

IAEA Analytical Quality in Nuclear Applications Series No. 23

Worldwide Laboratory Comparison on the Determination of Trace Elements in IAEA-452 Biota Sample



IAEA

International Atomic Energy Agency

**Worldwide Laboratory Comparison on the
Determination of Trace Elements in
IAEA-452 Biota Sample**

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Worldwide Laboratory Comparison on the Determination of Trace Elements in IAEA-452 Biota Sample

INTERNATIONAL ATOMIC ENERGY AGENCY
VIENNA, 2012

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FOREWORD

The primary goal of the IAEA Environment Laboratories (NAEL) is to help Member States understand, monitor and protect the marine environment. Thus, the major impact exerted by large coastal cities on marine ecosystems is an issue of primary concern for the IAEA and its Environment Laboratories. In this regard, it is noteworthy that marine pollution assessments depend on the accurate knowledge of contaminant concentrations in various environmental compartments.

Since the early 1970s, NAEL has been assisting national laboratories and regional laboratory networks through its reference material programme for the analysis of radionuclides, trace elements and organic compounds in marine samples. Relevant activities include global interlaboratory comparison exercises, regional proficiency tests, the production of marine reference materials, and the development of reference methods for trace elements and organic pollutants analysis in marine samples.

Two fundamental requirements for ensuring the reliability of analytical results are quality control (QC) and quality assurance (QA). Data that are not based on adequate QA/QC can be incorrect, and their misuse can lead to poor environmental management decisions. In this regard, the IAEA has a long history of organizing interlaboratory studies, which have evolved to include an ever increasing array of potential contaminants in the marine environment.

The Marine Environmental Studies Laboratory (MESL) of NAEL is actively assisting Member States with the organization of interlaboratory comparisons and the provision of reference materials.

This report summarizes the methodology and results of the IAEA-452 worldwide interlaboratory comparison exercise on the determination of trace elements in samples of scallop (*Pecten maximus*).

The IAEA officers responsible for this publication were E. Vasileva, S. Azemard and J. Oh of the IAEA Environment Laboratories.

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1. INTRODUCTION

The Marine Environmental Studies Laboratory (MESL) of the International Atomic Energy Agency'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 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 comparison exercises.

For nearly 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 methyl mercury [5–8]. This work has been conducted in collaboration with the UNEP Regional Seas Programme.

The goal of interlaboratory comparison is to demonstrate the measurement capabilities of laboratories participating in interlaboratory comparisons (ILCs) and proficiency tests (PTs). The results from ILCs or PTs are of crucial interest for laboratories as these provide clear information of its measurement capabilities. It should be pointed out that the participation is either voluntary or forced by external requirements (e.g. legal, accreditation, control bodies). NAEL's 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 test results.

Those 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. Continued membership has benefits in training and educational opportunities, enhanced mutual trust in results and methodology and objective evidence for accreditation purposes

The present ILC was designed in order to evaluate the measurement performance of participating laboratories for the analysis of trace elements in biota samples. The test material was distributed to 150 laboratories worldwide and the results from 143 laboratories in 59 countries were received by the end 2009.

The data reported by the laboratories, together with the technical and statistical evaluations of the results for each element, are included in this report.

The performance of the participant laboratories was assessed through evaluating Z-scores in accordance with ISO 13528 [9] and the International Harmonised Protocol for the Proficiency Testing of Analytical Chemistry Laboratories [10].

On the basis of the outcome from previous ILCs, organised by NAEL for the same population of laboratories, the standard deviation for proficiency assessment (also called target standard deviation) was fixed to 12.5%. Z-scores obtained from this ILC results should assist chemists to make appropriate modifications in their laboratory analytical procedures in order to improve data quality. All results were treated confidentially and each laboratory was identified with a code number for anonymity.

Further information concerning this report and the IAEA quality assurance programme can be obtained from the Marine Environmental Studies Laboratory, IAEA-NAEL, 4 Quai Antoine 1^{er}, MC 98000, Monaco or the web site: <http://www-naeb.iaea.org/naml/>.

2. SCOPE OF LABORATORY COMPARISON

In January 2009, 150 letters of invitation have been sent to laboratories that had expressed a wish to participate, or previously had participated, in an IAEA intercomparison exercise on the determination of trace elements in marine samples. Positive responses were received from 143 laboratories in 59 Member States and samples were duly dispatched to them. Each participating laboratory received one sample of the marine sediment material, designated IAEA-452, 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 16 elements: Ag, Al, As, Cd, Co, Cu, Fe, Hg (total and methyl mercury), Li, Mn, Sb, Se, Sn, Sr, V and Zn. The IAEA was also interested in receiving results for any other elements that the participating laboratories were willing to provide to enhance the characterisation of the matrix.

The deadline for returning the results was initially set at September 2009, but was later extended to the end of November 2009 due to delays of some laboratories in the reporting of results.

In total, 143 laboratories from 59 countries participated in this ILC exercise and reported results for up to 15 elements (including methyl mercury).

3. DESCRIPTION OF THE MATERIAL

A large quantity of scallop (*Pecten Maximus*) was collected in December 2007 and January 2008 by scuba diving in pertuis Breton, western France. Organisms were immediately dissected. Soft tissue (all but adductor muscle and gonads) were frozen and freeze dried for further processing and bottling. The dried material was hand grinded and sieved by MESL staff. The sieving cut-off value (250 µm) was selected to ensure that the physical properties of the material were sufficiently uniform whilst retaining sufficient material to make an adequate number of units.

Aliquots of about 8 g were packed into glass bottles with polyethylene caps and sealed in plastic bags. The homogeneity of this material for trace elements was tested using a standard protocol and found to be satisfactory for the purposes of this ILC (at or above an intake mass of 200 mg). Metal concentrations are expected to match the range normally found in the marine environment in this region.

4. HOMOGENEITY TESTS

Homogeneity was determined by MESL after the bottling of the sample material.

The within and between-bottle homogeneity was tested by the determination of the concentration of some typical elements (Cd, Cu, Fe, Hg, Mn, Pb and Zn) in sample aliquots of 0.25 g taken from 10 bottles, which were set aside at regular intervals during the whole period of bottling. From each bottle, three samples were prepared by digestion in a microwave oven using 5 ml HNO₃ and 2 ml HF. The samples were analysed by means of ETAAS. The measurements were performed under repeatability conditions and in a randomised way in order to be able to separate a potential analytical drift from a trend in the filling sequence. The determination of mercury was done in solid subsamples with solid mercury analyser. Both methods were previously validated in the inorganic chemistry laboratory of IAEA-NAEL.

Data were checked for presence of trends and outliers. Since no technical reasons were identified for the outlying results, all data were retained for statistical analysis. In the case of presence of trends and outlier averages, however the evaluation by ANOVA could not be the most appropriate and therefore an alternative approach for the estimation of homogeneity was followed. Obtained results were then evaluated in 2 different ways: 1) according to chapter 3.11.1 of the Harmonized Protocol [10] and 2) by one way analysis of variance ANOVA as recommended in ISO guide 35 [11].

The ANOVA allowed the calculation of the within S_{wb} and between-unit homogeneity S_{bb} . In the present study for several elements $MS_{between}$ was smaller than MS_{within} and S_{bb} could not be calculated. Instead U_{bb} , the heterogeneity that can be hidden by the method repeatability was calculated. Some results for within and between bottles homogeneity are presented in Table 1.

TABLE 1. WITHIN AND BETWEEN BOTTLE HOMOGENEITY FOR THE IAEA-452

Element	$U_{bb} \%$	$S_{wb} \%$
As	1.5	5.2
Cd	2.2	5.3
Cu	1.2	2.9
Fe	1.6	2.9
Hg	0.5	1.6
Mn	1.8	2.8
Zn	0.9	2.2

The variation of most of the investigated trace elements content between the ten different sample vials was not larger than the variation within the vials. The potential between-unit variation was generally below 0.5-2.5%. Usually the between-units variation was small enough compared to the method variability. The conclusion was that the homogeneity for most of analytes complied with the provisions given by the Harmonized Protocol. Hence it was concluded that the IAEA-452 test material sufficiently homogeneous for As, Cd, Cu, Fe, Mn, Hg and Zn.

However some exceptions have been noted for Ni (14%), Cr (13.4%) and Pb (14%). The high values for within bottle homogeneity were most probably a result of the presence of outliers. Ni, Cr and Pb were later on excluded from the list of elements on which is based the evaluation of the measurement performances of the laboratories participating in the IAEA-452.

5. STABILITY

The test material was monitored, using the measurement protocol applied for homogeneity study, at the beginning of the study and after receipt of the results from the participants as it is suggested in the Harmonized Protocol [10]. Statistically significant differences in the results of analysis obtained before dispatch of samples and after termination of the exercise were not found, thus indicating the stability of the test material. Test samples were kept at room temperature for the period of the study.

6. ANALYSES AND REPORTING

The participants in the IAEA-452 worldwide intercomparison exercise were requested to make at least three, preferably six, independent replicate determinations for each trace element in the sample by using the techniques routinely employed in their laboratories. They were requested to record all results in the reporting form and to provide a summary of the quality control procedures routinely employed (if any) in their laboratory and also the results of the reference materials analysed concurrently with the test material. Other information requested included the drying procedure and a short description of the analytical method used, comprising the pre-treatment or separation methods (i.e. mineralization/digestion procedure) and the way results were calculated.

All results were to be reported on a dry-weight basis and the residual moisture content of the sediment sample had to be indicated. The analyte concentrations were to be reported as net values (i.e. after blank correction *etc.*), leaving as many significant figures as justified by the precision of the method used. For each element, the participants were requested to report the average weight of the sample taken for analysis, the concentration of each independent replicate determination, the arithmetic mean, standard deviation (s_{n-1}) of the replicate determinations and detection limit of the method.

7. EVALUATION OF THE RESULTS

7.1. DATA TABLE TERM

All results reported by participating laboratories are presented in Appendix I (results are displayed as received, i.e. with all significant numbers reported). The main terms used in those tables are defined below.

Lab code: Each participant was identified by a unique code number known only by the respective laboratory and the organisers of the comparison.

Mean value: Arithmetic mean (\bar{x}) computed for each element from all individual results supplied by laboratory. Results given as below the detection limit (DL) are presented with the symbol < in the cases where the detection limit was reported. Results reported less than DL without giving DL or reported as Not Detected are not included in the tables.

Standard deviation: The standard deviation of the mean value reported by laboratory

Z-score: performance indicator

7.2. STATISTICAL EVALUATION OF THE RESULTS

7.2.1. Assigned value

In this ILC, the overall mean concentration assigned for each element was calculated using the IUPAC protocol for proficiency tests [10] and ISO standard 13528 [9]. Assigned values for trace elements in the IAEA-452 sample were established from the median of the participants' results, as suggested by the Harmonized Protocol. These values were compared to other robust estimates of the mean, which were calculated with an algorithm proposed by the Analytical Methods Committee of the Royal Society of Chemistry (AMC) [12].

This is rather a straight forward method and does not significantly differ from other methods, such as robust mean and other robust estimates of the mean, which were calculated with an algorithm proposed by the AMC.

The standard deviation for the proficiency assessment (also called target standard deviation), σ_p , was set to be fit for purpose, according to the Harmonised Protocol [10] and was fixed to 12.5% of the assigned values. The determination of target standard deviation was done on the basis of the outcome of previous ILCs organised by the MESL for the same population of laboratory. The appropriateness of this level of tolerated 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.

The standard uncertainties of the assigned values were determined in accordance with the Harmonized Protocol [10]. They correspond to the standard error of the consensus value, which is given by equation 3.1:

$$u = \frac{\hat{\sigma}}{\sqrt{n}} \quad (\text{Equation 3.1})$$

Where

$\hat{\sigma}$ is the robust standard deviation (obtained by AMC algorithm) [12];

n is number of results.

The relative expanded uncertainties for the assigned values of investigated elements were in the range of 8.5%.

7.2.2. Performance indicator and target standard deviation

The performance of an individual laboratory i was expressed by the z_i -score, which was calculated according to equation 3.2:

$$z_i = \frac{x_i - \hat{X}}{\sigma_p} \quad (\text{Equation 3.2})$$

Where

z_i is Z-score of laboratory i for the respective sample: Reported result of laboratory i for that sample, expressed as the mean of multiple determinations;

\hat{X} : Assigned value for the respective sample;

σ_p : Standard deviation for proficiency assessment.

The laboratory performance was evaluated using z-scores in accordance with ISO 13528 [9].

The acceptability of a laboratory's performance was evaluated according to the following generally accepted limits [10]:

	$ z \leq 2.0$	satisfactory
2.0 <	$ z < 3.0$	questionable
	$ z \geq 3.0$	unsatisfactory

Assigned values and the standard deviations for proficiency assessment for all measurants are presented in Table 2.

TABLE 2. ASSIGNED VALUES FOR THE IAEA-452 INTERCOMPARISON EXERCISE BIOTA SAMPLE

Analyte	Unit	Assigned value	Assigned std. dev.	N
Ag	mg kg ⁻¹	11.8	1.5	9
As	mg kg ⁻¹	17.5	2.2	76
Br	mg kg ⁻¹	500	62	9
Ca	mg kg ⁻¹	11300	1400	12
Cd	mg kg ⁻¹	29.6	3.7	121
Co	mg kg ⁻¹	1.62	0.20	65
Cu	mg kg ⁻¹	10.8	1.3	110
Fe	mg kg ⁻¹	1020	130	92
Hg	mg kg ⁻¹	0.15	0.02	80
K	mg kg ⁻¹	13140	1640	13
Li	mg kg ⁻¹	2.01	0.25	23
MeHg*	mg kg ⁻¹	0.0217	0.0027	20
Mg	mg kg ⁻¹	6380	800	22
Mn	mg kg ⁻¹	273	34	86
Na	mg kg ⁻¹	43960	5990	11
Rb	mg kg ⁻¹	7.85	0.98	9
Sb	mg kg ⁻¹	0.100	0.013	23
Se	mg kg ⁻¹	6.55	0.82	54
Sr	mg kg ⁻¹	82.9	10.3	42
V	mg kg ⁻¹	6.36	0.79	48
Zn	mg kg ⁻¹	166	21	115

* reported as Hg

8. RESULTS AND DISCUSSION

8.1. OVERVIEW OF THE RESULTS

One hundred and thirty nine laboratories provided results for the analysis of the IAEA-452 by the final deadline for the exercise. 143 sets of data were submitted (some laboratories reported data generated by multiple techniques) comprising 1131 analytical results for the determination of trace elements in the IAEA-452 for a suite of 20 elements and methyl mercury (Appendix I). Upon receipt of the datasets, the results were subject to technical evaluation.

