IAEA/AQ/54

IAEA Analytical Quality in Nuclear Applications Series No. 54

Certification of Massic Activities of Radionuclides in IAEA-412 Pacific Ocean Sediment

IAEA
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CERTIFICATION OF MASSIC ACTIVITIES OF RADIONUCLIDES IN IAEA-412 PACIFIC OCEAN SEDIMENT

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CERTIFICATION OF MASSIC ACTIVITIES OF RADIONUCLIDES IN IAEA-412 PACIFIC OCEAN SEDIMENT

INTERNATIONAL ATOMIC ENERGY AGENCY
VIENNA, 2018

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CERTIFICATION OF MASSIC ACTIVITIES OF RADIONUCLIDES IN IAEA-412 PACIFIC OCEAN SEDIMENT
IAEA, VIENNA, 2018
IAEA/AQ/54
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 Pacific Ocean 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 in the Pacific Ocean, elevated levels of long lived anthropogenic radionuclides (such as ^{137}Cs) were expected due to the influence of historical atmospheric nuclear weapon 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}
Transuranic	0.01–0.5 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 five laboratories belonging to CELLAR[1]) which will allow the IAEA-EL to produce a new certified reference material IAEA-412 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 Pacific Ocean 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. 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 spectrometry, alpha particle spectrometry and beta counting) and mass spectrometry measurement techniques (ICP-MS and AMS).

[1] Collaboration of European Low-level Underground Laboratories

3. DESCRIPTION OF THE MATERIAL

A total of 200 kg wet mass of sediment was collected from the Pacific Ocean (22º22' N, 152°41' E, water depth 5600 m) on 16 November 1997 during the "IAEA '97 Pacific Ocean Expedition" to the NW Pacific Ocean with research vessel *Bosei Maru*.

The sediment was collected using box coring down to 35 cm depth in the sediment. The sediment is red clay. It was noticed that the bulk sample contained a lot of water due to the difficulty of sample taken at very deeply sediment core at this Pacific Ocean position. It was first dried in open air and subsequently freeze dried, leaving a total dry mass of 18 kg. The sample was then ground into powder, sieved through a 250 µm mesh, homogenized by mixing in a nitrogen atmosphere, bottled in polyethylene sealed bottles ((100±5) g units) and coded as IAEA-412 for a total of 175 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 1.72% 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 should 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 spectrometry (placed in the underground laboratory of the Radiometrics laboratory, IAEA-EL-RML); alpha ray spectrometry (Ortec system at IAEA-EL-RML); and ICP-MS. The first homogeneity test between bottles was done for 14 bottles chosen at random at different masses of samples (100 g for gamma 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 the massic activities and uncertainties were done following ISO 18589-3 [7] and the procedure set up by Radiometric 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 ICPMS (Departamento de Fisica Applicada I, Universidad de Seville, Spain). 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 2, 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 ^{40}K, ^{137}Cs, and ^{214}Pb, 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 XFR. 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;
- Massic 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 if any 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 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 22, 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 5–26 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 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 $\mathbf{x}^*$ and $\mathbf{s}^*$ were updated by calculating:

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

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

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

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

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

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

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

Massic activities for 26 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 22, Appendix I, and Figures 4 to 19, Appendix II, while the less frequently measured radionuclides are presented in Table 23, Appendix I. The certified values obtained after statistical treatment are presented in Appendix I, Table 24, and information values are presented in Appendix I, Table 25.

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[2] unit Bq kg^{-1}.

[2] International System of Units (SI)

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, ^{137}Cs, ^{210}Pb (^{210}Po), ^{226}Ra, ^{228}Ra, ^{228}Th, ^{232}Th, ^{235}U, ^{238}U, ^{239}Pu, ^{240}Pu and $^{239+240}$Pu. The certified values are presented in Table 24, together with their expanded uncertainty.

For ^{230}Th, ^{234}Th, ^{234}U, ^{238}Pu and ^{241}Am the criteria were not fulfilled; robust mean and uncertainties for those radionuclides are given only as information values in Table 25.

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.

7. RESULTS AND DISCUSSION

7.1. ANTHROPOGENIC RADIONUCLIDES

Results of the determination of ^{137}Cs, ^{239}Pu, ^{240}Pu, $^{239+240}$Pu and ^{241}Am reported by participants are presented in Tables 4–7, Appendix I, and shown in Figures 4–8, Appendix II.

7.1.1. ^{137}Cs

Data were reported by 23 laboratories (Table 4, Appendix I and Fig. 4, Appendix II); all of them were accepted. The laboratories mainly used direct gamma 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

Six data sets were reported (Table 5, Appendix I), all were used for data evaluation.

7.1.2.2. $^{239+240}$Pu

Eighteen 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 determination. Most analyses were performed using conventional alpha particle spectrometry, while some results were combinations from ICP-MS and AMS methods.

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

Five laboratories determined separately ^{239}Pu and ^{240}Pu using mass spectrometry – ICP-MS and AMS (Table 6, Appendix I and Figs. 6 and 7, Appendix II). The robust mean, given as the certified value, is 0.358 ± 0.016 Bq kg^{-1} for ^{239}Pu and 0.240 ± 0.016 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 particle spectrometry technique (0.611 ± 0.028 Bq kg^{-1}).

7.1.3. ^{241}Am

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

7.2. NATURAL RADIONUCLIDES

7.2.1. Uranium series

7.2.1.1. ^{238}U

Nineteen data sets were reported (Table 8, 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. Seven other laboratories used direct gamma spectrometry technique. Four laboratories could determine their 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 difference between the two assigned values (see below for ^{226}Ra).

7.2.1.2. ^{234}Th

Eight data sets were reported (Table 9, Appendix I and Fig. 10, Appendix II). All participants used direct gamma spectrometry measurement.

7.2.1.3. ^{234}U

Eleven data sets were reported (Table 10, Appendix I and Fig. 11, Appendix II). All values were accepted. 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

All seven data sets reported (Table 11, Appendix I and Fig. 12, Appendix II) were accepted. Most participants used a conventional method based on sample treatment, ion-exchange separation followed by electro deposition and alpha particle spectrometry; two laboratories used direct gamma spectrometry.

7.2.1.5. ^{226}Ra

Data were reported from 22 laboratories (Table 12, Appendix I and Fig. 13, Appendix II). Most laboratories used direct gamma 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. The data showed good homogeneity.

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

Eleven and thirteen laboratories reported ^{214}Bi and ^{214}Pb results, respectively (Table 13, Appendix I). These data were determined by using gamma 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. 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 26 laboratories (Table 14, Appendix I and Fig. 14, Appendix II). ^{210}Pb and ^{210}Po were considered as 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 spectrometry to measure ^{210}Pb at 46.5 keV, eight 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

Eighteen data sets were reported (Table 15, Appendix I and Fig. 15, Appendix II). Nine participants used a conventional method based on sample treatment, ion-exchange separation followed by electro deposition and alpha particle spectrometry. Seven laboratories determined ^{235}U using direct gamma 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 16, Appendix I). These data were determined by using gamma spectrometry and are in the same range of ^{235}U mass activities (see above) showing that the ^{235}U and its progeny ^{227}Ac and are 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

Nine data sets were reported (Table 17, Appendix I and Fig.16, Appendix II). Four data sets were analysed by gamma 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 18 in Appendix I and Fig. 17 in Appendix II). All laboratories used direct gamma spectrometry to determine ^{228}Ra activity through their 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

Fifteen laboratories reported ^{228}Ac (Table 19, Appendix I). These data were determined by using gamma spectrometry and are about the same levels as ^{228}Th and ^{228}Ra massic activities (see above for ^{228}Ra and below for ^{228}Th and ^{208}Tl, respectively) showing that ^{228}Ra and its progeny ^{228}Ac, ^{228}Th and ^{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

Fourteen data sets were reported (Table 20, Appendix I and Fig. 18, Appendix II). Most participants used direct gamma 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). Five 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 six laboratories reported ^{212}Bi and ^{212}Pb results, respectively (Table 21, Appendix I). These data were determined by using gamma spectrometry and are in the same range with ^{228}Ra massic activity (see above for 7.2.3.2.) 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

Eleven laboratories reported results for ^{208}Tl (Table 19, Appendix I). These data were determined by using gamma spectrometry and are about the same levels as ^{232}Th, ^{228}Ra, ^{228}Ac, and ^{228}Th mass activities (see above), if taking into account the branching factor 35.93%. As mentioned above for ^{228}Ac, there is equilibrium in Thorium series (^{232}Th and its progeny ^{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 calculation.

7.2.4. ^{40}K

Data were reported from 24 laboratories (Table 22, 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 23, Appendix I.

7.3.1. ^{129}I

Two results were reported, using AMS technique, ranging from 0.26 to 0.35 mBq kg^{-1}.

7.3.2. ^{155}Eu

Two results were reported, using gamma spectrometry technique, ranging from 1.87 to 3.92 Bq kg^{-1}.

7.3.3. ^{211}Pb, ^{219}Rn, ^{223}Ra and ^{227}Th

One result was reported, using gamma spectrometry technique, given the value of (2.2 ± 0.3) Bq kg^{-1} for both radionuclides ^{211}Pb, ^{219}Rn; and (1.65 ± 0.26) Bq kg^{-1} for ^{223}Ra, which is in the same range with its precursor ^{235}U (see 7.2.2.1.).

Two laboratories reported ^{227}Th value in the range of 1.17 to 1.7 Bq kg^{-1}, which agrees with its precursor ^{235}U value.

7.3.4. ^{224}Ra

Three results were reported, using gamma spectrometry technique, ranging from 36 to 66 Bq kg^{-1}.

7.3.5. ^{236}U

Out of two results reported, using ICP-MS and AMS technique, only one result is significant (0.005 4 ± 0.008 Bq kg^{-1}), which is the average result of five values determined by AMS.

8. CONCLUSIONS

In this characterization study, selected 27 laboratories (including IAEA-EL and 5 laboratories from CELLAR group) reported mass activities of natural and anthropogenic radionuclides in a sediment sample from Pacific Ocean (IAEA-412). 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, ^{137}Cs, ^{210}Pb (^{210}Po), ^{226}Ra, ^{228}Ra, ^{228}Th, ^{232}Th, ^{235}U, ^{238}U, ^{239}Pu, ^{240}Pu and $^{239+240}$Pu and the information values are given to other radionuclides ^{230}Th, ^{234}Th, ^{234}U, ^{238}Pu and ^{241}Am. The agreement between the results confirms the absence of any significant method bias (if there is more than one methods 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 expended uncertainties for the most frequently reported anthropogenic and natural radionuclides could be found below as well as in Table 24 and Table 25, respectively in Appendix I.

APPENDIX I. TABLES

TABLE 1. SUMMARY TABLE. CERTIFIED AND INFORMATION VALUES FOR THE IAEA–412 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	561	14
^{137}Cs	6.50	0.28
^{210}Pb(^{210}Po)[c]	100.9	4.4
^{226}Ra	27.4	2.6
^{228}Ra	36.7	1.8
^{228}Th	38.3	3.8
^{232}Th	36.3	2.0
^{235}U	1.38	0.20
^{238}U	31.2	3.2
^{239}Pu	0.358	0.016
^{240}Pu	0.240	0.016
$^{239+240}$Pu	0.611	0.028

Radionuclide	Information value [Bq kg^{-1}]	Expanded uncertainty [Bq kg^{-1}]
^{230}Th	26	6
^{234}Th	37	8
^{234}U	29	6
^{238}Pu	0.019 9	0.004 0
^{241}Am	0.30	0.10

[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-412

Sample	^{40}K	^{137}Cs	^{210}Pb	^{214}Pb	^{228}Ra
1	0.87	0.96	0.84	0.95	0.96
2	0.89	0.97	0.88	0.97	0.98
3	0.94	0.98	0.91	0.99	0.98
4	0.97	0.98	0.92	0.99	0.99
5	0.98	0.98	0.94	1.00	1.00
6	1.00	1.00	0.95	1.00	1.00
7	1.02	1.01	1.02	1.00	1.00
8	1.03	1.01	1.02	1.00	1.00
9	1.04	1.01	1.07	1.01	1.00
10	1.05	1.01	1.07	1.01	1.01
11	1.05	1.02	1.10	1.01	1.01
12	1.05	1.02	1.13	1.02	1.02
13	1.05	1.02	1.16	1.03	1.03
14	1.07	1.03		1.03	1.03
Minimum	0.87	0.96	0.84	0.95	0.96
Maximum	1.07	1.03	1.16	1.03	1.03
Mean	1.00	1.00	1.00	1.00	1.00
Median	1.03	1.01	1.02	1.00	1.00
Std. dev.	0.06	0.02	0.10	0.02	0.02
Coeff. var. (%)	6	2	10	2	2

* Normalized activity = x/X (individual/mean values). The homogeneity test was performed in IAEA-EL-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-412

Radionuclide	Number of data reported	Radionuclide	Number of data reported
^{40}K	84	^{228}Ac	52
^{129}I	11	^{228}Th	49 (1)
^{137}Cs	84	^{230}Th	22
^{208}Tl	42	^{232}Th	27
^{210}Pb(^{210}Po)	79	^{234}Th	32
^{212}Pb	35	^{234}U	47
^{212}Bi	28	^{235}U	97 (1)
^{214}Pb	41	^{238}U	72
^{214}Bi	47	^{238}Pu	16 (1)
^{224}Ra	16	^{239}Pu	14
^{226}Ra	84	^{240}Pu	14
^{228}Ra	56	$^{239+240}$Pu	53
^{227}Ac	17	^{241}Am	26

