IAEA/AQ/53

IAEA Analytical Quality in Nuclear Applications Series No. 53

Certification of Massic Activities of Radionuclides in IAEA-410 Bikini Atoll Sediment

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CERTIFICATION OF MASSIC ACTIVITIES OF RADIONUCLIDES IN IAEA-410 BIKINI ATOLL SEDIMENT

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CERTIFICATION OF MASSIC ACTIVITIES OF RADIONUCLIDES IN IAEA-410 BIKINI ATOLL SEDIMENT

INTERNATIONAL ATOMIC ENERGY AGENCY
VIENNA, 2018

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CERTIFICATION OF MASSIC ACTIVITIES OF RADIONUCLIDES IN IAEA-410 BIKINI ATOLL SEDIMENT
IAEA, VIENNA, 2018
IAEA/AQ/53
ISSN 2074–7659

Printed by the IAEA in Austria
May 2018

FOREWORD

For almost 50 years, the Radiometrics Laboratory of the IAEA Environment Laboratories has been providing quality products and services for radionuclides in marine samples, including the organization of interlaboratory comparisons, the production of reference materials and certified reference materials, and training. The production of a new reference material is a long process, covering the identification of needs, sample collection, pretreatment, physical homogenization, bottling, homogeneity testing, distribution to laboratories, evaluation of data, preliminary reporting, additional analyses by expert laboratories, certification of material (including the determination of proper values and their uncertainties), and finally issuing the reference materials and certified reference materials. More than 45 reference materials have been produced, including a wide range of marine sample matrices and radionuclides.

As part of these activities, a new characterization study using different analytical methods was organized to provide sufficient data on a sediment sample with elevated radionuclide levels due to the influence of historical nuclear tests in the Bikini Atoll region. The reference material is aimed at the analysis of anthropogenic and natural radionuclides in the sediment. It is expected that the sample, after certification, will be issued as a certified reference material for radionuclides in sediment.

The IAEA officers responsible for this publication were M.K. Pham, A.V. Harms and I. Osvath of the IAEA Environment Laboratories.

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CONTENTS

1. INTRODUCTION

The accurate and precise determination of radionuclide concentrations in marine samples is an important aspect of marine radioactivity assessment and the use of radionuclides in studies of oceanographic processes. To address the problem of data quality, the IAEA Environment Laboratories (IAEA-EL) in Monaco regularly conduct characterization studies aimed to assign values to reference materials for radionuclides and other components in different matrices of marine samples as an integral part of the Sub-programme IAEA Reference Products for Science and Trade (see Refs [1, 2]).

The sediment was collected during the "IAEA '97 Pacific Ocean Expedition" to the NW Pacific Ocean on board of research vessel *Bosei Maru*, which took place from 21 October to 20 November 1997, with sampling stations at Bikini and Eniwetok atolls (See Ref. [3]). The Japan Meteorological Agency and Tokai University were among the collaborating institutes.

As the sample was collected offshore the Bikini Atoll, elevated levels of long-lived anthropogenic radionuclides (such as plutonium and americium isotopes) were expected due to the influence of the historical atmospheric nuclear weapons tests. Participants were informed that the expected activities for anthropogenic radionuclides would be in the ranges:

Gamma emitters	0.1–0.5 kBq kg^{-1}
Transuranics	0.01–10 Bq kg^{-1}

This report describes the results on anthropogenic and natural radionuclide determinations in sediment obtained from 27 selected laboratories (including IAEA-EL and 5 laboratories belonging to CELLAR[1]), which will allow the IAEA-EL to produce a new certified reference material IAEA-410 following ISO guidelines in Refs [4-7].

2. SCOPE OF THE CHARACTERIZATION STUDY

This characterization study was organized to obtain sufficient data using different analytical methods on a sediment sample with elevated radionuclide levels due to the influence of the historical nuclear test to the Bikini Atoll region.

The characterization study was designed for the analysis of anthropogenic and natural radionuclides. Participating laboratories were requested to determine as many radionuclides as possible among the following: ^{40}K, ^{137}Cs, ^{210}Pb, ^{210}Po, ^{226}Ra, ^{228}Ra, U, Th and Pu isotopes and ^{241}Am. Any additional measurements were welcome and would be included in the report as information values, unless sufficient data are available to justify statistical evaluation. The participating laboratories were chosen to allow both radiometric (gamma ray spectrometry, alpha particle spectrometry and beta counting) and mass spectrometry measurement techniques (e.g. ICP-MS, and AMS).

[1] Collaboration of European Low-level Underground Laboratories

3. DESCRIPTION OF THE MATERIAL

A total of 60 kg wet mass of sediment was collected from offshore Bikini Atoll (11º35' N, 165º20' E, water depth 4500 m) on 10 November 1997 by the IAEA during the "IAEA '97 Pacific Ocean Expedition" to the NW Pacific Ocean with the research vessel Bosei Maru [3].

The sediment was collected using box coring down to 24 cm depth in the bottom sediment. The sediment is coralligenous type. It was first dried in open air and subsequently freeze dried leaving a total dry mass of 16 kg. The sample was then ground into powder and sieved through a 250 µm mesh The sample was homogenized by mixing in a nitrogen atmosphere, bottled in glass sealed bottles ((100±5) g units) and coded as IAEA-410 (for a total of 150 bottles). All bottles were sterilized at 25 kGy (^{60}Co) in an irradiation facility.

The moisture content of the lyophilized material, determined by drying an aliquot 1 g to a constant mass at 105°C (firstly 48 hours of drying, then wait for plateau with a measure each 24 h until to a constant mass), was found to be approximately 1.65% at the time of the preparation of this sample. However, as the moisture content may change with the ambient humidity and temperature, it was recommended that it be determined again by the analysing laboratories by drying at 105°C to a constant mass at the time of analysis in the laboratory and to correct the results accordingly.

4. HOMOGENEITY AND STABILITY TESTS

Sample homogeneity was checked by the determination of ^{137}Cs, ^{40}K, ^{210}Pb (^{210}Po), ^{214}Bi, ^{214}Pb, ^{226}Ra, ^{228}Th, ^{230}Th, ^{232}Th, U isotopes and $^{239+240}$Pu activities (by using high-resolution low-background gamma ray spectrometry (placed in underground laboratory of the Radiometrics laboratory, IAEA-EL-RML) and alpha-ray spectrometry (Ortec system at IAEA-EL-RML) and Inductively Coupled Plasma Mass Spectrometry (ICP-MS). The first homogeneity test between bottles was done for 14 bottles chosen at random at different masses of samples (60 g for gamma ray spectrometry and between 1.5 and 10 g for alpha particle spectrometry). For the gamma emitter's determination, the sediment sample was sealed in a tin can geometry for three weeks (to get the equilibrium between radon daughters and mother) before gamma ray spectrometry measurement, the calculation of activity concentration and uncertainties were done following ISO 18589-3 [7] and procedure set up by the Radiometrics laboratory for gamma spectrometry accreditation. For alpha emitters, radiochemical purification was needed before alpha-ray spectrometry measurement. The second test within bottles was done for another 10 aliquots at 0.5–3 g of sample for Pu isotopes analysis by mass spectrometry AMS (Centro Nacional de Aceleradores, Universidad de Seville, Spain) and 0.5 g of material for U isotopes determination using ICP-MS (Departamento de Fisica Applicada I, Universidad de Seville, Spain), respectively. The procedure of determination of plutonium and uranium by mass spectrometry were set up by their laboratories respectively. Homogeneity was tested by using one-way analysis of variance (ANOVA). The coefficient variation was below 15% for all radionuclides determined (some examples are shown in Table 1, Appendix I). The "between samples" variances showed no significant differences from the "within sample" variances for the radionuclides tested. Results were identical within statistical

uncertainties. On the basis of the homogeneity tests (see Figs. 1, 2 and 3, Appendix II for ^{226}Ra, ^{214}Bi, and $^{239+240}Pu$, for instance), the material can be considered homogeneous for the radionuclides tested at the mass used. All analytical data obtained from homogeneity test were included in the final data reported for Radiometrics laboratory as mean value and their uncertainties as standard deviation.

An additional homogeneity test for major and trace elements (P, S, Cl, K, Ca, Fe, Ni, Cu, Zn, As, Br, Sr, I, Ba and Pb) for 4 g of sediment sample was done by XRF[2]. The coefficient of variation was below 10% for XRF determined elements.

For radionuclides in marine environment, the stability test is performed for gamma emitters during their life time in the stock whenever the CRM will be released, one per year. The gamma result (if necessary) is corrected for decay correction of reference date following the updated data from http://laraweb.free.fr/Spectro/ (See Ref. [8]).

5. SAMPLE DISPATCH AND DATA REPORTING

Each participant received 100 g of the sediment sample.

For each radionuclide analysed, the following information was requested:

- Average mass of sample;
- Number of analyses;
- Mass activity calculated in net values (i.e. corrected for blank, background, moisture content, etc.) and expressed in Bq kg^{-1};
- Estimate of the uncertainty;
- Description of chemical procedures and counting equipment;
- Reference standard solutions used; and
- Chemical recoveries, counting time, half-life (using the updated data from "laraweb" source: http://laraweb.free.fr/Spectro/

The massic activities were to be reported as net values (i.e. after correction for blank, background, etc.) calculated on a dry mass basis and expressed in Bq kg^{-1}. Results not statistically significant were to be reported as "less than" values.

The reference date was set at the 1st January 2013.

The samples were distributed to the selected 30 laboratories in February 2013. The selection of participants for this characterization study was based on the measurement performances demonstrated by laboratories in the previous IAEA inter-laboratory comparisons and certification campaigns on marine sediments. Only results of laboratories having a quality system in place, using validated methods, applying uncertainty and traceability

[2] X-ray fluorescence

concepts and having provided good results in previous IAEA inter-laboratory comparisons were accepted for the calculation of certified values and their uncertainties.

The deadline for reporting data was set for 31 August 2013. A reminder was sent to participants who did not submit the results in time extending the deadline to December 2013. A total of 26 laboratories sent their reports. The list of reported radionuclides is given in Table 3, Appendix I.

The list of contributing laboratories may be found at the end of the report.

6. EVALUATION OF RESULTS

6.1. DATA TREATMENT

The submitted results are shown under their laboratory code numbers in Tables 4 to 23, Appendix I. Laboratories' means and their uncertainties were calculated either as arithmetic means with corresponding standard deviations or as weighted means with weighted uncertainties in the case of large differences in the data.

6.2. STATISTICAL EVALUATION

The characterization campaign resulted in 6-24 results for the 24 radionuclides of interest. The obtained data were first checked for compliance with the certification requirements, and then for their validity based on technical reasoning. Robust statistics as described in ISO 13528 [6] were used for the determination of the assigned values, where the robust mean and robust standard deviations were calculated as per Algorithm A as detailed described in Annex C.21 of ISO 13528 [6].

Briefly, individual results were ranked in increasing order:

$(x_1, x_2, \quad x_i, \ldots x_n)$

Initial values of the robust average x^* and robust standard deviation s^* were calculated as:

$$x^* = \text{median of } x_i \quad (i=1,2,3\ldots.n) \qquad (Eq.\ 1)$$

Where the n is the number of reported results

$$s^* = 1.483 \text{ median } of\ |x_i - x^*| \quad (i=1,2,3\ldots..n) \qquad (Eq.\ 2)$$

The initial values x^* and s^* were updated by calculating:

$$\delta = 1.5 s^* \qquad (Eq.\ 3)$$

For each x_i $(i = 1, 2, 3 \ldots . n)$ calculate

$$x_i^* = \begin{cases} x^* - \delta, & \text{if } x_i < x^* - \delta \\ x^* + \delta, & \text{if } x_i > x^* + \delta \\ x_i\ , & \text{otherwise} \end{cases} \qquad (Eq.\ 4)$$

New values for x^* and s^* were calculated as:

$$\boldsymbol{x^* = \sum_1^n x_i^* / n} \qquad (Eq.\ 5)$$

$$\boldsymbol{s^* = 1.134\sqrt{\Sigma(x_i^* - x^*)^2/(n-1)}} \qquad (Eq.\ 6)$$

The robust estimates of $\boldsymbol{x^*}$ and $\boldsymbol{s^*}$ were calculated by iteration by updating the values of $\boldsymbol{x^*}$ and $\boldsymbol{s^*}$ until they converged to the third significant figure.

Massic activities for 27 radionuclides were reported and results are shown in Table 3, Appendix I, with the number of reporting laboratories for each radionuclide. The number of reported "less than" values is shown in parentheses. The results for the most frequently measured radionuclides can be found in Tables 4 to 23, Appendix I, and Figures 4 to 19, Appendix II, while the less frequently measured radionuclides are presented in Table 24, Appendix I. The certified values obtained after statistical treatment are presented in Appendix I, Table 25, and information values are presented in Appendix I, Table 26.

6.3. EXPLANATION OF TABLES

6.3.1. Laboratory code

Each laboratory was assigned an individual code number to ensure anonymity.

6.3.2. Method code

The analytical techniques employed by participants are specified with following codes:

Method code	Method	Detailed procedure
A	Alpha particle spectrometry	Treatment, evaporation/precipitation, ion exchange and electro deposition followed by alpha particle spectrometry
G	Gamma spectrometry	High resolution gamma ray-spectrometry using HP-Ge (High Purity Germanium) detectors
ICP-MS	Inductively Coupled Plasma Mass Spectrometry	Treatment, ion exchange, ICP-MS (Inductively Coupled Plasma Mass Spectrometry)
AMS	Accelerator Mass spectrometry	Leaching, treatment, AMS (Accelerator Mass Spectrometry)

6.3.3. Number of results

The number of determinations corresponds to the number of individual results received from each laboratory.

6.3.4. Massic activity

The activity corresponds to the arithmetical or weighted mean computed from all the individual results obtained from the participants with the corresponding standard deviation or weighted uncertainty. They are calculated as massic activities for each radionuclide respectively and expressed in the derived SI[3] unit Bq kg^{-1}.

6.4. EXPLANATION OF FIGURES

The figures (Figs. 4 to 19, Appendix II) present the data in order of ascending massic activity. In all figures the reported results are plotted with the robust mean denoted by a straight red line, while the dashed green lines represent the expanded uncertainty (k=2) associated with the robust mean (as calculated in equation 6). The error bars represent the expanded uncertainty as reported by participants.

6.5. CRITERIA FOR ASSIGNING CERTIFIED VALUES AND UNCERTAINTIES

A good agreement within the stated uncertainty was observed for results obtained with different methods. Therefore, all of them were considered in deriving certified values.

A certified value was assigned when at least 5 independent results were available and its relative expanded uncertainty (at k=2) was less than 15%. These criteria were fulfilled for ^{40}K, ^{210}Pb (^{210}Po), ^{226}Ra, ^{228}Ra, ^{228}Th, ^{232}Th, ^{234}U, ^{238}U, ^{239}Pu, $^{239+240}$Pu and ^{241}Am. The certified values are presented in Table 25, together with their expanded uncertainty.

For ^{137}Cs, ^{230}Th, ^{234}Th, ^{235}U, ^{238}Pu ^{240}Pu, the criteria were not fulfilled; robust mean and uncertainties for those radionuclides are given only as information values in Table 26.

6.6. METROLOGICAL TRACEABILITY

Only validated methods and calibration applied within stated scope were used by participating laboratories in this characterization study. All results obtained by different laboratories are checked if they are based on reliable measurement standards.

In the report form sent to the participants, they were asked to report the results following SI units (expressed as Bq/kg^{-1} dry mass) at the reference date, to provide the method determination of activity concentration and tracers and calibration solution (metrological traceability). This was provided in their individual reports. The individual results are therefore traceable to the SI. This is also confirmed by the agreement among the technically accepted datasets. As the assigned values are combinations of agreeing results individually traceable to the SI, the assigned quantity values are also traceable to the SI system of units.

[3] International System of Units (SI)

7. RESULTS AND DISCUSSION

7.1. ANTHROPOGENIC RADIONUCLIDES

Results of the determination of ^{137}Cs, $^{239+240}$Pu, ^{239}Pu, ^{240}Pu and ^{241}Am reported by participants are presented in Tables 4–8, Appendix I, and shown in Figures 4–8, Appendix II.

7.1.1. ^{137}Cs

Data were reported by 15 laboratories (Table 4, Appendix I and Fig. 4, Appendix II); nine of them were reported as below LLD (Low Limit Detection), six others were above LLD and could be used for data evaluation. The laboratories mainly used direct gamma ray spectrometry for the ^{137}Cs determination.

