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Worldwide Laboratory Comparison on the Determination of Radionuclides in IAEA-446 Baltic Sea Seaweed (*Fucus vesiculosus*)

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WORLDWIDE LABORATORY COMPARISON ON THE DETERMINATION OF RADIONUCLIDES IN IAEA-446 BALTIC SEA SEAWEED (*Fucus vesiculosus*)

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WORLDWIDE LABORATORY COMPARISON ON THE DETERMINATION OF RADIONUCLIDES IN IAEA-446 BALTIC SEA SEAWEED (*Fucus vesiculosus*)

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FOREWORD

The Radiometrics Laboratory of the IAEA Environment Laboratories in Monaco has been providing quality products and services for the past forty years, including the organization of interlaboratory comparisons, the production of reference and certified reference materials and the provision of training. More than 45 reference materials have been produced, including a wide range of marine sample matrices and radionuclide concentrations.

As part of these activities, a new interlaboratory comparison was organized to provide participating laboratories with the opportunity to test the performance of their analytical methods on a seaweed sample with elevated radionuclide levels due to the effects of the Chernobyl accident on the Baltic Sea region. The material used in the analysis of anthropogenic and natural radionuclides in seaweed was the bladder wrack (*Fucus vesiculosus*). It is expected that the sample, after successful certification, will be issued as a certified reference material for analysing radionuclides in seaweed.

The participating laboratories were informed that the IAEA publication would contain a list of the laboratories and the results and descriptions of the interlaboratory comparisons, but that the results would not be attributed to individual laboratories.

The IAEA officers responsible for this publication were Mai Khanh Pham and H. Nies of the IAEA Environment Laboratories.

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 inter-laboratory comparisons on radionuclides in marine samples as an integral part of the sub-programme IAEA Reference Products for Science and Trade [1-2].

In collaboration with HELCOM-MORS (Helsinki Commission's Project for Monitoring Radioactive Substances in the Baltic Sea), the Risø National Laboratory, Denmark (Risø) collected seaweed from the Baltic Sea in July 2006. Nine laboratories from HELCOM-MORS from 7 countries and 11 laboratories from 9 OSPAR contracting parties (some of them belonging to both organizations) participated in the exercise to test their performance in analysing radionuclides in seaweed. The results obtained from a total of 29 laboratories including expert laboratories and the Radiometrics Laboratory of the IAEA Environment Laboratories in Monaco (IAEA-EL-RML) will allow the IAEA-EL-RML to produce a new certified reference material.

As the sample was collected in the Baltic Sea, elevated levels of anthropogenic radionuclides were expected due to the influence of the historical Chernobyl accident to the Baltic Sea region. Participants were informed that the expected activities for anthropogenic radionuclides would be in the ranges:

Gamma-emitters	1–15 kBq kg^{-1}
Beta-emitters	0.1–1 Bq kg^{-1}
Transuranics	0.01–0.05 Bq kg^{-1}

This report describes the results on anthropogenic and natural radionuclide determinations in seaweed obtained from 29 laboratories including IAEA-EL-RML.

2. SCOPE OF THE INTERLABORATORY COMPARISON

This inter-laboratory comparison was organized to provide the participating laboratories with the possibility to test the performance of their analytical methods on a seaweed sample with elevated radionuclide levels due to the influence of the historical Chernobyl accident to the Baltic Sea region.

The inter-laboratory comparison material 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, ^{90}Sr, ^{99}Tc, ^{129}I, ^{137}Cs, ^{210}Pb, ^{210}Po, ^{226}Ra, ^{228}Ra, U, Th, Pu isotopes, etc. 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 spectrometry, beta counting…) and mass spectrometry measurement techniques (ICMPS, TIMS, AMS, etc.) analyses.

It is expected that the sample, after successful certification, will be issued as a certified reference material for radionuclides in seaweed.

3. DESCRIPTION OF THE MATERIAL

A total of 718 kg wet mass of seaweed, which is bladder wrack with the Latin name *Fucus vesiculosus* was collected from a coastline in the western part of the Baltic Sea (54°57' N, 11°59' E) between 11 and 27 July 2006 by the Risø National Laboratory, Denmark. The seaweed was first dried in open air and subsequently in heating cabinets at 85°C leaving a total dry mass of 105 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 (100g units) and coded as IAEA-446 (for a total of 900 bottles). All bottles were sterilized at 10 kGy (^{60}Co) in an irradiation facility (Isotron, France). The moisture content of the sample was found to be approximately 5.5%. As the results were reported on a dry mass basis, the moisture content of the samples should be determined prior to use by drying at 60°C in an oven until a constant mass is obtained and the results corrected accordingly.

4. HOMOGENEITY TESTS

Sample homogeneity was checked by the determination of ^{137}Cs, ^{40}K, ^{90}Sr, ^{210}Po, ^{214}Bi, ^{226}Ra, ^{228}Th, ^{230}Th, ^{232}Th and $^{239+240}$Pu activities (by using high-resolution low-background gamma spectrometry, low-level beta proportional counter and alpha spectrometry). The first homogeneity test between bottles was done for 10 × 3 aliquots chosen at random at different masses of samples (between 10 g and 20 g, between 50 g and 60 g for gamma spectrometry, beta counting, and alpha spectrometry, except a small volume of 0.5 g for ^{210}Po analysis by alpha spectrometry). The second test within bottles was done for another 10 × 3 aliquots at 5 g to 10 g of sample for gamma emitters and Pu isotopes analysis by alpha spectrometry and 0.2 g of material for U isotopes determination using ICP-MS, respectively. Homogeneity was tested by using one-way analysis of variance. The coefficient variation was below 15%–20% for all radionuclides determined (some examples are shown in Table 1, Appendix I), depending on their activity range. 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 (Figs. 1, 2 and 3, Appendix II for ^{137}Cs, ^{214}Bi, and $^{239+240}$Pu, for instance), the material can be considered homogeneous for the radionuclides tested at the mass used.

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 seaweed sample was done by XFR. The coefficient of variation was below 10% for XRF determined elements.

5. SAMPLE DISPATCH AND DATA RETURN

Each participant received 100 g of the seaweed sample. For each radionuclide analysed, the following information was requested:

- Average mass of sample
- The moisture content of the samples should be determined prior to use by drying at 60 °C in an oven until a constant mass is obtained
- Number of analyses
- Massic activity calculated in net values (i.e. corrected for blank, background etc.) and expressed in Bq kg^{-1}
- Estimate of the total uncertainty (counting and other uncertainties)
- Description of chemical procedures and counting equipment
- Reference standard solutions used
- Chemical recoveries, counting time, half-life

The massic activities should 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 that are not statistically significant should be reported as “less than” values. At least three independent analyses should be carried out and results should be reported for each analysis separately with their mean value and their combine uncertainties on the tables provided.

The reference date for reporting activities was 1 August 2006.

The samples were distributed to 40 laboratories in November 2010. The deadline for reporting data was set for 30 June 2011. A reminder was sent to participants, who did not submit the results in time. A total of 28 laboratories sent their reports. The list of reported radionuclides is given in Table 2, Appendix I.

Laboratories were informed that, after the completion of the exercise, an IAEA report describing the results of the inter-laboratory comparison would be issued, including their identities, but that the results would not be associated to each laboratory identity.

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 3 to 18, Appendix I. Laboratory means were calculated when necessary from individual results and are given either as arithmetic means with corresponding uncertainties when more than two results were reported, or as weighted means with weighted uncertainties in the case of only two results reported. All values have been rounded off to the most significant figure.

6.2. Statistical evaluation

Calculations are based on the assumption of non-parametric distribution of data to which distribution-free statistics are applicable. The "less than" values are segregated from the results and the remaining values are checked for the presence of outliers using a box and whisker plot test and Tukey's outlier method. Outliers are identified in the tables with an asterisk. Median values are calculated from all results passing the outlier test. These values are considered to be the most reliable estimates of the true values. Confidence intervals were taken from a non-parametric sample population. They represent a two-sided interval representing 95% confidence limits. The estimated expanded uncertainty for mean value was calculated according to the *Evaluation of measurement data – Guide to the expression of uncertainty in measurement*, (GUM with minor corrections) JCGM 100:2008 (2008) [4]. The weighted mean values and their uncertainties were calculated taking into account the uncertainties of measurements reported by individual laboratories.

Massic activities for 27 radionuclides were reported and results are shown in Table 2, Appendix I with the number of reporting laboratories for each radionuclide. The number of reported "less than" values are shown in parentheses. The results for the most frequently measured radionuclides can be found in Tables 3 to 17, Appendix I and Figures 4 to 19, Appendix II, while the less frequently measured radionuclides are presented in Table 18, Appendix I. The certified values and information values obtained after statistical treatment are presented in Table 19, Appendix I.

Following the recommendations for assessment of laboratory performance of the International Union of Pure and Applied Chemistry (IUPAC) [5] and the International Organization for Standardization (ISO) [6], the *z*-score methodology was used for the evaluation of the inter-laboratory results. The performance of a laboratory was considered to be acceptable if the difference between the robust mean of the laboratory and the assigned value is less than or equal to two. The warnings are for the values with *z*-score lying between 2 and 3. The analysis is regarded as being out of control when $|z|>3$.

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:

Alpha spectrometry

Code	*Method*
A	Treatment, evaporation/precipitation, ion exchange and electro deposition followed by alpha spectrometry

Beta counting

Code	*Method*
B	Precipitation (oxalate, hydroxide), scavenging, beta counting of Y-oxalate followed by beta counting (low level proportional gas counter)
B1	LSC (Liquid Scintillation Counting)

Gamma spectrometry

Code	*Method*
G	High resolution gamma-ray-spectrometry using HP-Ge detectors

Mass spectrometry

Code	*Method*
ICPMS	Treatment, ion exchange, electro deposition, leaching, ICP-MS (Inductively Coupled Plasma Mass Spectrometry)
AMS	Leaching, treatment, AMS (Accelerator Mass Spectrometry)

6.3.3. Number of results

The number of determinations corresponds to the number of individual results from which the laboratory mean was calculated. When no mention was made in a participant's report as to the number of measurements made, it was assumed to be one.

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 unit Bq kg^{-1}.

6.4. Explanation of figures

The figures (Figs. 4 to 19, Appendix II) present the data with the corresponding standard deviation or weighted uncertainty in order of ascending massic activity. Also shown are:

(i) The distribution medians (full lines) and corresponding confidence intervals (dashed horizontal lines)

(ii) The limits for accepted laboratory mean (vertical lines).

(iii) The warning points in yellow (and not included in the assigned values).

The performance of laboratories in terms of accuracy was expressed by *z*-scores, which were calculated for each radionuclide. Figures 20 to 35 in Appendix III present the *z*-scores for accepted values only. The distributions of *z*-scores are symmetric which indicates that the overall performance of the laboratories was satisfactory.

6.5. Criteria for assigning certified values

Median values and confidence intervals (95% significance level) were calculated as estimations of true massic activities. The median values of the data within the confidence interval were considered as the certified values when:

1. At least five laboratory means were available, calculated from at least three different laboratories.
2. The relative uncertainty of the median did not exceed ± 5% for activities higher than 100 Bq kg^{-1}, ± 10% for activities between 1 and 100 Bq kg^{-1} and ± 20% for activities lower than 1 Bq kg^{-1}.

An activity value was classified as an information value when it satisfies condition 1, but not condition 2.

Evidence on metrological traceability to the SI Units was provided by all laboratories in their individual reports.

7. RESULTS AND DISCUSSION

7.1. Anthropogenic radionuclides

Results of the determination of ^{90}Sr, ^{99}Tc, ^{137}Cs, $^{239+240}$Pu, ^{239}Pu and ^{240}Pu reported by participants are presented in Tables 4–6 and 16–17, Appendix I, and Figures 5–7, 17–19, Appendix II.

7.1.1. ^{90}Sr

Thirteen laboratories reported data for ^{90}Sr (Table 4, Appendix I and Fig. 5, Appendix II). One did not pass the outlier test. The data showed good homogeneity. The *z*-score (Fig. 21, Appendix III) is below 1.92, indicating a good performance. The median, given as the information value, is 5.1 Bq kg^{-1} (95% confidence interval is 4.5–5.3 Bq kg^{-1}).

7.1.2. ^{99}Tc

Data were reported from six laboratories (Table 5, Appendix I and Fig. 6, Appendix II). All the data sets, except one, passed the outlier test and showed good homogeneity. The *z*-score values are below 2 (Fig. 22, Appendix III). The median, given as the information value, is 14.8 Bq kg^{-1} (95% confidence interval is 13.8–20.2 Bq kg^{-1}).

7.1.3. ^{137}Cs

Data were reported from twenty five laboratories (Table 6, Appendix I and Fig. 7, Appendix II); two of them were not accepted. The laboratories mainly used direct gamma spectrometry for the ^{137}Cs determination. The data is homogenous within two standard deviations of the distribution mean. The *z*-score values are below 2, indicating a good performance (Fig. 23, Appendix III). The median, given as the certified value, is 18.8 Bq kg^{-1} (95% confidence interval is 18.2–19.2 Bq kg^{-1}).

7.1.4. Plutonium isotopes

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

7.1.4.1. ^{238}Pu

Nine data sets were reported (Table 16, Appendix I), but five of them could only be reported as LLD (Lower Limit of Detection), with results ranging from 0.019 to 0.056 Bq kg^{-1}.

7.1.4.2. $^{239+240}$Pu

Twenty data sets were reported (Table 16, Appendix I and Fig. 17, Appendix II), five failed the outlier test, two of them being warned. The data are homogeneous within two standard deviations of the distribution mean. The *z*-score values are below 1.6, revealing a good performance (Fig. 33, Appendix III). The median, given as the certified value, is 0.024 Bq kg $^{-1}$ (95% confidence interval is 0.022–0.026 Bq kg^{-1}).

7.1.4.3. ^{239}Pu and ^{240}Pu

Five laboratories could determine separately ^{239}Pu and ^{240}Pu activity concentrations using mass spectrometry (ICP-MS and AMS). The results are presented in Table 17, Appendix I and Figs. 18 and 19, Appendix II. The data are homogeneous within two standard deviations of the distribution mean. The *z*-score values are below 1.78 and 1.88, respectively (Figs. 34 and 35, Appendix III). The median, given as the information value, is 0.014 Bq kg^{-1} (95% confidence interval is 0.012–0.015 Bq kg^{-1}) for ^{239}Pu and 0.010 Bq kg^{-1} (95% confidence interval is 0.009–0.014 Bq kg^{-1}) for ^{240}Pu, respectively. It is worth noticing that the sum of the ^{239}Pu and ^{240}Pu activity concentrations is in agreement with the $^{239+240}$Pu value determined by alpha spectrometry technique (see above).

7.2. Natural radionuclides

7.2.1 ^{40}K

Data were reported from twenty-five laboratories (Table 3, Appendix I and Fig. 4, Appendix II). All results passed the outlier test, except one laboratory. The data showed good homogeneity. Results are between two standard deviations from the distribution mean.
The *z*-score values are below 1.95, showing a good performance by the laboratories (Fig. 20, Appendix III). The median, given as the certified value, is 660 Bq kg^{-1} (95% confidence interval is 626–671 Bq kg^{-1}).

7.2.2 ^{210}Pb (^{210}Po)

Data were reported from sixteen laboratories (Table 7, Appendix I and Fig. 8, Appendix II). Two did not pass the outlier test, one was warning. ^{210}Pb and ^{210}Po were considered as in equilibrium at the inter-laboratory comparison period (2011), when ten half-lives of ^{210}Po have passed, compared to the sampling time (2006) and the ^{210}Pb values were decay corrected back to the reference date at 1 August 2006. Half of the participants used alpha spectrometry with prior radiochemical purification of ^{210}Po, using electro deposition on a silver disk. The rest used direct gamma spectrometry to measure ^{210}Pb at 46.5 keV. The data showed good homogeneity. Results are between two standard deviations from the distribution mean.
The *z*-score values are below 1.69, showing a good performance by the laboratories (Fig. 24, Appendix III). The median, given as the information value, is 10.9 Bq kg^{-1} (95% confidence interval is 10.2–12.0 Bq kg^{-1}).

7.2.3 Radium isotopes

7.2.3.1 ^{226}Ra

Data were reported from sixteen laboratories (Table 8, Appendix I and Fig. 9, Appendix II). All results except two passed the outlier test, one was warning. While most of the 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; only one laboratory used LSC (Liquid Scintillation Counting) technique. The data showed good homogeneity. Results are between two standard deviations from the distribution mean. The *z*-score values are below1.82, showing a good performance by the laboratories (Fig. 25, Appendix III). The median, given as the information value, is 17.0 Bq kg^{-1} (95% confidence interval is 14.2–18.9 Bq kg^{-1}).

7.2.3.2 ^{228}Ra

Thirteen laboratories reported data for ^{228}Ra (Table 9, Appendix I and Fig. 10, Appendix II). All results passed the outlier test, except one laboratory. 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). The data showed good homogeneity. Results are between two standard deviations from the distribution mean. The *z*-score values are below 1.94, showing a good performance by the laboratories (Fig. 26, Appendix III). The median, given as the certified value, is 15.4 Bq kg^{-1} (95% confidence interval is 15.0–16.7 Bq kg^{-1}).

7.2.4 Thorium isotopes

7.2.4.1 ^{228}Th

Out of thirteen data sets reported (Table 10, Appendix I and Fig. 11, Appendix II), two failed the test for outliers. Half of the participants used a conventional method based on sample treatment, ion-exchange separation followed by electro deposition and alpha spectrometry. The rest 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). The data is homogeneous within two standard deviations of the distribution mean. The *z*-score values are below 1.93 (Fig. 27, Appendix III). The median, given as the information value, is 15.0 Bq kg^{-1} (95% confidence interval is 12.6–15.5 Bq kg^{-1}).

7.2.4.2 ^{230}Th

Out of eleven data sets reported (Table 11, Appendix I and Fig. 12, Appendix II), one did not pass the test for outliers, two of them were warning. All participants used a conventional method based on sample treatment, ion-exchange separation followed by electro deposition and alpha spectrometry. The data is homogeneous within two standard deviations of the distribution mean. The *z*-score values are below 1.82 (Fig. 28, Appendix III).
The median, given as the information value, is 0.36 Bq kg^{-1} (95% confidence interval is 0.22–0.38 Bq kg^{-1}).

7.2.4.3 ^{232}Th

Fourteen data sets were reported (Table 12, Appendix 4 and Fig.13, Appendix II). Four data sets were analysed by gamma spectrometry, and ten other used a conventional method based on sample treatment, ion-exchange separation followed by electro-deposition and alpha spectrometry. The former reported the higher level of activity, ranging from 13.4 to 25.4 Bq kg^{-1}. The latter reported the range of activity from 0.26 to 7.3 Bq kg^{-1}. Taking into account the large difference of activity concentrations between the two methods, data evaluation was done separately for alpha and gamma spectrometry. The data obtained from gamma spectrometry were not evaluated due to the limited number of data (4, see 6.5). There is a possible disequilibrium between ^{232}Th and its daughters ^{208}Tl, ^{212}Pb/^{212}Bi, ^{228}Th, ^{228}Ac and ^{228}Ra; the participant should then report their results for these daughters but not for ^{232}Th.

Out of ten data sets that used alpha spectrometry technique, one did not pass the outlier test. The accepted data is homogeneous within two standard deviations of the distribution mean. The *z*-score values are below 2.0 (Fig. 29, Appendix III). The median, given as the information value, is 0.38 Bq kg^{-1} (95% confidence interval is 0.30–0.46 Bq kg^{-1}).

7.2.5 Uranium isotopes

7.2.5.1 ^{234}U

Out of fifteen data sets reported (Table 13, Appendix I and Fig. 14, Appendix II), three were warning and one rejected by the test for outliers. Most participants used a conventional method based on sample treatment, ion-exchange separation followed by electro deposition and alpha spectrometry. Two laboratories could determine their activities using ICP-MS method, with prior radiochemical separation of the uranium isotopes. The data are homogeneous within two standard deviations of the distribution mean. The *z*-score values are below 1.6, showing a good performance by the laboratories (Fig. 30, Appendix III). The median, given as the certified value, is 10.5 Bq kg^{-1} (95% confidence interval is 10.0–11.0 Bq kg^{-1}).