Note: “Less than” values are shown in parentheses

TABLE 4. RESULTS FOR ^{137}Cs IN IAEA-412
(Reference date: 1 January 2013, unit: Bq kg^{-1})

Lab code	Method code	No. of results	Mass (g)	^{137}Cs
1	G	3	100.74	6.40 ± 0.40
2	G	2	88.51	6.80 ± 0.90
3	G	3	97.58	6.35 ± 0.98
4	G	3	100.6	6.59 ± 0.29
5	G	3	76	6.91 ± 0.51
6	G	5	100.19	7.74 ± 0.70
7	G	3	82.48	6.91 ± 0.83
8	G	3	34	5.70 ± 0.72
9	G	3	99.77	6.54 ± 0.22
10	G	1	56	6.90 ± 0.70
11	G	3	88	5.20 ± 0.54
12	G	10	83.5	5.95 ± 0.44
13	G	3	101	7.10 ± 0.60
14	G	3	39.55–101.45	5.52 ± 0.66
15	G	1	234.4	6.99 ± 0.34
16	G	2	27	6.25 ± 0.92
17	G	4	17; 80	6.40 ± 0.60
18	G	3	80.2	7.00 ± 0.60
20	G	4	7.2	6.80 ± 0.30
21	G	5	99.89	5.94 ± 0.27
23	G	1	100	6.00 ± 0.30
25	G	2	70.62	6.87 ± 0.40
26	G	14	100	6.35 ± 0.45

Number of reported laboratory means	23
Robust mean	6.50
Expanded uncertainty	0.28

TABLE 5. RESULTS FOR ^{238}Pu AND $^{239+240}$Pu IN IAEA-412
(Reference date: 1 January 2013; unit: Bq kg^{-1})

Lab code	Method code	No. of results	Mass (g)	^{238}Pu	$^{239+240}$Pu
3	A	3	2.97–3.44	—	0.58 ± 0.02
5	A	3	0.25	—	1.40 ± 0.52
6	A	2	15	0.022 ± 0.005	0.66 ± 0.02
7a	ICP-MS	3	10	—	0.60 ± 0.01
7b	A	3	10	—	0.61 ± 0.08
9	A	3	10–15	0.020 ± 0.006	0.62 ± 0.05
10a	ICP-MS	1	38.1	—	0.63 ± 0.06
10b	A	1	38	0.020 ± 0.004	0.63 ± 0.04
11	A	3	30	<0.045	0.57 ± 0.05
12	A	3	5-10	0.026 ± 0.016	0.63 ± 0.06
13	A	8	25.4–26.1	—	0.44 ± 0.07
14	A	3	10–14	—	0.65 ± 0.07
15	A	1	20.02	0.015 ± 0.009	0.62 ± 0.05
18	A	3	—	—	1.10 ± 0.60
24	AMS	3	1.5	—	0.60 ± 0.02
26a	A	4	3–10	0.017 ± 0.006	0.53 ± 0.04
26b	AMS	3	1.5	—	0.58 ± 0.02
26c	AMS	3	1.5	—	0.57 ± 0.02
Number of reported laboratory means				6	18
Robust mean				0.019 9	0.611
Expanded uncertainty				0.004 0	0.028

Note: Tables 5–25: —: data not available

TABLE 6. RESULTS FOR ^{239}Pu AND ^{240}Pu IN IAEA-412
(Reference date: 1 January 2013; unit: Bq kg^{-1})

Lab code	Method code	No. of results	Mass (g)	^{239}Pu	^{240}Pu
7	ICP-MS	3	10	0.36 ± 0.01	0.24 ± 0.01
10	ICP-MS	1	38.1	0.37 ± 0.04	0.26 ± 0.03
24	AMS	3	1.5	0.37 ± 0.01	0.23 ± 0.01
26a	AMS	3	1.5	0.34 ± 0.01	0.23 ± 0.01
26b	AMS	3	1.5	0.35 ± 0.01	0.24 ± 0.01
Number of reported laboratory means				5	5
Robust mean				0.358	0.240
Expanded uncertainty				0.016	0.016

TABLE 7. RESULTS FOR ^{241}Am IN IAEA-412
(Reference date: 1 January 2013, unit: Bq kg^{-1})

Lab code	Method code	No. of results	Mass (g)	^{241}Am
1	G	3	100.74	0.17 ± 0.24
2	G	2	88.51	<1.2
4	G	2	100.6	0.40 ± 0.09
9	G	3	99.77	<0.4
9	A	3	10–15	0.28 ± 0.02
10	A	1	38	0.25 ± 0.03
10	G	1	56	0.33 ± 0.12
11	A	3	30	0.20 ± 0.06
12	A	4	5	0.32 ± 0.07
20	G	4	7.2	1.0 ± 0.7
Number of reported laboratory means				8
Robust mean				0.30
Expanded uncertainty				0.10

TABLE 8. RESULTS FOR ^{238}U IN IAEA-412
(Reference date: 1 January 2013, unit: Bq kg^{-1})

Lab code	Method code	No. of results	Mass (g)	^{238}U
3	A	3	0.675–0.815	34.8 ± 0.6
4	G	3	100.6	33.4 ± 5.0
4	G	3	100.6	36.7 ± 2.8
5	A	4	0.25	9.3 ± 2.2
6	A	2	15	8.4 ± 0.5
7	A	3	2	28.3 ± 2.9
9	G	3	99.77	30.5 ± 1.8
10	ICP-MS	3	0.3–0.6	32.4 ± 3.6
12	A	3	0.5	36.5 ± 3.6
13	A	8	2.63; 2.7	33.4 ± 2.0
17	ICP-MS	2	1	17.2 ± 3.8
18	A	3	—	30.0 ± 6.0
18	G	3	80.2	33.0 ± 4.0
20	G	4	7.2	39.1 ± 8.2
21	G	5	99.89	34.2 ± 3.2
25	G	2	70.62	37.0 ± 7.0
26a	A	4	3–10	27.1 ± 1.7
26b	ICP-MS	4	0.15	27.0 ± 3.8
27	ICP-MS	10	0.5	29.3 ± 0.4
Number of reported laboratory means				19
Robust mean				31.2
Expanded uncertainty				3.2

TABLE 9. RESULTS FOR ^{234}Th IN IAEA-412
(Reference date: 1 January 2013, unit: Bq kg^{-1})

Lab code	Method code	No. of results	Mass (g)	^{234}Th
2	G	2	88.51	27.0 ± 6.1
9	G	3	99.77	30.5 ± 7.4
10	G	1	56	32 ± 4
13	G	3	101	54.1 ± 5.2
16	G	2	27	39.7 ± 6.9
18	G	3	80.2	33 ± 4
20	G	4	7.2	39.1 ± 8.2
26	G	14	100	44.6 ± 7.5
Number of reported laboratory means				8
Robust mean				37
Expanded uncertainty				8

TABLE 10. RESULTS FOR ^{234}U IN IAEA-412
(Reference date: 1 January 2013, unit: Bq kg^{-1})

Lab code	Method code	No. of results	Mass (g)	^{234}U
3	A	3	0.675–0.815	34.0 ± 0.7
5	A	4	0.25	14.7 ± 2.0
6	A	2	15	10.5 ± 0.5
7	A	3	2	27.5 ± 2.5
10	ICP-MS	3	0.3–0.6	30.6 ± 4.1
12	A	3	0.5	35.6 ± 3.6
13	A	8	2.63–2.7	31.6 ± 2.0
17	A	4	1	22.5 ± 6.3
18	A	3	—	46.0 ± 8.0
26	A	4	3–10	27.0 ± 1.9
27	ICP-MS	10	0.5	29.2 ± 0.5
Number of reported laboratory means				11
Robust mean				29
Expanded uncertainty				6

TABLE 11. RESULTS FOR ^{230}Th IN IAEA-412
(Reference date: 1 January 2013, unit: Bq kg^{-1})

Lab code	Method code	No. of results	Mass (g)	^{230}Th
3	A	3	0.675–0.815	32.0 ± 2.4
4	G	3	100.6	30.5 ± 7.7
7	A	3	2	30.4 ± 4.5
10	G	1	56	28.0 ± 12.0
17	A	4	1	18.3 ± 4.8
26	A	4	3–10	22.2 ± 7.5
26	A	4	3–10	21.6 ± 7.3
Number of reported laboratory means				7
Robust mean				26
Expanded uncertainty				6

TABLE 12. RESULTS FOR ^{226}Ra IN IAEA-412
(Reference date: 1 January 2013, unit: Bq kg^{-1})

Lab code	Method code	No. of results	Mass (g)	^{226}Ra
1	G	3	100.74	27.8 ± 1.0
2	G	2	88.51	23.0 ± 2.0
3	G	3	97.58	24.1 ± 3.4
3	A	1	0.675–0.815	26.7 ± 2.9
4	G	3	100.6	26.5 ± 3.0
6	G	5	100.19	29.5 ± 3.0
7	A	3	1	23.0 ± 1.0
8	G	5	34	52.1 ± 10.6
9	G	3	99.77	31.8 ± 3.4
10	G	1	56	32.0 ± 9.0
12	G	10	83.5	18.4 ± 0.9
13	G	3	101	30.5 ± 2.3
14	G	3	1; 101.45	42.3 ± 6.3
15	G	1	234.4	59.2 ± 6.8
17	G	4	17; 80	25.0 ± 4.0
18	G	3	80.2	27.1 ± 1.9
20	G	4	7.2	27.1 ± 0.7
21a	G	5	99.89	22.8 ± 1.5
21b	G	5	99.89	22.3 ± 1.3
23	G	1	100	23 ± 3
25	G	2	70.62	27.6 ± 1.4
26	G	14	100	28.1 ± 2.0
Number of reported laboratory means				22
Robust mean				27.4
Expanded uncertainty				2.6

TABLE 13. RESULTS FOR ^{214}Pb and ^{214}Bi IN IAEA-412
(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	100.74	27.6 ± 0.9	28.1 ± 1.1
2	G	2	88.51	25.0 ± 3.0	21.0 ± 2.6
5	G	3	76	—	25.2 ± 1.6
7	G	3	82.48	—	24.5 ± 3.8
9	G	3	99.77	27.2 ± 1.5	22.3 ± 0.9
13	G	3	101	31.0 ± 2.1	30.0 ± 2.6
15	G	1	234.4	29.2 ± 1.9	27.1 ± 2.6
16	G	2	27	28.7 ± 3.7	19.7 ± 2.4
18	G	3	80.2	27.1 ± 1.9	27.1 ± 1.9
19	G	2	27	29.5 ± 2.4	29.4 ± 2.6
20	G	4	7.2	27.1 ± 0.7	28.9 ± 3.7
21	G	4	53.8	22.8 ± 1.5	22.3 ± 1.3
26	G	14	100	28.1 ± 2.0	20.8 ± 1.5
Number of reported laboratory means				11	13
Robust mean				27.7	25.1
Expanded uncertainty				1.36	2.8

TABLE 14. RESULTS FOR ^{210}Pb (^{210}Po) IN IAEA-412
(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	100.74	89.3 ± 3.0
2	G	2	88.51	105 ± 13
3	G	1	97.58	90 ± 16
3 (Pb)	A	3	0.675–0.815	111 ± 4
3 (Po)	A	3	0.675–0.815	111 ± 4
4	G	3	100.6	106 ± 8
5	G	4	76	103 ± 14
7	A	3	1	94.9 ± 2.4
7	A	3	1	105 ± 9
8	G	3	34	71.5 ± 8.3
9	G	3	99.77	82.1 ± 2.0
10	G	1	56	107 ± 11
12 (Po)	A	3	0.5 – 1	95.5 ± 5.2
12	G	3	83.6	109 ± 10
12 (Pb)	A	3	0.5–1	113.5 ± 6.6
13	G	3	101	104.0 ± 9.0
14	G	3	39.55; 101.45	79.0 ± 7.7
17	G	2	17	97 ± 20
18 (Pb)	A	3	—	80 ± 14
18	G	3	80.2	98.0 ± 9.0
18 (Po)	A	3	—	102 ± 24
20	G	4	7.2	108.1 ± 8.7[b]
23	G	1	100	104 ± 9[b]
25	G	2	70.62	107 ± 12
26	G	14	10	103 ± 12
26	A	3	0.5	110 ± 22

Number of reported laboratory means	26
Robust mean	100.9
Expanded uncertainty	4.4

[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 15. RESULTS FOR ^{235}U IN IAEA-412
(Reference date: 1 January 2013, unit: Bq kg^{-1})