7.1.2. Plutonium isotopes

The majority of participants used a conventional method based on sample treatment, ion-exchange separation followed by electro deposition and alpha particle spectrometry. Some laboratories could determine separately ^{239}Pu and ^{240}Pu by using ICP-MS and AMS, after radiochemical separation of plutonium isotopes.

7.1.2.1. ^{238}Pu

Ten data sets were reported (Table 5, Appendix I), which were used for data evaluation.

7.1.2.2. $^{239+240}$Pu

Twenty-one data sets were reported (Table 5, Appendix I and Fig. *5*, Appendix II). Both alpha particle spectrometry and mass spectrometry techniques were used for $^{239+240}$Pu determinations. Most analyses were performed using conventional alpha particle spectrometry, while some results were combinations from ICP-MS and AMS methods.

The laboratory number 12 performed two series of samples for Pu isotopes (Table 5). The first one was done for 9 aliquots of samples at different weights (0.5; 1; 3; 5 g) using normal/partly leaching digestion technique with only HNO_3. The second one was done for 6 aliquots at weights 0.5 – 1 g using total digestion with HF/HNO3 and boric acids/HCl. The former gave the non-homogenous of Pu at weights 0.5, 1 and 5 g; however, the four values at 3 g are homogenous and reported as average value in Table 5. The latter gave the homogenous data of these 6 aliquots of samples, indicating that the non-homogeneity of samples could be due to the leaching techniques. This observation was confirmed by further investigation using two different leaching methods for 10 aliquots of 0.5 g of sample and then measuring the sample by AMS technique in CAN (Centro Nacional de Aceleradores, Seville, Spain). The difference between two methods could reach 15% (the average value of 5 aliquots using HNO_3/HF leaching (5.33 ± 0.29) Bq kg^{-1} compared to (4.70 ± 0.33) Bq kg^{-1} obtained by HNO_3 method leaching).

7.1.2.3. ^{239}Pu and ^{240}Pu

Six laboratories determined separately ^{239}Pu and ^{240}Pu mass activities using mass spectrometry (ICP-MS and AMS). The results are presented in Table 6, Appendix I and Figs. 6 and 7, Appendix II. The robust mean, given as the certified value, is 2.42 ± 0.26 Bq kg^{-1} for ^{239}Pu and 2.27 ± 0.40 Bq kg^{-1} for ^{240}Pu, respectively It is worth

noticing that the sum of the ^{239}Pu and ^{240}Pu mass activities is in agreement with the $^{239+240}$Pu value determined by alpha technique (4.68 ± 0.48 Bq kg^{-1}).

7.1.3. ^{241}Am

Twenty-three laboratories determined the ^{241}Am massic activity with seven using alpha particle spectrometry with prior radiochemical purification from rare earth elements, and 16 laboratories using direct gamma spectrometry measurement (Table 7 and 8, Appendix I and Fig. 8, Appendix II).

The laboratory number 12 performed two series of samples for Am isotopes (Table 7 and 8). The first one was done for 9 aliquots of samples at different weights (0.5; 1; 3; 5 g) using normal/partly leaching digestion technique with only HNO_3. The second one was done for 6 aliquots at weights 0.5–1 g using total digestion with HF/HNO3 and boric acids/HCl. The former gave the non-homogenous of ^{241}Am at weights 0.5, 1 and 5 g; however, the four values at 3 g are homogenous and reported as average value in the Tables 7 and 8. The latter gave the homogenous data of these 6 aliquots of samples, indicating that non-homogeneity of samples could be due to the leaching techniques, which again confirm the above observation for Pu case in the same analytical series (7.1.2.2.).

7.2. NATURAL RADIONUCLIDES

7.2.1. Uranium series

7.2.1.1. ^{238}U

Nineteen data sets were reported (Table 9, Appendix I and Fig. 9, Appendix II). Eight participants used a conventional method based on sample treatment, ion-exchange separation followed by electro deposition and alpha particle spectrometry. Six other laboratories used direct gamma ray spectrometry technique. Four laboratories could determine the activities using ICP-MS method, with prior radiochemical separation of uranium isotopes. There is apparently disequilibrium between ^{238}U and ^{226}Ra (and descendants such as their daughters ^{214}Pb and ^{214}Bi) resulting in a large difference between the two assigned values (see below for ^{226}Ra).

7.2.1.2. ^{234}Th

Six data sets were reported (Table 10, Appendix I and Fig. 10, Appendix II). All participants used direct gamma ray spectrometry measurement.

7.2.1.3. ^{234}U

Twelve data sets were reported (Table 11, Appendix I and Fig. 11, Appendix II). All values were accepted, except one. Most participants used a conventional method based on sample treatment, ion-exchange separation followed by electro deposition and alpha particle spectrometry. Two laboratories could determine the activities using ICP-MS method, with prior radiochemical separation of the uranium isotopes.

7.2.1.4. ^{230}Th

Six data sets reported (Table 12, Appendix I and Fig. 12, Appendix II). Half of the participants used a conventional method based on sample treatment, ion-exchange separation followed by electro deposition and alpha particle spectrometry; the rest used direct gamma ray spectrometry. The strong disequilibrium between ^{238}U and ^{230}Th (and ^{226}Ra, see below) is observed.

7.2.1.5. ^{226}Ra

Data were reported from twenty-two laboratories (Table 13, Appendix I, and Fig. 13, Appendix II). Most laboratories used direct gamma ray spectrometry to determine ^{226}Ra activity at 186 keV or through their daughters ^{214}Bi and ^{214}Pb peaks at 609 and 352 keV, respectively. Two laboratories used alpha particle spectrometry technique.

7.2.1.6. ^{214}Bi and ^{214}Pb

Ten and 13 laboratories reported ^{214}Bi and ^{214}Pb results, respectively (Table 14, Appendix I). These data were determined by using gamma ray spectrometry and are in the same range with ^{226}Ra mass activities (see above for 7.2.1.5. ^{226}Ra) showing that the ^{226}Ra and its progeny ^{214}Bi and ^{214}Pb are in equilibrium (but not with ^{238}U, see above). Those radionuclides are frequently used as daughters to determine indirectly ^{226}Ra activity concentration, for this reason they are mentioned in the table but they will later not be used for final calculation.

7.2.1.7 ^{210}Pb (^{210}Po)

Data were reported from 25 laboratories (Table 15, Appendix I and Fig. 14, Appendix II). ^{210}Pb and ^{210}Po were considered to be in equilibrium at the characterization study period (2013), when ten half-lives of ^{210}Po have passed, compared to the sampling time (1997) and the ^{210}Pb values were decay-corrected back to the reference date at 1 January 2013. While most participants used direct gamma ray spectrometry to measure ^{210}Pb at 46.5 keV, seven participants used alpha particle spectrometry with prior radiochemical purification of ^{210}Po, then electro deposition on a silver disk. The difference of values between ^{226}Ra and ^{210}Pb(^{210}Po) is due to the supported Pb during the decay processus of ^{226}Ra.

7.2.2 ^{235}U series

7.2.2.1. ^{235}U

Fifteen data sets were reported (Table 16, Appendix I and Fig. 15, Appendix II). Three participants reported results below the LLD. Seven participants used a conventional method based on sample treatment, ion-exchange separation followed by electro deposition and alpha particle spectrometry. Five laboratories determined ^{235}U using direct gamma ray spectrometry at 186 keV peak/line by subtracting the ^{226}Ra contribution in the same peak/line; or from 0.046-fold of the ^{234}Th (^{238}U) activity (determined from 63.3 and 92.5 keV lines). Three laboratories could determine the activities using ICP-MS method, with prior radiochemical separation of the uranium isotopes.

7.2.2.2. ^{227}Ac

Six laboratories reported ^{227}Ac (Table 17, Appendix I). These data were determined by using gamma ray spectrometry and are not in the same range of ^{235}U mass activities (see above) showing that the ^{235}U and its progeny

^{227}Ac are not in equilibrium. This radionuclide is frequently used as daughter to determine indirectly ^{235}U activity concentration, for this reason it is mentioned in the table but it will later not be used for final calculation.

7.2.3. Thorium series

7.2.3.1. ^{232}Th

Eight data sets were reported (Table 18, Appendix I and Fig.16, Appendix II). Three data sets were analysed by gamma ray spectrometry, four other used a conventional method based on sample treatment, ion-exchange separation followed by electro-deposition and alpha particle spectrometry; one data set was determined by ICP-MS method.

7.2.3.2. ^{228}Ra

Thirteen laboratories reported data for ^{228}Ra (Table 19, Appendix I and Fig. 17, Appendix II). All laboratories used direct gamma ray spectrometry to determine ^{228}Ra activity through daughters either ^{228}Ac at 911 keV or ^{228}Th at 238 keV or 583 keV. The equilibrium between ^{228}Ra and ^{228}Th is observed (see below the ^{228}Th results).

7.2.3.3. ^{228}Ac

Twelve laboratories reported ^{228}Ac (Table 20, Appendix I). These data were determined by using gamma ray spectrometry and are at about the same levels as ^{228}Th and ^{228}Ra (see above for ^{228}Ra and below for ^{228}Th and ^{208}Tl, respectively) showing that ^{228}Ra and its daughters ^{228}Ac, ^{228}Th, ^{208}Tl are in equilibrium (and also with their original precursor ^{232}Th, see above). This radionuclide is frequently used as daughter to determine indirectly ^{228}Ra activity concentration, for this reason it is mentioned in the table but it will later not be used for final calculation.

7.2.3.4. ^{228}Th

Twelve data sets were reported (Table 21, Appendix I and Fig. 18, Appendix II). Most participants used direct gamma ray spectrometry to determine ^{228}Th at two peaks 238 keV and 583 keV where the branching ratios are important (43.5% and 30.6%, respectively). Three laboratories used a conventional method based on sample treatment, ion-exchange separation followed by electro deposition and alpha particle spectrometry.

7.2.3.5. ^{212}Bi and ^{212}Pb

Nine and seven laboratories reported ^{212}Bi and ^{212}Pb results, respectively (Table 22, Appendix I). These data were determined by using gamma ray spectrometry and are in the same range with ^{228}Ra (see above for 7.2.3.2 ^{228}Ra) showing that the ^{228}Ra and its progeny ^{212}Bi and ^{212}Pb are in equilibrium (and also with its original precursor ^{232}Th see above). Those radionuclides are frequently used as daughters to determine indirectly ^{228}Ra activity concentration, for this reason they are mentioned in the table but they will later not be used for final calculation.

7.2.3.6. ^{208}Tl

Ten laboratories reported results for ^{208}Tl (Table 20, Appendix I). These data were determined by using gamma ray spectrometry and are about the same levels as ^{232}Th, ^{228}Ra, ^{228}Ac and ^{228}Th mass activities (see above), if the branching factor of 35.93% is taken into account. As mentioned above for ^{228}Ac, there is equilibrium in Thorium series (^{232}Th and its daughters ^{228}Ra, ^{228}Ac, ^{228}Th, ^{212}Bi, ^{212}Pb and ^{208}Tl). This radionuclide is frequently used as

daughter to determine indirectly ^{228}Ra activity concentration, for this reason it is mentioned in the table but it will later not be used for final data evaluation.

7.2.4. ^{40}K

Data were reported from twenty-four laboratories (Table 23, Appendix I and Fig. 19, Appendix II). The data showed good homogeneity.

7.3. LESS FREQUENTLY REPORTED RADIONUCLIDES

The results for the less frequently reported radionuclides are listed in Table 24, Appendix I.

7.3.1. ^{90}Sr

One laboratory reported three individual values for ^{90}Sr using beta technique, giving an average value of 1.2 ± 0.2 Bq kg^{-1}.

7.3.2. ^{129}I

One laboratory reported six individual values for ^{129}I using AMS technique, which gave an average value of (1.3 ± 0.04) × 10^{-5} Bq kg^{-1}.

7.3.3. ^{155}Eu

One laboratory reported three individual values for ^{155}Eu using gamma ray spectrometry technique, which gave an average value of (3.15 ± 0.50) Bq kg^{-1}.

7.3.4. ^{231}Pa, ^{223}Ra, ^{227}Th, and ^{207}Bi

Three results were reported for ^{231}Pa as well for ^{227}Th, using gamma ray spectrometry technique, giving ranges of values (from 8.7 to 10.2 Bq kg^{-1}) and (from 9.3 to 12.8 Bq kg^{-1}), respectively. Only one laboratory reported ^{223}Ra value (11.9 ± 2.4) Bq kg^{-1}. All these values are in the same range as the ^{227}Ac value (see 7.2.2.2.) and dissimilar from the precursor ^{235}U value (see 7.2.2.1. and Table 16, Appendix I), showing the disequilibrium of ^{235}U series.

One laboratory reported a ^{207}Bi value of (0.343 ± 0.039) Bq kg^{-1}, which is close to its precursor ^{235}U value (0.39 ± 0.10) Bq kg^{-1} (Table 16, Appendix I).

7.3.5. ^{224}Ra

One result using gamma ray spectrometry technique was reported for ^{224}Ra with a value of (9.2 ± 2.5) Bq kg^{-1}, which is in the same range with ^{228}Ra values (Table 19, Appendix I).

7.3.6. ^{234m}Pa

In three results reported for ^{234m}Pa, there is only one value of (20 ± 6) Bq kg^{-1}; two other results were reported as below the LLD. All participants used gamma ray spectrometry technique to determine ^{234m}Pa.

7.3.7. ^{236}U

Out of two results reported, using ICP-MS and AMS technique, only one result was above the LLD and given as (0.21 ± 0.01 Bq kg^{-1}), which is the average result of four individual values determined by AMS.

8. CONCLUSIONS

In this characterization study, the 27 selected laboratories (including IAEA-EL and five laboratories from CELLAR group) reported results of natural and anthropogenic radionuclides in a sediment sample from Bikini Atoll (IAEA-410). The robust mean mass activities for the sets of individual data were chosen as the most reliable estimates of the true values and are reported as certified and information values. The certified radionuclides include ^{40}K, ^{210}Pb (^{210}Po), ^{226}Ra, ^{228}Ra, ^{228}Th, ^{232}Th, ^{234}U, ^{238}U, ^{239}Pu, $^{239+240}Pu$ and ^{241}Am and the information values are given to other radionuclides ^{137}Cs, ^{230}Th, ^{234}Th, ^{235}U, ^{238}Pu and ^{240}Pu. The agreement between the results confirms the absence of any significant method bias (if there is more than one method used) and demonstrates the identity of the radionuclides. Radionuclides are clearly defined as total radionuclide mass fractions and independent of the measurement method. The participants used different methods for the sample preparation as well as for the final determination, demonstrating absence of measurement bias.

A summary of the certified and information values with expanded uncertainties for the most frequently reported anthropogenic and natural radionuclides could be found in the following summary table or in Table 25 and Table 26, respectively in Appendix I.

APPENDIX I. TABLES

TABLE 1. SUMMARY TABLE: CERTIFIED AND INFORMATION VALUES FOR THE IAEA-410 REFERENCE MATERIAL

(Reference date: 1 January 2013, unit: Bq kg^{-1})

Radionuclide	Certified value[a] [Bq kg^{-1}]	Expanded uncertainty[b] [Bq kg^{-1}]
^{40}K	115	6
^{210}Pb(^{210}Po)[c]	217	14
^{226}Ra	194	22
^{228}Ra	8.1	0.6
^{228}Th	8.3	1.0
^{232}Th	8.7	1.2
^{234}U	10.0	1.4
^{238}U	10.1	1.4
^{239}Pu	2.42	0.26
$^{239+240}$Pu	4.68	0.48
^{241}Am	4.12	0.28

Radionuclide	Information value [Bq kg-1]	Expanded uncertainty [Bq kg-1]
^{137}Cs	0.186	0.034
^{230}Th	4.4×10^2	0.8×10^2
^{234}Th	10.7	2.8
^{235}U	0.56	0.16
^{238}Pu	0.072	0.020
^{240}Pu	2.27	0.40

[a] The value is the robust mean (estimated in accordance with ISO 13528 [6]) of accepted sets of data. The certified values are reported on dry mass basis and are traceable to the SI.

[b] Expanded uncertainty with a coverage factor k=2 estimated in accordance with ISO 13528.

