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Worldwide Interlaboratory Comparison on the Determination of Trace Elements in Oyster Biota Sample IAEA-470

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WORLDWIDE INTERLABORATORY COMPARISON ON THE DETERMINATION OF TRACE ELEMENTS IN OYSTER BIOTA SAMPLE IAEA-470

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WORLDWIDE INTERLABORATORY COMPARISON ON THE DETERMINATION OF TRACE ELEMENTS IN OYSTER BIOTA SAMPLE IAEA-470

INTERNATIONAL ATOMIC ENERGY AGENCY
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FOREWORD

The primary goal of the IAEA Environment Laboratories is to help Member States understand, monitor and protect the marine environment. Thus, the major impact on marine ecosystems exerted by large coastal cities is an issue of primary concern for the IAEA and its Member States.

Marine pollution assessments depend on accurate knowledge of contaminant concentrations in various environmental compartments. Since the early 1970s, the IAEA Environment Laboratories have been assisting national laboratories and regional laboratory networks in the analysis of radionuclides, trace elements and organic contaminants in marine samples through global interlaboratory comparisons and regional proficiency tests, the production of marine certified reference materials and the development of analytical methods for analysing trace elements and organic pollutants in marine samples. The Marine Environmental Studies Laboratory in Monaco, part of the IAEA Environment Laboratories, actively assists Member States in organizing interlaboratory comparisons designed to monitor and demonstrate the performance and analytical capabilities of the participating laboratories, and to identify gaps and problem areas where further developments are needed.

The present publication summarizes the results of the IAEA-470 interlaboratory comparison on the determination of trace elements in oyster sample.

The IAEA wishes to thank the Korea Institute of Science and Technology for providing the raw sample material. The IAEA is also grateful to the the Government of Monaco for its support. The IAEA officers responsible for this publication were S. Azemard and E. Vasileva of the IAEA Environment Laboratories.

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1 INTRODUCTION

The Marine Environmental Studies Laboratory (MESL) of the IAEA's Environment Laboratories (IAEA-NAEL) has the programmatic responsibility to provide assistance to Member States' laboratories in maintaining and improving the reliability of analytical measurement results, both in trace elements and organic pollutants. This is accomplished through the provision of certified reference materials of marine origin, validated analytical procedures, training in the implementation of internal quality control, and through the evaluation of measurement performance by the organization of worldwide and regional interlaboratory comparisons. IAEAs subprogram 'Reference Products for Science and Trade' represents an important benchmark in upgrading the quality of laboratory performances and assessing the validity of the analytical methods used for marine monitoring studies in the IAEA Member States.

For more than thirty years, the MESL has conducted worldwide laboratory performance studies, also known as interlaboratory comparison [1, 2]. The results have been used to evaluate laboratory performance with respect to a wide range of organic [3, 4] and inorganic pollutants, including methylmercury [5, 6].

The goal of interlaboratory comparison is to demonstrate the measurement capabilities of laboratories participating in interlaboratory comparisons (ILCs) and targeted proficiency tests (PTs). The results from ILCs or PTs are of crucial interest for laboratories as they provide clear information of their measurement capabilities. NAEL Interlaboratory Comparison (ILC) and Proficiency Test (PT) schemes involve comparison of participant's results with an assigned value, which usually is delivered as a consensus value from the overall population of reported by participating laboratories test results.

These exercises are designed to monitor and demonstrate the performance and analytical capabilities of the participating laboratories, and to identify gaps and problem areas where further development is needed.

2 SCOPE OF THE INTERLABORATORY COMPARISON

The present interlaboratory comparison was designed to evaluate the measurement performance of participating laboratories for the analysis of trace elements in marine biota sample (oyster).

Invitation letters have been sent to 39 laboratories, previously participated in an IAEA ILC. Positive responses were received from 34 laboratories in 20 Member States and samples were duly dispatched to them.

Each participating laboratory received one bottle of the marine biota material, accompanied by an information sheet and a reporting form. Using the procedures, routinely applied in their laboratories, participants were requested to determine as many elements as possible from the following list: copper, cadmium, lead, lithium, zinc, mercury, methyl mercury nickel, vanadium, arsenic, silver, aluminium, iron, manganese, chromium, cobalt, tin and strontium. The organisers were also interested in receiving results for any other elements that the participating laboratories were willing to provide.

In total 30 laboratories (reporting 34 datasets) from 19 countries participated in this ILC and reported results for 35 elements.

The data reported by the laboratories, together with the technical and statistical evaluations of the results for each element, are included in this report. All results were treated confidentially and each laboratory was identified with a code number for anonymity.

3 DESCRIPTION OF THE MATERIAL

The interlaboratory comparison sample, distributed in this exercise was a Certified Reference Material (CRM) IAEA-470. All details about homogeneity, stability and characterization can be found in the certification report [7].

Reported measurement results were first evaluated with Kernel density plots [8]. Certified mass fraction values for selected trace elements and methylmercury, used in the evaluation process of reported results are shown in Table 1.

The assigned values for 5 additional elements, not included in Table 1, were calculated as described in the ISO 13528 standard, Annex C.21 [9]. Calculated robust mean and robust standard deviations are presented in Tables 2 and 3.

The uncertainties associated with the assigned values were calculated according to the ISO 35 standard [10]. The relative combined uncertainty of the assigned value of the CRM consists of uncertainty related to the characterization (u_{char}), between bottle heterogeneity (u_{hom}) and long–term stability (u_{stab}). Above mentioned contributions were combined to estimate the expanded uncertainty, as described in the Eq. (1).

$$U = k \times \sqrt{u_{char}^2 + u_{ho}^2 \quad + u_{stab}^2} \tag{1}$$

Where *k*: coverage factor 2, representing a level of confidence of about 95%.

u_{hom} – uncertainty on homogeneity of oyster sample is set at 3.5% and the estimation is based on the obtained results from homogeneity study.

u_{stab} - uncertainty on stability during the storage period was estimated at 3.5%.

More details on stability and homogeneity study for the IAEA-470 sample are described in the certification report [7].

u_{char} – uncertainty on characterisation of oyster sample are calculated following the Eq. (2)

$$u_{char} = 1.25 \times \frac{s^*}{\sqrt{n}} \tag{2}$$

Where s* is the robust standard deviation, calculated as described in the IS0 13528 [9] and n is the number of datasets used for calculation of the robust mean.

For Al and Ni obtained expanded uncertainties were higher than 25%. The reasons for the high uncertainties were the broad distribution of reported data for Al and insufficient homogeneity for Ni [7], respectively. As a consequence, those values were given as informative values and were not used for the evaluation of measurement performances of laboratories, participating in the IAEA-470 interlaboratory comparison.

TABLE 1. CERTIFIED VALUES FOR THE IAEA-470 OYSTER CRM

Element	Unit	Certified value[1]	Expanded uncertainty (k=2)[2]
Ag	mg kg^{-1}	1.29	0.10
As	mg kg^{-1}	11.9	0.9
Ca	mg kg^{-1}	2430	282
Cd	mg kg^{-1}	3.14	0.24
CH_3Hg	µg kg^{-1} as Hg	5.22	0.87
Co	mg kg^{-1}	0.201	0.025
Cr	mg kg^{-1}	0.965	0.113
Cu	mg kg^{-1}	146	13
Fe	mg kg^{-1}	131	12
Hg	µg kg^{-1}	21.1	2.1
Mg	mg kg^{-1}	3080	390
Mn	mg kg^{-1}	66.7	5.3
Na	mg kg^{-1}	19.7 10^3	2.3 10^3
Pb	mg kg^{-1}	0.361	0.053
Rb	mg kg^{-1}	5.14	0.59
Se	mg kg^{-1}	3.06	0.33
Sr	mg kg^{-1}	20.6	1.6
V	mg kg^{-1}	0.899	0.130
Zn	mg kg^{-1}	727	48

TABLE 2. ASSIGNED VALUE FOR THE IAEA-470 OYSTER SAMPLE

Element	Unit	Assigned value[1]	Expanded uncertainty (k=2)[2]
K	mg kg^{-1}	12720	2150
Li	mg kg^{-1}	0.532	0.094

TABLE 3. INFORMATIVE VALUE IN IAEA-470 OYSTER SAMPLE

Element	Unit	Informative value[1]	Expanded uncertainty (k=2)[2]
Al	mg kg^{-1}	72.9	19.9
Ni	mg kg^{-1}	0.849	0.291

4 EVALUATION OF ANALYTICAL PERFORMANCE

The individual laboratory performance was expressed in terms of *z*-score and Zeta-score, in accordance with the ISO 13528 [9].

The determination of target standard deviation was done on the basis of the outcome from the previous ILCs organised by the MESL for the same population of laboratories and similar sample matrix. The standard deviation for the proficiency assessment, σ_p, was fixed to 12.5 % from the assigned values. The appropriateness of this level of variability of results was confirmed by calculation of the robust standard deviation of the participants' results and the uncertainty of the assigned values for the respective measurants.

z-score, calculated following the Eq. (3), effectively expresses the difference between the mean of the laboratory and the assigned value in units of target standard deviation (σ_p).

Zeta-score, calculated according to the Eq. (4), indicates the agreement of the result of the individual participant with the ILC assigned value within the respective uncertainties. The denominator in the Eq. (4) is calculated from the combined uncertainty of the assigned value and the measurement uncertainty reported by the participating laboratory.

$$z = \frac{x_{lab} - x_{ref}}{\sigma_p} \quad (3)$$

$$\text{Zeta} = \frac{x_{lab} - x_{ref}}{\sqrt{u_{x_{lab}}^2 + u_{x_{ref}}^2}} \quad (4)$$

Where:
l_{ab} : Reported results by participant (express as the mean of multiple determination)
x_{ref}: Certified value or assigned value
σ_p: Target standard deviation
u_{xlab}: Standard uncertainty reported by participant
u_{ref}: Standard uncertainty of certified (or assigned) value

The interpretation of a laboratory's performance was evaluated according to the following generally accepted limits:

	$\lvert z \text{ or Zeta} \rvert \leq 2$	Satisfactory
$2 <$	$\lvert z \text{ or Zeta} \rvert < 3$	Questionable
	$\lvert z \text{ or Zeta} \rvert \geq 3$	Unsatisfactory

5 RESULTS AND DISCUSSION

5.1 OVERVIEW OF THE RESULTS

Thirty laboratories provided results for the analysis of the IAEA-470 oyster sample by the final deadline. Thirty-three sets of measurement results were submitted (some laboratories reported

data generated by multiple techniques) comprising 407 results for 35 different elements. *z*-scores and Zeta-scores were evaluated only for 21 elements.

From the 21 elements evaluated, 11 elements (As, Cd, Co, Cr, Cu, Fe, Hg, Mn, Pb, V and Zn) have been reported by more than 50% of participants.

For elements with more than 5 reported datasets, graphical presentations of *z*-score and Zeta-scores (except for Al and Ni) are presented in Appendix I, together with a Kernel density plot (if more than 8 data have been reported) and short summary on the statistical evaluation of results for the reported elements [8].

All reported data are presented in the Appendix II. For editorial purposes some results have been rounded to appropriate number of significant figures.

5.2 LABORATORY PERFORMANCES

5.2.1 *z*-scores:

Table 4 shows the *z*-scores of laboratories for all reported elements. The overall performance (*z*-scores) of participating laboratories is graphically presented on Figures 1 and 3.

The *z*-score compares the participant's deviation from the reference value with the target standard deviation (σp for proficiency assessment). σp is defined by the ILC organizer as the maximum acceptable standard deviation (25% in this case) of the assigned value for the investigated trace elements.

In total from the calculated 352 *z*-scores, 93% were $|z| \leq 2$, 96% $|z| < 3$, and only 3% were considered as unsatisfactory with $|z| > 3$.

Among 33 datasets, 17 (52%) were with $|z| \leq 2$ for all reported results and 25 (76%) with $|z| < 3$. All participants reported at least 50% of their measurement results with $|z| \leq 2$.

As shown on Figure 3 the part of accepted measurement results was 65% or higher for each of the evaluated elements. Methyl mercury, total mercury and lead appeared to be the elements reported with the highest number of unsatisfactory *z*-scores. This shows that the accurate determination of those elements in biological material is still challenging analytical task, particularly at low mass fraction levels.

This is underlined by the fact that a large part (72%) of $|z| > 3$ is positive, demonstrating positive bias. These biased results may arise from the contamination during sample preparation (e.g., digestion) step or from the instrumental steps. The laboratories concerned should carefully check sample preparation procedures (e.g., quality chemical reagents used) and try to improve their working laboratory environment. Laboratories should also develop an effective scheme for cleaning of the lab ware and regularly control this process.

Erroneous calibration standards could be another source of bias. It is important to note that losses in the working standard solutions at low concentration level can be the reason for the overestimation of the concentrations of investigated elements. Only standard solutions (CRMs) with stated SI traceability should be used for calibration purposes and in addition they should not be stored for an extended period of time.

Laboratories with questionable and unsatisfactory results should also carefully check all analytical procedures, laboratory equipment and instrumentation.

5.2.2 Zeta-scores:

Table 5 shows the overall performance (Zeta-scores) of laboratories for different trace elements. The overall performance (Zeta-scores) of participating laboratories are graphically presented on Figures 2 and 4.

The Zeta-score shows the level of agreement of the laboratory result with the assigned value within the respective uncertainties. The denominator in the Eq. (2) is the combined uncertainty of the assigned value and the uncertainty of measurement result, reported by the individual laboratory.

Five laboratories (15%) didn't report uncertainties and Zeta-scores for their results were not calculated.

As it can be easily seen in Figures 1 and 2, the comparison of laboratories performances evaluated with *z*-score and Zeta-score clearly indicate that the number of unsatisfactory Zeta-scores is considerably higher than the number of unsatisfactory *z*-scores (3% for *z*-scores and 8% for Zeta-scores). Only 10 laboratories (30%) could report 100% of their results with $|z|$ and $|$ Zeta-scores ≤ 2. As the Zeta-score is the parameter, reflecting all parts of the measurement procedure, laboratories with unsatisfactory Zeta-scores should invest additional efforts in the proper evaluation of measurement uncertainty of reported measurement results. Overall, obtained results indicates that they are still remaining problems with the realistic estimation of measurement uncertainty.

If should be mentioned here that an unsatisfactory Zeta-score can be also caused by wrong estimation of the mass fraction of the respective trace element.

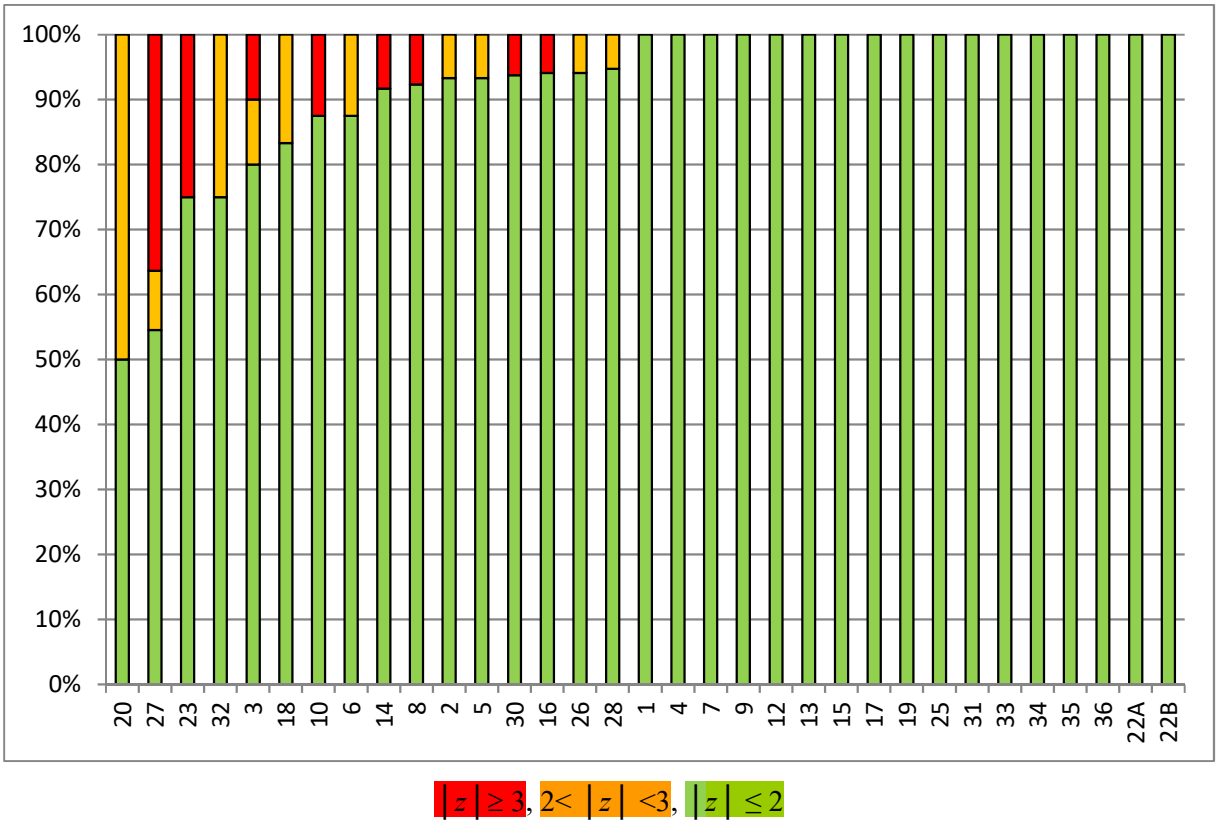


FIG. 1. The z-scores of results reported by the participants per laboratory.

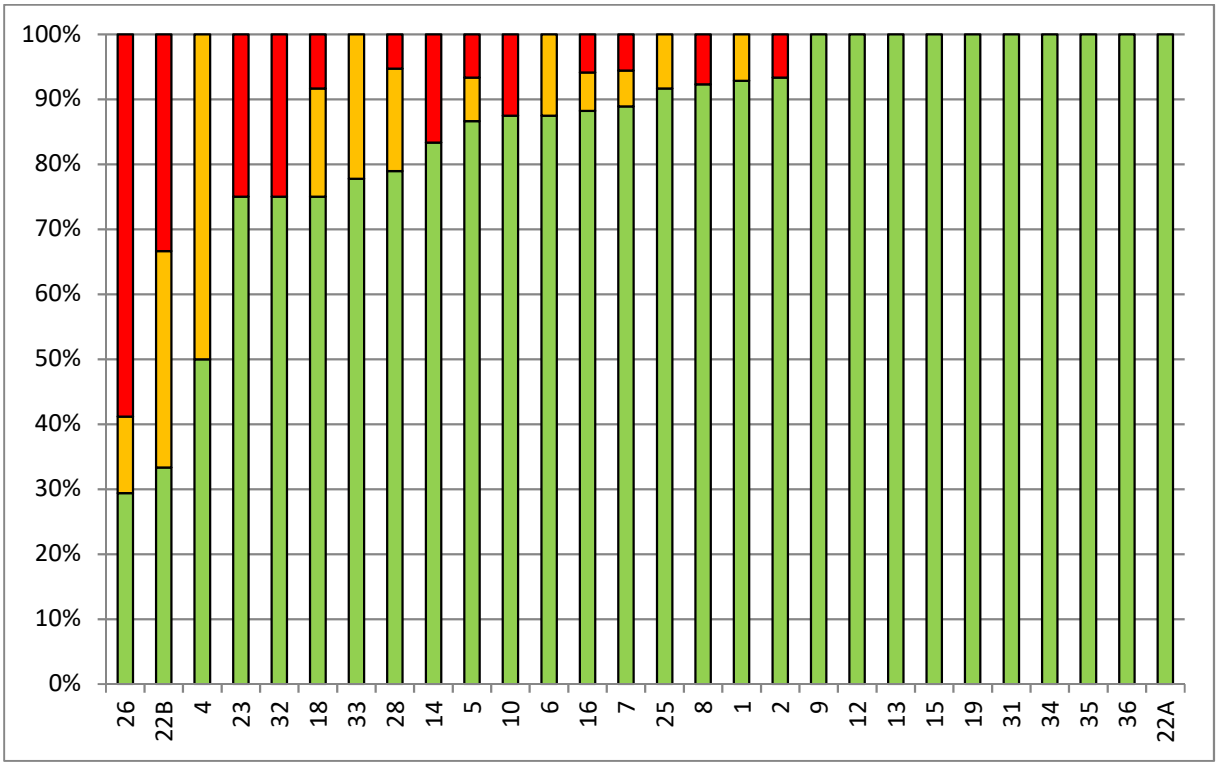


FIG. 2. The Zeta-scores of results reported by the participants per laboratory.

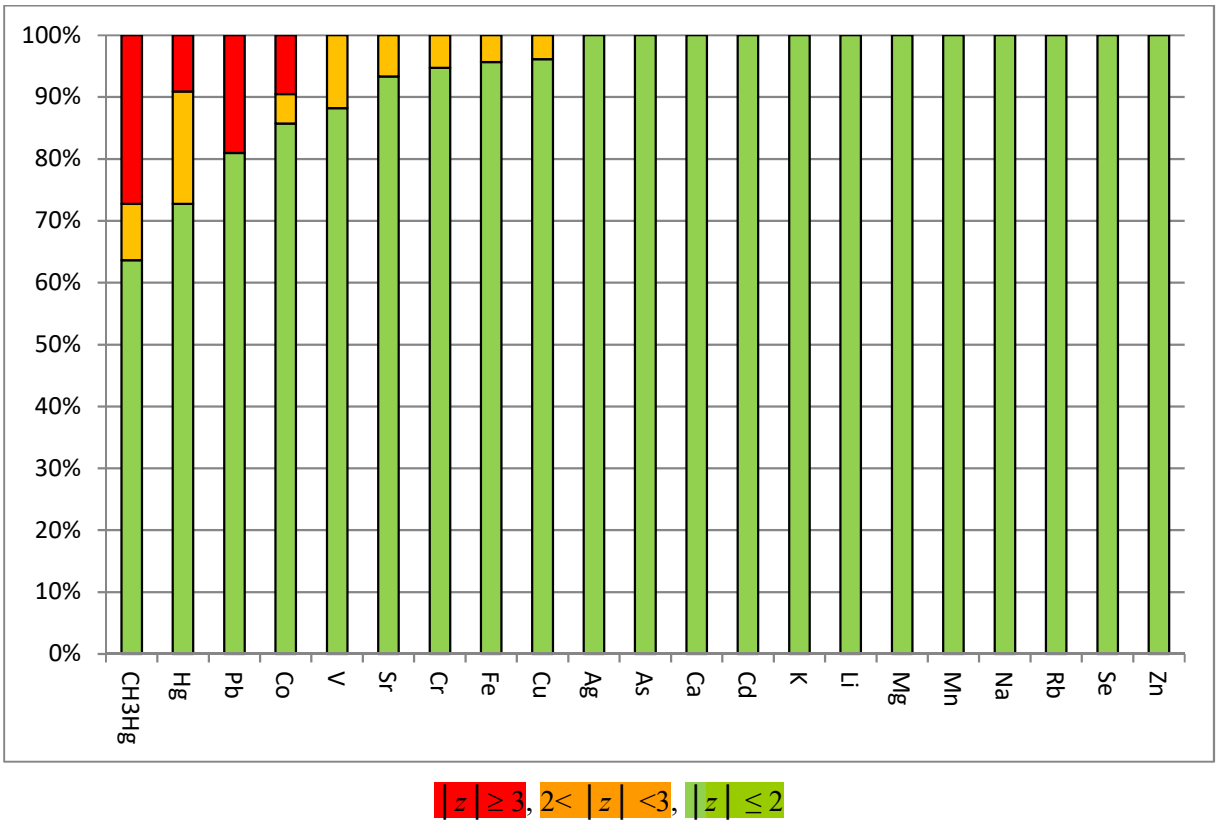


FIG. 3. The z-scores of results reported by the participants per element.