Lab code	Method code	No. of results	Mass (g)	^{235}U
1	G	3	100.74	1.7 ± 0.2
3	A	3	0.675–0.815	1.3 ± 0.3
5	A	4	0.25	0.82 ± 0.57
6	A	2	15	0.34 ± 0.02
7	A	3	2	1.30 ± 0.58
9	G	3	99.77	1.45 ± 0.30
10	G	1	56	1.2 ± 0.6
10	ICP-MS	3	0.3–0.6	1.50 ± 0.18
12	A	3	0.5	<0.2
13	A	8	2.63; 2.7	1.55 ± 0.39
17	ICP-MS	16	1	0.80 ± 0.16
18	A	3	—	1.39 ± 0.28
21a	A	5	99.89	1.7 ± 0.2
21b	G	5	99.89	2.2 ± 0.1
21c	G	5	99.89	2.2 ± 0.3
25	G	2	70.62	1.88 ± 0.40
26a	A	4	3–10	0.68 ± 0.09
26b	G	14	100	1.51 ± 0.12
27	ICP-MS	10	0.5	1.36 ± 0.02
Number of reported laboratory means				18
Robust mean				1.38
Expanded uncertainty				0.20

TABLE 16. RESULTS FOR ^{227}Ac IN IAEA-412
(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	1.17 ± 0.22
4	G	3	99.9	1.65 ± 0.26
9	G	3	99.77	2.12 ± 0.12
10	G	1	56	1.5 ± 0.6
21	G	5	99.89	1.7 ± 0.2
25	G	2	70.62	1.7 ± 0.4
Number of reported laboratory means				6
Robust mean				1.65
Expanded uncertainty				0.18

TABLE 17. RESULTS FOR ^{232}Th IN IAEA-412
(Reference date: 1 January 2013, unit: Bq kg^{-1})

Lab code	Method code	No. of results	Mass (g)	^{232}Th
3	A	3	0.675–0.815	35.3 ± 3.4
7	G	3	2	36.3 ± 8.7
13	G	3	101	42 ± 3
17	ICP-MS	2	1	34.9 ± 7.1
17	G	4	17; 80	37.0 ± 5.0
18	A	3	—	34.0 ± 5.0
23	G	1	100	33 ± 4
26a	A	4	3–10	37.5 ± 4.8
26b	A	4	3–10	38.7 ± 4.9
Number of reported laboratory means				9
Robust mean				36.3
Expanded uncertainty				2.0

TABLE 18. RESULTS FOR ^{228}Ra IN IAEA-412
(Reference date: 1 January 2013, unit: Bq kg^{-1})

Lab code	Method code	No. of results	Mass (g)	^{228}Ra
2	G	2	88.51	35.5 ± 5.2
3	G	1	97.58	33.9 ± 4.7
4	G	3	100.6	37.7 ± 1.5
6	G	5	100.19	45.6 ± 2.1
9	G	3	99.77	36.5 ± 1.6
12	G	10	83.5	34.0 ± 1.5
13	G	3	101	42.4 ± 2.9
15	G	1	234.4	38.2 ± 5.5
18	G	3	80.2	36.4 ± 2.9
20	G	4	7.2	39.3 ± 3.1
21	G	5	99.89	34.4 ± 0.9
25	G	2	70.62	40.2 ± 2.0
26	G	14	100	30.0 ± 2.2
Number of reported laboratory means				13
Robust mean				36.7
Expanded uncertainty				1.8

TABLE 19. RESULTS FOR ^{228}Ac and ^{208}Tl IN IAEA-412
(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	39.7 ± 2.1	12.1 ± 0.5*
2	G	3	88.51	35.5 ± 5.2	11.0 ± 1.3
4	G	3	100.6	37.7 ± 1.5	—
5	G	3	76	37.3 ± 2.0	12.9 ± 0.9
7	G	3	82.48	33.5 ± 1.9	11.5 ± 0.7
9	G	3	99.77	36.5 ± 1.6	10.9 ± 0.3*
10	G	1	56	37.0 ± 5.0	12.5 ± 1.5
13	G	3	101	42.4 ± 2.9	13.3 ± 0.9*
15	G	1	234.4	38.2 ± 5.5	—
16	G	2	27	36.3 ± 4.7	—
18	G	3	80.2	36.4 ± 2.9	13.0 ± 0.9*
20	G	4	7.2	39.3 ± 3.1	—
21	G	5	99.89	34.4 ± 2.2	11.7 ± 0.4*
23	G	1	100	33 ± 3	11.9 ± 1.2
26	G	14	100	30.0 ± 2.2	13.1 ± 1.2
Number of reported laboratory means				15	11
Robust mean				36.5	12.2
Expanded uncertainty				1.7	0.7

* Values corrected (by IAEA-EL-RML) for branching factor of 35.93%.

TABLE 20. RESULTS FOR ^{228}Th IN IAEA-412
(Reference date: 1 January 2013, unit: Bq kg^{-1})

Lab code	Method code	No. of results	Mass (g)	^{228}Th
4	G	3	100.6	36.4 ± 1.6
6	A	2	2	44.4 ± 7.9
7	A	3	2	43.5 ± 7.9
9	G	3	99.77	38.6 ± 0.4
10	G	1	56	<170
15	G	1	234.4	40.0 ± 4.2
17	A	4	1	29.0 ± 8.1
18	G	3	80.2	36.3 ± 2.6
20	G	4	7.2	35.6 ± 1.1
21a	G	5	99.89	32.9 ± 1.5
21b	G	5	99.89	36.2 ± 1.7
21c	G	5	99.89	32.6 ± 1.1
25	G	2	70.62	39.0 ± 2.0
26a	A	4	3–10	48.1 ± 5.0
26b	A	4	3–10	49.6 ± 5.2
Number of reported laboratory means				14
Robust mean				38.3
Expanded uncertainty				3.8

TABLE 21. RESULTS FOR ^{212}Pb and ^{212}Bi IN IAEA-412
(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	100.74	38.5 ± 0.8	—
9	G	3	99.77	38.6 ± 0.4	40.1 ± 2.6
10	G	1	56	37.0 ± 4.0	35.0 ± 5.0
13	G	3	101	42.6 ± 2.7	46.0 ± 5.0
15	G	1	234.4	39.5 ± 2.1	40.4 ± 3.1
16	G	2	27	38.3 ± 3.6	—
18	G	3	80.2	36.3 ± 2.6	—
21	G	5	99.89	32.9 ± 1.5	36.2 ± 1.7
26	G	14	100	38.2 ± 2.9	19.5 ± 1.8
Number of reported laboratory means				9	6
Robust mean				38.1	38.0
Expanded uncertainty				1.6	5.0

TABLE 22. RESULTS FOR ^{40}K IN IAEA-412
(Reference date: 1 January 2013, unit: Bq kg^{-1})

Lab code	Method code	No. of results	Mass (g)	^{40}K
1	G	3	100.74	640 ± 16
2	G	2	88.51	570 ± 63
3	G	1	97.58	513 ± 65
4	G	3	100.6	550 ± 23
5	G	3	76	574 ± 64
6	G	5	100.19	643 ± 49
7	G	3	82.48	544 ± 15
8	G	5	34	579 ± 41
9	G	3	99.77	558 ± 11
10	G	1	56	560 ± 60
11	G	3	88	460 ± 38
12	G	10	83.5	560 ± 14
13	G	3	101	567 ± 36
14	G	3	101.45	554 ± 25
15	G	1	234.4	563 ± 17
16	G	2	27	582 ± 47
17	G	2	80	531 ± 74
18	G	3	80.2	544 ± 60
19	G	3	25	678 ± 54
20	G	4	5.23	584 ± 21
21	G	5	99.89	499 ± 22
23	G	1	100	550 ± 30
25	G	1	70.62	588 ± 36
26	G	14	100	540 ± 40

Number of reported laboratory means	24
Robust mean	561
Expanded uncertainty	14

TABLE 23. RESULTS FOR THE LESS FREQUENTLY MEASURED RADIONUCLIDES REPORTED IN IAEA-412

(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	10	G	1	56	<0.4
^{129}I	17	AMS	5	5	$(3.5 \pm 0.7) \times 10^{-4}$
	22	AMS	6	0.5–5	$(2.6 \pm 0.1) \times 10^{-4}$
^{134}Cs	10	G	1	56	<0.32
^{155}Eu	6	G	4	100.19	3.92 ± 1.80
	9	G	3	99.77	1.87 ± 0.13
^{211}Pb	21	G	5	99.89	2.2 ± 0.3
^{219}Rn	21	G	5	99.89	2.2 ± 0.3
^{223}Ra	4	G	3	100.6	1.65 ± 0.26
^{224}Ra	10	G	1	56	36 ± 4
	15	G	1	234.4	66.2 ± 4.3
	26	G	14	100	66.4 ± 4.5
^{227}Th	4	G	3	100.6	1.17 ± 0.22
	21	G	5	99.89	1.7 ± 0.2
^{231}Pa	10	G	1	56	<6
^{234m}Pa	10	G	1	56	44 ± 14
^{236}U	10	ICP-MS	3	0.3–0.6	<0.003 9
	24	AMS	5	0.5	0.005 4 ± 0.000 8

TABLE 24. SUMMARY OF CERTIFIED VALUES FOR IAEA-412
(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	561	14	24
^{137}Cs	6.50	0.28	23
^{210}Pb(^{210}Po)[d]	100.9	4.4	26
^{226}Ra	27.4	2.6	22
^{228}Ra	36.7	1.8	13
^{228}Th	38.3	3.8	14
^{232}Th	36.3	2.0	9
^{235}U	1.38	0.20	18
^{238}U	31.2	3.2	19
^{239}Pu	0.358	0.016	5
^{240}Pu	0.240	0.016	5
$^{239+240}$Pu	0.611	0.028	18

Note: Tables 24–25:

[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 25. SUMMARY OF INFORMATION VALUES FOR IAEA-412
(Reference date: 1 January 2013, unit: Bq kg^{-1})

Radionuclide	Information value[a] [Bq kg^{-1}]	Expanded uncertainty[b] [Bq kg^{-1}]	N[c]
^{230}Th	26	6	7
^{234}Th	37	8	8
^{234}U	29	6	11
^{238}Pu	0.019 9	0.004 0	6
^{241}Am	0.30	0.10	8