7.2.5.2 ^{235}U

Out of fourteen data sets reported (Table 14, Appendix I and Fig. 15, Appendix II), only one data set was rejected by outliers test. Most participants used a conventional method based on sample treatment, ion-exchange separation followed by electro deposition and alpha spectrometry. Two laboratories could determine their activities using ICP-MS method, with prior radiochemical separation of the uranium isotopes. The data are homogeneous within two standard deviations of the distribution mean. The *z*-score values are below 1.8 (Fig. 31, Appendix III). The median, given as the information value, is 0.44 Bq kg^{-1} (95% confidence interval is 0.27–0.52 Bq kg^{-1}).

7.2.5.3 ^{238}U

Out of nineteen data sets reported (Table 15, Appendix I and Fig. 16, Appendix II), one data set was rejected by outliers test and four were warned. Most participants used a conventional method based on sample treatment, ion-exchange separation followed by electro deposition and alpha spectrometry. Two laboratories could determine their activities using ICP-MS method, with prior radiochemical separation of uranium isotopes. Four laboratories used a direct gamma spectrometry technique. 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 above for ^{226}Ra). The data is homogeneous within two standard deviations of the distribution mean. The *z*-score values are below 1.98, (Fig. 32, Appendix III). The median, given as the information value, is 9.34 Bq kg^{-1} (95% confidence interval is 8.98–10.96 Bq kg^{-1}).

7.3. Less frequently reported radionuclides

The results for the less frequently reported radionuclides are listed in Table 18, Appendix I.

7.3.1. ^{129}I

Two results were reported, using AMS technique, ranging from 0.1 to 0.13 Bq kg^{-1}.

7.3.2. ^{208}Tl

Four laboratories reported results for ^{208}Tl. The data were rather inconsistent and ranged from 4.5 to 15.3 Bq kg^{-1}. As mentioned above (see for ^{232}Th), there is probably a disequilibrium between ^{232}Th and its daughters ^{208}Tl, ^{212}Pb/^{212}Bi, ^{228}Th, ^{228}Ac, and ^{228}Ra and the precision of ^{208}Tl branching ratio used for ^{208}Tl activity concentration at 583 keV peak is probably interfered due to the presence of both ^{228}Th (and descendants, 30.6%) and ^{208}Tl (85.1%).

7.3.3. ^{212}Bi, ^{212}Pb, ^{224}Ra and ^{228}Ac

Two laboratories reported ^{212}Bi results ranging from 19.2 to 20.1 Bq kg^{-1}. Four laboratories reported ^{212}Pb results ranging from 15.0 to 17.8 Bq kg^{-1}. Two laboratories reported ^{224}Ra results ranging from 11.8 to 18.5 Bq kg^{-1}. Three laboratories reported ^{228}Ac results ranging from 12.5 to 15.4 Bq kg^{-1}. These data were determined by using gamma spectrometry and are about the same levels as ^{228}Th and ^{228}Ra activity concentrations (see above for ^{228}Th and ^{228}Ra, respectively) showing that ^{228}Ra and its daughters ^{228}Ac, ^{228}Th, ^{224}Ra, ^{212}Bi and ^{212}Pb are in equilibrium (but not with their original precursor ^{232}Th, see above).

7.3.4. ^{214}Bi, ^{214}Pb and ^{234}Th

Four laboratories reported ^{214}Bi and ^{214}Pb results ranging from 13.6 to 20.6 Bq kg^{-1} and from 13.4 to 21.7 Bq kg^{-1}, respectively. Only one laboratory reported a ^{234}Th value (16.9 ± 0.7 Bq kg^{-1}). These data were determined by using gamma spectrometry and are in the same range with ^{226}Ra activity concentrations (see above for 7.2.3.1 ^{226}Ra) showing that the ^{226}Ra and its progeny ^{214}Bi and ^{214}Pb are in equilibrium (but not with its original precursor ^{238}U, see above).

7.3.5. ^{236}U

Two results were reported, using ICP-MS technique, ranging from 3.4 to 59 mBq kg^{-1}.

7.3.6. ^{241}Am

Five laboratories determined ^{241}Am by alpha spectrometry with prior radiochemical purification from rare earth elements, one reported a “less than” value. The four other values were not consistent, ranging from 0.0073 to 3 Bq kg^{-1}.

8. CONCLUSIONS

In this inter-laboratory comparison, 29 laboratories including IAEA-RML reported concentrations of natural and anthropogenic radionuclides in a seaweed sample from the Baltic Sea (IAEA-446). The median concentrations 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.

A summary of the certified and information values with confidence intervals for the most frequently reported anthropogenic and natural radionuclides may be found in the summary table below as well as in Table 19, Appendix I.

SUMMARY TABLE: CERTIFIED AND INFORMATION VALUES FOR THE IAEA-446 REFERENCE MATERIAL.

(Reference date: 1 August 2006, unit: Bq kg^{-1})

Radionuclide	Median	Confidence interval (α = 0.05)	Mean	Expanded uncertainty (k = 2)$^{\&}$	Number of results*
	<u>Certified value</u>				
^{40}K	660	626 – 671	658	20	24
^{137}Cs	18.8	18.2 – 19.2	18.8	0.5	23
^{228}Ra	15.4	15.0 – 16.7	15.6	0.5	12
^{234}U	10.5	10.0 – 11.0	10.5	0.3	11
$^{239+240}Pu$	0.024	0.022 – 0.026	0.024	0.001	13
	<u>Information value</u>				
^{90}Sr	5.1	4.5 – 5.3	5.0	0.2	12
^{99}Tc	14.8	13.8 – 20.2	16.0	2.4	5
$^{210}Pb(^{210}Po)^{\$}$	10.9	10.2 – 12.0	11.1	0.5	13
^{226}Ra	17.0	14.2 – 18.9	16.8	1.6	13
^{228}Th	15.0	12.6 – 15.5	14.6	1.1	11
^{230}Th	0.36	0.22 – 0.38	0.37	0.07	8
^{232}Th	0.38	0.30 – 0.46	0.39	0.06	9
^{235}U	0.44	0.27 – 0.52	0.41	0.07	13
^{238}U	9.34	8.98 – 10.96	9.79	0.48	14
^{239}Pu	0.014	0.012 – 0.015	0.0135	0.001	5
^{240}Pu	0.010	0.009 – 0.014	0.011	0.002	5

& Expanded uncertainty for the mean value was calculated according to the "Evaluation of measurement data – Guide to the expression of uncertainty in measurement", JGCM 100:2008 (GUM with minor corrections), (2008) [4]

* Number of accepted laboratory means which were used to calculate the certification and information values and the confidence intervals

$ ^{210}Pb and ^{210}Po were considered as in equilibrium, and the ^{210}Pb is corrected for reference date at 1 August 2006

APPENDIX I
Data report – Tables

TABLE 1. HOMOGENEITY TESTS AS NORMALIZED ACTIVITY FOR RADIONUCLIDES IN IAEA-446(*).

Sample	^{137}Cs	^{40}K	^{210}Po	^{214}Bi	$^{239+240}Pu$
1	0.93	0.93	0.82	0.95	0.84
2	0.94	0.94	0.88	0.95	0.90
3	0.95	0.94	0.93	0.95	0.91
4	0.96	0.96	0.99	0.96	0.93
5	0.99	0.97	1.00	0.97	0.94
6	0.99	0.98	1.00	0.97	0.94
7	1.00	0.98	1.02	0.97	0.97
8	1.00	0.98	1.02	0.99	1.01
9	1.00	0.99	1.02	0.99	1.02
10	1.01	0.99	1.03	1.00	1.02
11	1.01	1.00	1.05	1.01	1.04
12	1.01	1.01	1.06	1.01	1.06
13	1.01	1.01	1.07	1.01	1.06
14	1.01	1.01	1.10	1.02	1.07
15	1.01	1.02		1.03	1.11
16	1.02	1.04		1.03	1.17
17	1.02	1.05		1.04	
18	1.04	1.06		1.05	
19	1.04	1.06		1.06	
20	1.06	1.09		1.07	
Minimum	0.93	0.93	0.82	0.95	0.84
Maximum	1.06	1.09	1.10	1.07	1.17
Mean	1.00	1.00	1.00	1.00	1.00
Median	1.01	0.98	1.02	0.98	1.01
Std. Dev.	0.02	0.03	0.07	0.03	0.09
Coef. Var. (%)	2	3	7	3	9

(*) Normalized activity = x/X (individual/mean values): initially expressed in this manner to assure confidentiality of results

TABLE 2. RADIONUCLIDES REPORTED FOR IAEA-446.

Radionuclide	Number of all results	Radionuclide	Number of all results
^{40}K	107	^{228}Ac	5
^{90}Sr	38(1)	^{228}Th	43
^{99}Tc	16	^{230}Th	34
^{129}I	18	^{232}Th	40
^{137}Cs	107	^{234}U	44
^{208}Tl	12	^{235}U	40
$^{210}Pb(^{210}Po)$	62	^{236}U	15
^{212}Pb	9	^{238}U	58
^{212}Bi	7	^{238}Pu	44(6)
^{214}Pb	29	^{239}Pu	17
^{214}Bi	29	^{240}Pu	17
^{224}Ra	7	$^{239+240}Pu$	79(1)
^{226}Ra	54	^{241}Am	22
^{228}Ra	48		

Note: "Less than" values are shown in parentheses

TABLE 3. RESULTS FOR ^{40}K IN IAEA-446.

(Reference date: 1 August 2006, unit: Bq kg^{-1})

Lab code	Method code	No. of results	Mass (g)	$^{40}K^{\#}$
1	G	4	85	676 ± 91
3	G	4	no information	802 ± 25*
4	G	1	56.5	665 ± 80
5	G	7	69.81	665 ± 40
6	G	3	38	568 ± 34
7	G	3	23.92	727 ± 29
7b	G	4	28.46	680 ± 26
8	G	1	100	630 ± 22
9	G	3	96.5	634 ± 13
10	G	3	46.8; 85.8	710 ± 17
11	G	2	101.6	609 ± 67
12	G	3	83.6	664 ± 40
13	G	1	43.62	610 ± 20
14	G	3	27	647 ± 30
15	G	2	14; 100	750 ± 60
16	G	3	45-50	670 ± 120
17	G	3	72	626 ± 13
18	G	2	30.8	620 ± 42
19	G	2	95.1	728 ± 19
21	G	1	88.46	618 ± 48
22	G	1	73	736 ± 40
23	G	1	62.56	592 ± 11
24	G	9	97	656 ± 17
27	G	40	8.77-19.54; 60	651 ± 6
28	G	1	100	671 ± 40

Number of reported lab. means	25
Number of accepted lab. means	24
Median	660
Mean	658
Weighted mean (uncertainty)	657 (16)
Confidence interval ($\alpha = 0.05$)	626 – 671
Expanded uncertainty ($k = 2$)	20

For Tables 3-17:

Uncertainties at 2σ

* Result rejected by the test for outliers

TABLE 4. RESULTS FOR ^{90}Sr IN IAEA-446.

(Reference date: 1August 2006, unit: Bq kg^{-1})

Lab code	Method code	No. of results	Mass (g)	^{90}Sr		
1	B	2	0.5	4.5	±	2.1
6	B	1	19	4.6	±	1.0
7	B	6	9.568	5.1	±	0.3
9	B	2	18.1; 20.0	5.3	±	0.6
10	B	3	9.4; 18.7	5.26	±	0.31
11	B	4	20	5.12	±	0.28
14	B	3	40.27-40.65	5.24	±	0.42
15	B	2	47	5.4	±	0.8
16	B	3	9.33	4.48	±	1.08
19	B	3	16-20	5.24	±	0.17
21	B	1	4.59	4.4	±	1.6
24	B	3	20	4.19	±	0.26!
27	B	5	10	5.1	±	0.3

Number of reported lab. means	13
Number of accepted lab. means	12
Median	5.1
Mean	5.0
Weighted mean (uncertainty)	5.0 (0.1)
Confidence interval ($\alpha = 0.05$)	4.5 – 5.3
Expanded uncertainty ($k = 2$)	0.2

For Tables 4-17:
! Results are warning, with z-scores between 2 and 3

TABLE 5. RESULTS FOR ^{99}Tc IN IAEA-446.

(Reference date: 1August 2006, unit: Bq kg^{-1})

Lab code	Method code	No. of results	Mass (g)	^{99}Tc
1	B1	2	0.5	69 ± 24*
3	B1	3	no information	20.2 ± 2.1
13	B1	1	10.38	13.8 ± 1.3
14	B1	3	9.67-9.69	14 ± 1
15	B1	6	5; 10	14.8 ± 0.8
18	B1	1	10	17 ± 2

Number of reported lab. means	6
Number of accepted lab. means	5
Median	14.8
Mean	16.0
Weighted mean (uncertainty)	14.9 (0.8)
Confidence interval ($\alpha = 0.05$)	13.8 – 20.2
Expanded uncertainty ($k = 2$)	2.4

TABLE 6. RESULTS FOR ^{137}Cs IN IAEA-446.

(Reference date: 1 August 2006, unit: Bq kg^{-1})

Lab code	Method code	No. of results	Mass (g)	^{137}Cs
1	G	4	85	21.5 ± 1.0*
3	G	4	no information	22.1 ± 0.6*
4	G	1	56.5	19.5 ± 1.4
5	G	7	69.81	19.2 ± 1.5
6	G	3	38	16.4 ± 1.2
7	G	3	23.92	21.3 ± 1.1
7b	G	4	28.46	18.8 ± 0.8
8	G	1	100	16.9 ± 0.9
9	G	3	96.5	18.1 ± 0.3
10	G	3	46.8; 85.8	20.1 ± 0.7
11	G	2	101.6	18.6 ± 1.9
12	G	3	83.6	19.3 ± 1.3
13	G	1	43.62	19.0 ± 0.9
14	G	3	27	18.0 ± 1.0
15	G	2	14; 100	19.5 ± 1.6
16	G	3	45-50	17.1 ± 3.1
17	G	3	72	18.2 ± 1.2
18	G	2	30.8	18.6 ± 1.2
19	G	2	95.1	18.4 ± 0.4
21	G	1	88.46	17.5 ± 1.5
22	G	1	73	21.0 ± 1.3
23	G	1	62.56	19.5 ± 0.6
24	G	9	97	19.0 ± 0.5
27	G	40	8.77-19.54; 60	18.5 ± 0.2
28	G	1	100	18.9 ± 0.6

Number of reported lab. means	25
Number of accepted lab. means	23
Median	18.8
Mean	18.8
Weighted mean (uncertainty)	18.8 (0.1)
Confidence interval (α = 0.05)	18.2 – 19.2
Expanded uncertainty (k = 2)	0.5

TABLE 7. RESULTS FOR $^{210}Pb(^{210}Po)$ IN IAEA-446.

(Reference date: 1 August 2006, unit: Bq kg^{-1})

Lab code	Method code	No. of results	Mass (g)	$^{210}Pb(^{210}Po)^{\$}$
1	G	4	85	12.4 ± 1.8
2	A	3	0.3	12.0 ± 0.6
3	A	3	no information	15.5 ± 0.7*
5	A	9	1.81-1.86	10.0 ± 1.3
8	A	1	1.039	10.6 ± 2.5
9	G	3	96.5	13.8 ± 1.4!
11	G	1	101.6	10.9 ± 3.1
12	A	3	5.1	10.4 ± 0.7
14	A	3	4.96-5.06	10.0 ± 0.8
15	G	1	14	9.8 ± 1.0
16	G	2	17.5; 50	12.0 ± 4.6
17	G	3	72	10.2 ± 1.4
22	A	1	2	16.6 ± 1.5*
23	A	2	1.996; 2.662	11.0 ± 0.4
24	G	9	97	12.2 ± 1.1
27	A	14	0.35-0.53	11.7 ± 0.2

Number of reported lab. means	16
Number of accepted lab. means	13
Median	10.9
Mean	11.1
Weighted mean	11.3 (0.2)
Confidence interval (α = 0.05)	10.2 – 12.0
Expanded uncertainty (k = 2)	0.5

$ ^{210}Pb and ^{210}Po were considered to be in equilibrium, and the ^{210}Pb values are corrected for reference date at 1 August 2006

TABLE 8. RESULTS FOR ^{226}Ra IN IAEA-446.

(Reference date: 1 August 2006, unit: Bq kg^{-1})

Lab code	Method code	No. of results	Mass (g)	^{226}Ra
1	G	4	85	30.0 ± 4.3*
4	G	1	56.5	17.0 ± 3.4
8	G	1	100	12.2 ± 2.5
9	G	3	96.5	21.2 ± 0.5
10	B1	3	4.68	16.2 ± 0.7
11	G	1	101.6	25.6 ± 7.7!
13	G	1	43.62	20.1 ± 1.6
15	G	2	14; 100	17.0 ± 2.0
16	G	1	50	16.1 ± 3.0
17	G	3	72	18.8 ± 1.5
18	G	1	30.8	14.2 ± 1.4
19	G	2	95.1	13.0 ± 0.5
22	G	1	73	16.0 ± 1.5
23	G	1	62.56	32.5 ± 1.8*
24	G	9	97	17.6 ± 1.4
27	G	20	8.77-19.54; 60	18.9 ± 0.2

Number of reported lab. means	16
Number of accepted lab. means	13
Median	17.0
Mean	16.8
Weighted mean (uncertainty)	18.2 (0.6)
Confidence interval (α = 0.05)	14.2 – 18.9
Expanded uncertainty (k = 2)	1.6

TABLE 9. RESULTS FOR ^{228}Ra IN IAEA-446.

(Reference date: 1 August 2006, unit: Bq kg^{-1})

Lab code	Method code	No. of results	Mass (g)	^{228}Ra
1	G	4	85	15.4 ± 1.2
4	G	1	56.5	15.0 ± 3.0
9	G	3	96.5	16.4 ± 0.5
13	G	1	43.62	14.6 ± 1.5
15	G	2	14; 100	17.0 ± 3.0
16	G	3	17.5-50	24.3 ± 5.0*
17	G	3	72	15.4 ± 0.6
18	G	2	30.8	16.5 ± 1.3
19	G	2	95.1	14.6 ± 0.7
22	G	1	73	15.0 ± 2.4
23	G	1	62.56	16.7 ± 2.0
24	G	9	97	15.3 ± 0.5
27	G	20	8.77-19.54; 60	15.7 ± 0.2

Number of reported lab. means	13
Number of accepted lab. means	12
Median	15.4
Mean	15.6
Weighted mean (uncertainty)	15.7 (0.2)
Confidence interval (α = 0.05)	15.0 – 16.7
Expanded uncertainty (k = 2)	0.5

TABLE 10. RESULTS FOR ^{228}Th IN IAEA-446.

(Reference date: 1 August 2006, unit: Bq kg^{-1})

Lab code	Method code	No. of results	Mass (g)	^{228}Th
3	A	3	no information	17.5 ± 0.3
8	A	2	0.98	12.2 ± 2.5
9	A	2	4.33; 4.75	12.6 ± 1.3
12	A	3	2.1	14.7 ± 0.7
13	G	1	43.62	98.4 ± 7.6*
14	A	3	29.6-29.8	15.0 ± 1.0
16	G	1	50	21.6 ± 10.5!
17	G	3	72	15.5 ± 0.4
18	G	2	30.8	15.0 ± 1.1
19	A	3	1.9	15.2 ± 0.6
23	G	1	62.56	15.4 ± 0.9
24	G	9	97	16.0 ± 0.5
27	A	10	15; 50	11.6 ± 0.4

Number of reported lab. means	13
Number of accepted lab. means	11
Median	15.0
Mean	14.6
Weighted mean (uncertainty)	15.2 (0.7)
Confidence interval (α = 0.05)	12.6 – 15.5
Expanded uncertainty (k = 2)	1.1

TABLE 11. RESULTS FOR ^{230}Th IN IAEA-446.

(Reference date: 1 August 2006, unit: Bq kg^{-1})

Lab code	Method code	No. of results	Mass (g)	^{230}Th
2	A	3	5	0.54 ± 0.06
3	A	3	no information	0.22 ± 0.01
8	A	2	0.98	22.6 ± 4.2*
9	A	2	4.33; 4.75	1.17 ± 0.12!
12	A	3	2.1	0.33 ± 0.07
14	A	3	29.6-29.8	0.36 ± 0.04
15	A	1	20	0.48 ± 0.10
19	A	3	1.9	0.38 ± 0.04
22	A	1	2	1.17 ± 0.41!
23	A	3	3.3-5.1	0.28 ± 0.01
27	A	10	15; 50	0.35 ± 0.01

Number of reported lab. means	11
Number of accepted lab. means	8
Median	0.36
Mean	0.37
Weighted mean (uncertainty)	0.29 (0.02)
Confidence interval (α = 0.05)	0.22 – 0.38
Expanded uncertainty (k = 2)	0.07

TABLE 12. RESULTS FOR ^{232}Th IN IAEA-446.