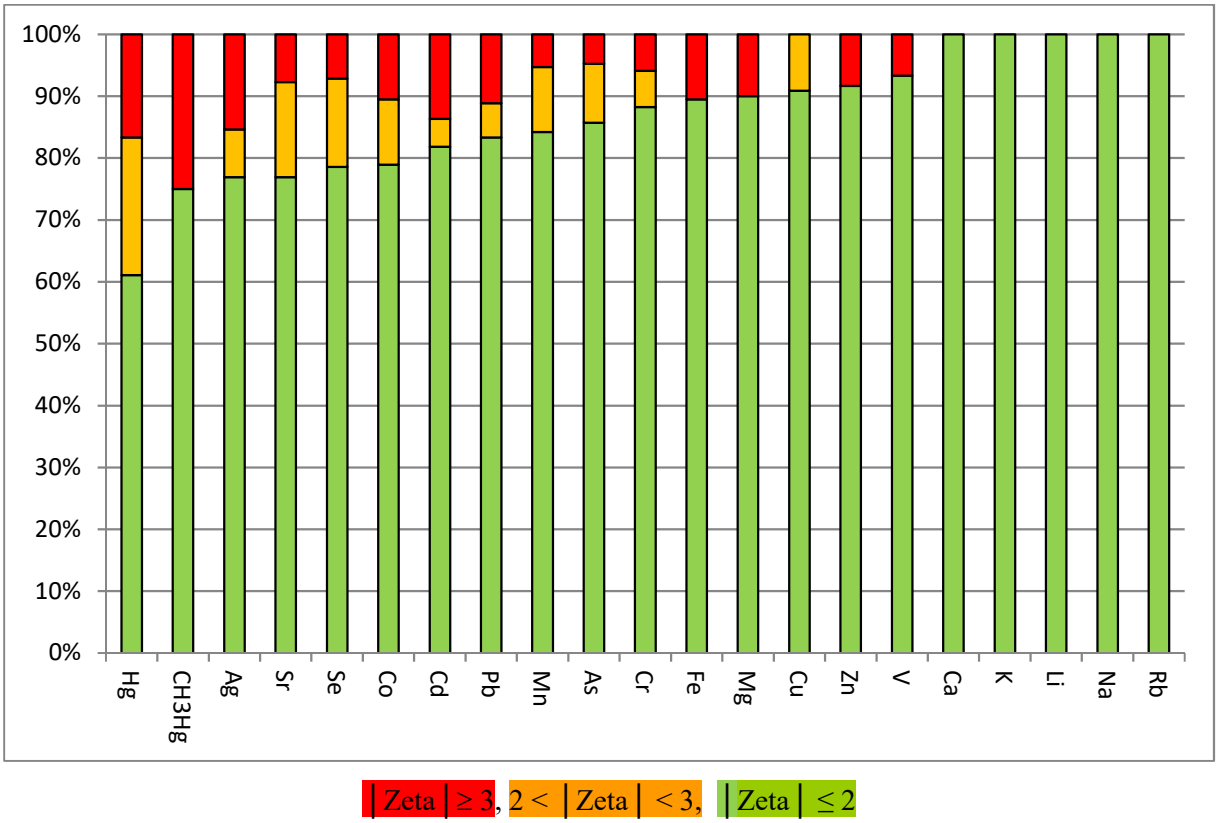


FIG. 4. The Zeta-scores of results reported by the participants per element.

TABLE 4. OVERALL ASSESSMENT OF LABORATORIES PERFORMANCE (*z*-SCORE) BY ELEMENTS

Lab code	Ag	As	Ca	Cd	CH_3Hg	Co	Cr	Cu	Fe	Hg	K	Li	Mg	Mn	Na	Pb	Rb	Se	Sr	V	Zn
1		0.47	0.63	0.08		-0.04	0.29	-0.11	0.37	-1.18			0.49	0.94		-0.47		1.75		0.19	0.51
2	0.11	0.31		0.45		0.48	2.12	0.35	-0.41			0.04	0.33	0.00	-0.83	0.33			0.19	0.69	0.32
3		-0.61		0.10			1.50	0.88	0.43					0.61		14.47			-1.38	-2.57	0.83
4		-0.94		-0.10		-1.55		-0.88						-0.53							-0.48
5		0.34	0.33	0.36	0.14	-0.08	-1.72	0.00	-0.37	-2.39			-0.13	-0.24		-0.13		0.26		-0.16	0.30
6		-0.07		-0.56				-0.16	0.00	2.24				-0.05		-0.62					-0.50
7	-1.77	0.47	0.14	0.31		0.28	-0.07	-0.41	0.07	-1.18	0.38		0.08	0.77	0.63	-0.71		-0.16	1.01	0.32	0.06
8	0.43	-0.09	-0.49		-3.09	-0.69	0.10		0.12	-0.45	-0.89				0.35		-0.02	0.41			0.32
8B										-0.38											
9	-0.87	-1.14	-0.62	-1.25		-0.16	-1.01	-0.82	-0.12	-0.04		-1.75	-0.29	-0.65		-0.13		-1.05	-0.12	-0.06	-1.32
10		-0.07		-0.54			-1.20	0.00	0.24					-0.20		12.61					-1.42
12					0.11					-0.91											
13	-0.33	-0.32		0.31		-0.09	0.00	-0.07	-0.46					0.14		0.20	-0.11	0.14	-0.22	-0.47	0.51
14		-0.47	0.35	-1.73		6.57	-1.04		-0.49	0.99					-0.35	0.44	-0.53	-0.42			0.17
15		0.58	0.39	0.81	1.96	0.18	-0.51	0.48	0.49	0.47	1.39		0.63	0.79	1.02	0.20	0.42	0.36	-0.04	0.58	0.56
16	-0.25	-0.74	-0.40	0.38		-0.92	-0.97	0.38	0.24	-5.57		-0.87	-0.31	0.16		-0.31		-0.37	-0.82	-0.69	0.15
17				-1.35				0.48	1.35	0.17				-0.32							
18		0.79		0.31		-0.06	-0.35	0.01	-0.23	2.30		1.05		1.70					2.54	0.18	0.36
19	0.12			0.23				0.58		-0.04						0.20					0.75
20					1.26					-2.39											
22A	-0.68	0.07				-0.76	0.21		-0.19						-0.04			-0.84			-0.30
22B				-1.71		1.75		-0.33								-1.13			-1.01		-1.17
23		0.47		-0.10				0.62								3.08					
25	1.12	0.07	-0.95						0.31		-0.96		-0.69	-0.44	0.23		0.25	0.37		-0.88	0.14
26	1.55	1.09	0.47	1.45	2.90	0.73	-0.02	0.82	1.36	1.47			1.58	1.34		0.43		1.65	1.61	0.53	0.95
27				-1.50	6.13	6.73	0.12	-2.73	-1.77	5.27				-1.72		-3.12				-0.35	-1.14

TABLE 4. OVERALL ASSESSMENT OF LABORATORIES PERFORMANCE (*z*-SCORE) BY ELEMENTS (cont.)

Lab code	Ag	As	Ca	Cd	CH_3Hg	Co	Cr	Cu	Fe	Hg	K	Li	Mg	Mn	Na	Pb	Rb	Se	Sr	V	Zn
28	-0.56	-0.85	-0.12	-0.32		-0.84	0.35	-0.51	2.30	-0.35	-0.54	0.92	-0.19	-0.78	-0.08	-1.43		-1.09	-0.67	-0.17	-0.39
30		1.75	0.16	0.00	3.80	-1.23		0.16	-0.31	-0.53	0.61		-0.05	0.07	0.97	0.42		0.34	-0.12		-0.10
31				-0.18				-0.71								0.75					0.02
32				-0.28		2.40		-0.21	0.64					0.34					0.43	2.34	-0.65
33	-0.28	-0.01		0.97			-1.57	-0.17		-1.74				-0.19						-1.19	0.33
34		0.47			-0.18	-1.47	0.54	0.22	0.00	0.53		0.37		0.79				-0.18	0.19	-1.01	0.13
35	0.12	0.27		0.69	-0.64	0.36		0.38		0.08				0.02		0.02			0.66		
36					0.21					1.02											

TABLE 5. OVERALL ASSESSMENT OF LABORATORIES PERFORMANCE (ZETA-SCORE) BY ELEMENTS

Lab code	Ag	As	Ca	Cd	CH3Hg	Co	Cr	Cu	Fe	Hg	K	Li	Mg	Mn	Na	Pb	Rb	Se	Sr	V	Zn
1		0.73	0.90	0.17		-0.05	0.32	-0.23	0.58	-1.14			0.77	1.70		-0.52		2.74		0.20	1.15
2	0.03	0.71		1.10		0.84	3.45	0.82	-0.87			0.06	0.59	0.01	-1.50	0.54			0.51	1.04	0.85
3																					
4		-2.91		-0.25		-2.67		-2.02						-1.28							-1.56
5		1.07	0.71	1.11	0.20	-0.15	-2.51	0.00	-0.90	-5.87			-0.24	-0.75		-0.22		0.48		-0.20	0.96
6		-0.18		-1.58				-0.44	0.00	2.61				-0.13		-0.98					-1.43
7	-3.88	1.05	0.24	0.56		0.49	-0.12	-0.94	0.18	-2.14	0.49		0.14	1.72	1.14	-0.98		-0.36	1.96	0.48	0.13
8	0.81	-0.20	-0.84		-4.41	-1.17	0.14		0.26	-1.03	-1.10				0.60		-0.04	0.75			0.81
8B										-0.66											
9	-0.59	-0.64	-0.51	-0.72		-0.10	-1.20	-1.03	-0.11	-0.01		-1.22	-0.23	-0.56		-0.11		-0.58	-0.09	-0.05	-1.83
10		-0.06		-0.54			-1.24	0.00	0.22					-0.20		4.77					-1.64
12					0.10					-1.21											
13	-0.28	-0.38		0.35		-0.10	0.00	-0.09	-0.68					0.27		0.17	-0.14	0.15	-0.33	-0.50	0.74
14		-1.16	0.58	-4.42		10.31	-1.52		-1.33	1.11					-0.73	0.43	-0.95	-0.62			0.38
15		0.20	0.50	1.02	1.64	0.21	-0.40	0.64	0.48	0.35	1.31		0.74	1.16	1.25	0.07	0.46	0.37	-0.06	0.31	0.86
16	-0.46	-2.41	-0.62	0.67		-1.41	-1.56	0.65	0.42	-13.27		-1.07	-0.46	0.28		-0.42		-0.59	-1.59	-0.96	0.28
17																					
18		1.58		0.62		-0.08	-0.56	0.02	-0.41	3.21		1.26		2.86					2.83	0.22	0.65
19	0.18			0.34				1.03		-0.04						0.22					1.46
20																					
22A	-1.48	0.12				-1.28	0.19		-0.40						-0.07			-1.44			-0.90
22B				-4.99		2.86		-0.84								-1.73			-2.91		-3.88
23		1.10		-0.26				1.26								3.47					
25	2.94	0.18	-1.13						0.65		-1.38		-1.21	-0.54	0.46		0.49	0.76		-1.46	0.43
26	4.74	3.26	0.98	4.41	3.80	1.44	-0.03	2.21	3.62	2.84			3.03	4.04		0.72		3.45	4.80	0.90	3.40

TABLE 5. OVERALL ASSESSMENT OF LABORATORIES PERFORMANCE (ZETA-SCORE) BY ELEMENTS (cont.)

Lab code	Ag	As	Ca	Cd	CH3Hg	Co	Cr	Cu	Fe	Hg	K	Li	Mg	Mn	Na	Pb	Rb	Se	Sr	V	Zn
27																					
28	-1.67	-1.69	-0.25	-0.91		-1.31	0.70	-1.30	5.96	-0.87	-0.75	1.20	-0.36	-2.31	-0.17	-2.37		-2.51	-1.84	-0.28	-1.41
30																					
31				-0.57				-1.91								0.60					0.08
32				-0.65		3.34		-0.45	1.00					0.73					0.94	3.29	-1.64
33	-0.32	-0.03		2.38			-1.29	-0.39		-2.90				-0.37						-1.16	0.81
34		0.64			-0.20	-1.82	0.57	0.39	0.00	1.01		0.38		1.02				-0.20	0.27	-1.12	0.27
35	0.22			1.16	-0.94	0.52		0.63						0.04		0.03			1.08		
36					0.24					1.41											

5.3 ANALYTICAL METHODS

As can be concluded from Figure 5, a wide range of analytical methodologies was used to provide results for the determination of trace elements mass fractions in oyster IAEA-470 sample. Generally, they can be divided to three groups: non-destructive techniques (NAA); plasma spectrometric methods (ICP-MS and ICP-OES) and atomic absorption spectrometry methods, representing 12%, 65% and 15% respectively. Abbreviation used in the figure 5 and appendix III are shown in Table 6.

TABLE 6. INSTRUMENTAL TECHNIQUES USED IN THE IAEA-470 INTERLABORATORY COMPARISON AND THEIR ABBREVIATIONS

Method code	Instrumental technique
Method code	Instrumental technique
CV-AAS	Cold Vapour Atomic Absorption Spectrometry
CV-AFS	Cold Vapour Atomic Fluorescence Spectrometry
ET-AAS	Electro Thermal Atomic Absorption Spectrometry
F-AAS	Flame Atomic Absorption Spectrometry
GC-AFS	Gas Chromatography Atomic Fluorescence Spectrometry
GC-ECD	Gas Chromatography Electron Capture Detector
HPLC-AFS	High Performance Liquid Chromatography Atomic Fluorescence Spectrometry
ICP-MS	Inductively Coupled Plasma Mass Spectrometry
ICP-OES	Inductively Coupled Plasma Optical Emission Spectrometry
ID-ICP-MS	Isotope Dilution Inductively Coupled Plasma Mass Spectrometry
NAA	Neutron Activation Analysis
Solid AAS	Solid Atomic Absorption Spectrometry
SV	Striping voltammetry

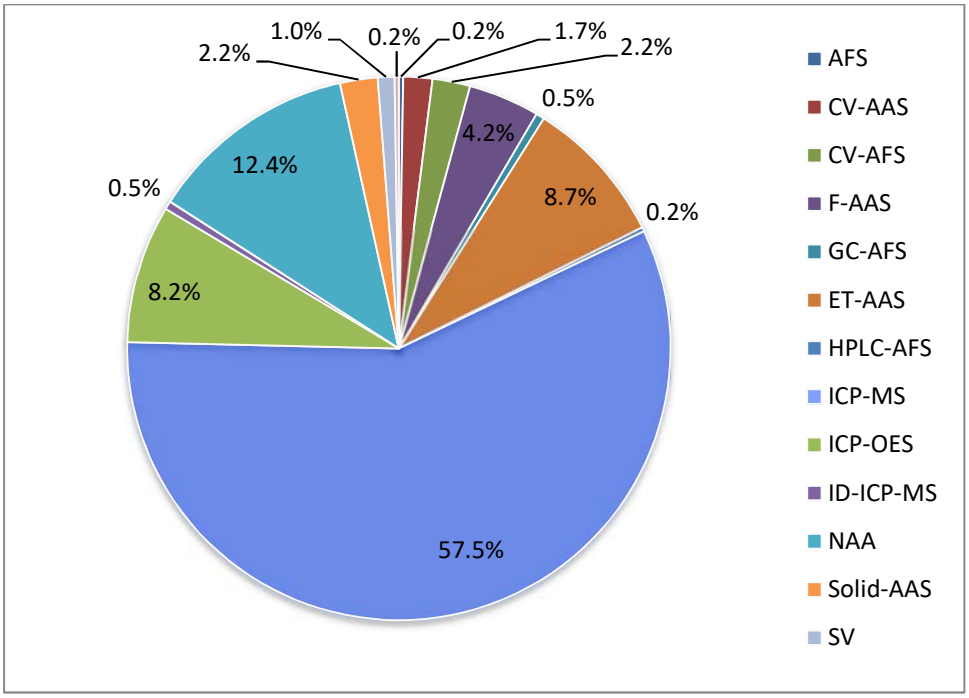


FIG. 5. Graphical distribution of instrumental techniques.

5.4 SAMPLE TREATMENT, IMPACT OF WATER CONTENT, USE OF CRM AND RECOVERY CORRECTIONS

In the IAEA-470 interlaboratory comparison, a wide range of sample preparation methods and instrumental set-ups were applied.

All laboratories stated using a CRM for quality control. Only one participating laboratory didn't report the results for the analysed CRM as requested in the reporting form. Five laboratories reported quality control results for selected elements only. An important principle for the selection of reference material by laboratories is the principle for matrix and concentration range matching. CRMs used in the IAEA-470 ILC were well selected, as all laboratories used biota of marine origin (i.e. TORT-2 lobster hepatopancreas from NRCC, IAEA 452 scallop from IAEA, NIST 1566 oyster from NIST etc).

Only 6 laboratories (18%) implemented correction for recovery for at least part of their reported results. 14 participants reported results for the obtained recovery rates. Most of recoveries reported were in the range of 100 ± 15%.

High proportion of the laboratories that didn't correct for recovery, still reported satisfactory results. They have correctly estimated that the recovery achieved by them was not significantly different from 100%.

IAEA-470 sample was subject to freeze drying as part of its preparation procedure. At the time of bottling, the moisture content of the oyster sample was around 6.5%. Depending on local storage conditions and humidity levels the ILC sample might absorb moisture from the laboratory environment. Consequently, the users were advised to make a separate determination of the moisture content in the oyster sample. As the moisture is operationally dependent parameter, a recommended procedure on moisture content determination in the test sample was preliminary provided to the participants [10]. The moisture content reported by the

laboratories that applied correction factor was in the range of 0.02 to 13%. Only 1 participant didn't correct for moisture content.

6 RECOMMENDATIONS

Participants got recommendation to review their data element by element, appraising whether the *z*-scores and Zeta-scores are less than or equal to 2. The use of *z*-scores and Zeta-scores evaluation parameters will help to identify systematic errors in the measurement results (*e.g.* from calibration, reagent contamination or incomplete digestion) and should ultimately improve data quality.

Some laboratories still need to improve quality assurance / quality control procedures. Interlaboratory comparisons represent only one aspect of data quality assurance and can only provide occasional indicators of data reliability. Another valuable approach is through the regular analysis of certified reference materials, and by plotting the resulting data on a quality control chart. This provides continuous feedback to the analyst and is an essential tool for monitoring data quality and assuring acceptable results in future exercises.

A full catalogue of available IAEA reference materials is published regularly and can be consulted on the IAEA website: http://www.iaea.org/programmes/aqcs

7 CONCLUSIONS

Although the overall performance of the laboratories is satisfactory, it should be pointed that for elements present at low to ultra-low concentration levels (i.e Pb, Hg, MeHg) in oyster is problematic. It is a subject of concern as these elements are toxics and the accurate determination of their concentration in seafood is of crucial importance.

An extra effort is still needed in the evaluation of measurement uncertainties, associated with the results, since the number of unsatisfactory Zeta-scores was systematically higher than the number of unsatisfactory *z*-scores for reported trace elements. The uncertainty associated with measurement results is of paramount importance in the frame of different regulations and international agreements, so it is fundamental for one laboratory to be able to report a sound uncertainty statement.

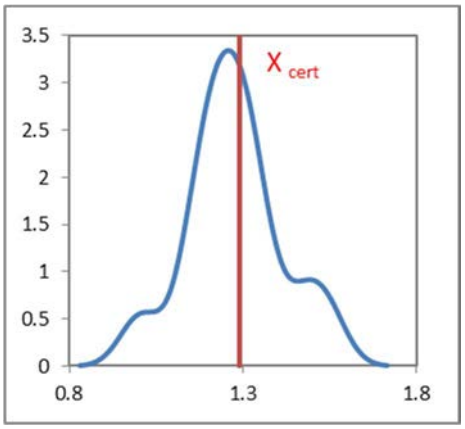
In general, laboratories should keep in mind that uncertainties evaluated only on the basis of measurement precision are frequently underestimated. In many cases, they just reflect variations coming from the measurement step and usually do not include uncertainty contributors, coming from other sources, namely uncertainty on recovery, procedural blank, moisture content etc.

The sample used in IAEA-470 interlaboratory comparison was further certified by expert laboratories for the mass fractions of 19 trace elements. Participants in the ILC could retain their ILC samples and request from the IAEA reference sheets with the certified mass fractions value of the IAEA-470 oyster sample.

APPENDIX I
PERFORMANCE EVALUATION BY ELEMENT IN IAEA-470

Reported data for Ag in IAEA-470

Kernel density Plot.



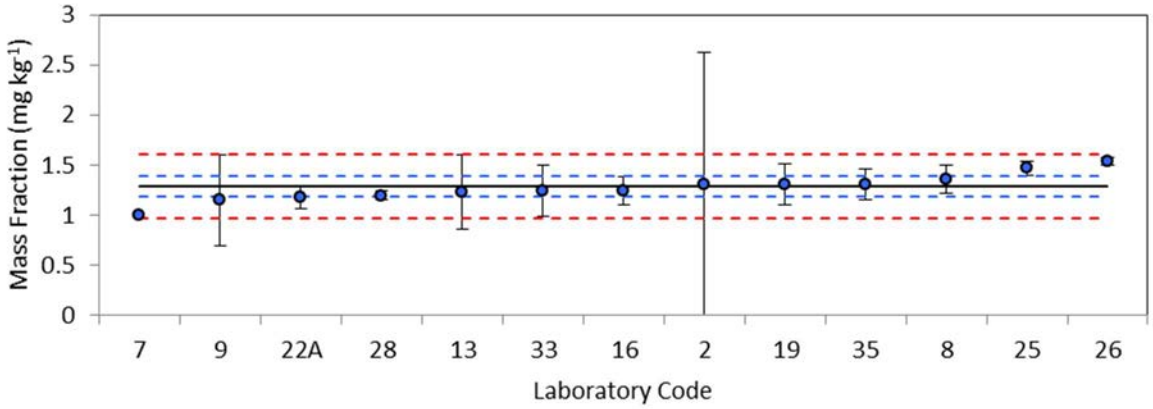
Summary of results:

	Satisfactory	Questionable	Unsatisfactory
z-score	100%	0%	0%
Zeta-score	77%	8%	15%

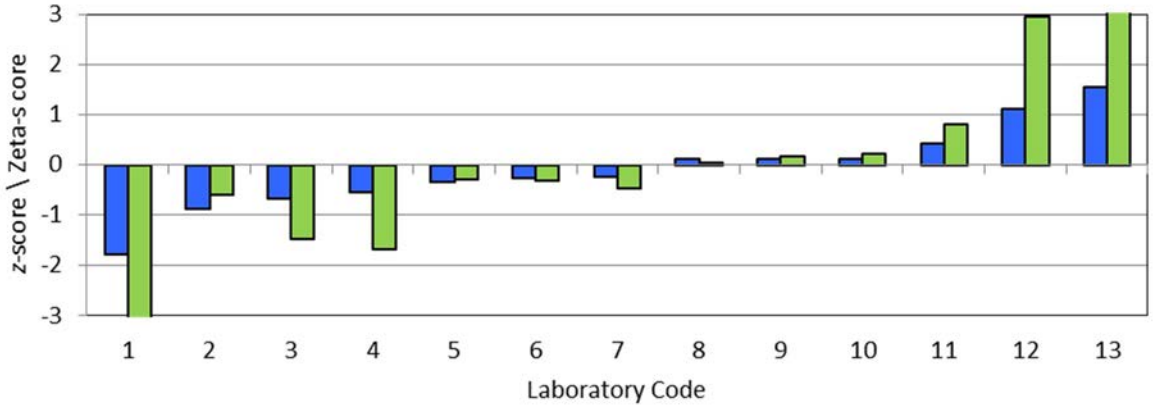
X_{cert}:	1.29 mg kg^{-1}
U_{cert} (k=2) :	0.10 mg kg^{-1}
$2\sigma_p$:	0.32 mg kg^{-1}
Number of results:	13
Number of method:	3

Reported results and expanded uncertainties.