[c] ^{210}Pb and ^{210}Po were considered as in equilibrium

TABLE 2. HOMOGENEITY TESTS (*) FOR RADIONUCLIDES IN IAEA-410

Sample ID	^{40}K	^{214}Bi	^{226}Ra	$^{239+240}$Pu
1	0.89	0.85	0.92	0.75
2	0.91	0.90	0.92	0.81
3	0.91	0.90	0.92	0.89
4	0.92	0.90	0.94	0.94
5	0.94	0.91	0.95	0.99
6	0.94	0.95	0.97	1.05
7	0.96	0.95	0.97	1.06
8	0.96	0.95	0.97	1.06
9	0.96	0.95	0.98	1.18
10	0.97	0.96	0.98	1.26
11	0.98	0.97	0.98	
12	0.98	0.98	1.00	
13	0.98	0.98	1.00	
14	0.98	0.98	1.00	
15	1.00	0.99	1.01	
16	1.00	0.99	1.01	
17	1.01	1.00	1.01	
18	1.02	1.02	1.02	
19	1.02	1.02	1.02	
20	1.03	1.03	1.02	
21	1.03	1.05	1.03	
22	1.03	1.05	1.03	
23	1.04	1.05	1.03	
24	1.04	1.06	1.04	
25	1.05	1.06	1.04	
26	1.05	1.06	1.04	
27	1.05	1.07	1.04	
28	1.07	1.08	1.05	
29	1.07	1.09	1.06	
30	1.08	1.09	1.07	
21	1.10	1.10	1.07	
32	1.10	1.12	1.07	
33	1.12	1.13	1.08	
Minimum	0.89	0.85	0.92	0.75
Maximum	1.12	1.13	1.08	1.26
Mean	1.00	1.00	1.00	1.00
Median	1.01	1.00	1.01	1.02
Std. Dev.	0.09	0.10	0.06	0.15
Coef. Var. (%)	9	10	6	15

(*) Normalized activity =x/X (individual/mean values). The homogeneity test was performed in RML (organizer of this characterization study) for different radionuclides using different techniques such as gamma-, alpha- and beta spectrometry as well as mass spectrometry, before dispatch of samples (see more detail in homogeneity test).

TABLE 3. RADIONUCLIDES REPORTED FOR IAEA-410

Radionuclide	Number of data reported	Radionuclide	Number of data reported
^{40}K	71	^{228}Ac	71
^{90}Sr	3	^{228}Th	55(1)
^{129}I	6	^{230}Th	16
^{137}Cs	59(35)	^{232}Th	21
^{208}Tl	66	^{234}Th	24(2)
^{210}Pb(^{210}Po)	71	^{234}U	52(1)
^{212}Pb	38	^{235}U	88(10)
^{212}Bi	37	^{238}U	80(3)
^{214}Pb	67	^{238}Pu	21
^{214}Bi	76	^{239}Pu	17
^{224}Ra	1	^{240}Pu	17
^{226}Ra	114	$^{239+240}$Pu	58
^{228}Ra	76	^{241}Am	57
^{227}Ac	18	^{241}Am (gamma)	46

(*) “Less than” values are shown in parentheses

TABLE 4. RESULTS FOR ^{137}Cs IN IAEA-410
(Reference date: 1 January 2013, unit: Bq kg^{-1})

Lab Code	Method code	No. of results	Mass (g)	^{137}Cs
1	G	3	100.44	0.20 ± 0.30
2	G	2	81.55	<0.9
4	G	3	99.9	0.17 ± 0.07
7	G	3	66.84	0.24 ± 0.43
8	G	5	30	<0.38
9	G	3	99.61	<0.3
10	G	1	41	<0.4
11	G	3	64	<1.2
12	G	10	70.1	<0.5
13	G	3	80	<0.82
14	G	3	86.74	<0.7
17	G	4	14–80	<0.4
21	G	1	4.59	0.16 ± 0.02
23	G	1	100	0.20 ± 0.12
26	G	14	60	0.155 ± 0.096

Number of reported laboratory means	6
Robust mean	0.186
Expanded uncertainty	0.034

TABLE 5. RESULTS FOR ^{238}Pu AND $^{239+240}$Pu IN IAEA-410
(Reference date: 1 January 2013; unit: Bq kg^{-1})

Lab.	Method code	No. of results	Mass (g)	^{238}Pu	$^{239+240}$Pu
3	A	3	1.8–2.4	–	3.94 ± 0.44
5	A	3	0.25	–	6.18 ± 1.68
6	A	2	5	0.084 ± 0.042	4.27 ± 0.42
7	A	3	10		2.60 ± 0.51
7	ICP-MS	3	10		2.38 ± 0.20
9	A	3	5	0.05 ± 0.01	4.40 ± 0.26
10	ICP-MS	1	14.3	0.075 ± 0.014	4.76 ± 0.39
10	A	1	14	0.075 ± 0.014	4.87 ± 0.19
11	A	3	30	0.052 ± 0.035	4.30 ± 0.24
12	A	4	3	0.02; 0.05; 0.062	4.52 ± 0.18^
12	A	6	0.5–1	–	4.92 ± 0.28
13	A	3	5.1–5.2	–	3.42 ± 0.59
14	A	3	5–9	–	4.42 ± 0.36
15	A	1	20.21	0.084 ± 0.015	5.87 ± 1.93
17	ICP-MS	3	1	±	4.68 ± 1.18
18	A	3		±	6.10 ± 1.60
19	A	3	10	±	4.60 ± 0.20
24	AMS	5	0.5	–	5.35 ± 0.22
26a	A	2	10	0.055 ± 0.014	5.33 ± 0.29
26b	AMS	5	0.5	–	4.70 ± 0.37
26c	A	4	3–10	0.101 ± 0.016	5.63 ± 0.53
Number of reported laboratory means				10	21
Robust mean				0.072	4.68
Expanded uncertainty				0.020	0.48

Note: Tables 5–24:

—: data not available.

(^) Laboratory reported that the sample is inhomogeneous for Pu isotopes when 9 aliquots of samples at different masses (0.5; 1; 3; 5 g) were analysed using normal/partly leaching digestion technique (as requested by laboratory, the data here is the mean value of 4 homogeneous values done for 3 g only). However, the second analysis of sample (6 aliquots of 0.5–1g) using total digestion with HF/HNO_3 and boric acids/HCl gave homogeneous data, resulting that the non-homogeneity of samples could be due to the leaching techniques (which is confirmed by IAEA and CNA in Seville for further investigation using two different leaching methods and then measured sample by AMS technique, see detail in the report).

TABLE 6. RESULTS FOR ^{239}Pu AND ^{240}Pu IN IAEA-410
(Reference date: 1 January 2013; unit: Bq kg^{-1})

Lab.	Method code	No. of results	Mass (g)	^{239}Pu	^{240}Pu
7	ICP-MS	3	10	1.22 ± 0.04	1.16 ± 0.05
10	ICP-MS	1	14.3	2.42 ± 0.28	2.34 ± 0.28
17	ICP-MS	3	1	2.45 ± 0.75	2.23 ± 0.50
24	AMS	5	0.5	2.70 ± 0.14	2.65 ± 0.11
26a	AMS	2	0.5	2.27 ± 0.18	2.09 ± 0.24
26b	AMS	3	0.5	2.59 ± 0.25	2.60 ± 0.25
Number of reported laboratory means				6	6
Robust mean				2.42	2.27
Expanded uncertainty				0.26	0.40

TABLE 7. RESULTS FOR ^{241}Am IN IAEA-410
(Reference date: 1 January 2013, unit: Bq kg^{-1})

Lab Code	Method code	No. of results	Mass (g)	^{241}Am
1	G	3	100.44	3.10 ± 0.30
2	G	2	81.55	3.70 ± 0.70
3	G	1	98.14	3.40 ± 1.00
4	G	3	99.9	4.13 ± 0.31
5	G	5	65	3.80 ± 1.10
6	G	2	99.68	4.80 ± 0.88
9	G	3	99.61	3.74 ± 0.58
9	A	3	5	3.90 ± 0.26
10	G	1	41	4.10 ± 0.50
10	A	1	14	3.77 ± 0.30
11	A	3	30	3.60 ± 0.30
12	A	3	3	4.13 ± 0.26^
12	A	6	0.5–1	4.38 ± 0.28
12	G	3	70.1	4.04 ± 0.62
13	G	3	80	4.80 ± 0.70
16	G	2	27	4.15 ± 1.15
17	G	4	14–80	4.90 ± 1.60
18	G	4	53.8	4.10 ± 0.50
19	G	3	25	3.70 ± 0.40
19	A	1	10	4.00 ± 0.50
20	G	3	5.23	5.10 ± 0.60
25	G	2	61.42	4.80 ± 1.10
26	A	2	10	4.47 ± 0.13

Number of reported laboratory means	23
Robust mean	4.12
Expanded uncertainty	0.28

(^) Laboratory reported that the sample is inhomogeneous for Am isotope when 9 aliquots of samples at different masses (0.5; 1; 3; 5 g) were analysed using normal/partly leaching digestion technique (as requested by laboratory, the data here is the mean value of 4 homogeneous values done for 3g only). However, the second analysis of sample (6 aliquots of 0.5–1g) using total digestion with HF/HNO_3 and boric acids/HCl gave homogeneous data, indicating that the non-homogeneity of samples could be due to the leaching techniques, which confirm the above observation for Pu results.

TABLE 8. RESULTS FOR ^{241}Am IN IAEA-410 (GAMMA AND ALPHA DATA GIVEN SEPARATELY)
(Reference date: 1 January 2013, unit: Bq kg^{-1})

Lab.	Method code	No. of results	Mass (g)	^{241}Am Gamma data	^{241}Am Alpha data
1	G	3	100.44	3.10 ± 0.30	—
2	G	2	81.55	3.70 ± 0.70	—
3	G	1	98.14	3.40 ± 1.00	—
4	G	3	99.9	4.13 ± 0.31	—
5	G	5	65	3.80 ± 1.10	—
6	G	2	99.68	4.80 ± 0.58	—
9	G, (A)	3, (3)	99.61 (5)	3.74 ± 0.58	3.90 ± 0.26
10	G, (A)	1, (1)	41, (14)	4.10 ± 0.50	3.77 ± 0.30
11	A	3	30	—	3.60 ± 0.30
12	G, (A)	1, (3)	70.1, (3)	4.04 ± 0.62	4.13 ± 0.26
12	A	6	0.5–1	—	4.38 ± 0.28
13	G	3	80	4.80 ± 0.70	—
16	G	2	27	4.15 ± 1.15	—
17	G	4	14-80	4.90 ± 1.60	—
18	G	4	53.8	4.10 ± 0.50	—
19	G, (A)	3, (1)	25, (10)	3.90 ± 0.70	4.00 ± 0.50
20	G	3	5.23	5.10 ± 0.60	—
25	G	2	61.42	4.80 ± 1.10	—
26	A	2	10	—	4.47 ± 0.13
Number of reported laboratory means				16	7
Robust mean				4.16	4.04
Expanded uncertainty				0.38	0.34

TABLE 9. RESULTS FOR ^{238}U IN IAEA-410
(Reference date: 1 January 2013, unit: Bq kg^{-1})

Lab code	Method code	No. of results	Mass (g)	^{238}U
3	A	3	0.635–0.797	16.8 ± 1.7
4	G	3	99.9	7.9 ± 1.8
5	A	4	0.25	8.1 ± 1.9
6	A	2	5	6.92 ± 0.15
7	A	3	2	9.3 ± 1.3
9	G	3	99.61	<175
9	G	3	99.61	10.9 ± 1.0
10	ICP-MS	3	0.29–0.6	9.0 ± 1.0
12	A	3	0.5	13.3 ± 1.7
13	A	8	2.53	10.1 ± 0.6
17	ICP-MS	2	1	9.8 ± 2.1
18	A	3	—	8.2 ± 1.6
18	G	1	53.8	10.0 ± 4.0
19	A	6	0.5–10	11.2 ± 0.8
21a	G	9	100.5	16.0 ± 2.0
21b	G	9	100.5	20.0 ± 6.0
25	G	2	61.42	8.9 ± 2.6
26a	A	4	3–10	12.0 ± 1.2
26b	ICP-MS	4	0.15	8.11 ± 1.14
27	ICP-MS	9	0.5	8.45 ± 0.08

Number of reported laboratory means	19
Robust mean	10.1
Expanded uncertainty	1.4

TABLE10. RESULTS FOR ^{234}Th IN IAEA-410
(Reference date: 1 January 2013, unit: Bq kg^{-1})

Lab code	Method code	No. of results	Mass (g)	^{234}Th
2	G	2	81.55	<11
4	G	3	99.9	7.9 ± 1.8
9	G	3	99.61	10.9 ± 1.0
10	G	1	41	8.7 ± 1.8
16	G	2	27	12.2 ± 3.9
18	G	4	53.8	10 ± 4
21	G	9	100.5	16 ± 2
Number of reported laboratory means				6
Robust mean				10.7
Expanded uncertainty				2.8

TABLE 11. RESULTS FOR ^{234}U IN IAEA-410
(Reference date: 1 January 2013, unit: Bq kg^{-1})

Lab code	Method code	No. of results	Mass (g)	^{234}U
3	A	3	0.635–0.797	16.7 ± 1.2
5	A	4	0.25	8.5 ± 2.0
6	A	2	5	7.64 ± 0.22
7	A	3	2	9.3 ± 1.7
10	ICP-MS	3	0.29–0.6	9.2 ± 1.2
10	G	1	41	<600
12	A	3	0.5	13.2 ± 1.7
13	A	8	2.53	10.5 ± 0.6
17	A	3	1	7.8 ± 3.2
18	A	3	—	9.7 ± 2.0
19	A	6	0.5–10	11.1 ± 0.8
26	A	4	3–10	12.2 ± 1.5
27	ICP-MS	9	0.5	8.77 ± 0.09
Number of reported laboratory means				12
Robust mean				10.0
Expanded uncertainty				1.4

TABLE 12. RESULTS FOR ^{230}Th IN IAEA-410
(Reference date: 1 January 2013, unit: Bq kg^{-1})

Lab code	Method code	No. of results	Mass (g)	^{230}Th
3	A	3	0.635–0.797	399 ± 12
4	G	3	99.9	483 ± 35
7	A	3	2	478 ± 29
10	G	1	41	510 ± 80
18	G	4	53.8	420 ± 50
26	A	2	4.33; 4.75	358 ± 44
Number of reported laboratory means				6
Robust mean				4.4×10^2
Expanded uncertainty				0.8×10^2

TABLE 13. RESULTS FOR ^{226}Ra IN IAEA-410
(Reference date: 1 January 2013, unit: Bq kg^{-1})

Lab code	Method code	No. of results	Mass (g)	^{226}Ra
1	G	3	100.44	161 ± 2
2	G	2	85.11	148 ± 12
3	A	3	0.635–0.797	184 ± 18
3	G	1	98.14	204 ± 25
4	G	3	99.9	244 ± 14
6	G	4	99.68	179 ± 14
7	A	3	1	222 ± 8
8	G	5	30	216 ± 12
9	G	3	99.61	271 ± 7
10	G	1	41	253 ± 26
12	G	10	70.1	115 ± 3
13	G	3	80	190 ± 12
14	G	3	86.74	220 ± 12
15	G	1	170.5	210 ± 18
17	G	4	14–80	152 ± 31
18	G	4	53.8	233 ± 13
20	G	3	5.23	217 ± 1
21a	G	9	100.5	120 ± 2
21b	G	9	100.5	125 ± 2
23	G	1	10	189 ± 10
25	G	2	61.42	199 ± 12
26	G	33	60	185 ± 12
Number of reported laboratory means				22
Robust mean				194
Expanded uncertainty				22

TABLE 14. RESULTS FOR ^{214}Pb and ^{214}Bi IN IAEA-410
(Reference date: 1 January 2013, unit: Bq kg^{-1})

Lab. code	Method code	No. of results	Mass (g)	^{214}Pb	^{214}Bi
1	G	3	100.44	162 ± 2	162 ± 2
2	G	2	81.55	157 ± 18	139 ± 16
5	G	3	65	–	136 ± 7
7	G	3	66.84	–	215 ± 4
9	G	3	99.61	196 ± 1	158 ± 1
13	G	3	80	194 ± 12	186 ± 12
15	G	1	170.5	218 ± 13	202 ± 13
16	G	2	27	158 ± 17	119 ± 11
18	G	4	53.8	233 ± 13	233 ± 13
19	G	3	25	137 ± 9	139 ± 10
20	G	3	5.23	–	212 ± 7
21	G	9	100.5	125 ± 2	120 ± 2
26	G	33	60	215 ± 3	171 ± 3
Number of reported laboratory means				10	13
Robust mean				179	168
Expanded uncertainty				33	28

TABLE 15. RESULTS FOR ^{210}Pb (^{210}Po) IN IAEA-410
(Reference date: 1 January 2013, unit: Bq kg^{-1})

Lab code	Method code	No. of results	Mass (g)	^{210}Pb(^{210}Po)[a]
1	G	3	100.44	168 ± 4
2	G	2	81.55	200 ± 23
3	G	1	98.14	236 ± 39
3 (Po, A)	A	3	0.635–0.797	242 ± 4
4	G	2	99.9	226 ± 17
5	G	5	65	205 ± 15
7 (Po)	A	3	1	232 ± 13
7	A	3	1	221 ± 28
8	G	5	30	142 ± 18
9	G	3	99.61	161 ± 5
10	G	1	41	238 ± 22
12	G	10	70.1	240 ± 16
12	A	3	0.5–1	267 ± 15
12 (Po, A)	A	3	0.5–1	238 ± 18
13	G	3	80	224 ± 17
14	G	3	86.74	149 ± 12
17	G	2	14;17	201 ± 40
18	G	4	53.8	205 ± 14
18 (Pb)	A	3	—	230 ± 50
18 (Po)	A	3	—	170 ± 30
19	G	3	25	184 ± 18
20	G	3	5.23	236 ± 20[b]
23	G	1	100	239 ± 12[b]
25	G	2	61.42	242 ± 28
26	G	16	60	216 ± 9

Number of reported laboratory means	25
Robust mean	217
Expanded uncertainty	14

[a] ^{210}Pb and ^{210}Po were considered as in equilibrium, and the ^{210}Pb values are corrected for reference date at 1 January 2013.
[b] Value is corrected by IAEA for the reference date.