Of the 143 participating laboratories, the number of results for the suite of analytes designated as being of greatest interest to the study was as follows: Ag (9), As (76), Br (9), Ca (12), Cd (121), Co (65), Cu (110), Fe (92), Hg (80), K (13), Li (23), Mg (22), Mn (86), Na (11) Rb (9), Sb (23) Se (54), Sr (42), V (48), Zn (115) and methyl mercury (20).

In addition to the compiled data sets given in Appendix I, graphical presentations of results sorted by element are presented in Appendix II. The datasets accepted on the technical grounds were tested for outliers using Dixon and Grubbs. The distribution of the results was checked also by kernel density estimations. Three sets of figures are provided for all investigated trace elements and MeHg in Appendix II. Each set includes a) the Kernel Density plot, b) Z-score for the reported results c) summary of the statistical evaluation of results for the respective element including assigned value and target standard deviation.

8.1.1. Moisture content

Participants were requested to evaluate the moisture content of the test material on a separate portion (not used for analysis) taken at same time as the sub-samples to be used for analysis. Results were to be reported to the IAEA after correction for the moisture content of the material.

IAEA-452 was subjected to freeze drying as part of its preparation. At the time of bottling, the moisture content of the material was below 1%. However, the material might absorb moisture from the atmosphere dependent upon local storage conditions and humidity levels. Consequently, users are advised to make a separate determination of the moisture content of the material whenever it is used for quality control purposes.

8.1.2. Analytical methods

In common with all open interlaboratory studies, a wide range of analytical methodologies and finishes was used to provide data for the determination of trace elements in marine biota IAEA-452. Broadly, this can be broken into three approximately equal groups, in this case: non-destructive techniques (XRF and NAA); plasma spectrometric methods (ICP-MS and ICP-AES) and atomic absorption methods, see Figure 1. The abbreviations of instrumental techniques used in this exercise are shown in Table 3

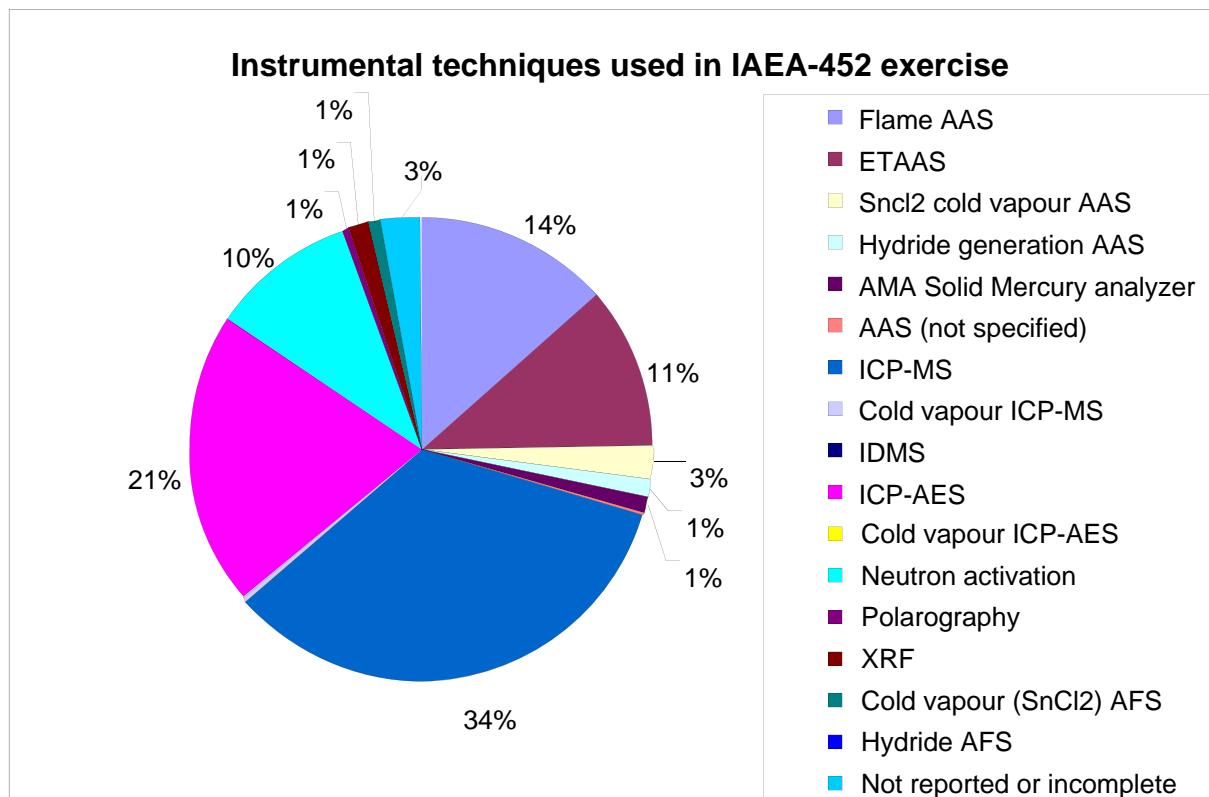


FIG. 1. Proportional distribution of analytical techniques used to analyse the IAEA-452.

TABLE 3. INSTRUMENTAL TECHNIQUES

Method code	Instrumental technique
AAS	Atomic Absorption Spectrometry
ETAAS	Electro Thermal Atomic Absorption Spectrometry
ICP-MS	Inductively Coupled Plasma Mass Spectrometry
IDMS	Isotope Dilution ICP-MS
ICP-AES	Inductively Coupled Plasma Optical Emission Spectrometry
AFS	Atomic Fluorescence Spectrometry
XRF	X-ray Fluorescence Spectrometry

8.1.3. Laboratory performances

Table 4 summarizes the overall performance of the participating laboratories in the IAEA-452 ILC by elements; the proportion of Z-scores falling in each category is given together with the percentage of participation of the laboratory in the determination of the respective element in the IAEA-452 sample.

TABLE 4. SUMMARY OF THE LABORATORY PERFORMANCES BY ELEMENTS

Element	Participation	$ Z \geq 3$	$2 < Z < 3$	$ Z \leq 2$
Ag	6%	11%	11%	78%
As	53%	13%	7%	80%
Br	6%	22%	0%	78%
Ca	8%	8%	17%	75%
Cd	85%	13%	5%	82%
Co	45%	11%	14%	75%
Cu	77%	9%	10%	81%
Fe	64%	5%	5%	89%
Hg	56%	18%	5%	78%
K	9%	15%	15%	69%
Li	16%	30%	26%	43%
Me Hg	14%	15%	10%	75%
Mg	15%	18%	9%	73%
Mn	60%	8%	6%	86%
Na	8%	9%	9%	82%
Rb	6%	22%	0%	78%
Sb	16%	17%	13%	70%
Se	38%	22%	11%	67%
Sr	29%	7%	10%	83%
V	34%	8%	13%	79%
Zn	80%	4%	6%	90%

Tables 5 and 6 give summaries on the performance of laboratories based on the obtained Z-scores. Detailed information reported by participants for all requested elements plus laboratory scoring provided by the organiser of the IAEA-452 ILC is summarized in Appendix I.

TABLE 5. SUMMARY OF THE LABORATORY PERFORMANCES BASED ON Z-SCORE

Lab code	N°of Z-Score	N°of $ Z \geq 3$	N°of $2 < Z < 3$	N°of $ Z \leq 2$
1	13	1	1	11
2	12	2	0	10
3	10	0	0	10
4	5	0	1	4
5	9	3	1	5
6	15	2	1	12
7	10	1	3	6
8	7	0	0	7
9	15	1	0	14
10	10	0	0	10
11	14	3	0	11
12	9	0	0	9
12	6	0	0	6
13	13	1	0	12
14	8	0	3	5
15	13	2	5	6
16	5	0	0	5
17	12	0	1	11
18	7	0	0	7
19	8	0	1	7
20	7	0	2	5
21	10	0	0	10
22	13	0	1	12
23	1	0	0	1
24	2	0	0	2
25	5	0	1	4
26	3	0	0	3
27	11	0	0	11
28	13	0	1	12
29	5	0	1	4
30	5	0	0	5
31	12	0	2	10
32	19	3	1	15
33	2	2	0	0

TABLE 5. SUMMARY OF THE LABORATORY PERFORMANCES BASED ON Z-SCORE (cont.)

Lab code	N°of Z-Score	N°of $ Z \geq 3$	N°of $2 < Z < 3$	N°of $ Z \leq 2$
34	15	0	1	14
34	1	0	0	1
35	3	0	0	3
36	6	0	0	6
37	10	0	0	10
38	5	0	0	5
39	10	0	0	10
40	14	0	0	14
41	13	3	4	6
42	6	1	2	3
43	16	1	0	15
44	3	0	0	3
45	3	0	0	3
46	4	0	0	4
47	9	3	0	6
48	12	2	0	10
49	13	7	1	5
50	4	1	0	3
51	5	0	0	5
52	10	1	0	9
53	3	0	0	3
54	14	0	1	13
55	3	2	0	1
55	1	0	0	1
56	2	0	2	0
57	6	0	0	6
58	9	0	1	8
59	8	0	0	8
60	13	0	0	13
61	2	1	0	1
62	6	2	0	4
63	5	0	0	5
64	5	1	0	4

TABLE 5. SUMMARY OF THE LABORATORY PERFORMANCES BASED ON Z-SCORE (cont.)

Lab code	N°of Z-Score	N°of $ Z \geq 3$	N°of $2 < Z < 3$	N°of $ Z \leq 2$
65	3	0	0	3
66	11	3	0	8
67	4	0	0	4
68	6	1	0	5
69	2	0	0	2
70	2	0	0	2
71	3	0	1	2
72	9	0	0	9
73	3	1	0	2
74	7	0	0	7
75	5	2	0	3
76	5	1	0	4
77	9	4	4	1
78	3	0	0	3
79	4	0	0	4
80	6	0	0	6
81	7	0	0	7
82	17	0	2	15
83	7	0	2	5
84	3	0	0	3
85	2	1	0	1
86	13	2	0	11
87	4	1	0	3
88	8	0	0	8
89	3	0	0	3
90	2	0	0	2
91	8	1	4	3
92	5	0	0	5
93	4	3	1	0
94	10	0	0	10
95	5	0	2	3
96	8	1	2	5
97	7	1	1	5
98	13	0	0	13

TABLE 5. SUMMARY OF THE LABORATORY PERFORMANCES BASED ON Z-SCORE (cont.)

Lab code	N°of Z-Score	N°of $ Z \geq 3$	N°of $2 < Z < 3$	N°of $ Z \leq 2$
99	8	0	0	8
100	13	3	5	5
101	2	0	0	2
102	12	0	1	11
103	7	0	1	6
104	6	0	1	5
105	9	0	1	8
106	9	2	3	4
107	9	0	1	8
108	11	1	0	10
109	1	0	0	1
110	3	1	0	2
111	3	1	0	2
112	8	3	0	5
113	6	0	1	5
114	4	0	2	2
115	4	0	0	4
116	4	0	0	4
117	8	1	0	7
118	9	4	2	3
119	8	0	2	6
120	3	0	0	3
121	5	0	0	5
122	13	5	2	6
123	11	0	0	11
124	3	3	0	0
125	5	0	0	5
126	1	0	0	1
127	5	0	0	5
128	7	5	0	2
129	7	6	0	1
130	3	0	1	2

TABLE 5. SUMMARY OF THE LABORATORY PERFORMANCES BASED ON Z-SCORE (cont.)

Lab code	N°of Z-Score	N°of $ Z \geq 3$	N°of $2 < Z < 3$	N°of $ Z \leq 2$
131	5	0	1	4
132	1	1	0	0
133	9	3	0	6
134	2	0	0	2
135	8	8	0	0
136	12	0	0	12
137	14	5	3	6
138	15	0	0	15
139	3	0	1	2

TABLE 6. OVERALL ASSESSMENT OF LABORATORIES PERFORMANCE BY ELEMENTS

Lab code	Ag	As	Br	Ca	Cd	Co	Cu	Fe	Hg	K	Li	Mg	Mn	Na	Rb	Sb	Se	Sr	V	Zn
1	-0.42			0.32	0.00	-7.24	0.46	-1.07		-0.06	0.26		-0.80	2.15	-0.01	0.15	-0.29			
2	0.77			-0.46	-0.25	0.50	0.56		-3.82	0.09	-0.38		3.04	-0.93	-0.23	0.17				
3	-0.69			-0.46	-0.44	-0.61	0.27			1.09			-0.33	0.85	0.67	1.35				
4	-2.53			-1.36		-1.32	-1.44											-1.64		
5	-7.57			-0.52	0.67	0.73	9.71			1.13			-3.09		2.36	-0.06				
6	-0.02			-0.29	0.40	-0.19	-3.98	-0.59	0.00	-1.38	-3.44	0.42	-0.24	-0.37	-0.71	-0.33	-2.75			
7	-1.96			-4.99	2.81	0.28	-1.94	0.43		0.46	-2.16		2.92		0.24					
8	0.24			0.24	-0.20	-0.90	0.37									0.20		-0.47		
9	0.17			0.16	0.25	0.13	-0.60	0.32	-5.42	-0.53	-0.01	0.04	-0.08	0.04	0.24	0.01	-0.05			
10	-0.74			0.11	-0.84	-1.49	-1.50				0.50		1.60	0.67	-0.45	0.19				
11	1.04			0.73	1.04	-0.09	-0.23	1.12	4.78		-0.28	0.06	3.20	-0.01	0.44	3.46	0.24			
12	-0.92			-0.54	1.11	-0.31	-0.02	0.11			-0.84		1.24				-0.67			
12	-0.01			-0.51	0.57	0.50							-0.45				-0.87			
13	-0.31			-0.06	0.10	-0.64	-0.23			-1.99		-1.03	0.25	1.60	12.56	0.77	1.23	-1.74		
14	2.50	-1.07	-2.06				1.39	0.00			0.71			-0.18		2.31				
15	-1.74		-2.00	-1.43	2.06	-2.58	-6.56		-3.86	-2.99	-1.17		-0.75	-2.17	-2.49	0.19				
16	0.00		0.76		0.36	0.00											0.34			
17	-1.29		-1.04	-1.68	-1.38	-1.08	-1.71		-2.91		-1.43		-1.29	-1.69	-0.92	-0.49				
18	1.45		0.00		1.46	-0.40					0.30			0.48		0.05				
19	1.27		1.38		0.95	1.17	0.53				0.88		2.63		0.22					
20			-1.11	-2.98	-1.72	-0.90					-2.21				-0.39	-1.58				

TABLE 6. OVERALL ASSESSMENT OF LABORATORIES PERFORMANCE BY ELEMENTS (cont.)