— X_{Cert} ; $X_{lab} \pm U_{lab}$; ---- $X_{Cert} \pm 2\sigma_p$; ---- $X_{Cert} \pm U_{Cert}(k=2)$

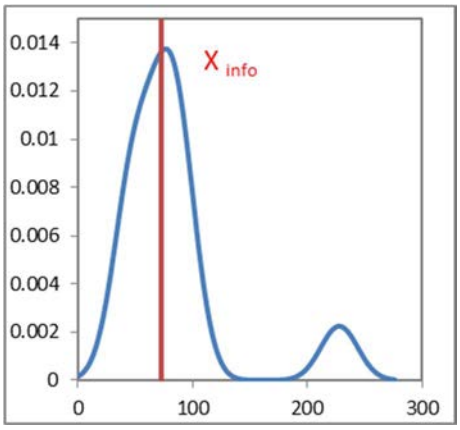


Performance evaluation: *z*-score Zeta-score



Reported data for Al in IAEA-470

Kernel density Plot.

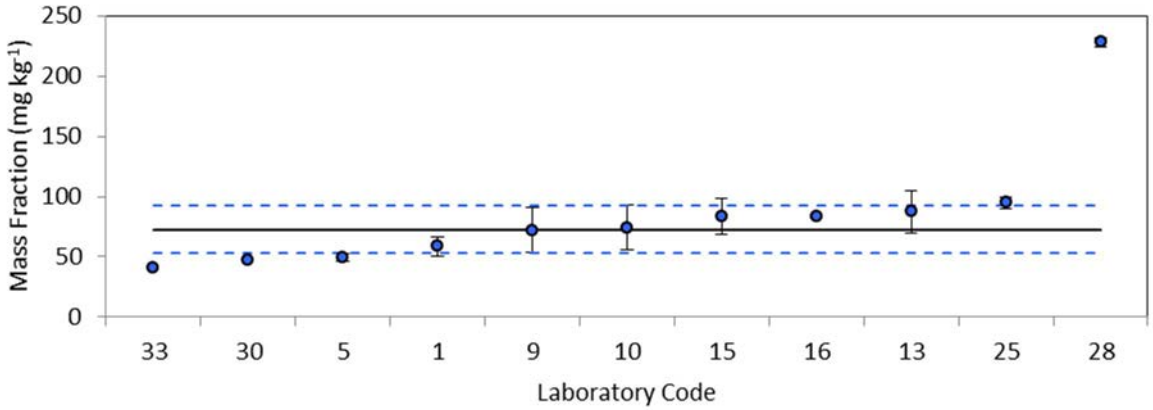


Summary of results:

X_{info}:	72.9 mg kg^{-1}
U_{info} (k=2) :	19.9 mg kg^{-1}
Number of results:	11
Number of method:	4

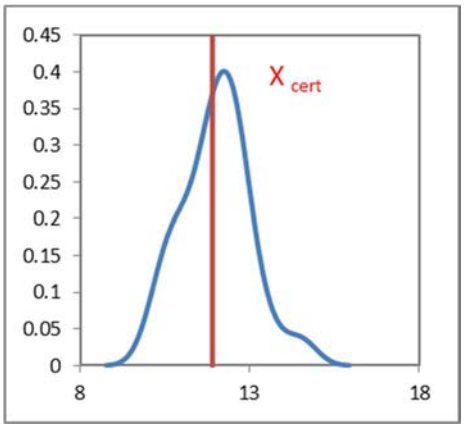
Reported results and expanded uncertainties.

— X_{Cert} ; $\overline{\bullet}$ $X_{lab} \pm U_{lab}$; ---- $X_{Cert} \pm 2\sigma_p$; ---- $X_{Cert} \pm U_{Cert}(k=2)$



Reported data for As in IAEA-470

Kernel density Plot.



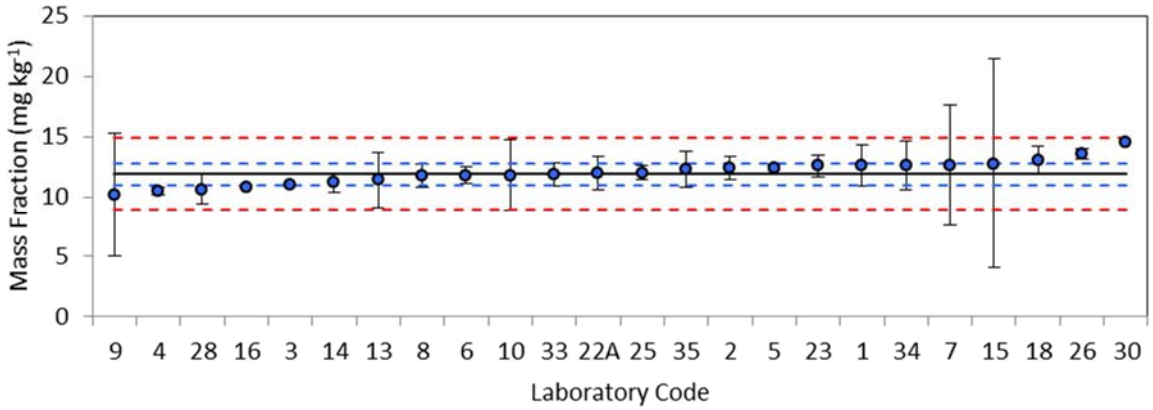
Summary of results:

	Satisfactory	Questionable	Unsatisfactory
z-score	100%	0%	0%
Zeta-score	86%	10%	5%

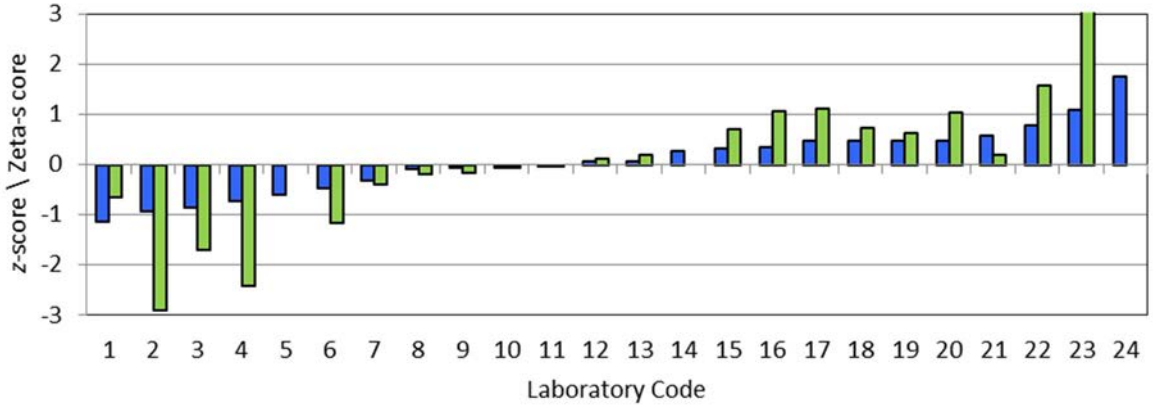
X_{cert}:	11.9 mg kg^{-1}
U_{cert} (k=2) :	0.9 mg kg^{-1}
$2\sigma_p$:	2.98 mg kg^{-1}
Number of results:	24
Number of method:	4

Reported results and expanded uncertainties.

— X_{Cert} ; $X_{lab} \pm U_{lab}$; ---- $X_{Cert} \pm 2\sigma_p$; ---- $X_{Cert} \pm U_{Cert}(k=2)$

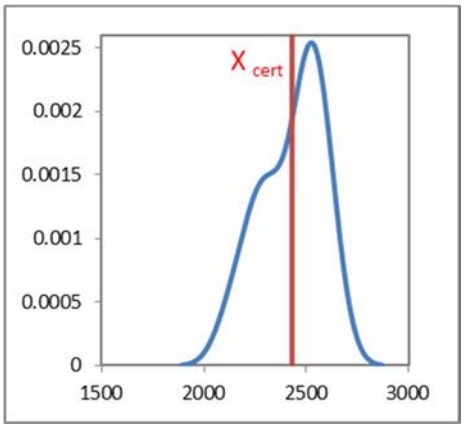


Performance evaluation: *z*-score Zeta-score.



Reported data for Ca in IAEA-470

Kernel density Plot.



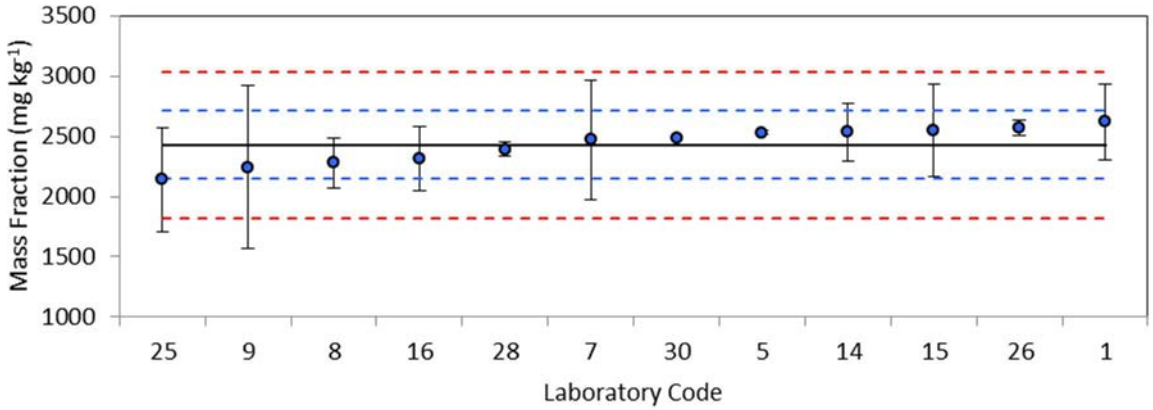
Summary of results:

	Satisfactory	Questionable	Unsatisfactory
z-score	100%	0%	0%
Zeta-score	100%	0%	0%

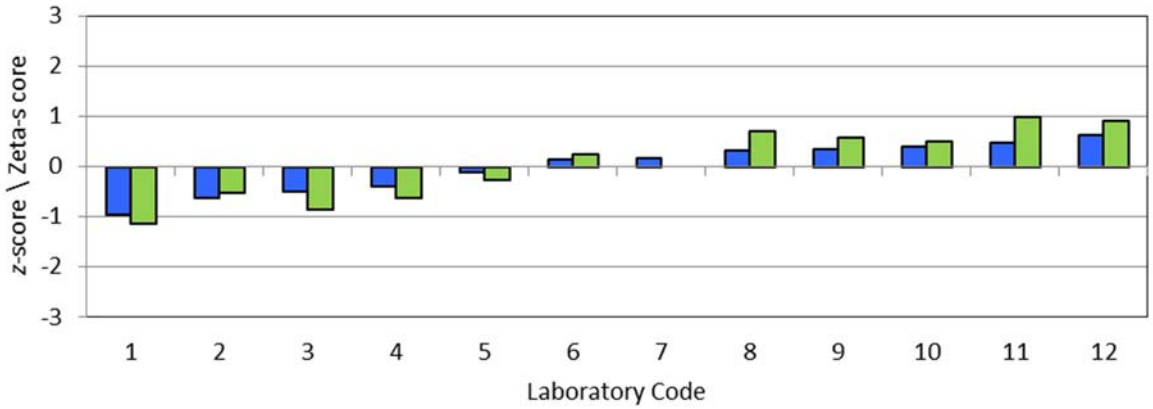
X_{cert}:	2430 mg kg^{-1}
U_{cert} (k=2) :	282 mg kg^{-1}
$2\sigma_p$:	607 mg kg^{-1}
Number of results:	12
Number of method:	3

Reported results and expanded uncertainties.

— X_{Cert} ; $X_{lab} \pm U_{lab}$; ---- $X_{Cert} \pm 2\sigma_p$; ---- $X_{Cert} \pm U_{Cert}(k=2)$

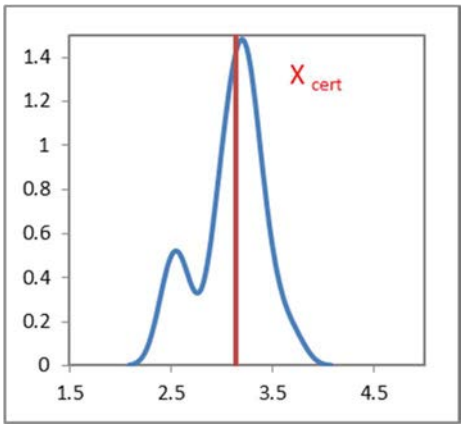


Performance evaluation: *z*-score Zeta-score.



Reported data for Cd in IAEA-470

Kernel density Plot.



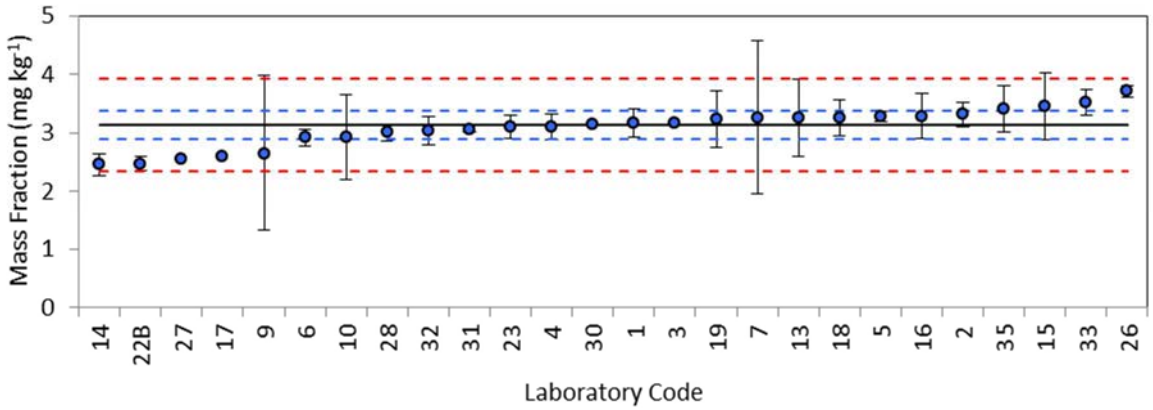
Summary of results:

	Satisfactory	Questionable	Unsatisfactory
z-score	100%	0%	0%
Zeta-score	82%	5%	14%

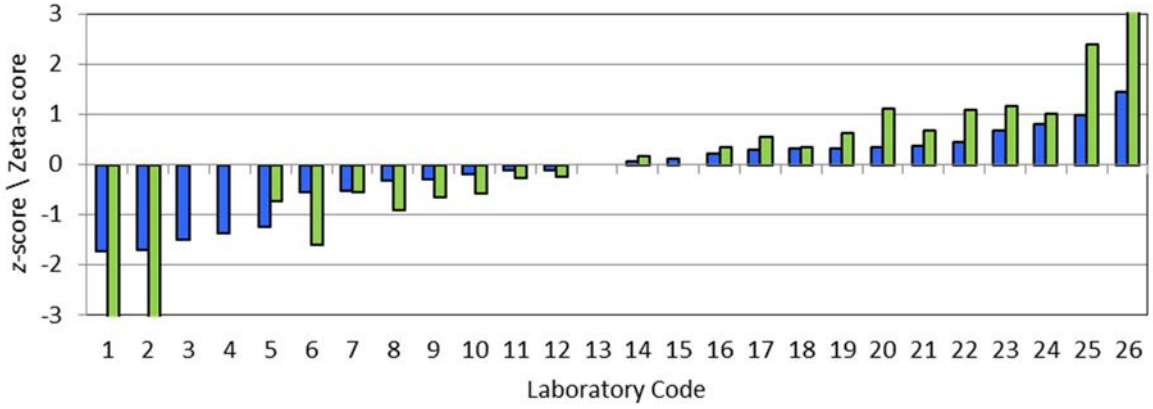
X_{cert}:	3.14 mg kg^{-1}
U_{cert} (k=2) :	0.24 mg kg^{-1}
$2\sigma_p$:	0.79 mg kg^{-1}
Number of results:	26
Number of method:	5

Reported results and expanded uncertainties.

— X_{Cert} ; $X_{lab} \pm U_{lab}$; ---- $X_{Cert} \pm 2\sigma_p$; ---- $X_{Cert} \pm U_{Cert}(k=2)$

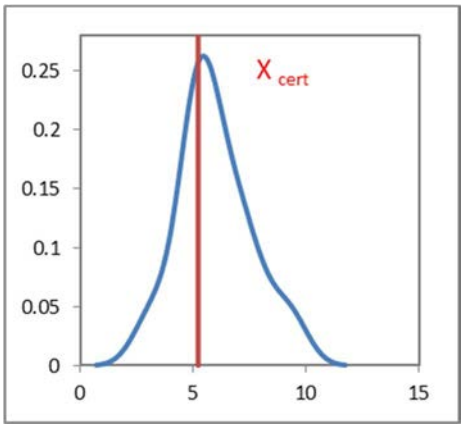


Performance evaluation: *z*-score Zeta-score.



Reported data for CH_3Hg in IAEA-470

Kernel density Plot.



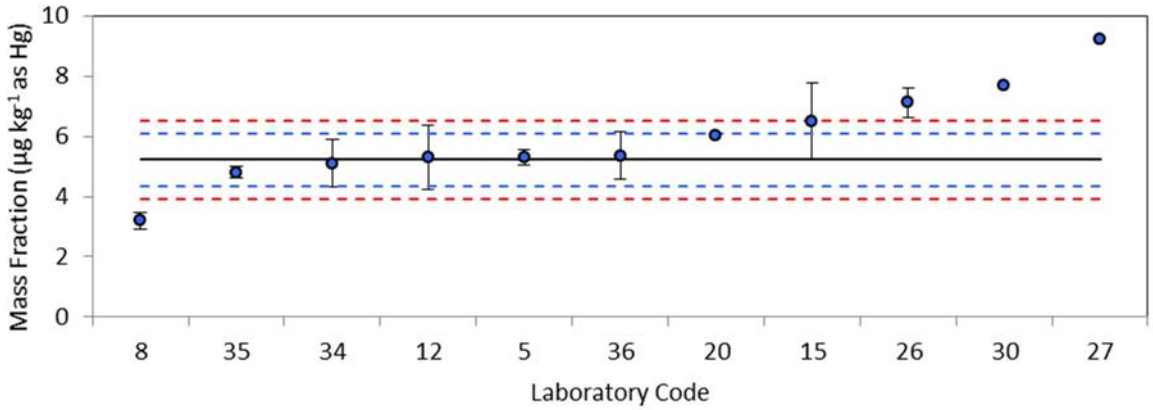
Summary of results:

	Satisfactory	Questionable	Unsatisfactory
z-score	64%	9%	27%
Zeta-score	75%	0%	25%

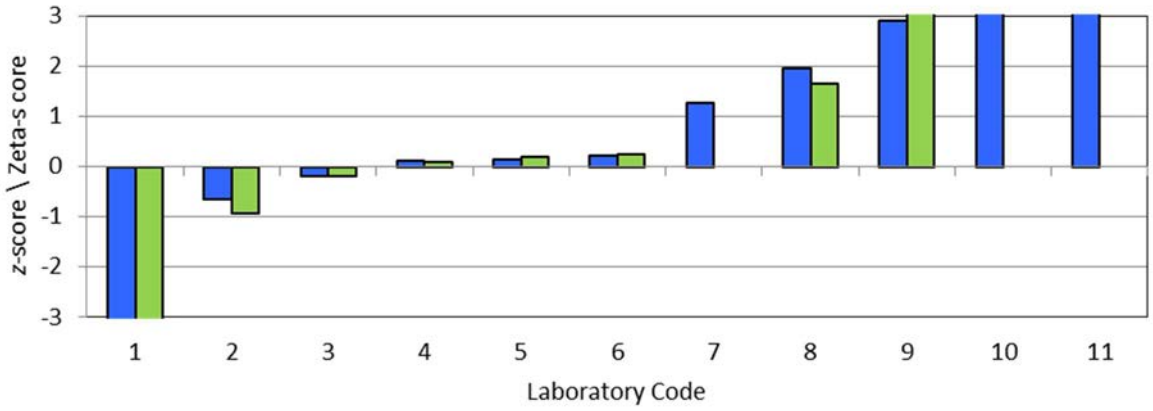
X_{cert}:	5.22 µg kg^{-1} as Hg
U_{cert} (k=2) :	0.87 µg kg^{-1} as Hg
$2\sigma_p$:	1.31 µg kg^{-1} as Hg
Number of results:	11
Number of method:	7

Reported results and expanded uncertainties.

— X_{Cert} ; $X_{lab} \pm U_{lab}$; ---- $X_{Cert} \pm 2\sigma_p$; ---- $X_{Cert} \pm U_{Cert}(k=2)$

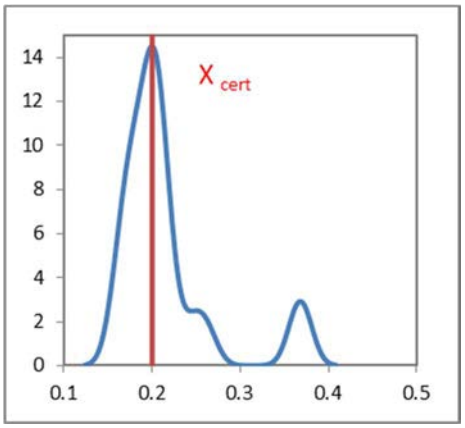


Performance evaluation: *z*-score Zeta-score.



Reported data for Co in IAEA-470

Kernel density Plot.



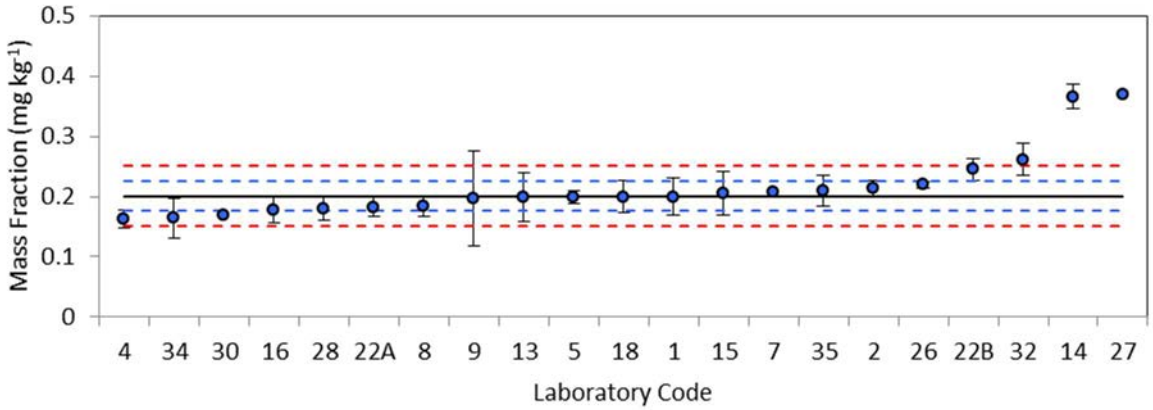
Summary of results:

	Satisfactory	Questionable	Unsatisfactory
z-score	85%	5%	10%
Zeta-score	79%	11%	11%

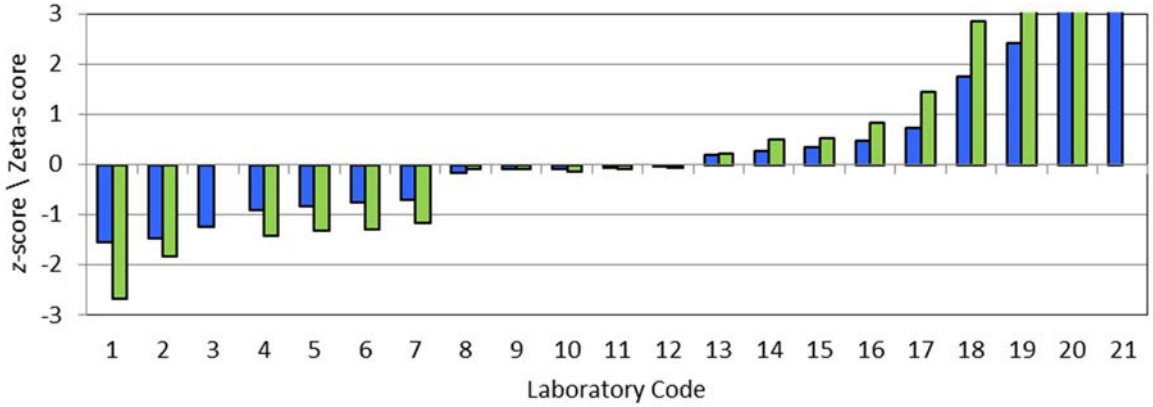
X_{cert}:	0.20 mg kg^{-1}
U_{cert} (k=2) :	0.03 mg kg^{-1}
$2\sigma_p$:	0.05 mg kg^{-1}
Number of results:	21
Number of method:	3

Reported results and expanded uncertainties.