(Reference date: 1 August 2006, unit: Bq kg^{-1})

Lab code	Method code	No. of results	Mass (g)	^{232}Th	^{232}Th
2	A	3	5	0.38 ± 0.04	
3	A	3	no information	0.320 ± 0.002	
8	A	2	0.98	7.3 ± 1.5*	
9	G	3	96.5		16.5 ± 0.5
9b	A	2	4.33; 4.75	0.57 ± 0.06	
11	G	2	101.6		25.4 ± 2.3
12	A	3	2.1	0.41 ± 0.10	
13	G	1	43.62		14.6 ± 1.5
14	A	3	29.6-29.8	0.46 ± 0.05	
15	A	1	20	0.38 ± 0.04	
19	A	3	1.9	0.42 ± 0.04	
23	A	3	3.3-5.1	0.26 ± 0.01	
27	A	10	15; 50	0.295 ± 0.012	
28	G	1	100		13.4 ± 0.5

Number of reported lab. means	10
Number of accepted lab. means	9
Median	0.38
Mean	0.39
Weighted mean (uncertainty)	0.32 (0.004)
Confidence interval ($\alpha = 0.05$)	0.29 – 0.46
Expanded uncertainty ($k = 2$)	0.06

TABLE 13. RESULTS FOR ^{234}U IN IAEA-446.

(Reference date: 1 August 2006, unit: Bq kg^{-1})

Lab code	Method code	No. of results	Mass (g)	^{234}U
1	A	4	0.5	11.0 ± 1.8
2	A	3	5	11.3 ± 0.3
3	A	3	no information	10.1 ± 0.4
8	A	2	0.25	37.5 ± 1.6*
9	A	2	4.62; 5.02	10.5 ± 1.1
12	A	3	2.1	10.5 ± 0.6
14	A	3	29.6-29.8	10.0 ± 0.6
15	A	1	20	11.0 ± 2.0
18	A	1	no information	16.3 ± 2.0!
19	A	3	1.9	10.0 ± 0.6
21	A	1	9.19	11.0 ± 0.3
22	A	1	2	8.4 ± 0.5!
23	A	2	2.08; 2.09	10.5 ± 0.3
26	ICP-MS	5	0.2	13.3 ± 0.9!
27	ICP-MS	10	0.2	9.85 ± 0.96

Number of reported lab. means	15
Number of accepted lab. means	11
Median	10.5
Mean	10.5
Weighted mean (uncertainty)	10.6 (0.1)
Confidence interval (α = 0.05)	10.0 – 11.0
Expanded uncertainty (k = 2)	0.3

TABLE 14. RESULTS FOR ^{235}U IN IAEA-446.

(Reference date: 1 August 2006, unit: Bq kg^{-1})

Lab code	Method code	No. of results	Mass (g)	^{235}U
2	A	3	5	0.44 ± 0.05
3	A	3	no information	0.27 ± 0.05
8	A	2	0.25	1.68 ± 0.07*
9	A	2	4.62; 5.02	0.49 ± 0.05
12	A	3	2.1	0.45 ± 0.10
14	A	3	29.6-29.8	0.28 ± 0.03
15	A	1	20	0.44 ± 0.1
18	A	1	no information	0.6 ± 0.3
19	A	3	1.9	0.52 ± 0.11
21	A	1	9.19	0.22 ± 0.03
22	A	1	2	0.23 ± 0.13
23	A	2	2.08; 2.09	0.39 ± 0.04
26	ICP-MS	5	0.2	0.535 ± 0.040
27	ICP-MS	10	0.2	0.482 ± 0.047

Number of reported lab. means	14
Number of accepted lab. means	13
Median	0.44
Mean	0.41
Weighted mean (uncertainty)	0.36 (0.03)
Confidence interval ($\alpha = 0.05$)	0.27 – 0.52
Expanded uncertainty ($k = 2$)	0.07

TABLE 15. RESULTS FOR ^{238}U IN IAEA-446.

(Reference date: 1 August 2006, unit: Bq kg^{-1})

Lab code	Method code	No. of results	Mass (g)	^{238}U
1	A	4	0.5	9.0 ± 0.9
2	A	3	5	9.5 ± 0.3
3	A	3	no information	9.0 ± 0.3
4	G	1	56.5	16.5 ± 7.3!
8	A	2	0.25	36.4 ± 1.5*
9	A	2	4.62; 5.02	9.2 ± 1.0
12	A	3	2.1	9.0 ± 0.5
14	A	3	29.6-29.8	8.9 ± 0.6
15	A	1	20	9.7 ± 1.0
17	G	3	72	11.3 ± 2.2
18	A	1	no information	13.2 ± 1.7!
19	A	3	1.9	8.6 ± 0.5
21	A	1	9.19	11.0 ± 0.3
22	A	1	2	6.4 ± 0.4!
23	A	2	2.08; 2.09	9.1 ± 0.2
24	G	9	97	11.0 ± 1.0
26	ICP-MS	5	0.2	11.6 ± 0.83
27	ICP-MS	10	0.2	10.4 ± 1.0
28	G	1	100	15.6 ± 0.6!

Number of reported lab. means	19
Number of accepted lab. means	14
Median	9.34
Mean	9.79
Weighted mean (uncertainty)	9.46 (0.21)
Confidence interval ($\alpha = 0.05$)	8.98 – 10.96
Expanded uncertainty ($k = 2$)	0.48

TABLE 16. RESULTS FOR ^{238}Pu AND $^{239+240}$Pu IN IAEA-446.

(Reference date: 1 August 2006; unit: Bq kg^{-1})

Lab. code	Method code	No. of results	Mass (g)	^{238}Pu$^{\#}$	$^{239+240}$Pu
1	A	4	0.5		0.51 ± 0.19*
3	A	3	no information		0.021 ± 0.003
8	A	2	9.48; 15.22	<0.023; 0.012	0.024 ± 0.01
9	A	2	9.82; 10.1	0.056 ± 0.006	0.0170 ± 0.0025!
10	A	3	9.4; 18.7		0.05 ± 0.01*
11	A	3	20	<0.0065	0.022 ± 0.01
12	A	3	10.1		0.035 ± 0.014!
12b	ICP-MS	3	10		0.0248 ± 0.0015
13	A	2	5.67; 6.5	<0.0142; <0.0149	0.838 ± 0.012*
14	A	3	40.27-40.65		0.0232 ± 0.0021
15	ICP-MS	2	20		0.0207 ± 0.0054
18	A	1	no information		0.022 ± 0.006
21	A	1	9.19	<0.05	0.054 ± 0.013*
22	A	1	4		0.0886 ± 0.0226*
23	A	3	3.3-5.7	<0.01	0.0274 ± 0.0045
24	A	3	14.3	0.0019 ± 0.0009	0.025 ± 0.0038
25	AMS	3	10		0.0241 ± 0.0023
27	A	18	50	0.0023 ± 0.0005	0.0265 ± 0.0037
27b	A	10	5-20	0.009 ± 0.0002	0.0259 ± 0.0023
27c	ICP-MS	9	10-20		0.0261 ± 0.0022

Number of reported lab. means	20
Number of accepted lab. means	13
Median	0.024
Mean	0.024
Weighted mean (uncertainty)	0.0246 (0.0005)
Confidence interval (α = 0.05)	0.022 – 0.026
Expanded uncertainty (k = 2)	0.001

TABLE 17. RESULTS FOR ^{239}Pu AND ^{240}Pu IN IAEA-446.

(Reference date: 1 August 2006; unit: Bq kg^{-1})

Lab. code	Method code	No. of results	Mass (g)	^{239}Pu	^{240}Pu
12	ICP-MS	3	10	0.0146 ± 0.0007	0.0102 ± 0.0013
15	ICP-MS	2	20	0.0119 ± 0.0012	0.0088 ± 0.0021
25	AMS	3	10	0.0147 ± 0.0006	0.0094 ± 0.0008
27d	ICP-MS	9	10-20	0.0144 ± 0.001	0.0115 ± 0.0007
27f	ICP-MS	3	10-20	0.0117 ± 0.001	0.0143 ± 0.0006
Number of reported lab. means				5	5
Number of accepted lab. means				5	5
Median				0.014	0.010
Mean				0.013	0.011
Weighted mean (uncertainty)				0.0140 (0.0006)	0.012 (0.001)
Confidence interval ($\alpha = 0.05$)				0.012 – 0.015	0.009 – 0.014
Expanded uncertainty ($k = 2$)				0.001	0.002

TABLE 18. RESULTS FOR THE LESS FREQUENTLY MEASURED RADIONUCLIDES REPORTED IN IAEA-446.

(Reference date: 1 August 2006, unit: Bq kg^{-1})

Isotope	Lab. code	Method code	No. of results	Mass (g)	Activity (Bq kg^{-1})
^{129}I	20	AMS	12	0.1	0.130 ± 0.001
-	25	AMS	6	0.5	0.100 ± 0.004
^{208}Tl	1	G	4	85	6.9 ± 0.5
-	9	G	3	96.5	15.3 ± 0.6
-	9b	G	3	96.5	5.5 ± 0.2
-	21	G	1	88.46	4.5 ± 0.5
-	28	G	1	100	12.6 ± 0.4
^{212}Bi	1	G	4	85	19.2 ± 3.2
-	9	G	3	96.5	20.1 ± 1.4
^{212}Pb	1	G	4	85	17.1 ± 1.3
-	9	G	3	96.5	17.8 ± 0.3
-	21	G	1	88.46	15.0 ± 1.3
-	28	G	1	100	15.3 ± 1.0
^{214}Bi	1	G	4	85	19.0 ± 1.1
-	9	G	3	96.5	20.6 ± 0.5
-	21	G	1	88.46	14.7 ± 1.3
-	27	G	20	8.77-19.54; 60	18.9 ± 0.2
-	28	G	1	100	13.6 ± 0.5
^{214}Pb	1	G	4	85	19.2 ± 1.2
-	9	G	3	96.5	21.7 ± 0.5
-	21	G	1	88.46	13.4 ± 1.2
-	27	G	20	8.77-19.54; 60	19.0 ± 0.2
-	28	G	1	100	16.2 ± 1.1
^{224}Ra	1	G	4	85	18.5 ± 5.1
-	9	G	3	96.5	11.8 ± 0.9
^{228}Ac	1	G	4	85	15.4 ± 1.2
-	21	G	1	88.46	12.6 ± 1.5
-	28	G	1	100	12.5 ± 1.2
^{234}Th	9	G	3	96.5	16.9 ± 0.7
^{236}U	26	ICP-MS	5	0.2	0.0034 ± 0.0002
-	27	ICP-MS	10	0.2	0.059 ± 0.006
^{241}Am	1	A	4	0.5	3.0 ± 0.6
-	8	A	2	9.48; 15.22	0.020 ± 0.018
-	21	A	1	9.19	<0.12
-	24	A	3	14.3	0.034 ± 0.008
-	27	A	12		0.0073 ± 0.0004

TABLE 19. SUMMARY OF CERTIFIED AND INFORMATION VALUES FOR IAEA-446

(Reference date: 1 August 2006, unit: Bq kg^{-1})

Radionuclide	Median	Confidence interval (α = 0.05)	Mean	Expanded uncertainty (k = 2)[&]	Number of results*
	Certified value				
^{40}K	660	626 – 671	658	20	24
^{137}Cs	18.8	18.2 – 19.2	18.8	0.5	23
^{228}Ra	15.4	15.0 – 16.7	15.6	0.5	12
^{234}U	10.5	10.0 – 11.0	10.5	0.3	11
$^{239+240}Pu$	0.024	0.022 – 0.026	0.024	0.001	13
	Information value				
^{90}Sr	5.1	4.5 – 5.3	5.0	0.2	12
^{99}Tc	14.8	13.8 – 20.2	16.0	2.4	5
$^{210}Pb(^{210}Po)$[$]	10.9	10.2 – 12.0	11.1	0.5	13
^{226}Ra	17.0	14.2 – 18.9	16.8	1.6	13
^{228}Th	15.0	12.6 – 15.5	14.6	1.1	11
^{230}Th	0.36	0.22 – 0.38	0.37	0.07	8
^{232}Th	0.38	0.30 – 0.46	0.39	0.06	9
^{235}U	0.44	0.27 – 0.52	0.41	0.07	13
^{238}U	9.34	8.98 – 10.96	9.79	0.48	14
^{239}Pu	0.014	0.012 – 0.015	0.0135	0.0010	5
^{240}Pu	0.010	0.009 – 0.014	0.011	0.002	5

[&] Expanded uncertainty for mean value was calculated according to the “Evaluation of measurement data – Guide to the expression of uncertainty in measurement”, JGCM 100:2008 (GUM with minor corrections), (2008) [4]

* Number of accepted laboratory means which were used to calculate the certification and information values and the confidence intervals

[$] ^{210}Pb and ^{210}Po were considered to be in equilibrium and the ^{210}Pb is corrected to a reference date of 1 August 2006

APPENDIX II
Data evaluation – Graphs

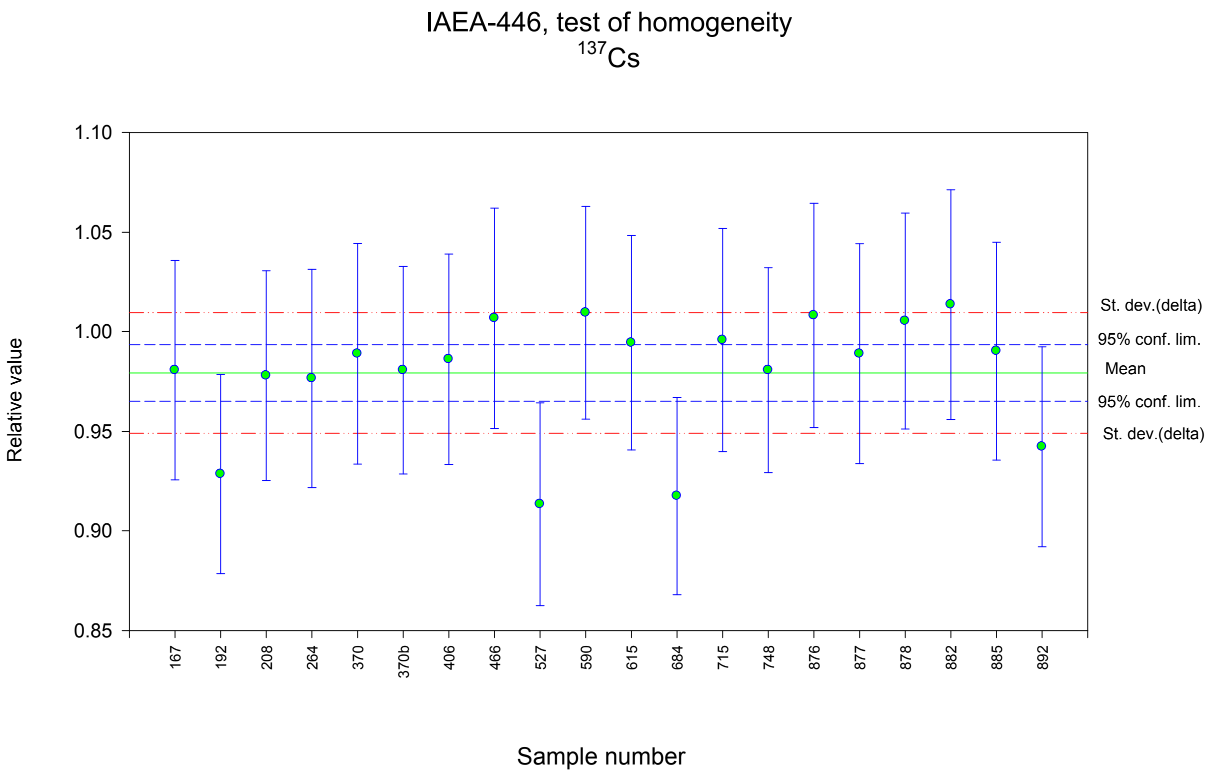

FIG.1. Homogeneity test for ^{137}Cs in IAEA-446.

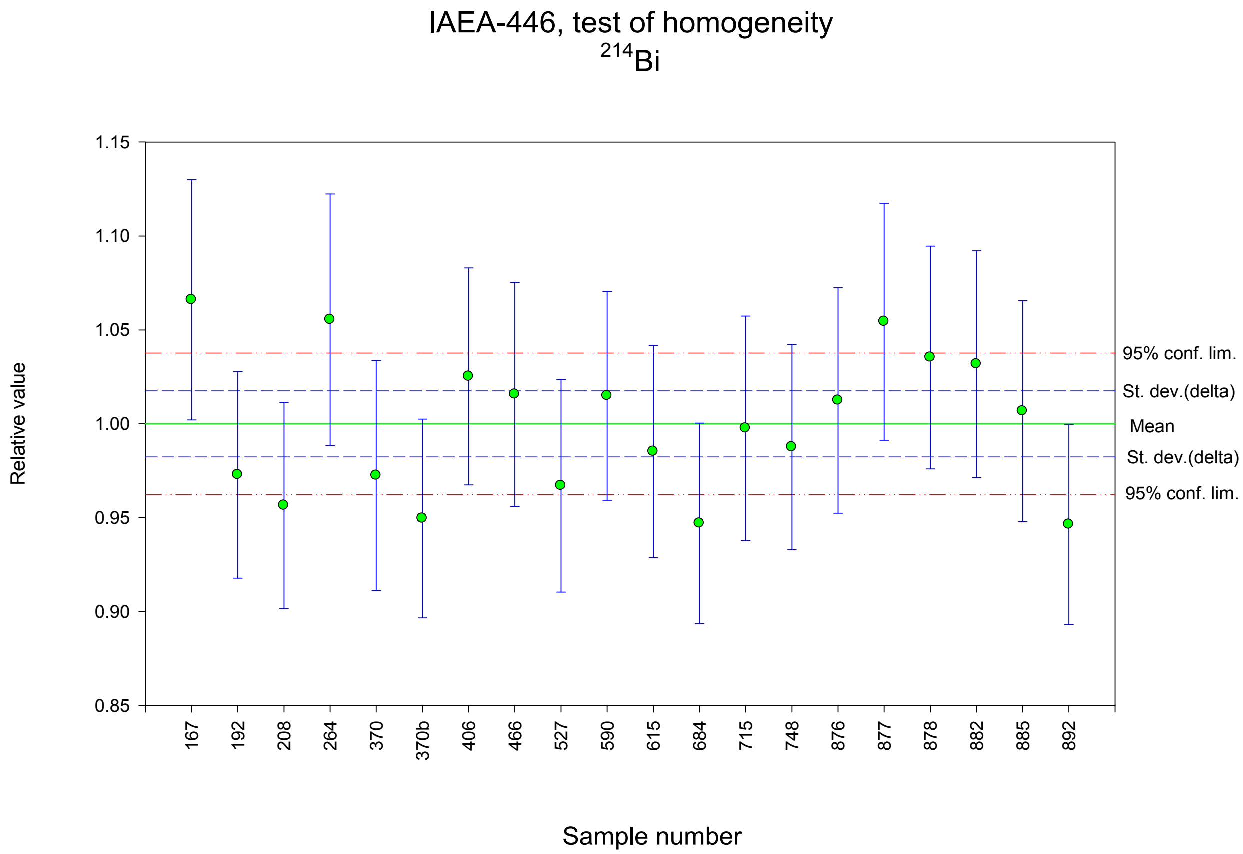

FIG.2. Homogeneity test for ^{214}Bi in IAEA-446.