TABLE 16. RESULTS FOR ^{235}U IN IAEA-410
(Reference date: 1 January 2013, unit: Bq kg^{-1})

Lab code	Method code	No. of results	Mass (g)	^{235}U
1	G	3	100.44	5.80 ± 0.40
3	A	3	0.635–0.797	0.61 ± 0.17
5	A	4	0.25	0.81 ± 0.98
6	A	2	5	0.26 ± 0.03
7	A	3	1	0.31 ± 0.04
9	G	3	99.61	<0.4
10	ICP-MS	3	0.29–0.6	0.42 ± 0.05
10	G	1	41	<18
12	A	3	0.5–1	<0.21
13	A	8	2.53	0.43 ± 0.10
17	ICP-MS	10	1	0.45 ± 0.10
18	A	3	—	0.39 ± 0.08
21a	G	9	100.5	9.30 ± 0.30
21b	G	9	100.5	5.10 ± 0.20
21c	G	9	100.5	11.0 ± 0.5
25	G	2	61.42	4.70 ± 1.00
26	A	4	3–10	0.30 ± 0.04
27	ICP-MS	9	0.5	0.39 ± 0.003

Number of reported laboratory means	15
Robust mean	0.56
Expanded uncertainty	0.16

TABLE 17. RESULTS FOR ^{227}Ac IN IAEA-410
(Reference date: 1 January 2013, unit: Bq kg^{-1})

Lab code	Method code	No. of results	Mass (g)	^{227}Ac
4	G	3	99.9	12.2 ± 1.0
4	G	3	99.9	11.2 ± 0.8
6	G	4	99.68	13.2 ± 1.6
9	G	2	0.25	12.2 ± 1.0
18	G	4	53.8	12.4 ± 0.9
25	G	2	61.42	13.2 ± 2.0
Number of reported laboratory means				6
Robust mean				12.4
Expanded uncertainty				0.9

TABLE 18. RESULTS FOR ^{232}Th IN IAEA-410
(Reference date: 1 January 2013, unit: Bq kg^{-1})

Lab code	Method code	No. of results	Mass (g)	^{232}Th
3	A	3	0.635–0.797	10.0 ± 1.2
7	A	3	2	8.4 ± 1.0
13	G	3	80	9.1 ± 1.2
17	G	4	14–80	8.5 ± 1.7
17	ICP-MS	3	1	9.2 ± 2.0
18	A	3	—	10.0 ± 2.0
23	G	1	100	6.9 ± 0.8
26	A	4	3–10	7.3 ± 2.0
Number of reported laboratory means				8
Robust mean				8.7
Expanded uncertainty				1.2

TABLE 19. RESULTS FOR ^{228}Ra IN IAEA-410
(Reference date: 1 January 2013, unit: Bq kg^{-1})

Lab code	Method code	No. of results	Mass (g)	^{228}Ra
2	G	2	81.55	7.5 ± 2.0
3	G	1	98.14	6.6 ± 2.2
4	G	3	99.9	7.6 ± 0.4
6	G	4	99.68	9.2 ± 1.8
9	G	3	99.61	8.3 ± 1.2
12	G	10	70.1	6.5 ± 1.4
13	G	3	80	9.8 ± 1.2
15	G	1	170.5	10.0 ± 2.7
18	G	4	53.8	8.3 ± 1.1
20	G	3	5.23	9.3 ± 1.9
21	G	9	100.5	7.2 ± 0.2
25	G	2	61.42	7.5 ± 2.2
26	G	31	60	7.0 ± 1.8
Number of reported laboratory means				13
Robust mean				8.1
Expanded uncertainty				0.6

TABLE 20. RESULTS FOR ^{228}Ac and ^{208}Tl IN IAEA-410
(Reference date: 1 January 2013, unit: Bq kg^{-1})

Lab code	Method code	No. of results	Mass (g)	^{228}Ac	^{208}Tl
1	G	3	100	7.7 ± 1.5	2.2 ± 0.3$^{\$}$
2	G	2	81.55	7.5 ± 2.0	2.3 ± 0.5
4	G	3	99.9	7.7 ± 0.4	—
5	G	3	65	7.7 ± 1.2	2.4 ± 0.5
7	G	3	66.84	8.5 ± 1.3	2.8 ± 0.5
9	G	3	99.61	8.3 ± 1.2	2.4 ± 0.2$^{\$}$
10	G	1	41	7.9 ± 1.2	2.8 ± 0.4
13	G	8	30	9.8 ± 1.2	2.9 ± 0.3$^{\$}$
16	G	2	27	8.6 ± 4.1	—
18	G	3	53.8	8.3 ± 1.1	3.0 ± 0.2$^{\$}$
21	G	9	100.5	7.2 ± 0.2	2.4 ± 0.1$^{\$}$
26	G	31	60	7.0 ± 1.8	2.5 ± 0.6$^{\$}$
Number of reported laboratory means				12	10
Robust mean				7.9	2.5
Expanded uncertainty				0.4	0.2

($^{\$}$) the values corrected (by RML) for branching factor of 35.93%

TABLE 21. RESULTS FOR ^{228}Th IN IAEA-410
(Reference date: 1 January 2013, unit: Bq kg^{-1})

Lab code	Method code	No. of results	Mass (g)	^{228}Th
6	G	4	99.68	8.2 ± 2.9
7	A	3	2	9.9 ± 2.0
9	G	3	99.61	8.6 ± 0.3
10	G	1	41	<70
15	G	1	170.5	9.6 ± 2.9
17	A	3	1	9.5 ± 2.6
18	G	4	53.8	8.3 ± 0.6
20	G	3	5.23	8.4 ± 0.4
21a	G	9	100.5	6.6 ± 0.2
21b	G	9	100.5	7.0 ± 0.4
21c	G	9	100.5	6.6 ± 0.2
25	G	2	61.42	8.0 ± 0.6
26	A	4	3–10	6.7 ± 2.2
Number of reported laboratory means				12
Robust mean				8.3
Expanded uncertainty				1.0

TABLE 22. RESULTS FOR ^{212}Pb and ^{212}Bi IN IAEA-410
(Reference date: 1 January 2013, unit: Bq kg^{-1})

Lab code	Method code	No. of results	Mass (g)	^{212}Pb	^{212}Bi
1	G	3	100.44	7.0 ± 0.8	—
9	G	3	99.61	8.6 ± 0.3	10.5 ± 3.9
10	G	1	41	8.2 ± 0.9	8.3 ± 2.2
13	G	3	80	9.4 ± 0.8	12.6 ± 2.9
15	G	1	170.5	9.2 ± 0.7	9.9 ± 2.9
16	G	2	27	9.4 ± 1.6	—
18	G	4	53.8	—	8.3 ± 0.6
21	G	9	100.5	6.6 ± 0.2	7.0 ± 0.4
26	G	16	60	9.2 ± 0.4	4.5 ± 1.1
Number of reported laboratory means				8	7
Robust mean				8.7	8.7
Expanded uncertainty				0.7	1.5

TABLE 23. RESULTS FOR ^{40}K IN IAEA-410
(Reference date: 1 January 2013, unit: Bq kg^{-1})

Lab code	Method code	No. of results	Mass (g)	^{40}K
1	G	3	100.44	125 ± 8
2	G	2	81.55	90 ± 15
3	G	1	98.14	113 ± 19
4	G	3	99.9	109 ± 5
5	G	3	65	109 ± 15
6	G	2	99.68	131 ± 21
7	G	3	66.84	123 ± 5
8	G	5	30	100 ± 19
9	G	3	99.61	116 ± 5
10	G	1	41	116 ± 13
11	G	3	83.6	100 ± 3
12	G	10	70.1	120 ± 7
13	G	3	80	118 ± 10
14	G	3	86.74	128 ± 18
15	G	1	170.5	117 ± 6
16	G	2	27	110 ± 17
17	G	2	80	107 ± 15
18	G	4	53.8	117 ± 12
19	G	3	25	106 ± 14
20	G	4	5.23	129 ± 13
21	G	9	100.5	94 ± 3
23	G	1	100	115 ± 6
25	G	2	61.42	121 ± 8
26	G	33	60	121 ± 8

Number of reported laboratory means	24
Robust mean	115
Expanded uncertainty	6

TABLE 24. RESULTS FOR THE LESS FREQUENTLY MEASURED RADIONUCLIDES REPORTED IN IAEA-410

(Reference date: 1 January 2013, unit: Bq kg^{-1})

Isotope	Lab. code	Method code	No. of results	Mass (g)	Activity (Bq kg^{-1})
^{60}Co	9	G	3	99.61	<0.4
^{90}Sr	3	B	3	30	1.2 ± 0.2
^{129}I	22	AMS	6	0.5–5	1.3×10^{-5} ± 0.04×10^{-5}
^{155}Eu	9	G	3	99.61	3.15 ± 0.50
^{207}Bi	4	G	3	99.9	0.343 ± 0.039
^{223}Ra	10	G	1	41	11.9 ± 2.4
^{224}Ra	10	G	1	41	9.2 ± 2.5
^{227}Th	9	G	3	99.61	12.2 ± 1.0
-	10	G	1	41	12.8 ± 2.1
-	21	G	9	100.5	9.3 ± 0.3
^{231}Pa	10	G	1	41	10.2 ± 3.1
-	18	G	4	53.8	13 ± 5
-	25	G	2	61.42	8.7 ± 1.8
^{234m}Pa	9	G	3	99.61	<175
-	10	G	1	41	<50
-	21	G	9	100.5	20 ± 6
^{236}U	10	ICP-MS	3	0.29–0.6	<0.0013
	24	ICP-MS	4	0.5	0.21 ± 0.01

TABLE 25. SUMMARY OF CERTIFIED VALUES FOR IAEA-410
(Reference date: 1 January 2013, unit: Bq kg^{-1})

Radionuclide	Certified value[a] [Bq kg^{-1}]	Expanded uncertainty[b] [Bq kg^{-1}]	N[c]
^{40}K	115	6	24
^{210}Pb(^{210}Po)[d]	217	14	25
^{226}Ra	194	22	22
^{228}Ra	8.1	0.6	13
^{228}Th	8.3	1.0	13
^{232}Th	8.7	1.2	8
^{234}U	10.0	1.4	12
^{238}U	10.1	1.4	19
^{239}Pu	2.42	0.26	6
$^{239+240}$Pu	4.68	0.48	21
^{241}Am	4.12	0.28	23

Note: Tables 25–26:

[a] The value is the robust mean (estimated in accordance with the ISO 13528) of accepted sets of data, each set being obtained by different laboratory. The certified values are reported on dry mass basis and are traceable to the SI.

[b] Expanded uncertainty with a coverage factor k=2 estimated in accordance with the ISO 13528.

[c] Number of accepted data for evaluation.

[d] ^{210}Pb and ^{210}Po were considered as in equilibrium.

TABLE 26. SUMMARY OF INFORMATION VALUES FOR IAEA-410
(Reference date: 1 January 2013, unit: Bq kg^{-1})

Radionuclide	Information value [Bq kg^{-1}]	Expanded uncertainty [Bq kg^{-1}]	N
^{137}Cs	0.186	0.034	6
^{230}Th	4.4×10^2	0.8×10^2	6
^{234}Th	10.7	2.8	6
^{235}U	0.56	0.16	15
^{238}Pu	0.072	0.020	10
^{240}Pu	2.27	0.40	6