APPENDIX II.
LABORATORY RESULTS – GRAPHS

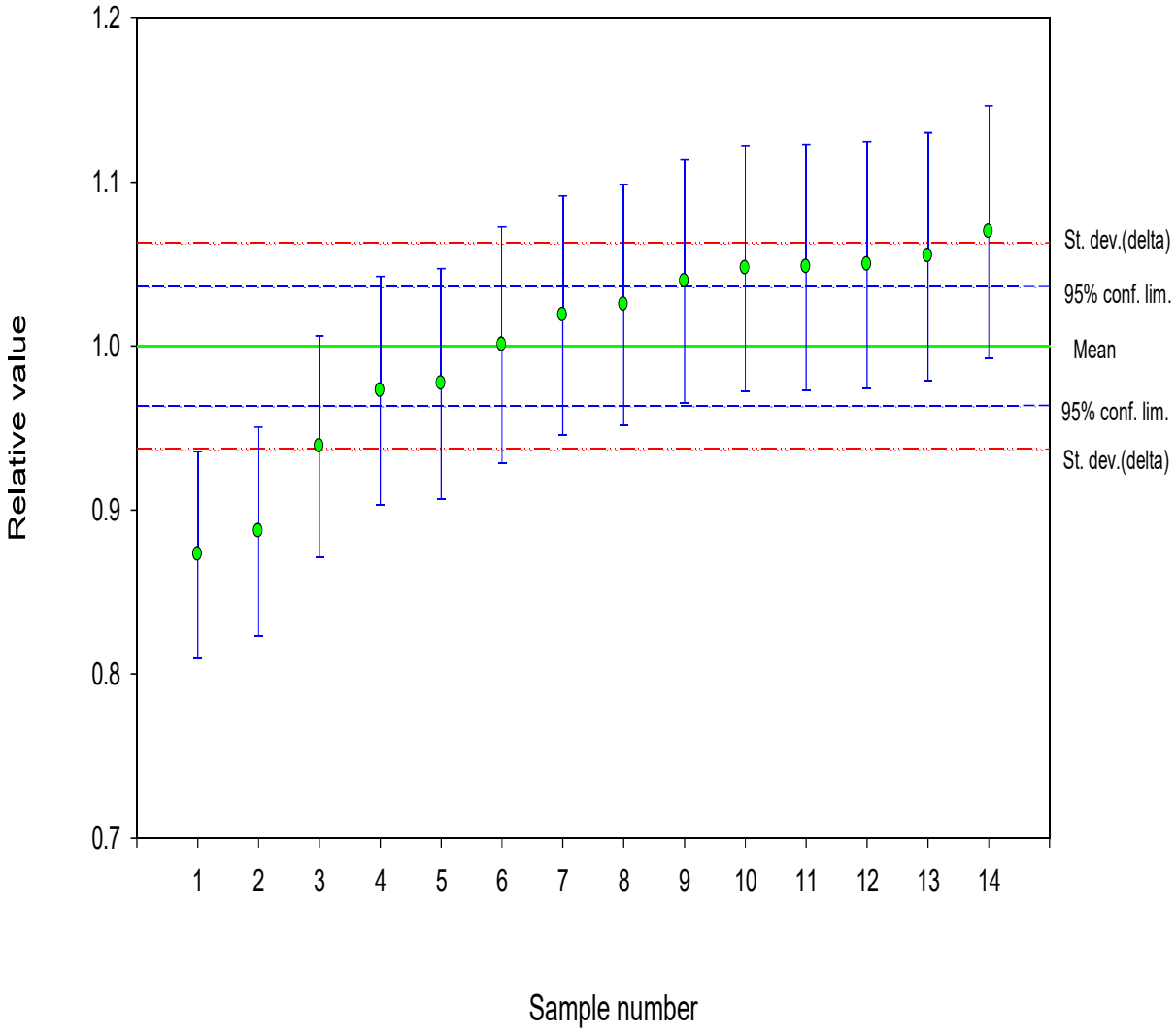

FIG.1. Homogeneity test for ^{40}K in IAEA-412

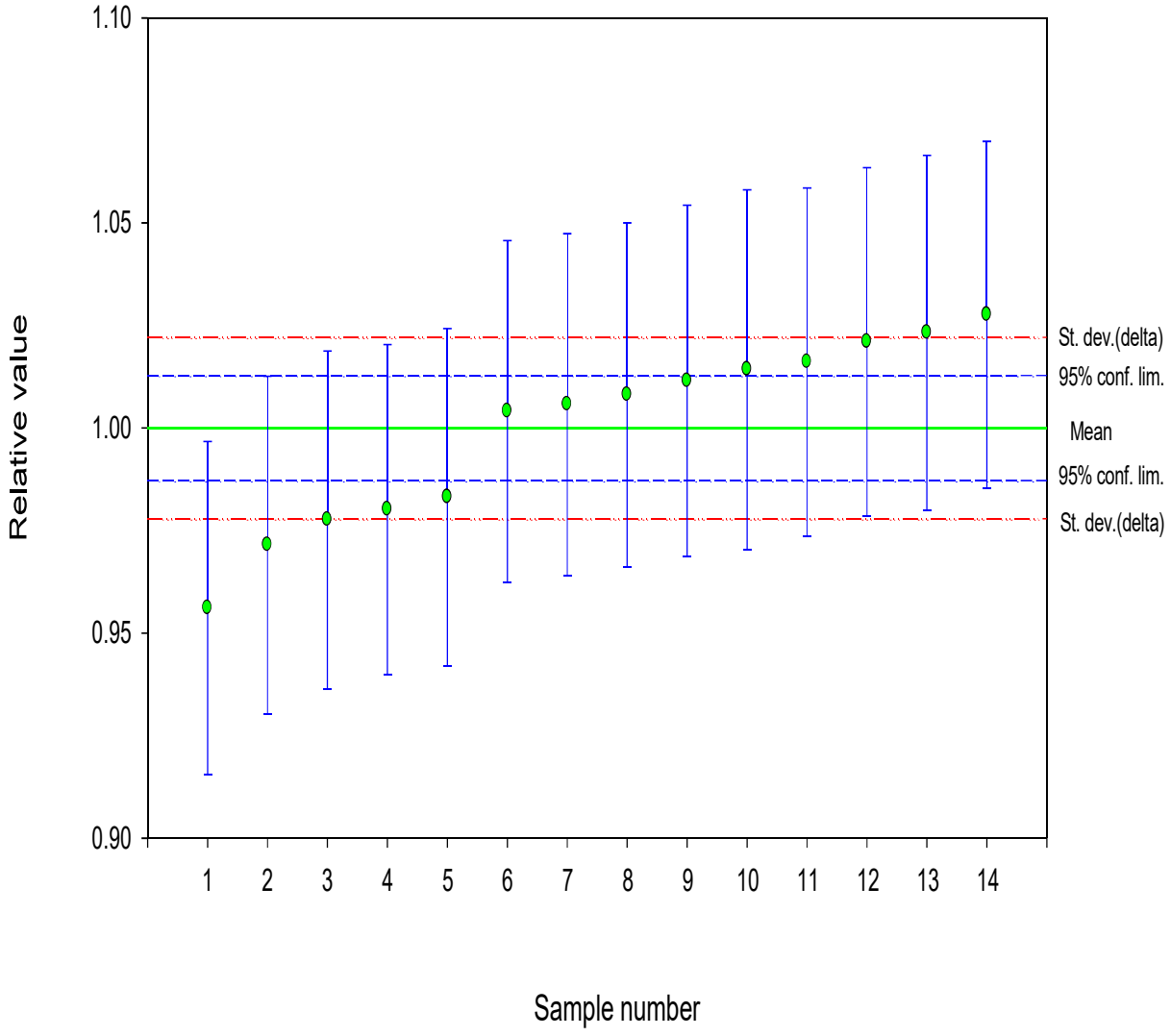

FIG.2. Homogeneity test for ^{137}Cs in IAEA-412

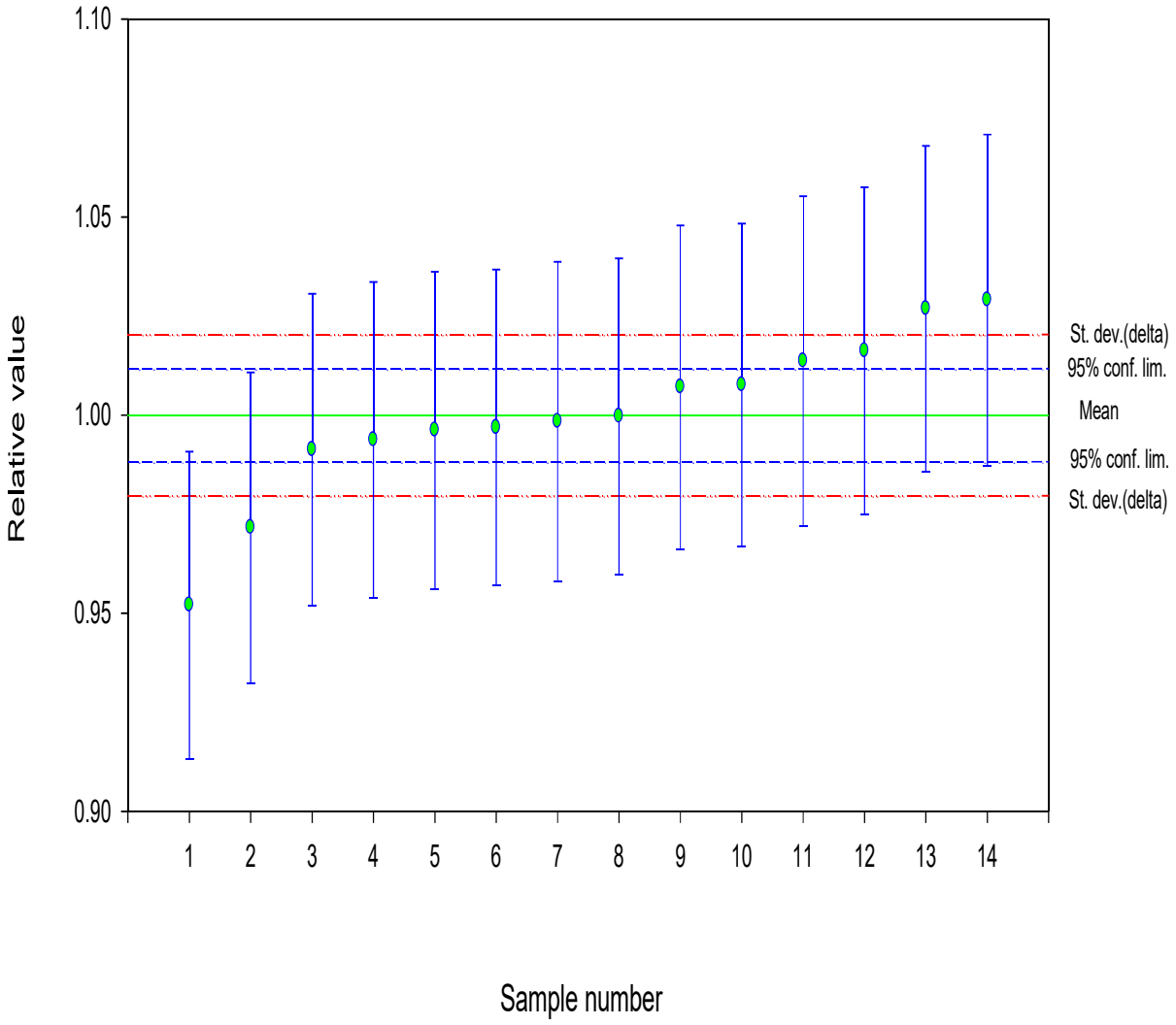

FIG.3. Homogeneity test for ^{214}Pb in IAEA-412