— X_{Cert} ; $X_{lab} \pm U_{lab}$; ---- $X_{Cert} \pm 2\sigma_p$; ---- $X_{Cert} \pm U_{Cert}(k=2)$

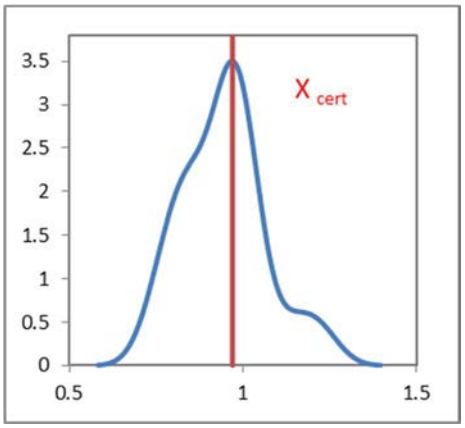


Performance evaluation: *z*-score Zeta-score.



Reported data for Cr in IAEA-470

Kernel density Plot.



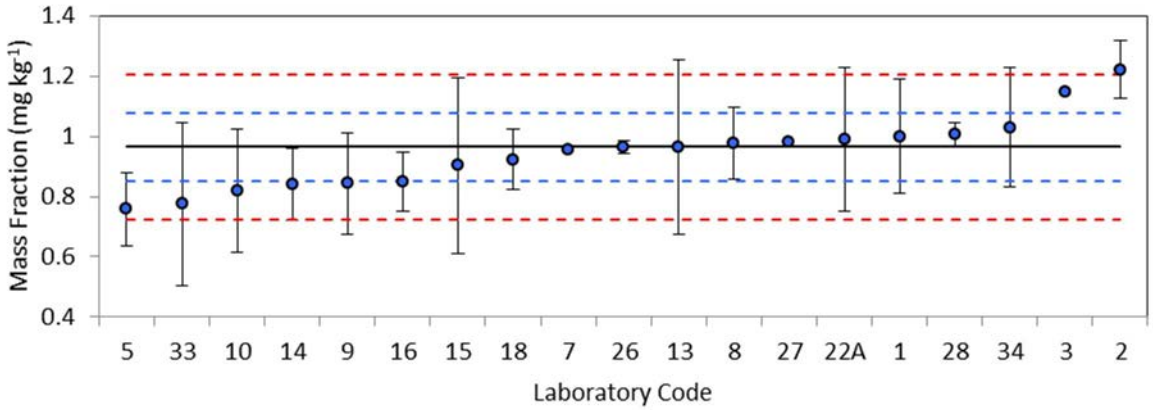
Summary of results:

	Satisfactory	Questionable	Unsatisfactory
z-score	95%	5%	0%
Zeta-score	88%	6%	6%

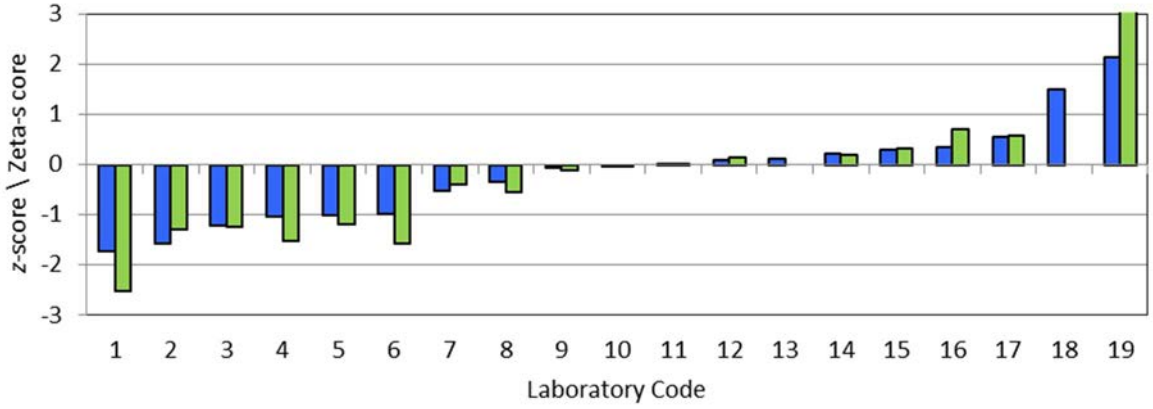
X_{cert}:	0.97 mg kg^{-1}
U_{cert} (k=2) :	0.11 mg kg^{-1}
$2\sigma_p$:	0.24 mg kg^{-1}
Number of results:	19
Number of method:	4

Reported results and expanded uncertainties.

— X_{Cert} ; $X_{lab} \pm U_{lab}$; ---- $X_{Cert} \pm 2\sigma_p$; ---- $X_{Cert} \pm U_{Cert}(k=2)$

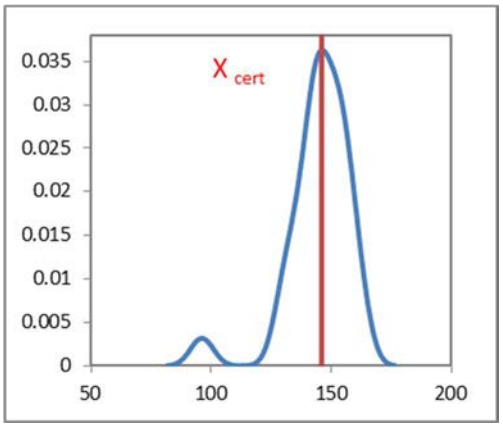


Performance evaluation: *z*-score Zeta-score.



Reported data for Cu in IAEA-470

Kernel density Plot.



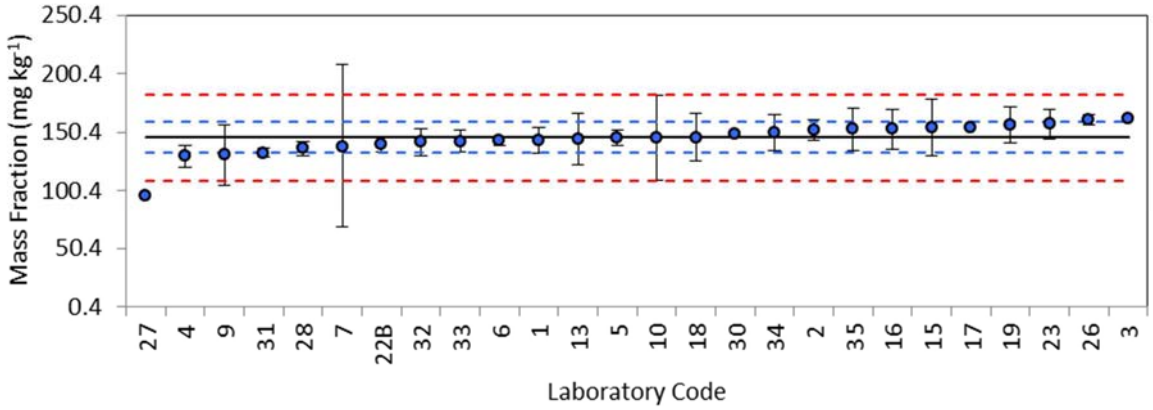
Summary of results:

	Satisfactory	Questionable	Unsatisfactory
z-score	96%	4%	0%
Zeta-score	91%	9%	0%

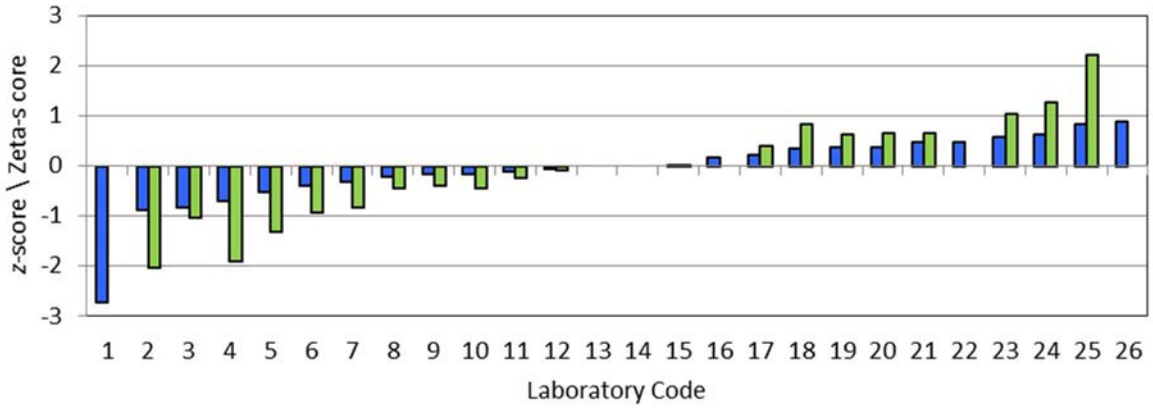
X_{cert}:	146 mg kg^{-1}
U_{cert} (k=2) :	13 mg kg^{-1}
$2\sigma_p$:	36.5 mg kg^{-1}
Number of results:	26
Number of method:	5

Reported results and expanded uncertainties.

— X_{Cert} ; $X_{lab} \pm U_{lab}$; ---- $X_{Cert} \pm 2\sigma_p$; ---- $X_{Cert} \pm U_{Cert}(k=2)$

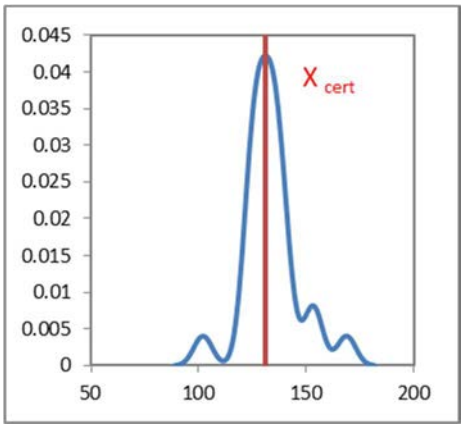


Performance evaluation: *z*-score Zeta-score.



Reported data for Fe in IAEA-470

Kernel density Plot.



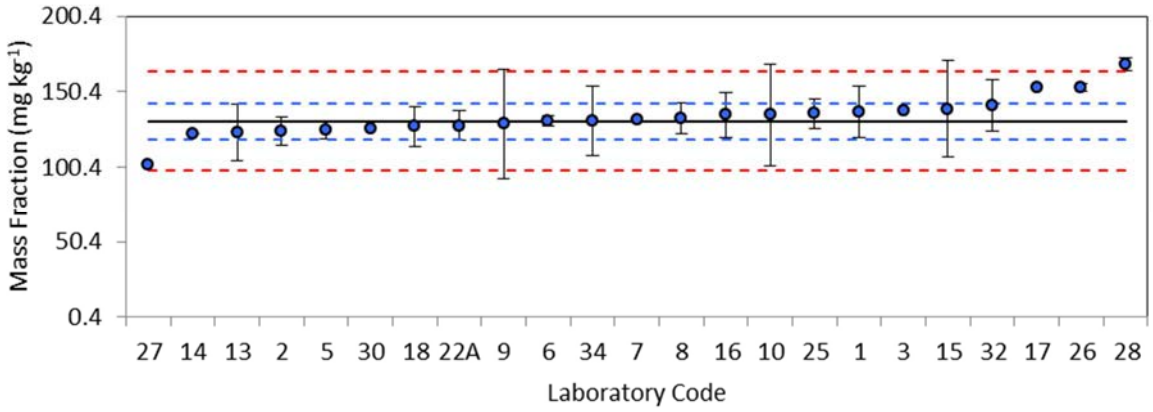
Summary of results:

	Satisfactory	Questionable	Unsatisfactory
z-score	96%	4%	0%
Zeta-score	89%	0%	11%

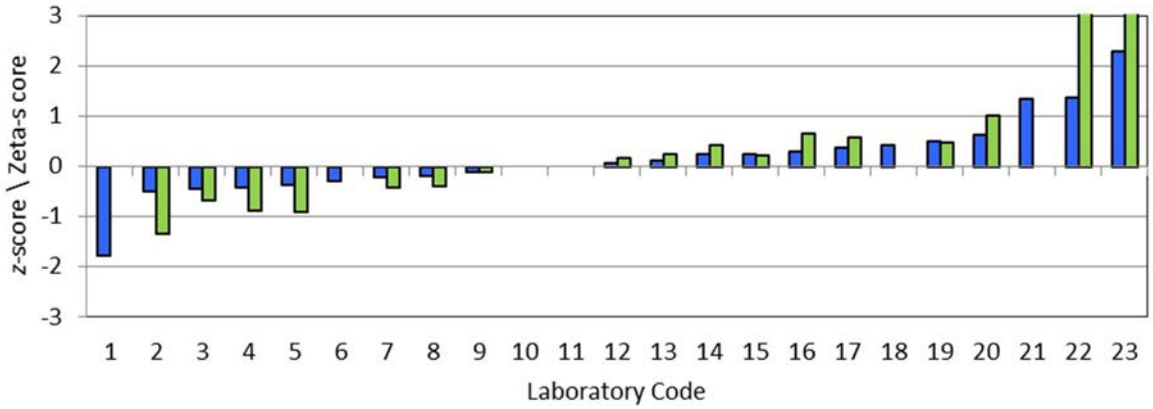
X_{cert}:	131 mg kg^{-1}
U_{cert} (k=2) :	12 mg kg^{-1}
$2\sigma_p$:	33 mg kg^{-1}
Number of results:	23
Number of method:	5

Reported results and expanded uncertainties.

— X_{Cert} ; $X_{lab} \pm U_{lab}$; ---- $X_{Cert} \pm 2\sigma_p$; ---- $X_{Cert} \pm U_{Cert}(k=2)$

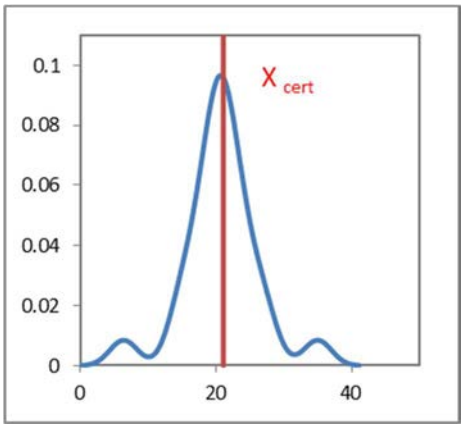


Performance evaluation: *z*-score Zeta-score.



Reported data for Hg in IAEA-470

Kernel density Plot.



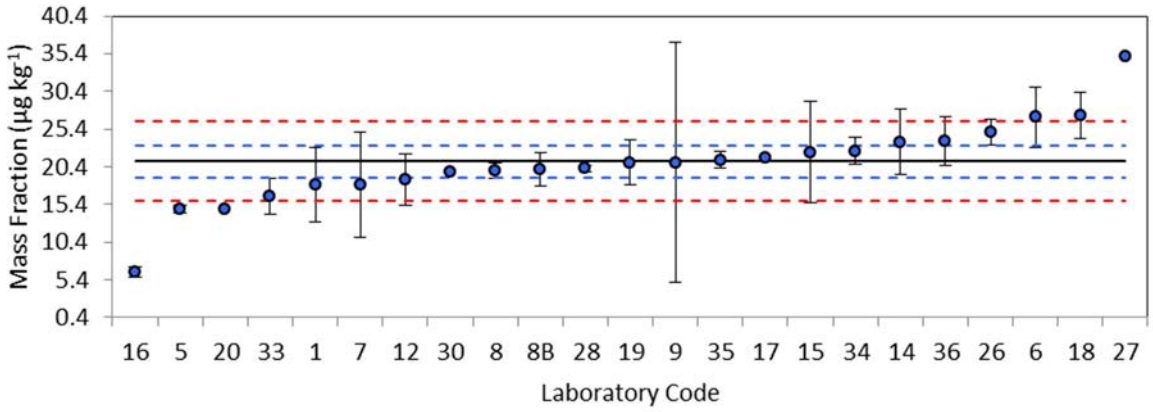
Summary of results:

	Satisfactory	Questionable	Unsatisfactory
z-score	73%	18%	9%
Zeta-score	61%	22%	17%

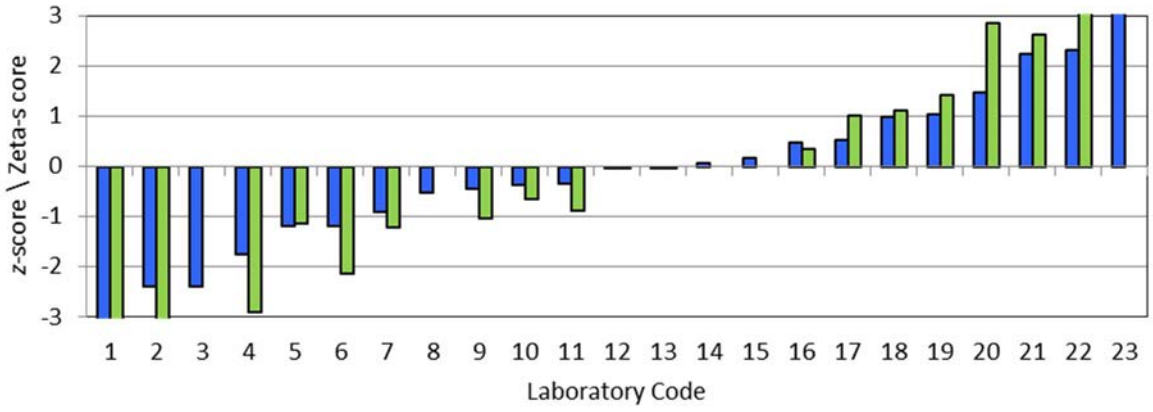
X_{cert}:	21.1 µg kg^{-1}
U_{cert} (k=2) :	2.1 µg kg^{-1}
$2\sigma_p$:	5.3 µg kg^{-1}
Number of results:	23
Number of method:	5

Reported results and expanded uncertainties.

— X_{Cert} ; $X_{lab} \pm U_{lab}$; ---- $X_{Cert} \pm 2\sigma_p$; ---- $X_{Cert} \pm U_{Cert}(k=2)$



Performance evaluation: *z*-score Zeta-score.



Reported data for K in IAEA-470

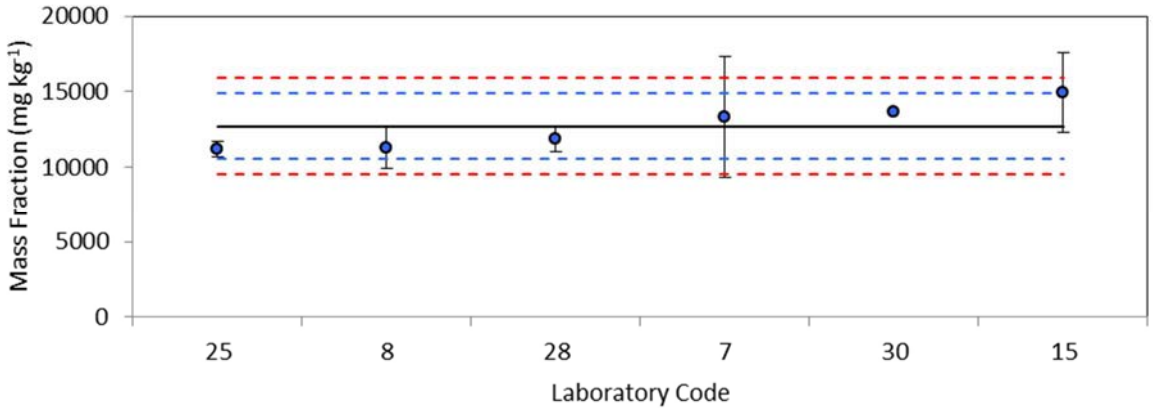
Summary of results:

	Satisfactory	Questionable	Unsatisfactory
z-score	100%	0%	0%
Zeta-score	100%	0%	0%

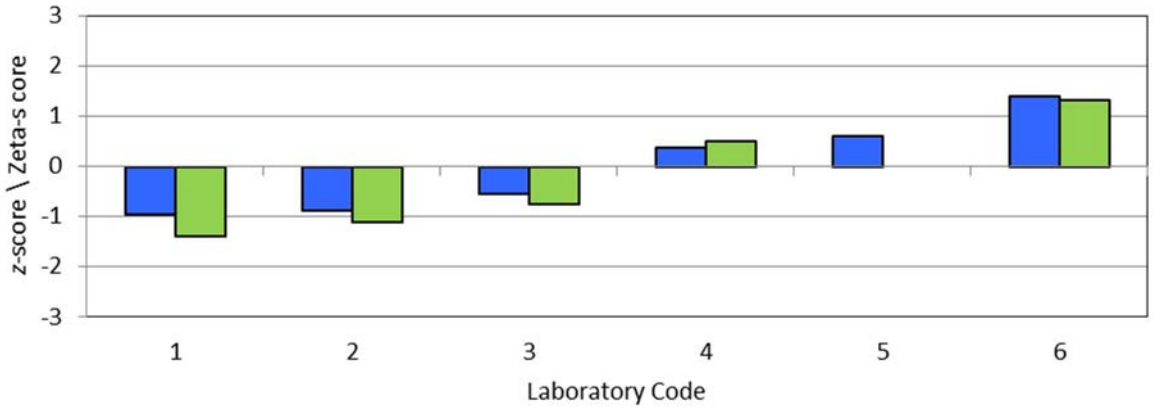
X_{ass}:	12720 mg kg^{-1}
U_{ass} (k=2) :	2150 mg kg^{-1}
$2\sigma_p$:	3180 mg kg^{-1}
Number of results:	6
Number of method:	3

Reported results and expanded uncertainties.

— X_{Cert} ; $X_{lab} \pm U_{lab}$; ---- $X_{Cert} \pm 2\sigma_p$; ---- $X_{Cert} \pm U_{Cert}(k=2)$



Performance evaluation: *z*-score Zeta-score.