APPENDIX II.
LABORATORY RESULTS – GRAPHS

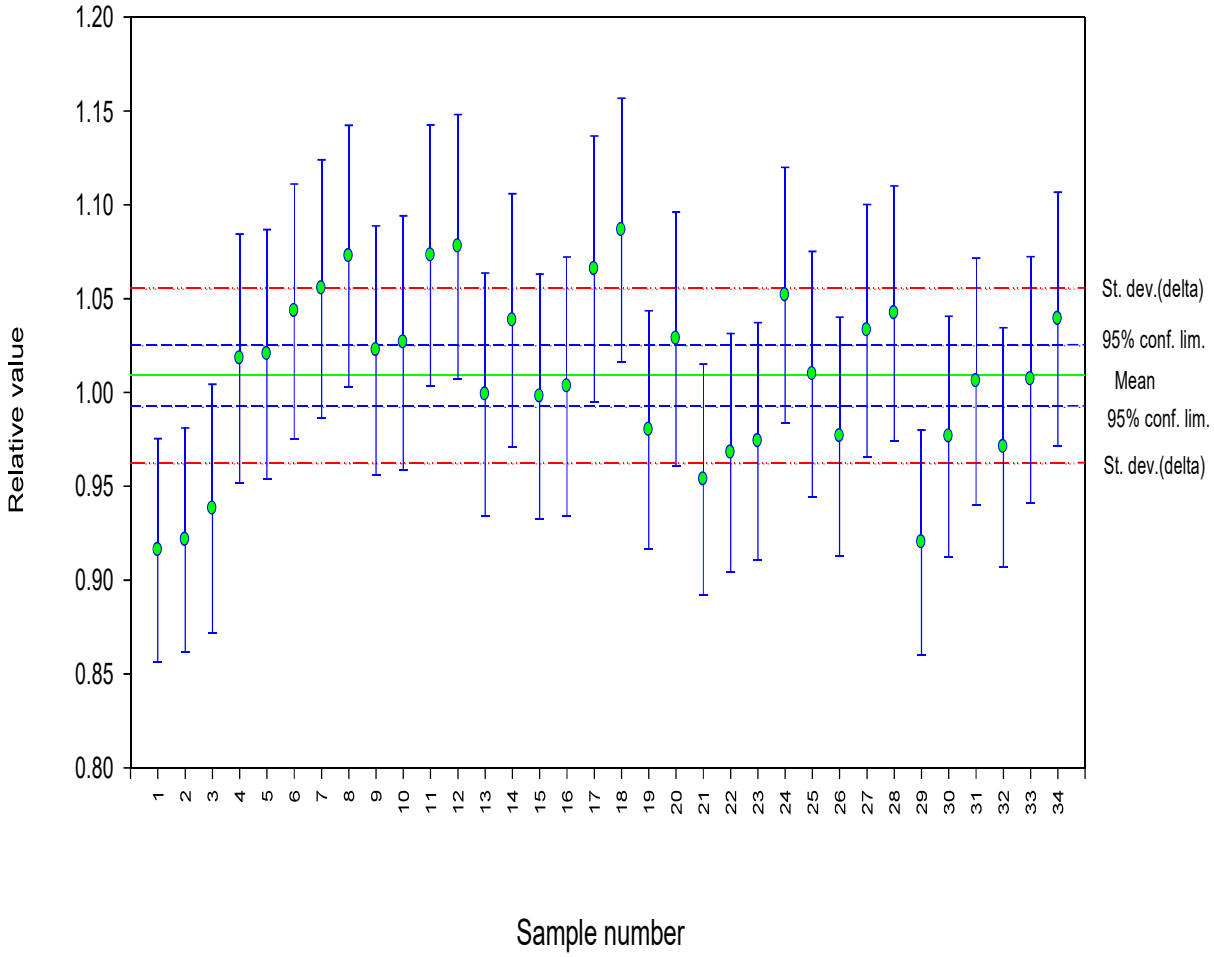

FIG.1. Homogeneity test for ^{226}Ra in IAEA-410

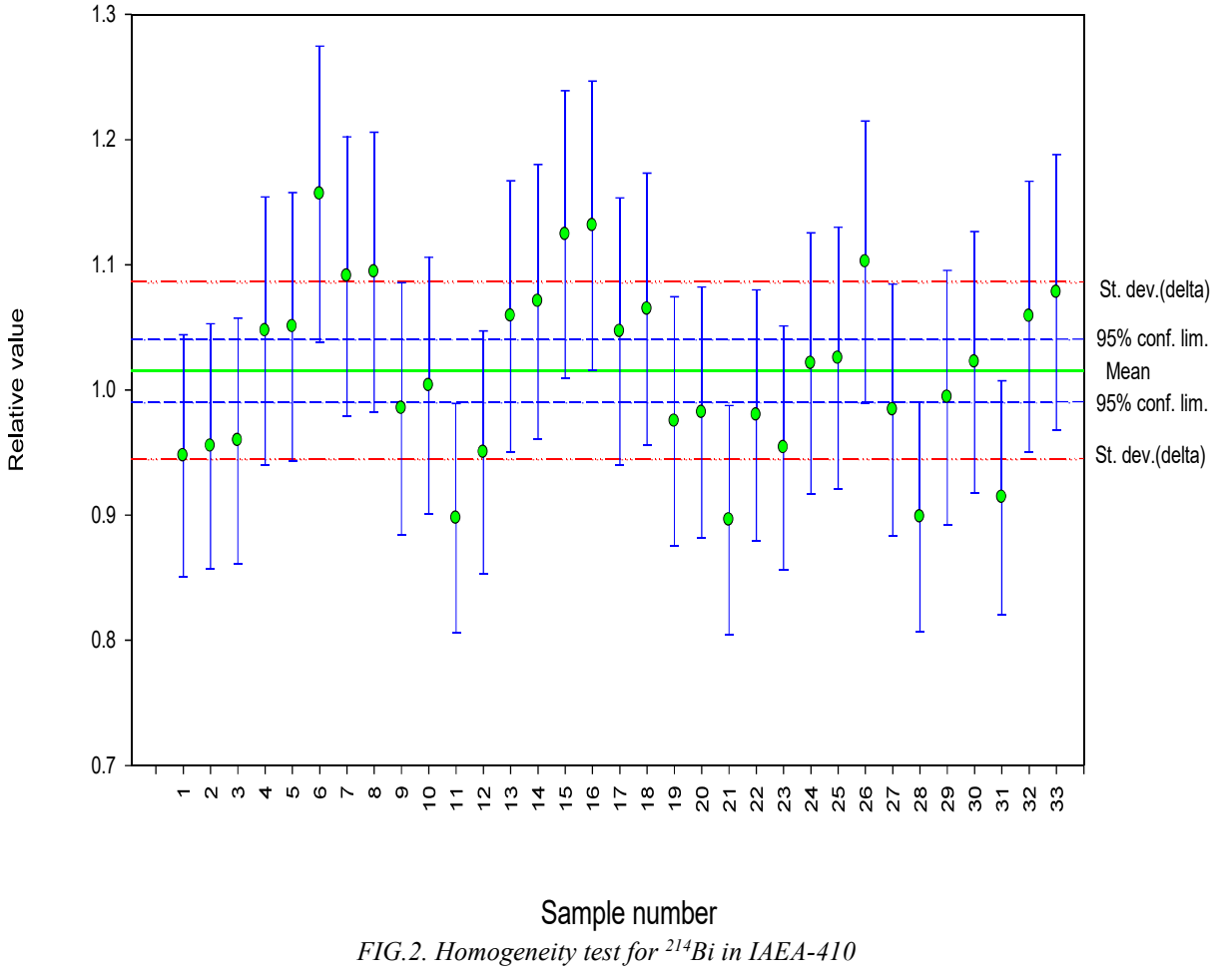

FIG.2. Homogeneity test for ^{214}Bi in IAEA-410

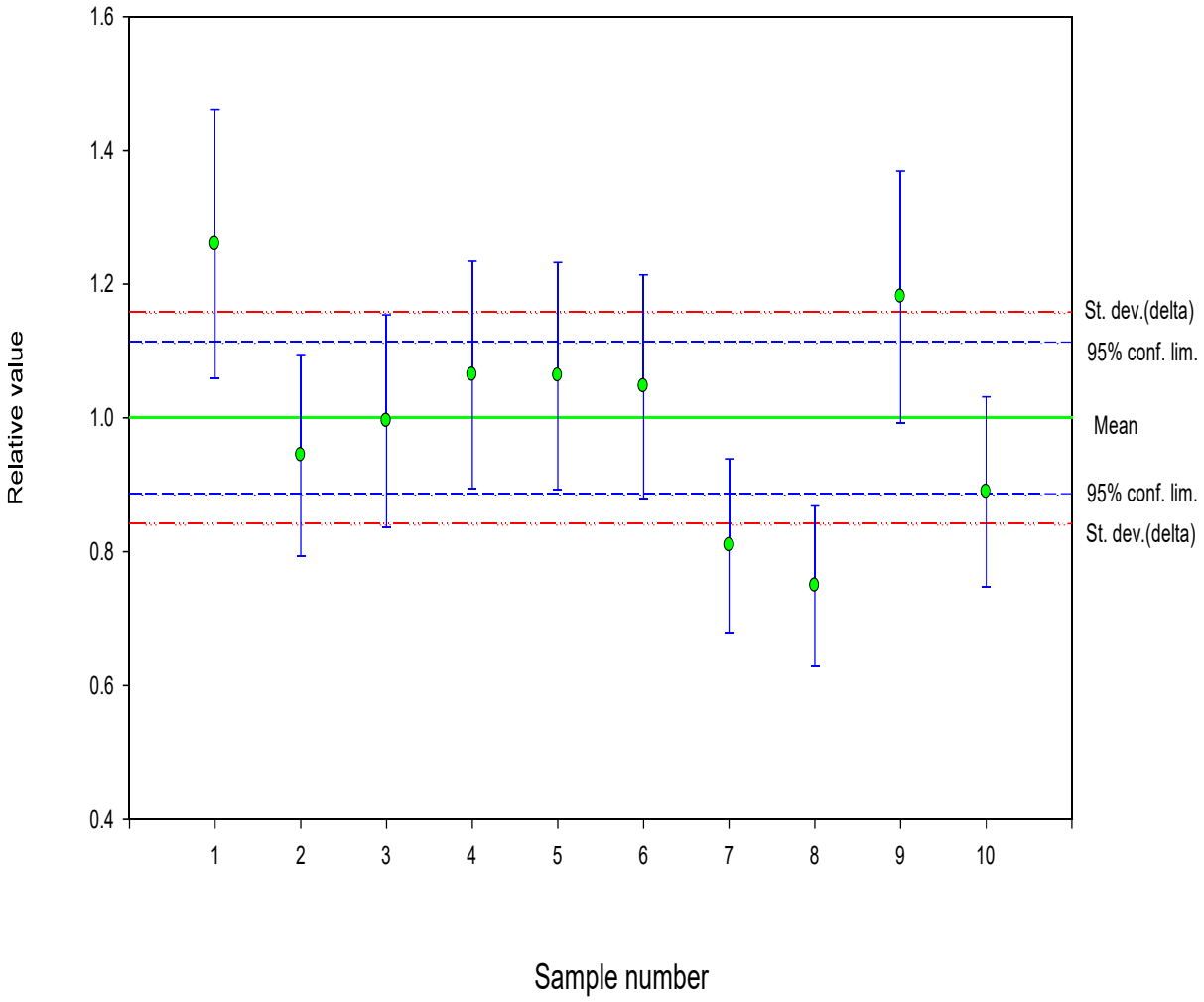

FIG.3. Homogeneity test for $^{239+240}Pu$ in IAEA-410

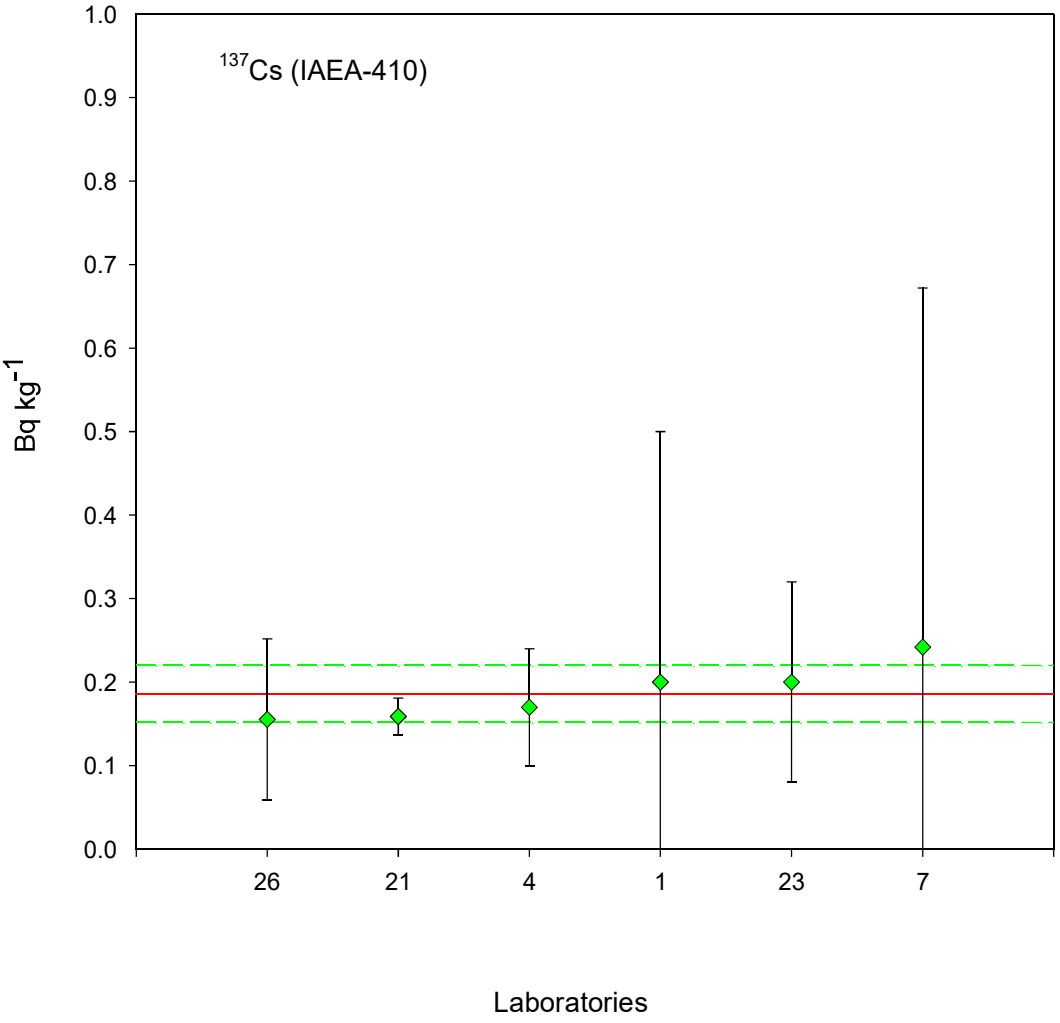

FIG. 4. Laboratory results for ^{137}Cs