Lab code	Ag	As	Br	Ca	Cd	Co	Cu	Fe	Hg	K	Li	MeHg	Mg	Mn	Na	Rb	Sb	Se	Sr	V	Zn
21	0.07			0.76	0.57	0.39	0.41					0.73					-0.38	0.26	0.97	0.42	
22	0.22			-0.70	0.35	0.13	0.08	-0.37		-0.32		0.77					-2.48	0.43	0.49	0.55	-1.20
23										-0.59											
24										-0.37											
25											-0.64										
26	-0.15			0.65							0.91						-0.67				0.08
27	0.40			-0.22	-0.54	-1.09	0.02	0.64				0.09					-1.28		-1.34	0.16	-0.32
28	-1.15			-0.97	-0.59	0.13	0.24	-1.55		-0.04		-0.85	-0.64				0.18	-2.02	0.55	-1.06	
29	0.14			0.08		-0.51		-2.11													-0.37
30	0.14			0.62		-0.01		1.17													0.10
31	0.88	0.58	0.00		-0.43	2.37	-1.27	0.04									2.19	0.36		1.40	
32	-1.90	-0.24	1.30	-3.05	-0.16	0.35	0.65	0.55	195.2	-0.39	2.55		-1.73	0.27		6.27	0.24	-1.03	-0.09	1.02	-0.48
32	0.04			0.32		2.35	0.54									0.53					
33						-7.14		-3.42													
34	0.68	0.26	0.11	0.48	1.30	0.44		0.79	-0.21	0.21						0.35	0.02	2.48	0.35	0.16	0.39
34									-0.05												
35	0.14			1.08						-0.69											
36											0.16						-0.36				0.22
37	0.35			0.40		0.04															
37				0.29		0.89	0.60	-0.53				0.75	0.39								
38						-0.15		0.69				0.43	0.50								
39	-0.15			0.32	-0.89	-0.90	0.44					-0.08									
40	0.40			0.22	-0.49	-0.68	0.50	1.17				0.61	0.01	-0.23			-0.28	0.64	-0.42	0.73	-0.53

TABLE 6. OVERALL ASSESSMENT OF LABORATORIES PERFORMANCE BY ELEMENTS (cont.)

Lab code	Ag	As	Br	Ca	Cd	Co	Cu	Fe	Hg	K	Li	MeHg	Mg	Mn	Na	Rb	Sb	Se	Sr	V	Zn
41		-3.63		-0.16	-2.12	-3.09	-1.31	1.44		-2.35		-2.22			-0.26	-4.81	-1.05	-0.57	-2.94		
42		73.5		1.15	1.93				-2.88						-0.64	-2.26					
43		0.22		0.30	-0.20	-0.88	-0.02	0.16		0.96	-1.16	0.39	0.50		-0.77	-0.16	3.25	0.04	-0.59	0.39	
44				0.98	0.55															0.76	
45				0.68	0.80															0.53	
46				0.53	0.83	-0.74								0.56							
47		-7.57		-0.17	4.05	-0.72	-0.42							3.81	0.15	0.64				-0.22	
48		-7.38		0.57	0.54	-0.01	0.83	1.44		1.00			0.50				-3.87	-0.66	1.04	0.43	
49		4.79	-3.02	1.60	-1.78		-1.81		-2.48					61.7	5.01	-1.19	3.19			-1.30	
50				0.35		11.22	0.05													1.27	
51				-0.30		-0.69			-0.53					-0.01						-0.86	
52				1.68	1.20	0.01	-0.17	-5.55	0.40					0.22	-1.46	0.00				0.77	
53				-0.06		-0.86														0.05	
54	0.00	0.08	0.26	-0.07	1.57	0.00		0.31		1.56				0.08	0.00	2.80	0.50	0.79	0.10		
55				-4.32		1.00			-4.19												
56						-0.23															
57		0.04			0.19		0.21	-0.49	0.59											0.00	
58		-0.69			-1.35	0.89	0.65	0.67						-0.38		-2.14	0.81	-0.14			
59		0.95			0.35	-0.84	-1.27	-0.16						0.04			-1.04	-0.43			
60		-1.27			-0.86	0.06	1.37	0.63	-0.96	0.33				0.32	0.47		-0.37	-0.57	0.30	0.20	
61										-0.53						-6.46					

TABLE 6. OVERALL ASSESSMENT OF LABORATORIES PERFORMANCE BY ELEMENTS (cont.)

Lab code	Ag	As	Br	Ca	Cd	Co	Cu	Fe	Hg	K	Li	MeHg	Mg	Mn	Na	Rb	Sb	Se	Sr	V	Zn
62					4.41		0.38	0.20	-3.17				0.11							0.34	
63					0.00		1.09	0.67				0.56								0.24	
64			-7.20		0.08		0.20													-0.74	
65		0.68			1.78															1.54	
66		3.42			-0.43	-1.58	1.61	-1.03	8.00			0.56								-0.43	
67					-1.62		-1.16					-0.20									-1.16
68					-1.54		-0.38	-1.63	100.2			0.03								-1.07	
69							0.59					0.06									
70							0.80					0.46									
71					-0.80			-2.05												0.67	
72		0.17			0.11	-1.04	-1.13	0.09	0.48			0.01								-0.67	
73					-3.14		-0.16													0.53	
74		-0.88			0.27		0.22	1.16	0.32			0.18								0.00	
75					4.94		-0.81	-0.81					-6.74							1.93	
76					-0.97		-1.49	-0.80					-6.84							-0.07	
77		4.03			6.38	4.84	2.71	1.31	2.13				-7.31							2.30	
78							0.64	0.64												0.50	
79					0.46		-0.38		0.21											0.43	
80					0.77	0.20	0.13	0.42					-0.11							0.44	
81		-0.33			0.08		-0.62	-0.18	0.91				-0.14							-1.11	
82	-2.08	-1.61			-0.36	-1.49	-1.78	-1.04	-1.23	0.00	-1.18	-2.35	-0.71	-0.84	-0.78	-1.03	-0.87	-1.81	-1.11		
83		-0.87			1.34	-2.21	2.23	0.07					0.62							0.50	

TABLE 6. OVERALL ASSESSMENT OF LABORATORIES PERFORMANCE BY ELEMENTS (cont.)

Lab code	Ag	As	Br	Ca	Cd	Co	Cu	Fe	Hg	K	Li	MeHg	Mg	Mn	Na	Rb	Sb	Se	Sr	V	Zn
84				0.54	0.21			0.64													
85		-7.46			-0.32	0.64	-0.29	-0.29	-0.11												
86			-6.49			0.54	-1.79				4.02										
87				0.59	-0.20	1.39	-0.79					1.47									
88		-0.33			-1.17							0.04									
89		-0.60																			
90						-0.32						0.13									
91			-0.34	-2.42		-0.38	-2.50	4.27					-2.95								
92			-0.17	-0.69	-0.30		0.81														
93			2.32		105.8				6.40											11.8	
94		0.90		-0.22	1.73		0.80	0.62	0.00	0.28											
95						0.68	-2.86	0.00													
96			-1.43	0.07		8.90	-2.09		-2.26												
97				0.38		0.13	-3.16														
98		-1.65		0.18	0.10	0.07	1.08			1.79											
99		-1.18		-0.58	-0.10	-1.04		-0.43													
100		-1.97	-2.16	-2.86	-0.98	-1.65	162.1		-4.06												
101						-0.21						0.17									
102	0.04		0.24	0.44	0.06	1.11			2.15				0.33								
103		2.37		0.60		-0.16	-0.81	0.37					0.09								
104				0.78			0.72		0.01		2.56			0.30							
105		-0.16		0.39	0.35	-0.07	1.58	-2.29													
																		-0.44	0.78	-0.10	

TABLE 6. OVERALL ASSESSMENT OF LABORATORIES PERFORMANCE BY ELEMENTS (cont.)

TABLE 6. OVERALL ASSESSMENT OF LABORATORIES PERFORMANCE BY ELEMENTS (cont.)

Lab code	Ag	As	Br	Ca	Cd	Co	Cu	Fe	Hg	K	Li	MeHg	Mg	Mn	Na	Rb	Sb	Se	Sr	V	Zn
128				-4.74				-5.29	-1.20		0.08			-7.08				-3.33	-7.97		
129					-3.80		591.5	-5.76			9.18		11.0					-0.24	-5.90		
130				-0.35															0.10		
131				2.35																	
132				2.35				-0.62		1.60											
133					-6.22																
134						-7.14	-6.34	-1.26	-1.10	0.27				-6.21							
135				-4.75		-5.84															
136						-1.01	0.64	0.42	-0.53					-1.35	-0.43			16.6		-3.88	
137						0.08												1.60	-0.22	0.30	-0.01
138						0.23	-0.11	18.37	2.30	-3.13	12.27		-2.79		-0.52	-0.90		28.8	3.91	1.15	2.63
139						0.19	0.12	0.06	-0.36	-0.21	-0.27		0.06	1.93	0.28	1.09		-0.72	1.16	0.34	0.27
																	2.15			0.65	

It should be noted that the results are considered as acceptable if $|Z| \leq 2$. Z-scores in the questionable range ($2 < |Z| < 3$) are technically acceptable but should be considered as a warning signal. Participants are encouraged to check their analytical processes, if all results reported are questionable or if they received questionable Z-scores for two exercises in a raw for the same element.

A total of 1027 Z-scores were calculated. 80% of data received were with Z-scores ≤ 2 and 84.8% of data received with Z-scores < 3 . For 11.9% of the data Z score ≥ 3 was calculated. This represents 139 dataset reported, in which 56 laboratories were able to produce datasets with all Z-score ≤ 2 , and 65 laboratories with all Z-score < 3 . If compared with two previous exercises (e.g. IAEA-405 [13] and IAEA-433 [14]), there is a noticeable improvement. The portion of dataset reporting 100% of data with Z-score < 3 in the previous exercise IAEA-405 and IAEA-433 was respectively 35% and 39%.

Also the proportion of laboratories that reported less than 50% of acceptable data represents only 5% (7 laboratories) compared to about 8% in the two previous exercises run on sediment samples [13, 14].

It should be noted that even if this is encouraging, part of these results could be also explained by the fact that the set of participants is not the same for the three exercises. Only a portion of participants is regularly participating, which makes the interpretation of the evolution of performance over time difficult.

It appears that more than 80% of laboratories measuring As, Cd, Cu, Fe, Mn, Na, Sr and Zn received Z-score ≤ 2 . This result shows that those elements are easily analysed. On the other hand Hg, Mg and Se have more than 15% of reported data outside of the accepted range (i.e. Z-score ≥ 3), probably reflecting unresolved analytical problems.

High-biased results could originate from contamination during either sample preparation (e.g. digestion step) or analysis. The laboratories concerned should carefully check analytical procedures (e.g. quality of purified water and reagents) and try to improve the cleanliness of the working environment. For example, dust is the most common atmospheric source of contaminants for trace elements in laboratories. Laboratories should also develop an effective scheme for cleaning laboratory-labware that generally includes a soap wash, an acid wash and thorough rinsing with purified water free from trace elements.

Out of the 139 sets of data received, 31 did not include results for QC as requested in the report form. 33% (455) from the total number of results (1379) and 60% from the total number of results with $Z \geq 3$ are obtained without applying QC steps. Unfortunately, even if the results obtained without QC turned out to be accurate, they are invalid in the absence of quality control data.

In order to validate their results, all laboratories should at least systematically realise the following QC steps: 1) analyse procedural blank to control the possible sources of contamination; 2) analyse standard reference materials (i.e. RMs) with a similar matrix and approximately the same concentration level as in the samples being analysed. This should be done for each series of analysis (i.e. batch of digested samples), and these quality control samples should be analysed at regular intervals during the measurements.

Detailed overview on the importance of the use of QC step for the quality of obtained results is presented in Table 7.

TABLE 7. Z-SCORES AND QUALITY CONTROL DATA FOR THE IAEA-452 COMPARISON

Element	Total number results	IAEA-452			Number of QC data	QC Data		
		Z ≥ 3	2 < Z < 3	Z ≤ 2		Z ≥ 3	2 < Z < 3	Z ≤ 2
Ag	9	1	1	7	1	0	0	1
As	76	10	5	61	55	2	1	52
Br	9	2	0	7	5	0	0	5
Ca	12	1	2	9	8	1	0	7
Cd	121	16	6	99	77	3	6	68
Co	65	7	9	49	34	1	2	31
Cs	7	0	0	7	1	0	0	1
Cu	110	10	11	89	77	2	0	75
Fe	92	5	5	82	60	2	3	55
Hg	80	14	4	62	60	2	1	57
K	13	2	2	9	7	0	0	7
Li	23	7	6	10	3	0	1	2
MeHg	20	3	2	15	19	5	0	14
Mg	22	4	2	16	10	0	1	9
Mn	86	7	5	74	52	2	0	50
Na	11	1	1	9	6	0	0	6
Rb	9	2	0	7	6	0	0	6
Sb	23	4	3	16	5	2	1	2
Se	54	12	6	36	36	3	1	32
Sr	42	3	4	35	21	0	0	21
V	48	4	6	38	17	0	0	17
Zn	115	5	7	103	85	1	0	84

It is satisfying to notice that major part of the participating laboratories have used as QC sample the reference materials from the IAEA-407, IAEA-436, IAEA-140. Reference materials from other producers such as EC JRC-IRMM, Belgium; NRCC, Canada; NIST USA are also used in the laboratory practice of participating in the IAEA-452 worldwide ILC and later on in the certification exercise organised with the same sample material [15]. An important principle for the selection of reference material by laboratories is the principle for matrix and analyte matching and when is possible the concentration range as well.