Reported data for Li in IAEA-470

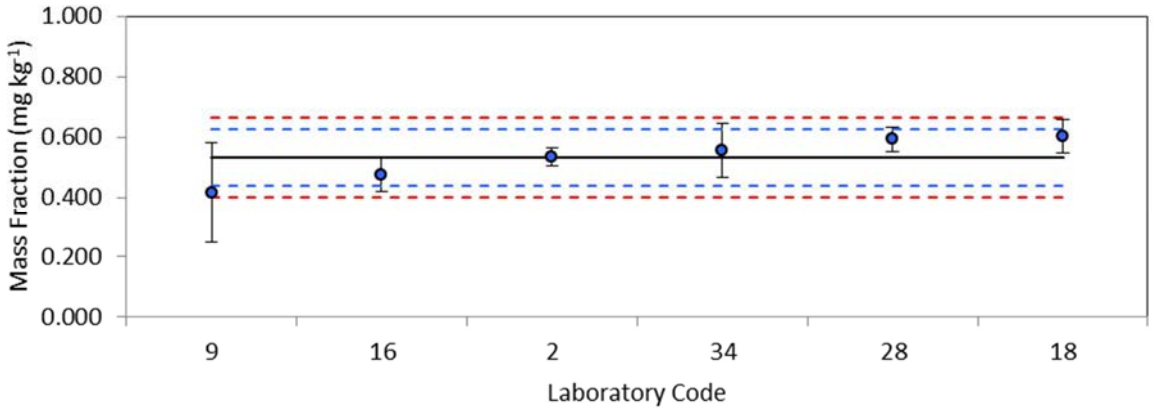
Summary of results:

	Satisfactory	Questionable	Unsatisfactory
z-score	100%	0%	0%
Zeta-score	100%	0%	0%

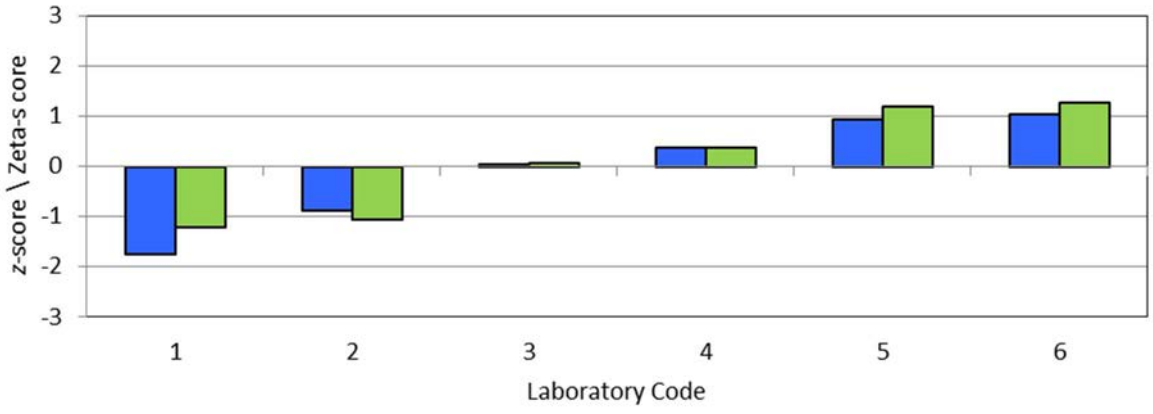
X_{ass}:	0.532 mg kg^{-1}
U_{ass} (k=2) :	0.094 mg kg^{-1}
$2\sigma_p$:	0.13 mg kg^{-1}
Number of results:	6
Number of method:	2

Reported results and expanded uncertainties.

— X_{Cert} ; $X_{lab} \pm U_{lab}$; ---- $X_{Cert} \pm 2\sigma_p$; ---- $X_{Cert} \pm U_{Cert}(k=2)$

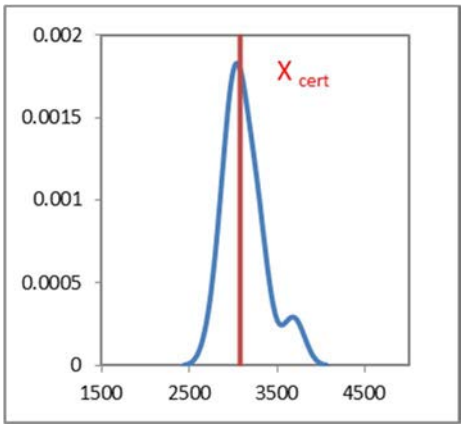


Performance evaluation: *z*-score Zeta-score.



Reported data for Mg in IAEA-470

Kernel density Plot.



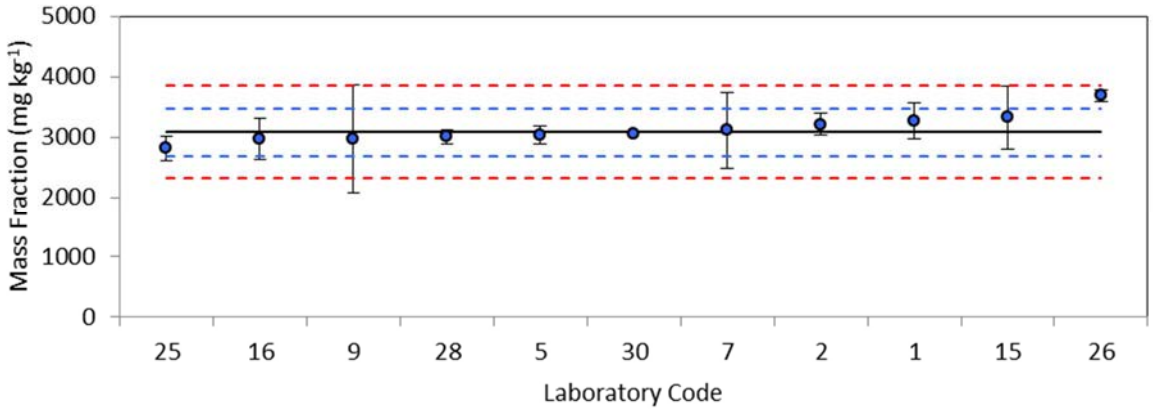
Summary of results:

	Satisfactory	Questionable	Unsatisfactory
z-score	100%	0%	0%
Zeta-score	90%	0%	10%

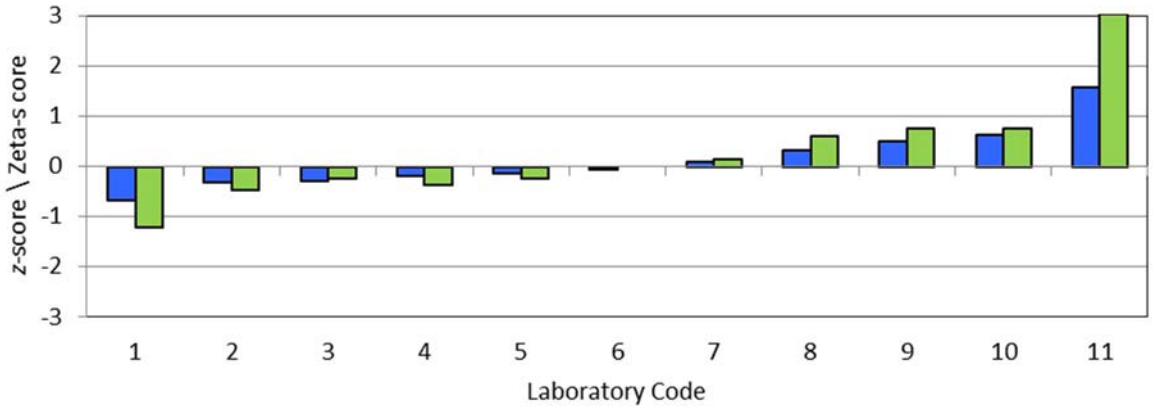
X_{cert}:	3080 mg kg^{-1}
U_{cert} (k=2) :	390 mg kg^{-1}
$2\sigma_p$:	770 mg kg^{-1}
Number of results:	11
Number of method:	3

Reported results and expanded uncertainties.

— X_{Cert} ; $X_{lab} \pm U_{lab}$; ---- $X_{Cert} \pm 2\sigma_p$; ---- $X_{Cert} \pm U_{Cert}(k=2)$

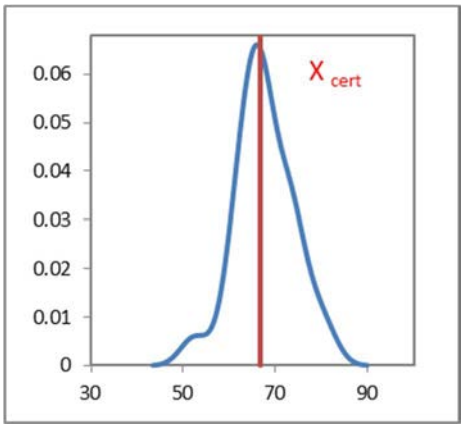


Performance evaluation: *z*-score Zeta-score.



Reported data for Mn in IAEA-470

Kernel density Plot.



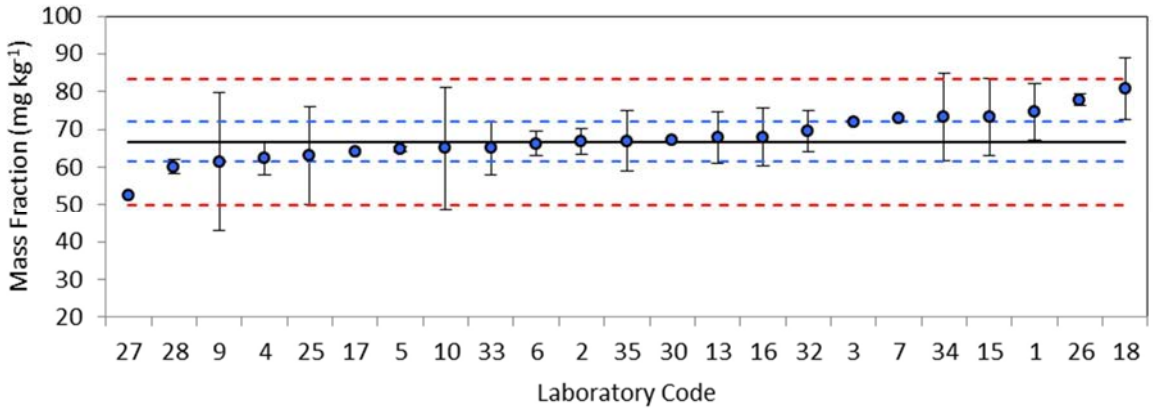
Summary of results:

	Satisfactory	Questionable	Unsatisfactory
z-score	100%	0%	0%
Zeta-score	84%	11%	5%

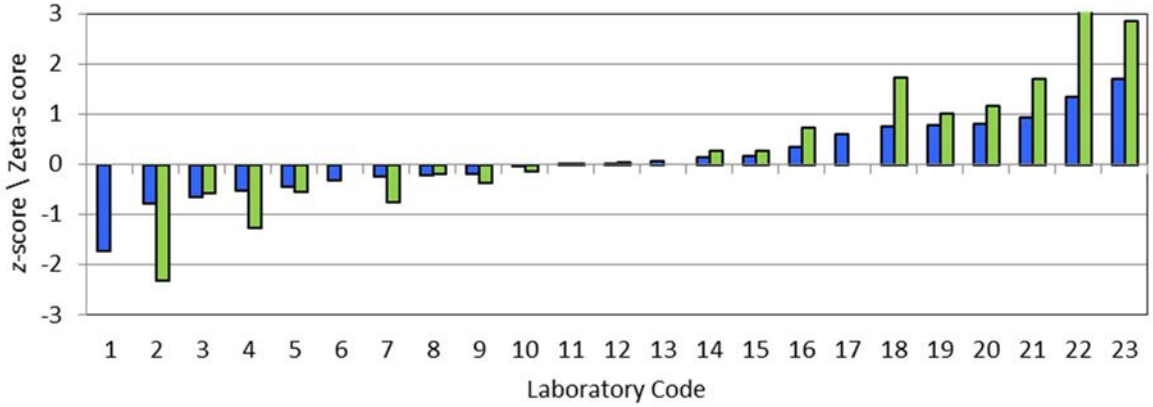
X_{cert}:	66.7 mg kg^{-1}
U_{cert} (k=2) :	5.3 mg kg^{-1}
$2\sigma_p$:	16.7 mg kg^{-1}
Number of results:	23
Number of method:	

Reported results and expanded uncertainties.

— X_{Cert} ; $\pm X_{lab} \pm U_{lab}$; ---- $X_{Cert} \pm 2\sigma_p$; ---- $X_{Cert} \pm U_{Cert}(k=2)$

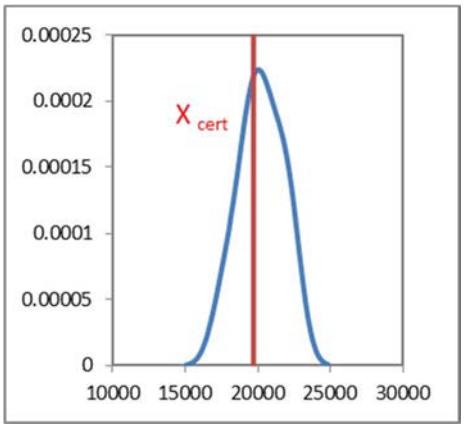


Performance evaluation: *z*-score Zeta-score.



Reported data for Na in IAEA-470

Kernel density Plot.



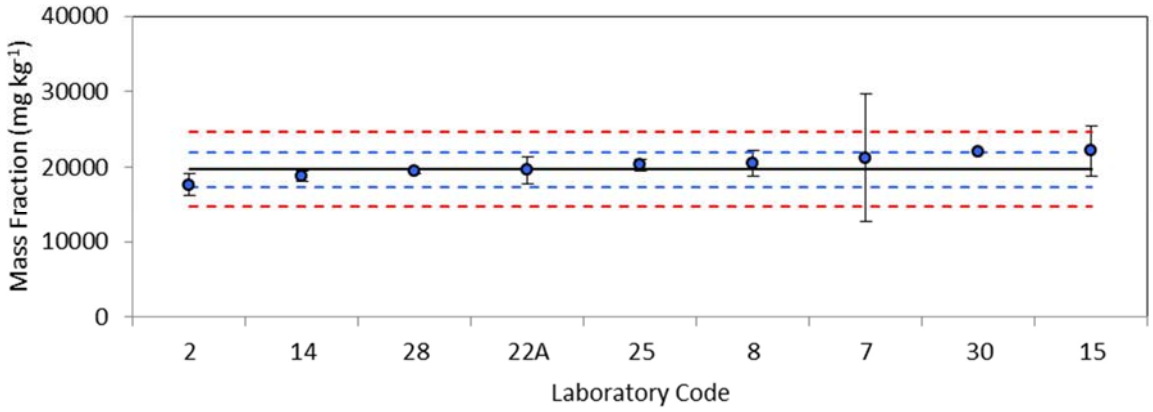
Summary of results:

	Satisfactory	Questionable	Unsatisfactory
z-score	100%	0%	0%
Zeta-score	100%	0%	0%

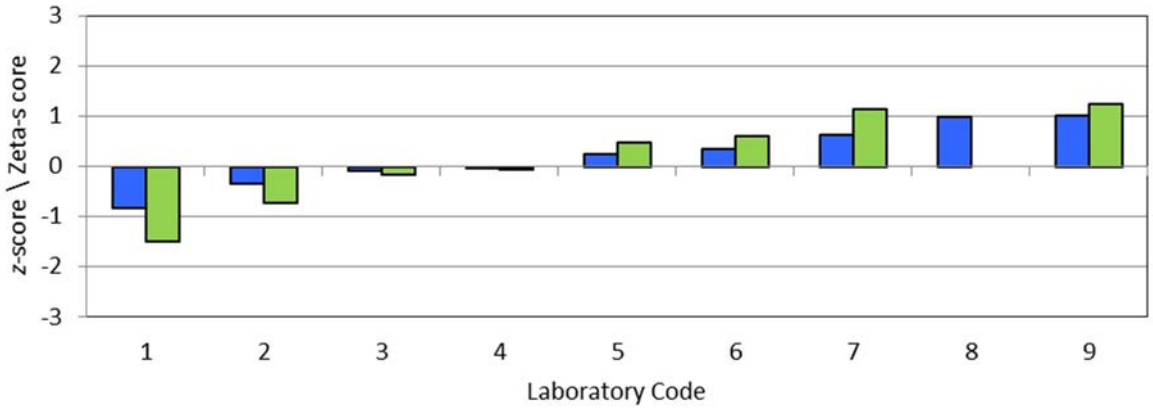
X_{cert}:	19700 mg kg^{-1}
U_{cert} (k=2) :	2300 mg kg^{-1}
$2\sigma_p$:	4925 mg kg^{-1}
Number of results:	9
Number of method:	3

Reported results and expanded uncertainties.

— X_{Cert} ; $X_{lab} \pm U_{lab}$; ---- $X_{Cert} \pm 2\sigma_p$; ---- $X_{Cert} \pm U_{Cert}(k=2)$

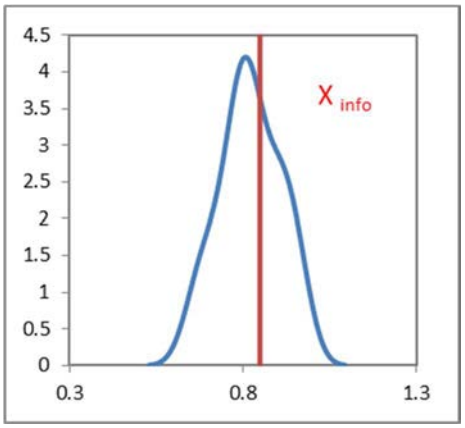


Performance evaluation: *z*-score Zeta-score.



Reported data for Ni in IAEA-470

Kernel density Plot.

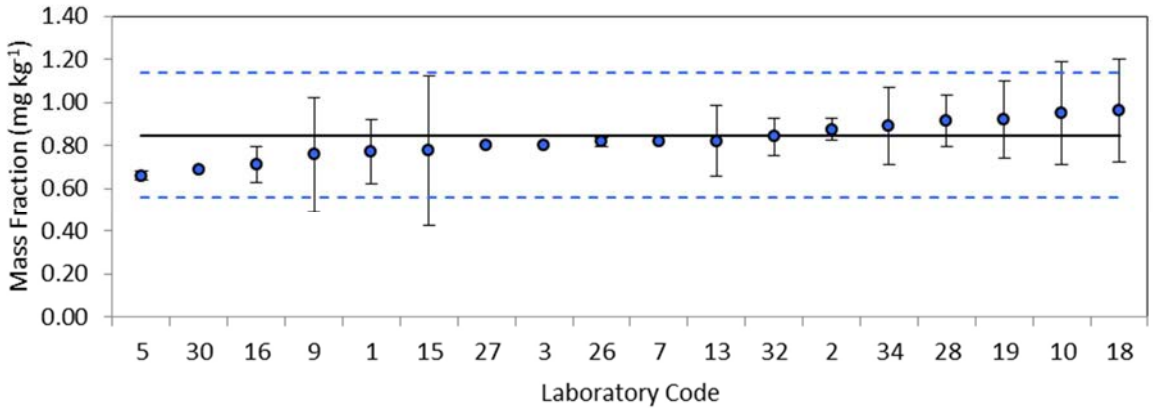


Summary of results:

X_{info}:	0.849 mg kg^{-1}
U_{info} (k=2) :	0.291 mg kg^{-1}
Number of results:	18
Number of method:	2

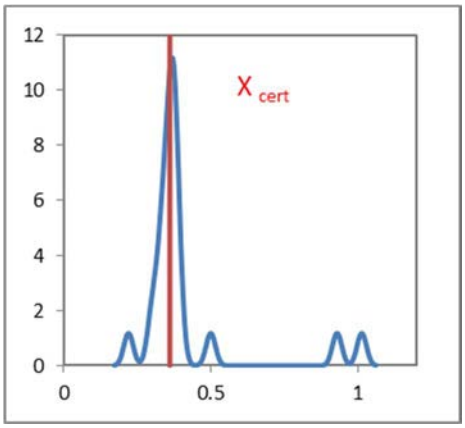
Reported results and expanded uncertainties.

— X_{Cert} ; $\overline{\bullet}$ $X_{lab} \pm U_{lab}$; ---- $X_{Cert} \pm 2\sigma_p$; ---- $X_{Cert} \pm U_{Cert}(k=2)$



Reported data for Pb in IAEA-470

Kernel density Plot.



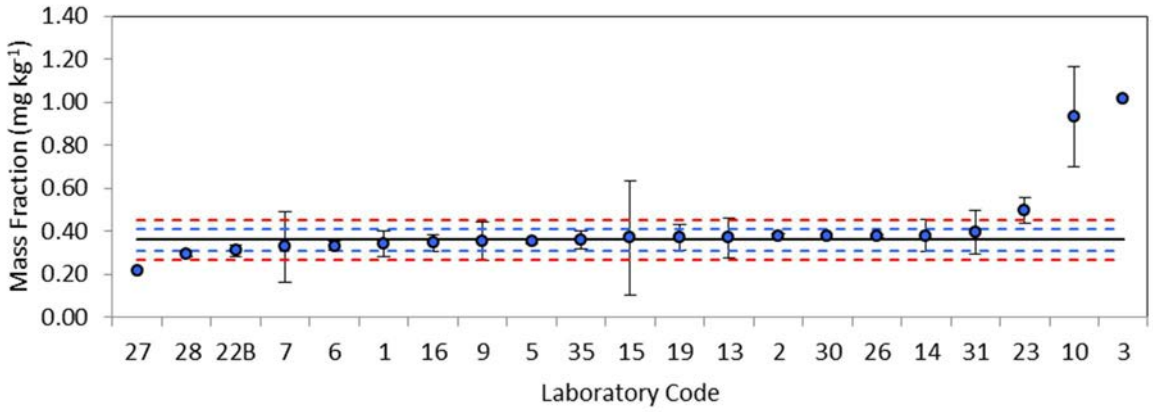
Summary of results:

	Satisfactory	Questionable	Unsatisfactory
z-score	81%	0%	19%
Zeta-score	83%	6%	11%

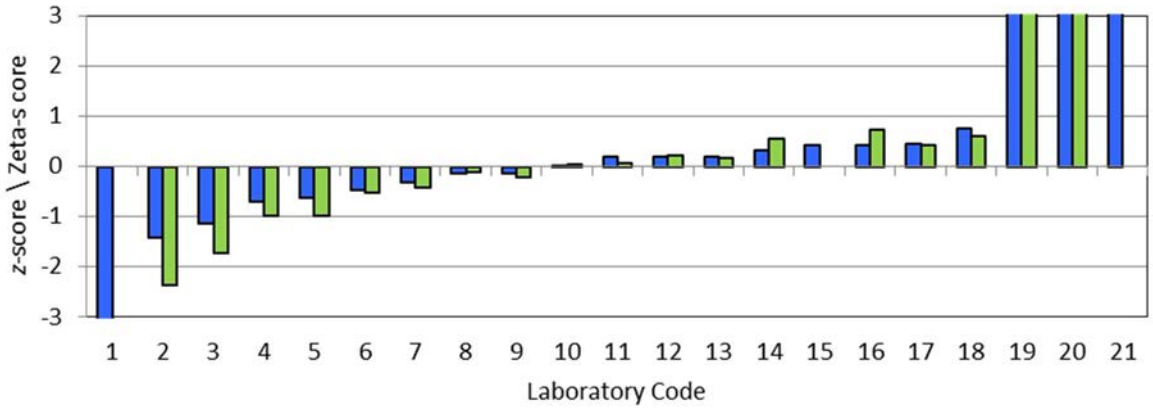
X_{cert}:	0.361 mg kg^{-1}
U_{cert} (k=2) :	0.053 mg kg^{-1}
$2\sigma_p$:	0.09 mg kg^{-1}
Number of results:	21
Number of method:	3

Reported results and expanded uncertainties.

