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REPORT ON THE WORLDWIDE INTERCOMPARISON

IAEA-414

RADIONUCLIDES

In Mixed Fish

From IRISH SEA and the NORTH SEA

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SUMMARY

The results of an intercomparison exercise, designed for the determination of anthropogenic and natural radionuclides in mixed fish sample from the Irish Sea and North Sea, IAEA-414, are reported. The data received from 90 laboratories have been evaluated.

The following are the recommended and information values (medians), with confidence intervals, for the set of radionuclides determined in the study. All the values are given for the reference date 1 January 1997 and expressed in Bq kg^{-1} dry weight.

Radionuclide	Median	Confidence interval ($\alpha = 0.05$)
	Recommended value	
^{137}Cs	5.14	5.00-5.27
^{238}Pu	0.023	0.022-0.025
$^{239+240}Pu$	0.120	0.113-0.125
^{241}Am*	0.196	0.189-0.199
	Information value	
^{40}K	480	461-498
^{90}Sr	0.28	0.10-0.54
$^{210}Pb(^{210}Po^{\#})$	2.22	1.55-2.60
^{226}Ra	1.40	0.59-1.76
^{232}Th	0.029	0.025-0.031
^{234}U	1.22	1.14-1.27
^{235}U	0.050	0.045-0.058
^{238}U	1.112	1.065-1.170

* The values should be corrected for in-growth from ^{241}Pu.
# ^{210}Po is in equilibrium with ^{210}Pb at the time of measurement.

1. INTRODUCTION

The accurate and precise determinations of radionuclide concentrations in marine samples are important aspects of marine radioactivity assessments and the use of radionuclides in studies of oceanographic processes. To address the problem of data quality, and to assist Member States in verifying the performance of their laboratories, the IAEA Marine Environment Laboratory (MEL) in Monaco has conducted intercomparison exercises on radionuclides in marine samples for many years as part of its contribution to the IAEA's programme of Analytical Quality Control Services (AQCS).

For this intercomparison exercise, IAEA-MEL with the help of the Fisheries Laboratory of Ministry for Agriculture Fisheries and Food (MAFF), (presently the Centre for Environment, Fisheries and Aquaculture Science (CEFAS)), Lowestoft, U.K., collected in 1996 fish flesh samples of mixed species in the eastern Irish Sea. As the quantity of material obtained was relatively small and measured activities of some anthropogenic radionuclides were relatively high, the sample was mixed with fish powder from North Sea. The sample aliquots were distributed during 2000 for intercomparison of anthropogenic and natural radionuclides. Over 100 laboratories world-wide agreed to participate. Of these, 90 sent the results which could be used in the evaluation of this intercomparison exercise.

As the sample was collected in the Irish Sea, elevated levels of anthropogenic radionuclides (e.g. ^{90}Sr, ^{99}Tc, ^{129}I, ^{137}Cs, Pu isotopes) were expected due to discharges from the Sellafield reprocessing plant. Participants were informed that the expected activities for anthropogenic and natural radionuclides would be in the ranges (Bq/kg dry weight):

gamma emitters : 0.1 – 500

beta emitters : 0.1-1

transuranics : 0.01 – 0.3

This information was given to the participants with the sample.

This report describes the results obtained from 90 laboratories on anthropogenic and natural radionuclide determinations in Irish and North Seas fish.

2. SCOPE OF THE INTERCOMPARISON EXERCISE

This intercomparison exercise was organised with the aim of providing the participating laboratories with the possibility of testing the performance of their analytical methods on a fish sample with elevated radionuclide levels due to the contamination of fish species by the reprocessing nuclear plant.

The intercomparison material was designed for analysis of anthropogenic and natural radionuclides. Participating laboratories were requested to determine as many radionuclides as possible by gamma spectrometry and any possible transuranium radionuclides and other radionuclides requiring radiochemical separation and alpha or beta counting, as well as ICPMS, AMS and TIMS analyses.

It was expected that the sample, after successful certification, could be issued as a reference material for radionuclides in marine fish.

3. DESCRIPTION OF THE MATERIAL

About 350 kg of mixed fish species were collected in the eastern Irish Sea by the Fisheries Laboratory of the Ministry for Agriculture Fisheries and Foods (MAAF), (presently the Center for Environment, Fisheries and Aquaculture Science (CEFAS)), Lowestoft, U.K., in 1996. The skin was removed, the fish filleted and freeze-dried, and sent to IAEA-MEL for processing.

The sample, which was reduced by freeze-drying to about 69 kg, was then ground to powder, sieved through a 0.5 mm mesh and homogenized by mixing under nitrogen atmosphere. The sample was freeze-dried once more, ground and sieved at 250 μm to obtain a final amount of 10 kg. As the quantity of material obtained was relatively small and the measured activities of some anthropogenic radionuclides were relatively high, the sample was mixed with 100 kg of North Sea fish powder (a fraction below 250 μm) to get the required quantity of material. To ensure the homogenization of the final sample, both samples (fractions <250 μm) were mixed using the jet pulverizing technique in a nitrogen atmosphere.

The samples were then packed into brown glass bottles (100 g per bottle) under nitrogen gas, screwed with polyethylene caps and labeled with the code number IAEA-414.

The sealed glass bottles containing 100 g each of fish powder were sterilized as requested by ISO 2001 [1] at 10 kGy in an irradiation facility.

The average moisture content of the lyophilized sample after bottling, determined by drying several aliquots in an oven at 80 ºC to constant weight (1-2 days), was found to be approximately 3%. Since the moisture content can vary with the ambient humidity and temperature, it was recommended that the water content should be checked prior to analysis and that all results should be reported on a dry-weight basis.

4. HOMOGENEITY TESTS

The homogeneity of the sample was checked by measuring the activity of ^{40}K, ^{137}Cs, ^{210}Po, ^{235}U, ^{238}U, ^{238}Pu, $^{239+240}$Pu and ^{241}Am on 16-30 bottles taken at random. Gamma-spectrometric measurements were performed on 30 to 100 g of samples, ^{210}Po, ^{235}U, ^{238}U, ^{238}Pu, $^{239+240}$Pu and ^{241}Am were determined by alpha spectrometry on 0.1 to 20 g of samples. Results for some selected transuranic and natural radionuclides as ^{137}Cs, ^{210}Po, ^{238}U, ^{238}Pu, $^{239+240}$Pu and ^{241}Am and expressed as relative activities are shown in Annex I, Tables I&II. Homogeneity was determined using one-way analysis of variance. The coefficient of variation was below 10% for both gamma and alpha-spectrometrically determined radionuclides. The "between samples" variances showed no significant differences from the "within sample" variances for all radionuclides tested.

IAEA-414 intercomparison sample was used in a national intercomparison exercise organised by CIEMAT (Centro de Investigaciones Energeticas, Medioambientales y Tecnologicas, Madrid, Spain) for Spanish laboratories [2]. During the course of exercise we were notified about possible inhomogeneities in the sample claimed for several radionuclides. However, the Spanish report [2] did not confirm any existence of inhomogeneities in IAEA-414. Additional analyses carried out in IAEA-MEL during the certification procedure also did not find any inhomogeneities. Thus the material could be considered sufficiently homogeneous for the tested radionuclides at the weights used.

5. SAMPLE DISPATCH AND DATA RETURN

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

- Average weight of sample used for analysis
- Number of analyses
- Massic activity calculated in net values (i.e. corrected for blank, background etc.) and expressed in Bq kg^{-1} dry weight
- Estimate of the total uncertainty (counting and other uncertainties)
- Description of chemical procedure and counting equipment
- Reference standard solutions used
- Chemical recoveries, counting time, decay corrections

The reference date for reporting activities was set for 1 January 1997.

The samples were distributed to over 100 laboratories. The deadline for reporting data was set for 31 December 2000. As some participants expressed their intention to report later, the deadline was extended to 30 September 2001. A reminder was sent to late participants in order to obtain more data. Requests for more information about chemical procedures, counting systems, standards, units etc., were also sent to many participants who did not provide enough information on time.

A total of 90 sets of results were received from participants and included in the evaluation. The list of reported radionuclides is given in Table III.

The list of contributing laboratories can be found in Annex IV.

6. EVALUATION OF RESULTS

6.1. DATA TREATMENT

The list of reported radionuclides with the number of reporting laboratories for each radionuclide is given in Annex I, Table III. The number of reported "less than" values are shown in parentheses. The massic activities of anthropogenic and natural radionuclides were reported. Laboratory means were calculated when necessary from individual results and are given as weighted means with weighted uncertainties. All values have been rounded off to the most significant figure keeping in mind the necessity of maintaining the uniformity of presentation.

6.2. STATISTICAL EVALUATION

The principles and applications of the statistical programme used for the evaluation of data have been described in previous reports. Briefly, the data treatment consists of identifying and eliminating the outlying values, calculating the median and the confidence intervals. 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. Outliers are identified in different tables with an asterisk. Median values are calculated from all results passing the test. These values are considered to be the most reliable estimates of the true values.

The results for the most frequently measured radionuclides are found in Annex I, Tables IV to XIV and Figures 1 to 12. The activity ratios obtained for ^{238}Pu/$^{239+240}$Pu and for ^{241}Am/$^{239+240}$Pu are given in Table XV and Figures 13 and 14. The other less frequently measured atom ratios are presented in Table XVI. The less frequently measured radionuclides are presented in Table XVII. The recommended values and information values obtained after statistical treatment of data are summarised in Table XVIII.

Confidence intervals were taken from a non-parametric sample population. They represent a two-sided interval representing 95% confidence limits.

Following the IUPAC [3] and ISO [4] recommendations for assessment of laboratory performance, Z-score methodology has been used in the evaluation of intercomparison results. The Z-score is calculated according to the formula:

$$Z = (X_i - X_a) / S_b,$$

Where

X_i is the robust mean of the reported values of massic activity in the sample,
X_a is the assigned value (a mean value of accepted results),
S_b is the target standard deviation.

The performance of a laboratory is considered to be acceptable if the difference between the robust mean of the laboratory and the assigned value (in s_b units) is less than or equal to two. The analysis is regarded as being out of control when $|Z|>3$. The Z-score evaluation represents a simple method which gives participating laboratories a normalized performance score for bias.

The selection of the right target value depends on the objectives of the exercise. For radionuclide analysis, laboratories are required to have a relative bias below 20% (s_b <10%). The uncertainty of the assigned value should be taken into account using the formula:

$$Z = (X_i - X_a) / \sqrt{s_b^2 + s_{tu}^2}$$

Where

s_{tu} is the uncertainty of the assigned value [5].

Z-score graphs are given in Annex III, from Figure 15 to Figure 26.

6.3. EXPLANATION OF TABLES

6.3.1. Laboratory code

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

6.3.2. Method code

The analytical techniques employed by the participants are:

Alpha spectrometry

Code	*Method*
A	Not specified or not enough information.
A2	Treatment, evaporation/precipitation, ion exchange, electro-deposition.
A10	Treatment, ion exchange, electro-deposition.
A11	Ca oxalate, TRU resins, SCN- anion exchange, electro-deposition.
A12	Double columns: UTEVA + TRU resins, electro-deposition.
A13	$Fe(OH)_3$ co-precipitation, electro-deposition on silver, nickel or stainless steel discs, alpha-counting.
A14	Sulphate/iron hydroxide precipitations, Fe extraction with di-isopropyl ether, anion exchange in HCl, electro-deposition.
A15	Treatment, ion exchange in xylene, NdF_3 co-precipitation, electro-deposition.
A17	Pu separation and purification with anionite (Av-17, Russian reagent), electro-deposition.
A18	Treatment, extraction by TBP, anion-exchange resin, electro-deposition.
A19	Treatment, TOPO extraction in cyclo-hexane, LaF_3 co-precipitation, anion exchange, electro-deposition.
A22	Treatment, anion exchange, UTEVA resins, electro-deposition.
A23	Leaching, double columns, UTEVA + TRU resins, electro-deposition.
A25	TRU resins, LaF3 co-precipitation.
A26	Treatment, extraction, cation exchange, LaF3 precipitation.
A27	Leaching, UTEVA+TRU resins, NdF3 micro-co-precipitation.
A28	Digestion, lead and barium precipitation.
A29	Ba-$RaSO_4$ precipitation, TTA extraction in benzene.
A30	Wet oxidation, filtration and dilution to 1M, repeated Rn emanation strips into quartz glass cells.

Beta counting

Code	*Method*
B	Not specified or not enough information.
B1	Precipitation (oxalate), fuming nitric acid separation, $PbCr_2O_4$ ppt.
B5	Precipitation (hydroxide, oxalate, carbonate, sulphate), separation with conc. or fuming nitric acid, scavenging of Ra and Fe, 2 weeks ingrowth period, precipitation (hydroxide, oxalate, carbonate), beta counting of ^{90}Y (as Y oxalate, Y_2O_3,...) or liquid scintillation counting.
B7	Precipitation (oxalate, hydroxide), scavenging, beta counting of Y oxalate.
B11	Precipitation (oxalate), cationic resin with DCTA, sodium malonate, atomic absorption.
B14	Sr separation in presence of EDTA, $SrSO_4$ precipitation.
B15	Oxalate precipitation, Sr crown ether resin, Sr carbonate precipitation.
B16	Digestion in HCl, ion exchange separation, Cs chloroplatinate (Cs_2PtCl_6).
B17	$BaCrO_4$ co-precipitation, iron scavenging, Y oxalate.
B18	$Sr(NO_3)_2$ precipitation, $PbBaCrO_4$ precipitation, iron scavenging, Y oxalate.
B19	EDTA precipitation, ion-exchange chromatography, carbonate precipitation.
B20	HCl leaching, Eichrom Sr-spec columns, LSC counting.
B21	(Carbonate, oxalate and nitrate) co-precipitation, cation exchange, resin columns, iron hydroxide co-precipitation.
B22	HNO_3, HCl leaching, anion-exchange or separation on Sr resin, electro-deposition, gas flow proportional counting.
B23	HCl, H_2SO_4 dissolved, extraction in tri-n-butyl phosphate, electro-deposition, gas flow proportional counting.
B24	Lead sulphate precipitation, gravimetric analysis of lead.
B25	$PbBaCrO_4$ precipitation, LaF_3 precipitation.
B26	Treatment, ion exchange.

Gamma spectrometry

Code	*Method*
G	Not specified.
G1	High resolution Ge spectrometry.
G2	Treatment, purification, AMP, high resolution Ge spectrometry.

Mass spectrometry

Code	*Method*
M1	Treatment, ion exchange, electro-deposition, leaching, ICPMS.
M2	Double columns: UTEVA + TRU resins, ICPMS.
M3	Treatment, ICPMS.
M5	Extraction, purification using TBP/CCl_4 solvent, ICPMS.
M7	Digestion, iron oxide matrix, evaporation and baking to Fe_2O_3, mixing with Al powder, AMS.
M8	Treatment, AMS.

Other methods

Code	*Method*
N	No information.
NAA	Neutron Activation Analysis.
DNC	Delayed neutron counting.
F	Treatment, anion-exchange, laser fluometer.

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 has been assumed to be one.

6.3.4. Massic activity

The activity corresponds to the weighted mean computed from all the individual results obtained from the participants with the corresponding standard deviation or weighted uncertainty.

6.4. EXPLANATION OF FIGURES

The figures (Figs. 1 to 12) in Annex II present the tabulated 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 means (vertical lines).

Figs. 13 and 14 show $^{238}Pu/^{239+240}Pu$ and $^{241}Am/^{239+240}Pu$ activity ratios, respectively.

The performance of laboratories in terms of accuracy has been expressed by Z-scores which were calculated for each radionuclide. Performance is considered satisfactory if the Z-score is equal to or less than 2. A Z-score from 2 to 3 indicates that the results are of questionable quality. If $|Z|>3$, the analysis is out of accepted range. Figs. 15 to 26 in Annex III present the Z-scores for only accepted value. The presented distributions of Z-scores are symmetric which indicates that the performances of the laboratories were satisfactory.

6.5. CRITERIA FOR CERTIFICATION

Following ISO Guide 35 [1], a preliminary certification of IAEA-414 was carried out using criteria as strict as in the previous intercomparison exercise IAEA-384 [6]. The certification will be completed when all data from expert laboratories participating in the certification procedure are available. For data sets comprising 5 or more accepted laboratory means, median values and confidence intervals were calculated as estimations of true massic activities. The median values of the data within the confidence interval, were considered as the **recommended values** when:

1. At least 5 laboratory means were available, calculated from at least 3 different laboratories.
2. The relative uncertainty of the median did not exceed ± 5% for activities higher than 100 Bq kg^{-1}, ± 10% for activities from 1-100 Bq kg^{-1} and ± 20% for activities lower than 1 Bq kg^{-1}.

An activity value was classified as an **information value** when at least 5 laboratory means calculated from the results of at least 2 different laboratories were available.

7. RESULTS AND DISCUSSION

7.1. ANTHROPOGENIC RADIONUCLIDES

Results of analysis of ^{90}Sr, ^{137}Cs, ^{238}Pu, $^{239+240}Pu$, ^{241}Am data in IAEA-414 fish samples as supplied by participants are presented in Annex I, Tables IV to VII and in Annex II, Figures 1 to 5. Laboratory averages were calculated when necessary from individual results and are given as weighted means with corresponding standard deviations. The evaluation of the full data set consisted of identifying and eliminating outlying values, then calculating the median and the confidence intervals (95% significance level). The performance of laboratories expressed by Z-scores is presented in Annex III, Figures 15 to 19.

7.1.1. ^{90}Sr

Data were reported from 28 laboratories (Table IV, Fig. 1) in which sixteen laboratories reported only detection limits. Four laboratories submitted outlying results. The rest of the data show good homogeneity, all data fall less than two standard deviations from the distribution mean. Z-score values are below 1.5 showing good performances by the laboratories (Fig.15). The median given as the information value is 0.28 Bq kg^{-1} dry weight (95% confidence interval is (0.10 – 0.54) Bq kg^{-1}dw).

7.1.2. ^{137}Cs

81 laboratories reported ^{137}Cs results (Table V, Fig. 2). The laboratories mainly used direct gamma spectrometry for ^{137}Cs analysis, one laboratory reported detection limit only. Nineteen results were identified as outliers. The rest of the data is homogenous within two standard deviations of the distribution mean. Z-score values are below 2.2, showing good performances by the laboratories (Fig. 16). The median, given as the recommended value, is 5.14 Bq kg^{-1} dw (95% confidence interval is (5.00 – 5.27) Bq kg^{-1} dw).

7.1.3. Plutonium isotopes

The majority of participants used a rather conventional method based on sample treatment, ion-exchange separation followed by electro-deposition and alpha-spectrometry. Some laboratories combined ion-exchange separation with liquid-liquid extraction, or used only liquid-liquid extraction. Resins (a single TRU column or double UTEVA + TRU columns) for separation and subsequent electro-deposition and alpha-spectrometry (^{238}Pu) or for direct ICPMS, AMS analysis (^{239}Pu, ^{240}Pu, ^{241}Pu, ^{242}Pu) have also been used. The samples for mass spectrometry were either leached from stainless steel discs after alpha-spectrometry measurements, or analysed directly by ICPMS and/or AMS. Generally, good agreement was found between alpha-spectrometry and mass spectrometry results, and between ICPMS and AMS.