Erroneous calibration standards may be another source of bias. For instance, it is important to note that losses can occur in low-concentration working standard solutions, which would result in overestimates of the concentrations of elements in the samples (e.g. standard solutions should not be stored for an extended period of time). Only standards (CRM) with stated SI traceability should be used for calibration purposes.

Laboratories with poor results should carefully check all laboratory procedures, equipment and instruments.

8.1.4. Laboratory performance for methyl mercury

Twenty results were reported for MeHg. The summary of the methods used for sample preparation for MeHg given in Table 8, reveals differences within the methodologies used. The distribution of the data could not be explained solely on the basis of the method, at least in the absence of very detailed description of the entire analytical procedures. It is possible that minor changes in the protocol might influence the final results.

TABLE 8. ANALYTICAL METHODS USED FOR METHYL MERCURY DETERMINATIONS

Lab code	Sample prep	Detection ^a	Z score
1	KOH/MeOH, aqueous phase ethylation, purge onto Tenax trap	GC-AFS	0.64
6	KOH/MeOH, aqueous phase ethylation, purge onto Tenax trap	GC-AFS	-0.8
7	Alkaline digestion, extraction-derivatisation in pentane with NaBEt ₄ after addition of propylmercury as internal standard	GC-ICPMS	1.2
9	KOH/MeOH, aqueous phase ethylation, purge onto Tenax trap	GC-AFS	0.12

TABLE 8. ANALYTICAL METHODS USED FOR METHYL MERCURY DETERMINATIONS (cont.)

Lab code	Sample prep	Detection ^a	Z-score
24	Extraction Toluene, back-extraction cysteine, mineralisation HNO ₃	Cold Vapour-AFS	0
37	KOH/MeOH	HPLC-AFS	1.52
40	KOH/MeOH, aqueous phase ethylation, purge onto Tenax trap	GC-AFS	1.36
43	Distillation, BrCl oxidation	Cold Vapour – AAS	-0.56
61	KOH/MeOH, aqueous phase ethylation, purge onto Tenax trap (use of 3mg of sample)	GC-AFS	-6.32
69	KOH/MeOH, aqueous phase ethylation, purge onto Tenax trap	GC-AFS	0.76
70	KOH/MeOH, aqueous phase ethylation, purge onto Tenax trap	GC-AFS	1.2
85	Alkaline digestion, extraction CH ₂ Cl ₂ , cleanup Thiosulfide, back-extraction CH ₂ Cl ₂	GC-AFS	-3.08
90	Distillation, aqueous phase ethylation, purge and trap	GC-ID-ICPMS	0.84
101	TMAH open microwave, extraction-derivatisation in isoctane with NaB ₄ E ₄	GC-ID-ICPMS	0.88
107	H ₂ SO ₄ /CuSO ₄ /KBr leaching followed by Toluene extraction, Grignard	GC-AED	-1.76
118	Leaching 10N HCl (report inorganic Hg)	CVAAS	72.4
119	Organic Hg (no precision)	Solid AAS	3.4
123	H ₂ SO ₄ /CuSO ₄ /KBr leaching followed by Toluene extraction, back-extraction in aqueous thiosulfate solution	Solid AAS	0.64

TABLE 8. ANALYTICAL METHODS USED FOR METHYL MERCURY DETERMINATIONS
(cont.)

Lab code	Sample prep	Detection ^a	Z-score
134	Dithizone-toluene extraction	GC-ECD	0.32
138	KOH/MeOH, aqueous phase ethylation, purge onto Tenax trap	GC-AFS	2.8

^a GC: Gas Chromatography; AFS: Atomic Fluorescence Spectrometry; AED: Atomic Emission Detector, ECD: Electron Capture Detector; ICP-MS: Induced Coupled Plasma Mass Spectrometry, ID-ICP-MS: Isotope Dilution Induced Coupled Plasma Mass Spectrometry, AAS: Atomic Absorption Spectrometry, HPLC: High Pressure Liquid Chromatography.

9. RECOMMENDATIONS

Participants are recommended to review their data element-by-element, appraising whether the Z-score is less than or equal to 2. The use of the Z-scores 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 their QA/QC procedures. Interlaboratory studies 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.

Laboratories are encouraged to refer to IAEA reference methods in order to improve their analytical methods and their QC procedures. These methods, listed on the website: <http://www-naweb.iaea.org/naml/aqcsmethods.asp> are available free of charge from IAEA-NAEL in Monaco. 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>

10. CONCLUSIONS

The current intercomparison exercise for the determination of trace elements in biota sample attracted a large number of international participants. Although the overall performance of the laboratory is quite satisfactory for trace elements and MeHg, it must be pointed that a number of laboratories have a problem with the proper use of reference material and internal quality control. The material used in the IAEA-452 ILC was further certified for the amount content of 15 trace elements and MeHg. Participants in the intercomparison could retain their samples

and use them as a reference material in their laboratory practice. The reference material is suitable for the validation of analytical procedure when biota samples need to be analysed for trace elements.

The IAEA-452 reference material is now available from the International Atomic Energy Agency (IAEA), AQCS, PO Box 100, A-1400 Vienna, Austria.

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APPENDIX I

DATA REPORT OF RESULTS SORTED BY ELEMENTS

TABLE I.1. SILVER IN THE IAEA-452 SAMPLE: INFORMATION REPORTED BY PARTICIPANTS PLUS LABORATORY SCORING PROVIDED BY THE ORGANISER
 Ag - assigned value: 11.8 mg kg^{-1}

Lab code	Mean value	Standard deviation	Z-score	Instrumental technique
30	12.0	0.2	0.14	ICP-MS
31	13.1	0.74	0.88	Neutron activation
32	9		-1.90	XRF
34	12.8	0.1	0.68	Neutron activation
54	11.8	0.15	0.00	Neutron activation
60	9.93	0.30	-1.27	Neutron activation
82	8.73	0.53	-2.08	ICP-MS
113	12.8	0.39	0.68	ETAAS
135	4.80	0.85	-4.75	Neutron activation

TABLE I.2. ARSENIC IN THE IAEA-452 SAMPLE: INFORMATION REPORTED BY PARTICIPANTS PLUS LABORATORY SCORING PROVIDED BY THE ORGANISER
 As - Assigned value: 17.52 mg kg⁻¹

Lab code	Mean value	Standard deviation	Z-score	Instrumental technique
1	16.6	1.19	-0.42	ICP-MS
2	19.2	1.49	0.77	ICP-AES
3	16.0	0.3	-0.69	ICP-MS
4	11.98	0.25	-2.53	ICP-AES
5	0.942	0.074	-7.57	Hydride generation AAS
6	17.48	0.28	-0.02	ICP-MS
7	13.22	1.29	-1.96	ICP-MS
8	18.04	0.21	0.24	ICP-MS
9	17.9	0.4	0.17	ICP-MS
10	15.9	0.5	-0.74	ICP-MS
11	19.8	0.944	1.04	ICP-MS
12	15.5	0.61	-0.92	ICP-MS
12	17.5	0.6	-0.01	ICP-MS
13	16.85	0.47	-0.31	ICP-MS
14	23	5	2.50	XRF
15	13.7	1.08	-1.74	ICP-AES
16	17.53	1.05	0.00	ETAAS
17	14.69	0.36	-1.29	ICP-AES
18	20.7	0.98	1.45	ICP-AES
19	20.3	1.51	1.27	ICP-MS
21	17.68	0.14	0.07	ICP-MS
22	18	0.5	0.22	ICP-MS
26	17.2	0.505	-0.15	ICP-MS
27	18.4	0.30	0.40	ICP-MS
28	15		-1.15	ICP-AES
29	17.828	0.47	0.14	ICP-MS
31	18.8	1.5	0.58	Neutron activation
32	17		-0.24	XRF
32	17.6	0.6	0.04	ICP-MS
34	18.1	0.2	0.26	Neutron activation
35	17.82	0.12	0.14	ETAAS
37	18.29	0.57	0.35	ICP-MS
39	17.2	0.4	-0.15	ICP-AES

TABLE I.2. ARSENIC IN THE IAEA-452 SAMPLE: INFORMATION REPORTED BY PARTICIPANTS PLUS LABORATORY SCORING PROVIDED BY THE ORGANISER (cont.)

Lab code	Mean value	Standard deviation	Z-score	Instrumental technique
40	18.4	0.221	0.40	ICP-MS
41	9.57	0.31	-3.63	ICP-MS
42	178.6	2.2	73.55	ICP-MS
43	18.0	0.2	0.22	ICP-MS
47	0.94	0.05	-7.57	Hydride generation AAS
48	1.35	0.163	-7.38	ICP-AES
49	28	5	4.79	Neutron activation
54	17.7	0.46	0.08	Neutron activation
57	17.6	1.6	0.04	Hydride generation AAS
58	16	2.0	-0.69	Neutron activation
59	19.6	0.5	0.95	ETAAS
64	1.75	0.0557	-7.20	Hydride generation AAS
65	19.0	0.724	0.68	ICP-MS
66	25	0.5	3.42	ICP-AES
72	17.9	0.9	0.17	ETAAS
74	15.6	0.34	-0.88	ETAAS
77	26.35	1.60	4.03	Hydride generation AAS
81	16.8	2.68	-0.33	ETAAS
82	14.0	0.5	-1.61	ICP-MS
83	15.62	1.83	-0.87	Hydride generation AAS
86	1.18	0.079	-7.46	ICP-MS
88	16.8	1.1	-0.33	ICP-AES
89	16.2	0.64	-0.60	ETAAS
94	19.5	1.09	0.90	ETAAS
98	13.91	0.53	-1.65	ICP-MS
99	14.93	0.30	-1.18	ICP-MS
100	13.21	2.4	-1.97	XRF
102	17.6	0.3	0.04	ICP-MS
103	22.72	1.64	2.37	ETAAS
105	17.18	0.42	-0.16	ICP-MS
106	21.3	1.2	1.73	ICP-MS
107	17.0	0.5	-0.24	Hydride generation AAS
108	18.2	1.7	0.31	Neutron activation
113	16.6	0.52	-0.42	Hydride generation AAS

TABLE I.2. ARSENIC IN THE IAEA-452 SAMPLE: INFORMATION REPORTED BY PARTICIPANTS PLUS LABORATORY SCORING PROVIDED BY THE ORGANISER (cont.)

Lab code	Mean value	Standard deviation	Z-score	Instrumental technique
118	12.26	1.27	-2.40	ETAAS
119	11.30	0.25	-2.84	ETAAS
121	18.1	0.4	0.26	ICP-MS
122	15	0	-1.15	Not reported
123	16.2	1.3	-0.60	ICP-MS
133	19.3	0.735	0.81	ICP-MS
136	17.7	0.52	0.08	Neutron Activation
137	18.03	0.56	0.23	ICP-AES
138	17.933	0.207	0.19	ICP-MS

TABLE I.3. BROMINE IN THE IAEA-452 SAMPLE: INFORMATION REPORTED BY PARTICIPANTS PLUS LABORATORY SCORING PROVIDED BY THE ORGANISER

Br - Assigned value: 500 mg kg⁻¹

Lab code	Mean value	Standard deviation	Z-score	Instrumental technique
14	433	5	-1.07	XRF
31	500	35	0.00	Neutron activation
32	581		1.30	XRF
34	507	1	0.11	Neutron activation
49	311	20	-3.02	Neutron activation
54	516	1.8	0.26	Neutron activation
96	410.9	18.8	-1.43	XRF
108	553	21	0.85	Neutron activation
135	134.9	11.1	-5.84	Neutron activation

TABLE I.4. CALCIUM IN THE IAEA-452 SAMPLE: INFORMATION REPORTED BY PARTICIPANTS PLUS LABORATORY SCORING PROVIDED BY THE ORGANISER
 Ca - Assigned value: 11305 mg kg⁻¹

Lab code	Mean value	Standard deviation	Z-score	Instrumental technique
14	8400	800	-2.06	XRF
32	7000		-3.05	XRF
34	11979	493	0.48	Neutron activation
49	13570	2430	1.60	Neutron activation
52	13677	971	1.68	Flame AAS
54	11200	680	-0.07	Neutron activation
82	10800	400	-0.36	ICP-AES
94	11000	306	-0.22	Flame AAS
96	11409.3	903.4	0.07	XRF
104	12410	320	0.78	Flame AAS
108	12952	1499	1.17	Neutron activation
122	8008	611	-2.33	Not reported

TABLE I.5. CADMIUM IN THE IAEA-452 SAMPLE: INFORMATION REPORTED BY PARTICIPANTS PLUS LABORATORY SCORING PROVIDED BY THE ORGANISER
 Cd - Assigned value: 29.60 mg kg⁻¹

Lab code	Mean value	Standard deviation	Z-score	Instrumental technique
1	30.8	2.86	0.32	ICP-MS
2	27.9	0.99	-0.46	ICP-AES
3	27.9	0.7	-0.46	ICP-MS
4	24.57	0.43	-1.36	ICP-AES
5	27.66	1.41	-0.52	Flame AAS
6	28.51	0.26	-0.29	ICP-MS
7	11.12	0.39	-4.99	ETAAS
8	30.50	0.34	0.24	ICP-MS
9	30.2	0.5	0.16	ICP-MS
10	30	1.5	0.11	ICP-MS
11	32.3	2.43	0.73	ICP-MS
12	27.6	0.9	-0.54	ICP-MS
12	27.7	0.39	-0.51	ICP-MS
13	29.37	0.88	-0.06	ICP-MS

TABLE I.5. CADMIUM IN THE IAEA-452 SAMPLE: INFORMATION REPORTED BY PARTICIPANTS PLUS LABORATORY SCORING PROVIDED BY THE ORGANISER (cont.)