— X_{Cert} ; $X_{lab} \pm U_{lab}$; ---- $X_{Cert} \pm 2\sigma_p$; ---- $X_{Cert} \pm U_{Cert}(k=2)$



Performance evaluation: *z*-score Zeta-score



Reported data for Rb in IAEA-470

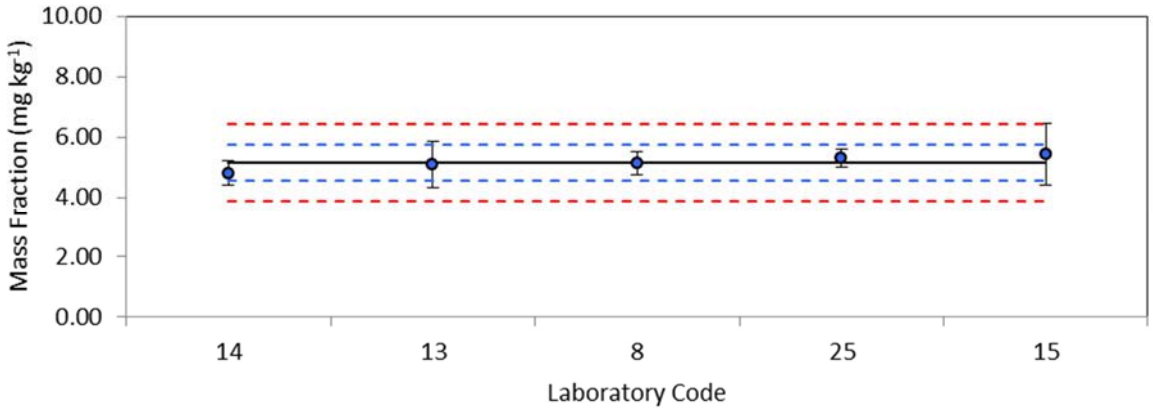
Summary of results:

	Satisfactory	Questionable	Unsatisfactory
z-score	100%	0%	0%
Zeta-score	100%	0%	0%

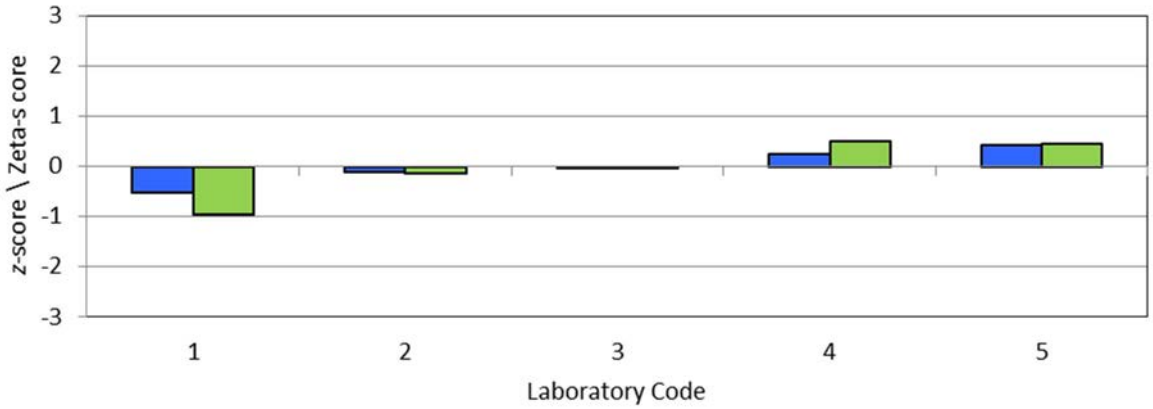
X_{cert}:	5.14 mg kg^{-1}
U_{cert} (k=2) :	0.59 mg kg^{-1}
$2\sigma_p$:	1.29 mg kg^{-1}
Number of results:	5
Number of method:	2

Reported results and expanded uncertainties.

— X_{Cert} ; $X_{lab} \pm U_{lab}$; ---- $X_{Cert} \pm 2\sigma_p$; ---- $X_{Cert} \pm U_{Cert}(k=2)$

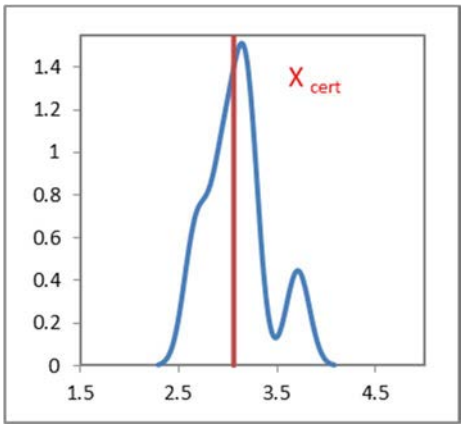


Performance evaluation: *z*-score Zeta-score.



Reported data for Se in IAEA-470

Kernel density Plot.



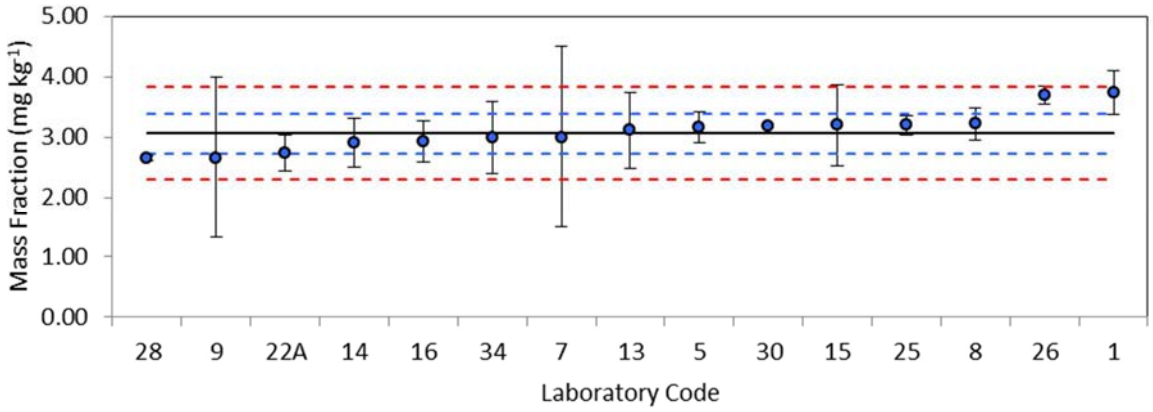
Summary of results:

	Satisfactory	Questionable	Unsatisfactory
z-score	100%	0%	0%
Zeta-score	79%	14%	7%

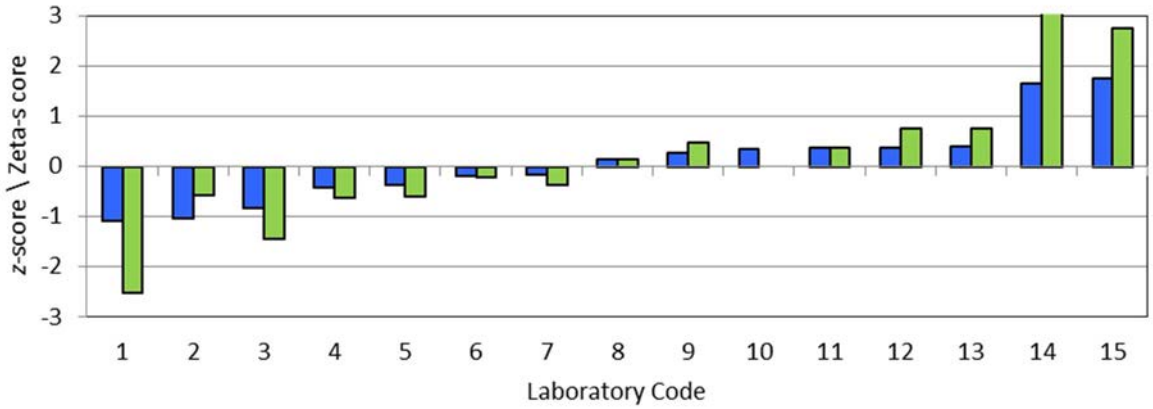
X_{cert}:	3.06 mg kg^{-1}
U_{cert} (k=2) :	0.33 mg kg^{-1}
$2\sigma_p$:	0.77 mg kg^{-1}
Number of results:	15
Number of method:	4

Reported results and expanded uncertainties.

— X_{Cert} ; $X_{lab} \pm U_{lab}$; ---- $X_{Cert} \pm 2\sigma_p$; ---- $X_{Cert} \pm U_{Cert}(k=2)$

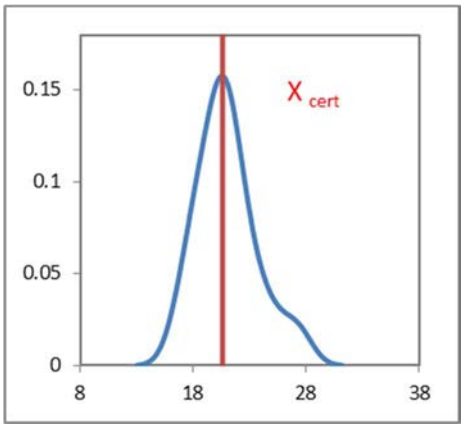


Performance evaluation: *z*-score Zeta-score.



Reported data for Sr in IAEA-470

Kernel density Plot.



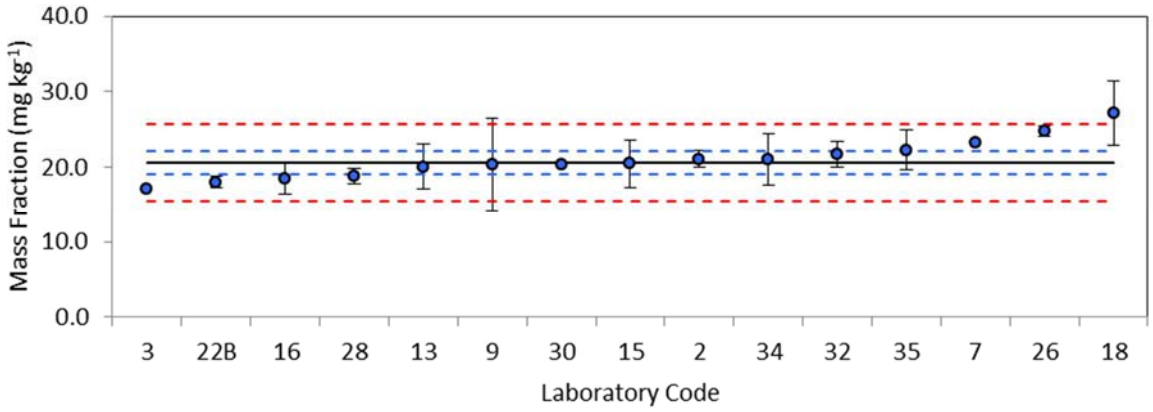
Summary of results:

	Satisfactory	Questionable	Unsatisfactory
z-score	93%	7%	0%
Zeta-score	77%	15%	8%

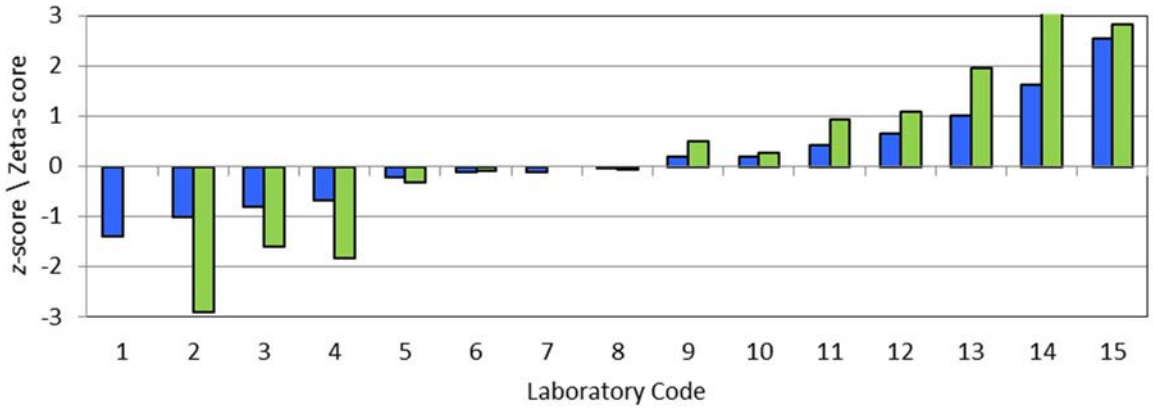
X_{cert}:	20.6mg kg^{-1}
U_{cert} (k=2) :	1.6 mg kg^{-1}
$2\sigma_p$:	5.15 mg kg^{-1}
Number of results:	15
Number of method:	3

Reported results and expanded uncertainties.

— X_{Cert} ; $X_{lab} \pm U_{lab}$; ---- $X_{Cert} \pm 2\sigma_p$; ---- $X_{Cert} \pm U_{Cert}(k=2)$

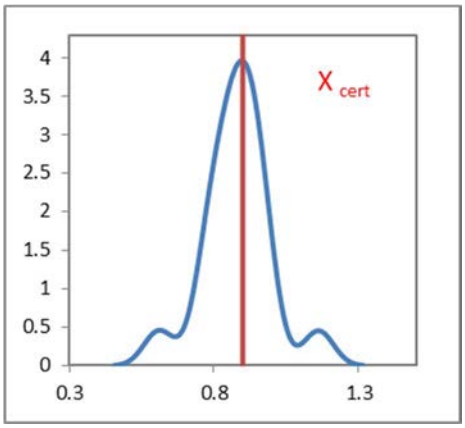


Performance evaluation: *z*-score Zeta-score.



Reported data for V in IAEA-470

Kernel density Plot.



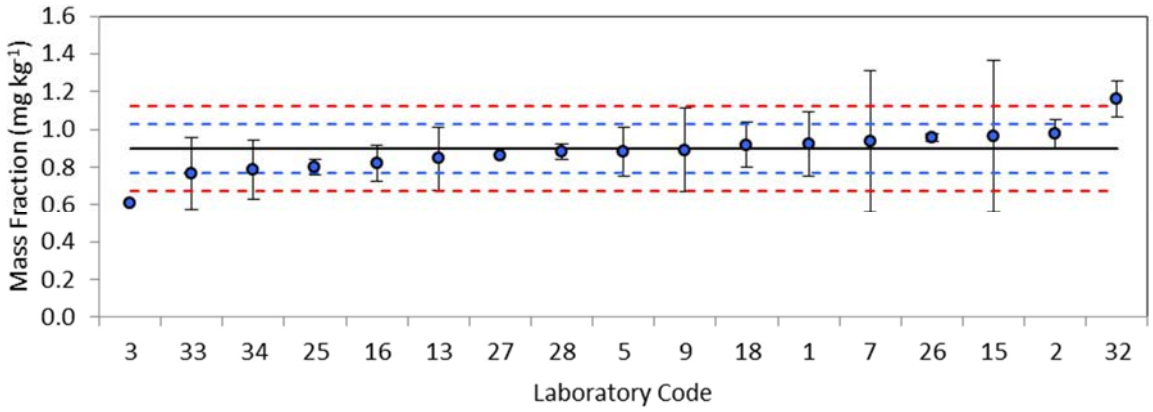
Summary of results:

	Satisfactory	Questionable	Unsatisfactory
z-score	88%	12%	0%
Zeta-score	77%	15%	8%

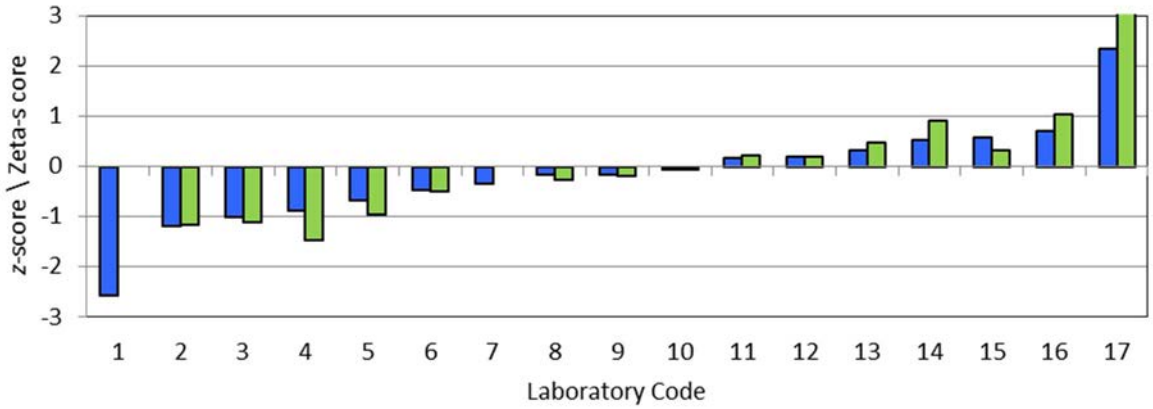
X_{cert}:	0.90 mg kg^{-1}
U_{cert} (k=2) :	0.13 mg kg^{-1}
$2\sigma_p$:	0.22 mg kg^{-1}
Number of results:	17
Number of method:	4

Reported results and expanded uncertainties.

— X_{Cert} ; $X_{lab} \pm U_{lab}$; ---- $X_{Cert} \pm 2\sigma_p$; ---- $X_{Cert} \pm U_{Cert}(k=2)$

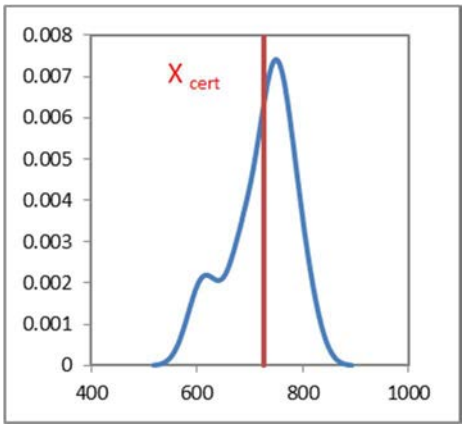


Performance evaluation: *z*-score Zeta-score.



Reported data for Zn in IAEA-470

Kernel density Plot.



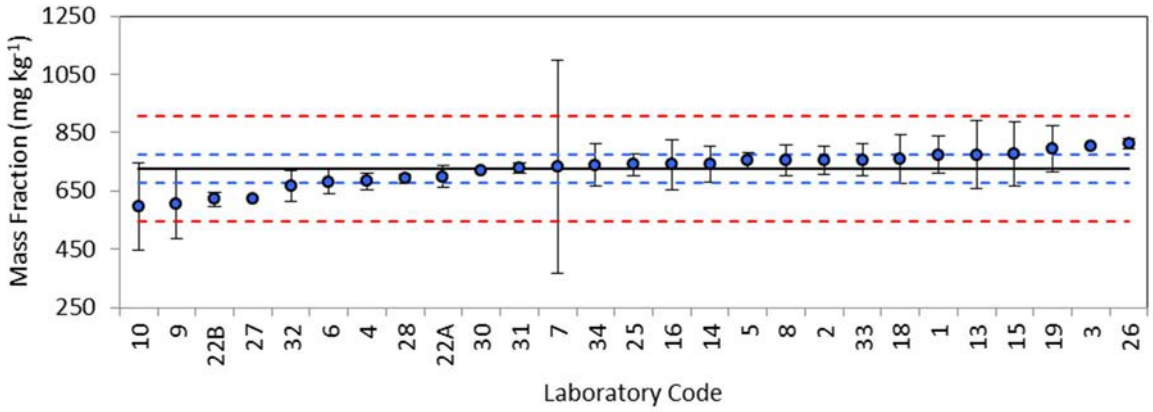
Summary of results:

	Satisfactory	Questionable	Unsatisfactory
z-score	100%	0%	0%
Zeta-score	92%	0%	8%

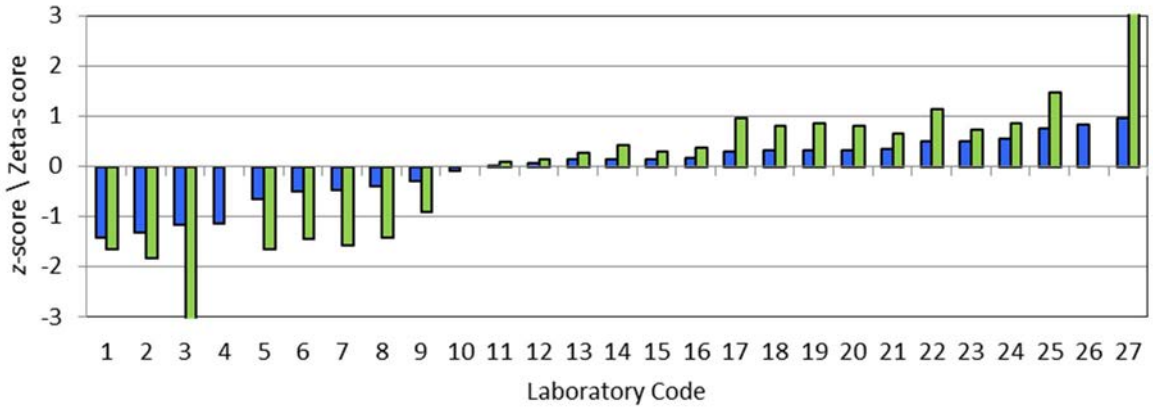
X_{cert}:	727 mg kg^{-1}
U_{cert} (k=2) :	48 mg kg^{-1}
$2\sigma_p$:	182 mg kg^{-1}
Number of results:	27
Number of method:	6

Reported results and expanded uncertainties.

— X_{Cert} ; $X_{lab} \pm U_{lab}$; ---- $X_{Cert} \pm 2\sigma_p$; ---- $X_{Cert} \pm U_{Cert}(k=2)$



Performance evaluation: *z*-score Zeta-score.



APPENDIX II

REPORTED RESULTS BY PARTICIPANTS

TABLE 7. REPORTED RESULTS BY PARTICPANTS

Lab Code	Element	Unit	Instrumental Method	Lab Mean	Lab u	Lab U	*k*	*z*-score	Zeta-score	QC
2	Ag	mg kg-1	ICP-MS	1.31	0.66	1.32	2.00	0.1	0.0	NIST 1566b
7	Ag	mg kg-1	ICP-MS	1.01	0.05			-1.8	-3.9	
8	Ag	mg kg-1	NAA	1.36	0.07	0.14	2.00	0.4	0.8	NIST 1547
9	Ag	mg kg-1	ICP-MS	1.15	0.23	0.46	2.00	-0.9	-0.6	
13	Ag	mg kg-1	ICP-MS	1.24	0.19	0.37	2.00	-0.3	-0.3	DORM-2
16	Ag	mg kg-1	ICP-MS	1.25	0.07	0.14	2.00	-0.2	-0.5	DOLT-4
19	Ag	mg kg-1	ICP-MS	1.31	0.10	0.20	2.00	0.1	0.2	NIST 1566b
25	Ag	mg kg-1	NAA	1.47	0.04	0.07	2.00	1.1	2.9	NIST 1566b
26	Ag	mg kg-1	ICP-MS	1.54	0.02	0.03	2.00	1.6	4.7	NIST 1566b
28	Ag	mg kg-1	ICP-MS	1.20	0.02	0.04	2.00	-0.6	-1.7	ESA-2
33	Ag	mg kg-1	ICP-OES	1.25	0.13	0.26	2.00	-0.3	-0.3	DOLT-4
35	Ag	mg kg-1	ICP-MS	1.31	0.08	0.16	2.00	0.1	0.2	NIST 2976
22A	Ag	mg kg-1	NAA	1.18	0.06	0.11	2.00	-0.7	-1.5	DOLT-4
1	Al	mg kg-1	ICP-MS	58.6	4.1	8.1	2.0	-1.6	-1.3	NIST 1515
5	Al	mg kg-1	ICP-MS	49.0	1.6	3.2	2.0	-2.6	-2.4	NIST 1547
9	Al	mg kg-1	ICP-OES	72.3	9.4	18.8	2.0	-0.1	0.0	
10	Al	mg kg-1	ET-AAS	74.2	9.3	18.6	2.0	0.1	0.1	NIST 2976
13	Al	mg kg-1	ICP-MS	87.8	8.8	17.6	2.0	1.6	1.1	DORM-2
15	Al	mg kg-1	ICP-MS	83.7	7.5	15.1	2.0	1.2	0.9	TORT-3
16	Al	mg kg-1	ICP-MS	83.9	0.6	1.1	2.0	1.2	1.1	NIST 1548
25	Al	mg kg-1	NAA	95.0	2.5	5.0	2.0	2.4	2.2	NIST 1566b
28	Al	mg kg-1	ICP-MS	228.0	2.0	4.0	2.0	17	15	ESA-2
30	Al	mg kg-1	ICP-MS	47.5				-2.8		TORT-2

TABLE 7. REPORTED RESULTS BY PARTICPANTS (cont.)