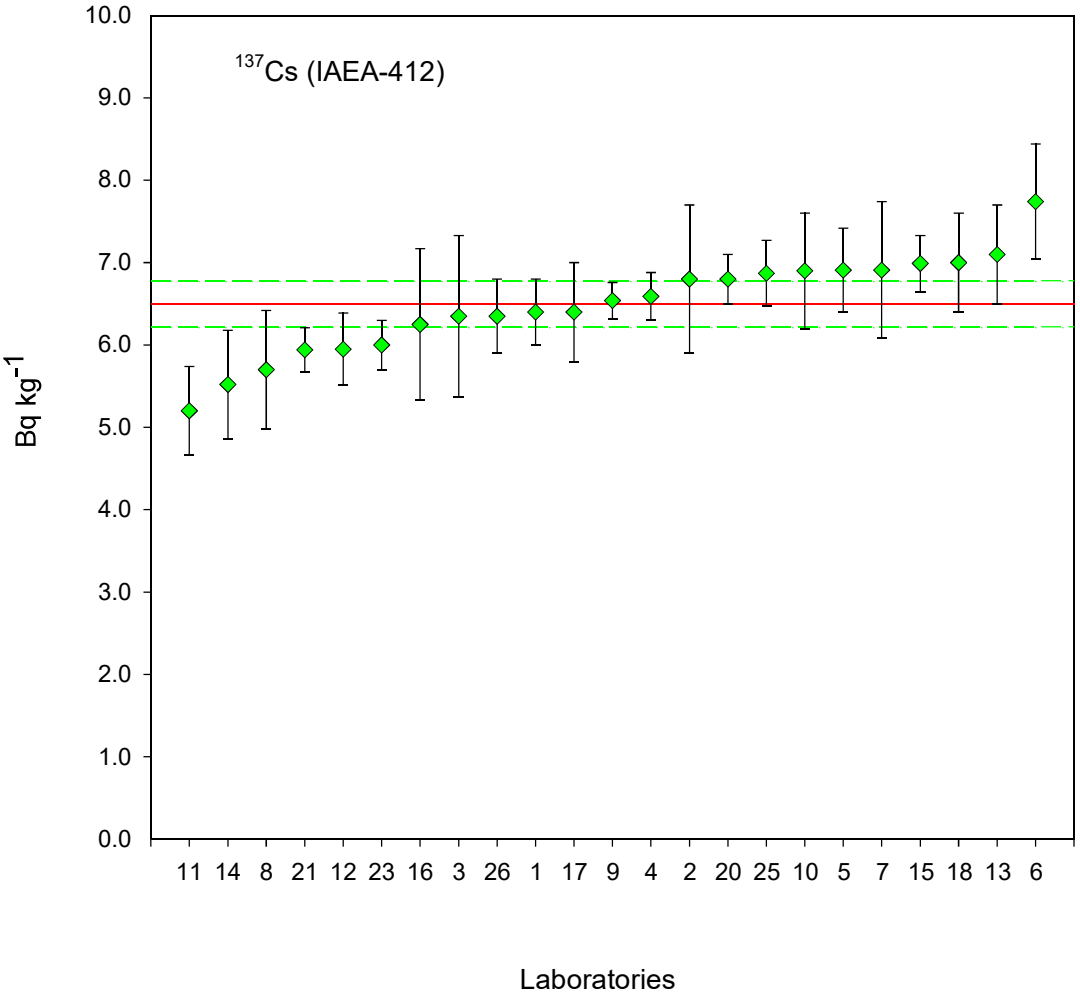

FIG.4. Laboratory results for ^{137}Cs

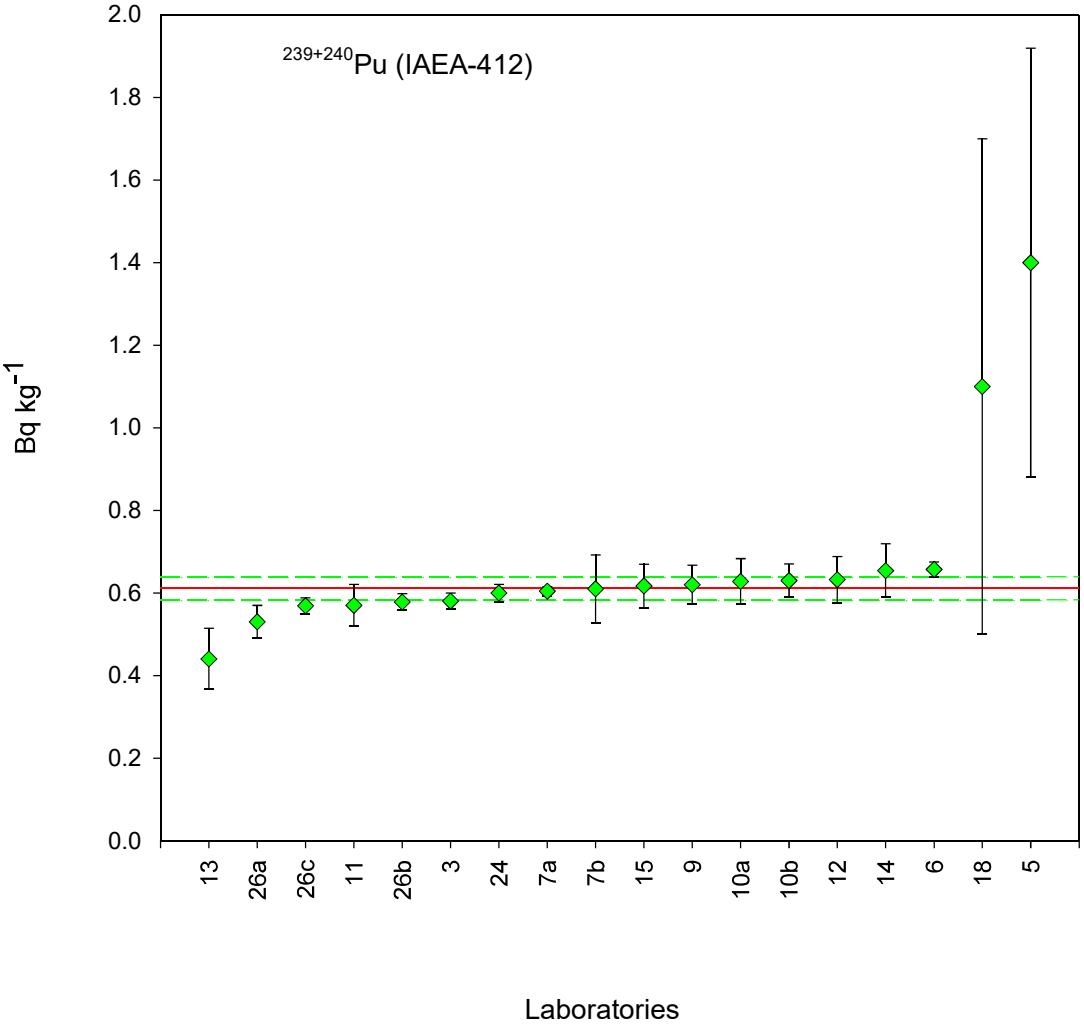

FIG.5. Laboratory results for $^{239+240}Pu$

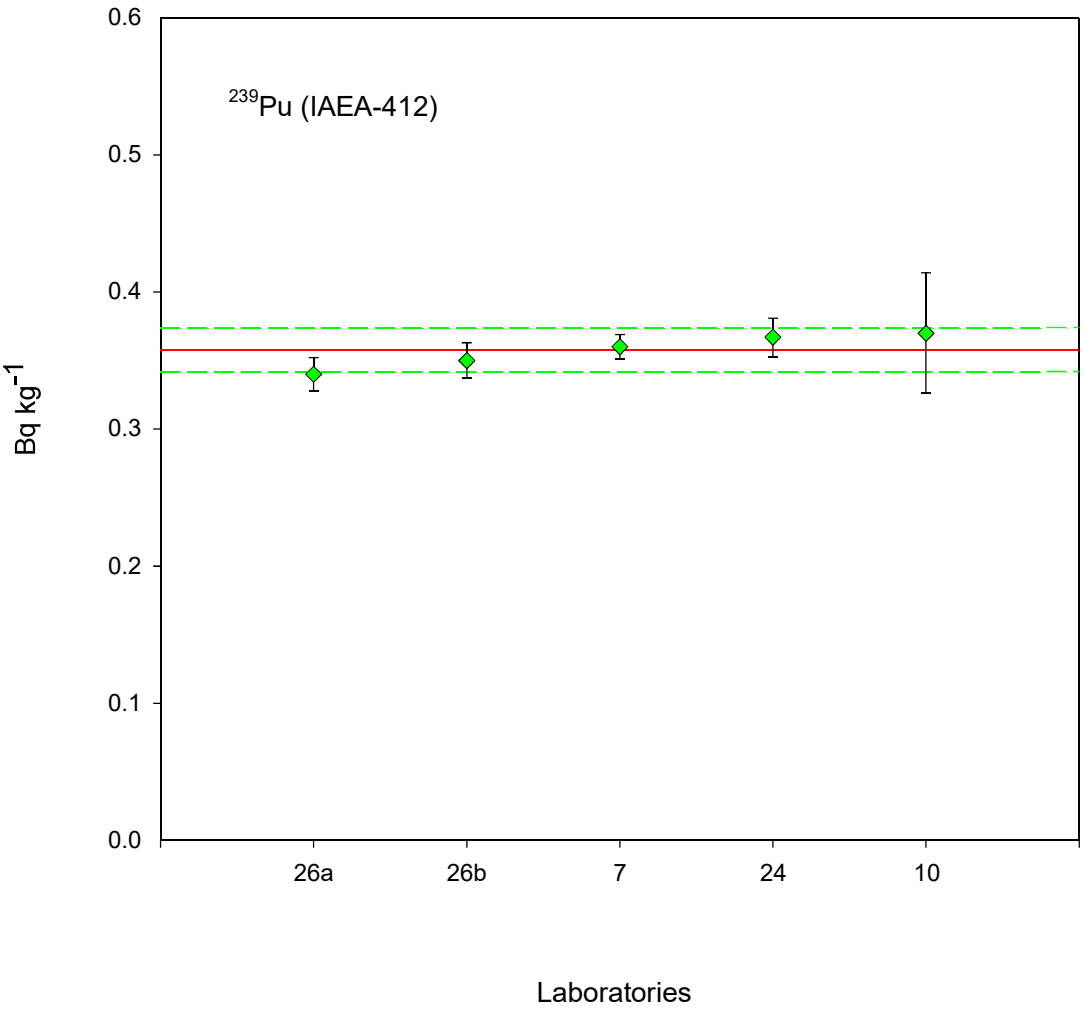

FIG.6. Laboratory results for ^{239}Pu

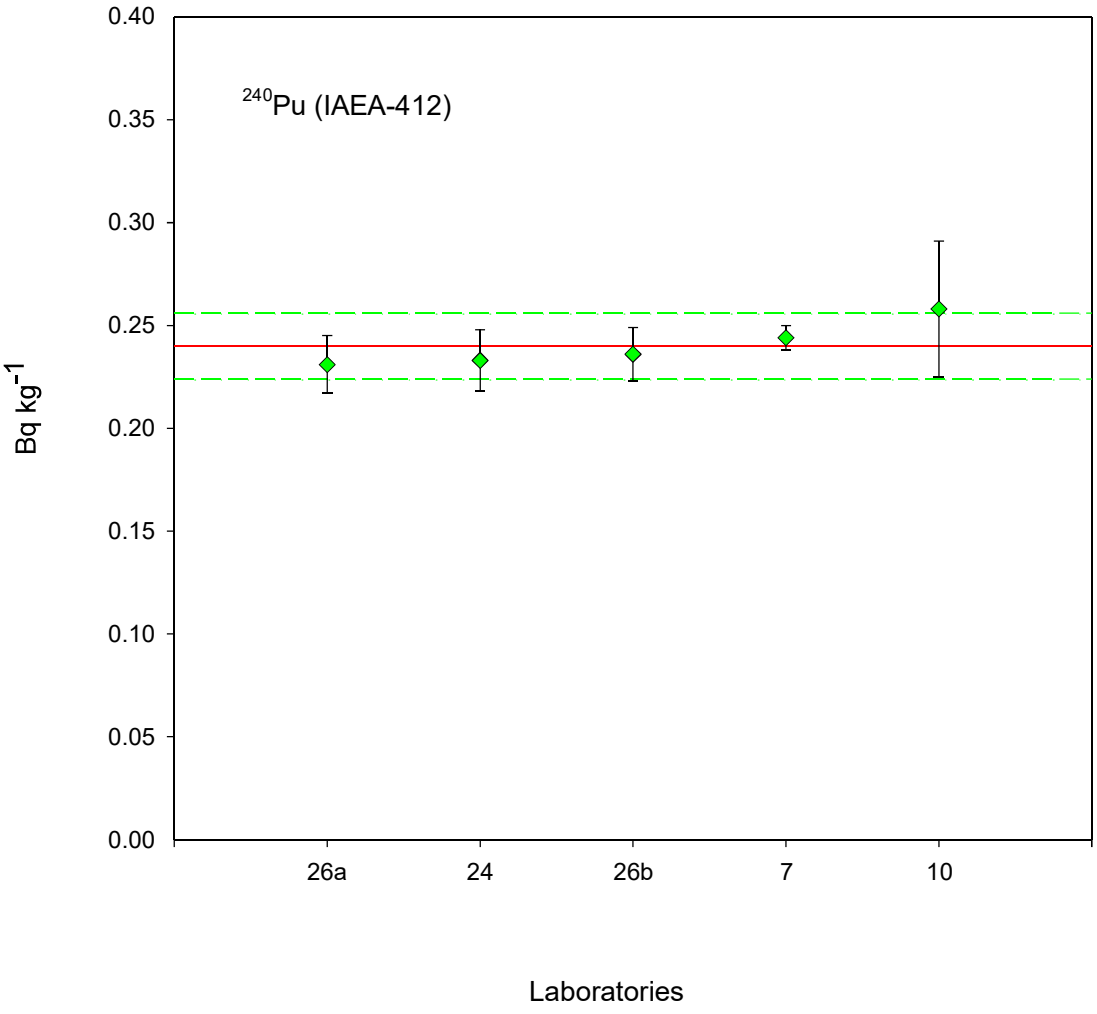

FIG.7. Data evaluation for ^{240}Pu