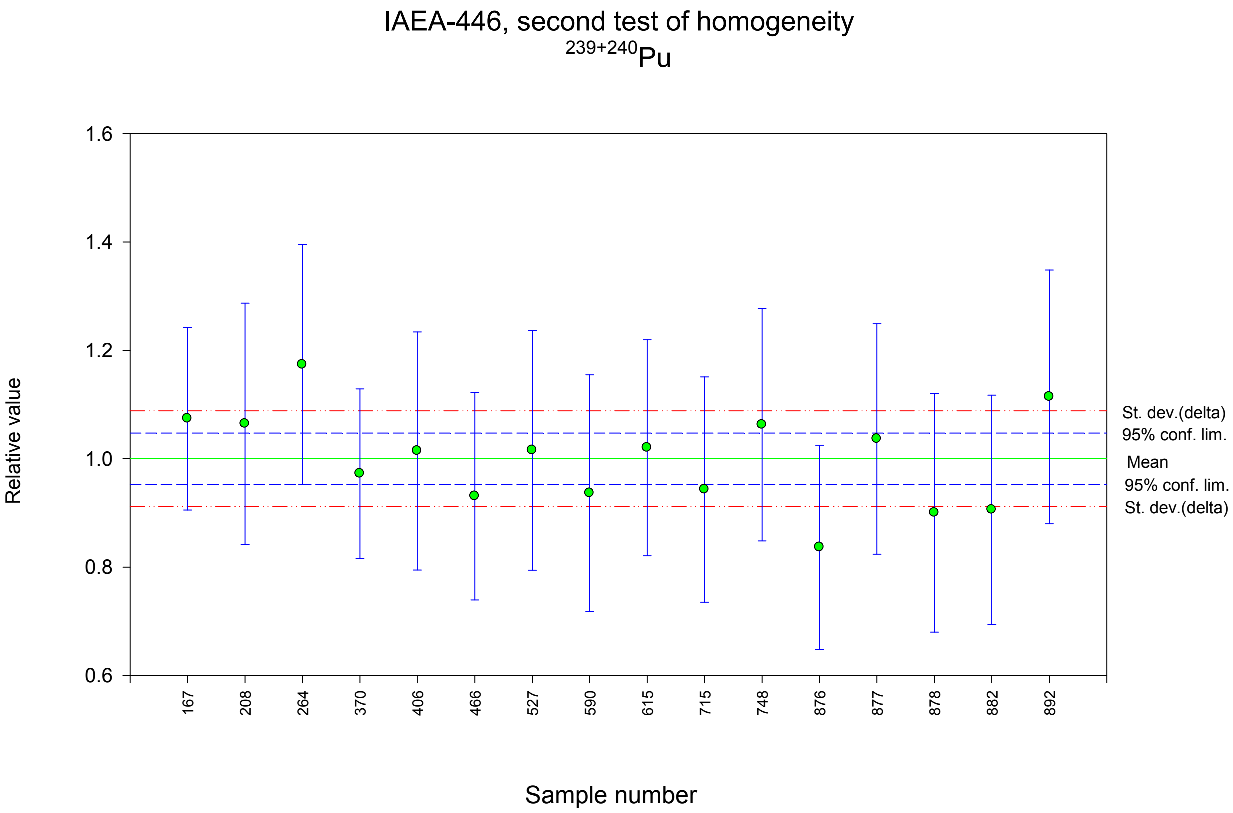

FIG.3. Homogeneity test for $^{239+240}$Pu in IAEA-446.

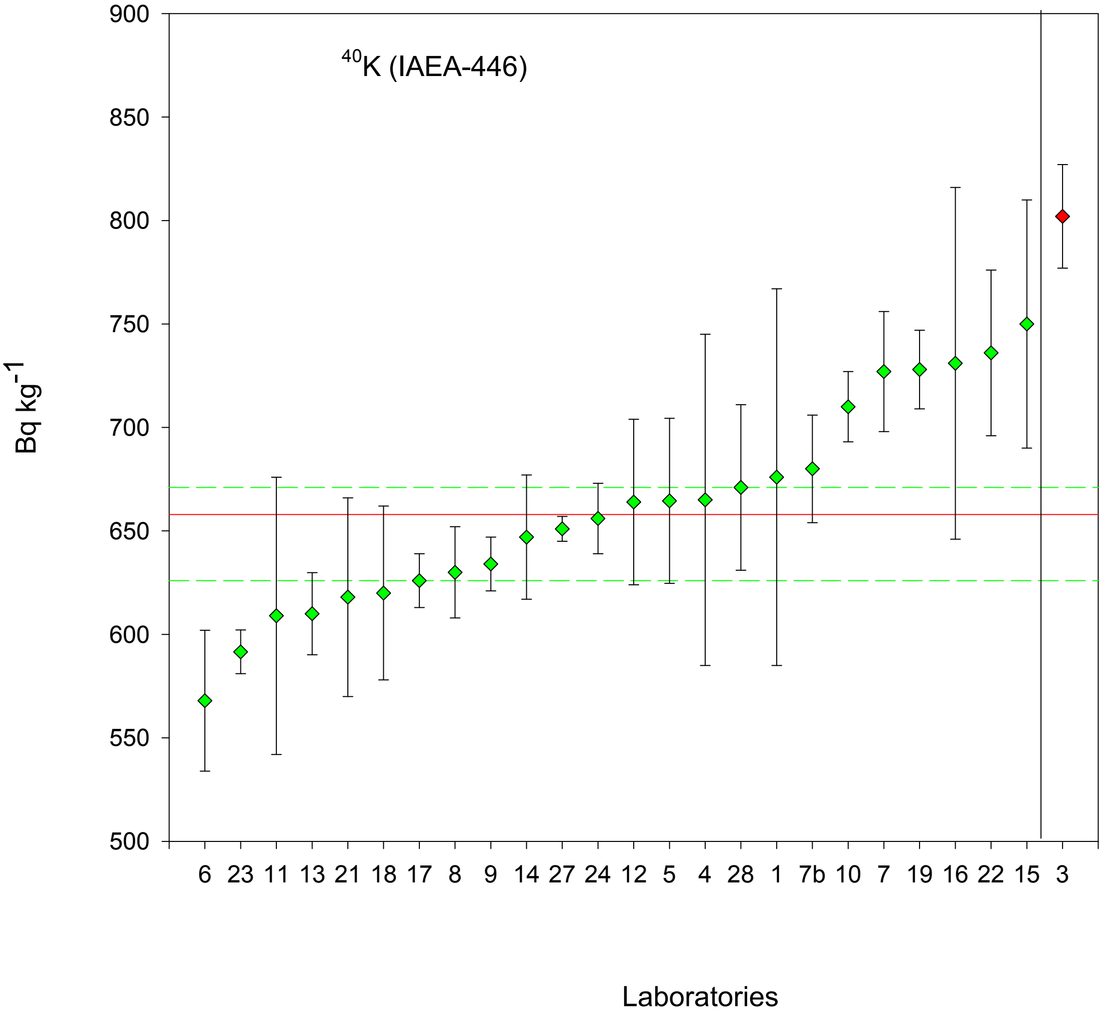

FIG.4. Data reported for ^{40}K.

Note: Figs 4–19: yellow points are warning, red points are outliers, which are not included in the certification process.

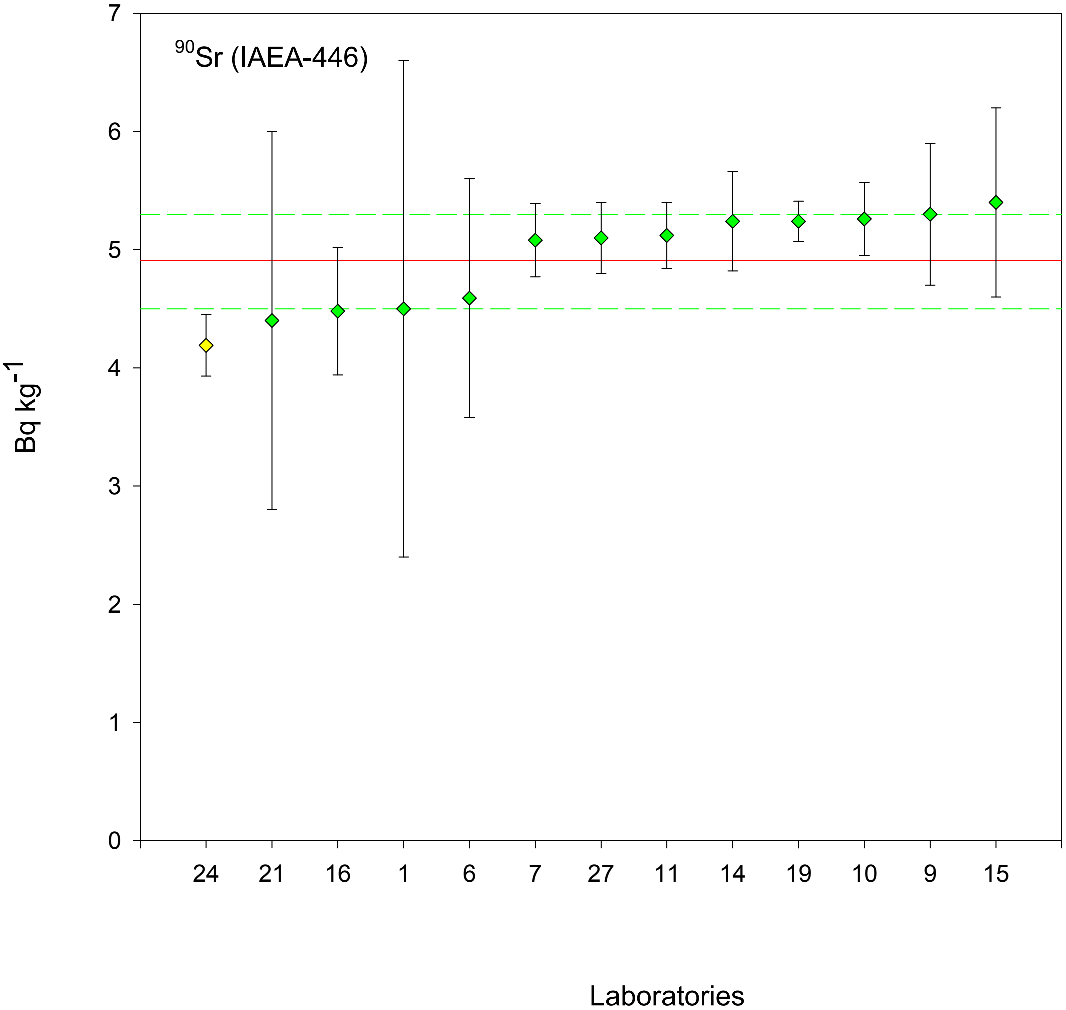

FIG.5. Data reported for ^{90}Sr.

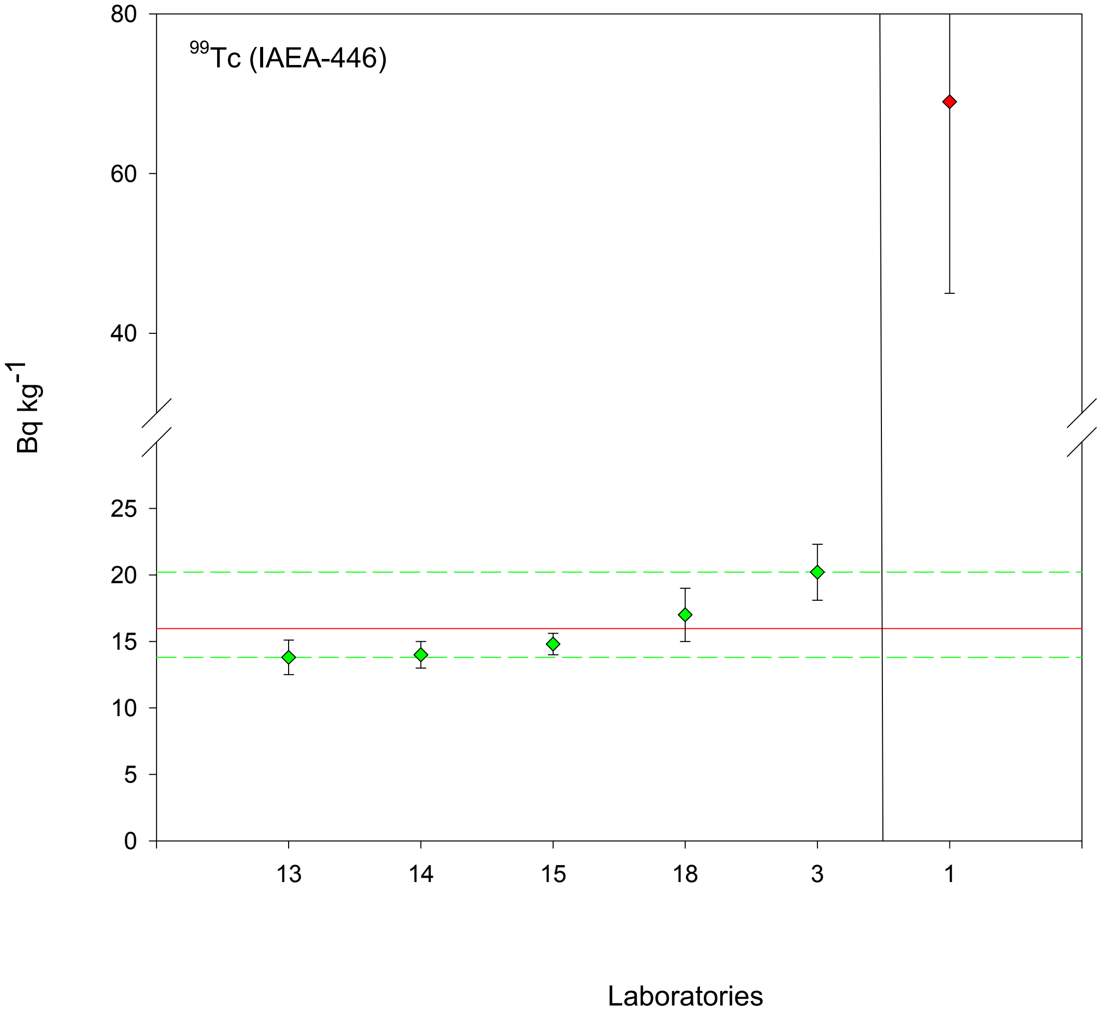

FIG.6. Data reported for ^{99}Tc.

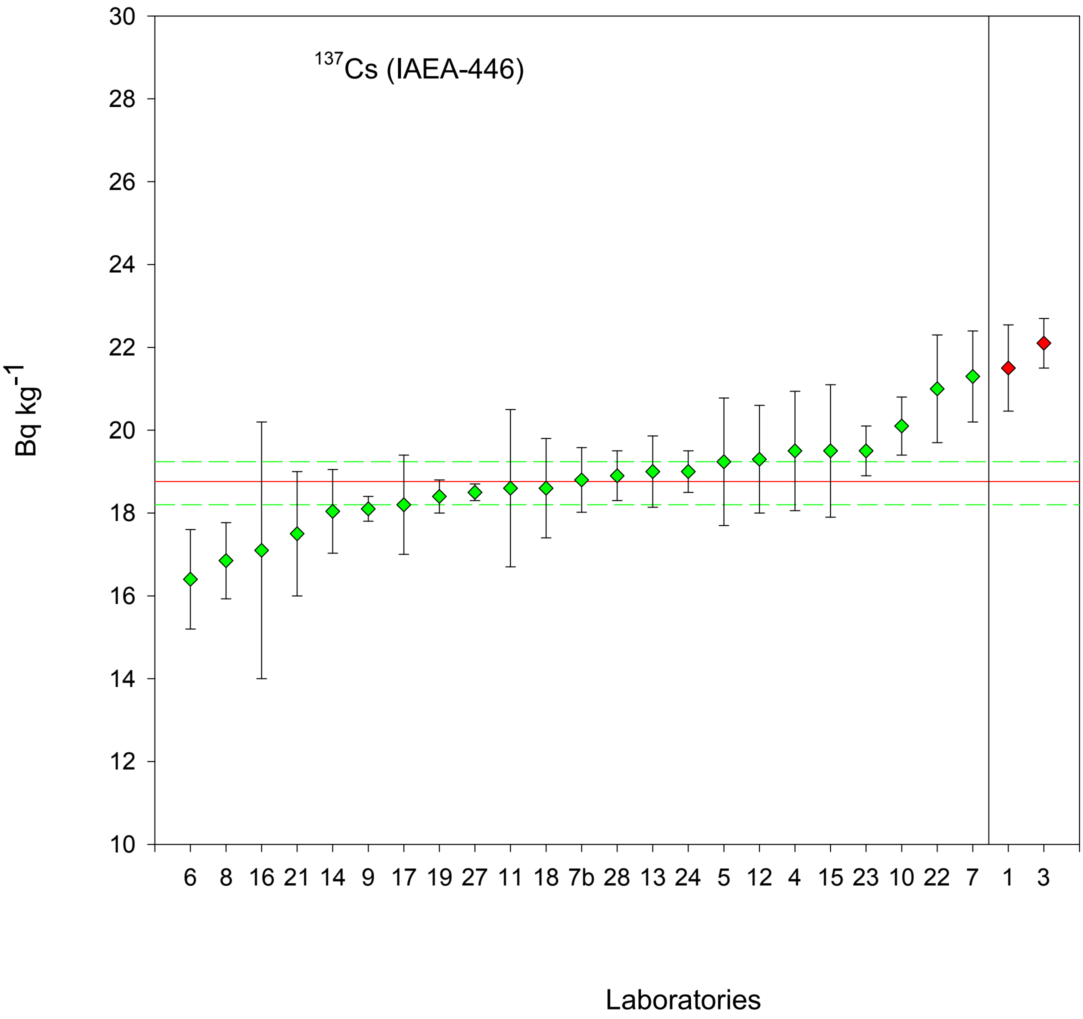

FIG.7. Data reported for ^{137}Cs.

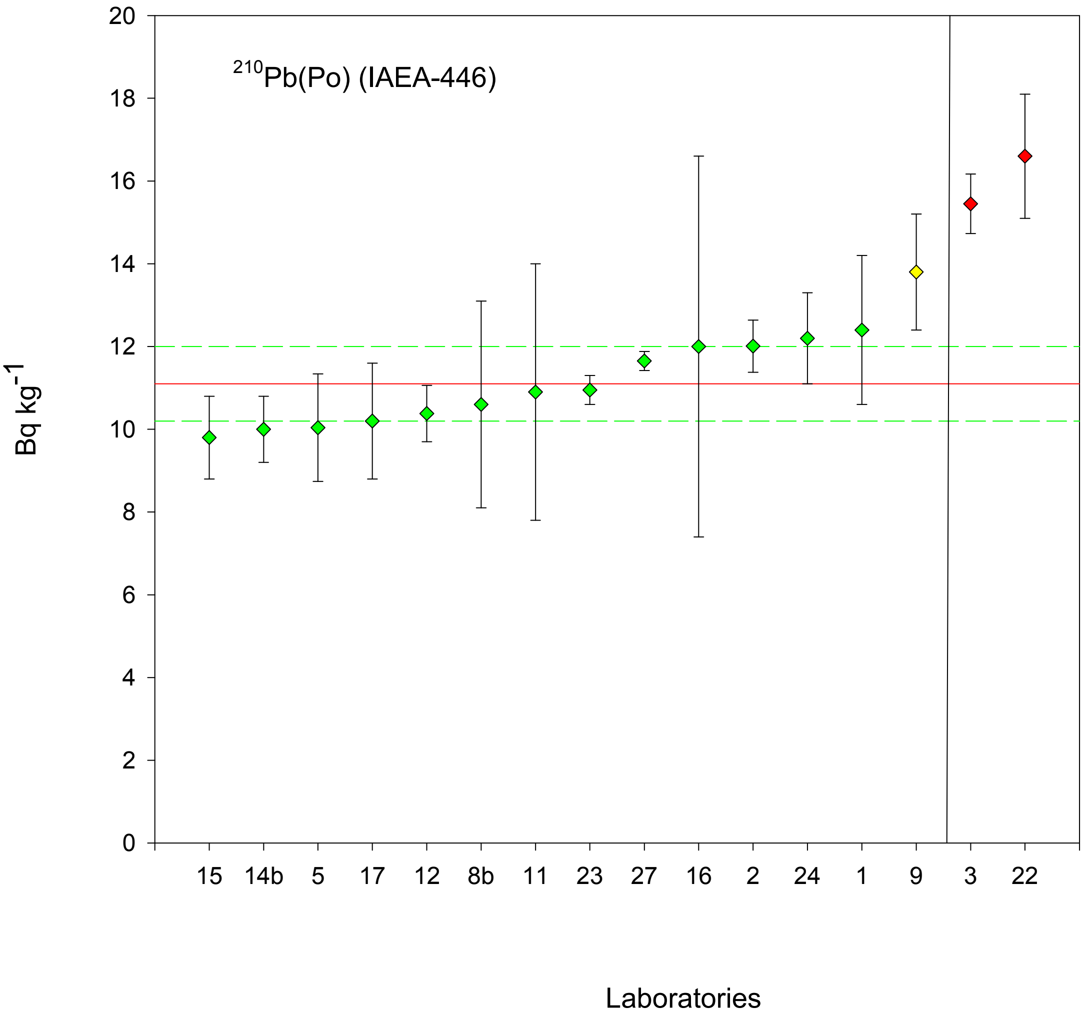

FIG.8. Data reported for ^{210}Pb (^{210}Po).