^{238}Pu

Thirty-two data sets were reported (Table VI, Fig. 3). Ten results did not pass the outlier test. Four other laboratories reported detection limits only. Some laboratories reported their results with rather high analytical uncertainties. The rest of the data is homogeneous, within two standard deviations of the distribution mean. Z-score values are below 2.0 showing reasonable performances by the laboratories (Fig. 17). The median, given as the recommended value, is 0.023 Bq kg^{-1} dw (95% confidence interval is (0.022 – 0.025) Bq kg^{-1} dw).

$^{239+240}$Pu

Forty-two data sets (combined alpha-spectrometry, ICPMS and AMS results) were reported (Table VI, Fig. 4). Nine results were identified as outliers. The rest of the data is homogeneous, within two standard deviations of the distribution mean. Z-score values are below 2.0 showing good performances by the laboratories (Fig. 18). The median, given as the recommended value is 0.12 Bq kg^{-1} dw (95% confidence interval is (0.113 – 0.125) Bq kg^{-1} dw).

7.1.4. ^{241}Am

Thirty-four results (13 obtained by gamma-spectrometry and 21 by alpha-spectrometry) were reported in which twenty-two (8 for gamma results and 14 for alpha results) were available for statistical treatment (Table VII, Fig. 5). Six laboratories reported detection limits only. Five alpha-spectrometry results and one gamma-spectrometry result did not pass the outlier test. The rest fall less than 2.2 standard deviations from the distribution mean. The data evaluation was separately done for gamma-spectrometry results and alpha-spectrometry results, but only alpha-spectrometry results were used for certification process due to too high analytical uncertainties in gamma-spectrometry. The Z-score values are below 2.2 (Fig. 19). The median, given as the recommended value, is 0.196 Bq kg^{-1} dw (95% confidence interval is (0.189 – 0.199) Bq kg^{-1} dw).

7.2. NATURAL RADIONUCLIDES

Results of analysis of ^{40}K, ^{232}Th, ^{234}U, ^{235}U, ^{238}U, ^{226}Ra, ^{210}Pb(^{210}Po) in IAEA-414 fish samples as supplied by participants are presented in Annex I, Tables VIII to XIV and in Annex II, Figures 6 to 12. Laboratory averages were calculated when necessary from individual results and are given as weighted means with corresponding standard deviations. The evaluation of the full data set consisted of identifying and eliminating outlying values, then calculating the median and the confidence intervals

(95% significance level). The performance of laboratories expressed by Z-scores is presented in Annex III, Figures 20 to 26.

7.2.1. ^{40}K

Data were reported from 73 laboratories (Table VIII, Fig. 6). One laboratory reported a detection limit only, and five results did not pass the outlier test. These laboratories might have problems with the correct estimation of their background under the ^{40}K photopeak. The remaining data show reasonable homogeneity. Z-score values of accepted data are below 2.2, showing reasonable performances by the laboratories (Fig. 20). The median, given as the information value, is 480 Bq kg^{-1} dw (95% confidence interval is (461 – 498) Bq kg^{-1} dw).

7.2.2. ^{232}Th

From 11 reported laboratory means, only 6 results were accepted in the evaluation, 5 results did not pass the outlier test (Table IX, Fig. 7). Three laboratories reported detection limits only. The data were homogeneous within two standard deviations of the distribution mean. Both non-destructive gamma-spectrometry and alpha-spectrometry techniques were used, but only the results obtained by alpha-spectrometry technique have shown good results. The Z-score values (Fig. 21) are below 1.6. The median, given as the information value is 0.029 Bq kg^{-1} dw (95% confidence interval is (0.025 – 0.031) Bq kg^{-1}dw).

7.2.3. Uranium isotopes

^{234}U

From 23 reported laboratory means, 7 results did not pass the outlier test (Table X, Fig. 8). Mostly total dissolution followed by alpha-spectrometry, with the exception of 3 results obtained by ICPMS, was used in the analysis. The Z-score values were below 2.3 showing good performances by the laboratories (Fig. 22). The median, given as the information value is 1.22 Bq kg^{-1} dw (95% confidence interval is (1.14 – 1.27) Bq kg^{-1}dw).

^{235}U

Eighteen results were reported (Table XI, Fig. 9) using alpha-spectrometry and ICPMS with chemical purification and enrichment of uranium beforehand. One result was obtained by delayed neutron measurement. Almost all results obtained by gamma spectrometry were reported as detection limits only or as outlying values. The difficulty dealing with gamma-spectrometry measurements was principally due to interference of ^{226}Ra. In 17 results obtained by alpha spectrometry, only one did not pass the outlier test. Two of three results obtained by using ICPMS technique fell in the confidence interval range (95%). The Z-score values (Fig. 23) are below 2. The median given as the information value is 0.05 Bq kg^{-1} dw (95% confidence interval is (0.045 – 0.058) Bq kg^{-1}dw).

^{238}U

Twenty-seven results were reported (Table XII, Fig. 10) using gamma-spectrometry, alpha-spectrometry and ICPMS. Most results obtained by gamma-spectrometry were reported as detection limits only or as outlying values. Twenty laboratories reported results obtained by alpha-spectrometry (14 were accepted). Two of three results obtained by ICPMS technique were in confidence interval range (95%). The Z-score values (Fig. 24) are below 1.8, showing very good performances by the laboratories.

The median gives the information value is 1.112 Bq kg^{-1} dw (95% confidence interval is (1.065 – 1.170) Bq kg^{-1}dw).

7.2.4. ^{226}Ra

Eighteen laboratory means (13 accepted) mostly obtained by gamma-spectrometry were reported (Table XIII, Fig. 11). Several laboratories overestimated the ^{226}Ra concentrations due to an improper calibration, a background estimation and possible interferences in gamma-spectra at 186 keV energy from ^{235}U. Ten laboratories, which reported detection limits only, used gamma-spectrometry techniques. The Z-score values of accepted data (Fig. 25) are below 1.7. The median given as the information value is 1.40 Bq kg^{-1} dw (95% confidence interval is (0.59 – 1.76) Bq kg^{-1}dw).

7.2.5. ^{210}Pb(^{210}Po)

Twenty-seven laboratory means were reported for ^{210}Pb and ^{210}Po (Table XIV, Fig. 12). Mostly gamma-spectrometry and alpha-spectrometry (^{210}Po in-growth) were used for ^{210}Pb. Eight laboratories reported gamma-spectrometry results as outlying values or detection limits only. This showed evidently the difficulty dealing with low energy gamma-rays due to self-absorption problems and the energy and efficiency calibration. The Z-score values (Fig. 26) for ^{210}Pb&^{210}Po are below 1.9. The symmetry of Z-scores showed the good performance of laboratories. The median given as the information values is 2.22 Bq kg^{-1} dw (95% confidence interval (1.55 – 2.60) Bq kg^{-1}dw).

Taking into account the time elapsed between collection and analysis of the samples, it seems reasonable to assume that ^{210}Pb and ^{210}Po are in secular equilibrium.

7.3. ISOTOPE RATIOS

^{238}Pu/$^{239+240}$Pu activity ratios are shown in Table XV and Fig. 13. The weighted mean value is 0.193 ± 0.006 (higher than a global fallout value 0.028) documenting that plutonium found in IAEA-414 sample is significantly contaminated by nuclear discharges from the Sellafield reprocessing plant.

^{241}Am/$^{239+240}$Pu activity ratios are shown in Table XV and Fig. 14. The weighted mean value is 1.59 ± 0.05, much higher than the global fallout ratio (0.37) due to the enhanced concentration of ^{241}Am in the fish sample.

^{240}Pu/^{239}Pu atom ratios from 5 different measurements made by ICPMS and AMS are shown in Table XVI. The weighted mean value is 0.21 ± 0.01, that does not differ significantly from the global fallout ratio (0.18).

^{234}U/^{238}U and ^{235}U/^{238}U atom ratios are given in Table XVI. One result obtained by ICPMS gave a value of (5.8 ± 0.3) 10^{-5} for ^{234}U/^{238}U and (7.1 ± 0.2) 10^{-3} for

$^{235}U/^{238}U$, respectively. These ratios are consistent with the values for natural uranium atom ratios (5.64 10^{-5} for $^{234}U/^{238}U$ and 7.26 10^{-3} for $^{235}U/^{238}U$).

7.4. LESS FREQUENTLY REPORTED RADIONUCLIDES

Results for the less frequently reported radionuclides (^{60}Co, ^{99}Tc, ^{129}I, $^{228}Ra(^{228}Ac)$, ^{228}Th, ^{230}Th,^{237}Np, ^{239}Pu, ^{240}Pu, ^{241}Pu) in IAEA-414 are given in Table XVII.

^{60}Co

Eight laboratories reported results for ^{60}Co, most of them are lower limit detection or reported with very high statistic error, so we prefer to note them as LLD.

^{99}Tc

Using beta-counting with chemical pre-treatment beforehand, five laboratories reported ^{99}Tc results. Four values were rather homogenous (from 5.5 to 7.9 Bq kg^{-1} dw), one laboratory submitted an outlying results.

^{129}I

Three laboratories reported ^{129}I values, determined by ICPMS in the range from 0.008 to 0.012 Bq kg^{-1} dw.

^{228}Ra(^{228}Ac)

Two laboratories reported results (9.2 ± 1.8) and (2.0 ± 0.3) Bq kg^{-1} dw for ^{228}Ra and one laboratory reported results (2.0 ± 0.3) Bq kg^{-1} dw for ^{228}Ac, respectively. All other laboratories reported detection limits only

^{228}Th

Five laboratories reported consistent ^{228}Th results obtained by alpha-spectrometry (from 0.17 to 0.22 Bq kg^{-1}dw).

^{230}Th

Seven laboratories reported ^{230}Th results with two different ranges, one of low values (0.037 to 0.06 Bq kg^{-1}dw), the other in higher values (0.5 to 1.95 Bq kg^{-1}dw). Two other laboratories reported LLD (below 0.04 Bq.kg^{-1} dw).

^{237}Np

Two results obtained by alpha and gamma-spectrometry, ranging from 0.32 to 1.5 Bq kg^{-1} dw were reported. Two other laboratories reported detection limits (below 0.4 Bq.kg^{-1} dw)

^{239}Pu&^{240}Pu

Four laboratories reported ^{239}Pu and ^{240}Pu results obtained by ICPMS. The results obtained by ICPMS method are rather homogenous (0.064 to 0.082 Bq kg^{-1} dw) for ^{239}Pu and (0.047 to 0.065 Bq kg^{-1} dw) for ^{240}Pu, respectively

^{241}Pu

Four results were reported (from 1.33 to 4.5 Bq kg^{-1} dw) obtained by beta-spectrometry, alpha-spectrometry of the ^{241}Am daughter, or ICPMS analysis. 9 individual results obtained by ICPMS are on the higher side of the data set (in comparison with the results obtained by other methods in the same laboratory).

8. CONCLUSIONS

In this world-wide intercomparison exercise, using mixed fish from the Irish Sea and the North Sea (IAEA-414), data on natural and anthropogenic radionuclide concentration measurements have been reported from 90 laboratories world-wide. The median activities for the sets of accepted values were chosen as the most reliable estimates of the true values and were given as recommended values and information values.

A summary of the recommended values and information values with confidence intervals for the most frequently reported anthropogenic and natural radionuclides is given in Table XVIII.

The certification procedure for IAEA-414 will be completed when all analyses from expert laboratories are evaluated. It is expected that IAEA-414 will be released as an IAEA Reference Material for radionuclides in the marine environment.

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ANNEX I

Data report - tables

TABLE I. HOMOGENEITY TESTS(*) FOR TRANSURANICS IN IAEA-414.

No. of sample	^{241}Am	^{238}Pu	$^{239+240}Pu$
1	0.92	0.84	0.90
2	0.93	0.92	0.95
3	0.94	0.93	0.95
4	0.96	0.97	0.96
5	0.98	0.98	0.96
6	0.99	0.98	0.97
7	1.00	1.00	0.99
8	1.02	1.04	1.02
9	1.03	1.06	1.05
10	1.08	1.10	1.09
11	1.15	1.16	1.17
12	1.04	1.04	0.96
13	1.00	0.82	0.99
14	1.06	1.15	1.07
15	1.04	1.05	0.95
16	1.07	1.05	1.11
17	0.92	0.94	0.93
18	0.88	0.94	0.99
19	1.05	0.90	0.98
20	0.99	0.94	0.91
21	0.97	0.90	0.97
22	1.00	1.17	1.00
23	1.01	1.20	1.07
24	0.98	0.90	1.07
Minimum	0.88	0.82	0.90
Maximum	1.15	1.20	1.17
Mean	1.00	1.00	1.00
Median	1.00	0.98	0.99
Standard dev	0.06	0.1	0.07
Coef. var. (%)	6	10	7

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

TABLE II. HOMOGENEITY TESTS(*) FOR SOME ANTHROPOGENIC AND NATURAL RADIONUCLIDES IN IAEA-414.

No. of sample	^{137}Cs	^{210}Po		^{238}U
1	0.70	0.66	0.99	0.89
2	0.90	0.81	1.01	0.90
3	0.92	0.81	1.05	0.90
4	0.92	0.87	1.06	0.91
5	0.99	0.87	1.06	0.92
6	1.00	0.89	1.08	0.92
7	1.01	0.89	1.08	0.93
8	1.02	0.91	1.09	0.99
9	1.04	0.91	1.14	0.99
10	1.04	0.96	1.17	0.99
11	1.06	0.96	1.18	1.01
12	1.06	0.97	1.20	1.01
13	1.07	0.97	1.23	1.03
14	1.08	0.98	1.25	1.03
15	1.09	0.98		1.05
16	1.09	0.98		1.08
17				1.22
18				1.23
Minimum	0.70	0.66		0.89
Maximum	1.09	1.25		1.23
Mean	1.00	1.00		1.00
Median	1.03	0.98		0.99
Standard dev	0.10	0.13		0.098
Coef. var. (%)	10	13		10

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

TABLE III. RADIONUCLIDES REPORTED FOR IAEA-414.

Radionuclides	Number of all results	Radionuclides	Number of all results
^{40}K	228(3*)	^{234}U	65
^{60}Co	(35)	^{235}U	89(18)
^{90}Sr	55(30)	^{238}U	92(19)
^{99}Tc	8	^{237}Np	9(4)
^{129}I	26(9)	^{238}Pu	83(4)
^{137}Cs	240(1)	^{239}Pu	27
$^{210}Pb(^{210}Po)$	107(17)	^{240}Pu	27
^{226}Ra	76(19)	$^{239+240}Pu$	124(1)
$^{228}Ra(^{228}Ac)$	39(36)	^{241}Pu	24
^{228}Th	37(13)	^{241}Am	94(10)
^{230}Th	24(3)	^{243}Am	2(2)
^{232}Th	32(5)	^{243}Cm	(1)
^{232}U	2	^{244}Cm	(1)

*) "Less than" values are shown in parenthesis.

TABLE IV. RESULTS[#] FOR ^{90}Sr IN IAEA-414.
(Reference date: 1 January 1997, unit: Bq kg^{-1} dry weight)

Lab Code	Method Code	No. of Results	Weight (g)	^{90}Sr
1	B7	2	10	14.8 ± 3.7*
2	B17	2	30; 45	<0.5
14	B17	2	48	<0.4
15	B17	2	10	0.14 ± 0.01
19	B17	2	20	2.3 ± 1.0*
21	B18	2	29.30; 29.82	<0.4
23	B17	2	44.03; 46.2	<0.1
26	B15	1	40	<0.5
27	B11	2	30	<0.25
29	B17	2	21.93	<0.3
40	B18	2	10.07	<2.6
41	B17	2	28.42	6.2 ± 1.1*
42	B19	2	19; 20	< 0.15
53	B14	2	24	<0.12
54	B17	2	6	1.25 ± 0.14*
64	B20	2	10	<0.4
68	B5	2	41.2; 43.5	<0.3
71A	B7	4	50	0.159 ± 0.014
71B	B7	1	100	0.08 ± 0.04
74	B21	2	30	0.54 ± 0.22
75	B	2	75.1	0.44 ± 0.07
78	B15	2	30.45; 37.45	<0.2
79	B	2	35	0.103 ± 0.025
82	B7	2	46	<0.3
83	B21	2	40	<0.4
85	B7	1	24.662	<0.6
87	B	2	100	0.6 ± 0.2
88	B5	2	20; 40	0.40 ± 0.17

Number of reported lab. Means	12
Number of accepted lab. Means	8
Median	0.28
Confidence interval ($\alpha = 0.05$)	0.10 – 0.54

Uncertainties at 2σ
* Results rejected by the test for outliers

TABLE V. RESULTS# FOR ^{137}Cs IN IAEA-414.
(Reference date: 1 January 1997, unit: Bq kg^{-1} dry weight)

Lab Code	Method code	No. of Results	Weight (g)	^{137}Cs
1	G1	3	20	7.4 ± 1.9*
2	G1	2	30; 45	4.9 ± 0.5
3	G1	2	62.5	5.5 ± 0.5
4	G1	2	28.39	4.33 ± 0.43
5	G1	2	246.5	5.45 ± 0.21
6	G1	5	90.7	5.22 ± 0.31
7	G1	2	32.46	5.7 ± 1.5
8	G1	2	45	5.14 ± 0.44
9	G1	1	68.85	4.8 ± 0.2
10	G1	2	69.8	6.8 ± 1.0*
11	G1	2	28.2	5.7 ± 0.6
12	G1	2	25.7; 69.1	5.6 ± 1.0
13	G1	2	38; 57	5.05 ± 0.42
14	G1	4	96.71	4.95 ± 0.14
15	G1	2	10	225 ± 16*
16	G1	2	96.7; 99.3	5.17 ± 0.26
17	G1	2	30.67	6.0 ± 0.9
19	G1	1	20	5.4 ± 1.4
21	G1	1	97.635	<0.5
22	G1	2	96.81	5.6 ± 0.4
23	G1	3	44-76.4	6.5 ± 0.8*
24	G1	2	22.24; 22.45	5.3 ± 0.6
25	G1	2	31.3145	5.34 ± 0.40
26	G1	3	56.3	5.4 ± 0.7
27	G1	2	20-24	7.9 ± 1.3*
28	G1	2	14.55; 15.89	4.9 ± 0.4
29	G1	2	44.59	4.6 ± 0.5
30	G1	4	58.5	5.5 ± 0.4
31	G1	1	2.79	16.3 ± 2.4*
32	G1	1	98	5.4 ± 1.3
33	G1	1	64.2	2.4 ± 0.2*
34	G1	4	93.4	5.2 ± 0.3
35	G1	2	14.92; 14.95	7.6*
36	G1	4	22.66	4.85 ± 0.42
37	G1	2	83.386	5.00 ± 0.17
38	G1	4	38	4.4 ± 0.7
39	G1	2	25.0	5.0 ± 1.6
41	G1	2	22.24	5.49 ± 0.43
42	G1	2	29.7	6.2 ± 1.0
43	G1	2	32.4; 33.7	7.2 ± 1.4*
44	G1	2	88.44	4.9 ± 0.7
45	G1	13	41.42-43.38	4.7 ± 0.7
46	G1	2	109	6.16 ± 0.29
47	G1	2	2.334	6.2 ± 0.1
48	G1	3	23.78-24.9	6.03 ± 0.32
49	G1	2	42.85; 44.26	5.0 ± 0.7
51	G1	2	97.98; 98.11	4.47 ± 0.36
52	G1	5	71.45	5.21 ± 0.37
53	G1	9	62.15-62.18	5.04 ± 0.23
55	G1	2	15.4; 25.5	5.80 ± 0.32
56	G1	2	27.6	6.7 ± 0.6*
57	G1	2	28; 30	4.22 ± 0.47