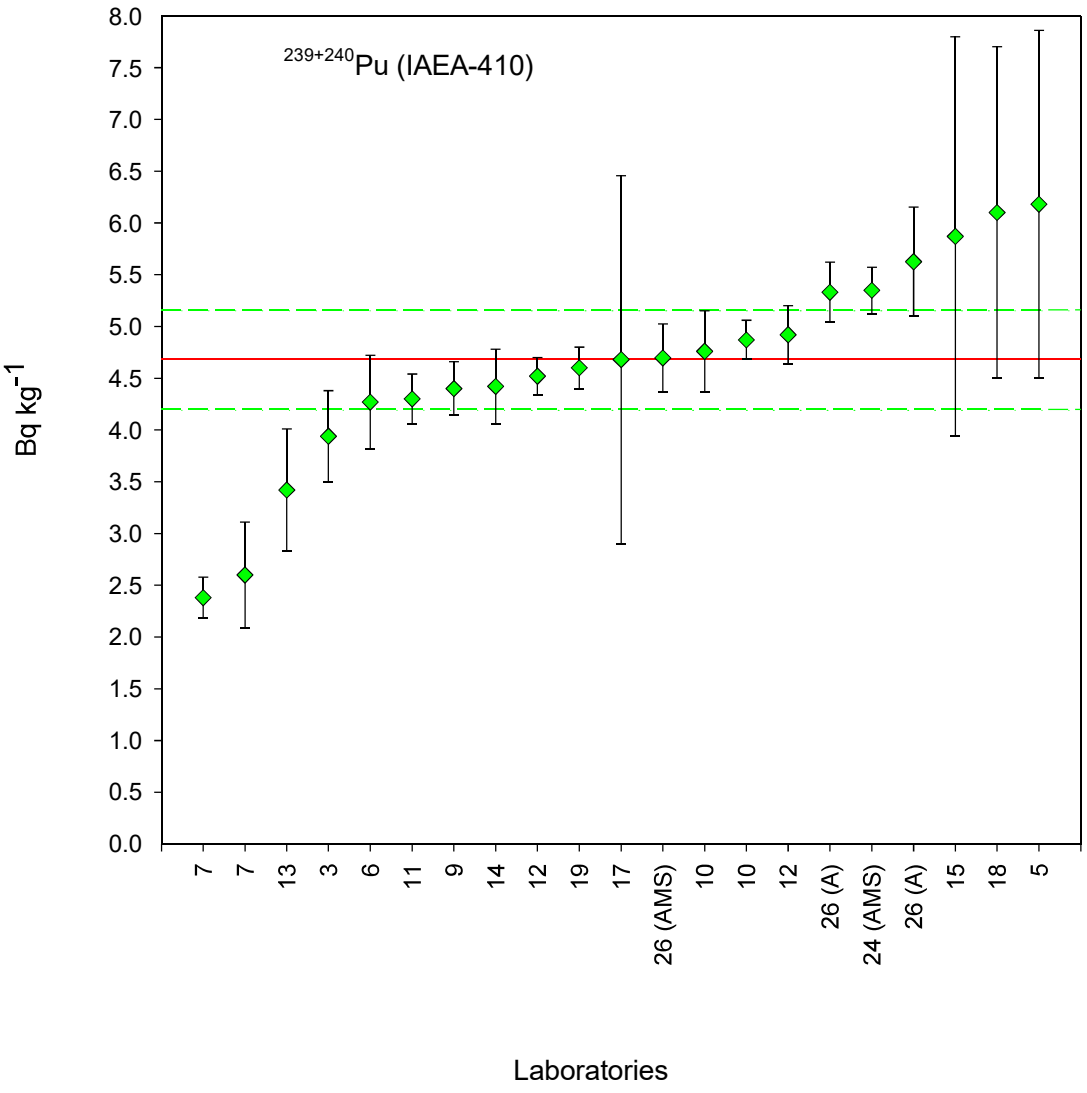

FIG.5. Laboratory results for $^{239+240}Pu$

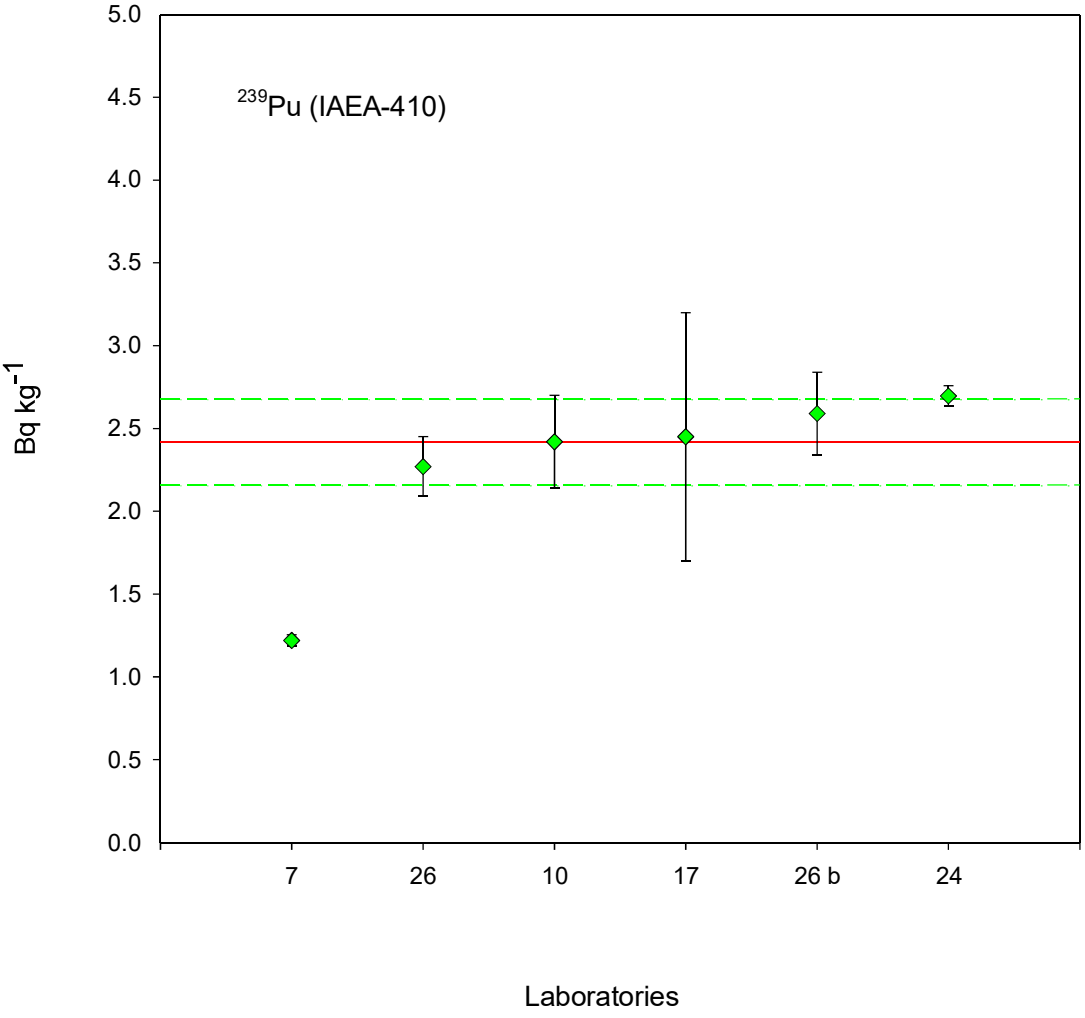

FIG.6. Laboratory results for ^{239}Pu

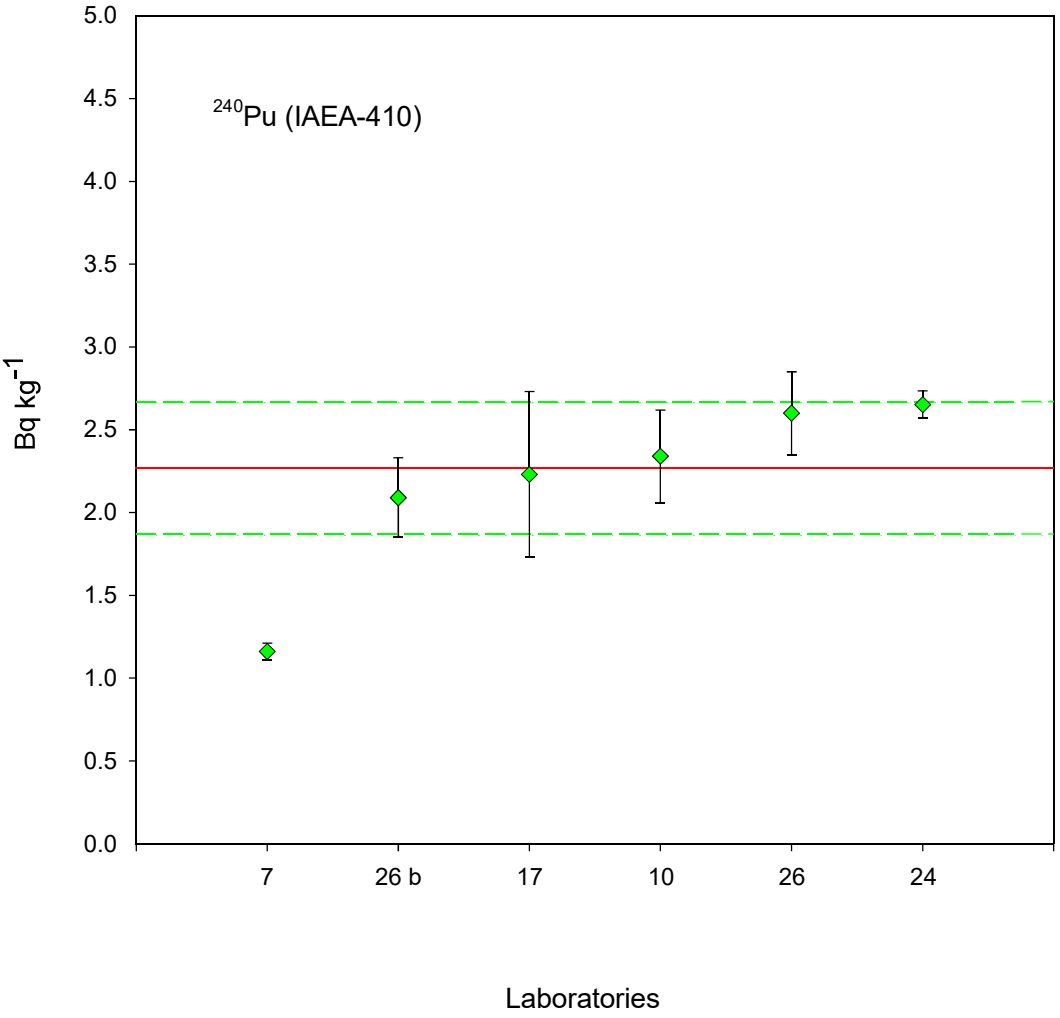

FIG. 7. Laboratory results for ^{240}Pu

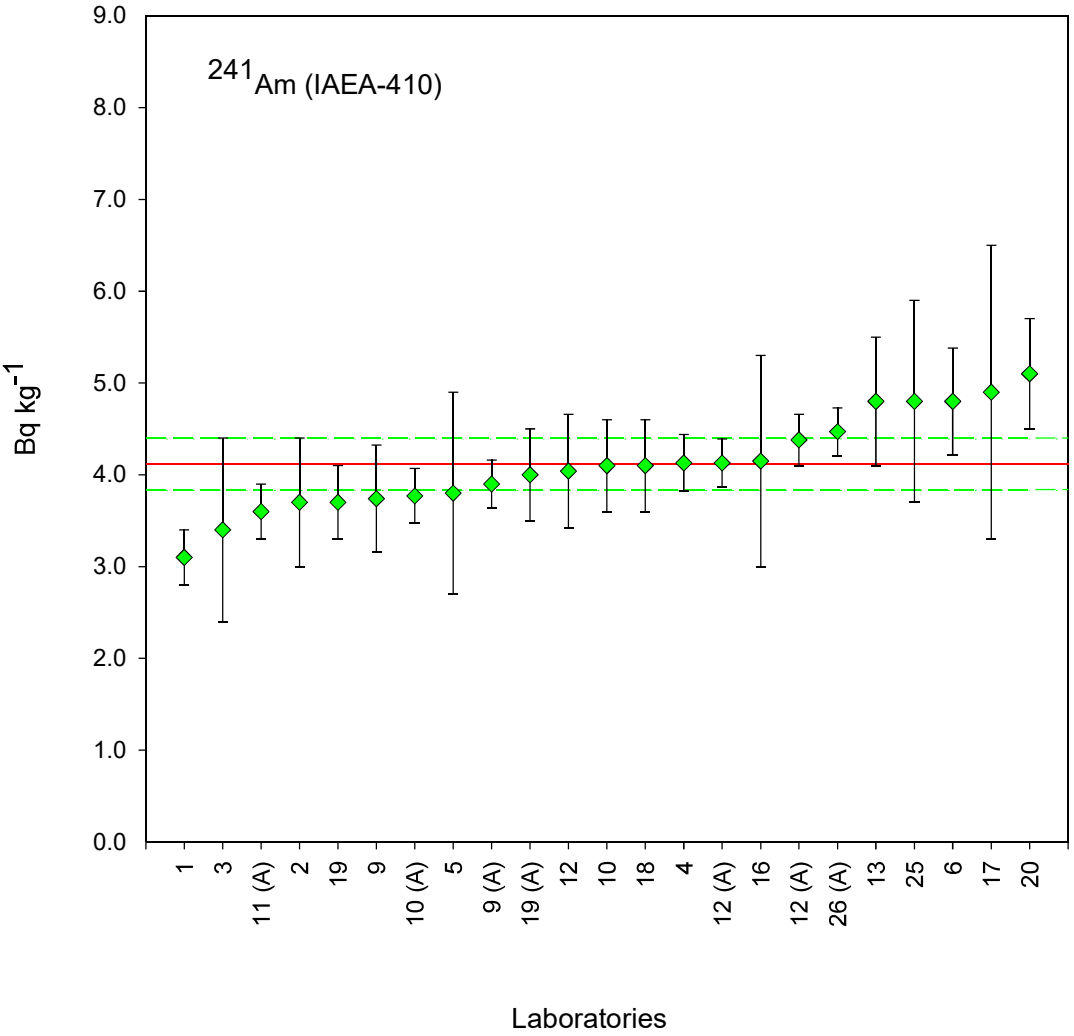

FIG. 8. Laboratory results for ^{241}Am

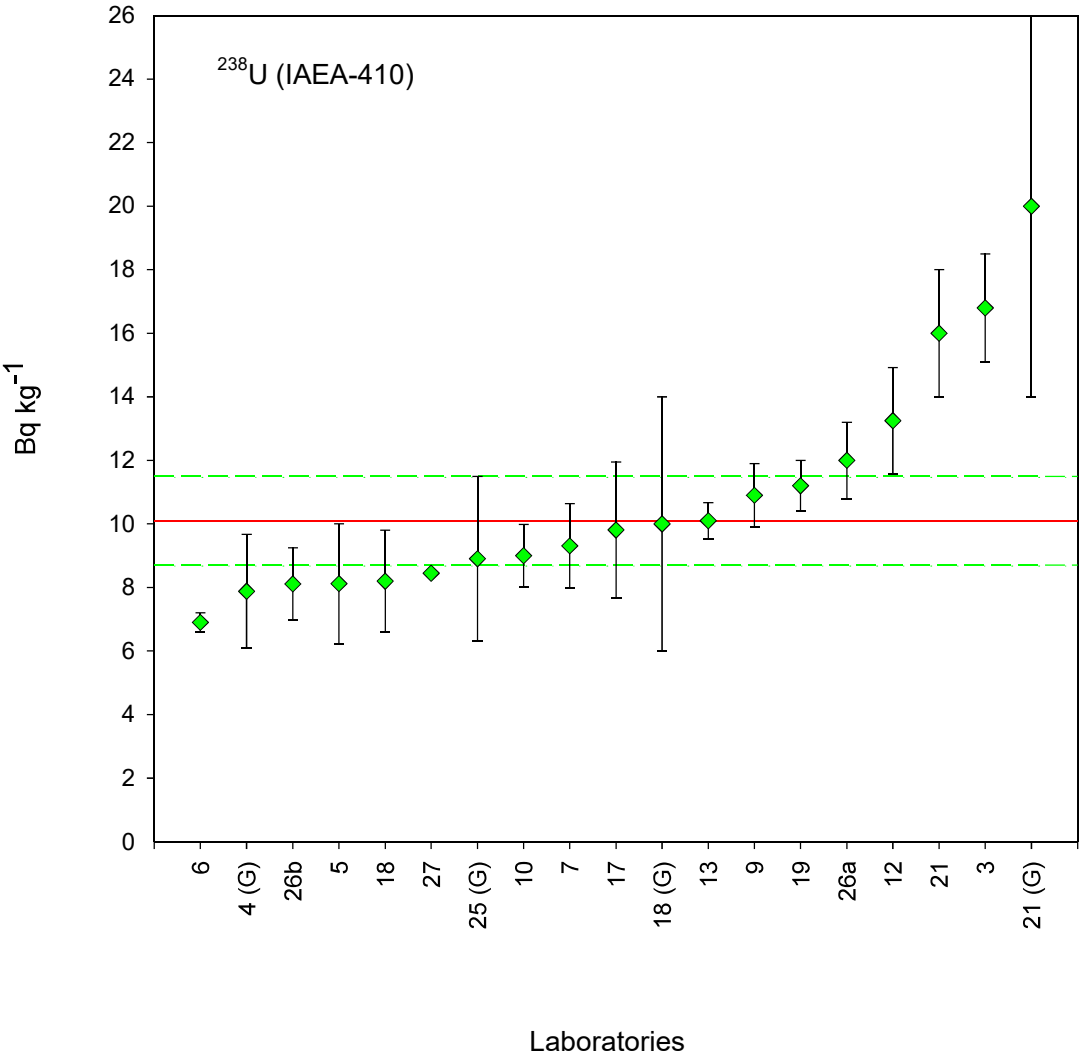

FIG. 9. Laboratory results for ^{238}U