Lab code	Mean value	Standard deviation	Z-score	Instrumental technique
15	22.2	0.44	-2.00	ICP-AES
16	32.43	0.50	0.76	ETAAS
17	25.75	0.58	-1.04	ICP-AES
18	29.6	0.79	0.00	ICP-AES
19	34.7	2.68	1.38	ICP-MS
20	25.5	1.21	-1.11	ICP-AES
21	32.42	0.36	0.76	ICP-MS
22	27	0.3	-0.70	ICP-MS
25	39.433	1.3	2.66	ETAAS
26	32.0	3.23	0.65	ICP-MS
27	28.8	0.17	-0.22	ICP-MS
28	26		-0.97	ICP-AES
29	29.907	1.59	0.08	ICP-MS
30	31.9	0.5	0.62	ICP-MS
31	28.0	0.84	-0.43	Flame AAS
32	29		-0.16	XRF
32	30.8	0.5	0.32	ICP-MS
33	3.18	0.17	-7.14	ETAAS
34	34.4	0.5	1.30	Neutron activation
35	33.6	0.4	1.08	ETAAS
36	31.07	0.80	0.40	Flame AAS
37	30.67	0.62	0.29	ICP-MS
39	30.8	0.5	0.32	ICP-AES
40	30.4	0.297	0.22	ICP-MS
41	29	0.6	-0.16	ICP-MS
42	33.87	0.32	1.15	ICP-MS
43	30.7	0.5	0.30	ICP-MS
44	33.212	0.053	0.98	ICP-AES
45	32.1	2.95	0.68	Flame AAS
46	31.573	0.363	0.53	Flame AAS
47	28.98	2.03	-0.17	ETAAS
48	31.7	3.56	0.57	ICP-AES
50	30.9	0.086	0.35	Flame AAS
51	28.48	2.1	-0.30	ICP-MS

TABLE I.5. CADMIUM IN THE IAEA-452 SAMPLE: INFORMATION REPORTED BY PARTICIPANTS PLUS LABORATORY SCORING PROVIDED BY THE ORGANISER (cont.)

Lab code	Mean value	Standard deviation	Z-score	Instrumental technique
52	34.05	1.33	1.20	ETAAS
53	29.38	0.35	-0.06	Flame AAS
54	35.4	0.78	1.57	Neutron activation
55	13.631	0.160	-4.32	ETAAS
55	28.76	0.93	-0.23	Flame AAS
56	22.179	0.588	-2.01	Flame AAS
57	30.3	1.2	0.19	Flame AAS
58	24.6	0.4	-1.35	Flame AAS
59	30.9	0.85	0.35	ETAAS
60	26.4	1.0	-0.86	ICP-MS
62	45.92	0.71	4.41	ETAAS
63	29.6	0.6	0.00	Flame AAS
64	29.89	0.4225	0.08	ICP-AES
65	36.2	0.615	1.78	ICP-MS
66	28	0.8	-0.43	ICP-AES
67	23.6	1.4	-1.62	ETAAS
68	23.9	0.66	-1.54	Flame AAS
71	26.64	7.58	-0.80	Flame AAS
72	30.0	0.5	0.11	Flame AAS
73	18.0	2.0	-3.14	ETAAS
74	30.6	0.53	0.27	Flame AAS
75	47.89	1.91	4.94	Flame AAS
76	26.0	4.34	-0.97	Flame AAS
77	53.2	3.60	6.38	ETAAS
79	31.310	0.875	0.46	Flame AAS
80	32.44	0.81	0.77	Flame AAS
81	29.9	1.559	0.08	Flame AAS
82	24.1	1.0	-1.49	ICP-MS
83	34.56	0.57	1.34	ETAAS
84	31.58	0.693	0.54	ETAAS
86	28.4	0.579	-0.32	ICP-MS
87	5.57	0.133	-6.49	ETAAS
88	31.8	2.0	0.59	ICP-AES
89	25.28	0.917	-1.17	ETAAS
91	28.36	2.26	-0.34	ICP-AES

TABLE I.5. CADMIUM IN THE IAEA-452 SAMPLE: INFORMATION REPORTED BY PARTICIPANTS PLUS LABORATORY SCORING PROVIDED BY THE ORGANISER (cont.)

Lab code	Mean value	Standard deviation	Z-score	Instrumental technique
92	28.96	0.57	-0.17	Polarography
93	38.2	0.6	2.32	ETAAS
94	36.0	1.60	1.73	ETAAS
97	31.0	0.896	0.38	Flame AAS
98	30.28	1.51	0.18	ICP-MS
99	27.45	0.32	-0.58	ICP-AES
100	21.59	7.8	-2.16	ETAAS
102	30.5	0.3	0.24	ICP-MS
103	31.81	0.326	0.60	ETAAS
105	31.04	1.13	0.39	ICP-MS
106	22.4	1.4	-1.95	ICP-MS
107	29.04	0.5	-0.15	Flame AAS
108	31.9	1.8	0.62	ETAAS
110	0.028833	0.003125	-7.99	ETAAS
111	2.44	1.39	-7.34	ETAAS
112	16.72	0.83	-3.48	ETAAS
113	29.1	0.86	-0.14	ETAAS
114	18.8813	0.48	-2.90	ETAAS
115	33.97	0.0035	1.18	Flame AAS
116	33.0	1.5	0.92	ETAAS
117	29.330	2.437	-0.07	Flame AAS
118	3.52	0.22	-7.05	Flame AAS
119	28.25	0.74	-0.36	ETAAS
120	31.35	0.62	0.47	ETAAS
121	32.1	0.6	0.68	ICP-MS
122	25.7	0.5	-1.05	Not reported
123	28.2	1.8	-0.38	ICP-MS
124	315.1	4.4	77.16	ICP-AES
126	28.3	1.34	-0.35	Neutron activation
127	32.2	1.22	0.70	ICP-MS
128	12.06	2.09	-4.74	Flame AAS
130	28.3	0.4	-0.35	Flame AAS
131	38.3	4.06	2.35	ETAAS
132	6.591	0.1032	-6.22	AAS (not specified)
133	3.18	0.131	-7.14	ETAAS

TABLE I.5. CADMIUM IN THE IAEA-452 SAMPLE: INFORMATION REPORTED BY PARTICIPANTS PLUS LABORATORY SCORING PROVIDED BY THE ORGANISER (cont.)

Lab code	Mean value	Standard deviation	Z-score	Instrumental technique
136	25.88	2.04	-1.01	Neutron Activation
137	29.18	0.99	-0.11	ICP-AES
138	30.033	0.151	0.12	ICP-MS

TABLE I.6. COBALT IN THE IAEA-452 SAMPLE: INFORMATION REPORTED BY PARTICIPANTS PLUS LABORATORY SCORING PROVIDED BY THE ORGANISER
Co - Assigned value: 1.62 mg kg⁻¹

Lab code	Mean value	Standard deviation	Z-score	Instrumental technique
1	1.62	0.074	0.00	ICP-MS
2	1.57	0.082	-0.25	ICP-AES
3	1.53	0.02	-0.44	ICP-MS
6	1.70	0.07	0.40	ICP-MS
7	2.19	0.32	2.81	ICP-MS
8	1.58	0.04	-0.20	ICP-MS
9	1.67	0.09	0.25	ICP-MS
10	1.45	0.04	-0.84	ICP-MS
11	1.83	0.0173	1.04	ICP-MS
12	1.736	0.0507	0.57	ICP-MS
12	1.844	0.055	1.11	ICP-MS
13	1.64	0.13	0.10	ICP-MS
15	1.33	0.02	-1.43	ICP-AES
17	1.28	0.04	-1.68	ICP-AES
20	1.017	0.041	-2.98	ICP-AES
21	1.736	0.070	0.57	ICP-MS
22	1.69	0.07	0.35	ICP-MS
27	1.51	0.026	-0.54	ICP-MS
28	1.5		-0.59	ICP-AES
31	2.1	0.17	2.37	Neutron activation
32	1.69	0.07	0.35	ICP-MS
34	1.71	0.02	0.44	Neutron activation
38	1.589	0.102	-0.15	ICP-AES
39	1.44	0.04	-0.89	ICP-AES
40	1.52	0.0349	-0.49	ICP-AES

TABLE I.6. COBALT IN THE IAEA-452 SAMPLE: INFORMATION REPORTED BY PARTICIPANTS PLUS LABORATORY SCORING PROVIDED BY THE ORGANISER (cont.)

Lab code	Mean value	Standard deviation	Z-score	Instrumental technique
41	1.19	0.04	-2.12	ICP-MS
42	2.010	0.120	1.93	ICP-MS
43	1.58	0.03	-0.20	ICP-MS
47	2.44	0.61	4.05	Flame AAS
48	1.73	0.161	0.54	ICP-AES
49	1.26	0.13	-1.78	Neutron activation
54	1.62	0.059	0.00	Neutron activation
58	1.8	0.14	0.89	Neutron activation
59	1.45	0.08	-0.84	ETAAS
60	1.632	0.047	0.06	Neutron activation
66	1.3	0.05	-1.58	ETAAS
72	1.41	0.1	-1.04	ETAAS
77	2.6	0.50	4.84	ETAAS
80	1.661	0.103	0.20	ETAAS
82	1.26	0.08	-1.78	ICP-MS
83	1.172	0.352	-2.21	ETAAS
86	1.75	0.025	0.64	ICP-MS
88	1.58	0.10	-0.20	ICP-AES
91	1.13	0.28	-2.42	ICP-AES
92	1.48	0.15	-0.69	Polarography
98	1.64	0.07	0.10	ICP-MS
99	1.60	0.01	-0.10	ICP-AES
100	1.04	0.21	-2.86	ETAAS
102	1.71	0.06	0.44	ICP-MS
105	1.69	0.0	0.35	ICP-MS
106	2.15	0.16	2.62	ICP-MS
108	1.750	0.22	0.64	Neutron activation
112	1.987	0.45	1.81	ETAAS
117	0.860	0.063	-3.75	ICP-AES
118	1.06	0.09	-2.77	Flame AAS
122	2	0	1.88	Not reported
123	1.56	0.14	-0.30	ICP-MS
125	1.71	0.10	0.44	Neutron activation
127	1.82	0.09	0.99	ICP-MS

TABLE I.6. COBALT IN THE IAEA-452 SAMPLE: INFORMATION REPORTED BY PARTICIPANTS PLUS LABORATORY SCORING PROVIDED BY THE ORGANISER (cont.)

Lab code	Mean value	Standard deviation	Z-score	Instrumental technique
129	0.85	0.05	-3.80	Neutron activation
133	0.336	0.019	-6.34	ICP-MS
135	0.543	0.065	-5.32	Neutron activation
136	1.75	0.08	0.64	Neutron Activation
137	5.34	1.18	18.37	ICP-AES
138	1.633	0.023	0.06	ICP-MS

TABLE I.7. COPPER IN THE IAEA-452 SAMPLE: INFORMATION REPORTED BY PARTICIPANTS PLUS LABORATORY SCORING PROVIDED BY THE ORGANISER
Cu - Assigned value: 10.82mg kg⁻¹

Lab code	Mean value	Standard deviation	Z-score	Instrumental technique
1	1.03	0.03	-7.24	ICP-MS
2	11.5	0.294	0.50	ICP-AES
3	10.0	0.10	-0.61	ICP-MS
5	11.73	0.82	0.67	Flame AAS
6	10.56	0.09	-0.19	ICP-MS
7	11.2	0.5	0.28	Flame AAS
8	9.60	0.12	-0.90	ICP-MS
9	11.0	0.5	0.13	ICP-MS
10	8.8	0.1	-1.49	ICP-MS
11	10.7	0.423	-0.09	ICP-AES
12	10.4	0.7	-0.31	ICP-MS
12	11.5	0.4	0.50	ICP-MS
13	9.95	0.34	-0.64	ICP-MS
15	13.6	0.48	2.06	ICP-AES
16	11.31	0.19	0.36	ETAAS
17	8.96	0.05	-1.38	ICP-AES
18	12.8	0.46	1.46	ICP-AES
19	12.1	0.55	0.95	ICP-MS
20	8.49	0.058	-1.72	ICP-AES
21	11.35	0.26	0.39	ICP-MS
22	11	0.4	0.13	ICP-MS
25	10.852	0.44	0.02	ETAAS

TABLE I.7. COPPER IN THE IAEA-452 SAMPLE: INFORMATION REPORTED BY PARTICIPANTS PLUS LABORATORY SCORING PROVIDED BY THE ORGANISER (cont.)

Lab code	Mean value	Standard deviation	Z-score	Instrumental technique
27	9.35	0.040	-1.09	ICP-MS
28	11		0.13	ICP-AES
29	10.127	0.23	-0.51	ICP-MS
30	10.8	0.2	-0.01	ICP-MS
31	9.1	0.17	-1.27	Flame AAS
32	11.7	0.2	0.65	ETAAS
32	14		2.35	XRF
33	6.20	0.13	-3.42	ETAAS
36	10.88	0.17	0.04	Flame AAS
37	12.02	0.23	0.89	ICP-MS
39	9.6	0.2	-0.90	ICP-AES
40	9.9	0.212	-0.68	ICP-MS
41	6.64	0.16	-3.09	ICP-MS
43	9.63	0.11	-0.88	ICP-MS
44	11.565	0.288	0.55	ICP-AES
45	11.9	0.8	0.80	Flame AAS
46	11.943	0.246	0.83	Flame AAS
47	9.84	0.78	-0.72	Flame AAS
48	10.8	1.25	-0.01	ICP-AES
50	26	3.3	11.22	Flame AAS
51	9.89	1.0	-0.69	ICP-MS
52	10.84	0.82	0.01	ETAAS
53	9.66	0.56	-0.86	Flame AAS
55	12.171	0.009	1.00	Flame AAS
56	7.850	0.405	-2.20	Flame AAS
57	11.1	0.26	0.21	Flame AAS
58	11.7	0.3	0.65	Flame AAS
59	9.10	0.11	-1.27	ETAAS
60	9.04	0.373	1.37	ICP-MS
62	11.34	0.12	0.38	Flame AAS
63	12.3	0.6	1.09	Flame AAS
66	13	21	1.61	ICP-AES
67	9.25	0.54	-1.16	ETAAS
68	10.3	0.189	-0.38	Flame AAS
72	9.29	0.12	-1.13	ETAAS

TABLE I.7. COPPER IN THE IAEA-452 SAMPLE: INFORMATION REPORTED BY PARTICIPANTS PLUS LABORATORY SCORING PROVIDED BY THE ORGANISER (cont.)