TABLE V (cont'd). RESULTS# FOR ^{137}Cs IN IAEA-414.
(Reference date: 1 Janσσuary 1997, unit: Bq kg^{-1} dry weight)

Lab code	Method code	No. of results	Weight (g)	^{137}Cs		
58	G1	7	97.56	5.58	±	0.38
60	G1	4	66.74	5.1	±	1.6
61	G1	3	34.72-36.8	4.83	±	0.47
62	G1	3	35.24	7.5	±	1.2*
63	G1	2	27.88; 31.39	3.95	±	1.33*
64	G1	2	27.13; 38.41	6.4	±	0.6*
65	G1	3	24.58	5.14	±	0.41
66	G1	2	41.4; 102.4	5.0	±	1.0
67A	G1	2	14.3	3.9	±	1.2*
67B	G1	2	14.3	2.7	±	1.2*
68	G1	2	42.2; 43.5	5.8	±	0.8
69	G1	2	100	3.28	±	0.42*
70	G1	1	37.5	5.1	±	0.7
71A	G1	16	30	5.07	±	0.13
71B	G2	2	10	4.47	±	0.50
72	G1	2	25	5.26	±	0.40
73	G1	3	14.3	4.3	±	1.2
74A	G1	3	61.31-68.23	4.9	±	0.5
74B	B16	2	30	5.0	±	0.8
75	G1	4	32.05-33.57	6.04	±	0.18
76	G1	5	37-93	6.1	±	0.5
77	G1	16	96.4-96.8	5.21	±	0.12
78	G1	2	16.9; 72.1	4.75	±	0.21
79	G1	4	11-95	5.27	±	0.43
81	G1	2	50	4.4	±	0.9
82	G1	2	102	19.9	±	2.7*
83	G1	1	150.6	6.5	±	0.2*
85	G1	2	70.6	5.1	±	0.8
87	G1	3	100	3.32	±	0.33*

Number of reported lab. Means	80
Number of accepted lab. Means	61
Median	5.14
Confidence interval (α = 0.05)	5.00– 5.27

Uncertainties at 2σ
* Results rejected by the test for outliers

TABLE VI. RESULTS[#] FOR ^{238}Pu AND $^{239+240}$Pu IN IAEA-414.
(Reference date: 1 January 1997; unit : Bq kg^{-1} dry weight)

Lab.	Method Code	No. of results	Weight (g)	^{238}Pu	$^{239+240}$Pu
1	A25	2	10		0.54 ± 0.22*
2	A2	1	30	0.07 ± 0.01*	0.29 ± 0.02*
3	A10	2	10; 10.3		0.144 ± 0.013
9	A	1	10	<0.04	0.134 ± 0.024
9	M3	1			0.121 ± 0.035
15	A2	2	10	0.027	
17	A2	2	10; 20	0.032 ± 0.008*	0.139 ± 0.018
20	A2	6	-	0.021 ± 0.004	0.113 ± 0.008
21	A2	2	29; 30	0.025 ± 0.014	0.39 ± 0.06*
23	A2	2	44; 46		0.119 ± 0.033
25	A2	4	3-3.139		0.108 ± 0.025
26	A23	1	10	<0.05	<0.05
27	A23	2	15	0.012 ± 0.002*	0.11 ± 0.01
29	A2	2	21.93	0.023 ± 0.011	0.125 ± 0.026
30	A	3	11.5-11.7		0.115 ± 0.015
31	A15	2	26.4; 29	0.54 ± 0.30*	0.154 ± 0.039*
31	A15	1	29	<0.25	
40	A2	3	3		0.20 ± 0.02*
42	A2	2	22		0.11 ± 0.02
49	A12	2	42; 44	0.02 ± 0.01	0.114 ± 0.019
49	A12	1	42.79	<0.03	
50	A11	2	4.8; 4.86	0.066 ± 0.016*	0.07 ± 0.02*
53	A15	2	49; 53	0.021 ± 0.004	0.105 ± 0.010
55	A2	2	10; 20		0.103 ± 0.007
68	A2	2	42; 43	0.028 ± 0.003	0.143 ± 0.008
70	A2	2	15	0.11 ± 0.03*	0.17 ± 0.02*
71A	A2	13	20	0.022 ± 0.001	0.122 ± 0.004
71B	M1	14	10		0.111 ± 0.003
71C	M7	3	10		0.146 ± 0.015
71D	A22	5	10	0.023 ± 0.001	0.115 ± 0.003
72	A13	3	50	0.023 ± 0.002	0.123 ± 0.006
73	A	2	2.7	0.05 ± 0.03*	0.08 ± 0.03*
74	A2	2	20	0.023 ± 0.008	0.145 ± 0.019
75	A	2	14	0.027 ± 0.004	0.134 ± 0.009
76	A2	3	50	0.025 ± 0.007	0.107 ± 0.012
77	A19	3	32-33	0.022 ± 0.002	0.121 ± 0.005
78	A2	2	30; 37	0.025 ± 0.002	0.108 ± 0.005
79	A	4	10	0.022 ± 0.002	0.125 ± 0.013
80	A2	3	25	0.046 ± 0.003*	0.125 ± 0.008
83	A2	2	40	0.03 ± 0.003*	0.120 ± 0.009
84A	A2	3	5	0.04 ± 0.01*	0.12 ± 0.02
84B	M3	2	10		0.13 ± 0.01
85	A2	1	24.662	0.022 ± 0.011	0.136 ± 0.022
88	A2	2	20; 40	0.025 ± 0.007	0.16 ± 0.02*
90	M3	5	10		0.116 ± 0.022

Number of reported lab. Means	28	41
Number of accepted lab. Means	18	32
Median	0.023	0.120
Confidence interval (α = 0.05)	0.022 – 0.025	0.113 – 0.125

Uncertainties at 2σ; * Results rejected by the test for outliers

TABLE VII. RESULTS# FOR ^{241}Am IN IAEA-414.
(Reference date: 1 January 1997; unit : Bq kg^{-1} dry weight)

Lab Code	Method code	No. of Results	Weight (g)	^{241}Am (gamma)	^{241}Am (alpha)
1	A25	2	10		<0.1
5	G1	2	246.5	0.26 ± 0.03	
7	G1	2	32.46	<1	
9	G1	1	68.85	0.28 ± 0.21	
12	G1	2	25.7; 69.1	0.6 ± 0.3*	
15	A2	2	10		0.02*
16	G1	2	96.7; 99.3	0.29 ± 0.18	
20	A2	2	-		0.194 ± 0.022
22	G1	2	96.81	0.25 ± 0.08	
26	A23	1	10		<0.5
27	A23	2	15		0.196 ± 0.007
29	A15	2	21.93		0.19 ± 0.02
31	A27	1	26.4		0.57 ± 0.17*
34	G1	3	93.4	0.24 ± 0.05	
37	G1	2	83.386	0.22 ± 0.08	
44	G1	2	88.4	<0.6	
49	A12	2	42; 44		0.16 ± 0.03
50	A11	2	4.8; 4.86		0.062 ± 0.013*
53	A15	2	49; 53		0.20 ± 0.03
62	G1	3	35.24		
68	A2	2	42; 43	<2	0.233 ± 0.013
70	G1	1	37.5	<2	
71A	A2	13	20		0.20 ± 0.01
71D	A22	5	10		0.189 ± 0.002
71C	G1	11	30	0.25 ± 0.01	
72	A13	3	50		0.197 ± 0.012
74	A2	2	20		0.185 ± 0.012
75	G1	4	32; 33	0.38 ± 0.13	
76	A2	2	50		0.180 ± 0.014
77	A19	3	32; 33		0.194 ± 0.008
78	A2	1	37.45		0.30 ± 0.03*
79	A	4	10		0.20 ± 0.02
83	A2	3	20-40		0.20 ± 0.01
85	A2	1	24.662		0.39 ± 0.06*
Number of reported lab. Means				9	19
Number of accepted lab. Means				8	14
Median				0.26	0.196
Confidence interval (α = 0.05)				0.24 – 0.29	0.189 – 0.199

Uncertainties at 2σ
* Results rejected by the test for outliers

TABLE VIII. RESULTS# FOR ^{40}K IN IAEA-414.
(Reference date: 1 January 1997, unit: Bq kg^{-1} dry weight)

Lab Code	Method code	No. of Results	Weight (g)	^{40}K
1	G1	3	20	<115
2	G1	2	30; 45	580 ± 100
3	G1	2	62.5	507 ± 20
4	G1	2	28.39	509 ± 14
5	G1	2	246.5	508 ± 20
6	G1	5	90.7	427 ± 4
7	G1	2	32.46	480 ± 50
8	G1	2	45	494 ± 24
9	G1	1	68.85	398 ± 18
10	G1	2	69.8	454 ± 20
11	G1	2	28.2	463 ± 20
12	G1	2	25.7; 69.1	514 ± 24
13	G1	2	38; 57	453 ± 13
14	G1	4	96.71	558 ± 4
16	G1	2	96.7; 99.3	486 ± 21
17	G1	2	30.67	537 ± 38
19	G1	2	15.382; 16.735	395 ± 100
21	G1	1	97.635	326 ± 25*
22	G1	2	96.81	527 ± 26
23	G1	3	44-76.4	470 ± 30
24	G1	2	22.24; 22.45	430 ± 25
25	G1	2	31.3145	558 ± 16
26	G1	3	56.3	420 ± 34
27	G1	2	20; 24	593 ± 45
28	G1	2	14.55; 15.89	465 ± 21
29	G1	2	44.59	405 ± 10
30	G1	4	58.5	480 ± 50
31	G1	1	2.79	528 ± 20
34	G1	4	93.4	453 ± 18
35	G1	2	14.92; 14.95	690*
36	G1	4	22.66	444 ± 40
37	G1	2	83.386	434 ± 17
38	G1	4	38	700 ± 120*
39	G1	2	25.0	381 ± 37
41	G1	2	22.24	501 ± 18
42	G1	2	29.7	517 ± 58
43	G1	2	32.4; 33.7	532 ± 33
44	G1	2	88.44	490 ± 57
45	G1	13	41.42-43.38	470 ± 20
46	G1	2	109	465 ± 10
47	G1	2	2.334	484 ± 15
48	G1	3	23.78-24.9	484 ± 41
49	G1	2	42.85; 44.26	440 ± 16
51	G1	2	97.98; 98.11	449 ± 17
52	G1	5	71.45	442 ± 16
53	G1	9	62.15-62.18	504 ± 13
56	G1	2	27.6	454 ± 17
57	G1	2	28; 50	432 ± 30
58	G1	7	97.56	510 ± 20
59	G1	2	21.61	580 ± 100
60	G1	4	66.74	493 ± 15
61	G1	3	34.72-36.8	483 ± 33

TABLE VIII (cont'd). RESULTS# FOR ^{40}K IN IAEA-414.
(Reference date: 1 January 1997, unit: Bq kg^{-1} dry weight)

Lab Code	Method code	No. of Results	Weight (g)	^{40}K
62	G1	3	35.24	498 ± 67
63	G1	2	27.88; 31.39	400 ± 35
64	G1	2	27.13; 38.41	540 ± 35
65	G1	3	24.58	461 ± 32
66	G1	2	41.4; 102.4	504 ± 81
67	G1	4	14.3	507 ± 37
68	G1	2	42.2; 43.5	552 ± 43
69	G1	2	100	368 ± 32
70	G1	1	37.5	522 ± 26
71	G1	16	30	480 ± 10
72	G1	2	25	426 ± 12
74	G1	3	61.31-68.23	460 ± 20
75	G1	4	32.05-33.57	498 ± 15
76	G1	5	37-93	609 ± 41*
77	G1	16	96.4-96.8	453 ± 9
78	G1	2	16.9; 72.1	436 ± 25
79	G1	4	11-95	507 ± 51
82	G1	2	102	382 ± 44
83	G1	1	150.6	510 ± 30
85	G1	2	70.6	479 ± 24
87	G1	3	100	353 ± 35*

Number of reported lab. Means	72
Number of accepted lab. Means	67
Median	480
Confidence interval ($\alpha = 0.05$)	461 – 498

Uncertainties at 2σ
* Results rejected by the test for outliers

TABLE IX. RESULTS[#] FOR ^{232}Th IN IAEA-414.
(Reference date: 1 January 1997, unit: Bq kg^{-1} dry weight)

Lab code	Method code	No. of results	Weight (g)	^{232}Th
1	G1	1	20	<0.6
3	A18	2	10.1; 10.2	0.025 ± 0.005
5	G1	2	246.5	<1.0
10	G1	2	69.8	1.85 ± 1.03*
22	G1	2	96.81	<20
45	A	1	1.56	0.9 ± 0.6*
50	A11	1	4.8	0.031 ± 0.015
57	G1	1	28	1.45 ± 1.03*
71	A22	8	10	0.044 ± 0.002*
72	A10	2	14.96; 15.06	0.030 ± 0.004
74	A10	2	5	0.026 ± 0.012
76	A2	2	50	0.030 ± 0.014
79	A	4	10	0.029 ± 0.003
81	G1	2	50	2.7 ± 1.1*
Number of reported lab. Means				11
Number of accepted lab. Means				6
Median				0.029
Confidence interval ($\alpha = 0.05$)				0.025 – 0.031

Uncertainties at 2σ
* Results rejected by the test for outliers

TABLE X. RESULTS# FOR ^{234}U IN IAEA-414.
(Reference date: 1 January 1997, unit: Bq kg^{-1} dry weight)

Lab code	Method code	No. of results	Weight (g)	^{234}U
1	A26	2	5	1.4 ± 0.3
3	A18	2	10	1.27 ± 0.08
9	M3	1	9.55	1.14 ± 0.09
15	A2	2	10	0.02*
23	A2	1	46.2	0.04 ± 0.02*
25	A22	5	3.0-3.6	1.45 ± 0.07
26	A23	3	1	3.5 ± 0.3*
29	M5	2	7.31	0.97 ± 0.16
31	A27	2	26.4; 29	1.14 ± 0.09
40	A2	3	1.0	3.8 ± 0.2*
45	A	1	1.56	2.6 ± 0.6*
49	A23	2	42; 44	0.25 ± 0.04*
50	A11	2	4.82; 4.86	0.82 ± 0.07
68	A22	2	42.1; 43.5	1.16 ± 0.05
71B	M1	3	10	1.24 ± 0.02
71C	A22	16	10	1.23 ± 0.02
72	A18	2	14.8; 14.93	1.15 ± 0.07
74	A18	2	5	1.20 ± 0.14
75	A	2	4.243; 6.277	1.32 ± 0.05
78	A14	2	7.6	2.15 ± 0.07*
79	A	4	10	1.26 ± 0.13
80	A2	3	25	1.06 ± 0.04
85	A22	1	24.662	1.28 ± 0.09

Number of reported lab. Means	23
Number of accepted lab. Means	16
Median	1.22
Confidence interval (α = 0.05)	1.14– 1.27

Uncertainties at 2σ
* Results rejected by the test for outliers

TABLE XI. RESULTS# FOR ^{235}U IN IAEA-414.
(Reference date: 1 January 1997, unit: Bq kg^{-1} dry weight)

Lab code	Method Code	No. of Results	Weight (g)	^{235}U
1	DNC	2	0.2	0.065 ± 0.019
1	A26	2	5	<0.1
3	A18	2	10	0.062 ± 0.016
9	M3	1	9.55	0.050 ± 0.002
14	G1	4	96.71	3.86 ± 0.14*
15	A2	2	10	0.029 ± 0.006
25	A22	5	3.0-3.6	0.07 ± 0.02
26	A23	3	1	<0.1
29	M5	2	3.66	0.036 ± 0.002
31	A27	1	26.4	0.05 ± 0.04
38	G1	4	38	<2.0
44	G1	2	88.44	<0.2
50	A11	2	4.82; 4.86	<0.04
57	G1	2	28; 50	<1
62	G1	3	35.24	<24
68	A22	2	42.1; 43.5	0.042 ± 0.005
71A	A22	2	10	0.045 ± 0.005
71B	M2	3	10	0.052 ± 0.001
71C	A22	18	10	0.0525 ± 0.0004
71D	G1	16	30	0.045 0.002
72	A18	2	14.8; 14.93	0.038 ± 0.008
74	A18	2	5	0.051 ± 0.020
75	A	2	4.243; 6.277	0.086 ± 0.015
79	A	4	10	0.046 ± 0.004
85	A22	1	24.662	0.058 ± 0.012

Number of reported lab. Means	18
Number of accepted lab. Means	17
Median	0.050
Confidence interval (α = 0.05)	0.045 – 0.058

Uncertainties at 2σ
* Results rejected by the test for outliers

TABLE XII. RESULTS# FOR ^{238}U IN IAEA-414.
(Reference date: 1 January 1997, unit: Bq kg^{-1} dry weight)

Lab code	Method Code	No. of results	Weight (g)	^{238}U
1	DNC	2	0.2	1.4 ± 0.3
1	A26	2	5	1.4 ± 0.4
3	A18	2	10	1.15 ± 0.08
5	G1	2	246.5	<2.0
9	M3	1	9.55	1.07 ± 0.05
10	G1	2	26.2	21.0 ± 5.2*
15	A2	2	10	0.026*
23	A2	1	46.2	0.05 ± 0.02*
25	A22	5	3.0-3.6	1.33 ± 0.06
26	A23	3	1	2.8 ± 0.4*
29	M5	2	7.31	0.94 ± 0.02
31	A27	2	26.4; 29	0.85 ± 0.06
38	G1	1	38	0.9 ± 0.4
45	A	1	1.56	3.5 ± 0.8*
45	G1	13	41-44	<3
49	A23	2	42; 44	0.14 ± 0.03*
50	A11	2	4.82; 4.86	0.82 ± 0.08
57	G1	2	28; 50	<19
66	G1	2	41; 102	<34
68	A22	2	42.1; 43.5	1.07 ± 0.05
71A	A22	2	10	1.10 ± 0.05
71B	M2	3	10	1.112 ± 0.020
71C	A22	18	10	1.12 ± 0.01
72	A18	2	14.8; 14.93	1.07 ± 0.07
74	A18	2	5	1.15 ± 0.15
75	A	2	4.243; 6.277	1.19 ± 0.04
78	A14	2	7.6	1.95 ± 0.07*
79	A	4	10	1.17 ± 0.12
80	A2	3	25	1.02 ± 0.03
81	G1	2	50	17.8 ± 7.7*
85	A22	1	24.662	1.22 ± 0.09
Number of reported lab. Means				27
Number of accepted lab. Means				19
Median				1.112
Confidence interval ($\alpha = 0.05$)				1.065 – 1.170

Uncertainties at 2σ
* Results rejected by the test for outliers

TABLE XIII. RESULTS[#] FOR ^{226}Ra IN IAEA-414.
(Reference date: 1 January 1997, unit: Bq kg^{-1} dry weight)