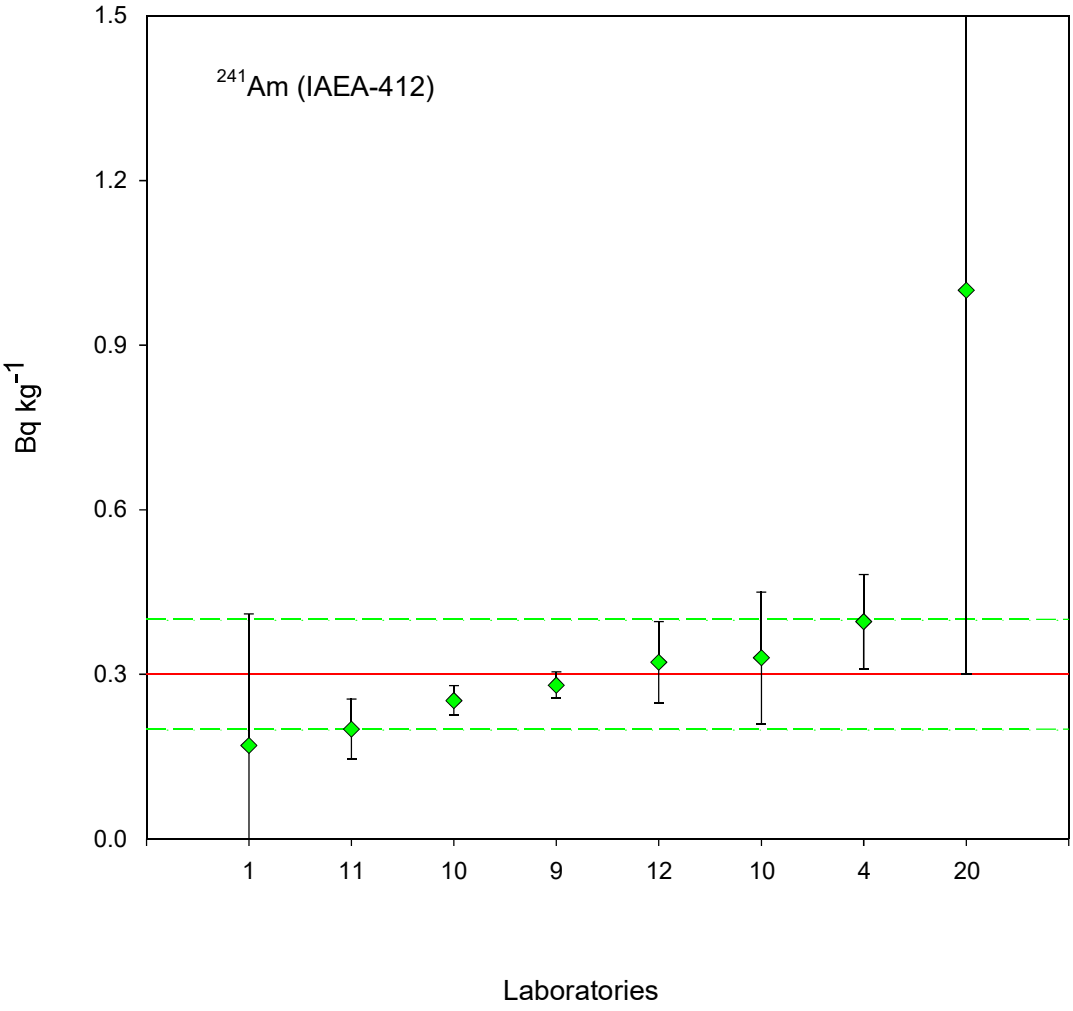

FIG.8. Laboratory results for ^{241}Am

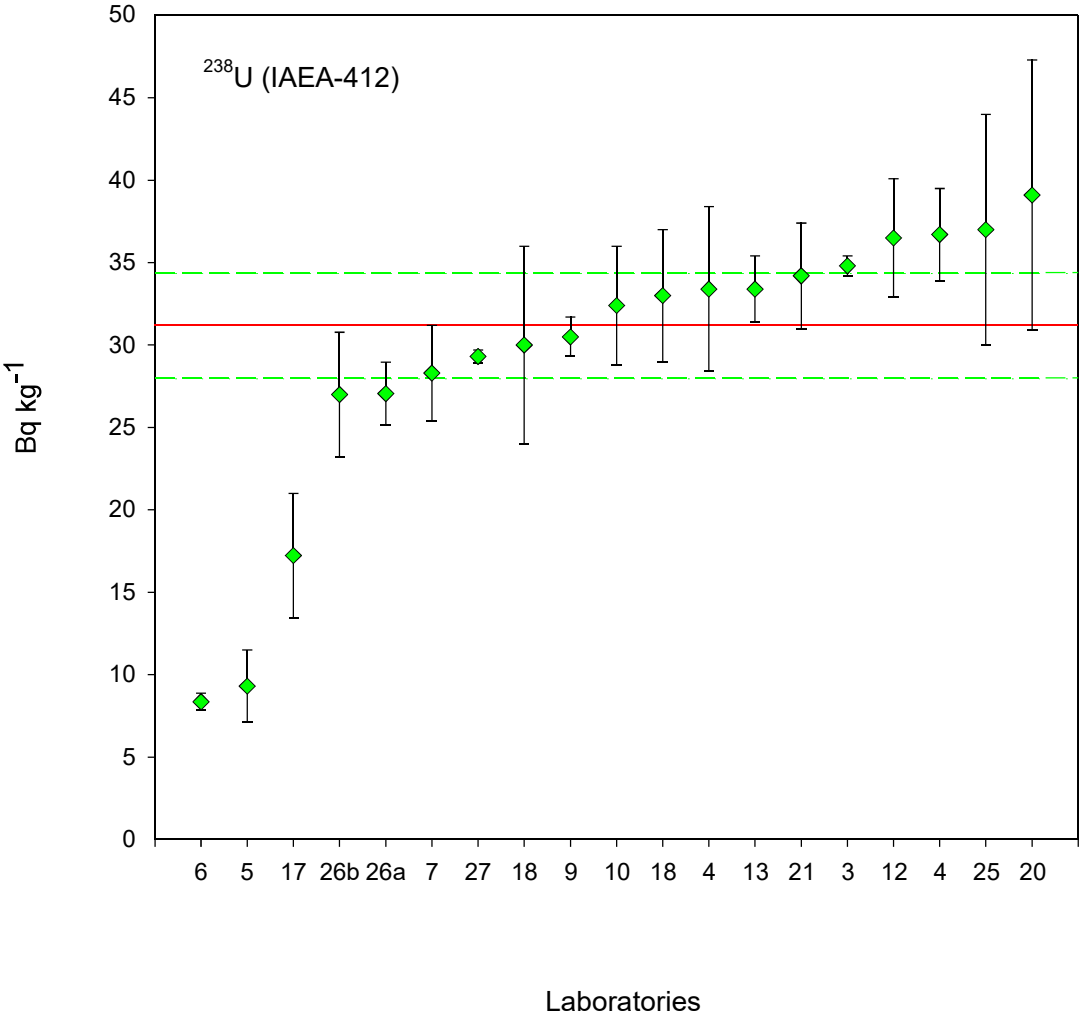

FIG.9. Laboratory results for ^{238}U

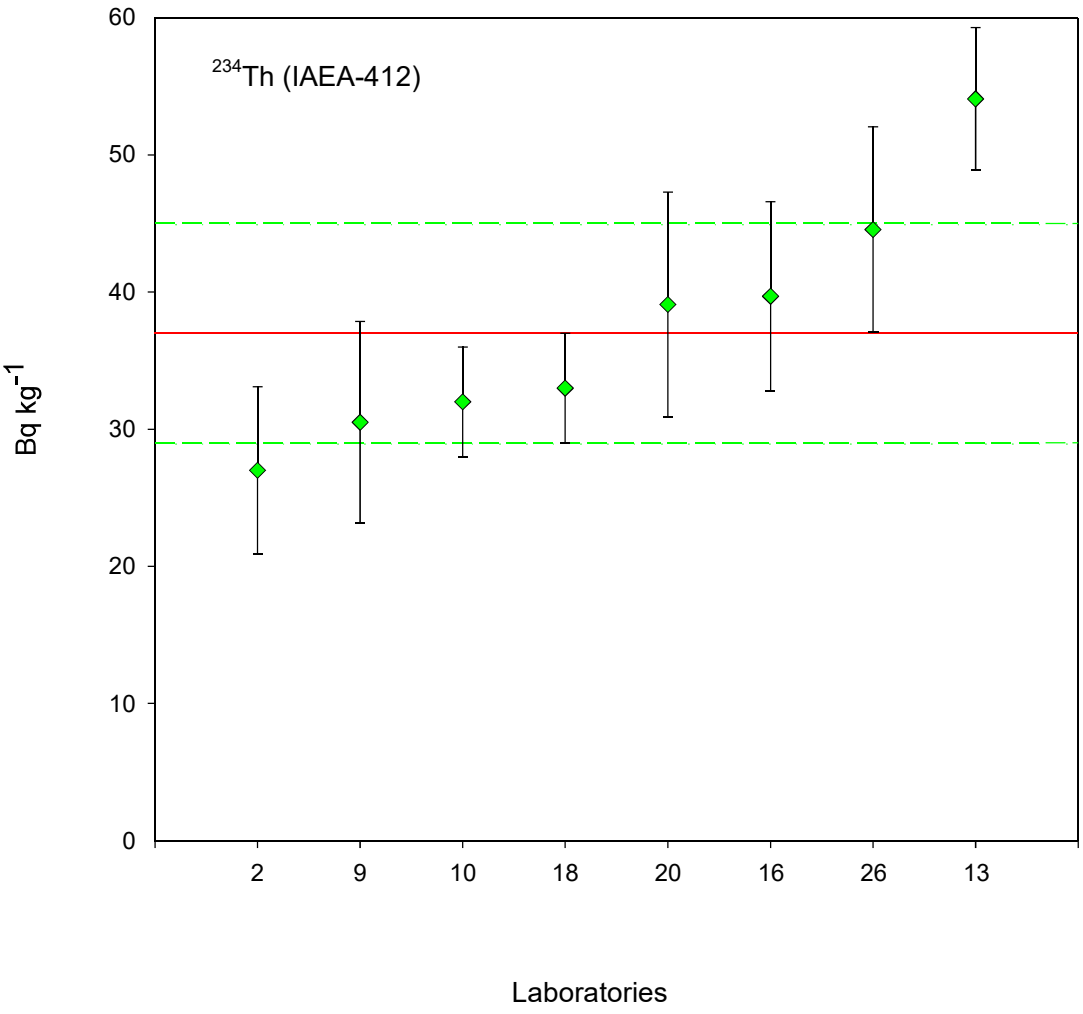

FIG.10. Laboratory results for ^{234}Th

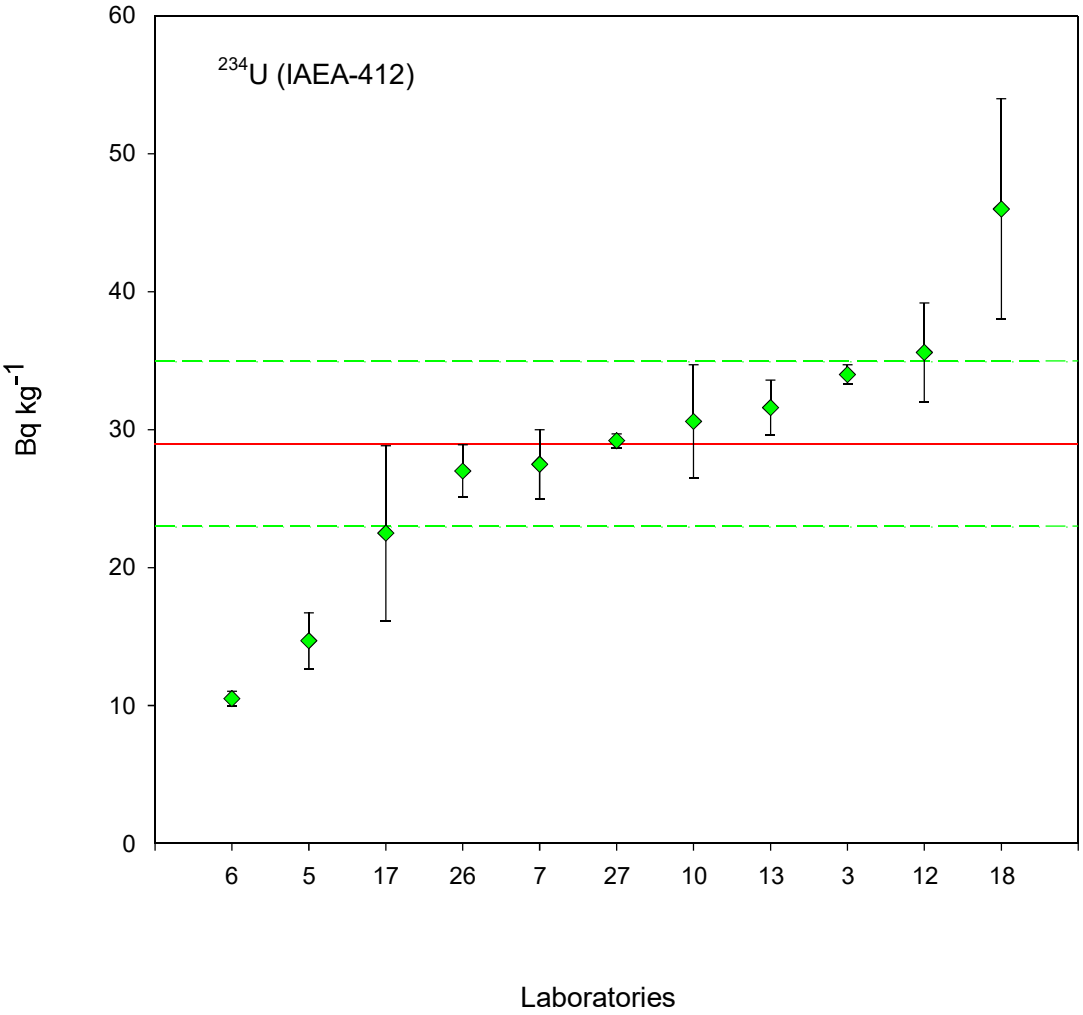

FIG.11. Laboratory results for ^{234}U