Lab code	Mean value	Standard deviation	Z-score	Instrumental technique
73	10.6	0.5	-0.16	ETAAS
74	11.12	0.26	0.22	Flame AAS
75	9.72	0.52	-0.81	Flame AAS
76	8.81	1.80	-1.49	Flame AAS
77	14.49	1.45	2.71	ETAAS
78	11.68	0.14	0.64	ICP-AES
79	10.3	0.39	-0.38	Flame AAS
80	11.00	1.59	0.13	ETAAS
81	9.98	0.438	-0.62	ETAAS
82	9.41	0.42	-1.04	ICP-MS
83	13.84	1.30	2.23	Flame AAS
84	11.11	0.548	0.21	ETAAS
86	10.43	0.111	-0.29	ICP-MS
87	11.55	0.276	0.54	ETAAS
88	12.7	1.0	1.39	ICP-AES
91	10.3	0.63	-0.38	ICP-AES
92	10.42	0.23	-0.30	Polarography
93	154	24	105.86	ETAAS
94	11.9	0.736	0.80	ETAAS
95	11.74	0.94	0.68	Flame AAS
96	22.86	1.35	8.90	XRF
97	11	0.6	0.13	ETAAS
98	10.91	0.37	0.07	ICP-MS
99	9.41	0.10	-1.04	ICP-AES
100	9.50	5.2	-0.98	ETAAS
102	10.9	0.05	0.06	ICP-MS
103	10.61	0.104	-0.16	Flame AAS
105	10.73	0.14	-0.07	ICP-MS
106	14.2	0.45	2.50	ICP-MS
107	8.3	0.3	-1.86	ETAAS
110	9.658	0.316812	-0.86	ETAAS
111	11.99	1.56	0.87	ETAAS
112	8.268	1.69	-1.89	ETAAS
113	7.62	0.22	-2.37	ETAAS
114	13.6638	1.0511	2.10	Flame AAS

TABLE I.7. COPPER IN THE IAEA-452 SAMPLE: INFORMATION REPORTED BY PARTICIPANTS PLUS LABORATORY SCORING PROVIDED BY THE ORGANISER (cont.)

Lab code	Mean value	Standard deviation	Z-score	Instrumental technique
115	12.63	0.45	1.34	Flame AAS
116	10.5	0.3	-0.24	ETAAS
117	12.80	0.421	1.46	Flame AAS
118	4.7	0.35	-4.52	ETAAS
119	11.0	0.65	0.13	Flame AAS
120	8.77	0.27	-1.52	ETAAS
121	11.4	0.2	0.43	ICP-MS
122	8	0	-2.09	Not reported
123	10.1	0.7	-0.53	ICP-MS
124	89.4	3.0	58.10	ICP-AES
127	11.43	0.57	0.45	ICP-MS
128	3.66	0.44	-5.29	Flame AAS
129	810.82	28.88	591.50	Neutron activation
130	14.0	1.9	2.35	ETAAS
131	9.98	1.31	-0.62	ETAAS
133	9.11	0.245	-1.26	ETAAS
137	13.93	0.09	2.30	ICP-AES
138	10.333	0.266	-0.36	ICP-MS

TABLE I.8. IRON IN THE IAEA-452 SAMPLE: INFORMATION REPORTED BY PARTICIPANTS PLUS LABORATORY SCORING PROVIDED BY THE ORGANISER
Fe - Assigned value: 1021 mg kg⁻¹

Lab code	Mean value	Standard deviation	Z-score	Instrumental technique
1	962	67.8	0.46	ICP-MS
2	1092	161.0	0.56	ICP-AES
3	1056	12	0.27	ICP-MS
4	852	20.1	-1.32	ICP-AES
5	1114.3	56.5	0.73	Flame AAS
6	513	26	-3.98	ICP-MS
7	774	96	-1.94	ICP-MS
8	1068	23	0.37	ICP-MS
9	945	27	-0.60	ICP-MS
10	830	6	-1.50	ICP-AES

TABLE I.8. IRON IN THE IAEA-452 SAMPLE: INFORMATION REPORTED BY PARTICIPANTS PLUS LABORATORY SCORING PROVIDED BY THE ORGANISER (cont.)

Lab code	Mean value	Standard deviation	Z-score	Instrumental technique
11	992	91.1	-0.23	ICP-AES
12	1019	41	-0.02	ICP-AES
13	992	45	-0.23	ICP-MS
14	1198	20	1.39	XRF
15	692	12.4	-2.58	ICP-AES
17	882.98	21.01	-1.08	ICP-AES
18	970	25.6	-0.40	ICP-AES
19	1170.2	60.9	1.17	ICP-AES
20	906	33.5	-0.90	ICP-AES
21	1073	17	0.41	ICP-MS
22	1031	40	0.08	ICP-AES
25	1216.522	121.2	1.53	Flame AAS
27	1023	11.5	0.02	ICP-AES
28	1052		0.24	Flame AAS
31	1026	34	0.04	Flame AAS
32	1090		0.54	XRF
32	1091	18	0.55	ICP-AES
34	1122	25	0.79	Neutron activation
37	1098	20	0.60	ICP-MS
38	1108.5	51.96	0.69	ICP-AES
39	1077	15	0.44	ICP-AES
40	1085	15.7	0.50	ICP-AES
41	854	20	-1.31	ICP-MS
43	1019	26	-0.02	ICP-MS
46	926.815	11.406	-0.74	Flame AAS
47	967	27.64	-0.42	Flame AAS
48	1127	131	0.83	ICP-AES
49	790	120	-1.81	Neutron activation
52	999	49	-0.17	Flame AAS
54	1060	25	0.31	Neutron activation
57	959	22	-0.49	Flame AAS
58	1107	116	0.67	Neutron activation
59	1000	63	-0.16	ETAAS
60	1102	61	0.63	Neutron activation
62	1046.0	16.11	0.20	Flame AAS

TABLE I.8. IRON IN THE IAEA-452 SAMPLE: INFORMATION REPORTED BY PARTICIPANTS PLUS LABORATORY SCORING PROVIDED BY THE ORGANISER (cont.)

Lab code	Mean value	Standard deviation	Z-score	Instrumental technique
63	1107	13	0.67	Flame AAS
64	1046	21.2	0.20	ICP-AES
66	890	51	-1.03	Flame AAS
68	813	15.6	-1.63	Flame AAS
71	760	70	-2.05	Flame AAS
72	1033	39.0	0.09	Flame AAS
74	1169	37.00	1.16	Flame AAS
75	917.76	116.63	-0.81	Flame AAS
76	919	10.40	-0.80	Flame AAS
77	1187.7	92.10	1.31	Flame AAS
78	1103.24	40.90	0.64	ICP-AES
80	1075.0	26.9	0.42	Flame AAS
81	998	31.475	-0.18	Flame AAS
82	864	33	-1.23	ICP-AES
83	1030.4	35.2	0.07	Flame AAS
86	984	8.62	-0.29	ICP-AES
87	792.03	7.985	-1.79	Flame AAS
88	920	63	-0.79	ICP-AES
91	702.23	57.38	-2.50	ICP-AES
94	1100	58	0.62	Flame AAS
95	655.36	18.43	-2.86	Flame AAS
96	753.92	39.58	-2.09	XRF
97	618.0	21.0	-3.16	Flame AAS
98	1159	48	1.08	ICP-MS
100	810	57	-1.65	ETAAS
102	1163	27	1.11	ICP-MS
103	917.9	2.55	-0.81	Flame AAS
104	1113.46	16.76	0.72	Flame AAS
105	1222.4	0.03	1.58	Flame AAS
107	1155	34	1.05	Flame AAS
108	1080	47	0.46	Neutron activation
112	1116	64.80	0.74	Flame AAS
116	845	24	-1.38	Flame AAS
117	1137.7	33.72	0.91	Flame AAS
118	961	29.60	-0.47	ETAAS

TABLE I.8. IRON IN THE IAEA-452 SAMPLE: INFORMATION REPORTED BY PARTICIPANTS PLUS LABORATORY SCORING PROVIDED BY THE ORGANISER (cont.)

Lab code	Mean value	Standard deviation	Z-score	Instrumental technique
119	1029	29.00	0.06	Flame AAS
122	880	29	-1.10	Not reported
123	1023	50	0.02	ICP-MS
125	1099.2	33.4	0.61	Neutron activation
128	868.26	46.07	-1.20	Flame AAS
129	285.5	10.85	-5.76	Neutron activation
133	880	25.1	-1.10	Flame AAS
135	400.6	54.3	-4.86	Neutron activation
136	1075	60	0.42	Neutron Activation
137	621	2.55	-3.13	ICP-AES
138	993.833	11.303	-0.21	ICP-AES
139	1037.4	18.7	0.13	ICP-AES

TABLE I.9. MERCURY IN THE IAEA-452 SAMPLE: INFORMATION REPORTED BY PARTICIPANTS PLUS LABORATORY SCORING PROVIDED BY THE ORGANISER

Hg - Assigned value: 0.150 mg kg⁻¹

Lab code	Mean value	Standard deviation	Z-score	Instrumental technique
1	0.130	0.003	-1.07	Cold vapour AFS
4	0.123	0.004	-1.44	Cold Vapour AAS
5	0.332	0.016	9.71	Cold Vapour AAS
6	0.139	0.010	-0.59	Cold vapour AFS
7	0.158	0.008	0.43	Hydride AFS
9	0.156	0.005	0.32	Cold vapour AFS
11	0.171	0.0035	1.12	ICP-MS
12	0.152	0.004	0.11	AMA Solid Mercury analyzer
15	0.027	0.003	-6.56	ICP-AES
16	0.150	0.009	0.00	Cold Vapour AAS
17	0.118	0.01	-1.71	Cold Vapour AAS
19	0.16	0.02	0.53	ICP-MS
22	0.143	0.015	-0.37	Cold Vapour AAS
23	0.139	0.0034	-0.59	AMA Solid Mercury analyzer
24	0.143	0.003	-0.37	Cold vapour AFS
26	0.167	0.00599	0.91	ICP-MS
27	0.162	0.0012	0.64	Cold Vapour AAS

TABLE I.9. MERCURY IN THE IAEA-452 SAMPLE: INFORMATION REPORTED BY PARTICIPANTS PLUS LABORATORY SCORING PROVIDED BY THE ORGANISER (cont.)

Lab code	Mean value	Standard deviation	Z-score	Instrumental technique
28	0.121		-1.55	Cold vapour ICP-AES
29	0.110	0.01922	-2.11	Cold vapour AFS
30	0.172	0.001	1.17	AMA Solid Mercury Analyzer
32	3.81	0.32	195.20	Cold Vapour AAS
34	0.146	0.027	-0.21	Neutron activation
34	0.149	0.006	-0.05	Cold vapour AFS
35	0.137	0.002	-0.69	AMA Solid Mercury Analyzer
36	0.153	0.002	0.16	Cold Vapour AAS
37	0.140	0.004	-0.53	AMA Solid Mercury Analyzer
40	0.172	0.00378	1.17	Cold Vapour AAS
41	0.177	0.006	1.44	ICP-MS
42	0.096	0.003	-2.88	ICP-MS
43	0.153	0.008	0.16	ICP-MS
48	0.177	0.020	1.44	ICP-AES
50	0.151	0.0044	0.05	Cold Vapour AAS
51	0.14	0.015	-0.53	Cold Vapour AAS
52	0.046	0.009	-5.55	Cold Vapour AAS
55	0.071	0.014	-4.19	AMA Solid Mercury Analyzer
57	0.161	0.013	0.59	Cold Vapour AAS
60	0.132	0.0057	-0.96	Neutron activation
61	0.140	0.002	-0.53	Cold vapour AFS
62	0.0905	0.0042	-3.17	Cold Vapour AAS
66	0.3	0.05	8.00	Cold Vapour AAS
68	2.03	0.136	100.27	Cold Vapour AAS
69	0.161	0.0027	0.59	Cold vapour AFS
70	0.165	0.00857	0.80	Cold vapour AFS
72	0.159	0.002	0.48	Cold Vapour AAS
74	0.156	0.001	0.32	Cold vapour AFS
77	0.19	0.02	2.13	Cold Vapour AAS
79	0.154	0.002	0.21	AMA Solid Mercury Analyzer
81	0.167	0.0019	0.91	AMA Solid Mercury Analyzer
82	0.15	0.006	0.00	Cold Vapour AAS
84	0.162	0.0015	0.64	Cold Vapour AAS
85	0.154	0.0016	0.21	AMA Solid Mercury Analyzer
86	0.148	0.0028	-0.11	Cold Vapour AAS

TABLE I.9. MERCURY IN THE IAEA-452 SAMPLE: INFORMATION REPORTED BY PARTICIPANTS PLUS LABORATORY SCORING PROVIDED BY THE ORGANISER (cont.)