Lab Code	Method Code	No. of results	Weight (g)	^{226}Ra
1	G1	2	20	<15
1	A28	2	1	5.0 ± 1.3*
3	G1	2	62.5	<3
4	G1	2	28.39	<0.1
5	G1	2	246.5	0.59 ± 0.06
6	G1	5	90.7	7.7 ± 0.5*
9	G1	1	68.85	<2
10	G1	2	69.8	<6
21	B25	2	29; 30	<0.1
22	G1	2	96.81	<2
25	A2	4	3.1-3.6	1.48 ± 0.14
25	G1	2	31.31	1.8 ± 0.5
32	G1	1	98	0.6 ± 0.4
34	G1	2	93.4	0.38 ± 0.15
37	G1	2	83.386	0.54 ± 0.24
38	G1	4	0.0383	12.1 ± 1.3*
40	A29	3	1.0	1.6 ± 0.7
44	G1	2	88.44	<0.2
45	G1	5	41-43	1.0 ± 0.5
56	G1	2	27.6	<2
57	G1	1	28	10 ± 6*
64	G1	1	38.41	1.3 ± 0.5
69	G1	2	100	1.76 ± 0.20
71	G1	14	30	1.40 ± 0.35
72	A30	2	14; 15	1.5 ± 0.4
74	A28	2	5	<0.6
81	G1	2	50	12 ± 6*
87	G1	3	100	2.6 ± 0.3
Number of reported lab. Means				18
Number of accepted lab. Means				13
Median				1.40
Confidence interval ($\alpha = 0.05$)				0.59 – 1.76

Uncertainties at 2σ
* Results rejected by the test for outliers

TABLE XIV. RESULTS# FOR $^{210}Pb(^{210}Po)$ IN IAEA-414.
(Reference date: 1 January 1997, unit: Bq kg^{-1} dry weight)

Lab code	Method code	No. of results	Weight (g)	$^{210}Pb(^{210}Po)$
1	A13	3	1	3.3 ± 1.0
3A	A13	2	1.33; 1.36	12 ± 1*
3B	A13	2	1.33; 1.36	11 ± 1*
5	G1	2	246.5	<3
9	G1	1	68.85	<5
10	G1	2	69.8	<15
16	G1	2	96.7; 99.3	5.1 ± 2.3*
19A	A28	1	5.1638	9 ± 4*
19B	A28	1	10	2.0 ± 0.7
20	A13	3	-	1.83 ± 0.04
22	G1	2	96.81	<3.6
25A	A13	5	3.05-3.66	2.85 ± 0.12
25B	A13	5	3.05-3.66	2.54 ± 0.11
34	G1	2	93.4	1.3 ± 0.9
37	G1	2	83.386	2.3 ± 0.9
40	A13	4	1.0-1.02	3.0 ± 0.1
44	G1	2	88.44	<170
47	G1	2	2.334	<10
50	A13	2	4.79; 4.86	0.76 ± 0.15
55A	B24	2	17.5; 19.2	2.9 ± 0.5
55B	A13	2	7.7; 8.8	2.44 ± 0.22
56	G1	2	27.6	6 ± 2*
62	G1	3	35	<25
64	G1	3	27-38	<1.6
68	A13	2	4.2; 9.6	2.9 ± 0.3
70	A13	2	1.5; 3.7	2.5 ± 0.2
71	A13	30	0.3	2.14 ± 0.07
72A	A13	2	14.79; 14.89	1.35 ± 0.13
72B	A13	2	14.79; 14.89	1.92 ± 0.11
74A	B16	1	30	1.1 ± 0.9
74B	A13	2	1	1.55 ± 0.25
79	A	6	3-10	2.77 ± 0.23
80	A13	4	25	1.61 ± 0.03
82	A	2	1.01; 1.06	0.65 ± 0.19
83	B22	2	40	2.6 ± 0.7

Number of reported lab. Means	27
Number of accepted lab. Means	22
Median	2.22
Confidence interval (α = 0.05)	1.55 – 2.60

Uncertainties at 2σ
* Results rejected by the test for outliers

TABLE. XV. RESULTS FOR $^{238}Pu/^{239+240}Pu$ AND $^{241}Am/^{239+240}Pu$ ACTIVITY RATIOS IN IAEA-414

Lab. Code	$^{238}Pu/^{239+240}Pu$	$^{241}Am/^{239+240}Pu$
2	0.24 ± 0.04	
17	0.23 ± 0.06	
20	0.21 ± 0.03	1.7 ± 0.2
27		1.78 ± 0.17
29	0.18 ± 0.10	1.52 ± 0.37
49	0.18 ± 0.09	1.4 ± 0.4
68	0.196 ± 0.024	1.63 ± 0.13
71	0.180 ± 0.011	1.64 ± 0.10
72	0.187 ± 0.019	1.60 ± 0.12
74	0.16 ± 0.06	1.28 ± 0.19
75	0.201 ± 0.033	
76	0.23 ± 0.07	1.7 ± 0.2
77	0.179 ± 0.015	1.6 ± 0.6
78	0.228 ± 0.022	
79	0.177 ± 0.026	1.6 ± 0.2
83	0.25 ± 0.03	1.67 ± 0.15
85	0.16 ± 0.08	
88	0.156 ± 0.021	
Weighted mean value	0.193 ± 0.006	1.59 ± 0.05

TABLE XVI. OTHER LESS FREQUENTLY MEASURED ATOM RATIOS REPORTED IN IAEA-414

Isotope	Lab. code	Method Code	No. of results	Weight (g)	Atom ratios
$^{240}Pu/^{239}Pu$	9	M3	1	-	0.22 ± 0.09
-	71B	M1	14	10	0.21 ± 0.01
-	71C	M7	9	10	0.23 ± 0.05
-	85	M3	1	2.758	0.191 ± 0.016
-	90	M3	5	10	0.19 ± 0.05
$^{234}U/^{238}U$	9	M3	1	9.55	$(5.8 \pm 0.3)\ 10^{-5}$
$^{235}U/^{238}U$	9	M3	1	9.55	$(7.1 \pm 0.2)\ 10^{-3}$

TABLE XVII. RESULTS[#] FOR THE LESS FREQUENTLY MEASURED RADIONUCLIDES REPORTED IN IAEA-414.
(Reference date: 1 January 1997, unit: Bq kg^{-1} dry weight)

Isotope	Lab. code	Method Code	No. of results	Weight (g)	Activity (Bq kg^{-1})
^{60}Co	26	G1	3	56.3	<2
-	32	G1	1	98	<0.6
-	33	G1	1	64.2	<1.3
-	36	G1	4	22.66	<1
-	57	G1	2	28; 50	<1
-	62	G1	3	35.24	<3
-	71	G1	11	60	<0.5
-	91	G1	10	102	<0.1
^{99}Tc	2	B22	1	30	2.2 ± 0.8
-	68	B23	2	49.34; 49.84	6.55 ± 0.45
-	72	B22	2	10	5.5 ± 0.4
-	79	B	2	10	7.0 ± 1.0
-	84	B20	1	10	7.9 ± 2.6
^{129}I	1	G1	1	6	<16
-	9	G1	1	68.85	<3.9
-	22	G1	2	96.81	<1.5
-	34	G1	2	93.4	<0.1
-	44	G1	2	88.44	<27
-	62	G1	3	35.24	<2
-	86	M8	6	1.086-1.099	0.0080 ± 0.0002
-	89	M8	6	2.48	0.0090 ± 0.0002
-	91	M8	3	8.1416	0.012 ± 0.003
^{228}Ra*(^{228}Ac)	1	G1	1	20	<40
	3	G1	2	62.5	<3
	4	G1	2	28.39	2.0 ± 0.3
	10	G1	2	69.8	<2
-	21	B25	2	29; 30	<0.4
-	22	G1	2	96.81	<2
-	24	G1	1	22.45	9.2 ± 1.8
-	25	G1	2	31.3145	<1.2
-	34	G1	2	93.4	<0.2
	44	G1	2	88.44	<0.5
-	45	G1	13	41.42-43.38	<0.4
	56	G1	2	27.6	<1
	62	G1	3	35.24	<22
-	64	G1	3	27-38	<0.9
^{228}Th*	1	G1, A26	3	2.5 - 20	<0.5
-	24	G1	1	22.45	14.0 ± 2.7
-	38	G1	4	38	<17
-	47	G1	2	2.334	<5
-	50	A11	2	4.8; 4.82	<0.03
-	64	G1	2	27; 38	<0.8
-	71	A22	11	10	0.176 ± 0.002
-	72	A10	2	14.96; 15.06	0.175 ± 0.014
-	74	A10	2	5	0.22 ± 0.03
-	76	A2	2	50	0.75 ± 0.14
-	79	A	4	10	0.193 ± 0.018

TABLE XVII (cont'd). RESULTS[#] FOR THE LESS FREQUENTLY MEASURED RADIONUCLIDES REPORTED IN IAEA-414
(Reference date: 1 January 1997, unit: Bq kg^{-1} dry weight)

Isotope	Lab. code	Method Code	No. of results	Weight (g)	Activity (Bq kg^{-1})
^{230}Th	1	G1, A26	3	2.5 - 20	1.95 ± 0.48
-	3	A18	2	10.1; 10.2	0.50 ± 0.03
-	45	A	1	1.56	1.3 ± 0.6
-	50	A11	2	4.8; 4.82	<0.04
-	71	A22	8	10	0.060 ± 0.001
-	72	A10	2	14.96; 15.06	0.040 ± 0.008
-	74	A10	1	5	<0.015
-	74	A10	1	5	0.037 ± 0.018
-	79	A	4	10	0.043 ± 0.004
^{236}U	9	M3	1	9.55	<1.2 10^{-4}
^{237}Np	1	A25	2	10	<0.4
-	31	A27	2	26.4; 29	0.32 ± 0.06
-	44	G1	2	88.44	<0.3
-	71	G1	3	30	1.5 ± 0.3
^{239}Pu	9	M3	1	-	0.070 ± 0.025
-	71B	M1	14	10	0.0640 ± 0.0003
-	71C	M7	9	10	0.082 ± 0.001
-	84	M3	2	10	0.072 ± 0.006
^{240}Pu	9	M3	1	-	0.051 ± 0.024
-	71B	M1	14	10	0.0470 ± 0.0003
-	71C	M7	9	10	0.065 ± 0.003
-	84	M3	2	10	0.053 ± 0.004
^{241}Pu	29	B26	2	21.93	4.5 ± 3.1
-	71B	M1	9	20	2.916 ± 0.003
-	71E	B1	8	20	1.95 ± 0.10
-	71F	A2	5	20	1.33 ± 0.15
^{243}Cm	26	A23	1	10	<0.5
^{243}Am	44	G1	2	88.44	<0.2
^{244}Cm	26	A23	1	10	<0.5

Uncertainties at 2σ
* At the time of the measurement

TABLE XVIII. SUMMARY OF RECOMMENDED AND INFORMATION VALUES FOR IAEA-414
(Reference Date : 1 January 1997, unit: Bq kg^{-1} dry weight)

Radionuclide	Median	Confidence interval ($\alpha = 0.05$)
	Recommended value	
^{137}Cs	5.14	5.00-5.27
^{238}Pu	0.023	0.022-0.025
$^{239+240}Pu$	0.120	0.113-0.125
^{241}Am*	0.196	0.189-0.199
	Information value	
^{40}K	480	461-498
^{90}Sr	0.28	0.10-0.54
$^{210}Pb(^{210}Po^{\#})$	2.22	1.55-2.60
^{226}Ra	1.40	0.59-1.76
^{232}Th	0.029	0.025-0.031
^{234}U	1.22	1.14-1.27
^{235}U	0.050	0.045-0.058
^{238}U	1.112	1.065-1.170

* The values should be corrected for in-growth from ^{241}Pu.
# ^{210}Po is in equilibrium with ^{210}Pb at the time of measurement.