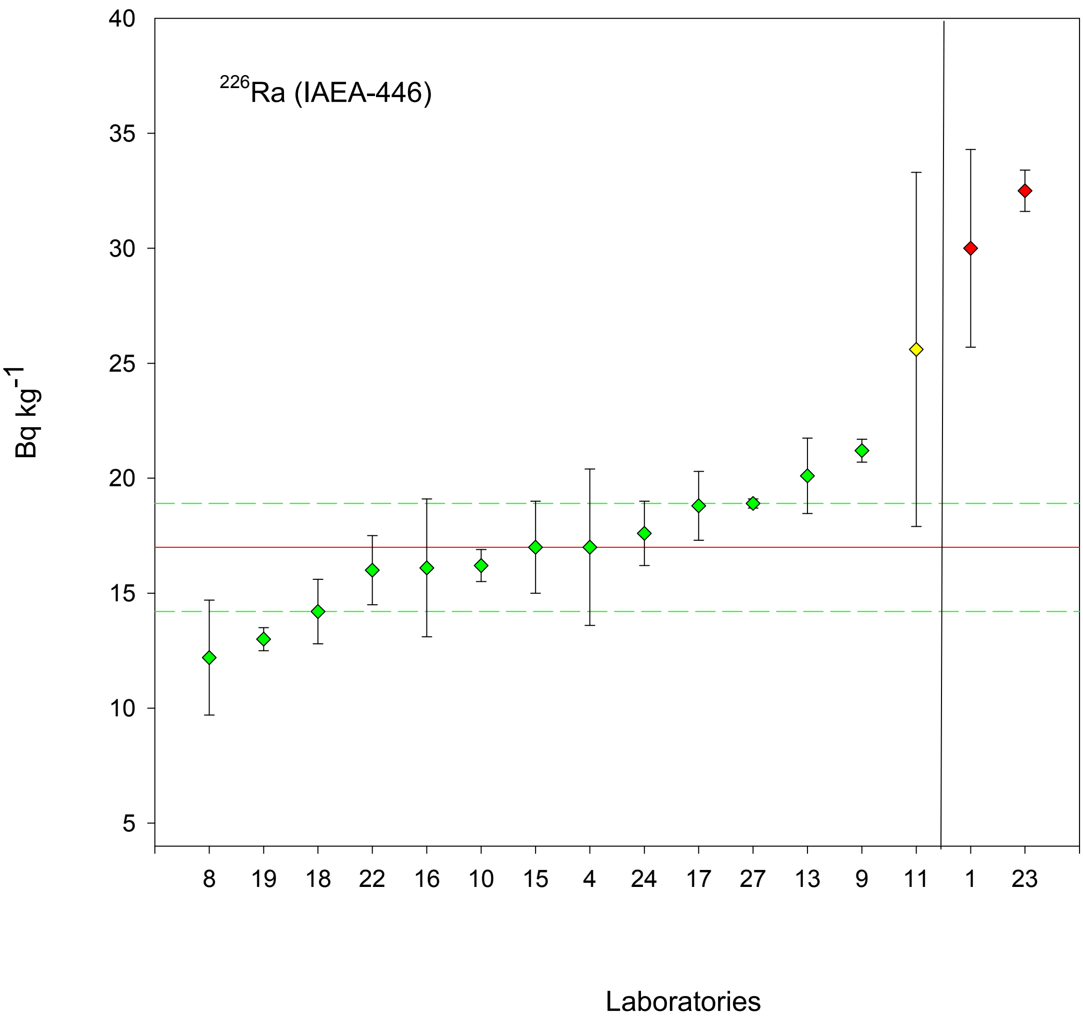

FIG.9. Data reported for ^{226}Ra.

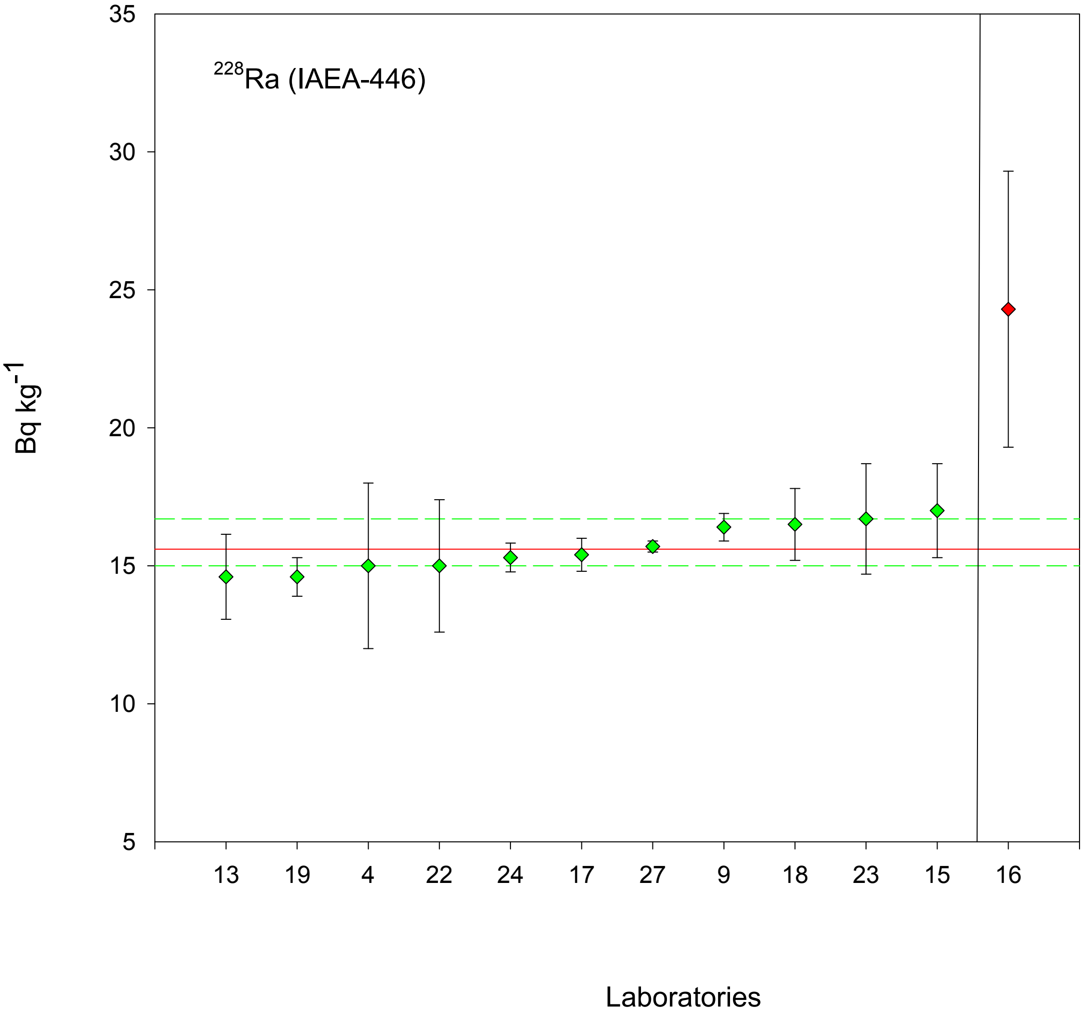

FIG.10. Data reported for ^{228}Ra.

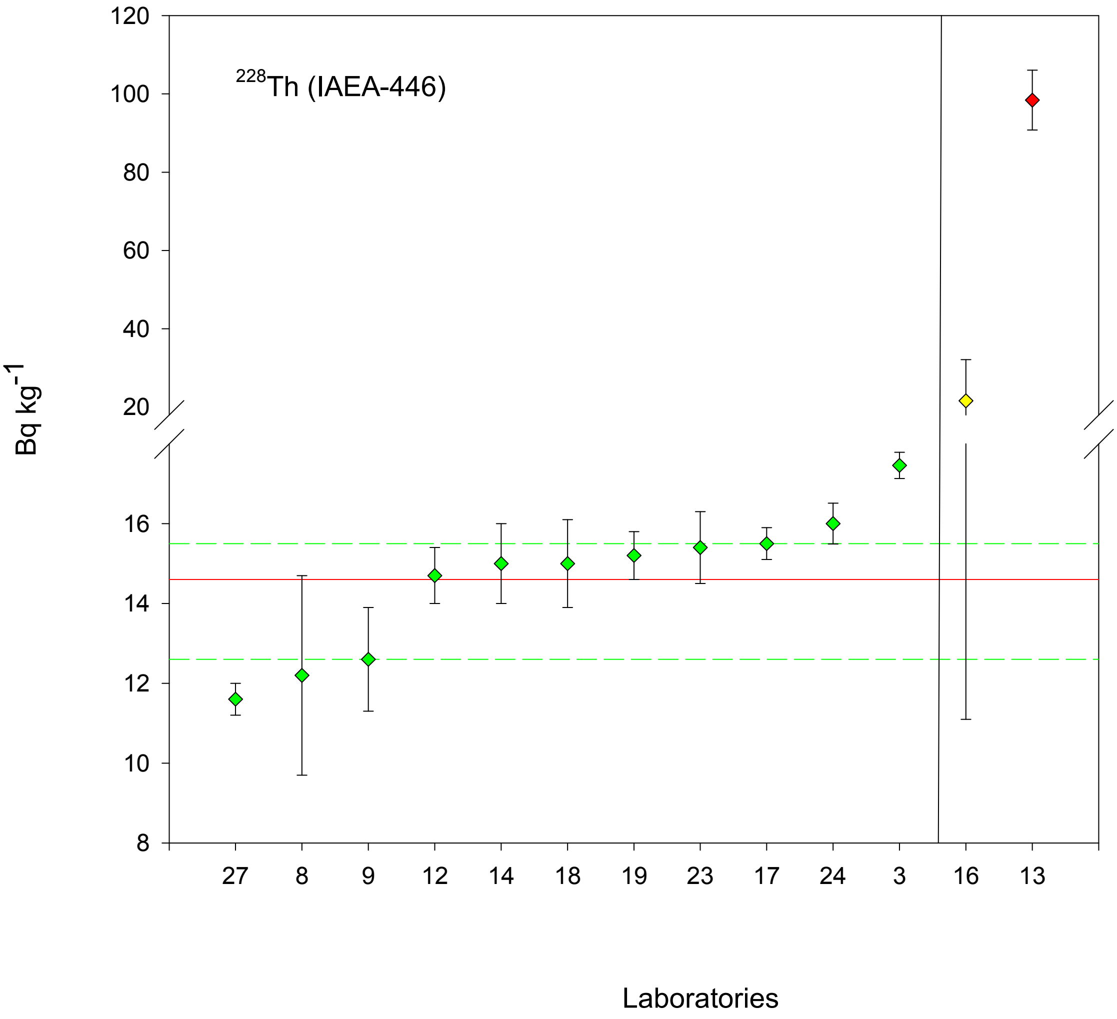

FIG.11. Data reported for ^{228}Th.

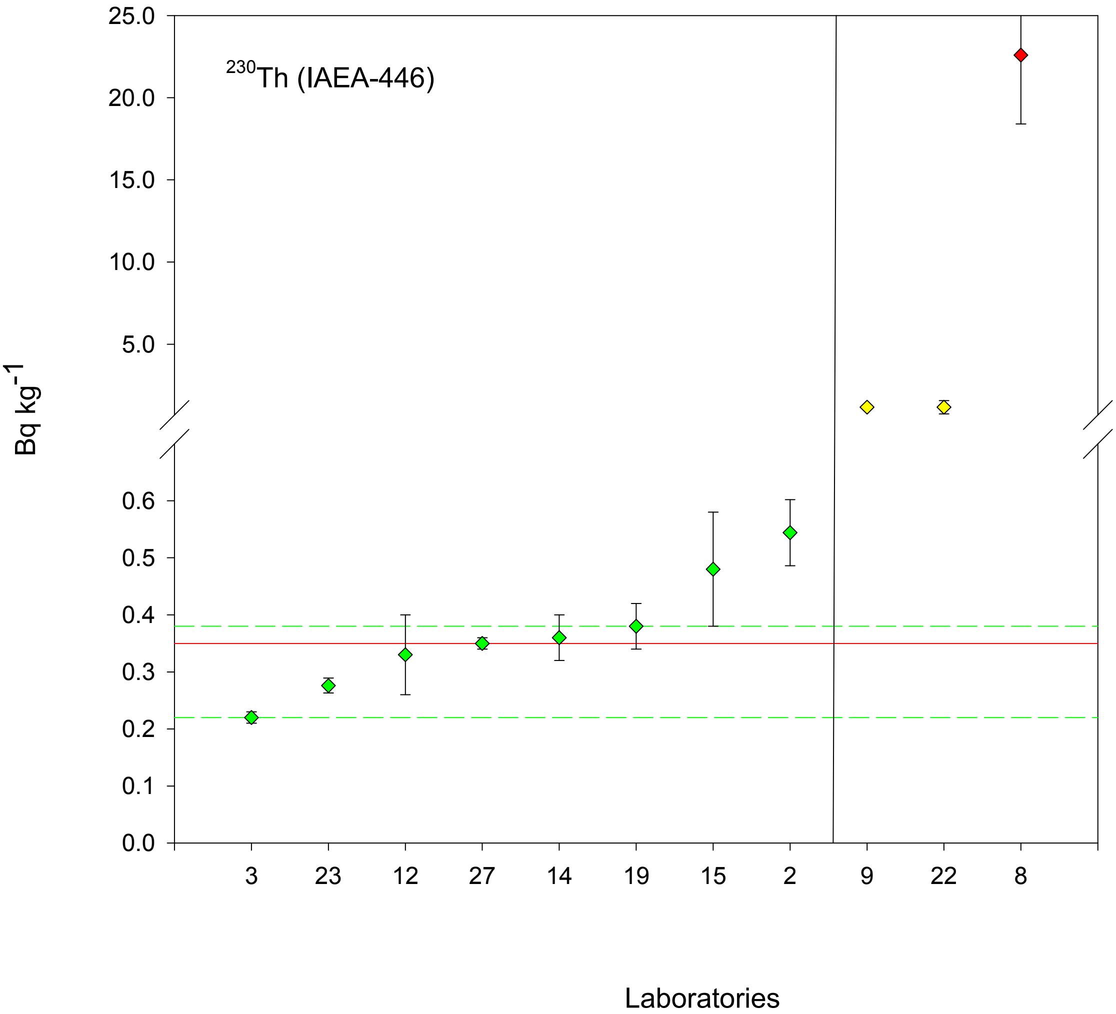

FIG.12. Data reported for ^{230}Th.

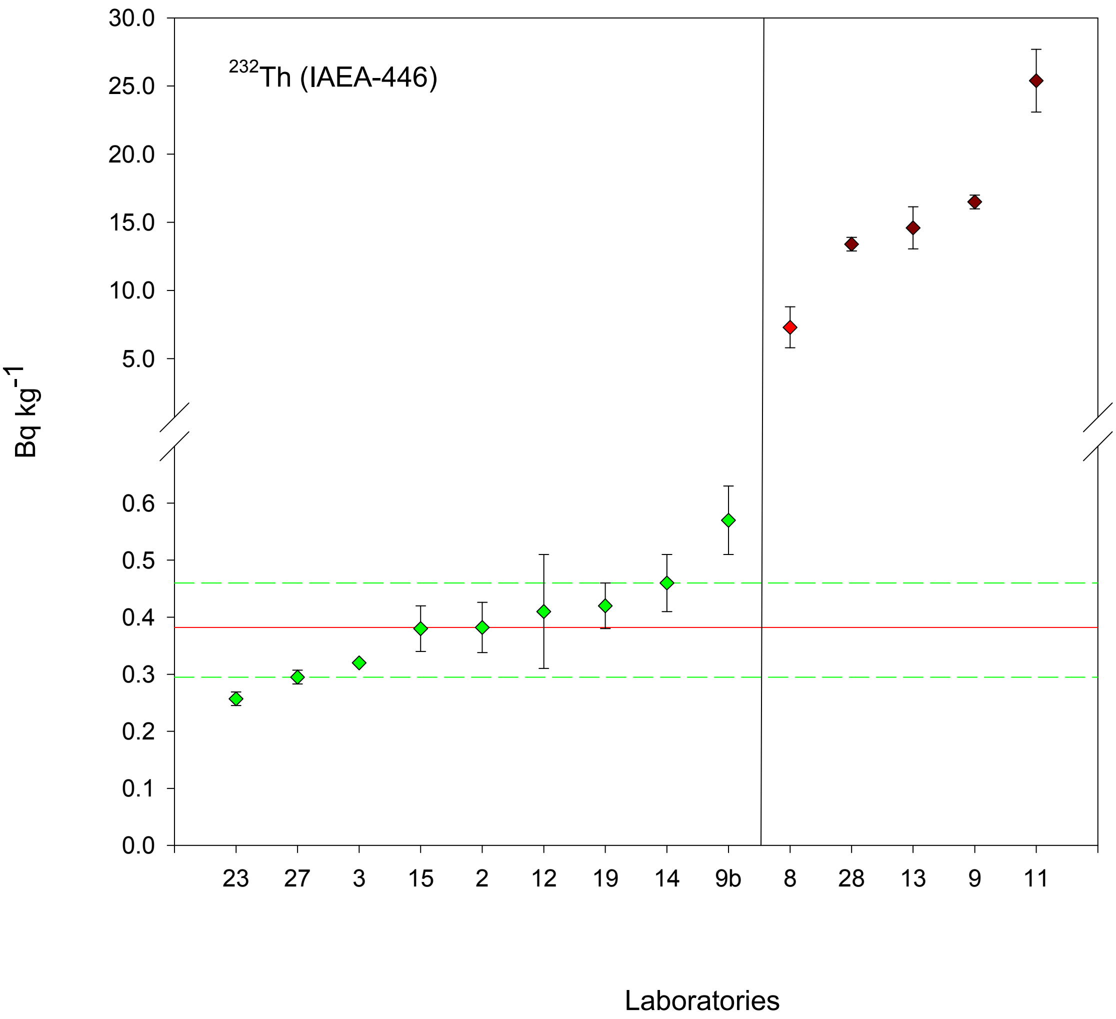

FIG.13. Data reported for ^{232}Th.

Note: dark points determined by gamma spectrometry, which are not included in the data evaluation (see above 7.2.4)

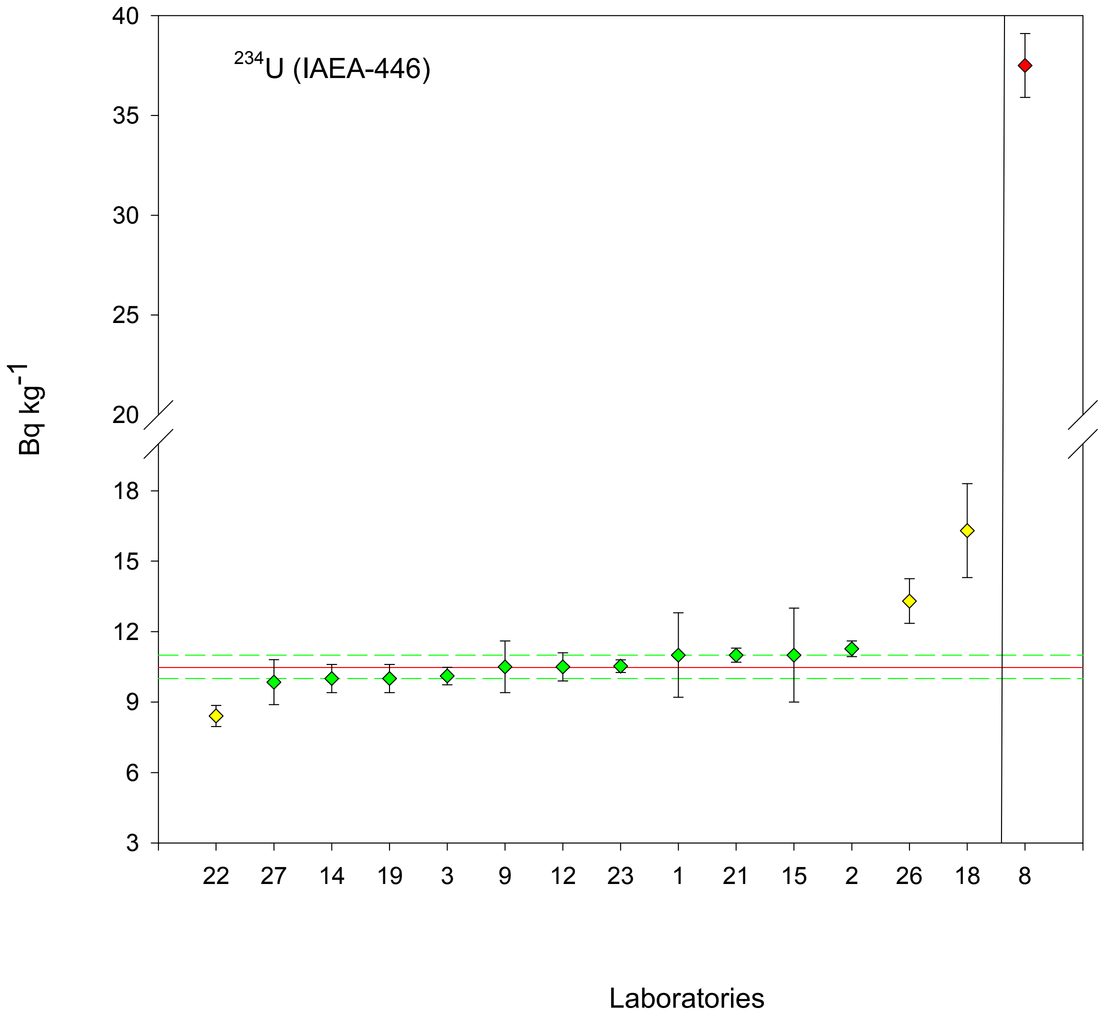

FIG.14. Data reported for ^{234}U.