Lab code	Mean value	Standard deviation	Z-score	Instrumental technique
89	0.1593	0.0012	0.50	AMA Solid Mercury Analyzer
90	0.144	0.003	-0.32	Cold vapour ICP-MS
91	0.23	0.09	4.27	ICP-AES
92	0.1652	0.00145	0.81	Cold Vapour AAS
93	0.27	0.03	6.40	Cold Vapour AAS
94	0.15	0.0052	0.00	Cold Vapour AAS
95	0.15	0.01	0.00	Cold Vapour AAS
99	0.142	0.002	-0.43	Cold Vapour AAS
100	3.19	0.96	162.13	XRF
101	0.146	0.0032	-0.21	IDMS
103	0.157	0.0036	0.37	AMA Solid Mercury Analyzer
105	0.107	0.0	-2.29	Hydride generation AAS
106	0.253	0.028	5.49	ICP-MS
107	0.144	0.004	-0.32	Cold vapour AFS
109	0.1549	0.0003	0.26	AMA Solid Mercury Analyzer
110	0.1161683	0.01132814	-1.80	Cold Vapour AAS
113	0.15	0.02	0.00	Cold Vapour AAS
117	0.130	0.032	-1.07	Cold Vapour AAS
118	0.223	0.01	3.89	Cold Vapour AAS
119	0.156	0.01	0.32	AMA Solid Mercury Analyzer
120	0.141	0.0052	-0.48	Cold Vapour AAS
123	0.146	0.003	-0.21	AMA Solid Mercury Analyzer
131	0.18	0.01	1.60	Cold Vapour AAS
133	0.155	0.004	0.27	ICP-MS
134	0.1335	0.00194	-0.88	AAS (not specified)
136	0.14	0.008	-0.53	Neutron Activation
137	0.38	0.02	12.27	ICP-AES
138	0.145	0.002	-0.27	Cold Vapour AAS

TABLE I.10. POTASSIUM IN THE IAEA-452 SAMPLE: INFORMATION REPORTED BY PARTICIPANTS PLUS LABORATORY SCORING PROVIDED BY THE ORGANISER
 K - Assigned value: 13140 mg kg⁻¹

Lab code	Mean value	Standard deviation	Z-score	Instrumental technique
14	13140	160	0.00	XRF
32	12500		-0.39	XRF
34	13492	1022	0.21	Neutron activation
49	9060	430	-2.48	Neutron activation
52	13798	500	0.40	Flame AAS
54	15700	2200	1.56	Neutron activation
60	13685	740	0.33	Neutron activation
82	11200	100	-1.18	ICP-AES
94	13600	606	0.28	Flame AAS
96	9422.3	243.4	-2.26	XRF
104	13150	260	0.01	Flame AAS
108	1.33	0.08	-8.00	Neutron activation
135	1752	170	-6.93	Neutron activation

TABLE I.11. LITHIUM IN THE IAEA-452 SAMPLE: INFORMATION REPORTED BY PARTICIPANTS PLUS LABORATORY SCORING PROVIDED BY THE ORGANISER
 Li - Assigned value: 2.01 mg kg⁻¹

Lab code	Mean value	Standard deviation	Z-score	Instrumental technique
2	1.05	0.16	-3.82	ICP-AES
6	2.01	0.08	0.00	ICP-MS
9	0.647	0.096	-5.42	ICP-MS
11	3.21	0.163	4.78	ICP-MS
13	1.51	0.14	-1.99	ICP-MS
15	1.04	0.06	-3.86	ICP-AES
17	1.28	0.06	-2.91	ICP-AES
22	1.93	0.15	-0.32	ICP-MS
28	2.0		-0.04	ICP-AES
32	2.65	0.005	2.55	ICP-MS
41	1.42	0.09	-2.35	ICP-MS
43	2.25	0.03	0.96	ICP-MS
48	2.26	0.205	1.00	ICP-AES
82	1.42	0.07	-2.35	ICP-MS

TABLE I.11. LITHIUM IN THE IAEA-452 SAMPLE: INFORMATION REPORTED BY PARTICIPANTS PLUS LABORATORY SCORING PROVIDED BY THE ORGANISER (cont.)

86	3.02	0.052	4.02	ICP-MS
98	2.46	0.10	1.79	ICP-MS
100	0.99	0.30	-4.06	ETAAS
102	2.55	0.07	2.15	ICP-MS
111	2.20	0.46	0.76	ETAAS
122	66	2	254.69	Not reported
128	2.03	0.29	0.08	Flame AAS
137	1.31	0.24	-2.79	ICP-AES
138	2.025	0.058	0.06	ICP-MS

TABLE I.12. METHYLMERCURY IN THE IAEA-452 SAMPLE: INFORMATION REPORTED BY PARTICIPANTS PLUS LABORATORY SCORING PROVIDED BY THE ORGANISER

MeHg - Assigned value: 0.0217 mg kg⁻¹

Lab code	Mean value	Standard deviation	Z-score	Instrumental technique
1	0.0216	0.0053	0.64	See detail in Table 6
6	0.018	0.0022	-0.80	See detail in Table 6
7	0.023	0.004	1.20	See detail in Table 6
9	0.0203	0.0016	0.12	See detail in Table 6
24	0.020	0.002	0.00	See detail in Table 6
37	0.0238	0.0017	1.52	See detail in Table 6
40	0.0234	0.0020	1.36	See detail in Table 6
43	0.0186	0.0004	-0.56	See detail in Table 6
61	0.0042	0.0005	-6.32	See detail in Table 6
69	0.0219	0.00047	0.76	See detail in Table 6
70	0.0230	0.0014	1.20	See detail in Table 6
85	0.0123	0.0014	-3.08	See detail in Table 6
90	0.0221	0.0004	0.84	See detail in Table 6
101	0.0222	0.0006	0.88	See detail in Table 6
107	0.0156	0.0018	-1.76	See detail in Table 6
118	0.201	0.01	72.40	See detail in Table 6
119	0.0285	0.0048	3.40	See detail in Table 6
123	0.0216	0.0016	0.64	See detail in Table 6
134	0.0208	0.00247	0.32	See detail in Table 6
138	0.027	0.001	2.80	See detail in Table 6

TABLE I.13. MAGNESIUM IN THE IAEA-452 SAMPLE: INFORMATION REPORTED BY PARTICIPANTS PLUS LABORATORY SCORING PROVIDED BY THE ORGANISER
Mg - Assigned value: 6380 mg kg⁻¹

Lab code	Mean value	Standard deviation	Z-score	Instrumental technique
2	6450	623.7	0.09	ICP-AES
6	3640	150	-3.44	ICP-MS
9	6370	410	-0.01	ICP-MS
11	6155	284	-0.28	ICP-AES
13	5557	223	-1.03	ICP-MS
15	3999	396	-2.99	ICP-AES
28	5700		-0.85	Flame AAS
32	5000		-1.73	XRF
38	6722.2	86.7	0.43	ICP-AES
40	6385	83.2	0.01	ICP-AES
43	6687	110	0.39	ICP-MS
47	9420	247	3.81	Flame AAS
49	55600	7470	61.72	Neutron activation
52	6551	233	0.22	Flame AAS
82	5810	80	-0.71	ICP-AES
94	6550	40	0.21	Flame AAS
97	6374	191.0	-0.01	Flame AAS
104	8420	740	2.56	Flame AAS
129	13700	400	9.18	Neutron activation
136	5300	316	-1.35	Neutron Activation
137	5961.33	4.27	-0.52	ICP-AES
138	6601.667	60.47	0.28	ICP-MS

TABLE I.14. MANGANESE IN THE IAEA-452 SAMPLE: INFORMATION REPORTED BY PARTICIPANTS PLUS LABORATORY SCORING PROVIDED BY THE ORGANISER
Mn - Assigned value: 273 mg kg⁻¹

Lab code	Mean value	Standard deviation	Z-score	Instrumental technique
1	264	14.1	0.26	ICP-MS
2	260	4.55	-0.38	ICP-AES
3	310	11	1.09	ICP-MS
5	311.4	17.0	1.13	Flame AAS
6	287	7	0.42	ICP-MS

TABLE I.14. MANGANESE IN THE IAEA-452 SAMPLE: INFORMATION REPORTED BY PARTICIPANTS PLUS LABORATORY SCORING PROVIDED BY THE ORGANISER (cont.)

Lab code	Mean value	Standard deviation	Z-score	Instrumental technique
7	199	22	-2.16	ICP-MS
9	274	19	0.04	ICP-MS
10	290	15	0.50	ICP-AES
11	275	15.4	0.06	ICP-AES
12	244	11	-0.84	ICP-MS
13	281.19	8.32	0.25	ICP-MS
14	297	24	0.71	XRF
15	233	6.10	-1.17	ICP-AES
17	223.99	5.49	-1.43	ICP-AES
18	283	5.92	0.30	ICP-AES
19	302.7	13.8	0.88	ICP-AES
20	197.3	2.52	-2.21	ICP-AES
21	297.7	9.5	0.73	ICP-MS
22	299	11	0.77	ICP-AES
25	249.93	6.9	-0.67	Flame AAS
27	276	9.8	0.09	ICP-AES
28	251		-0.64	ICP-AES
31	263	5	-0.29	Flame AAS
32	282	5	0.27	ICP-AES
32	291		0.53	XRF
36	260.65	6.66	-0.36	Flame AAS
37	286.2	14.3	0.39	ICP-MS
38	289.9	4.79	0.50	ICP-AES
39	270	5	-0.08	ICP-AES
40	265	7.17	-0.23	ICP-AES
41	197	4	-2.22	ICP-MS
43	290	10	0.50	ICP-MS
46	291.909	8.2	0.56	Flame AAS
47	278	7.36	0.15	Flame AAS
48	290	35	0.50	ICP-AES
49	102	31	-5.01	Neutron activation
51	272.6	11.8	-0.01	ICP-MS
52	223	7	-1.46	Flame AAS
58	260	22	-0.38	Neutron activation
59	274	13.8	0.04	ETAAS

TABLE I.14. MANGANESE IN THE IAEA-452 SAMPLE: INFORMATION REPORTED BY PARTICIPANTS PLUS LABORATORY SCORING PROVIDED BY THE ORGANISER (cont.)

Lab code	Mean value	Standard deviation	Z-score	Instrumental technique
60	283.8	8.5	0.32	Neutron activation
62	276.6	2.93	0.11	Flame AAS
63	292	10	0.56	Flame AAS
66	292	13	0.56	Flame AAS
67	266	5.1	-0.20	Flame AAS
68	273.8	10.2	0.03	Flame AAS
72	273	4	0.01	Flame AAS
74	279	6.62	0.18	Flame AAS
75	42.8	2.01	-6.74	Flame AAS
76	39.6	3.16	-6.84	Flame AAS
77	23.6	2.40	-7.31	Flame AAS
80	269.0	9.3	-0.11	Flame AAS
81	268	4.509	-0.14	Flame AAS
82	244	11	-0.84	ICP-MS
83	293.8	7.17	0.62	Flame AAS
86	261	3.89	-0.35	ICP-AES
87	322.92	5.151	1.47	Flame AAS
88	274	6	0.04	ICP-AES
91	172.31	29.06	-2.95	ICP-AES
95	221.92	5.90	-1.49	Flame AAS
96	216.83	7.69	-1.64	XRF
97	270.0	4.4	-0.08	Flame AAS
98	300.1	12.5	0.80	ICP-MS
99	235.5	3.90	-1.09	ICP-AES
100	229	18	-1.28	ETAAS
102	284	3.6	0.33	ICP-MS
103	275.8	3.23	0.09	Flame AAS
106	261	14	-0.35	ICP-MS
107	300	11.7	0.80	Flame AAS
112	260	22.7	-0.38	Flame AAS
114	317.07	6.743	1.30	Flame AAS
115	317	3.6	1.30	Flame AAS
116	209	7	-1.87	Flame AAS
117	253.8	6.063	-0.56	Flame AAS
118	244	12.40	-0.84	Flame AAS

TABLE I.14. MANGANESE IN THE IAEA-452 SAMPLE: INFORMATION REPORTED BY PARTICIPANTS PLUS LABORATORY SCORING PROVIDED BY THE ORGANISER (cont.)

Lab code	Mean value	Standard deviation	Z-score	Instrumental technique
119	273.9	1.30	0.03	Flame AAS
121	284	9	0.33	ICP-AES
122	225	9	-1.40	Not reported
123	263	23	-0.29	ICP-MS
128	31.25	1.25	-7.08	Flame AAS
129	649.77	13.80	11.05	Neutron activation
133	61.2	0.674	-6.21	Flame AAS
136	258	8	-0.43	Neutron Activation
137	242	1.92	-0.90	ICP-AES
138	309.833	3.92	1.09	ICP-MS
139	346.2	1.4	2.15	ICP-AES

TABLE I.15. SODIUM IN THE IAEA-452 SAMPLE: INFORMATION REPORTED BY PARTICIPANTS PLUS LABORATORY SCORING PROVIDED BY THE ORGANISER
Na - Assigned value: 43959 mg kg⁻¹

Lab code	Mean value	Standard deviation	Z-score	Instrumental technique
31	56000	2500	2.19	Neutron activation
34	45885	218	0.35	Neutron activation
49	37410	1210	-1.19	Neutron activation
52	43959	1879	0.00	Flame AAS
54	44400	230	0.08	Neutron activation
60	46569	1077	0.47	Neutron activation
82	39700	400	-0.78	ICP-AES
94	41300	1250	-0.48	Flame AAS
104	45620	430	0.30	Flame AAS
108	43000	1000	-0.17	Neutron activation
135	3823	224	-7.30	Neutron activation

TABLE I.16. RUBIDIUM IN THE IAEA-452 SAMPLE: INFORMATION REPORTED BY PARTICIPANTS PLUS LABORATORY SCORING PROVIDED BY THE ORGANISER
 Rb - Assigned value: 7.85 mg kg⁻¹

Lab code	Mean value	Standard deviation	Z-score	Instrumental technique
31	8.2	0.43	0.36	Neutron activation
32	14		6.27	XRF
34	7.87	0.24	0.02	Neutron activation
43	7.09	0.14	-0.77	ICP-MS
49	10.98	1.87	3.19	Neutron activation
54	7.85	0.26	0.00	Neutron activation
98	7.06	0.1	-0.81	ICP-MS
102	6.97	0.18	-0.90	ICP-MS
108	7.6	0.5	-0.25	Neutron activation

TABLE I.17. ANTIMONY IN THE IAEA-452 SAMPLE: INFORMATION REPORTED BY PARTICIPANTS PLUS LABORATORY SCORING PROVIDED BY THE ORGANISER
 Sb - Assigned value: 0.099 mg kg⁻¹

Lab code	Mean value	Standard deviation	Z-score	Instrumental technique
1	0.09	0.01	-0.80	ICP-MS
6	0.097	0.003	-0.24	ICP-MS
9	0.099	0.015	-0.08	ICP-MS
10	0.12	0.01	1.60	ICP-MS
11	0.140	0.0116	3.20	ICP-MS
13	0.12	0.01	1.60	ICP-MS
22	0.069	0.003	-2.48	ICP-MS
27	0.084	0.0026	-1.28	ICP-MS
32	0.103	0.004	0.24	ICP-MS
34	0.131	0.005	2.48	Neutron activation
40	0.0965	0.0041	-0.28	ICP-MS
41	0.0968	0.0042	-0.26	ICP-MS
42	0.092	0.003	-0.64	ICP-MS
43	0.098	0.007	-0.16	ICP-MS
54	0.135	0.0048	2.80	Neutron activation
86	0.108	0.0079	0.64	ICP-MS
98	0.093	0.011	-0.56	ICP-MS
100	1.06	0.27	76.80	XRF

TABLE I.17. ANTIMONY IN THE IAEA-452 SAMPLE: INFORMATION REPORTED BY PARTICIPANTS PLUS LABORATORY SCORING PROVIDED BY THE ORGANISER (cont.)