ANNEX II

Data evaluation - graphs

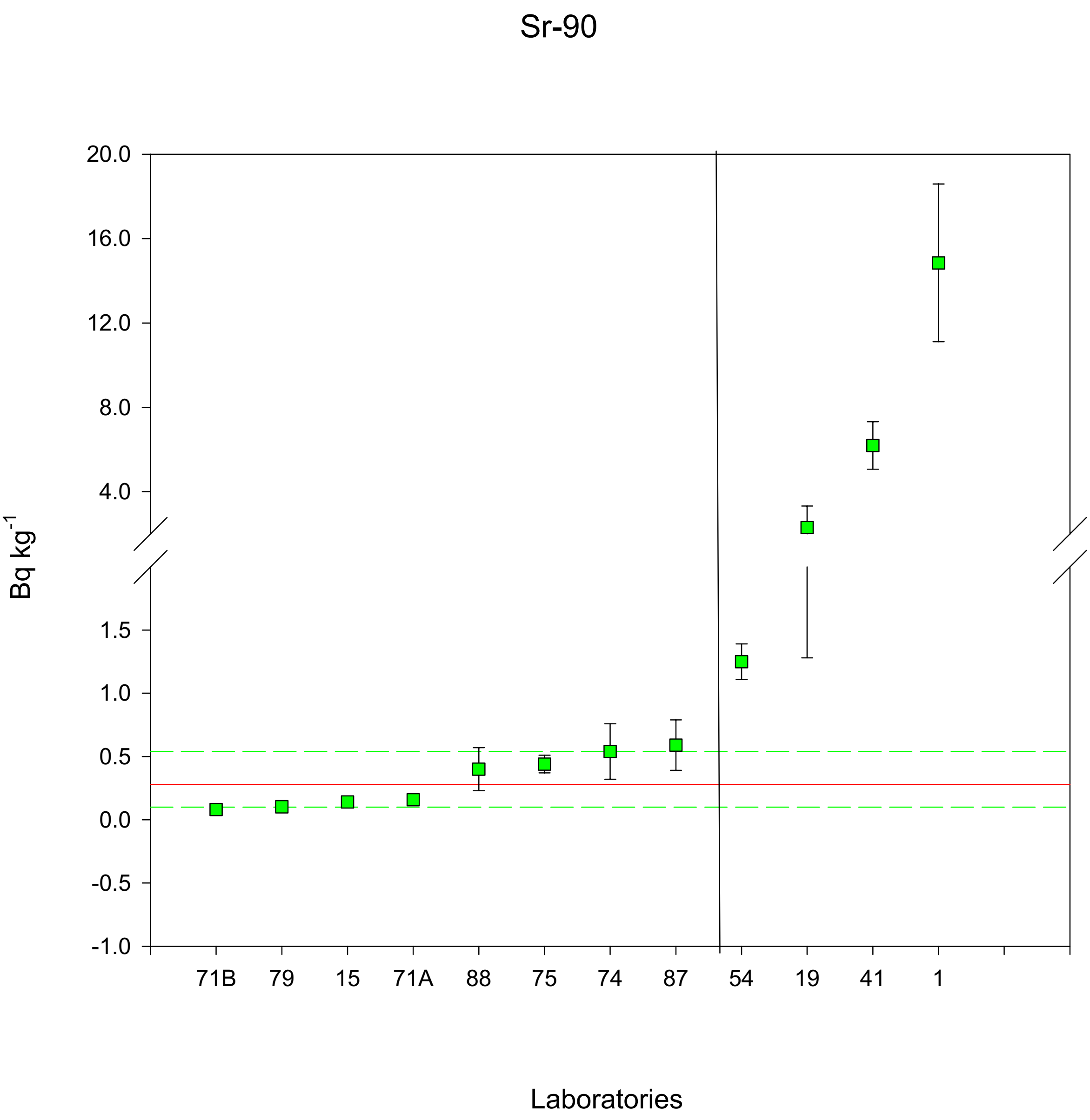

Fig. 1. Data evaluation for ^{90}Sr

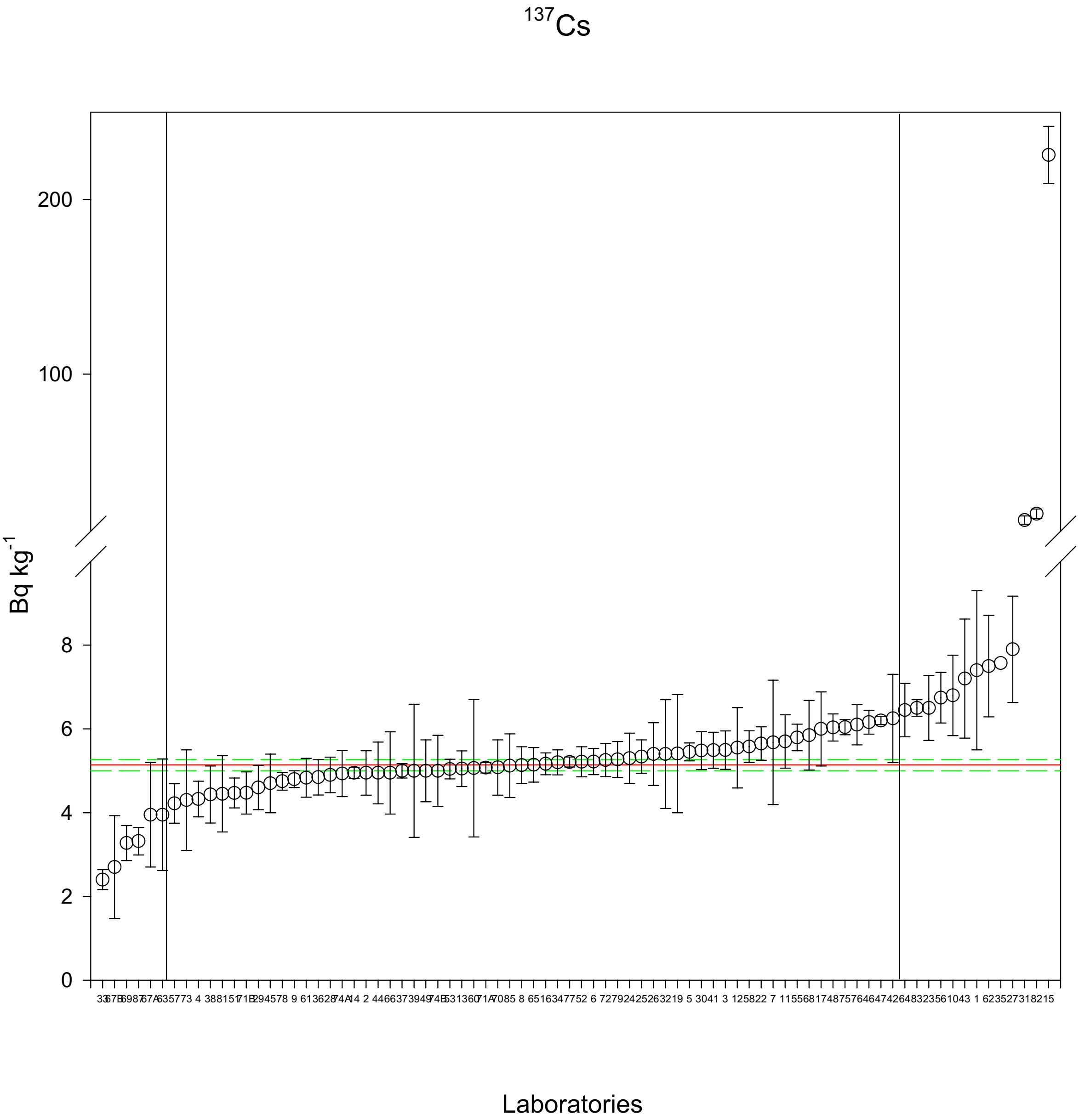

Fig. 2. Data evaluation for ^{137}Cs

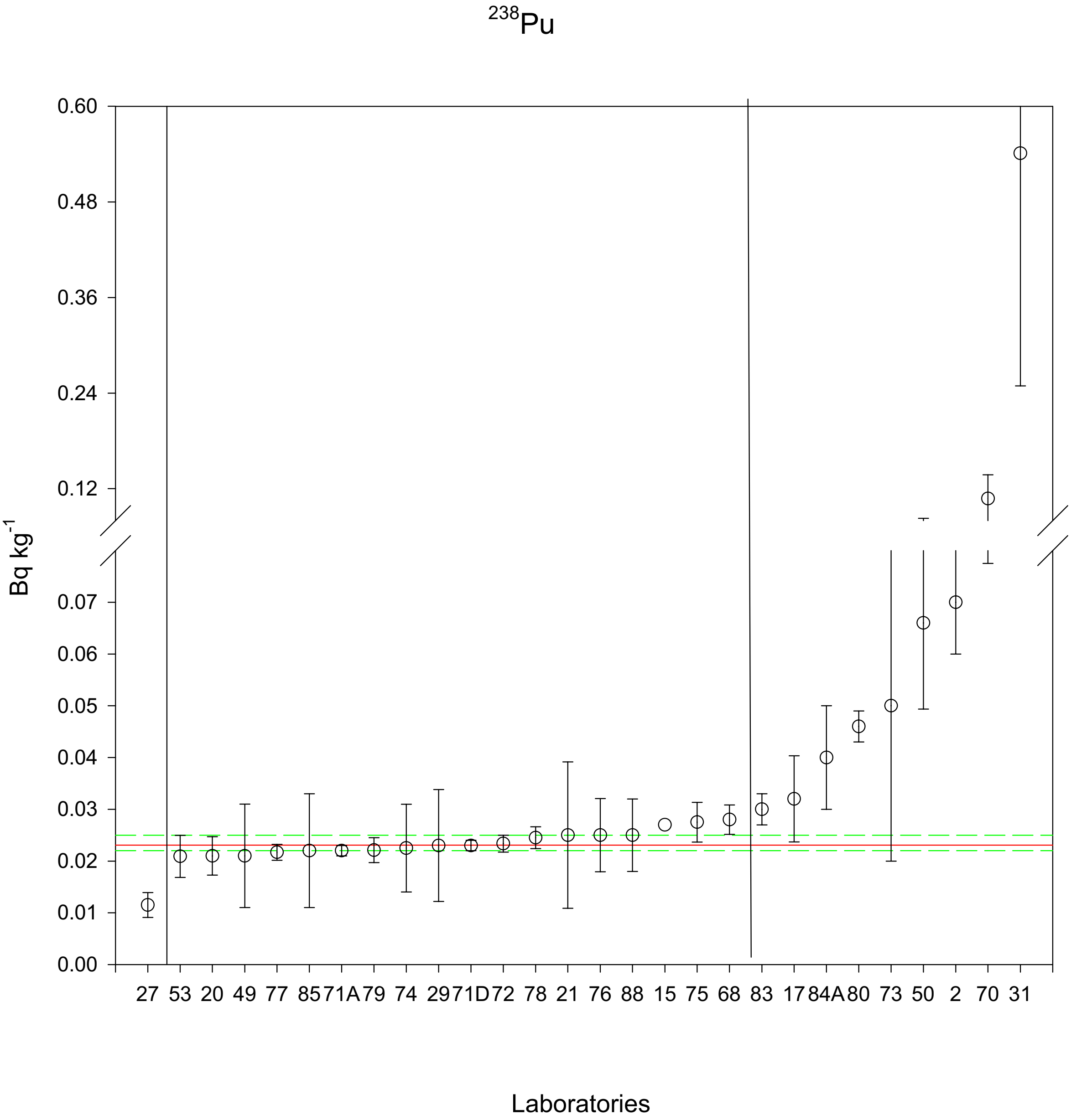

Fig. 3. Data evaluation for ^{238}Pu

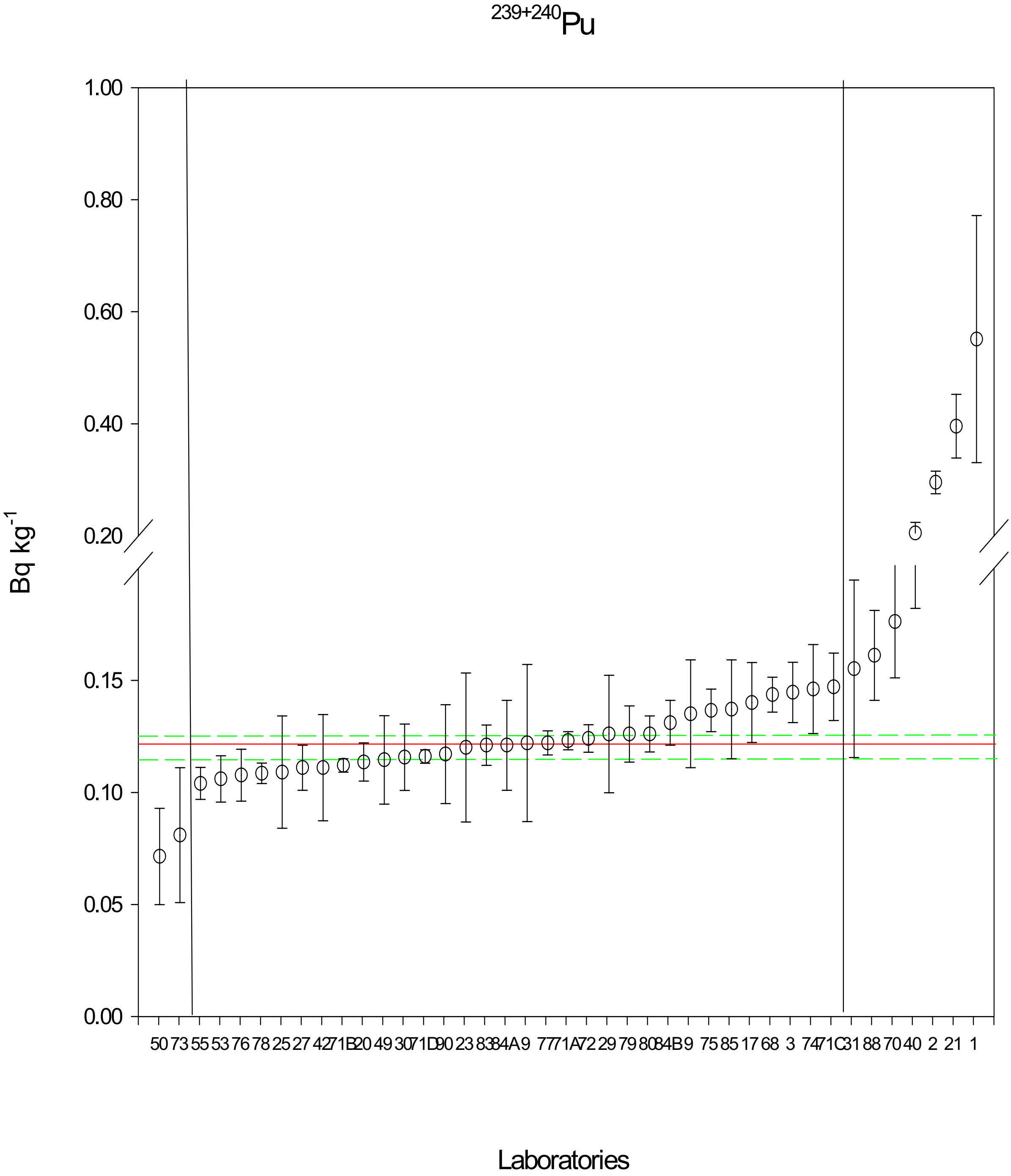

Fig. 4. Data evaluation for $^{239+240}$Pu

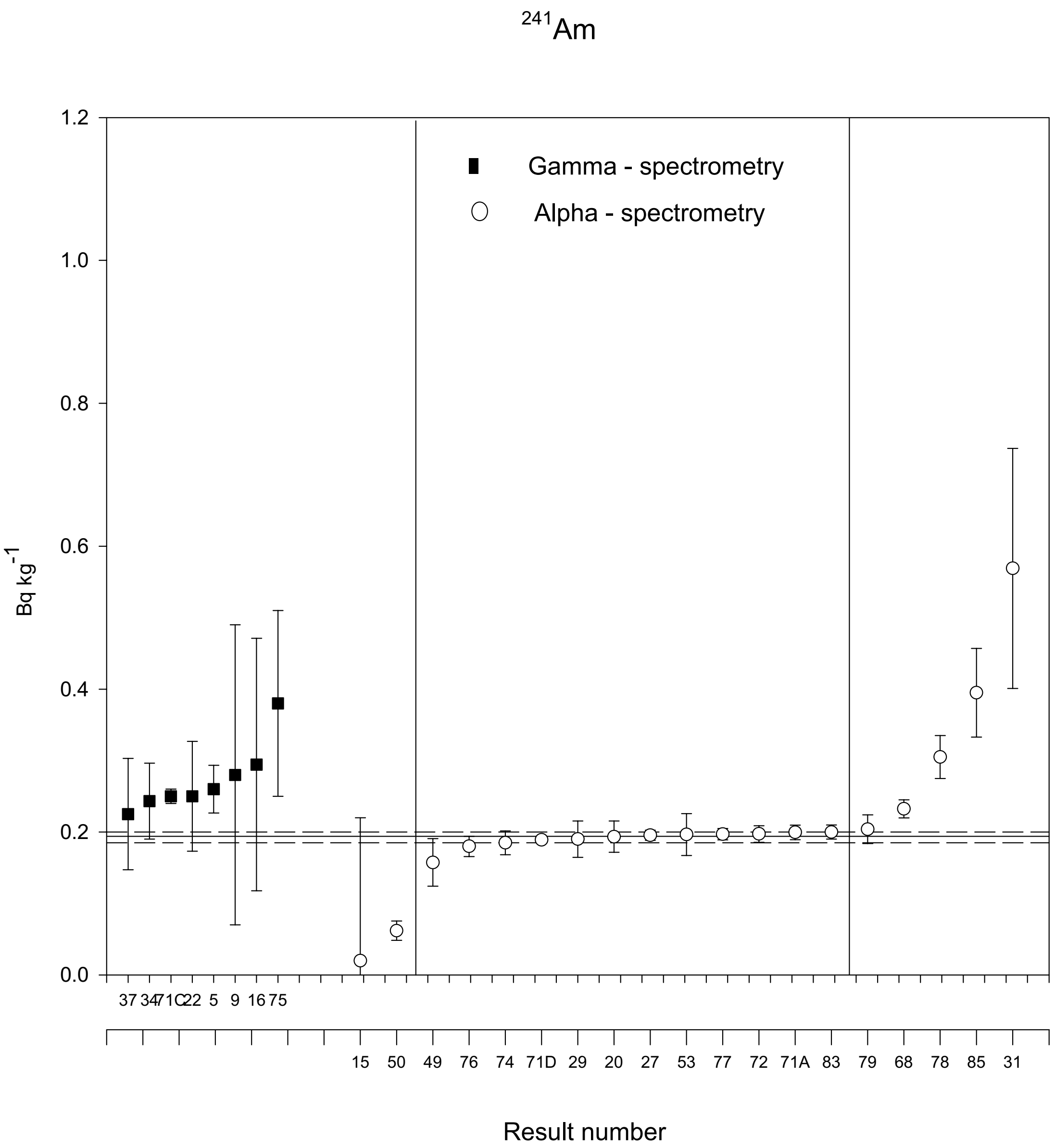

Fig. 5. Data evaluation of ^{241}Am

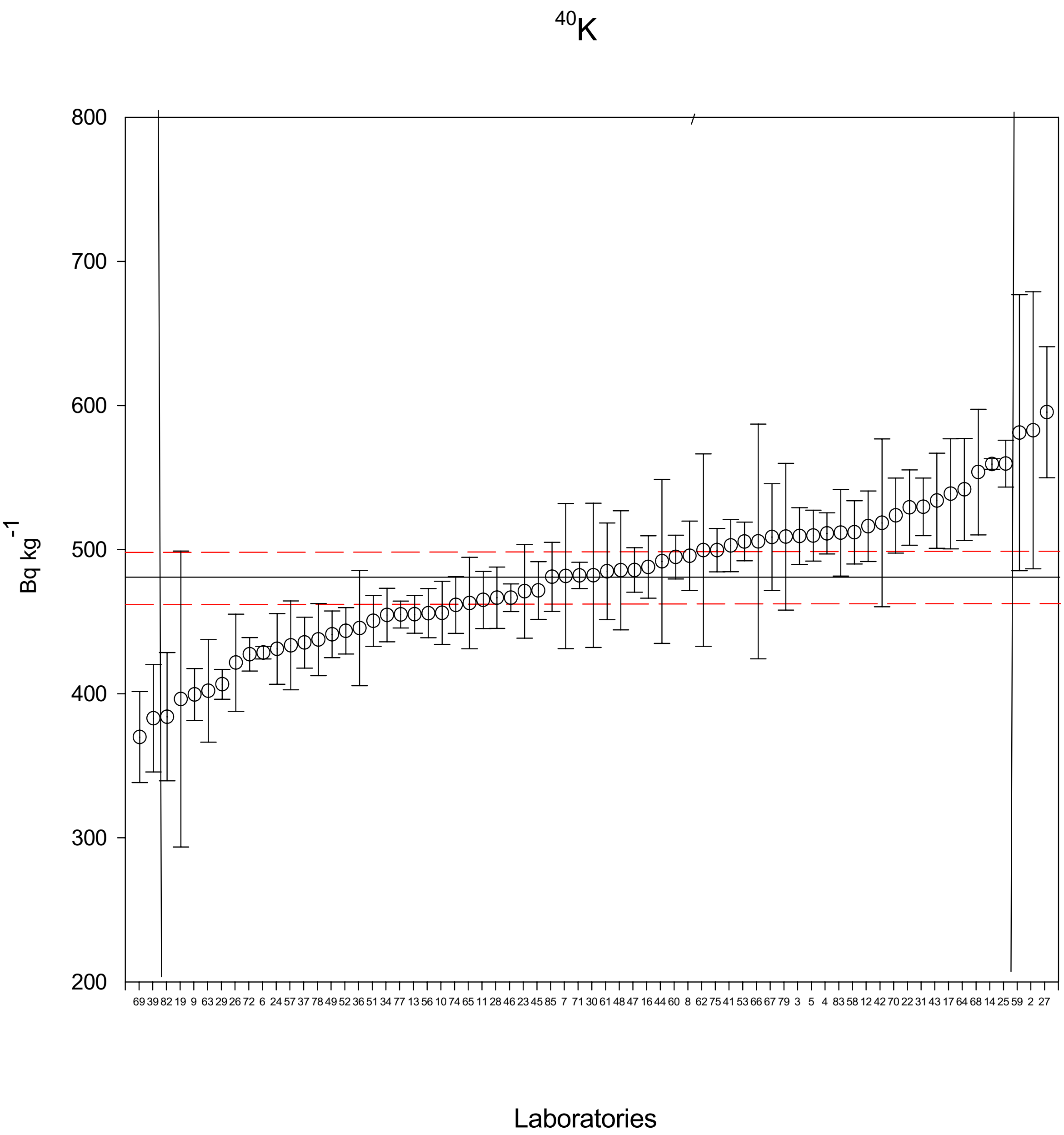

Fig. 6. Data evaluation for ^{40}K

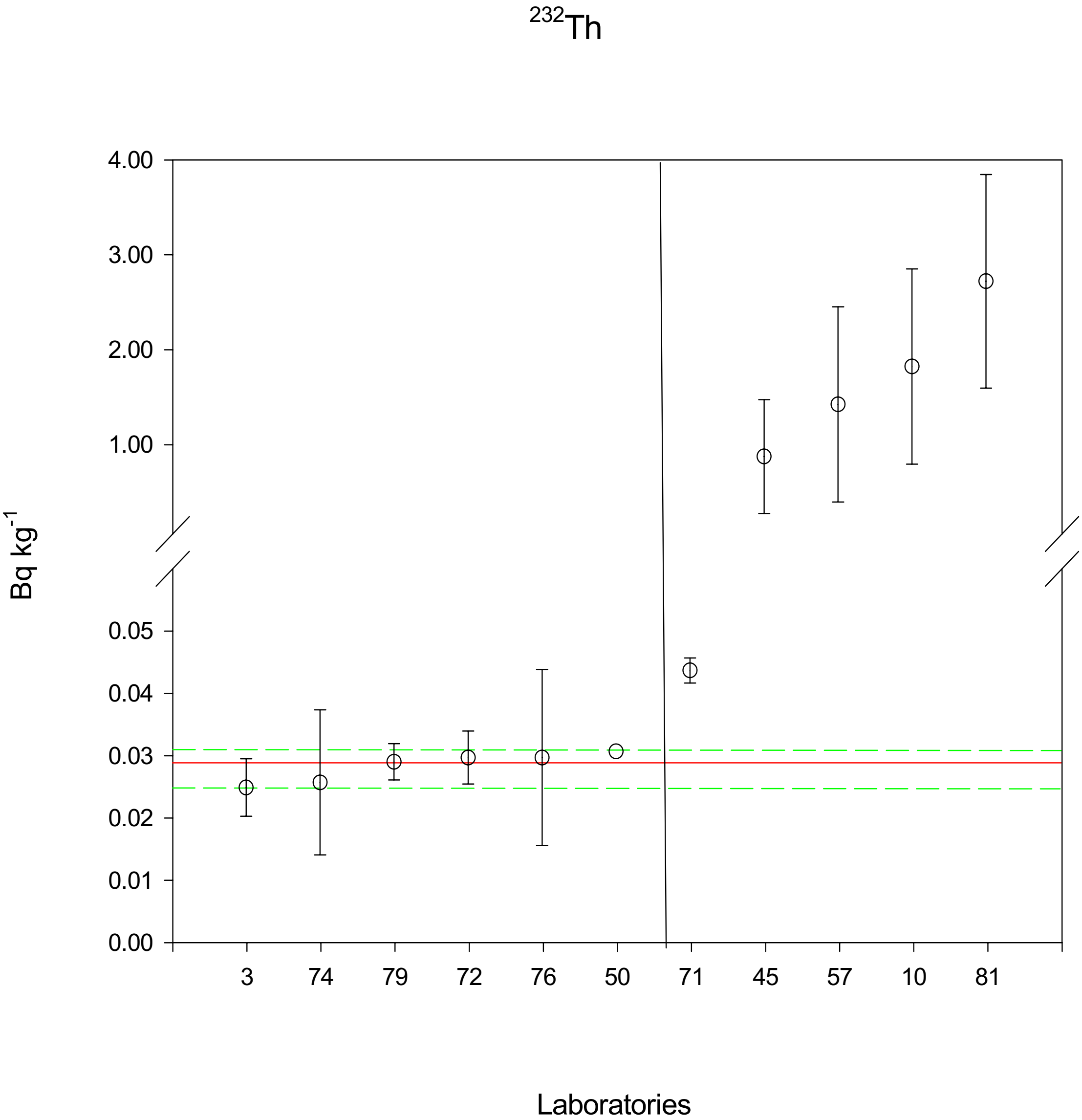