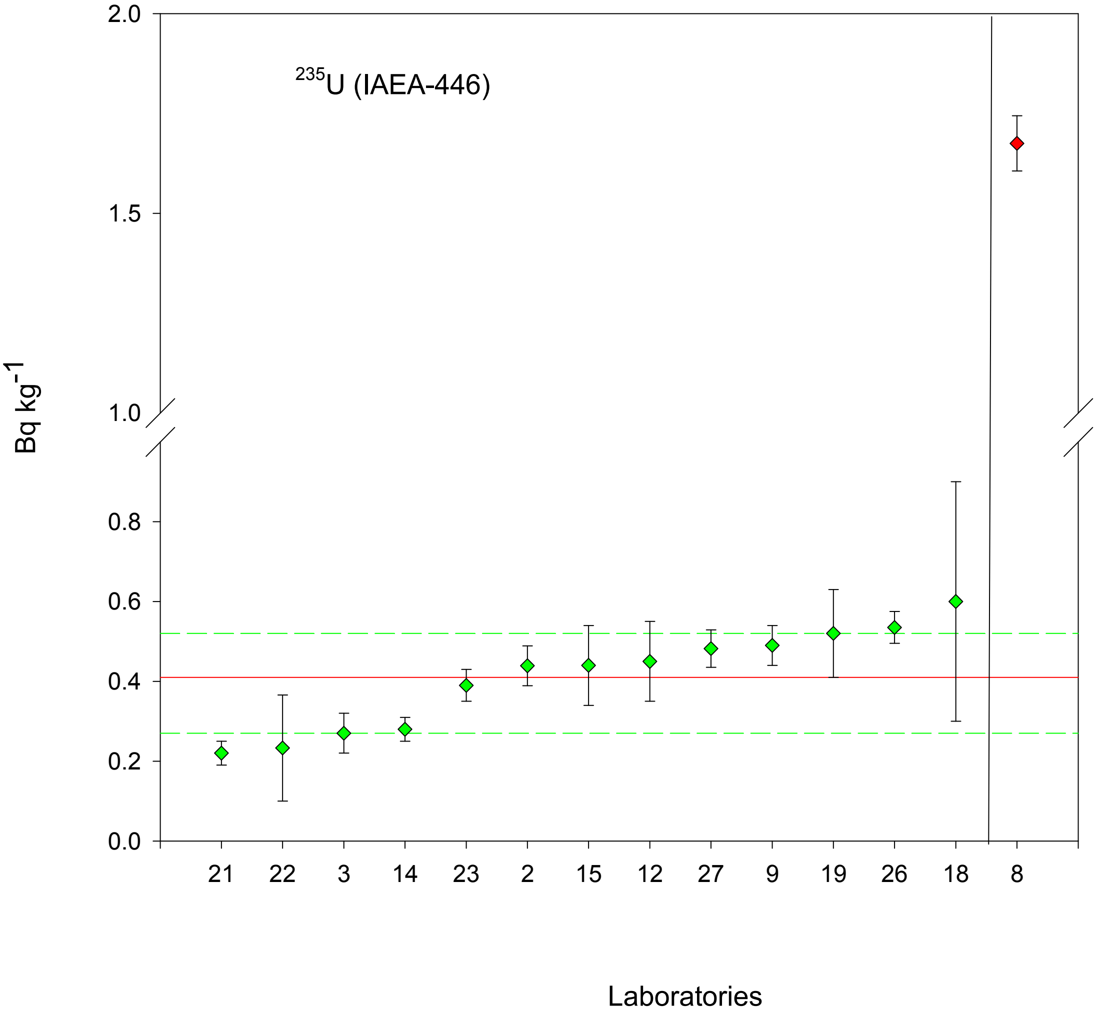

FIG.15. Data reported for ^{235}U.

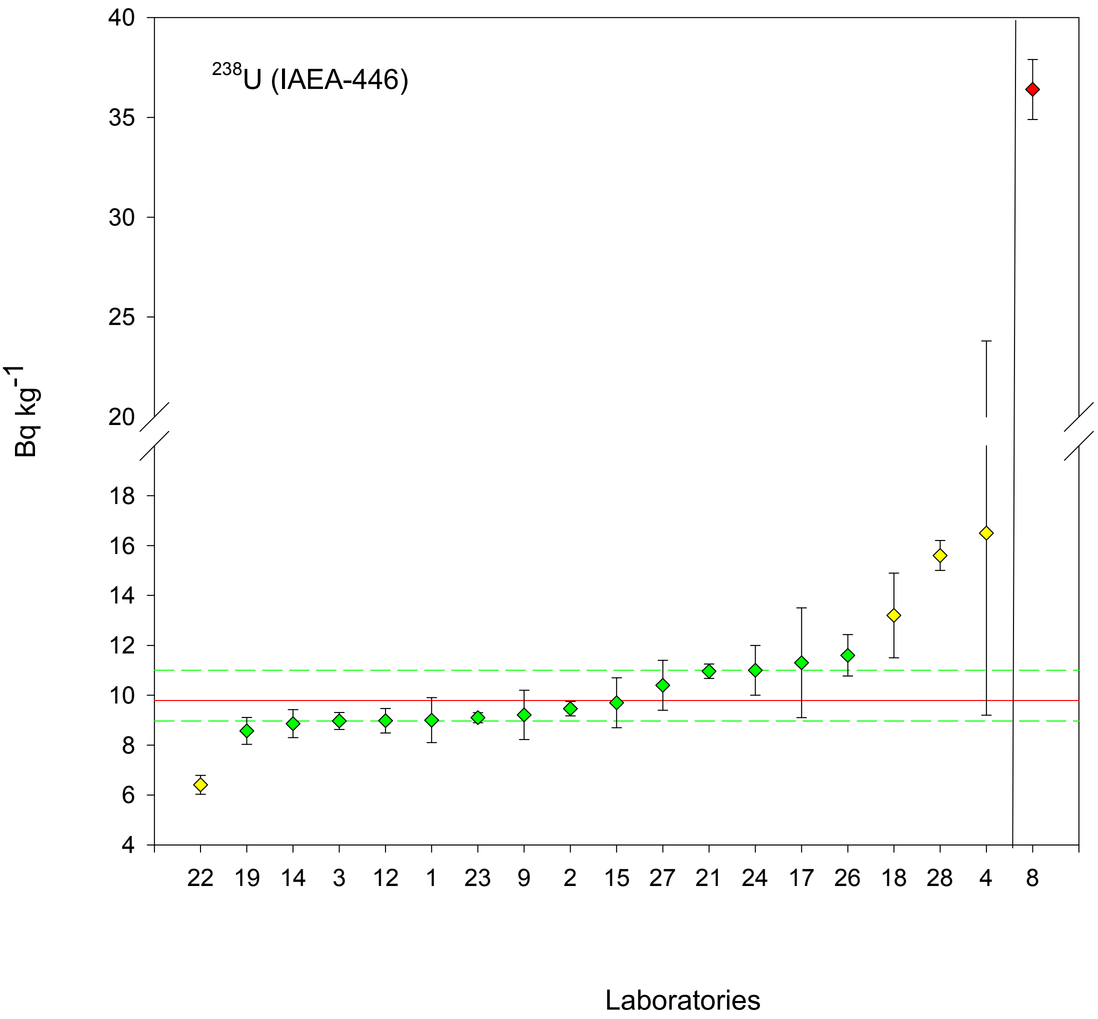

FIG.16. Data reported for ^{238}U.

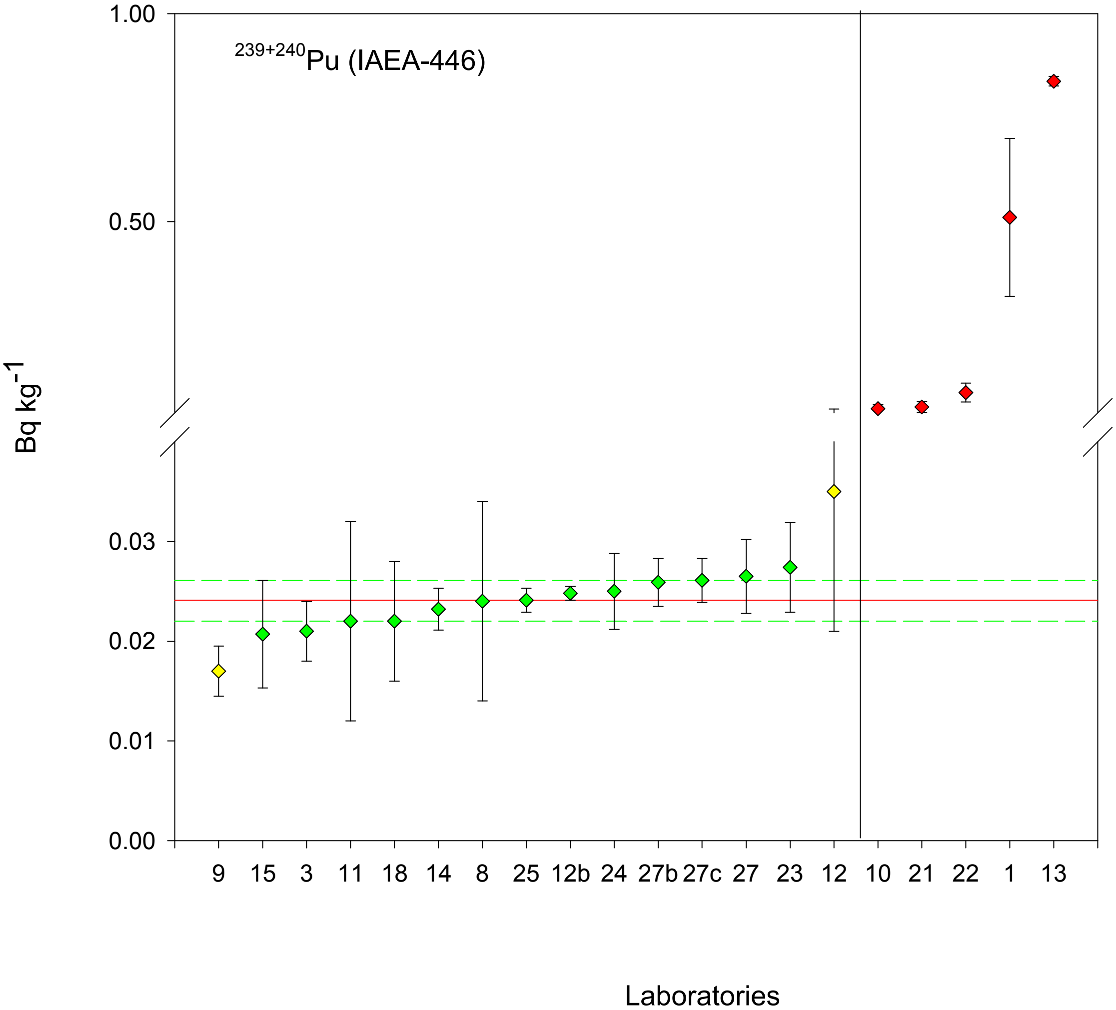

FIG.17. Data reported for $^{239+240}Pu$.

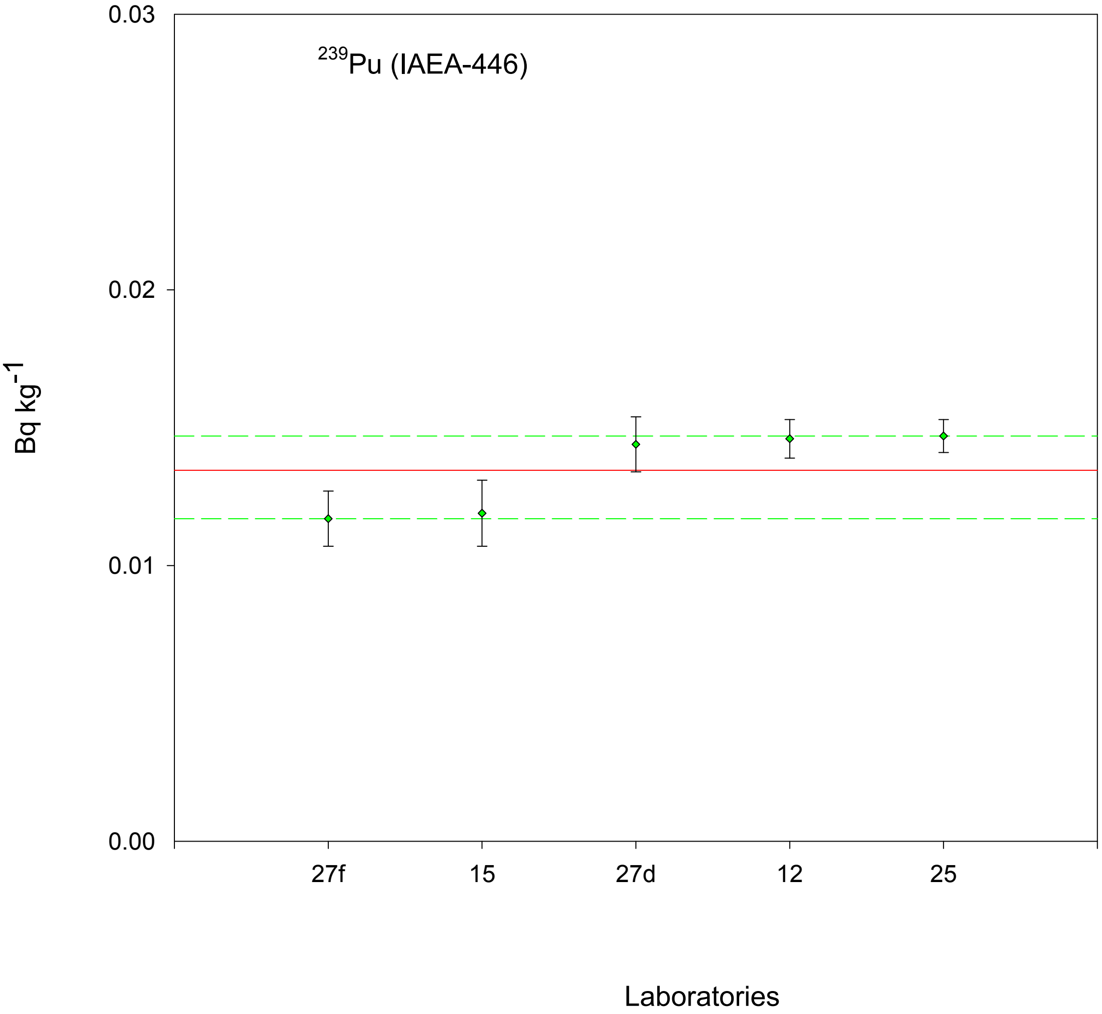

FIG.18. Data reported for ^{239}Pu.

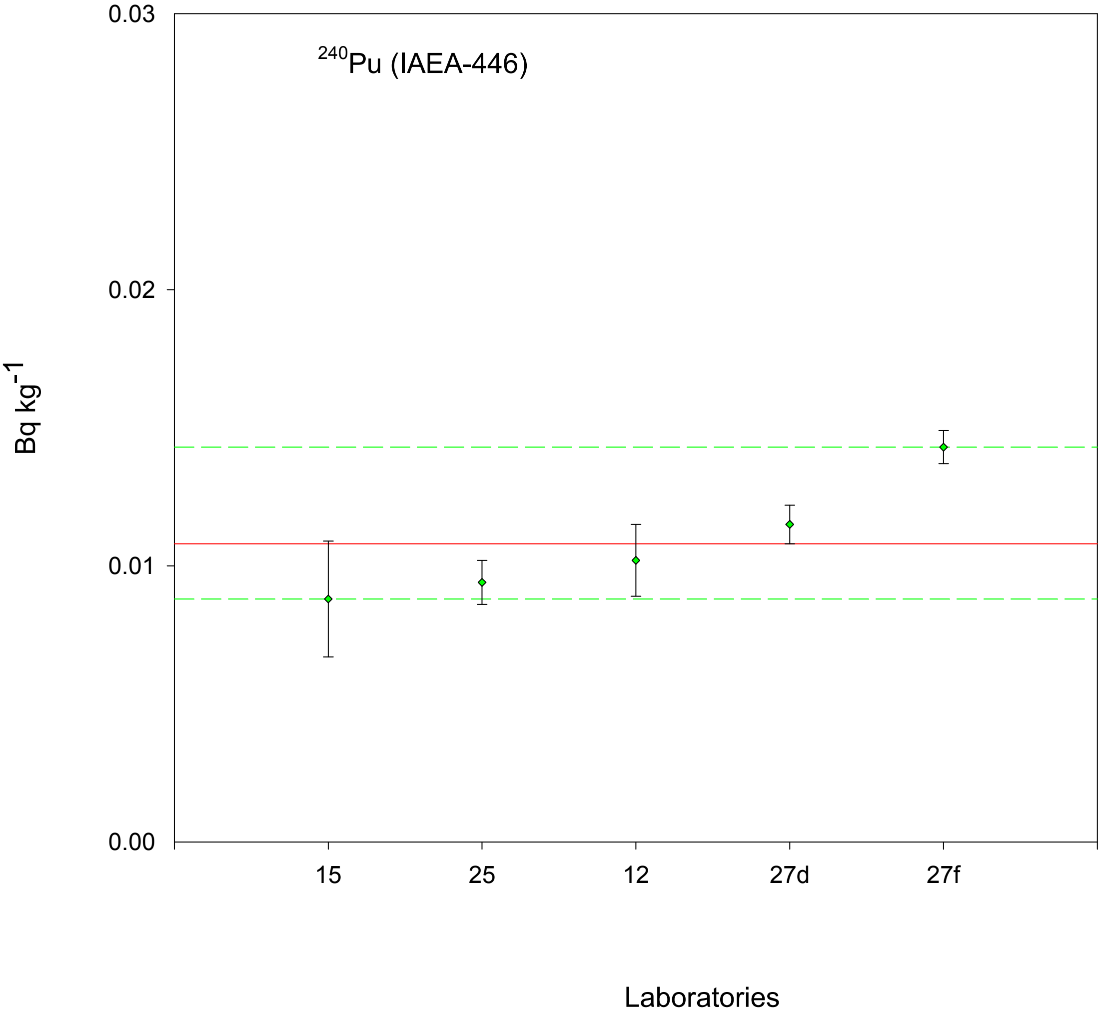

FIG.19. Data reported for ^{240}Pu.