Lab Code	Element	Unit	Instrumental Method	Lab Mean	Lab u	Lab U	*k*	*z*-score	Zeta-score	QC
33	Al	mg kg-1	ICP-OES	40.5				-3.5		TORT-2
1	As	mg kg-1	ICP-MS	12.6	0.9	1.7	2.0	0.5	0.7	TORT-2
2	As	mg kg-1	ICP-MS	12.4	0.5	1.0	2.0	0.3	0.7	NIST 1566b
3	As	mg kg-1	ICP-OES	11.0				-0.6		IAEA-452
4	As	mg kg-1	ICP-MS	10.5	0.2	0.3	2.0	-0.9	-2.9	TORT-2
5	As	mg kg-1	ICP-MS	12.4	0.1	0.3	2.0	0.3	1.1	DORM-3
6	As	mg kg-1	ET-AAS	11.8	0.3	0.7	2.0	-0.1	-0.2	DORM-3
7	As	mg kg-1	ICP-MS	12.6	0.5	5.0	2.0	0.5	1.0	TORT-2
8	As	mg kg-1	NAA	11.8	0.5	1.0	2.0	-0.1	-0.2	NIST 1547
9	As	mg kg-1	ICP-MS	10.2	2.6	5.1	2.0	-1.1	-0.6	IAEA-SL-1
10	As	mg kg-1	ET-AAS	11.8	1.5	3.0	2.0	-0.1	-0.1	NIST 2976
13	As	mg kg-1	ICP-MS	11.4	1.1	2.3	2.0	-0.3	-0.4	DORM-2
14	As	mg kg-1	NAA	11.2	0.4	0.8	2.0	-0.5	-1.2	NIST 2976
15	As	mg kg-1	ICP-MS	12.8	4.3	8.7	2.0	0.6	0.2	TORT-3
16	As	mg kg-1	ICP-MS	10.8	0.1	0.2	2.0	-0.7	-2.4	NIST 1548
18	As	mg kg-1	ICP-MS	13.1	0.6	1.2	2.0	0.8	1.6	IAEA-407
23	As	mg kg-1	ICP-MS	12.6	0.5	0.9	2.0	0.5	1.1	ERM-CE278K
25	As	mg kg-1	NAA	12.0	0.3	0.6	2.0	0.1	0.2	NIST 1566b
26	As	mg kg-1	ICP-MS	13.5	0.2	0.4	2.0	1.1	3.3	NIST 1566b
28	As	mg kg-1	ICP-MS	10.6	0.6	1.2	2.0	-0.9	-1.7	ESA-2
30	As	mg kg-1	ICP-MS	14.5				1.7		TORT-2
33	As	mg kg-1	ICP-OES	11.9	0.5	1.0	2.0	0.0	0.0	TORT-2

TABLE 7. REPORTED RESULTS BY PARTICPANTS (cont.)

Lab Code	Element	Unit	Instrumental Method	Lab Mean	Lab u	Lab U	*k*	*z*-score	Zeta-score	QC
34	As	mg kg-1	ET-AAS	12.6	1.0	2.0	2.0	0.5	0.6	TORT-2
35	As	mg kg-1	ICP-MS	12.3		1.5	2.0	0.3		NIST 2976
22A	As	mg kg-1	NAA	12.0	0.7	1.4	2.0	0.1	0.1	DOLT-4
15	B	mg kg-1	Other	20.5	2.6	5.1	2.0			TORT-3
30	B	mg kg-1	ICP-OES	20.9						TORT-2
7	Ba	mg kg-1	ICP-MS	0.554	0.010					
15	Ba	mg kg-1	ICP-MS	0.494	0.037	0.074	2			TORT-3
28	Ba	mg kg-1	ICP-MS	0.910	0.040	0.080	2			ESA-2
8	Br	mg kg-1	NAA	195.3	7.0	14.0	2.0			NIST 1547
14	Br	mg kg-1	NAA	203.0	7.0	14.0	2.0			NIST 2976
25	Br	mg kg-1	NAA	194.0	4.5	9.0	2.0			NIST 1566b
22A	Br	mg kg-1	NAA	170.0	8.0	16.0	2.0			IAEA-407
1	Ca	mg kg-1	ICP-MS	2620	156	312	2	0.6	0.9	NIST 1515
5	Ca	mg kg-1	ICP-MS	2530	8	15	2	0.3	0.7	NIST 1547
7	Ca	mg kg-1	ICP-MS	2473	115	495	2	0.1	0.2	TORT-2
8	Ca	mg kg-1	NAA	2282	104	208	2	-0.5	-0.8	NIST 1547
9	Ca	mg kg-1	ICP-OES	2243	337	673	2	-0.6	-0.5	
14	Ca	mg kg-1	NAA	2537	120	240	2	0.4	0.6	NIST 2976
15	Ca	mg kg-1	ICP-MS	2548	192	384	2	0.4	0.5	TORT-3
16	Ca	mg kg-1	ICP-MS	2310	134	267	2	-0.4	-0.6	NIST 1548
25	Ca	mg kg-1	NAA	2140	214	430	2	-1.0	-1.1	NIST 1566b
26	Ca	mg kg-1	ICP-MS	2571	32	64	2	0.5	1.0	NIST 1566b

TABLE 7. REPORTED RESULTS BY PARTICPANTS (cont.)

Lab Code	Element	Unit	Instrumental Method	Lab Mean	Lab u	Lab U	*k*	*z*-score	Zeta-score	QC
28	Ca	mg kg-1	ICP-MS	2393	30	60	2	-0.1	-0.3	ESA-2
30	Ca	mg kg-1	ICP-OES	2480				0.2		TORT-2
1	Cd	mg kg-1	ICP-MS	3.17	0.13	0.25	2.00	0.1	0.2	TORT-2
2	Cd	mg kg-1	ICP-MS	3.32	0.11	0.21	2.00	0.4	1.1	NIST 1566b
3	Cd	mg kg-1	ET-AAS	3.18				0.1		IAEA-452
4	Cd	mg kg-1	ICP-MS	3.10	0.11	0.22	2.00	-0.1	-0.2	TORT-2
5	Cd	mg kg-1	ICP-MS	3.28	0.04	0.08	2.00	0.4	1.1	DORM-3
6	Cd	mg kg-1	F-AAS	2.92	0.07	0.14	2.00	-0.6	-1.6	NIST 1566b
7	Cd	mg kg-1	ICP-MS	3.26	0.18	1.30	2.00	0.3	0.6	TORT-2
9	Cd	mg kg-1	ICP-MS	2.65	0.67	1.33	2.00	-1.2	-0.7	IAEA-SL-1
10	Cd	mg kg-1	ET-AAS	2.93	0.37	0.73	2.00	-0.5	-0.5	NIST 2976
13	Cd	mg kg-1	ICP-MS	3.26	0.33	0.65	2.00	0.3	0.4	IAEA-452
14	Cd	mg kg-1	ET-AAS	2.46	0.10	0.19	2.00	-1.7	-4.4	NIST 2976
15	Cd	mg kg-1	ICP-MS	3.46	0.29	0.58	2.00	0.8	1.0	TORT-3
16	Cd	mg kg-1	ICP-MS	3.29	0.19	0.38	2.00	0.4	0.7	TORT-2
17	Cd	mg kg-1	ET-AAS	2.61			2.00	-1.4		DOLT-2
18	Cd	mg kg-1	ICP-MS	3.26	0.16	0.31	1.96	0.3	0.6	IAEA-140
19	Cd	mg kg-1	ICP-MS	3.23	0.24	0.48	2.00	0.2	0.3	NIST 1566b
23	Cd	mg kg-1	ICP-MS	3.10	0.10	0.20	2.00	-0.1	-0.3	ERM-CE278K
26	Cd	mg kg-1	ICP-MS	3.71	0.05	0.09	2.00	1.4	4.4	NIST 1566b
27	Cd	mg kg-1	F-AAS	2.55				-1.5		TORT-2
28	Cd	mg kg-1	ICP-MS	3.01	0.07	0.14	2.00	-0.3	-0.9	ESA-2

TABLE 7. REPORTED RESULTS BY PARTICPANTS (cont.)

Lab Code	Element	Unit	Instrumental Method	Lab Mean	Lab u	Lab U	*k*	*z*-score	Zeta-score	QC
30	Cd	mg kg-1	ICP-MS	3.14				0.0		TORT-2
31	Cd	mg kg-1	SV	3.07	0.03	0.06	2.00	-0.2	-0.6	NIST 1566b
32	Cd	mg kg-1	ICP-MS	3.03	0.12	0.24	2.00	-0.3	-0.6	NIST 1566a
33	Cd	mg kg-1	ICP-OES	3.52	0.11	0.21	2.00	1.0	2.4	TORT-2
35	Cd	mg kg-1	ICP-MS	3.41	0.20	0.40	2.00	0.7	1.2	NIST 2976
22B	Cd	mg kg-1	ICP-MS	2.47	0.06	0.12	2.00	-1.7	-5.0	NIST 1566B
15	Ce	mg kg-1	Other	0.025	0.003	0.006	2			TORT-3
5	CH_3Hg	µg kg-1 as Hg	CV-AFS	5.31	0.13	0.26	2.00	0.1	0.2	DOLT-4
8	CH_3Hg	µg kg-1 as Hg	CV-AFS	3.21	0.14	0.28	2.00	-3.1	-4.4	T-38
12	CH_3Hg	µg kg-1 as Hg	CV-AFS	5.29	0.53	1.06	2.00	0.1	0.1	DORM-2
15	CH_3Hg	µg kg-1 as Hg	GC-AFS	6.50	0.64	1.29	2.00	2.0	1.6	TORT-3
20	CH_3Hg	µg kg-1 as Hg	GC-ECD	6.04				1.3		IAEA-452
26	CH_3Hg	µg kg-1 as Hg	HPLC-AFS	7.11	0.24	0.48	2.00	2.9	3.8	NIST 1566b
27	CH_3Hg	µg kg-1 as Hg	CV-AAS	9.22				6.1		TORT-2
30	CH_3Hg	µg kg-1 as Hg	Solid-AAS	7.70				3.8		TORT-2
34	CH_3Hg	µg kg-1 as Hg	Solid-AAS	5.10	0.40	0.80	2.00	-0.2	-0.2	IAEA-452
35	CH_3Hg	µg kg-1 as Hg	ID-ICP-MS	4.80	0.10	0.20	2.00	-0.6	-0.9	IAEA-461
36	CH_3Hg	µg kg-1 as Hg	GC-AFS	5.36	0.40	0.80	2.00	0.2	0.2	IAEA-452
1	Co	mg kg-1	ICP-MS	0.200	0.015	0.030	2	0.0	-0.1	
2	Co	mg kg-1	ICP-MS	0.213	0.007	0.014	2	0.5	0.8	NIST 1566b

TABLE 7. REPORTED RESULTS BY PARTICPANTS (cont.)

Lab Code	Element	Unit	Instrumental Method	Lab Mean	Lab u	Lab U	*k*	*z*-score	Zeta-score	QC
4	Co	mg kg-1	ICP-MS	0.162	0.008	0.015	2	-1.6	-2.7	TORT-2
5	Co	mg kg-1	ICP-MS	0.199	0.005	0.011	2	-0.1	-0.1	NIST 1547
7	Co	mg kg-1	ICP-MS	0.208	0.007			0.3	0.5	TORT-2
8	Co	mg kg-1	NAA	0.184	0.008	0.016	2	-0.7	-1.2	NIST 1547
9	Co	mg kg-1	ICP-MS	0.197	0.040	0.079	2	-0.2	-0.1	IAEA-SL-1
13	Co	mg kg-1	ICP-MS	0.199	0.020	0.040	2	-0.1	-0.1	DORM-2
14	Co	mg kg-1	NAA	0.366	0.010	0.020	2	6.6	10.3	NIST 2976
15	Co	mg kg-1	ICP-MS	0.206	0.018	0.035	2	0.2	0.2	TORT-3
16	Co	mg kg-1	ICP-MS	0.178	0.011	0.021	2	-0.9	-1.4	TORT-2
18	Co	mg kg-1	ICP-MS	0.200	0.014	0.027	2	-0.1	-0.1	IAEA-407
26	Co	mg kg-1	ICP-MS	0.219	0.002	0.005	2	0.7	1.4	NIST 1566b
27	Co	mg kg-1	ET-AAS	0.370				6.7		TORT-2
28	Co	mg kg-1	ICP-MS	0.180	0.010	0.020	2	-0.8	-1.3	ESA-2
30	Co	mg kg-1	ICP-MS	0.170				-1.2		TORT-2
32	Co	mg kg-1	ICP-MS	0.261	0.013	0.026	2	2.4	3.3	NIST 1566a
34	Co	mg kg-1	ET-AAS	0.164	0.016	0.033	2	-1.5	-1.8	TORT-2
35	Co	mg kg-1	ICP-MS	0.210	0.012	0.025	2	0.4	0.5	NIST 2976
22A	Co	mg kg-1	NAA	0.182	0.008	0.016	2	-0.8	-1.3	DOLT-4
22B	Co	mg kg-1	ICP-MS	0.245	0.009	0.018	2	1.8	2.9	NIST 1566B
1	Cr	mg kg-1	ICP-MS	1.00	0.10	0.19	2.00	0.3	0.3	TORT-2
2	Cr	mg kg-1	ICP-MS	1.22	0.05	0.10	2.00	2.1	3.5	NIST 1566b
3	Cr	mg kg-1	ICP-OES	1.15				1.5		IAEA-140

TABLE 7. REPORTED RESULTS BY PARTICPANTS (cont.)

Lab Code	Element	Unit	Instrumental Method	Lab Mean	Lab u	Lab U	*k*	*z*-score	Zeta-score	QC
5	Cr	mg kg-1	ICP-MS	0.758	0.060	0.120	2	-1.7	-2.5	DORM-3
7	Cr	mg kg-1	ICP-MS	0.956	0.047			-0.1	-0.1	TORT-2
8	Cr	mg kg-1	NAA	0.977	0.060	0.120	2	0.1	0.1	NIST 1547
9	Cr	mg kg-1	ICP-MS	0.843	0.085	0.169	2	-1.0	-1.2	IAEA-SL-1
10	Cr	mg kg-1	ET-AAS	0.820	0.103	0.205	2	-1.2	-1.2	NIST 2976
13	Cr	mg kg-1	ICP-MS	0.965	0.145	0.290	2	0.0	0.0	DORM-2
14	Cr	mg kg-1	NAA	0.840	0.060	0.120	2	-1.0	-1.5	NIST 2976
15	Cr	mg kg-1	ICP-MS	0.903	0.146	0.291	2	-0.5	-0.4	TORT-3
16	Cr	mg kg-1	ICP-MS	0.848	0.049	0.098	2	-1.0	-1.6	TORT-2
18	Cr	mg kg-1	ICP-MS	0.923	0.051	0.099	2	-0.4	-0.6	IAEA-407
26	Cr	mg kg-1	ICP-MS	0.963	0.011	0.022	2	0.0	0.0	
27	Cr	mg kg-1	ET-AAS	0.980				0.1		TORT-2
28	Cr	mg kg-1	ICP-MS	1.01	0.02	0.04	2.00	0.3	0.7	ESA-2
33	Cr	mg kg-1	ICP-OES	0.776	0.135	0.270	2	-1.6	-1.3	TORT-2
34	Cr	mg kg-1	ET-AAS	1.03	0.10	0.20	2.00	0.5	0.6	TORT-2
22A	Cr	mg kg-1	NAA	0.990	0.120	0.240	2	0.2	0.2	DORM-2
8	Cs	mg kg-1	NAA	0.034	0.001	0.002	2			NIST 1547
14	Cs	mg kg-1	NAA	0.027	0.005	0.010	2			NIST 2976
1	Cu	mg kg-1	ICP-MS	144.0	5.5	11.0	2.0	-0.1	-0.2	TORT-2
2	Cu	mg kg-1	ICP-MS	152.4	4.3	8.6	2.0	0.4	0.8	NIST 1566b
3	Cu	mg kg-1	ICP-OES	162.0				0.9		
4	Cu	mg kg-1	ICP-MS	130.0	4.5	9.0	2.0	-0.9	-2.0	TORT-2

TABLE 7. REPORTED RESULTS BY PARTICPANTS (cont.)

Lab Code	Element	Unit	Instrumental Method	Lab Mean	Lab u	Lab U	*k*	*z*-score	Zeta-score	QC
5	Cu	mg kg-1	ICP-MS	146.0	3.4	6.9	2.0	0.0	0.0	DORM-3
6	Cu	mg kg-1	F-AAS	143.0	2.0	3.9	2.0	-0.2	-0.4	DORM-3
7	Cu	mg kg-1	ICP-MS	138.6	4.4	69.3	2.0	-0.4	-0.9	TORT-2
9	Cu	mg kg-1	ICP-MS	131.0	13.0	26.0	2.0	-0.8	-1.0	IAEA-SL-1
10	Cu	mg kg-1	ET-AAS	146.0	18.3	36.5	2.0	0.0	0.0	NIST 2976
13	Cu	mg kg-1	ICP-MS	144.8	10.9	21.7	2.0	-0.1	-0.1	IAEA-452
15	Cu	mg kg-1	ICP-MS	154.7	11.9	23.8	2.0	0.5	0.6	TORT-3
16	Cu	mg kg-1	ICP-MS	153.0	8.5	17.0	2.0	0.4	0.7	TORT-2
17	Cu	mg kg-1	ET-AAS	154.8			2.0	0.5		DOLT-2
18	Cu	mg kg-1	F-AAS	146.2	10.3	20.3	2.0	0.0	0.0	IAEA-407
19	Cu	mg kg-1	ICP-MS	156.5	7.8	15.7	2.0	0.6	1.0	NIST 1566b
23	Cu	mg kg-1	ICP-MS	157.3	6.2	12.4	2.0	0.6	1.3	ERM-CE278K
26	Cu	mg kg-1	ICP-MS	161.0	2.0	4.0	2.0	0.8	2.2	NIST 1566b
27	Cu	mg kg-1	F-AAS	96.2				-2.7		TORT-2
28	Cu	mg kg-1	ICP-MS	136.7	3.0	6.0	2.0	-0.5	-1.3	ESA-2
30	Cu	mg kg-1	ICP-OES	149.0				0.2		TORT-2
31	Cu	mg kg-1	SV	133.0	2.0	4.0	2.0	-0.7	-1.9	NIST 1566b
32	Cu	mg kg-1	ICP-MS	142.1	5.7	11.4	2.0	-0.2	-0.5	NIST 1566a
33	Cu	mg kg-1	ICP-OES	142.9	4.6	9.2	2.0	-0.2	-0.4	TORT-2
34	Cu	mg kg-1	F-AAS	150.0	8.0	15.0	2.0	0.2	0.4	TORT-2
35	Cu	mg kg-1	ICP-MS	153.0	9.0	18.0	2.0	0.4	0.6	NIST 2976
22B	Cu	mg kg-1	ICP-MS	140.0	3.0	6.0	2.0	-0.3	-0.8	NIST 1566B

TABLE 7. REPORTED RESULTS BY PARTICPANTS (cont.)

Lab Code	Element	Unit	Instrumental Method	Lab Mean	Lab u	Lab U	*k*	*z*-score	Zeta-score	QC
1	Fe	mg kg-1	ICP-MS	137.0	8.5	17.0	2.0	0.4	0.6	TORT-2
2	Fe	mg kg-1	ICP-MS	124.3	4.8	9.6	2.0	-0.4	-0.9	NIST 1566b
3	Fe	mg kg-1	ICP-OES	138.0				0.4		IAEA-452
5	Fe	mg kg-1	ICP-MS	125.0	3.0	5.9	2.0	-0.4	-0.9	DORM-3
6	Fe	mg kg-1	F-AAS	131.0	1.6	3.2	2.0	0.0	0.0	DORM-3
7	Fe	mg kg-1	ICP-MS	132.1	1.2			0.1	0.2	TORT-2
8	Fe	mg kg-1	NAA	133.0	5.0	10.0	2.0	0.1	0.3	NIST 1547
9	Fe	mg kg-1	ICP-MS	129.0	18.0	36.0	2.0	-0.1	-0.1	IAEA-SL-1
10	Fe	mg kg-1	ET-AAS	135.0	16.9	33.8	2.0	0.2	0.2	NIST 2976
13	Fe	mg kg-1	ICP-MS	123.5	9.3	18.5	2.0	-0.5	-0.7	DORM-2
14	Fe	mg kg-1	NAA	123.0	4.0	8.0	2.0	-0.5	-1.3	NIST 2976
15	Fe	mg kg-1	ICP-OES	139.1	15.8	31.6	2.0	0.5	0.5	TORT-3
16	Fe	mg kg-1	ICP-MS	135.0	7.5	15.0	2.0	0.2	0.4	GBW10014
17	Fe	mg kg-1	ET-AAS	153.2			2.0	1.4		DOLT-2
18	Fe	mg kg-1	F-AAS	127.3	6.9	13.5	2.0	-0.2	-0.4	IAEA-407
25	Fe	mg kg-1	NAA	136.0	4.9	10.0	2.0	0.3	0.6	NIST 1566b
26	Fe	mg kg-1	ICP-MS	153.3	1.4	2.7	2.0	1.4	3.6	NIST 1566b
27	Fe	mg kg-1	F-AAS	102.0				-1.8		TORT-2
28	Fe	mg kg-1	ICP-MS	168.7	2.0	4.0	2.0	2.3	6.0	ESA-2
30	Fe	mg kg-1	ICP-OES	126.0				-0.3		TORT-2
32	Fe	mg kg-1	ICP-MS	141.4	8.5	17.0	2.0	0.6	1.0	NIST 1566a
34	Fe	mg kg-1	F-AAS	131.0	12.0	23.0	2.0	0.0	0.0	TORT-2

TABLE 7. REPORTED RESULTS BY PARTICPANTS (cont.)

Lab Code	Element	Unit	Instrumental Method	Lab Mean	Lab u	Lab U	*k*	*z*-score	Zeta-score	QC
22A	Fe	mg kg-1	NAA	127.9	4.8	9.6	2.0	-0.2	-0.4	DOLT-4
1	Hg	µg kg-1	CV-AFS	18.0	2.5	5.0	2.0	-1.2	-1.1	TORT-2
5	Hg	µg kg-1	CV-AFS	14.8	0.2	0.4	2.0	-2.4	-5.9	DORM-3
6	Hg	µg kg-1	CV-AFS	27.0	2.0	4.0	2.0	2.2	2.6	DORM-3
7	Hg	µg kg-1	ICP-MS	18.0	1.0	7.0	2.0	-1.2	-2.1	TORT-2
8	Hg	µg kg-1	Solid-AAS	19.9	0.5	1.0	2.0	-0.5	-1.0	NIST 1566b
9	Hg	µg kg-1	CV-AAS	21.0	8.0	16.0	2.0	0.0	0.0	IAEA-SL-1
12	Hg	µg kg-1	CV-AFS	18.7	1.7	3.4	2.0	-0.9	-1.2	DORM-3
14	Hg	µg kg-1	CV-AAS	23.7	2.1	4.3	2.0	1.0	1.1	NIST 1566b
15	Hg	µg kg-1	CV-AFS	22.3	3.4	6.7	2.0	0.5	0.3	TORT-3
16	Hg	µg kg-1	ICP-MS	6.41	0.35	0.70	2.00	-5.6	-13.3	TORT-2
17	Hg	µg kg-1	Solid-AAS	21.5			2.0	0.2		IAEA-452
18	Hg	µg kg-1	CV-AAS	27.2	1.6	3.1	2.0	2.3	3.2	IAEA-140
19	Hg	µg kg-1	CV-AAS	21.0	2.0	3.0	2.0	0.0	0.0	IAEA-142
20	Hg	µg kg-1	Solid-AAS	14.8				-2.4		NIST 2976
26	Hg	µg kg-1 as Hg	ICP-MS	25.0	0.9	1.7	2.0	1.5	2.8	
27	Hg	µg kg-1 as Hg	CV-AAS	35.0				5.3		TORT-2
28	Hg	µg kg-1	Solid-AAS	20.2	0.2	0.4	2.0	-0.4	-0.9	ESA-2
30	Hg	µg kg-1	Solid-AAS	19.7				-0.5		TORT-2
33	Hg	µg kg-1	Solid-AAS	16.5	1.2	2.4	2.0	-1.7	-2.9	TORT-2
34	Hg	µg kg-1	Solid-AAS	22.5	0.9	1.8	2.0	0.5	1.0	NIST 2976
35	Hg	µg kg-1	ID-ICP-MS	21.3		1.1	2.0	0.1		

TABLE 7. REPORTED RESULTS BY PARTICPANTS (cont.)