Fig. 7. Data evaluation for ^{232}Th

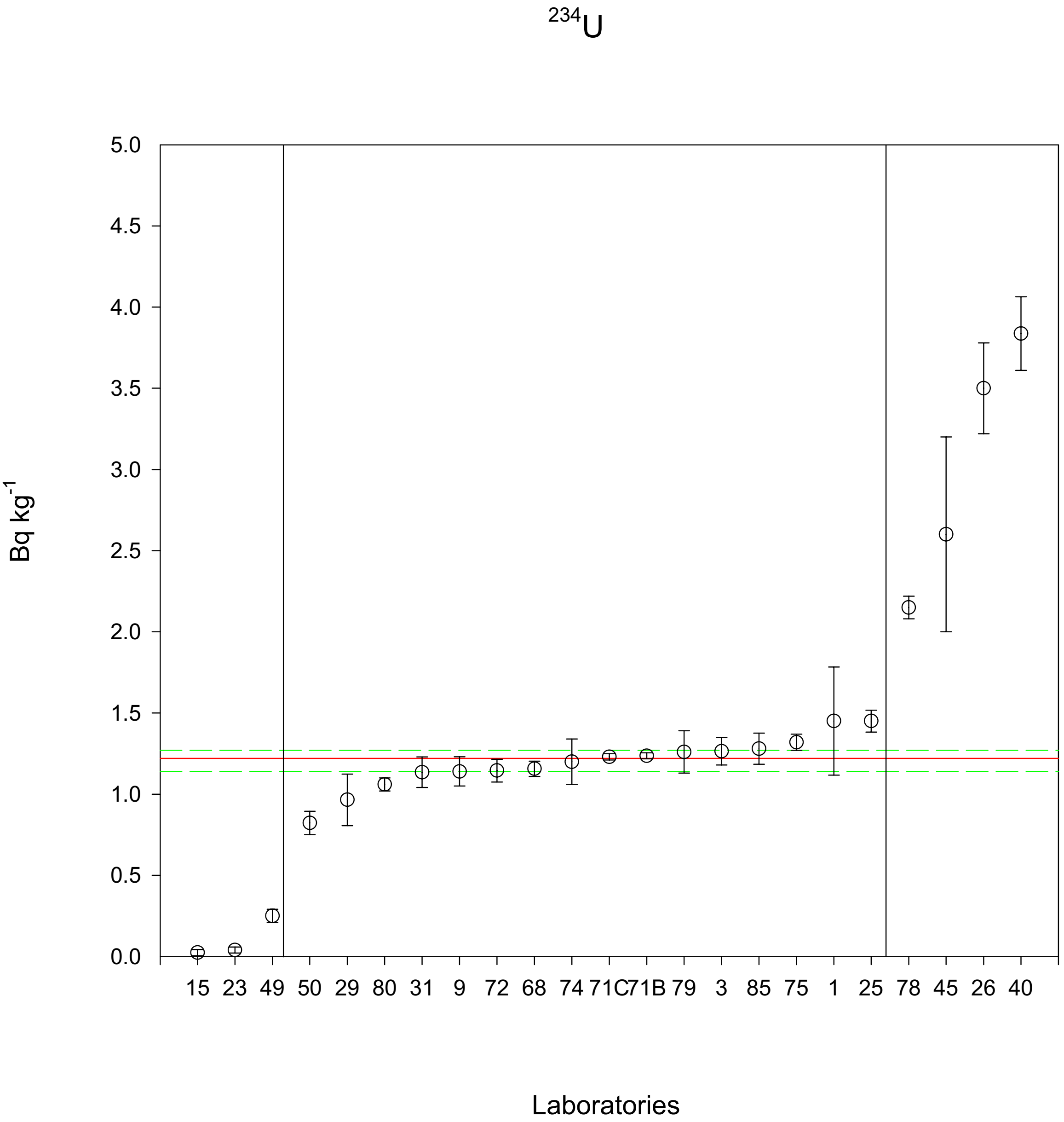

Fig. 8. Data evaluation for ^{234}U

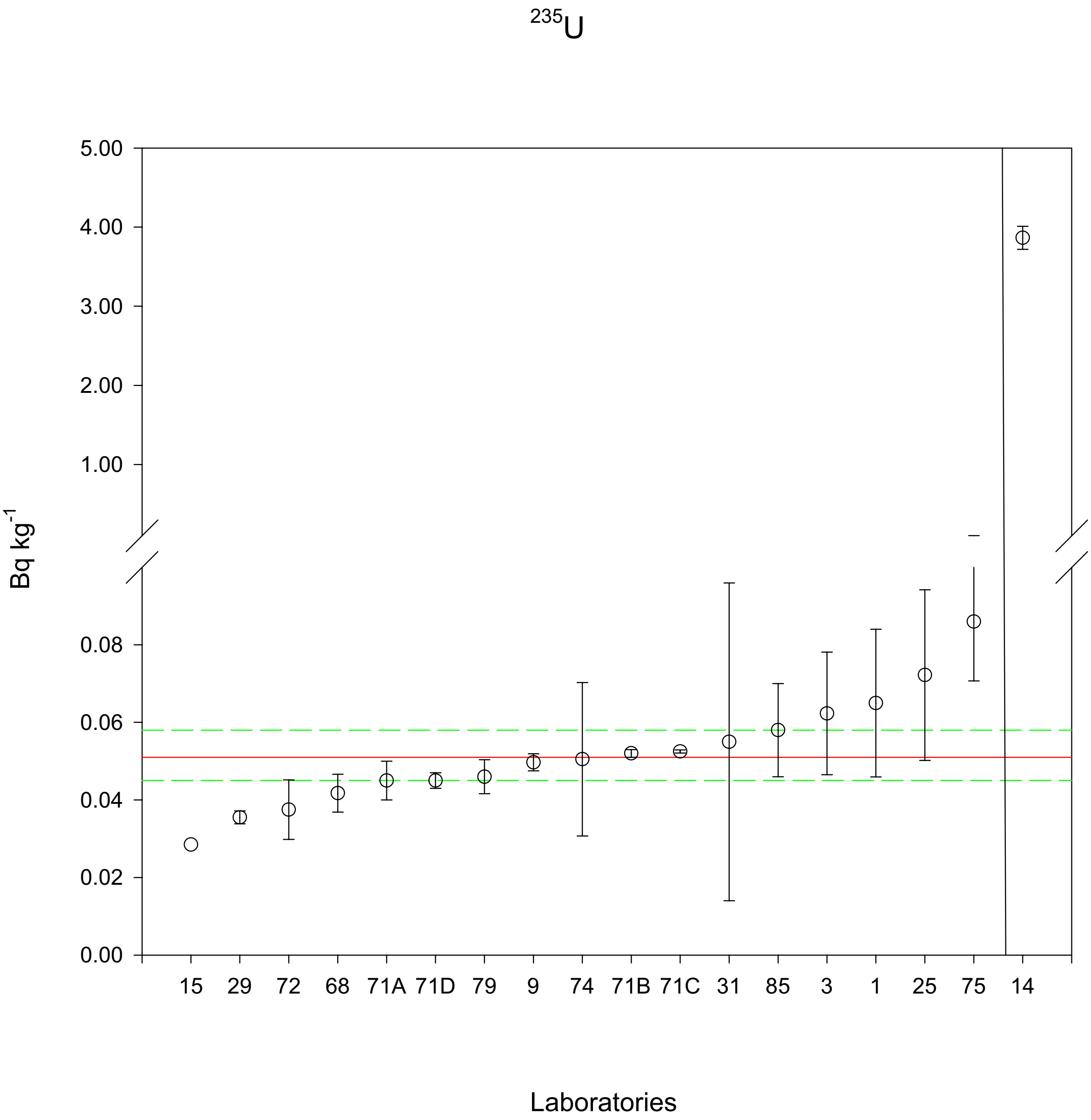

Fig. 9. Data evaluation for ^{235}U

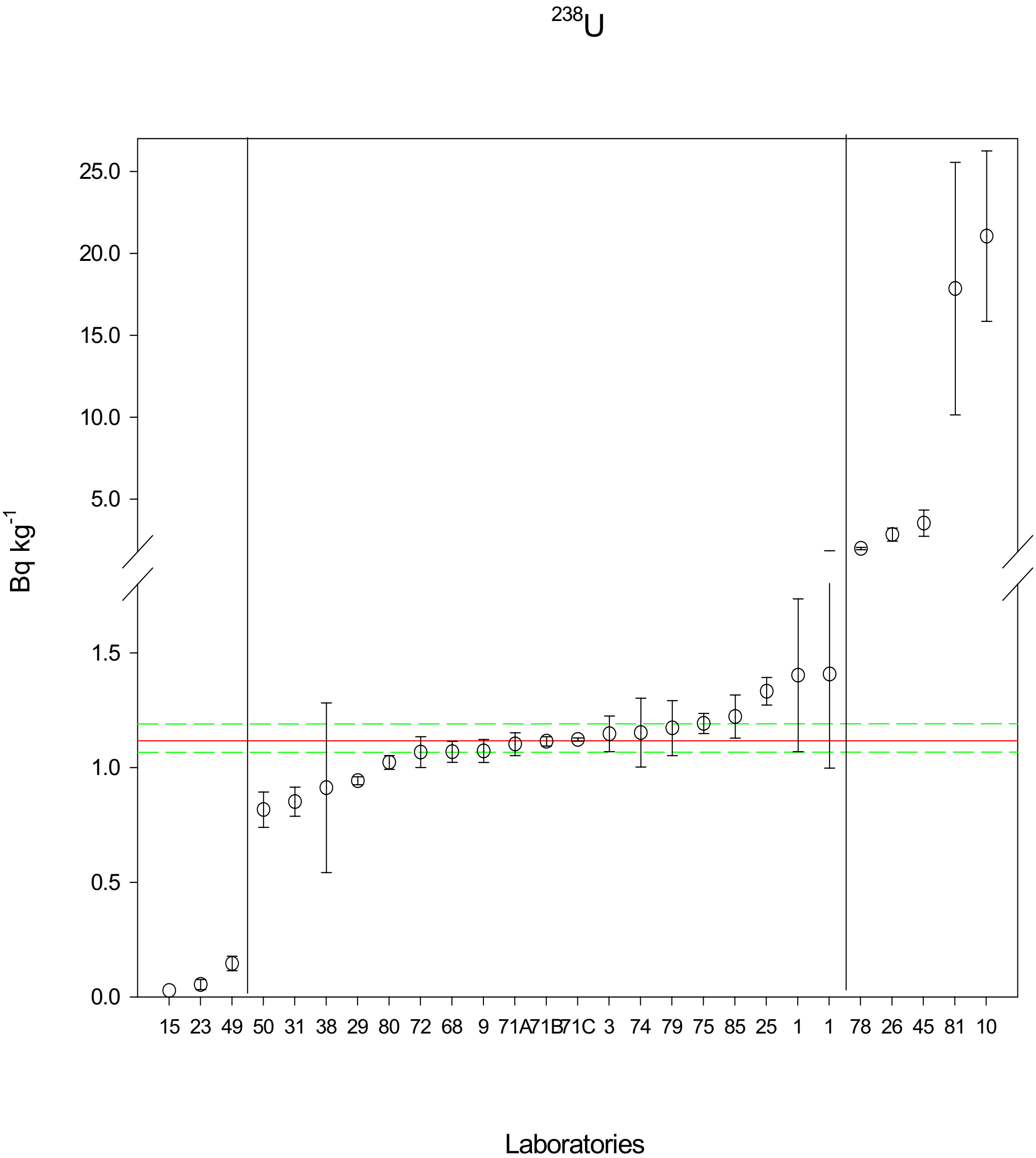

Fig. 10. Data evaluation for ^{238}U

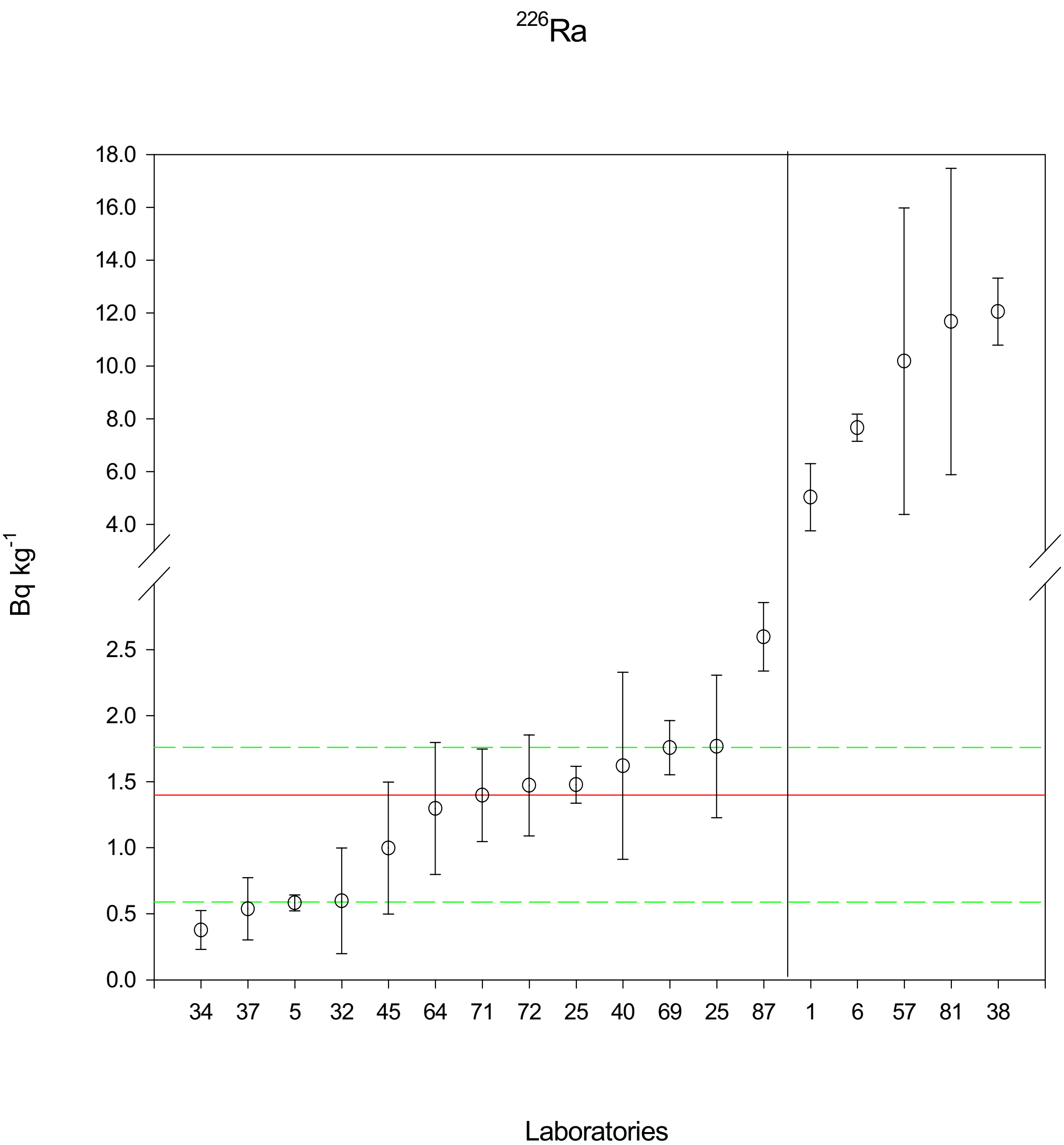

Fig. 11. Data evaluation for ^{226}Ra

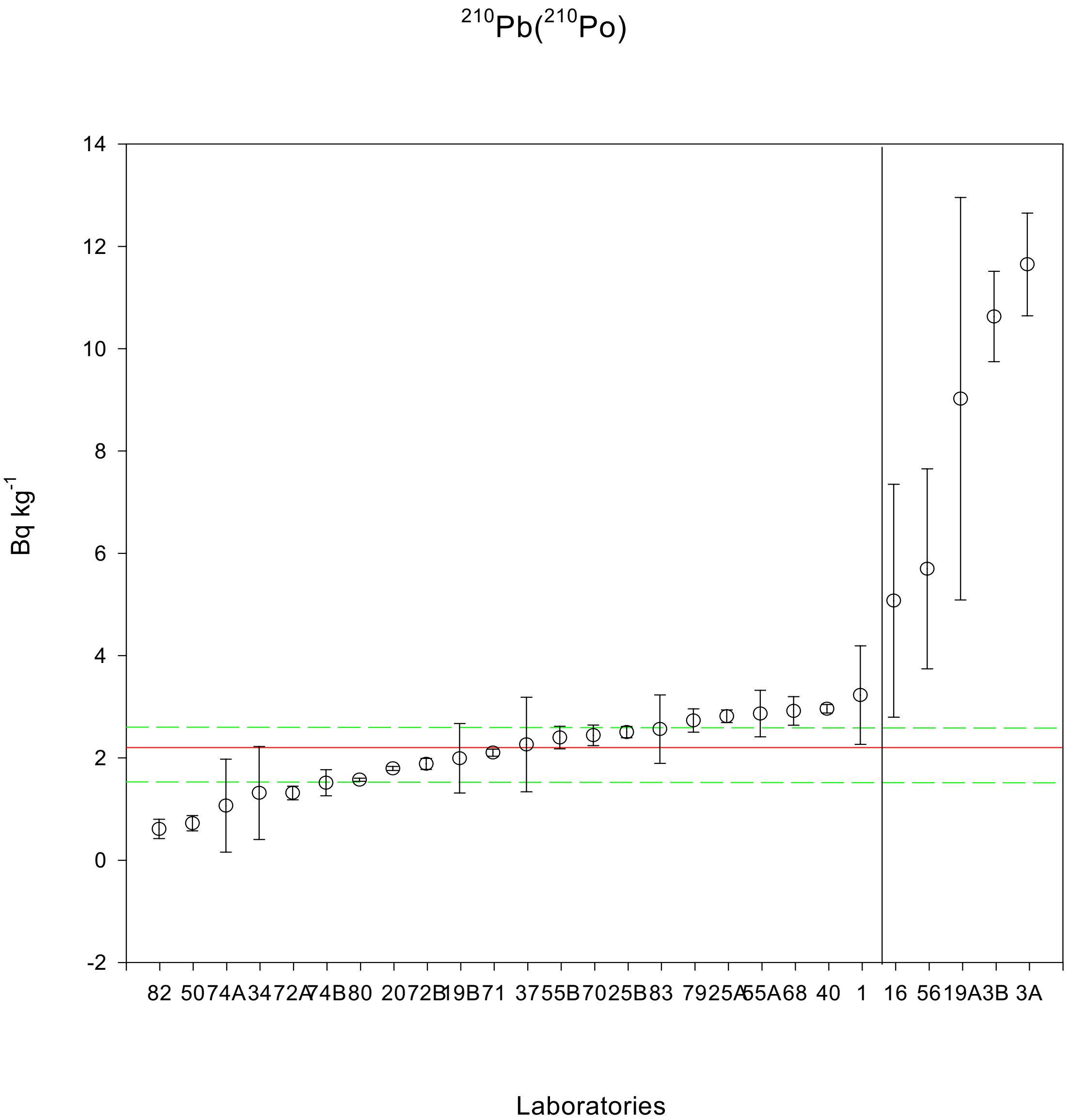

Fig. 12. Data evaluation for $^{210}Pb(^{210}Po)$

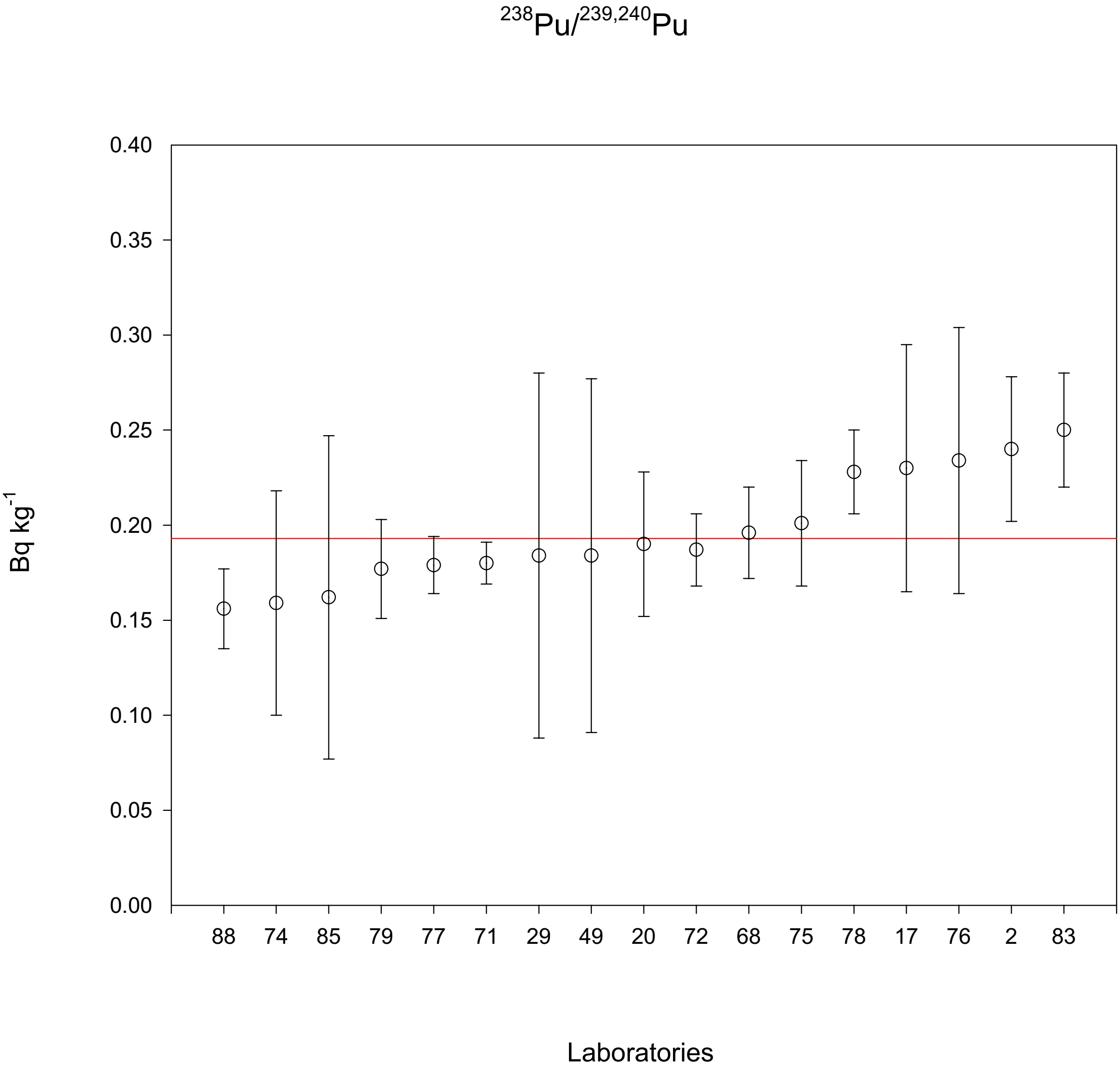

Fig. 13. $^{238}Pu/^{239+240}Pu$ activity ratio