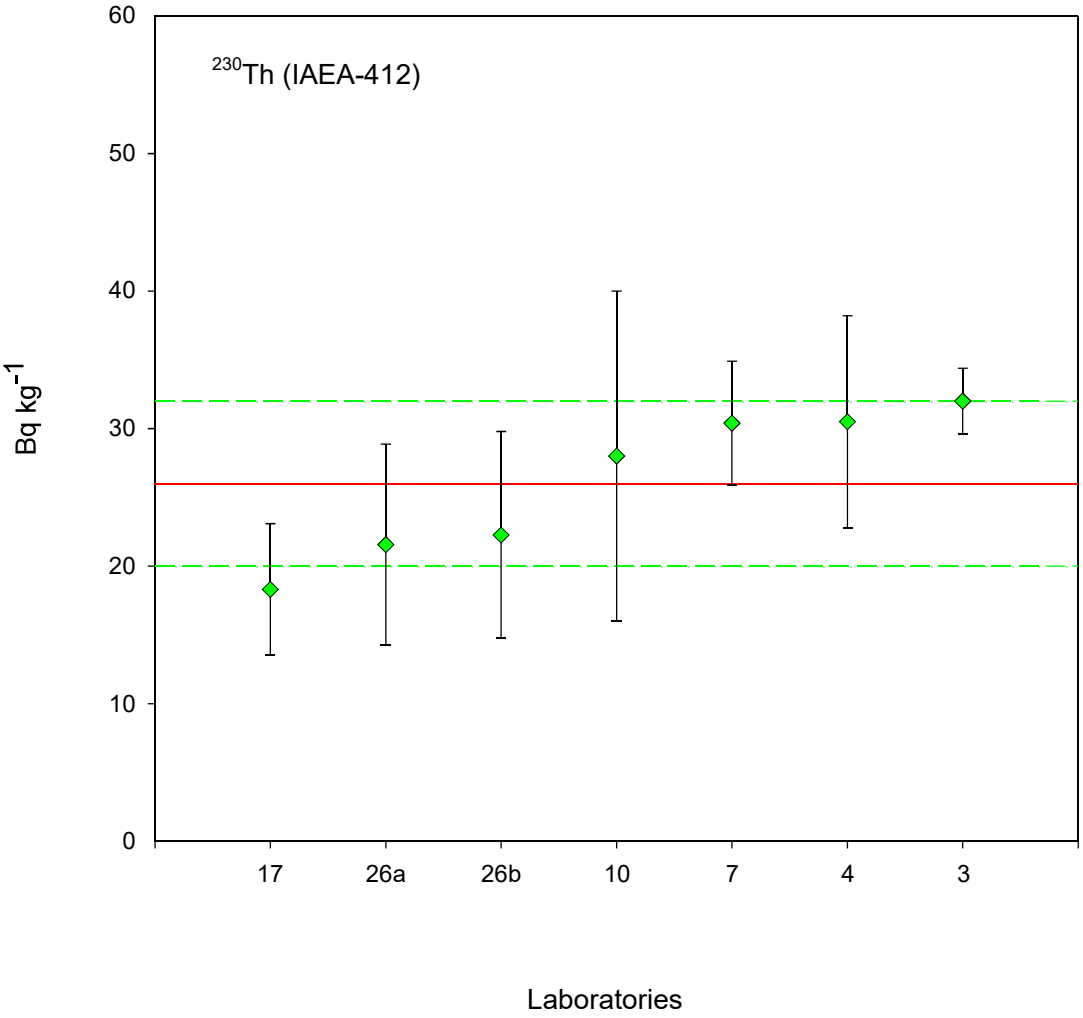

FIG.12. Laboratory results for ^{230}Th

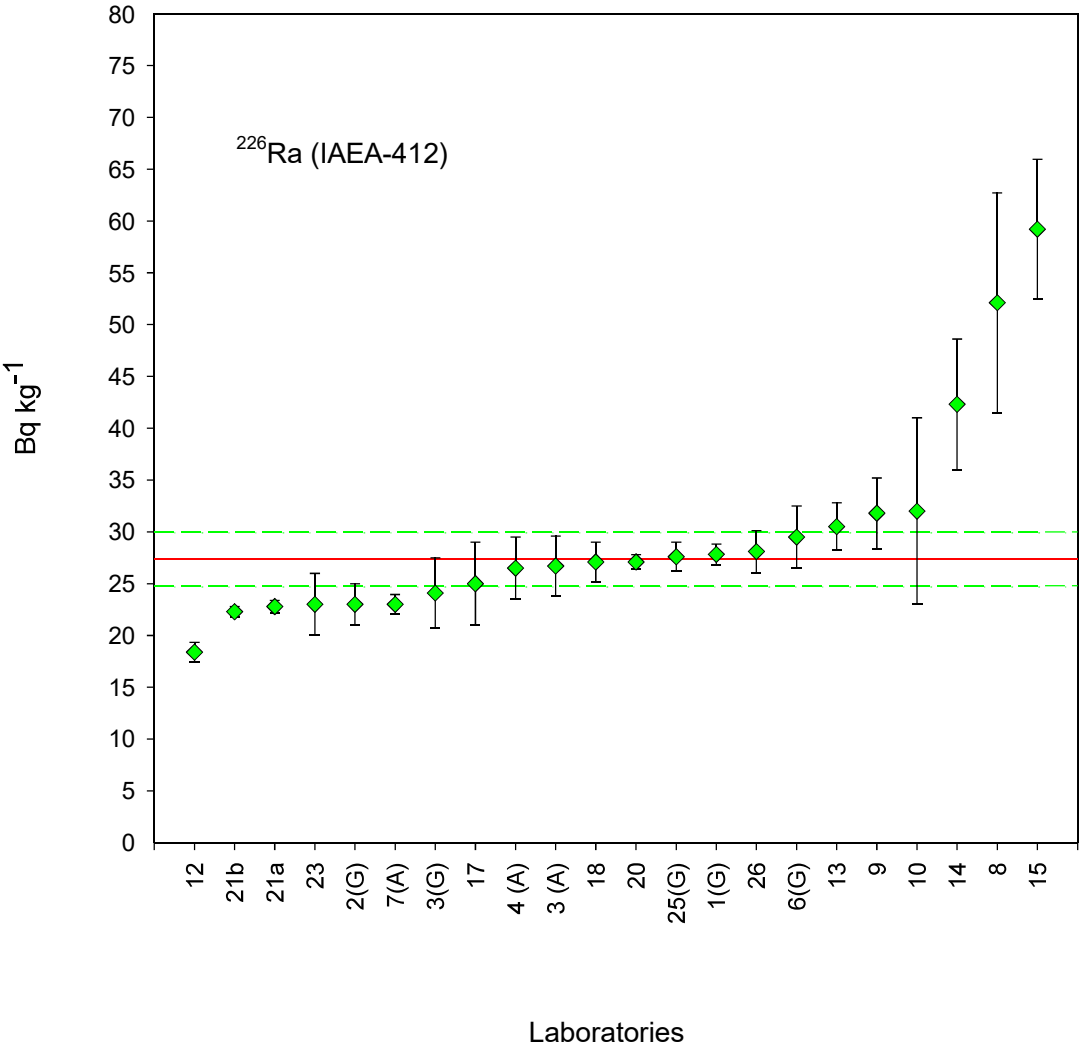

FIG.13. Laboratory results for ^{226}Ra

(Figs. 13–19: A = alpha spectrometry; G = gamma spectrometry)

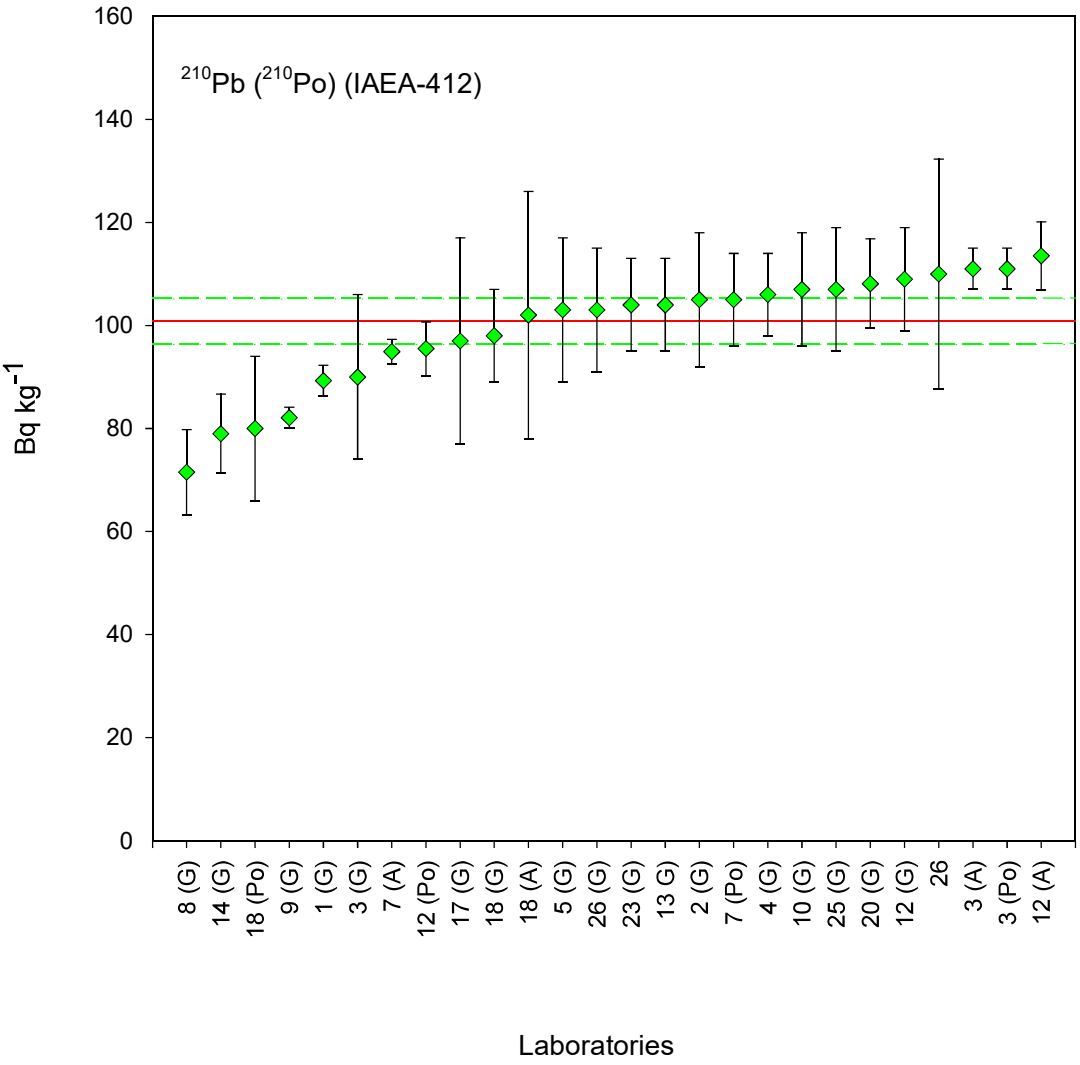

FIG.14. Laboratory results for ^{210}Pb (^{210}Po)