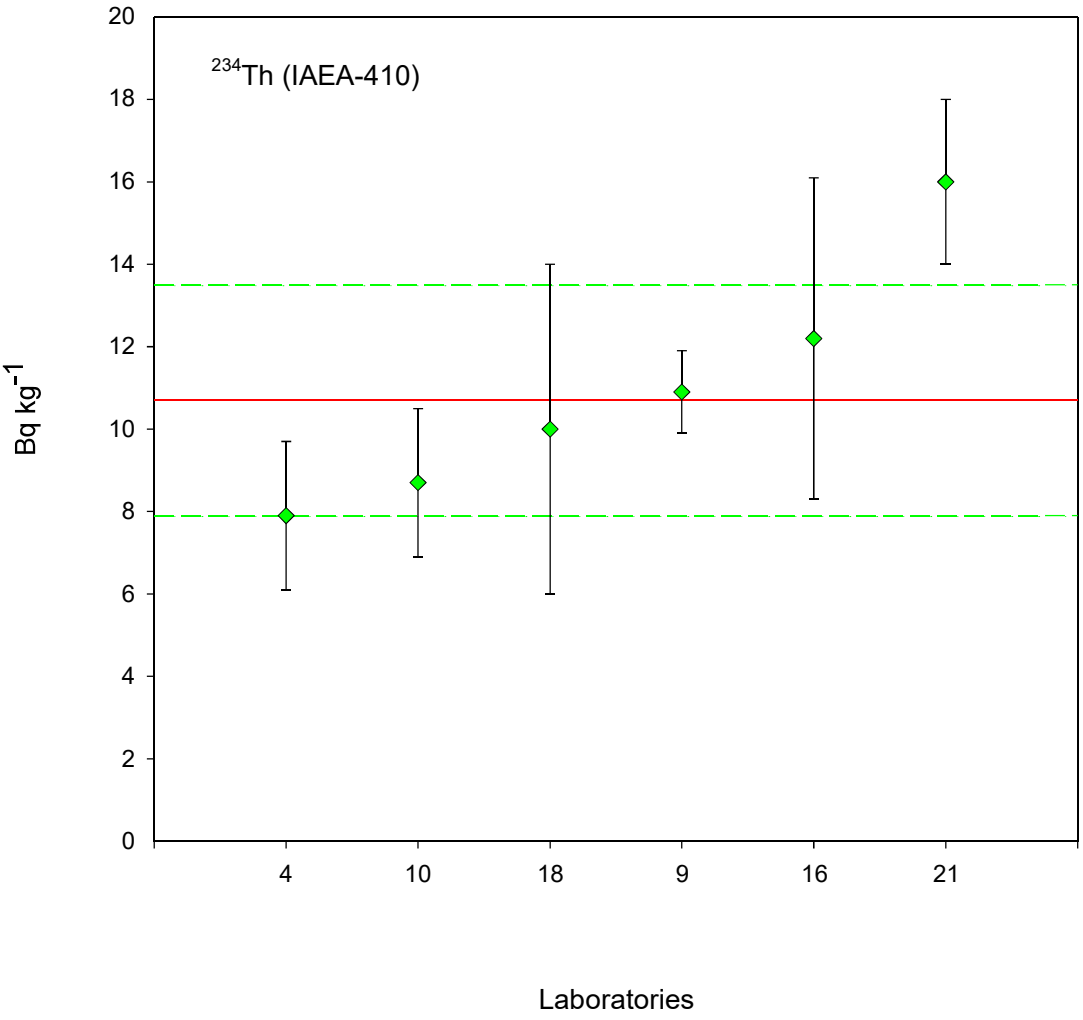

FIG. 10. Laboratory results for ^{234}Th

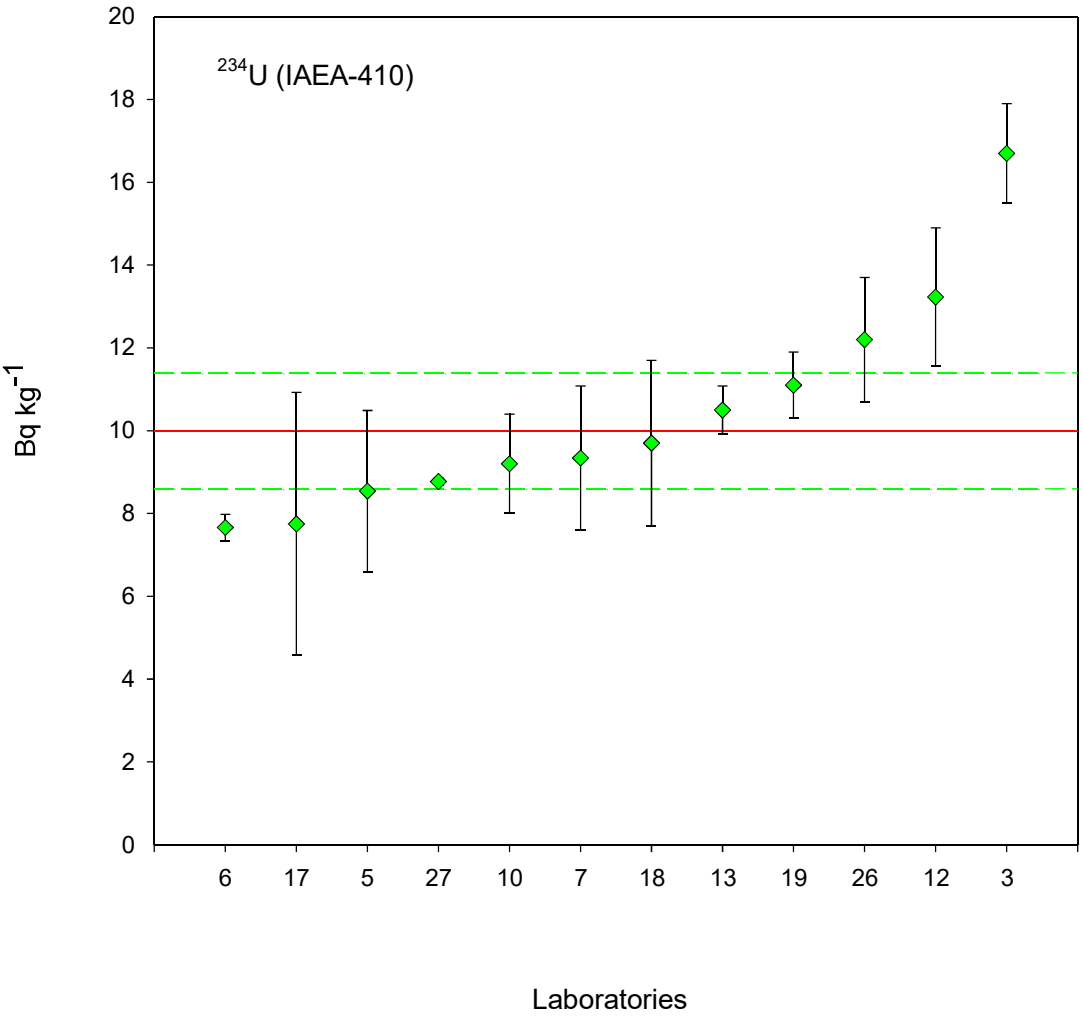

FIG. 11. Laboratory results for ^{234}U

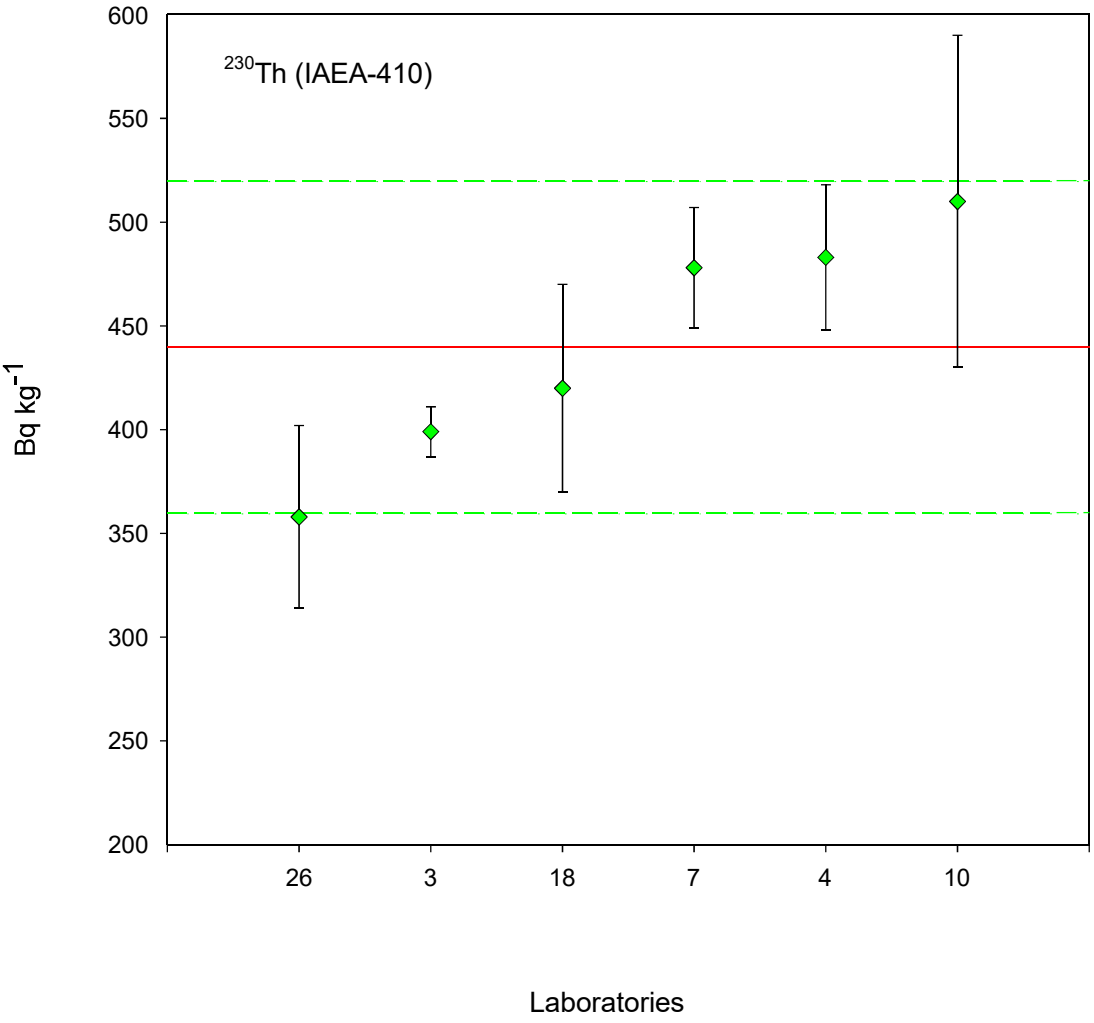

FIG. 12. Laboratory results for ^{230}Th

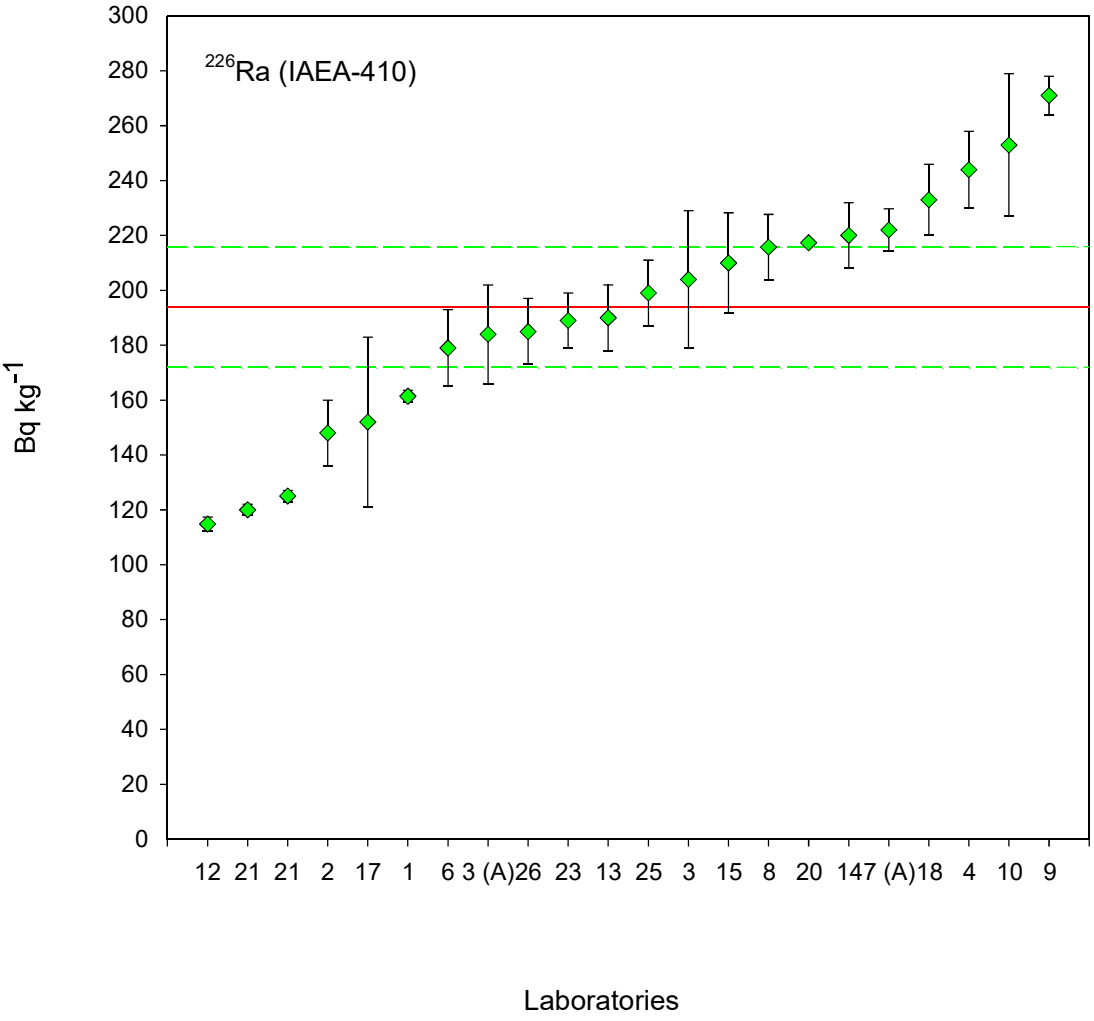

FIG. 13. Laboratory results for ^{226}Ra

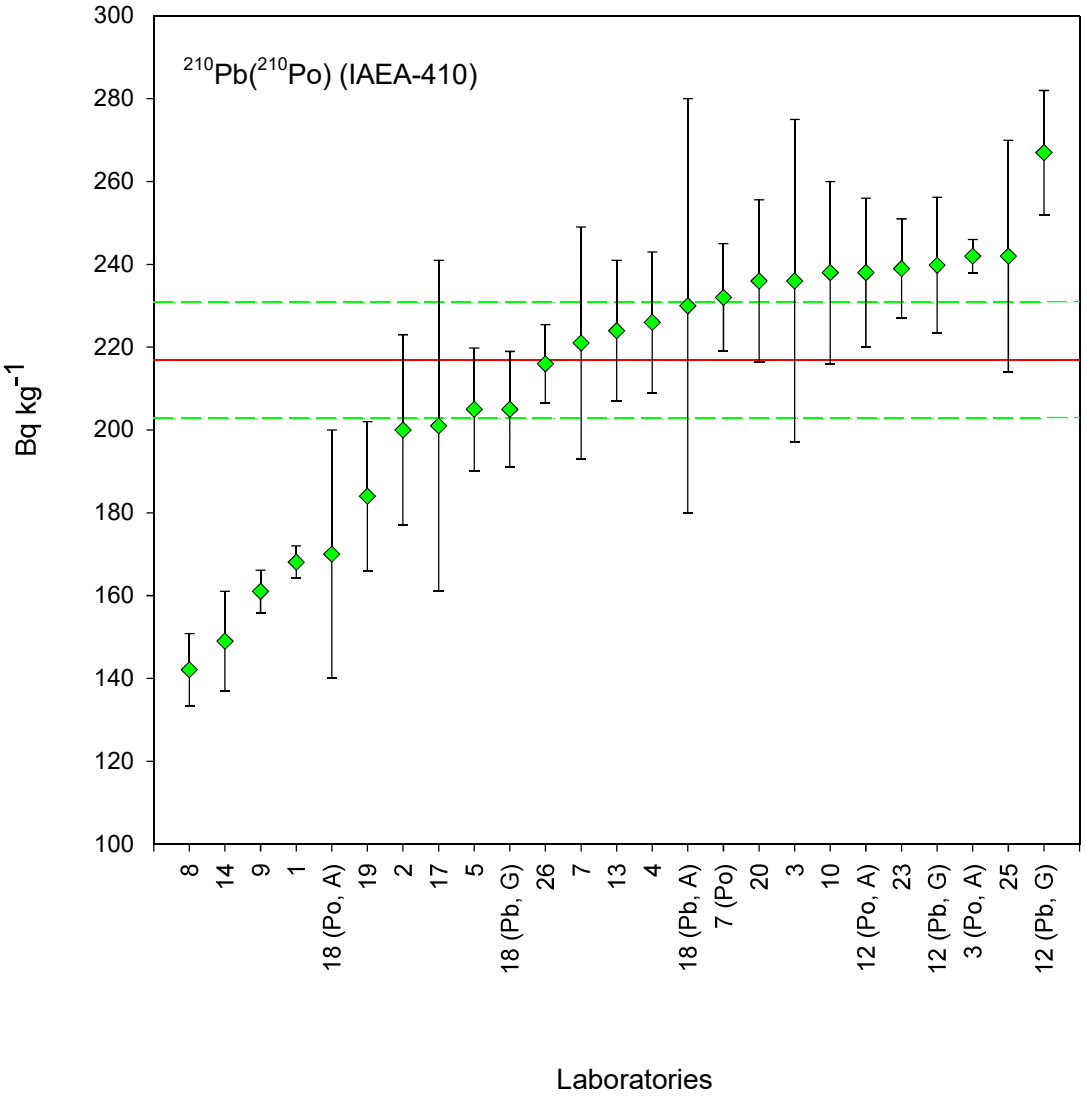

FIG. 14. Laboratory results for ^{210}Pb (^{210}Po)