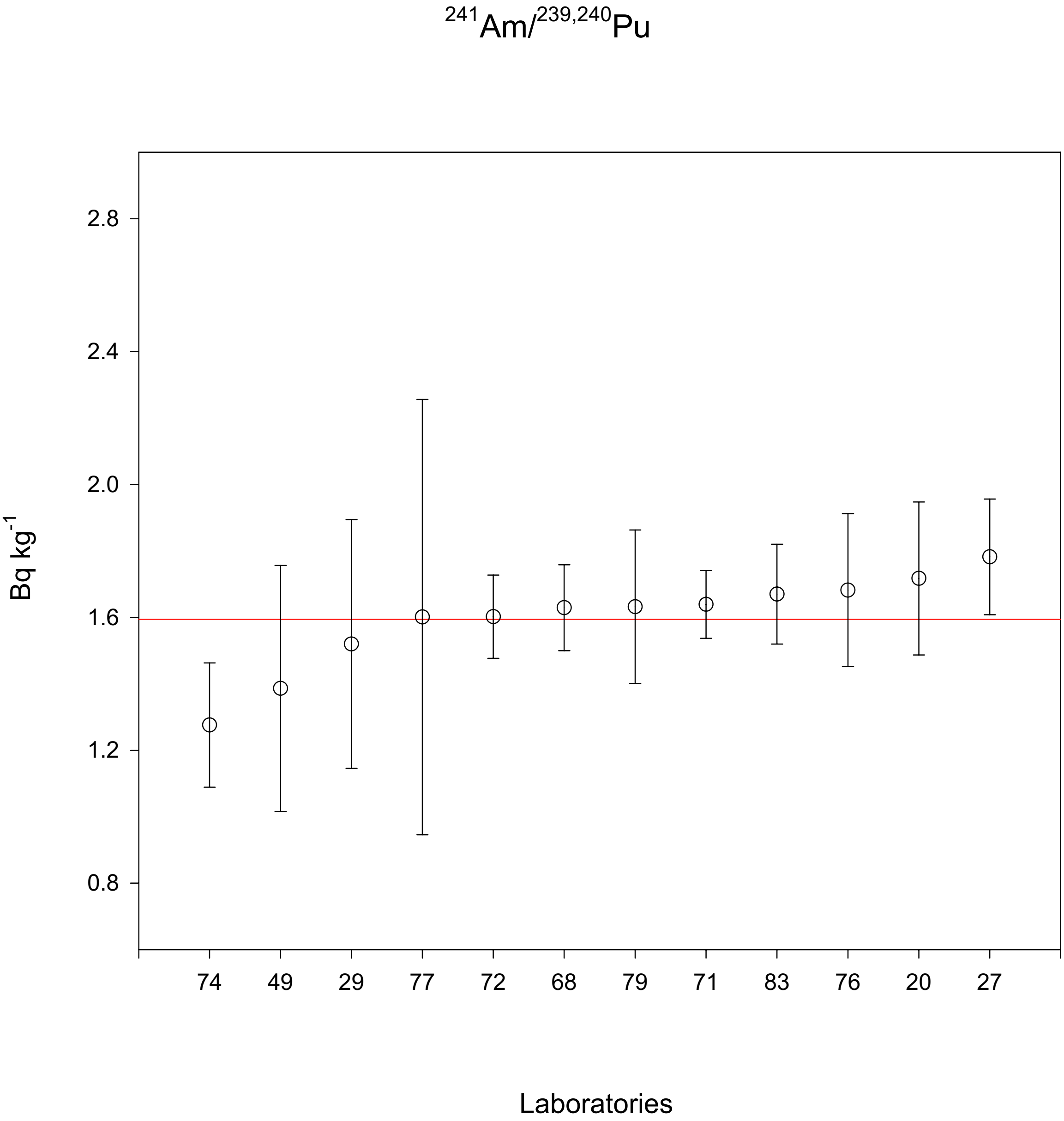

Fig. 14. $^{241}Am/^{239+240}Pu$ activity ratio

ANNEX III

Z-Scores

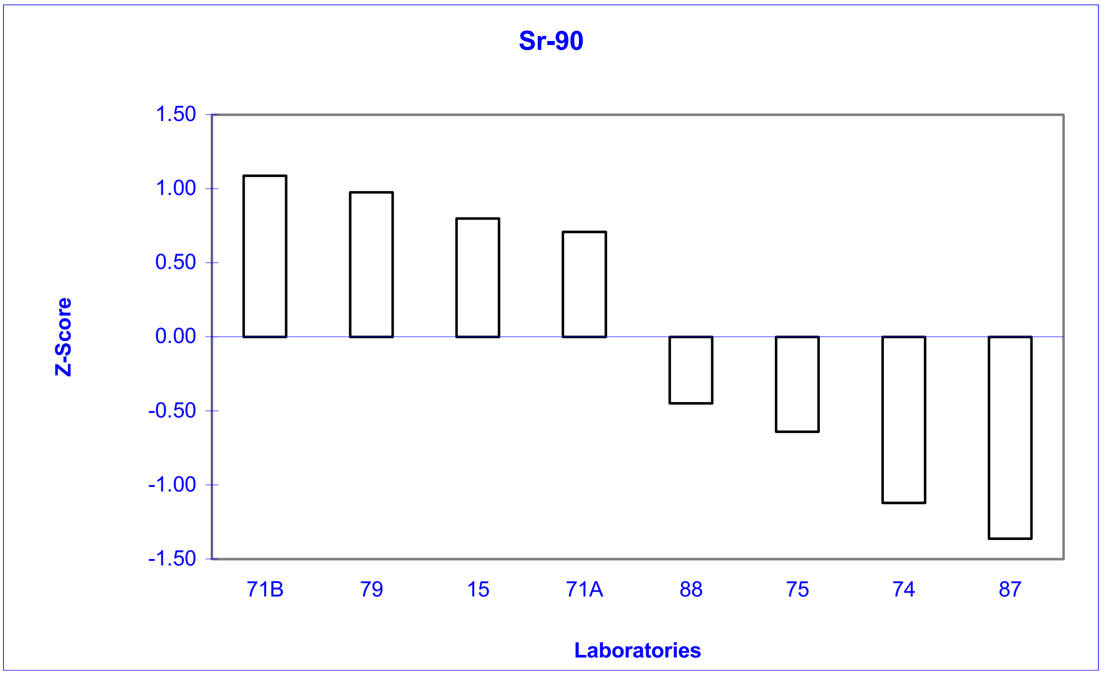

Fig.15. Z-score values for ^{90}Sr

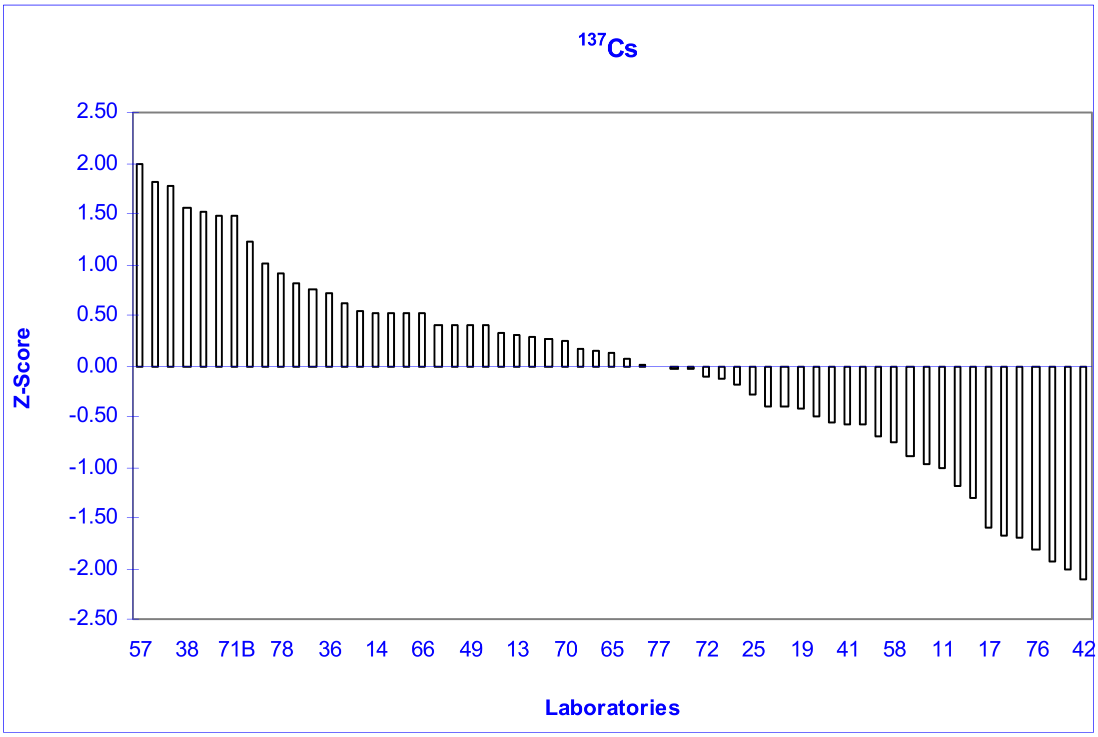

Fig.16. Z-score values for ^{137}Cs

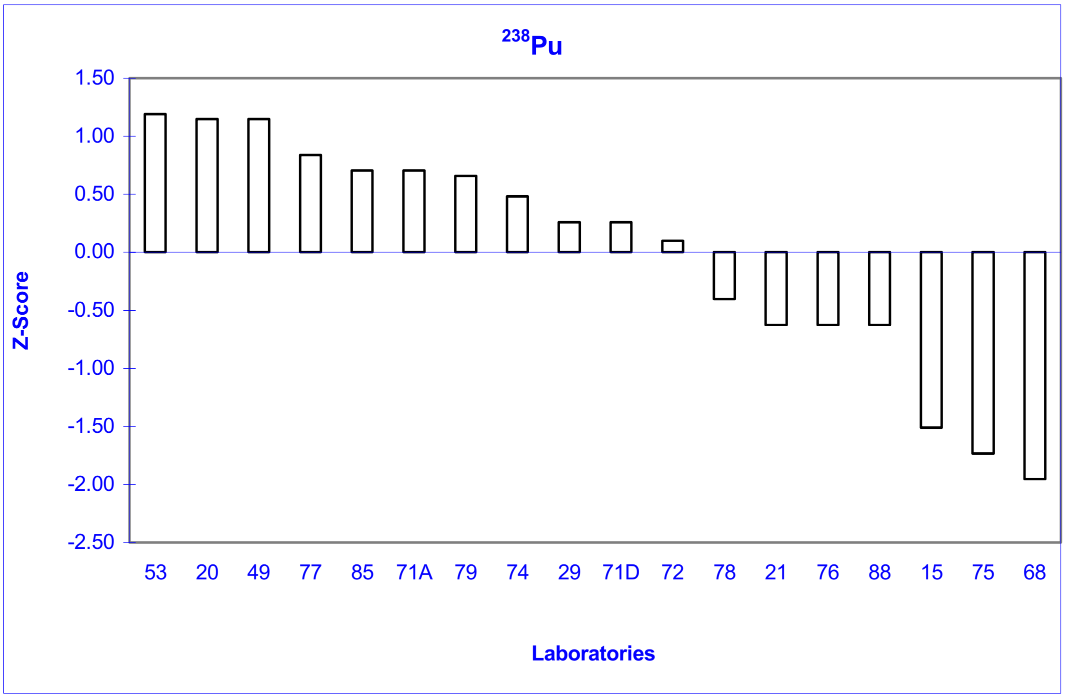

Fig.17. Z-score values of ^{238}Pu

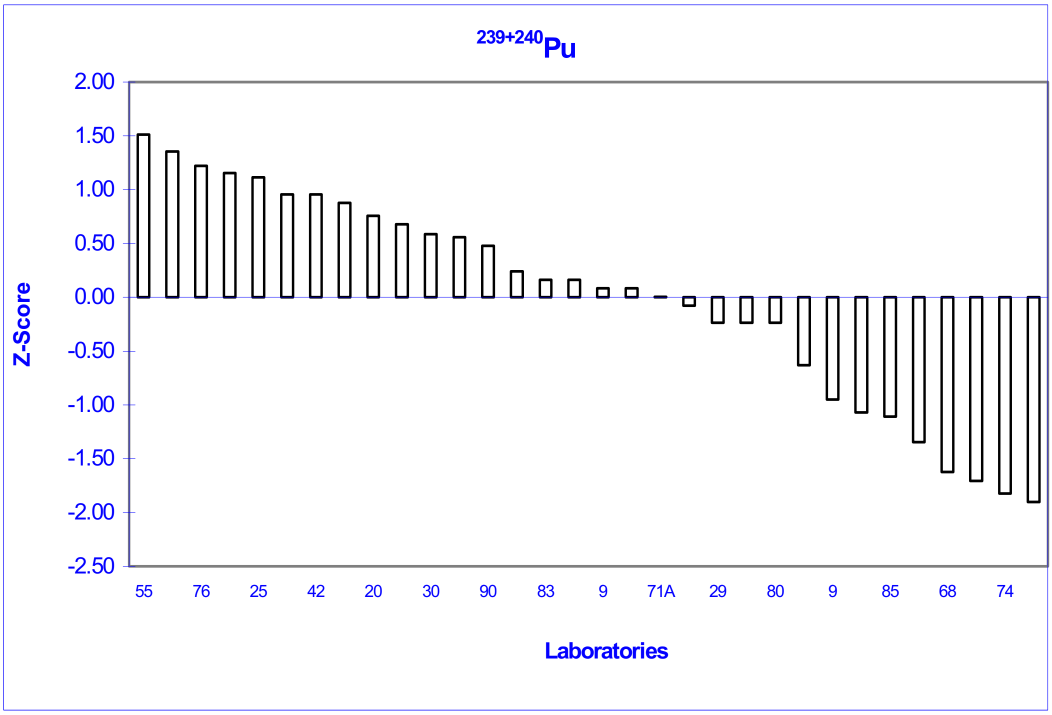

Fig.18. Z-score values of $^{239+240}$Pu

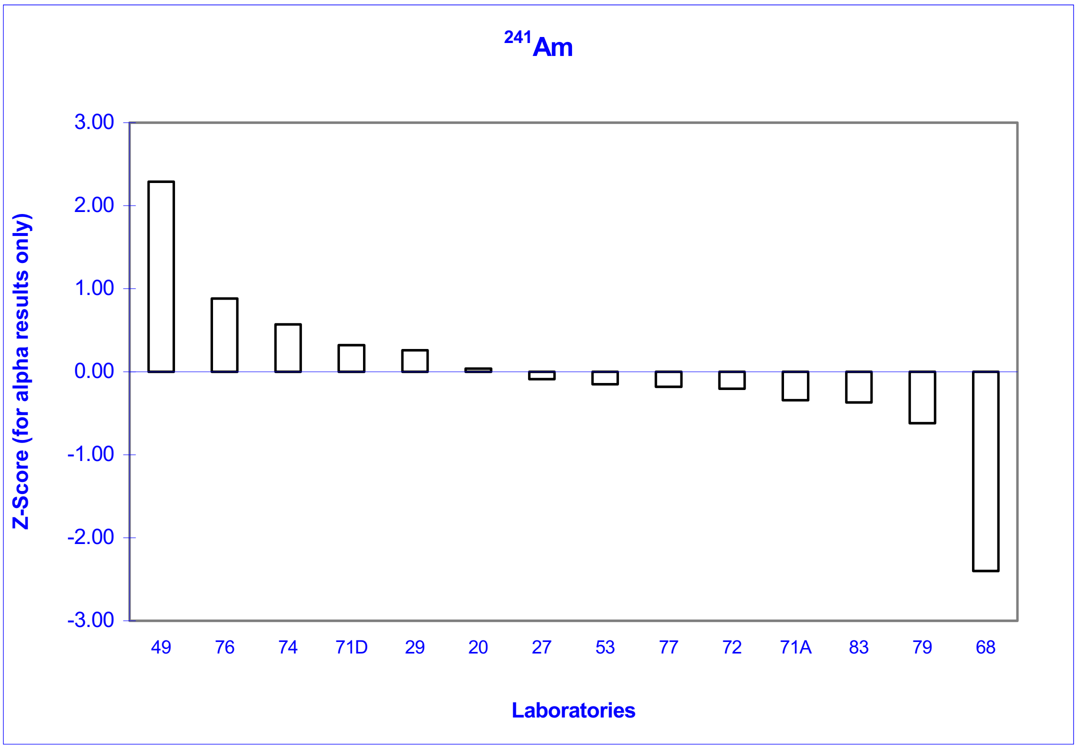

Fig.19. Z-score values of ^{241}Am

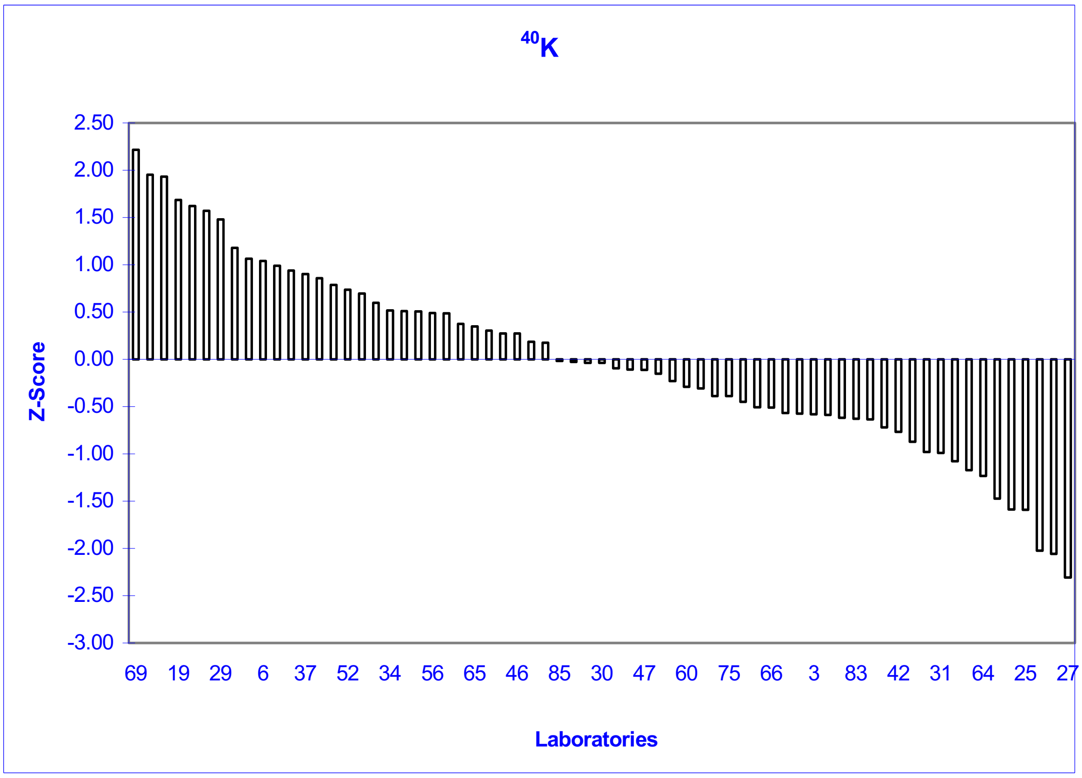