APPENDIX III
z-scores – Graphs

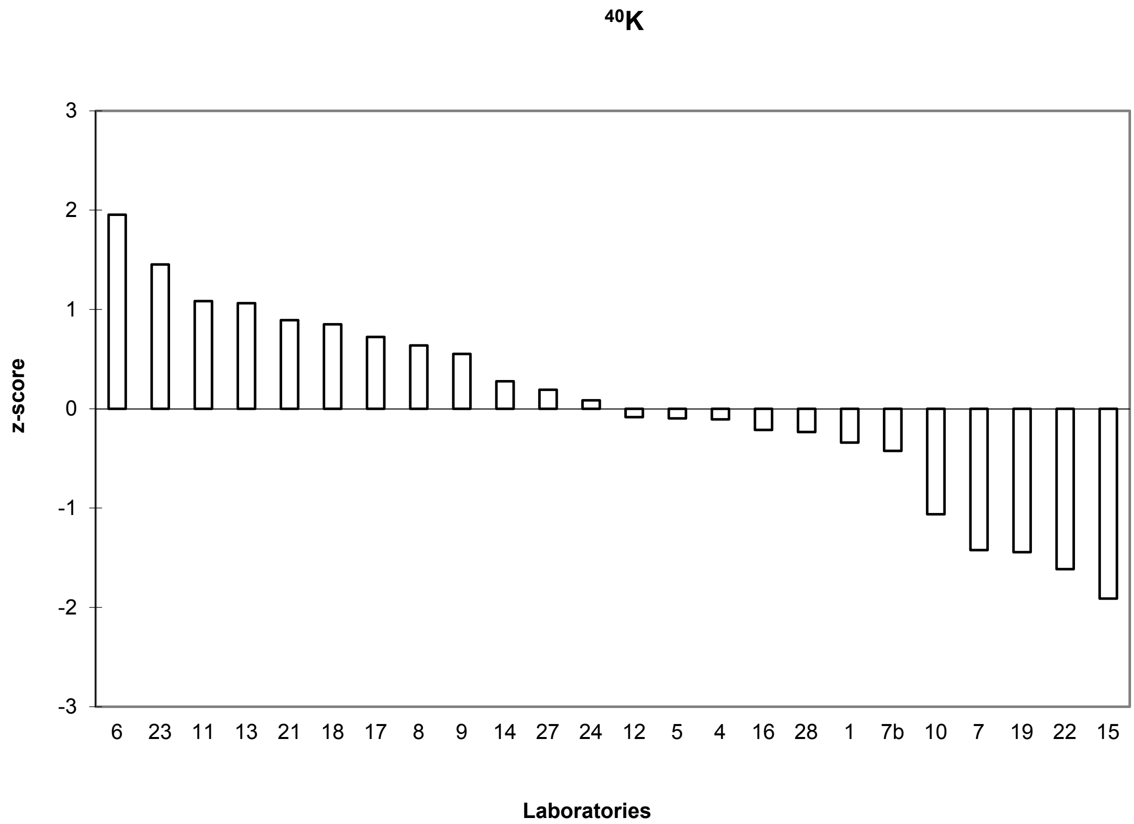

FIG.20. z-score values for ^{40}K.

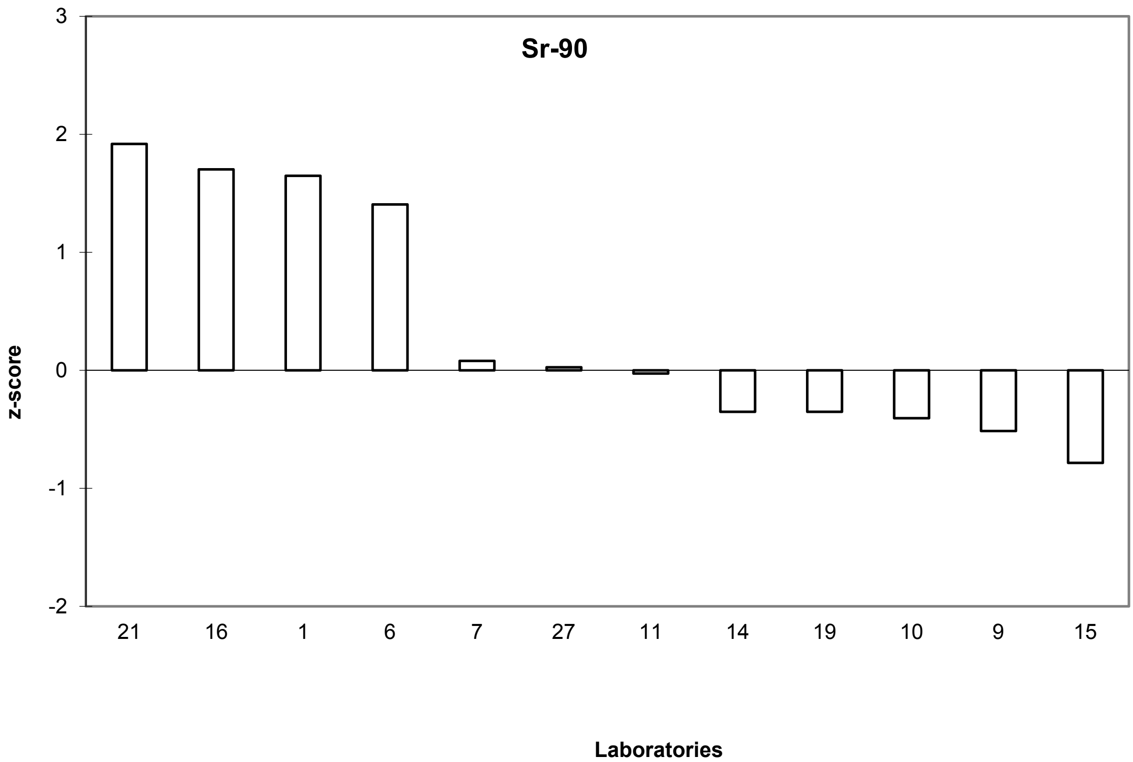

FIG.21. z-score values for ^{90}Sr.

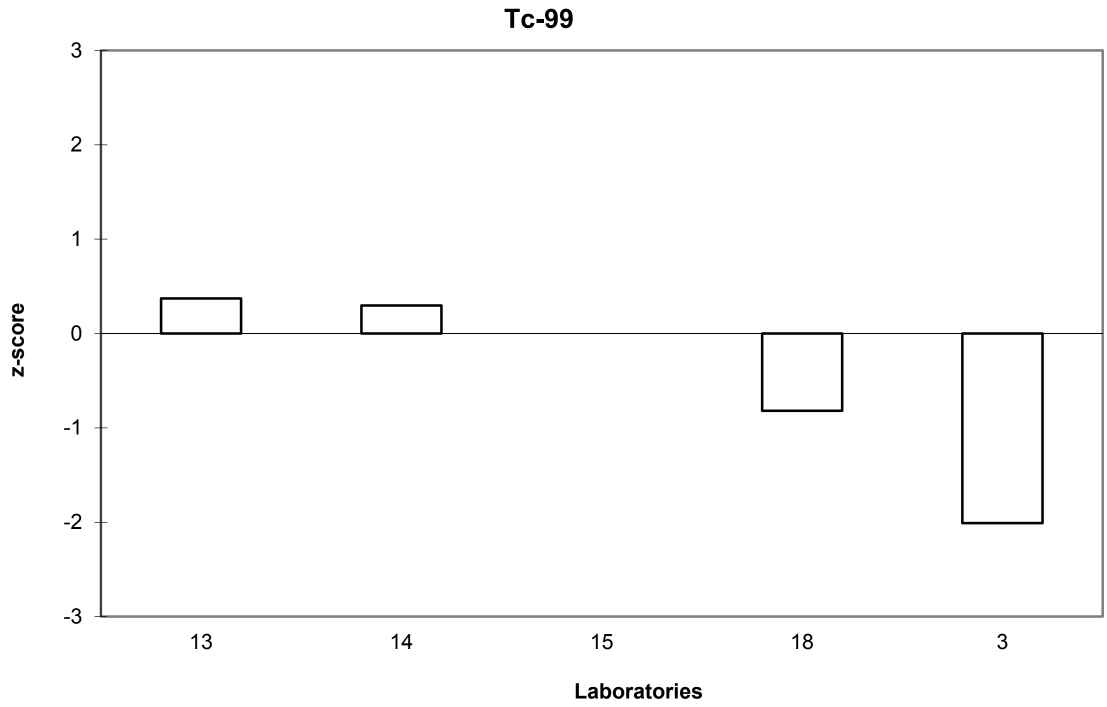

FIG.22. z-score values for ^{99}Tc.

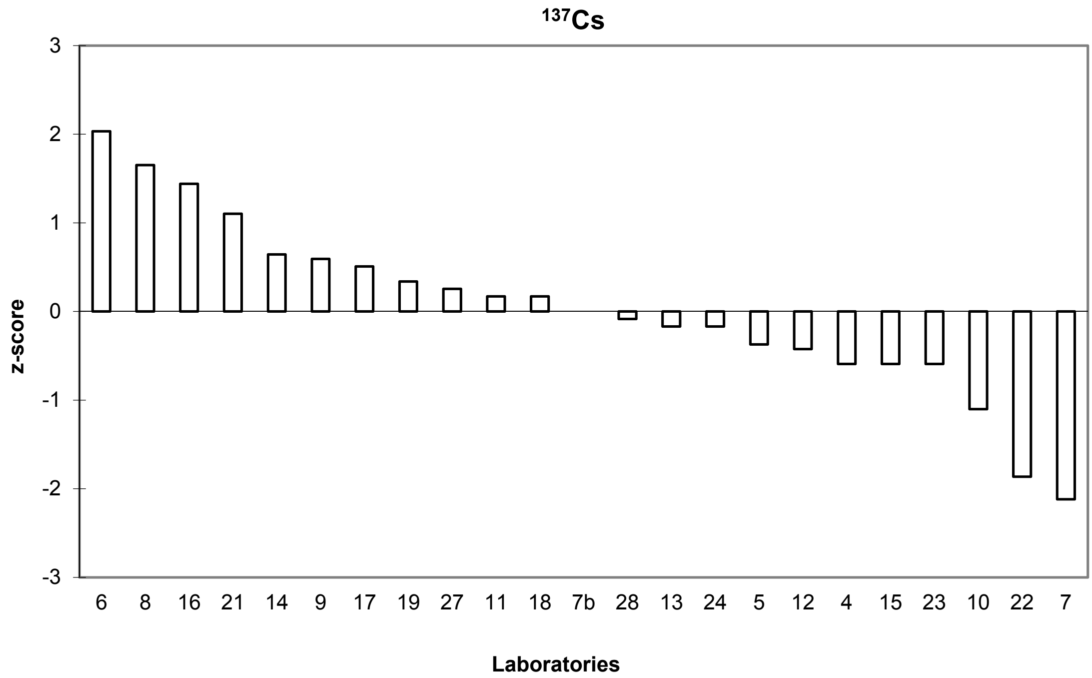

FIG.23. z-score values of ^{137}Cs.

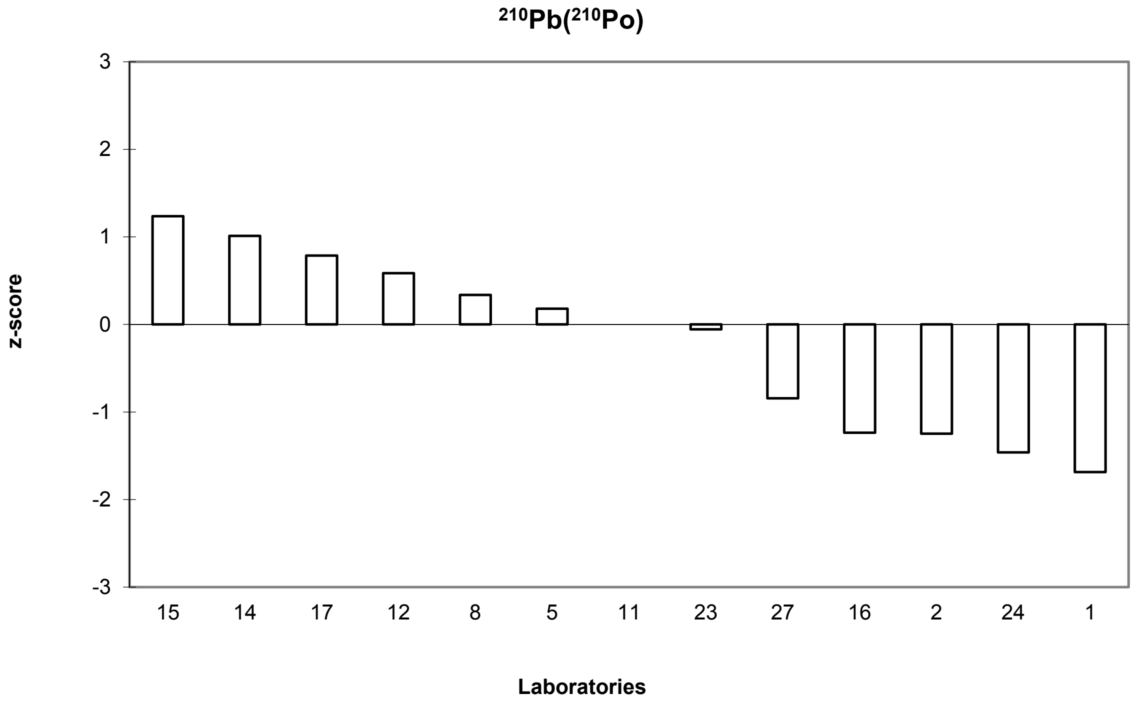

FIG.24. z-score values of ^{210}Pb (^{210}Po).

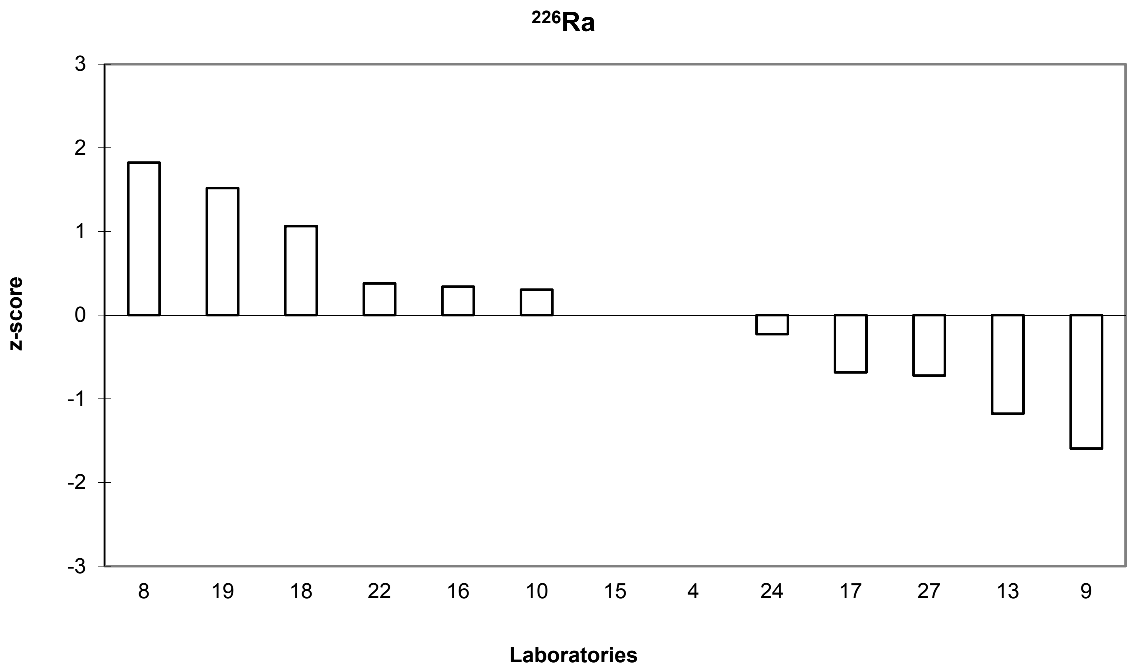

FIG.25. z-score values of ^{226}Ra.

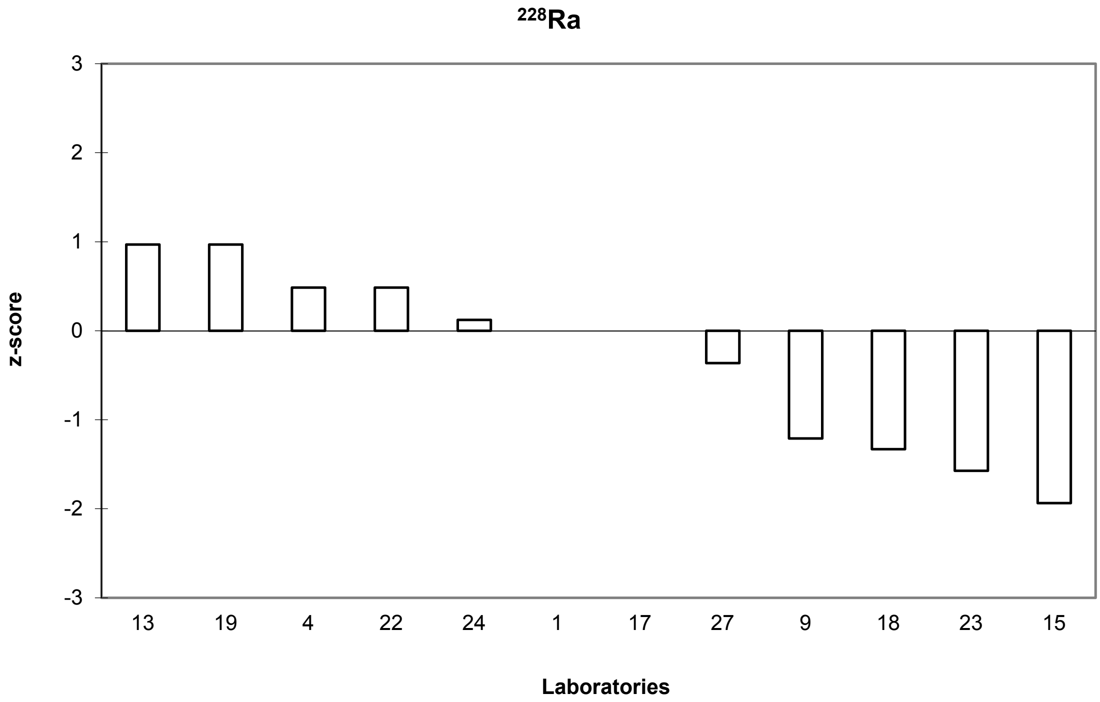

FIG.26. z-score values of ^{228}Ra.

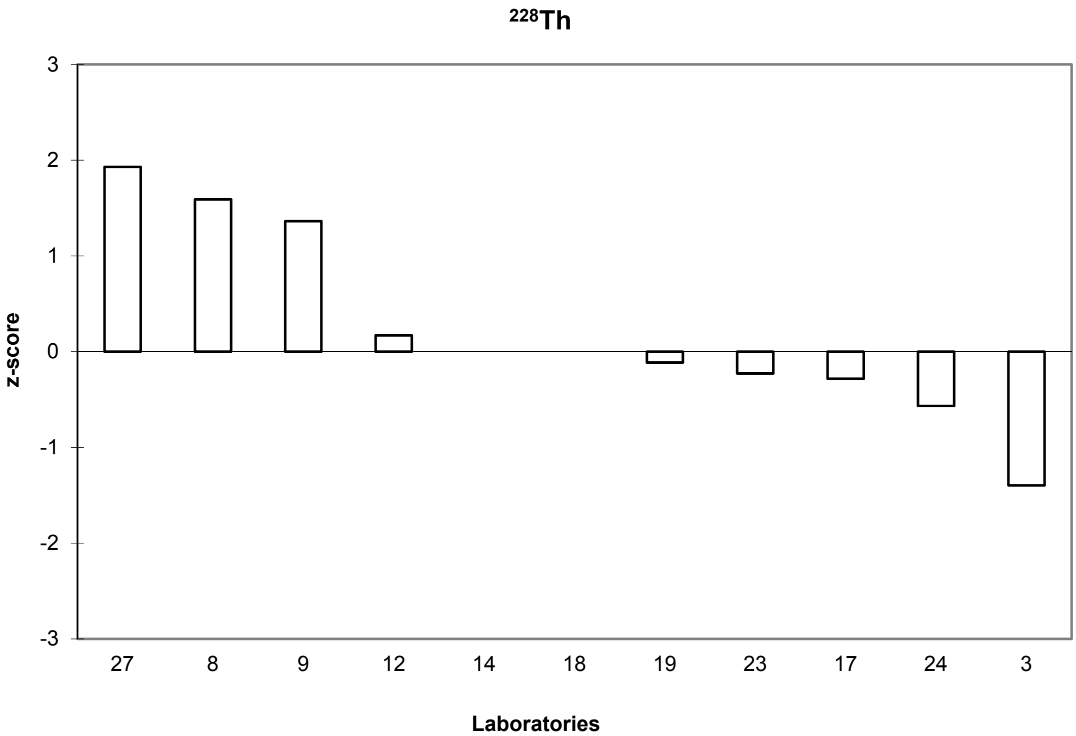

FIG.27. z-score values of ^{228}Th.