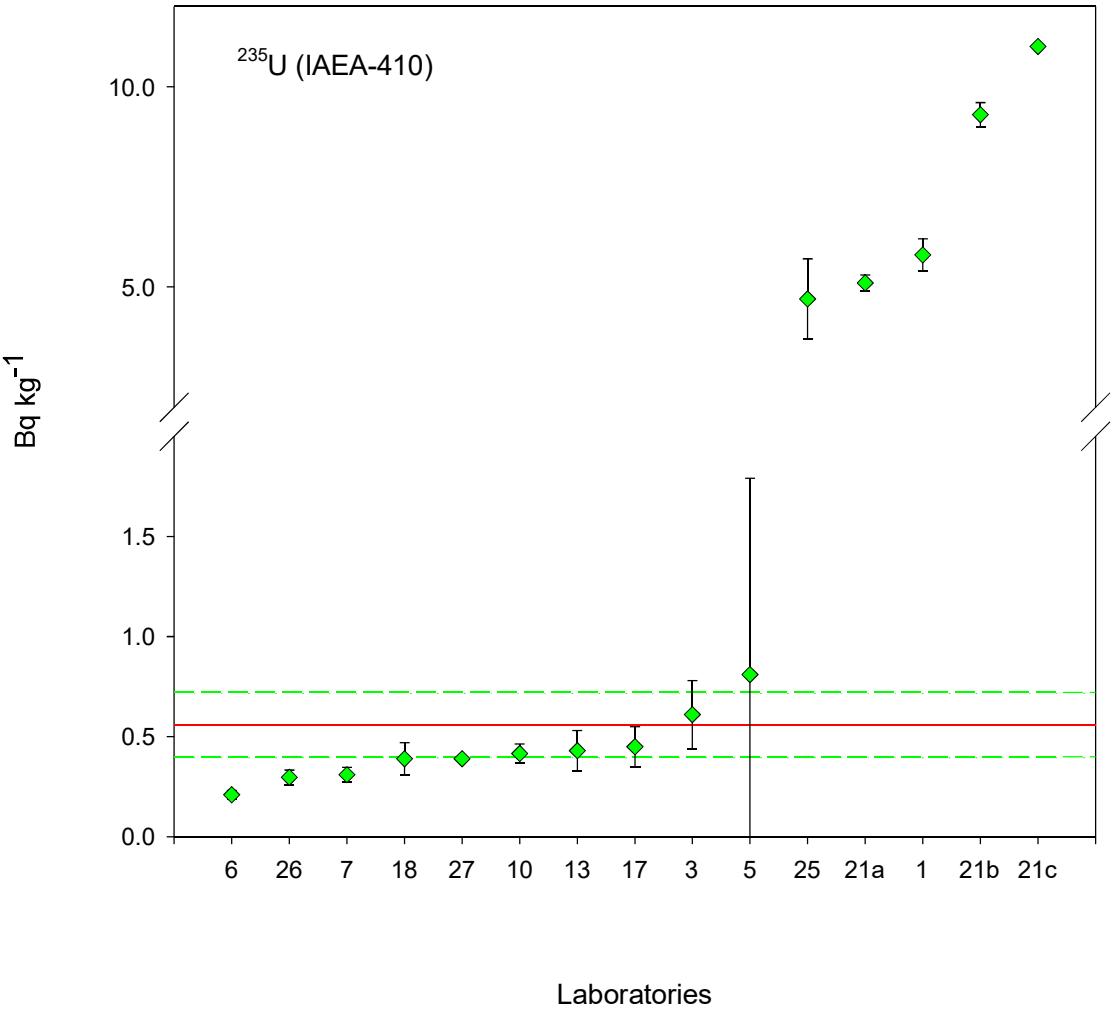

FIG. 15. Laboratory results for ^{235}U

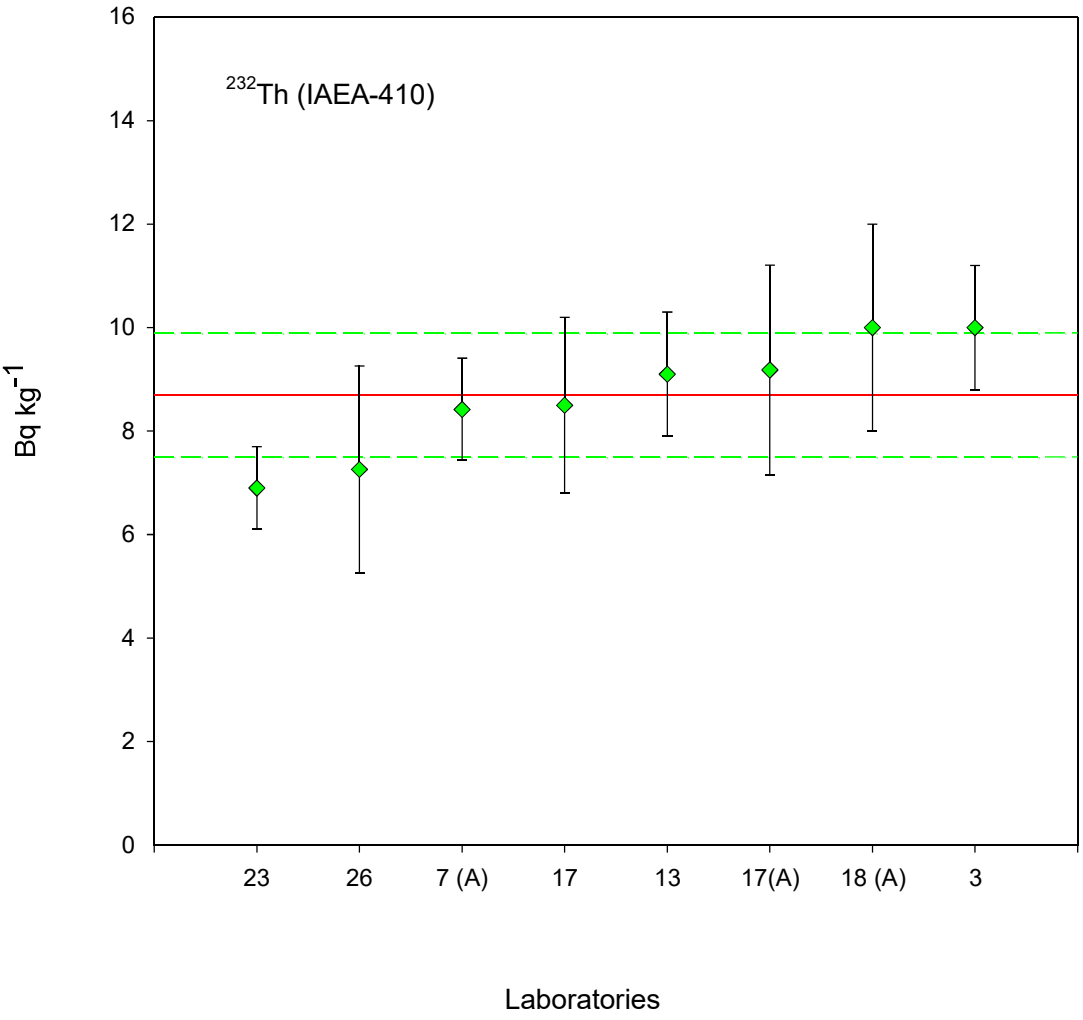

FIG. 16. Laboratory results for ^{232}Th

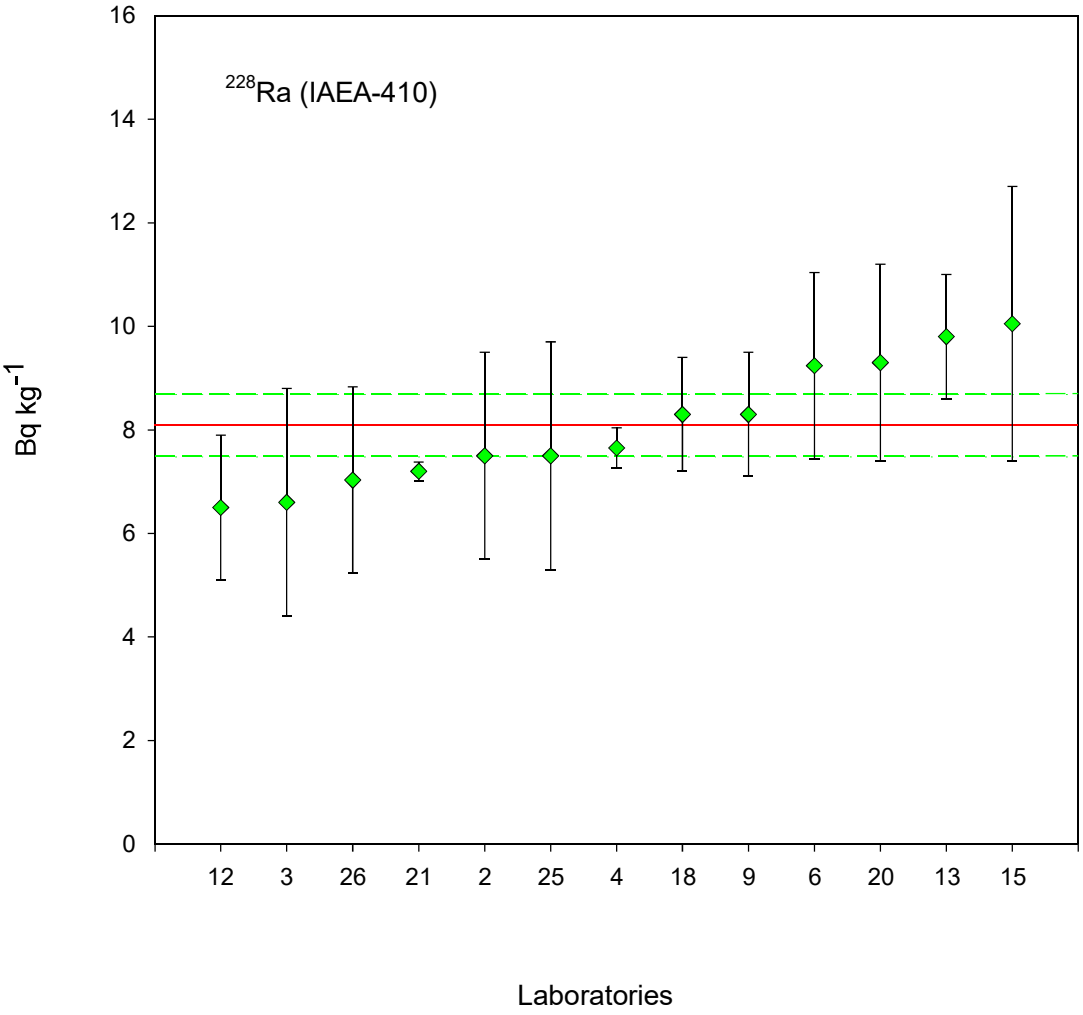

FIG. 17. Laboratory results for ^{228}Ra

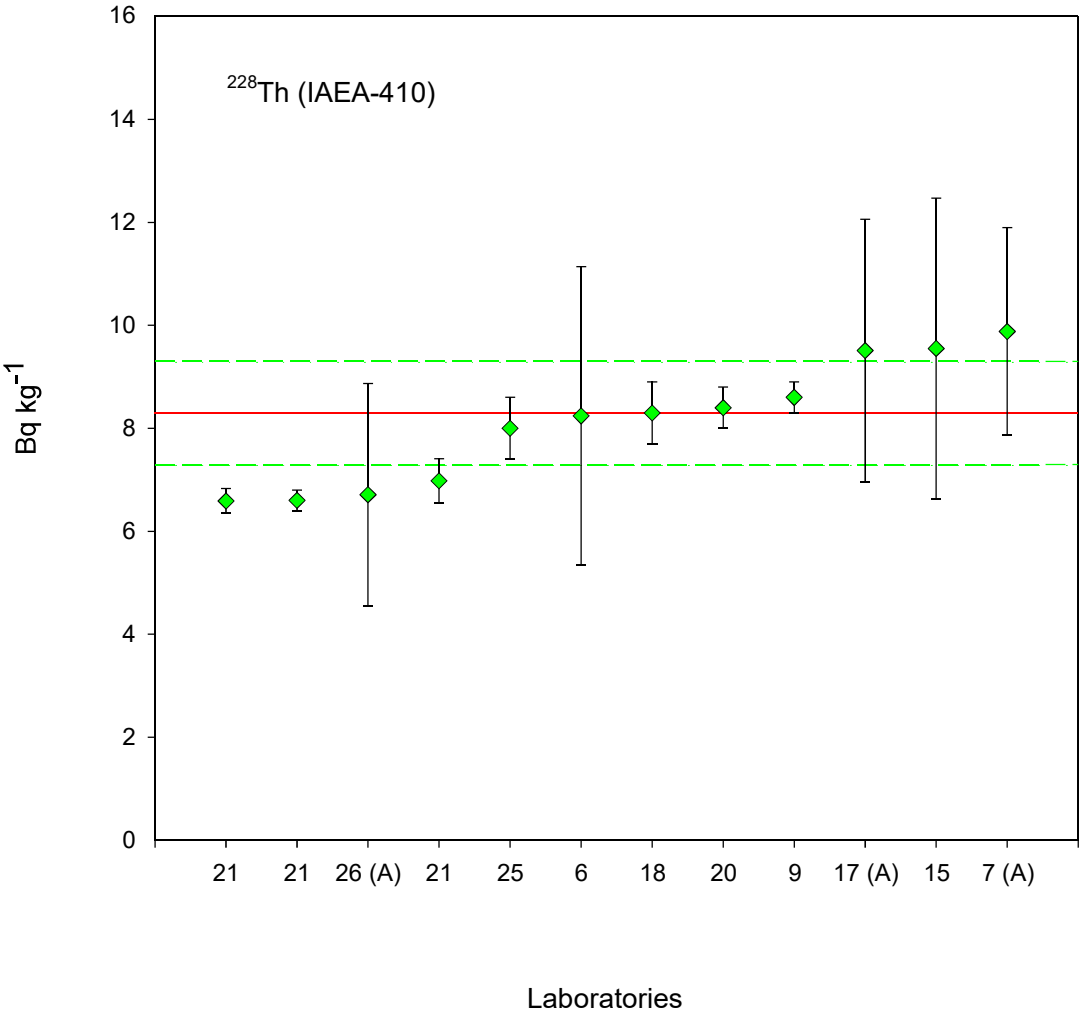

FIG.18. Laboratory results for ^{228}Th

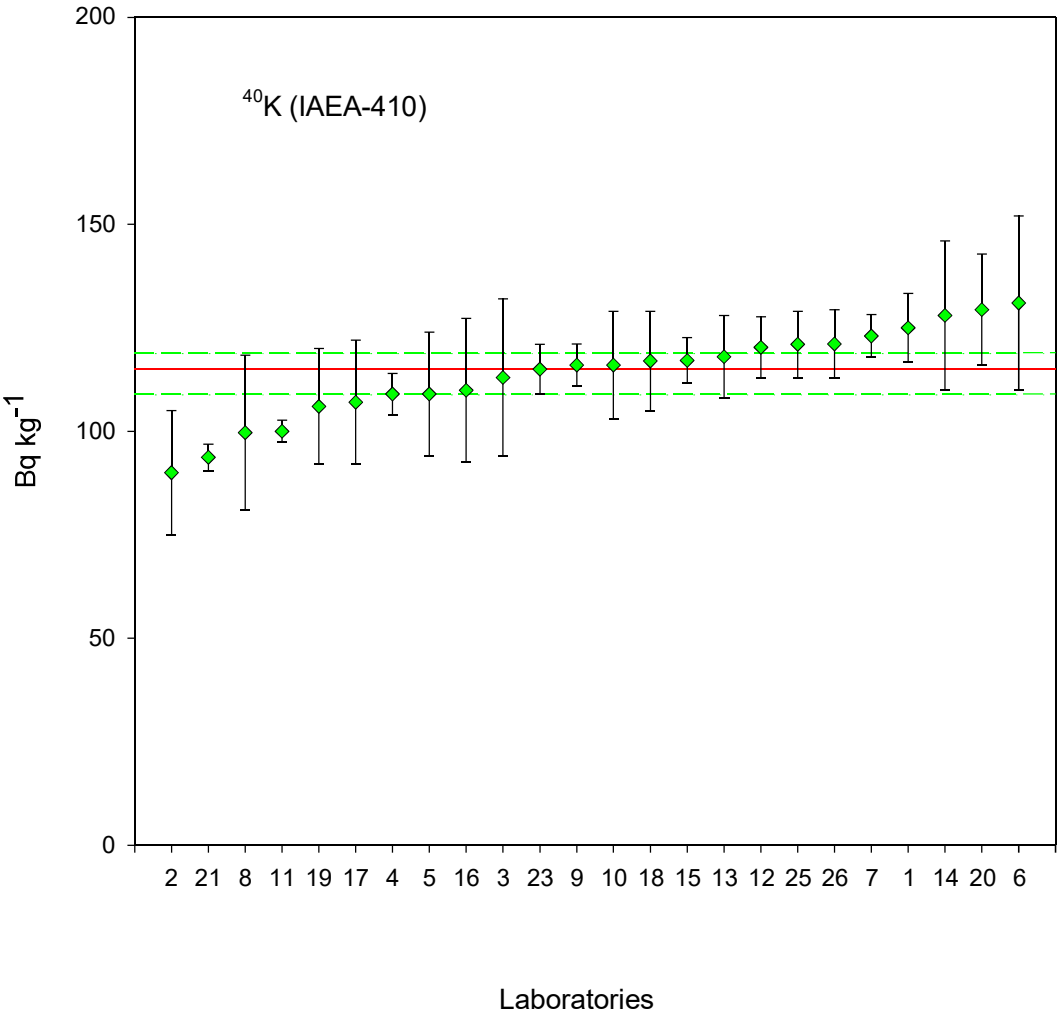

FIG.19. Laboratory results for ^{40}K

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ACKNOWLEDGEMENTS

The International Atomic Energy Agency is grateful to the participants and laboratories taking part in this characterization study and contributing their time and facilities to the present work.

The International Atomic Energy Agency is grateful to the Government of the Principality of Monaco for the support provided to its Environment Laboratories.

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ISSN 2074–7659