Fig.20. Z-score values of ^{40}K

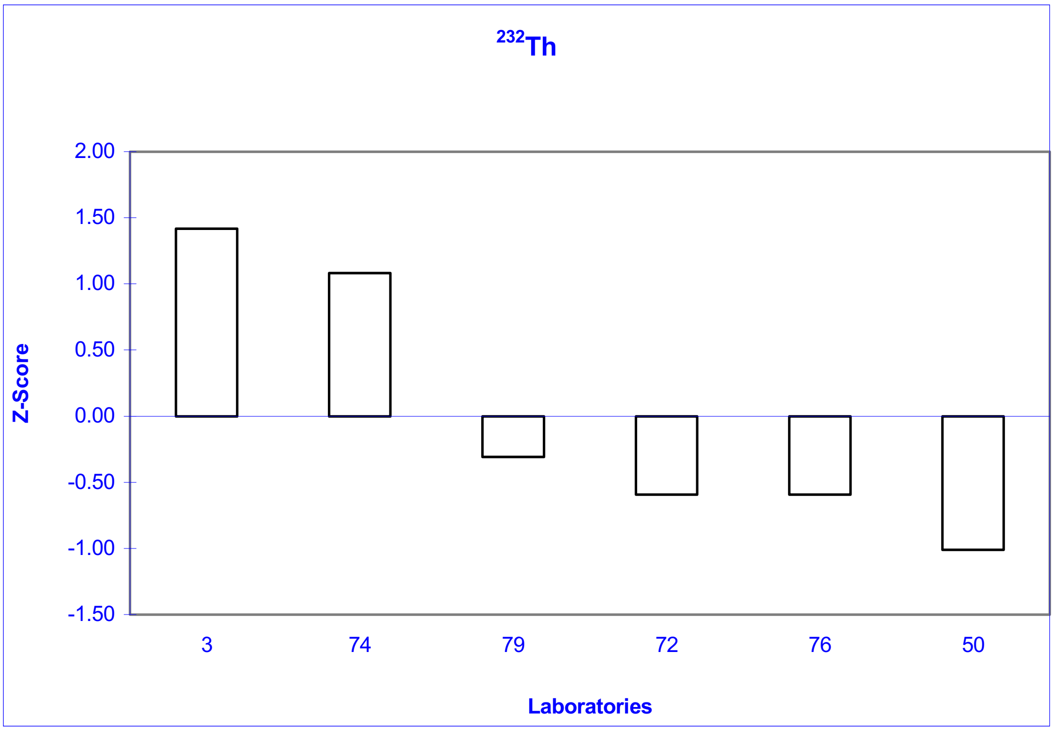

Fig.21. Z-score values of ^{232}Th

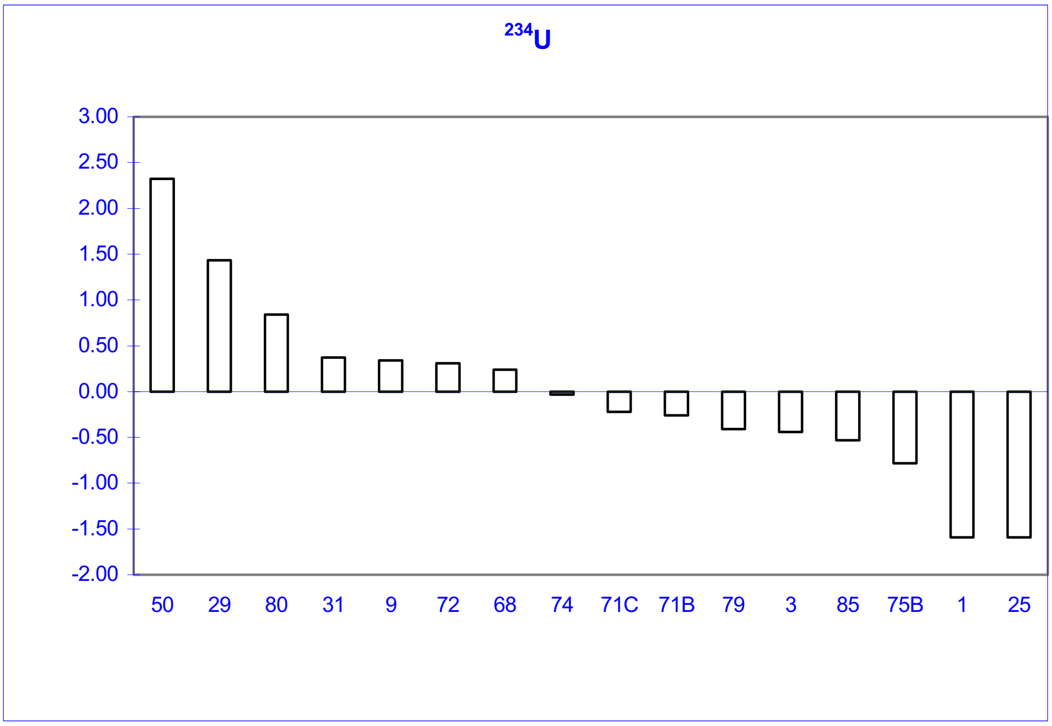

Fig.22. Z-score values of ^{234}U

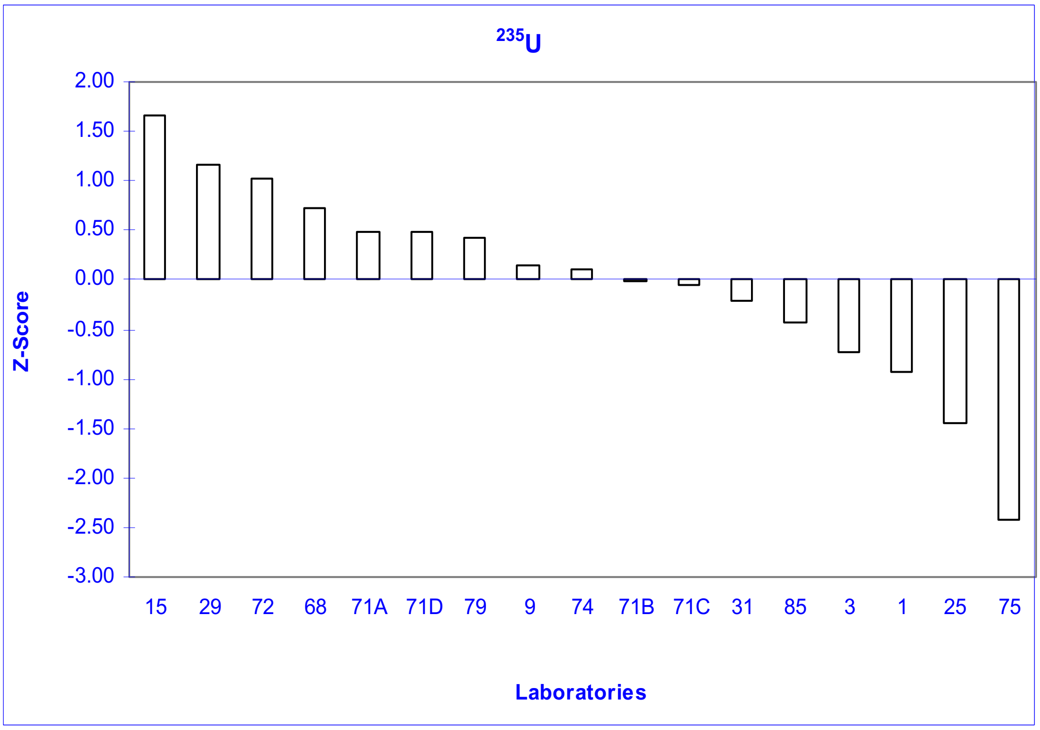

Fig.23. Z-score values of ^{235}U

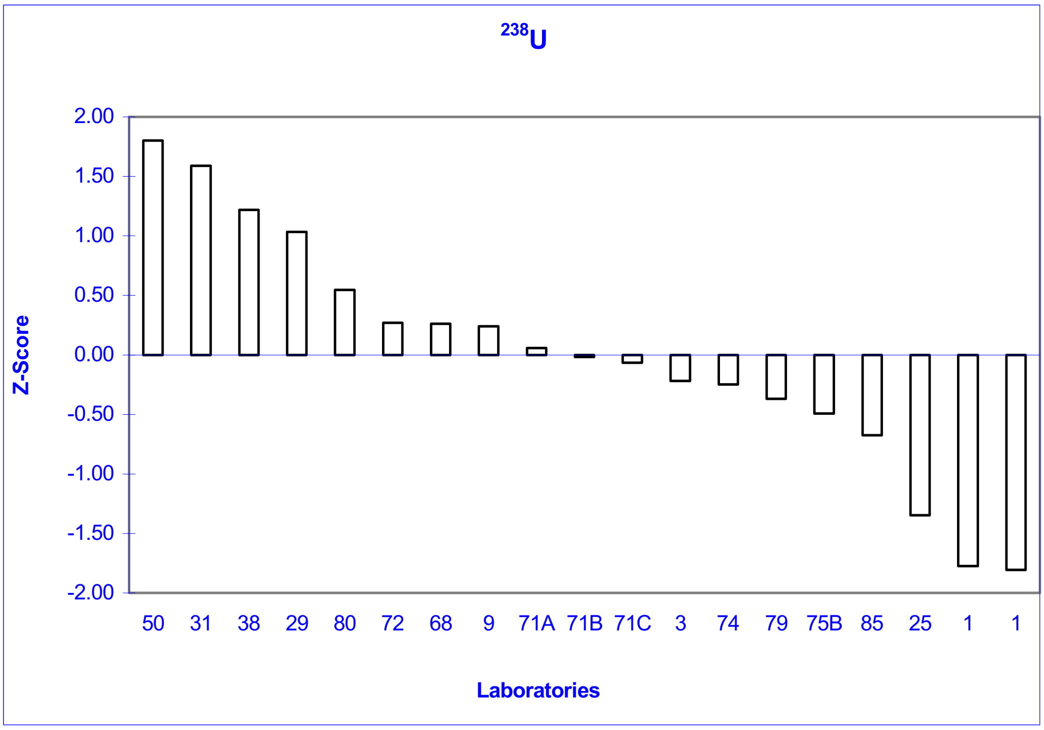

Fig.24. Z-score values of ^{238}U

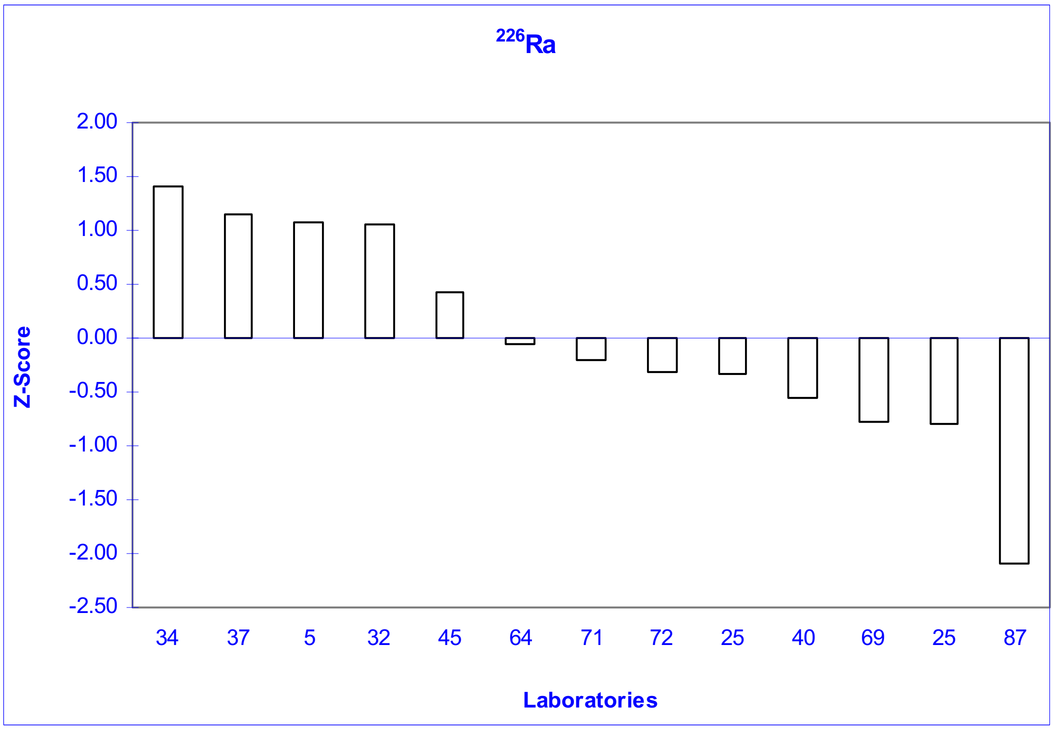

Fig.25. Z-score values of ^{226}Ra

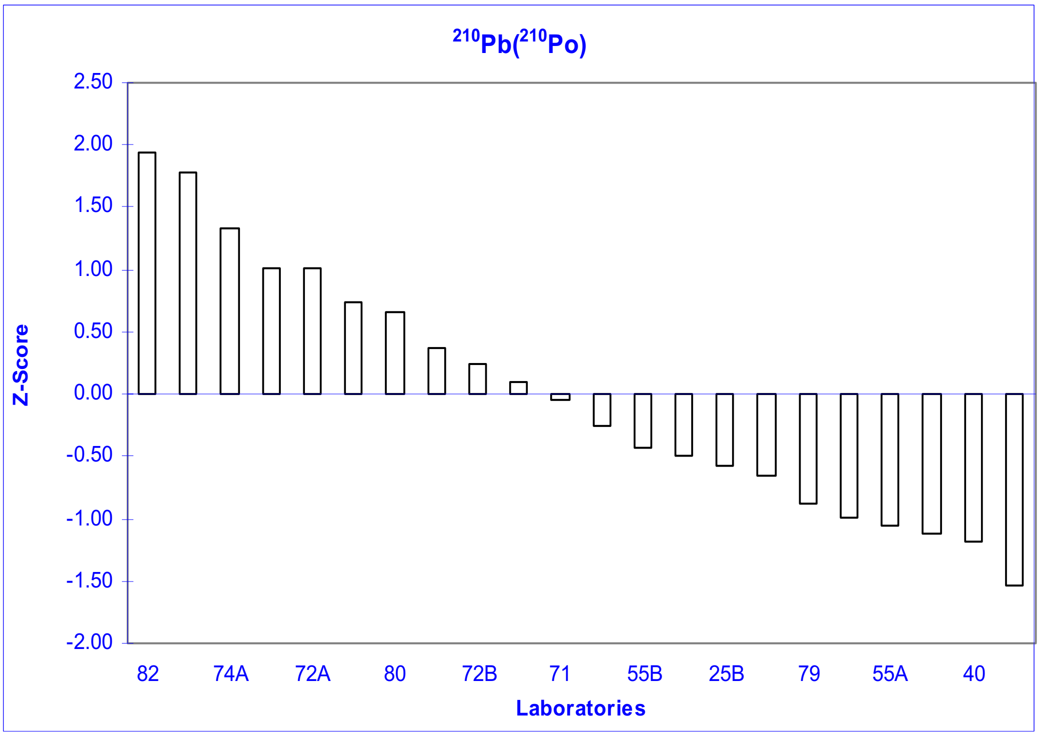

Fig.26. Z-score values of ^{210}Pb (^{210}Po)

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