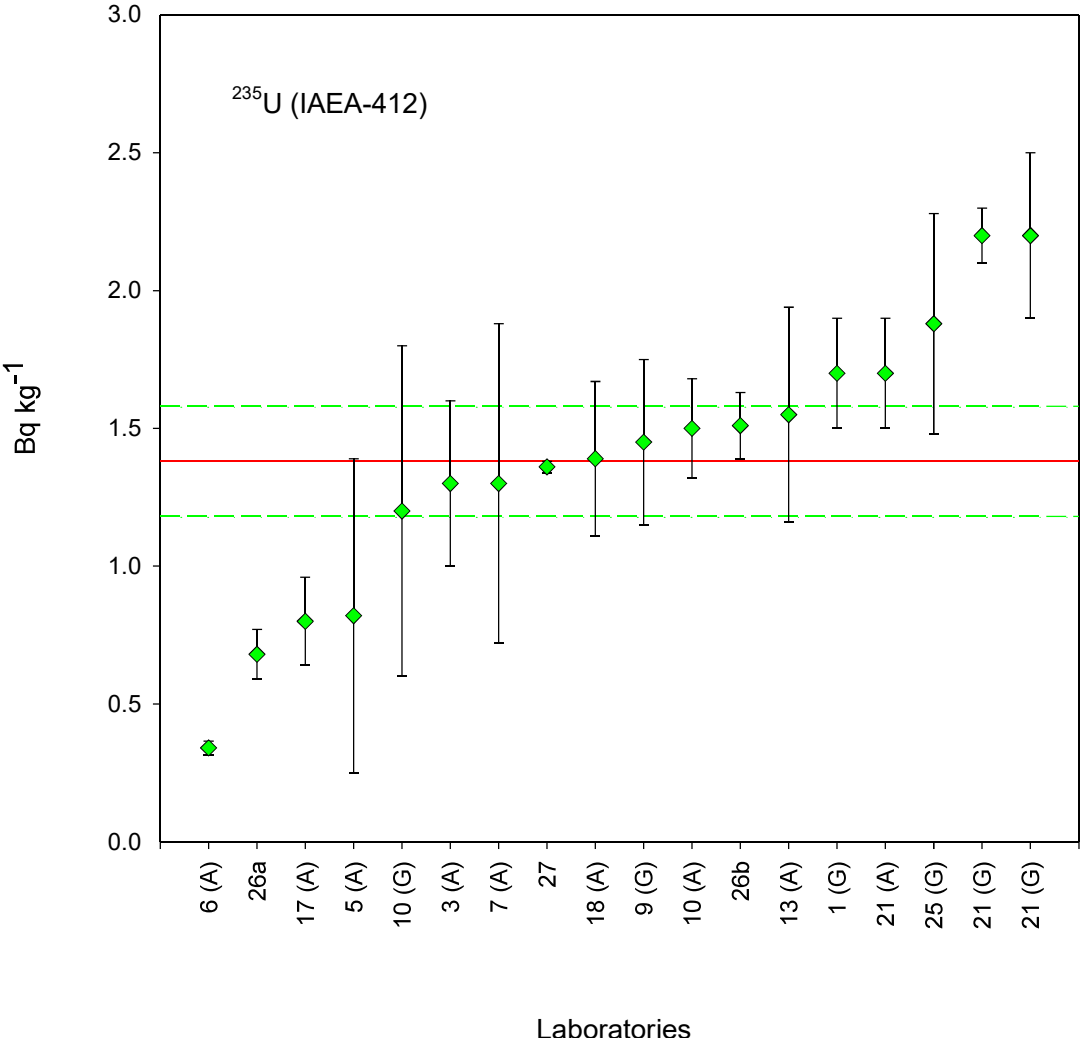

FIG.15. Data evaluation for ^{235}U

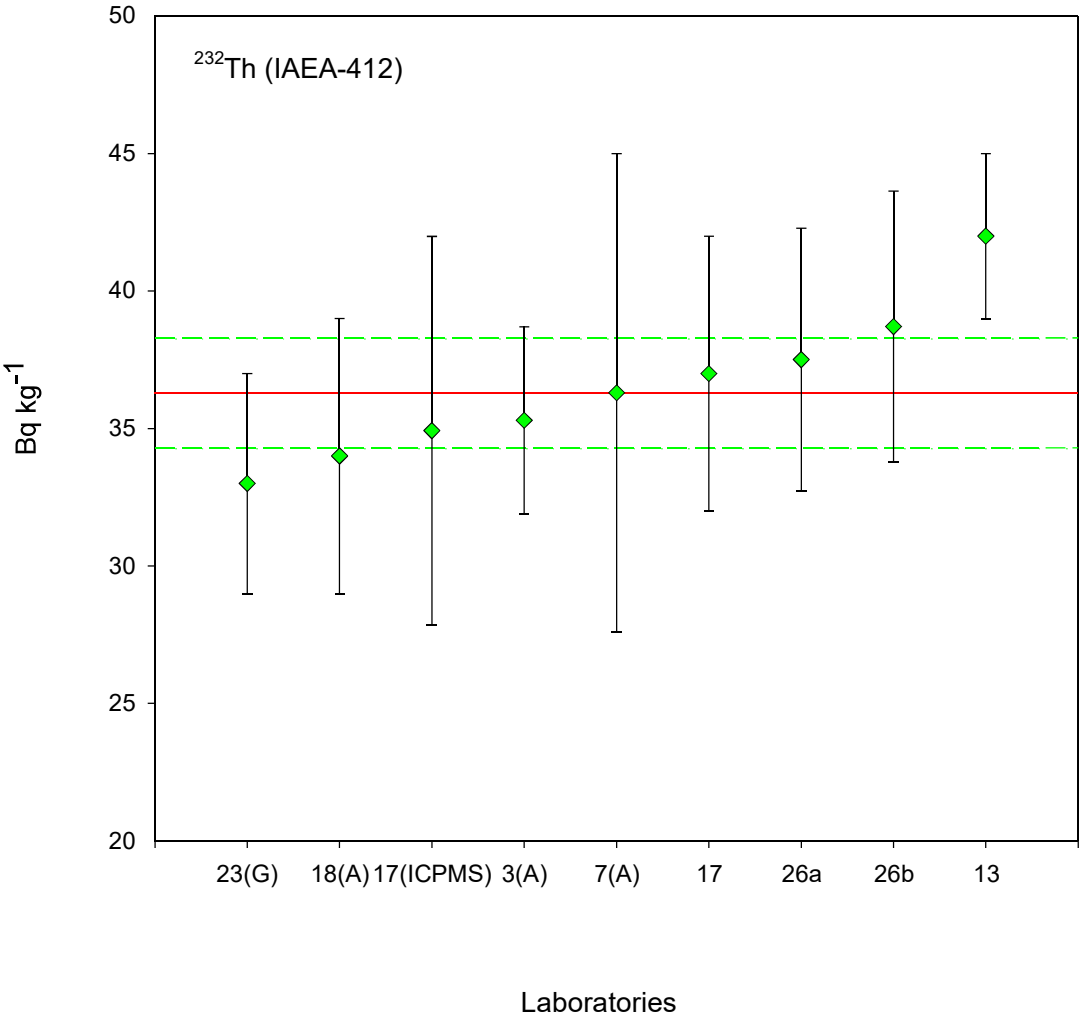

FIG.16. Laboratory results for ^{232}Th

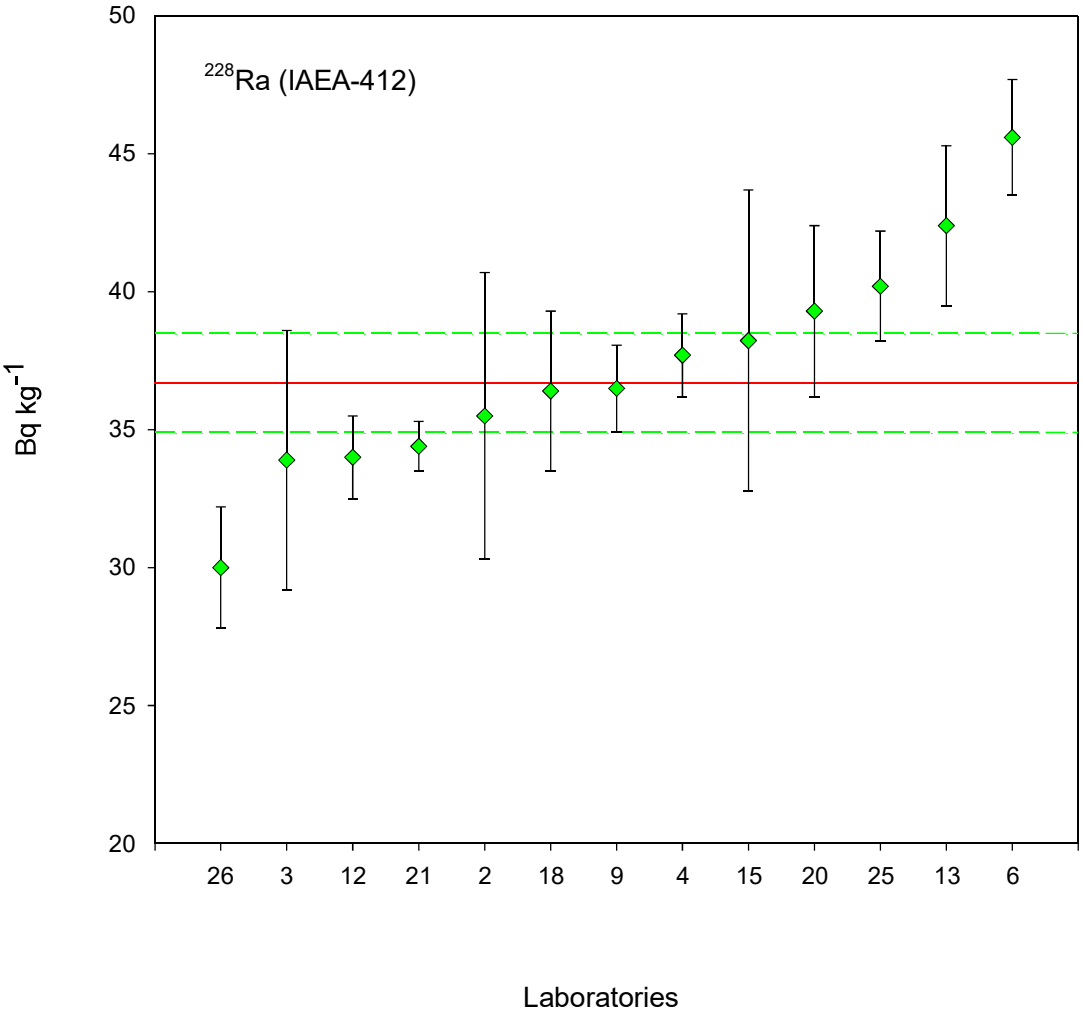

FIG.17. Laboratory results for ^{228}Ra

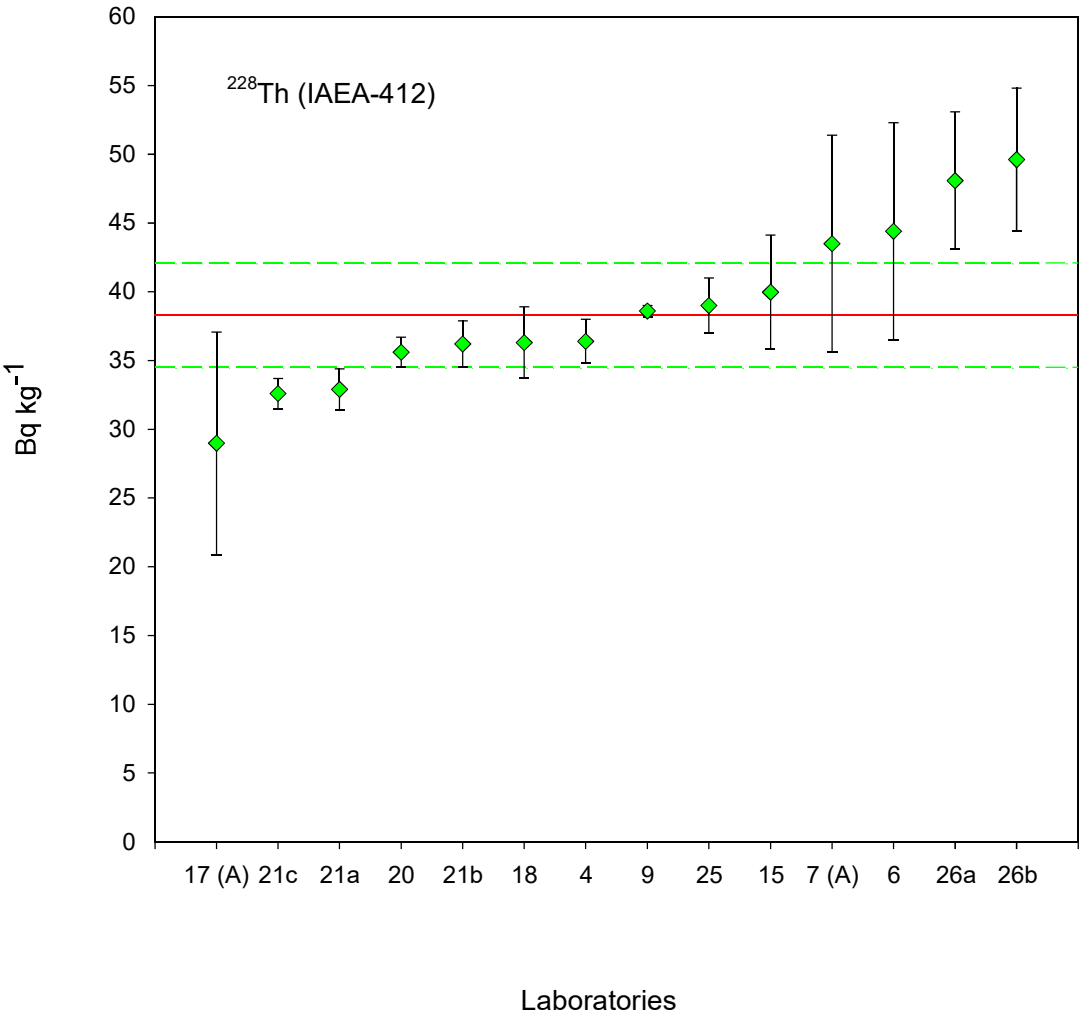

FIG.18. Laboratory results for ^{228}Th

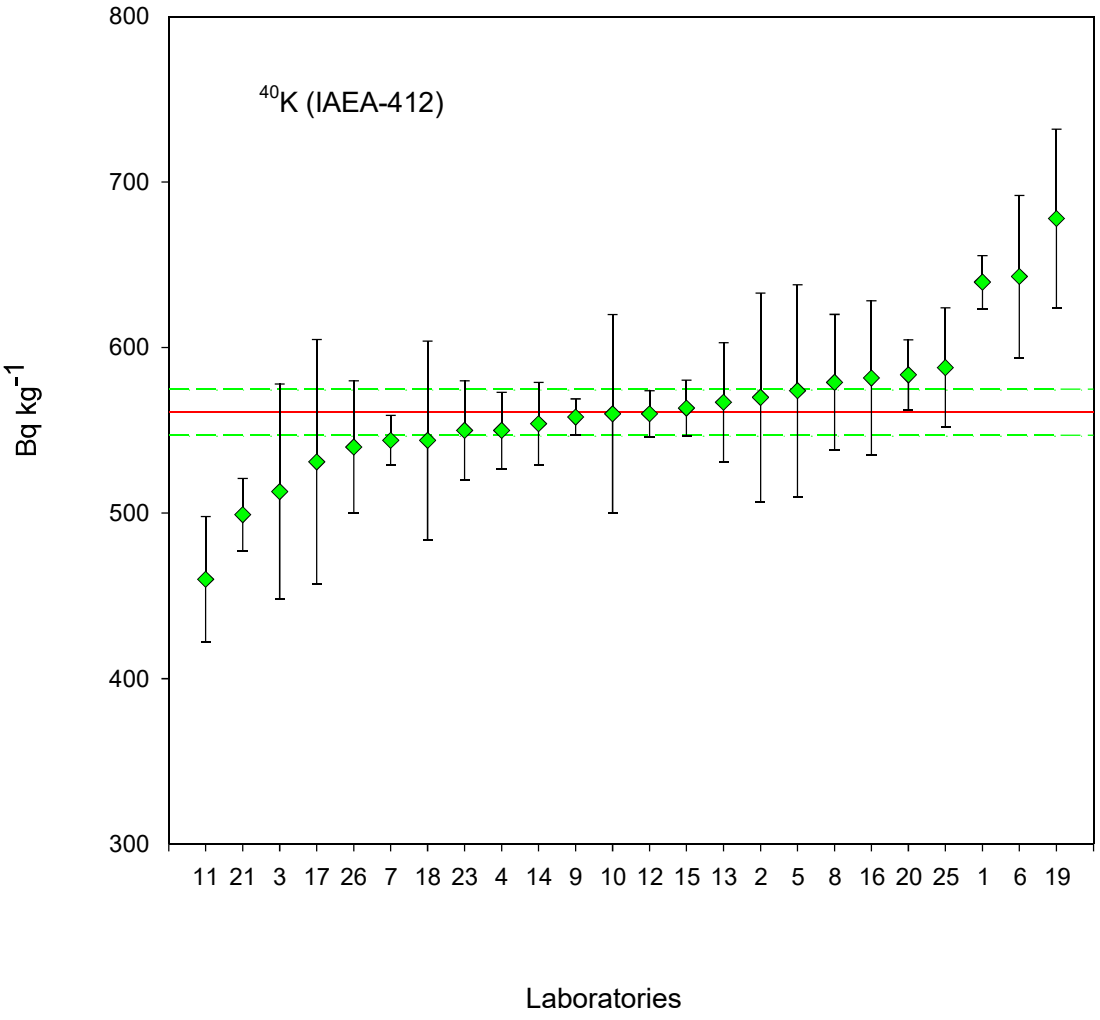

FIG.19. Laboratory results for ^{40}K

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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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