$$d = (x_{meas} - x_{spike}) \quad (1) \qquad U(d) = 2 * \sqrt{u(x_{meas})^2 + u(x_{spike})^2} \quad (2)$$

Where:
x_{meas} is the measurement result of the spiked amount,
$x_{spike.}$ is the value of the spiked amount based on the dilution factor,
d_i is the difference $(x_{meas} - x_{spike})$,
$u(x_{meas})$ is the standard uncertainty of the measurement result of the spiked amount,
$u(x_{spike})$ is the standard uncertainty associated with x_{spike},
U(d) is the expanded uncertainty ($k = 2$) of the difference d at a 95% level of confidence.
$d_i/U(d_i)$ is the degree of equivalence

Tables 4 and 5 show the calculation results of the degree of equivalence for the IAEA-444 and IAEA-445 respectively. The measurement results show that an acceptable degree of equivalence ($d_i/U(d_i)$<2) was demonstrated for all nuclides of interest in both CRMs.

TABLE 4. CALCULATION OF DEGREE OF EQUIVALANCE FOR THE CERTIFIED VALUES OF MEASURANDS OF THE IAEA-444

Nuclide	$x_{spike.}$ Bq kg^{-1}	$u(x_{spike})$ Bq kg^{-1}	x_{meas} Bq kg^{-1}	$u(x_{meas})$ Bq kg^{-1}	d Bq kg^{-1}	U(d) Bq kg^{-1}	$d_i/U(d_i)$
Mn-54	61.0	1.3	65.5	1.6	4.5	4.14	1.08
Co-60	82.6	2	84.9	2.1	2.3	5.87	0.40
Zn-65	29.9	1	30.3	1.5	0.4	3.60	0.11
Cd-109	248.7	5.2	257.9	7.0	9.2	17.46	0.53
Cs-134	59.4	1.7	60.9	1.8	1.5	4.93	0.31
Cs-137	68.5	1.4	71.5	1.8	3.0	4.62	0.65
Pb-210	48.0	1.6	52.0	3.3	4.0	7.24	0.55
Am-241	55.6	1.6	59.0	1.1	3.4	3.87	0.89

TABLE 5. CALCULATION OF DEGREE OF EQUIVALANCE FOR THE CERTIFIED VALUES OF MEASURANDS OF THE IAEA-445

Nuclide	$x_{spike.}$ Bq kg^{-1}	$u(x_{spike})$ Bq kg^{-1}	x_{meas} Bq kg^{-1}	$u(x_{meas})$ Bq kg^{-1}	d Bq kg^{-1}	U(d) Bq kg^{-1}	$d_i/U(d_i)$
Mn-54	4.74	0.05	4.98	0.12	0.24	0.26	0.92
Co-60	7.52	0.07	7.93	0.13	0.41	0.30	1.39
Zn-65	13.06	0.15	12.99	0.92	0.07	1.86	0.04
Cd-109	34.96	0.35	36.69	0.93	1.73	1.99	0.87
Cs-134	7.65	0.10	7.59	0.19	0.06	0.43	0.14
Cs-137	8.12	0.08	8.54	0.10	0.42	0.26	1.64
Pb-210	29.34	0.5	29.95	0.59	0.61	1.55	0.39
Am-241	7.11	0.07	6.92	0.25	0.19	0.52	0.37

3. INSTRUCTIONS TO THE END USERS

3.1. Metrological traceability and uncertainty of the assigned values

The quantity values assigned to the IAEA-444 and IAEA-445 certified reference materials are massic activities of Mn-54, Co-60, Zn-65, Cd-109, Cs-134, Cs-137, Pb-210 and Am-241, expressed in the derived SI unit Bq kg^{-1}. Values were established based on the certified activity values of the standard solutions used for spiking, taking into account the successive dilution steps, the mass of spiking solutions and the amount of matrix being spiked. For all values used in the calculation of the assigned values and associated uncertainties, the evidence on metrological traceability to the SI Units was provided.

3.2. Intended use

IAEA-444 and IAEA-445 can be used for the development and validation of analytical work and for training purposes when measuring Mn-54, Co-60, Zn-65, Cd-109, Cs-134, Cs-137, Pb-210 and Am-241 massic activities in soil or water respectively.

Based on the defined metrological traceability and the relatively small uncertainty of the assigned quantity of massic activities values, the IAEA-444 and IAEA-445 could be used as a calibrator when soil and water with similar levels of activities and matrix are analysed.

The information value of Pb-210 in the IAEA-444 can not be used for calibration purposes; it could be used for quality assurance/control of the analytical results and for method validation.

It is not recommended to dilute the IAEA-445 water certified reference material to fit the measuring geometry, as this will increase the uncertainty of the reference values.

3.3. 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.

3.4. Expiry date

Based on the experience with similar materials the reference values for Mn-54, Co-60, Zn-65, Cd-109, Cs-134, Cs-137, Pb-210 and Am-241 are valid until 31 December 2015, provided the original bottle is handled and stored in accordance with the instructions given in this reference sheet (see “Storage”). This certification is nullified if the bottle is damaged. Reference values as stated in this reference sheet may be updated if more information becomes available. Users of this reference material should ensure that the reference sheet in their possession is current. This can be accomplished by contacting the appropriate web page at the IAEA main web site: http://www.iaea.org/

The IAEA is monitoring the long term stability of the material and customers will be informed in case of any observed change.

3.5. Legal disclaimer

Although great care has been taken to maintain the accuracy of information contained in this reference sheet, the IAEA assumes no responsibility for consequences which may arise from its use.

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