Lab Code	Element	Unit	Instrumental Method	Lab Mean	Lab u	Lab U	*k*	*z*-score	Zeta-score	QC
36	Hg	µg kg-1	CV-AFS	23.8	1.6	3.2	2.0	1.0	1.4	IAEA-452
8B	Hg	µg kg-1	CV-AAS	20.1	1.1	2.2	2.0	-0.4	-0.7	NIST 1566b
7	K	mg kg-1	ICP-MS	13332	600	4000	2	0.4	0.5	TORT-2
8	K	mg kg-1	NAA	11305	714	1428	2	-0.9	-1.1	NIST 1547
15	K	mg kg-1	ICP-OES	14942	1302	2603	2	1.4	1.3	TORT-3
25	K	mg kg-1	NAA	11200	258	511	2	-1.0	-1.4	NIST 1566b
28	K	mg kg-1	ICP-MS	11867	400	800	2	-0.5	-0.7	ESA-2
30	K	mg kg-1	ICP-OES	13700				0.6		TORT-2
2	Li	mg kg-1	ICP-MS	0.535	0.015	0.031	2	0.0	0.1	NIST 1566b
9	Li	mg kg-1	ICP-MS	0.416	0.083	0.166	2	-1.7	-1.2	
16	Li	mg kg-1	ICP-MS	0.474	0.028	0.055	2	-0.9	-1.1	GBW10014
18	Li	mg kg-1	ICP-MS	0.602	0.029	0.056	2	1.0	1.3	IAEA-407
28	Li	mg kg-1	ICP-MS	0.593	0.020	0.040	2	0.9	1.2	ESA-2
34	Li	mg kg-1	ET-AAS	0.557	0.045	0.090	2	0.4	0.4	TORT-2
1	Mg	mg kg-1	ICP-MS	3270	153	305	2	0.5	0.8	NIST 1515
2	Mg	mg kg-1	ICP-MS	3208	91	182	2	0.3	0.6	NIST 1566b
5	Mg	mg kg-1	ICP-MS	3030	73	146	2	-0.1	-0.2	NIST 1547
7	Mg	mg kg-1	ICP-MS	3112	124	622	2	0.1	0.1	TORT-2
9	Mg	mg kg-1	ICP-OES	2968	445	890	2	-0.3	-0.2	
15	Mg	mg kg-1	ICP-MS	3322	260	521	2	0.6	0.7	TORT-3
16	Mg	mg kg-1	ICP-MS	2960	170	340	2	-0.3	-0.5	NIST 1548
25	Mg	mg kg-1	NAA	2815	101	203	2	-0.7	-1.2	NIST 1566b

TABLE 7. REPORTED RESULTS BY PARTICPANTS (cont.)

Lab Code	Element	Unit	Instrumental Method	Lab Mean	Lab u	Lab U	*k*	*z*-score	Zeta-score	QC
26	Mg	mg kg-1	ICP-MS	3687	46	93	2	1.6	3.0	NIST 1566b
28	Mg	mg kg-1	ICP-MS	3007	60	120	2	-0.2	-0.4	ESA-2
30	Mg	mg kg-1	ICP-OES	3060				-0.1		TORT-2
1	Mn	mg kg-1	ICP-MS	74.5	3.8	7.5	2.0	0.9	1.7	TORT-2
2	Mn	mg kg-1	ICP-MS	66.7	1.7	3.4	2.0	0.0	0.0	NIST 1566b
3	Mn	mg kg-1	ICP-OES	71.8				0.6		IAEA-140
4	Mn	mg kg-1	ICP-MS	62.3	2.2	4.4	2.0	-0.5	-1.3	TORT-2
5	Mn	mg kg-1	ICP-MS	64.7	0.4	0.7	2.0	-0.2	-0.7	NIST 1547
6	Mn	mg kg-1	F-AAS	66.3	1.7	3.3	2.0	0.0	-0.1	NIST 1566b
7	Mn	mg kg-1	ICP-MS	73.1	2.6			0.8	1.7	TORT-2
9	Mn	mg kg-1	ICP-OES	61.3	9.2	18.4	2.0	-0.6	-0.6	IAEA-SL-1
10	Mn	mg kg-1	ET-AAS	65.0	8.1	16.3	2.0	-0.2	-0.2	NIST 2976
13	Mn	mg kg-1	ICP-MS	67.9	3.4	6.8	2.0	0.1	0.3	IAEA-452
15	Mn	mg kg-1	ICP-MS	73.3	5.0	10.1	2.0	0.8	1.2	TORT-3
16	Mn	mg kg-1	ICP-MS	68.0	3.9	7.8	2.0	0.2	0.3	GBW10014
17	Mn	mg kg-1	ET-AAS	64.0			2.0	-0.3		DOLT-2
18	Mn	mg kg-1	ICP-MS	80.8	4.2	8.2	2.0	1.7	2.9	IAEA-140
25	Mn	mg kg-1	NAA	63.0	6.3	13.0	2.0	-0.4	-0.5	NIST 1566b
26	Mn	mg kg-1	ICP-MS	77.9	0.8	1.5	2.0	1.3	4.0	NIST 1566b
27	Mn	mg kg-1	F-AAS	52.4				-1.7		TORT-2
28	Mn	mg kg-1	ICP-MS	60.2	1.0	2.0	2.0	-0.8	-2.3	ESA-2
30	Mn	mg kg-1	ICP-OES	67.3				0.1		TORT-2

TABLE 7. REPORTED RESULTS BY PARTICPANTS (cont.)

Lab Code	Element	Unit	Instrumental Method	Lab Mean	Lab u	Lab U	*k*	*z*-score	Zeta-score	QC
32	Mn	mg kg-1	ICP-MS	69.5	2.8	5.6	2.0	0.3	0.7	NIST 1566a
33	Mn	mg kg-1	ICP-OES	65.1	3.5	7.0	2.0	-0.2	-0.4	TORT-2
34	Mn	mg kg-1	F-AAS	73.3	5.9	11.7	2.0	0.8	1.0	TORT-2
35	Mn	mg kg-1	ICP-MS	66.9	4.0	8.0	2.0	0.0	0.0	NIST 2976
15	Mo	mg kg-1	ICP-MS	0.316	0.051	0.101	2			TORT-3
30	Mo	mg kg-1	ICP-MS	0.490						TORT-2
2	Na	mg kg-1	ICP-MS	17660	720	1430	2	-0.8	-1.5	NIST 1566b
7	Na	mg kg-1	ICP-MS	21248	718	8499	2	0.6	1.1	TORT-2
8	Na	mg kg-1	NAA	20553	829	1658	2	0.3	0.6	NIST 1547
14	Na	mg kg-1	NAA	18828	339	678	2	-0.4	-0.7	NIST 2976
15	Na	mg kg-1	ICP-OES	22213	1658	3315	2	1.0	1.2	TORT-3
25	Na	mg kg-1	NAA	20276	487	810	2	0.2	0.5	NIST 1566b
28	Na	mg kg-1	ICP-MS	19500	200	400	2	-0.1	-0.2	ESA-2
30	Na	mg kg-1	ICP-OES	22100				1.0		TORT-2
22A	Na	mg kg-1	NAA	19600	900	1800	2	0.0	-0.1	IAEA-407
1	Ni	mg kg-1	ICP-MS	0.770	0.075	0.150	2	-0.7	-0.5	TORT-2
2	Ni	mg kg-1	ICP-MS	0.876	0.026	0.052	2	0.3	0.2	NIST 1566b
3	Ni	mg kg-1	ET-AAS	0.802				-0.4		IAEA-140
5	Ni	mg kg-1	ICP-MS	0.660	0.010	0.020	2	-1.8	-1.3	DORM-3
7	Ni	mg kg-1	ICP-MS	0.820	0.005			-0.3	-0.2	TORT-2
9	Ni	mg kg-1	ICP-MS	0.758	0.133	0.265	2	-0.9	-0.5	IAEA-SL-1
10	Ni	mg kg-1	ET-AAS	0.950	0.119	0.238	2	1.0	0.5	NIST 2976

TABLE 7. REPORTED RESULTS BY PARTICPANTS (cont.)

Lab Code	Element	Unit	Instrumental Method	Lab Mean	Lab u	Lab U	*k*	*z*-score	Zeta-score	QC
13	Ni	mg kg-1	ICP-MS	0.820	0.082	0.164	2	-0.3	-0.2	DORM-2
15	Ni	mg kg-1	ICP-MS	0.776	0.174	0.348	2	-0.7	-0.3	TORT-3
16	Ni	mg kg-1	ICP-MS	0.713	0.041	0.082	2	-1.3	-0.9	NIST 1548
18	Ni	mg kg-1	ICP-MS	0.963	0.122	0.240	2	1.1	0.6	IAEA-407
19	Ni	mg kg-1	ICP-MS	0.920	0.090	0.180	2	0.7	0.4	NIST 1566b
26	Ni	mg kg-1	ICP-MS	0.817	0.011	0.022	2	-0.3	-0.2	NIST 1566b
27	Ni	mg kg-1	ET-AAS	0.800				-0.5		TORT-2
28	Ni	mg kg-1	ICP-MS	0.913	0.060	0.120	2	0.6	0.4	ESA-2
30	Ni	mg kg-1	ICP-MS	0.690				-1.5		TORT-2
32	Ni	mg kg-1	ICP-MS	0.840	0.042	0.084	2	-0.1	-0.1	NIST 1566a
34	Ni	mg kg-1	ET-AAS	0.891	0.090	0.180	2	0.4	0.2	TORT-2
1	Pb	mg kg-1	ICP-MS	0.340	0.030	0.060	2	-0.5	-0.5	TORT-2
2	Pb	mg kg-1	ICP-MS	0.376	0.008	0.016	2	0.3	0.5	NIST 1566b
3	Pb	mg kg-1	ET-AAS	1.01				14		IAEA-140
5	Pb	mg kg-1	ICP-MS	0.355	0.005	0.010	2	-0.1	-0.2	NIST 1547
6	Pb	mg kg-1	ET-AAS	0.333	0.011	0.022	2	-0.6	-1.0	DORM-3
7	Pb	mg kg-1	ICP-MS	0.329	0.019	0.165	2	-0.7	-1.0	TORT-2
9	Pb	mg kg-1	ICP-MS	0.355	0.045	0.089	2	-0.1	-0.1	IAEA-SL-1
10	Pb	mg kg-1	ET-AAS	0.930	0.116	0.233	2	13	4.8	NIST 2976
13	Pb	mg kg-1	ICP-MS	0.370	0.046	0.093	2	0.2	0.2	DORM-2
14	Pb	mg kg-1	ET-AAS	0.381	0.038	0.076	2	0.4	0.4	NIST 2976
15	Pb	mg kg-1	ICP-MS	0.370	0.133	0.265	2	0.2	0.1	TORT-3

TABLE 7. REPORTED RESULTS BY PARTICPANTS (cont.)

Lab Code	Element	Unit	Instrumental Method	Lab Mean	Lab u	Lab U	*k*	*z*-score	Zeta-score	QC
16	Pb	mg kg-1	ICP-MS	0.347	0.020	0.040	2	-0.3	-0.4	TORT-2
19	Pb	mg kg-1	ICP-MS	0.370	0.030	0.060	2	0.2	0.2	NIST 1566b
23	Pb	mg kg-1	ICP-MS	0.500	0.030	0.060	2	3.1	3.5	ERM-CE278K
26	Pb	mg kg-1	ICP-MS	0.380	0.003	0.007	2	0.4	0.7	NIST 1566b
27	Pb	mg kg-1	ET-AAS	0.220				-3.1		TORT-2
28	Pb	mg kg-1	ICP-MS	0.297	0.006	0.012	2	-1.4	-2.4	ESA-2
30	Pb	mg kg-1	ICP-MS	0.380				0.4		TORT-2
31	Pb	mg kg-1	SV	0.395	0.050	0.100	2	0.8	0.6	NIST 1566b
35	Pb	mg kg-1	ICP-MS	0.362	0.020	0.043	2	0.0	0.0	NIST 2976
22B	Pb	mg kg-1	ICP-MS	0.310	0.013	0.026	2	-1.1	-1.7	NIST 1566B
8	Rb	mg kg-1	NAA	5.13	0.19	0.38	2.00	0.0	0.0	NIST 1547
13	Rb	mg kg-1	ICP-MS	5.07	0.38	0.76	2.00	-0.1	-0.1	IAEA-452
14	Rb	mg kg-1	NAA	4.80	0.20	0.40	2.00	-0.5	-1.0	NIST 2976
15	Rb	mg kg-1	Other	5.41	0.51	1.02	2.00	0.4	0.5	TORT-3
25	Rb	mg kg-1	NAA	5.30	0.14	0.30	2.00	0.2	0.5	NIST 1566b
15	Sb	mg kg-1	Other	0.013	0.002	0.005	2			TORT-3
8	Sc	mg kg-1	NAA	0.026	0.001	0.002	2			NIST 1547
14	Sc	mg kg-1	NAA	0.026	0.0002	0.0004	2			NIST 2976
1	Se	mg kg-1	ICP-MS	3.73	0.18	0.36	2.00	1.8	2.7	TORT-2
5	Se	mg kg-1	ICP-MS	3.16	0.13	0.26	2.00	0.3	0.5	DORM-3
7	Se	mg kg-1	ICP-MS	3.00	0.02	1.50	2.00	-0.2	-0.4	TORT-2
8	Se	mg kg-1	NAA	3.22	0.13	0.26	2.00	0.4	0.7	NIST 1547

TABLE 7. REPORTED RESULTS BY PARTICPANTS (cont.)

Lab Code	Element	Unit	Instrumental Method	Lab Mean	Lab u	Lab U	*k*	*z*-score	Zeta-score	QC
9	Se	mg kg-1	ICP-MS	2.66	0.67	1.33	2.00	-1.0	-0.6	IAEA-SL-1
13	Se	mg kg-1	ICP-MS	3.11	0.31	0.62	2.00	0.1	0.1	DORM-2
14	Se	mg kg-1	NAA	2.90	0.20	0.40	2.00	-0.4	-0.6	NIST 2976
15	Se	mg kg-1	ICP-MS	3.20	0.34	0.68	2.00	0.4	0.4	TORT-3
16	Se	mg kg-1	ICP-MS	2.92	0.17	0.34	2.00	-0.4	-0.6	TORT-2
25	Se	mg kg-1	NAA	3.20	0.08	0.16	2.00	0.4	0.8	NIST 1566b
26	Se	mg kg-1	ICP-MS	3.69	0.08	0.16	2.00	1.6	3.5	NIST 1566b
28	Se	mg kg-1	AFS	2.64	0.02	0.04	2.00	-1.1	-2.5	ESA-2
30	Se	mg kg-1	ICP-MS	3.19				0.3		TORT-2
34	Se	mg kg-1	ET-AAS	2.99	0.30	0.60	2.00	-0.2	-0.2	TORT-2
22A	Se	mg kg-1	NAA	2.74	0.15	0.30	2.00	-0.8	-1.4	DOLT-4
7	Sn	mg kg-1	ICP-MS	0.034	0.0005					
9	Sn	mg kg-1	ICP-MS	0.316	0.063	0.126	2			
16	Sn	mg kg-1	ICP-MS	0.000036	0.000002	0.000004	2			NIST 1548
2	Sr	mg kg-1	ICP-MS	21.1	0.5	1.1	2.0	0.2	0.5	NIST 1566b
3	Sr	mg kg-1	ICP-OES	17.1				-1.4		IAEA-140
7	Sr	mg kg-1	ICP-MS	23.2	1.1			1.0	2.0	TORT-2
9	Sr	mg kg-1	ICP-MS	20.3	3.1	6.1	2.0	-0.1	-0.1	IAEA-SL-1
13	Sr	mg kg-1	ICP-MS	20.0	1.5	3.0	2.0	-0.2	-0.3	IAEA-452
15	Sr	mg kg-1	ICP-MS	20.5	1.6	3.1	2.0	0.0	-0.1	TORT-3
16	Sr	mg kg-1	ICP-MS	18.5	1.1	2.1	2.0	-0.8	-1.6	GBW10014
18	Sr	mg kg-1	ICP-MS	27.1	2.2	4.2	2.0	2.5	2.8	IAEA-407

TABLE 7. REPORTED RESULTS BY PARTICPANTS (cont.)

Lab Code	Element	Unit	Instrumental Method	Lab Mean	Lab u	Lab U	*k*	*z*-score	Zeta-score	QC
26	Sr	mg kg-1	ICP-MS	24.8	0.3	0.7	2.0	1.6	4.8	NIST 1566b
28	Sr	mg kg-1	ICP-MS	18.9	0.5	1.0	2.0	-0.7	-1.8	ESA-2
30	Sr	mg kg-1	ICP-MS	20.3				-0.1		TORT-2
32	Sr	mg kg-1	ICP-MS	21.7	0.9	1.7	2.0	0.4	0.9	NIST 1566a
34	Sr	mg kg-1	ET-AAS	21.1	1.7	3.4	2.0	0.2	0.3	TORT-2
35	Sr	mg kg-1	ICP-MS	22.3	1.4	2.7	2.0	0.7	1.1	NIST 2976
22B	Sr	mg kg-1	ICP-MS	18.0	0.4	0.8	2.0	-1.0	-2.9	NIST 1566B
30	Ti	mg kg-1	ICP-MS	10.3						TORT-2
15	Tl	mg kg-1	Other	0.006	0.001	0.002	2			TORT-3
8	U	mg kg-1	NAA	0.146	0.005	0.010	2			NIST 1547
15	U	mg kg-1	ICP-MS	0.164	0.036	0.073	2			TORT-3
32	U	mg kg-1	ICP-MS	0.171	0.007	0.014	2			NIST 1566a
1	V	mg kg-1	ICP-MS	0.920	0.085	0.170	2	0.2	0.2	
2	V	mg kg-1	ICP-MS	0.977	0.037	0.073	2	0.7	1.0	NIST 1566b
3	V	mg kg-1	ET-AAS	0.610				-2.6		IAEA-140
5	V	mg kg-1	ICP-MS	0.881	0.065	0.130	2	-0.2	-0.2	NIST 1547
7	V	mg kg-1	ICP-MS	0.935	0.036	0.374		0.3	0.5	TORT-2
9	V	mg kg-1	ICP-MS	0.892	0.112	0.223	2	-0.1	-0.1	IAEA-SL-1
13	V	mg kg-1	ICP-MS	0.846	0.085	0.169	2	-0.5	-0.5	IAEA-452
15	V	mg kg-1	ICP-MS	0.964	0.201	0.402	2	0.6	0.3	TORT-3
16	V	mg kg-1	ICP-MS	0.822	0.048	0.095	2	-0.7	-1.0	TORT-2
18	V	mg kg-1	ICP-MS	0.919	0.062	0.121	2	0.2	0.2	IAEA-407

TABLE 7. REPORTED RESULTS BY PARTICPANTS (cont.)

Lab Code	Element	Unit	Instrumental Method	Lab Mean	Lab u	Lab U	*k*	*z*-score	Zeta-score	QC
25	V	mg kg-1	NAA	0.800	0.020	0.040	2	-0.9	-1.5	NIST 1566b
26	V	mg kg-1	ICP-MS	0.958	0.010	0.019	2	0.5	0.9	NIST 1566b
27	V	mg kg-1	ET-AAS	0.860				-0.3		TORT-2
28	V	mg kg-1	ICP-MS	0.880	0.020	0.040	2	-0.2	-0.3	ESA-2
32	V	mg kg-1	ICP-MS	1.16	0.05	0.09	2.00	2.3	3.3	NIST 1566a
33	V	mg kg-1	ICP-OES	0.766	0.094	0.189	2	-1.2	-1.2	TORT-2
34	V	mg kg-1	ET-AAS	0.785	0.078	0.157	2	-1.0	-1.1	TORT-2
1	Zn	mg kg-1	ICP-MS	773.0	32.0	64.0	2.0	0.5	1.2	TORT-2
2	Zn	mg kg-1	ICP-MS	756.0	24.2	48.4	2.0	0.3	0.9	NIST 1566b
3	Zn	mg kg-1	ICP-OES	802.0				0.8		IAEA-452
4	Zn	mg kg-1	ICP-MS	683.0	14.8	29.6	2.0	-0.5	-1.6	TORT-2
5	Zn	mg kg-1	ICP-MS	754.0	14.4	28.8	2.0	0.3	1.0	DORM-3
6	Zn	mg kg-1	F-AAS	682.0	20.3	40.6	2.0	-0.5	-1.4	DORM-3
7	Zn	mg kg-1	ICP-MS	732.1	29.6	366.1	2.0	0.1	0.1	TORT-2
8	Zn	mg kg-1	NAA	755.7	26.0	52.0	2.0	0.3	0.8	NIST 1547
9	Zn	mg kg-1	ICP-OES	607.0	61.0	121.0	2.0	-1.3	-1.8	IAEA-SL-1
10	Zn	mg kg-1	ET-AAS	598.0	74.8	149.5	2.0	-1.4	-1.6	NIST 2976
13	Zn	mg kg-1	ICP-MS	773.5	58.0	116.0	2.0	0.5	0.7	IAEA-452
14	Zn	mg kg-1	NAA	742.0	31.0	62.0	2.0	0.2	0.4	NIST 2976
15	Zn	mg kg-1	ICP-MS	778.1	54.5	109.0	2.0	0.6	0.9	TORT-3
16	Zn	mg kg-1	ICP-MS	741.0	43.0	86.0	2.0	0.2	0.3	TORT-2
18	Zn	mg kg-1	F-AAS	759.5	43.7	85.6	2.0	0.4	0.7	IAEA-407

TABLE 7. REPORTED RESULTS BY PARTICPANTS (cont.)

Lab Code	Element	Unit	Instrumental Method	Lab Mean	Lab u	Lab U	*k*	*z*-score	Zeta-score	QC
19	Zn	mg kg-1	ICP-MS	795.0	40.0	80.0	2.0	0.7	1.5	NIST 1566b
25	Zn	mg kg-1	NAA	740.0	18.5	37.0	2.0	0.1	0.4	NIST 1566b
26	Zn	mg kg-1	ICP-MS	813.6	8.4	16.9	2.0	1.0	3.4	NIST 1566b
27	Zn	mg kg-1	F-AAS	623.0				-1.1		TORT-2
28	Zn	mg kg-1	ICP-MS	691.7	7.0	14.0	2.0	-0.4	-1.4	ESA-2
30	Zn	mg kg-1	ICP-OES	718.0				-0.1		TORT-2
31	Zn	mg kg-1	SV	729.0	8.6	17.2	2.0	0.0	0.1	NIST 1566b
32	Zn	mg kg-1	ICP-MS	668.1	26.7	53.4	2.0	-0.6	-1.6	NIST 1566a
33	Zn	mg kg-1	ICP-OES	756.7	27.5	54.9	2.0	0.3	0.8	TORT-2
34	Zn	mg kg-1	F-AAS	739.0	37.0	74.0	2.0	0.1	0.3	TORT-2
22A	Zn	mg kg-1	NAA	700.0	18.0	36.0	2.0	-0.3	-0.9	DOLT-4
22B	Zn	mg kg-1	ICP-MS	621.0	13.0	26.0	2.0	-1.2	-3.9	NIST 1566B

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