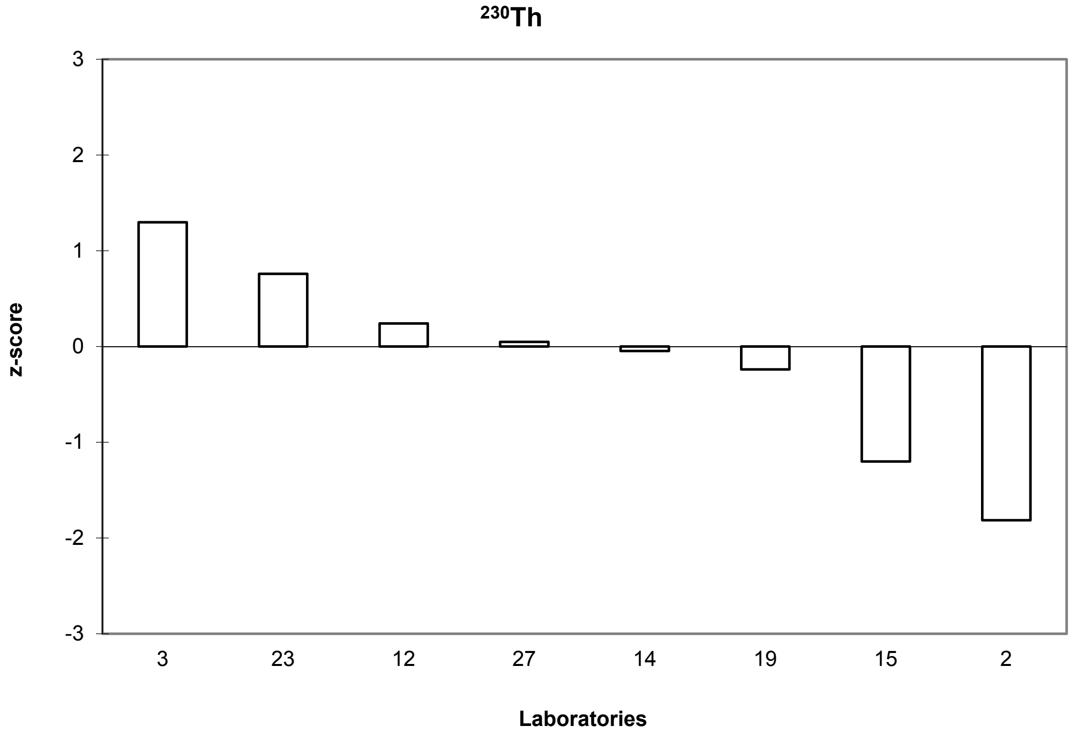

FIG.28. z-score values of ^{230}Th.

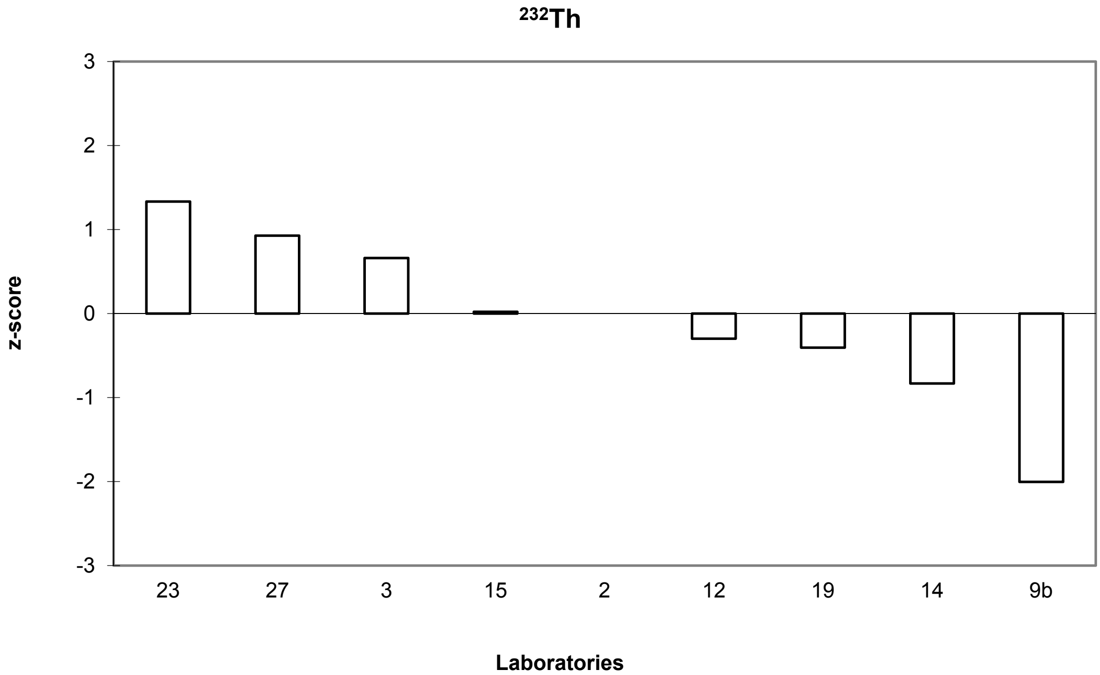

FIG.29. z-score values of ^{232}Th.

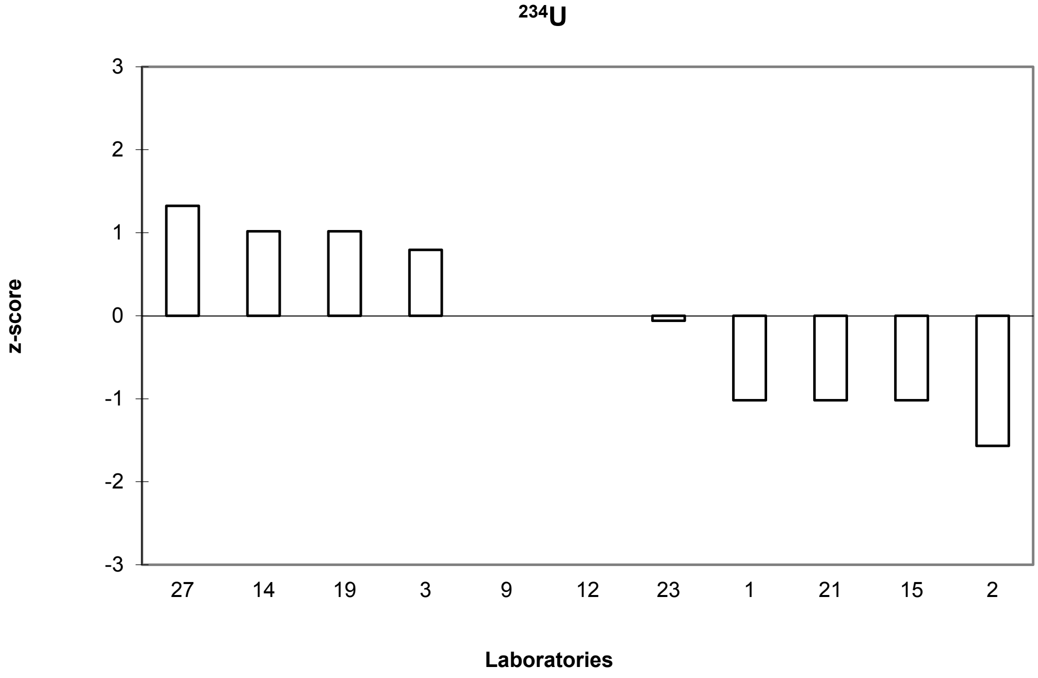

Fig.30. z-score values of ^{234}U.

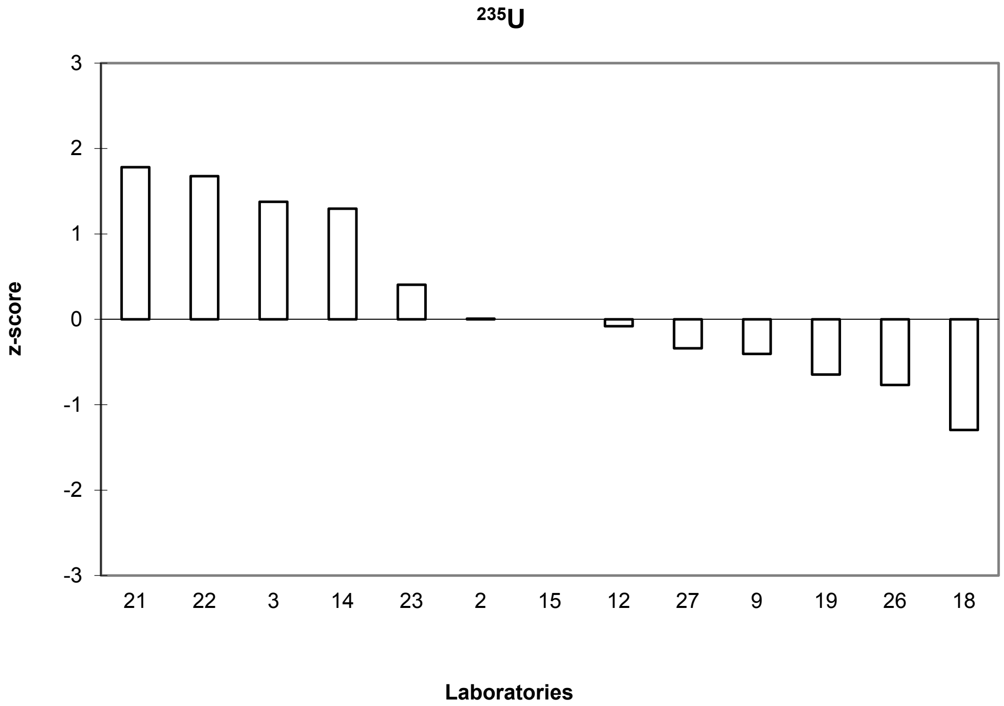

FIG.31. z-score values of ^{235}U.

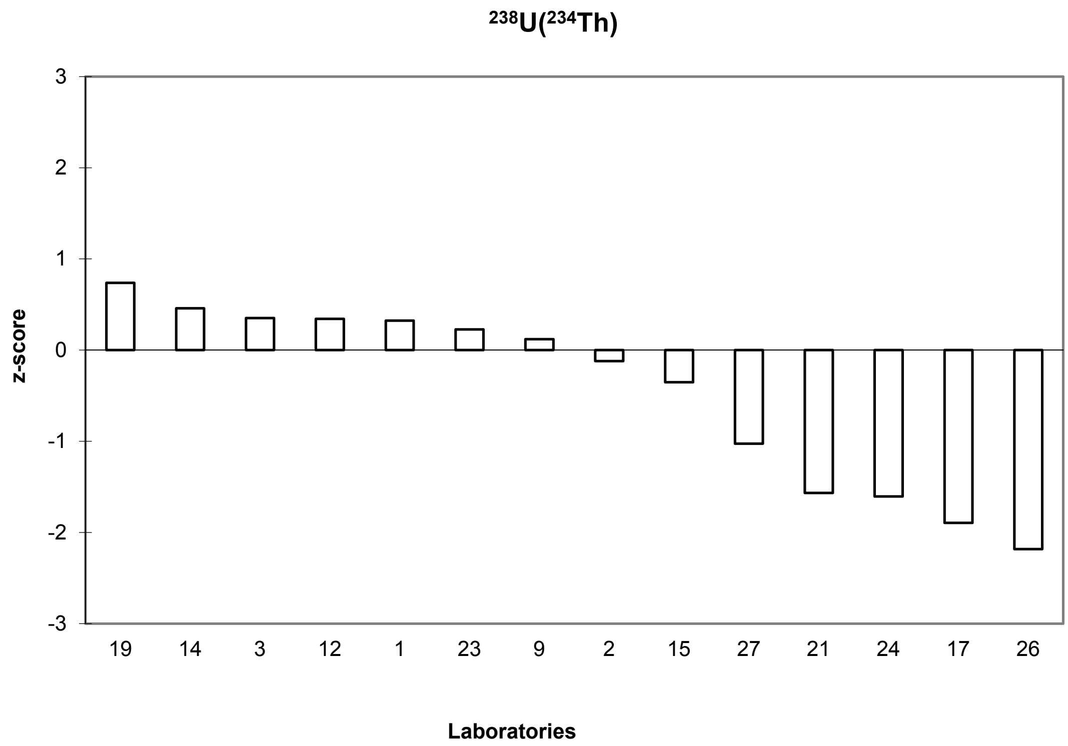

FIG.32. z-score values of ^{238}U.

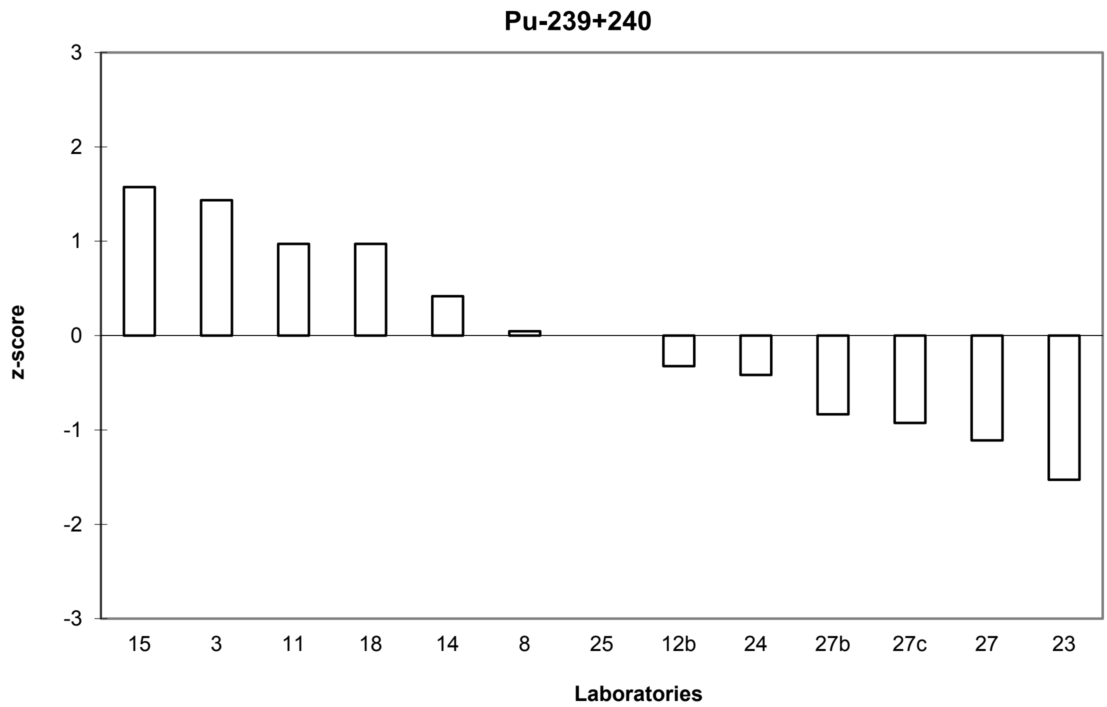

FIG.33. z-score values of $^{239+240}Pu$.

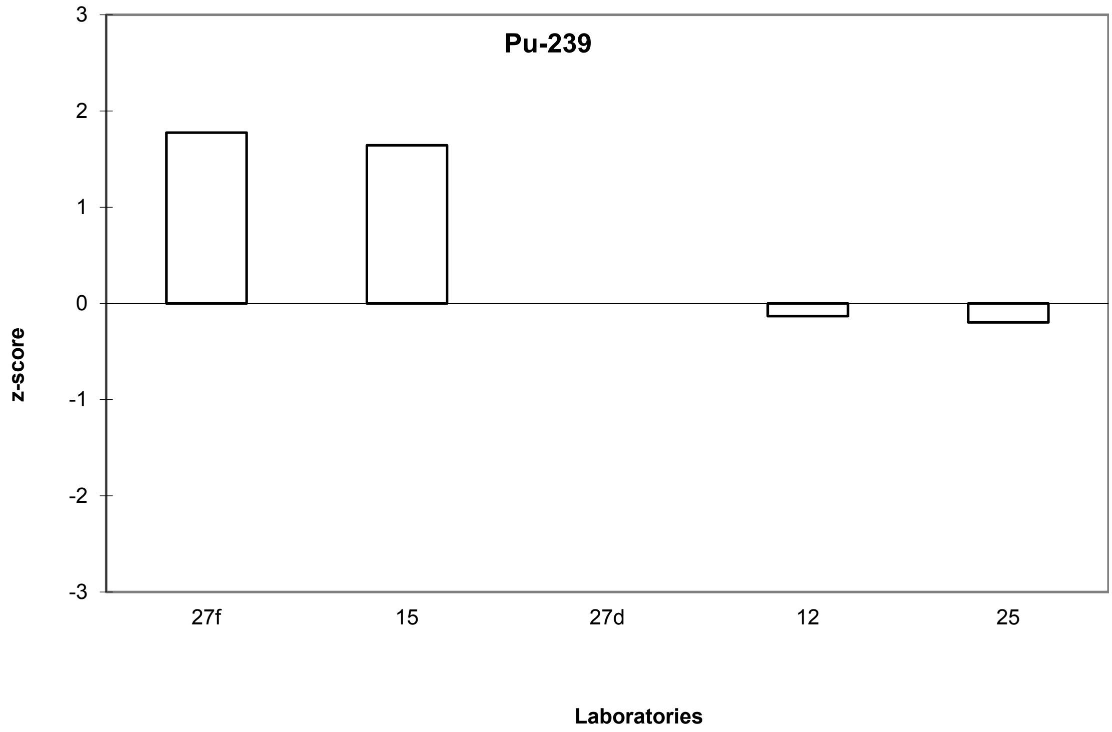

FIG.34. z-score values of ^{239}Pu.

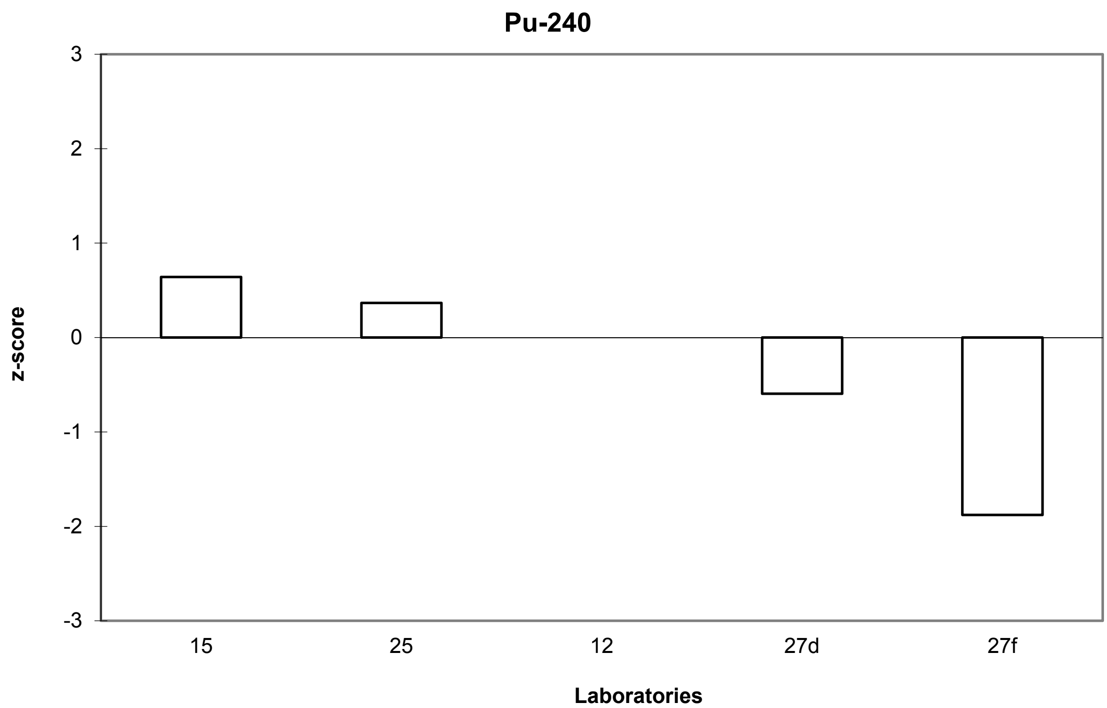

FIG.35. z-score values of ^{240}Pu.

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