122	0.06	0.01	-3.20	Not reported
125	0.115	0.018	1.20	Neutron activation
136	0.12	0.01	1.60	Neutron Activation
137	0.46	0.01	28.80	ICP-AES
138	0.091	0.003	-0.72	ICP-MS

TABLE I.18. SELENIUM IN THE IAEA-452 SAMPLE: INFORMATION REPORTED BY PARTICIPANTS PLUS LABORATORY SCORING PROVIDED BY THE ORGANISER

Se - Assigned value: 6.55 mg kg⁻¹

Lab code	Mean value	Standard deviation	Z-score	Instrumental technique
1	8.31	1.89	2.15	ICP-MS
2	9.04	1.36	3.04	ICP-AES
3	6.28	0.17	-0.33	ICP-MS
5	4.017	0.239	-3.09	Hydride generation AAS
6	6.25	0.10	-0.37	ICP-MS
9	6.58	0.39	0.04	ICP-MS
10	7.1	0.1	0.67	ICP-MS
11	6.54	0.191	-0.01	ICP-MS
12	6.182	0.195	-0.45	ICP-MS
12	7.565	0.127	1.24	ICP-MS
13	16.83	1.3	12.56	ICP-MS
15	5.94	0.51	-0.75	ICP-AES
17	5.49	0.33	-1.29	ICP-AES
19	8.7	1.33	2.63	ICP-MS
21	6.242	0.077	-0.38	ICP-MS
22	6.90	0.15	0.43	ICP-MS
28	6.7		0.18	ICP-AES
31	7.7	0.36	1.40	Neutron activation
32	5.71	0.13	-1.03	ICP-MS
32	6		-0.67	XRF
34	6.84	0.09	0.35	Neutron activation
36	6.48	0.26	-0.09	Hydride generation AAS
37	7.68	0.21	1.38	ICP-MS
39	6.8	0.4	0.31	ICP-AES
40	7.07	0.116	0.64	ICP-MS

TABLE I.18. SELENIUM IN THE IAEA-452 SAMPLE: INFORMATION REPORTED BY PARTICIPANTS PLUS LABORATORY SCORING PROVIDED BY THE ORGANISER (cont.)

Lab code	Mean value	Standard deviation	Z-score	Instrumental technique
41	2.61	0.34	-4.81	ICP-MS
42	4.7	0.127	-2.26	ICP-MS
43	9.21	0.44	3.25	ICP-MS
47	7.07	0.42	0.64	Hydride generation AAS
48	3.38	0.39	-3.87	ICP-AES
49	1.64	0.5	-6.00	Neutron activation
54	6.96	0.058	0.50	Neutron activation
58	4.8	0.4	-2.14	Flame AAS
60	6.25	0.28	-0.37	Neutron activation
66	12	0	6.66	ICP-AES
77	4.11	0.39	-2.98	Hydride generation AAS
82	5.71	0.18	-1.03	ICP-MS
86	4.92	0.283	-1.99	ICP-MS
97	4.5	0.3	-2.50	ETAAS
98	5.8	0.2	-0.92	ICP-MS
100	6.04	1.51	-0.62	XRF
102	6.71	0.27	0.20	ICP-MS
106	9.44	0.65	3.53	ICP-MS
107	6.2	0.24	-0.43	Hydride generation AAS
108	6.284	0.249	-0.32	Neutron activation
122	4	0	-3.11	Not reported
123	7.50	0.94	1.16	ICP-MS
125	6.82	0.09	0.33	Neutron activation
127	7.63	0.72	1.32	ICP-MS
133	6.56	0.155	0.01	ICP-MS
135	20.2	4.0	16.67	Neutron activation
136	6.37	0.26	-0.22	Neutron Activation
137	9.75	1.07	3.91	ICP-AES
138	7.5	0.126	1.16	ICP-MS

TABLE I.19. STRONTIUM IN THE IAEA-452 SAMPLE: INFORMATION REPORTED BY PARTICIPANTS PLUS LABORATORY SCORING PROVIDED BY THE ORGANISER
 Sr - Assigned value: 82.9 mg kg⁻¹

Lab code	Mean value	Standard deviation	Z-score	Instrumental technique
1	82.8	5.02	-0.01	ICP-MS
2	73.3	3.89	-0.93	ICP-AES
3	91.7	4.2	0.85	ICP-MS
6	75.5	2.1	-0.71	ICP-MS
7	113.2	8.11	2.92	ICP-MS
8	84.97	2.29	0.20	ICP-MS
9	85.4	2.7	0.24	ICP-MS
11	87.5	5.24	0.44	ICP-MS
13	90.84	4.97	0.77	ICP-MS
14	81	2	-0.18	XRF
15	60.4	1.81	-2.17	ICP-AES
17	65.36	2.85	-1.69	ICP-AES
18	87.9	2.09	0.48	ICP-AES
21	85.58	2.39	0.26	ICP-MS
22	88	8	0.49	ICP-AES
27	69.0	2.77	-1.34	ICP-MS
28	62		-2.02	ICP-AES
32	82.0	3.1	-0.09	ICP-AES
32	83		0.01	XRF
34	84.6	3.3	0.16	Neutron activation
39	85	3	0.20	ICP-AES
40	78.5	0.886	-0.42	ICP-MS
41	72	2.7	-1.05	ICP-MS
43	83.3	2.6	0.04	ICP-MS
48	76.1	12.1	-0.66	ICP-AES
49	35	9	-4.62	Neutron activation
54	91.1	3	0.79	Neutron activation
60	77.0	5.8	-0.57	Neutron activation
66	71	3.5	-1.15	ICP-AES
82	73.9	4.8	-0.87	ICP-MS
86	70.6	1.63	-1.19	ICP-AES
96	71.64	6.92	-1.09	XRF
98	84.8	7.3	0.18	ICP-MS
100	58	11.6	-2.40	ETAAS

TABLE I.19. STRONTIUM IN THE IAEA-452 SAMPLE: INFORMATION REPORTED BY PARTICIPANTS PLUS LABORATORY SCORING PROVIDED BY THE ORGANISER (cont.)

Lab code	Mean value	Standard deviation	Z-score	Instrumental technique
102	83.2	2.2	0.03	ICP-MS
105	78.29	2.78	-0.44	ICP-MS
112	44.58	6.800	-3.70	Flame AAS
122	295	9	20.47	Not reported
123	83.4	3.6	0.05	ICP-MS
136	86	4.9	0.30	Neutron Activation
137	94.83	1.47	1.15	ICP-AES
138	86.433	2.336	0.34	ICP-MS

TABLE I.20. VANADIUM IN THE IAEA-452 SAMPLE: INFORMATION REPORTED BY PARTICIPANTS PLUS LABORATORY SCORING PROVIDED BY THE ORGANISER
V - Assigned value: 6.36 mg kg^{-1}

Lab code	Mean value	Standard deviation	Z-score	Instrumental technique
1	6.48	0.47	0.15	ICP-MS
2	6.18	0.47	-0.23	ICP-AES
3	6.89	0.08	0.67	ICP-MS
5	8.234	0.528	2.36	ETAAS
6	6.10	0.09	-0.33	ICP-MS
9	6.37	0.21	0.01	ICP-MS
10	6	0.1	-0.45	ICP-MS
11	9.11	0.313	3.46	ICP-MS
13	7.34	0.29	1.23	ICP-MS
15	4.38	0.28	-2.49	ICP-AES
17	5.63	0.18	-0.92	ICP-AES
20	6.05	0.02	-0.39	ICP-AES
21	7.128	0.132	0.97	ICP-MS
22	6.80	0.36	0.55	ICP-MS
27	6.49	0.04	0.16	ICP-MS
28	6.8		0.55	ICP-AES
32	7.17	0.11	1.02	ICP-MS
37	6.96	0.1	0.75	ICP-MS
39	6.87	0.07	0.64	ICP-AES
40	6.94	0.0421	0.73	ICP-AES

TABLE I.20. VANADIUM IN THE IAEA-452 SAMPLE: INFORMATION REPORTED BY PARTICIPANTS PLUS LABORATORY SCORING PROVIDED BY THE ORGANISER (cont.)

Lab code	Mean value	Standard deviation	Z-score	Instrumental technique
41	5.91	0.12	-0.57	ICP-MS
43	5.89	0.09	-0.59	ICP-MS
48	7.19	0.796	1.04	ICP-AES
58	7	1.0	0.81	Neutron activation
59	5.53	0.06	-1.04	ETAAS
60	6.6	1.2	0.30	Neutron activation
64	5.77	0.1079	-0.74	ICP-AES
66	6	0	-0.45	ETAAS
72	5.83	0.11	-0.67	ETAAS
82	4.92	0.26	-1.81	ICP-MS
86	6.97	0.183	0.77	ICP-MS
88	5.53	0.15	-1.04	ICP-AES
91	4.60	0.23	-2.21	ICP-AES
98	7.02	0.11	0.83	ICP-MS
99	4.77	0.10	-2.00	ICP-MS
100	4.04	0.77	-2.92	XRF
102	7.51	0.15	1.45	ICP-MS
105	6.98	0.07	0.78	ICP-MS
106	4.54	0.46	-2.29	ICP-MS
112	2.51	0.574	-4.84	ETAAS
117	5.26	0.082	-1.38	ICP-AES
122	2.77	0.18	-4.52	Not reported
128	3.71	0.41	-3.33	Flame AAS
129	6.17	0.10	-0.24	Neutron activation
131	6.90	0.43	0.68	ETAAS
136	6.35	0.38	-0.01	Neutron Activation
137	8.45	0.14	2.63	ICP-AES
138	6.725	0.096	0.46	ICP-MS

TABLE I.21. ZINC IN THE IAEA-452 SAMPLE: INFORMATION REPORTED BY PARTICIPANTS PLUS LABORATORY SCORING PROVIDED BY THE ORGANISER
 Zn - Assigned value: 166 mg kg⁻¹

Lab code	Mean value	Standard deviation	Z-score	Instrumental technique
1	160	9.87	-0.29	ICP-MS
2	169.5	5.32	0.17	ICP-AES
3	194	4	1.35	ICP-MS
4	132	1.73	-1.64	ICP-AES
5	164.8	5.84	-0.06	Flame AAS
6	109	14	-2.75	ICP-MS
7	170.9	2.2	0.24	Flame AAS
8	156.2	0.9	-0.47	ICP-MS
9	165	3.9	-0.05	ICP-MS
10	170	6	0.19	ICP-AES
11	171	9.57	0.24	ICP-AES
12	148	2.9	-0.87	ICP-MS
12	152	4	-0.67	ICP-MS
13	129.81	3.58	-1.74	ICP-MS
14	214	7	2.31	XRF
15	170	4.33	0.19	ICP-AES
16	173	3.6	0.34	Flame AAS
17	155.76	2.00	-0.49	ICP-AES
18	167	5.08	0.05	ICP-AES
19	170.6	11.9	0.22	ICP-MS
20	133.3	1.15	-1.58	ICP-AES
21	174.8	4.0	0.42	ICP-MS
22	141	2	-1.20	ICP-AES
25	167.695	4.4	0.08	Flame AAS
27	159.3	3.1	-0.32	ICP-MS
28	144		-1.06	ICP-AES
29	158.289	3.80	-0.37	ICP-MS
30	168	2	0.10	ICP-MS
31	156	4	-0.48	Flame AAS
32	168	1	0.10	ICP-AES
32	213		2.27	XRF
34	174	3	0.39	Neutron activation
36	170.63	6.66	0.22	Flame AAS
37	174.2	4.5	0.40	ICP-MS

TABLE I.21. ZINC IN THE IAEA-452 SAMPLE: INFORMATION REPORTED BY PARTICIPANTS PLUS LABORATORY SCORING PROVIDED BY THE ORGANISER (cont.)

Lab code	Mean value	Standard deviation	Z-score	Instrumental technique
38	173.4	5.417	0.36	ICP-AES
39	169	3	0.14	ICP-AES
40	155	3.47	-0.53	ICP-MS
41	105	2	-2.94	ICP-MS
43	174	2	0.39	ICP-MS
44	181.826	1.4390	0.76	ICP-AES
45	177	14.0	0.53	Flame AAS
47	161.52	8.79	-0.22	Flame AAS
48	175	21.0	0.43	ICP-AES
49	139	9	-1.30	Neutron activation
50	192.4	0.3	1.27	Flame AAS
51	148.2	5.9	-0.86	ICP-MS
52	182	5	0.77	Flame AAS
53	167.03	1.48	0.05	Flame AAS
54	168	1.1	0.10	Neutron activation
57	166	4.4	0.00	Flame AAS
58	163	12	-0.14	Neutron activation
59	157	6.4	-0.43	ETAAS
60	170.1	6.2	0.20	ICP-MS
62	173.1	0.52	0.34	Flame AAS
63	171	5	0.24	Flame AAS
64	179	1.528	0.63	ICP-AES
65	197.9	3.28	1.54	ICP-MS
66	190	9.8	1.16	Flame AAS
67	157	9.2	-0.43	Flame AAS
68	143.8	1.25	-1.07	Flame AAS
71	180	20	0.67	Flame AAS
72	165	3	-0.05	Flame AAS
73	177	6.8	0.53	Flame AAS
74	166	3.82	0.00	Flame AAS
75	206.14	10.01	1.93	Flame AAS
76	164.65	7.81	-0.07	Flame AAS
77	213.7	11.8	2.30	Flame AAS
78	176.39	3.50	0.50	ICP-AES
79	175	6	0.43	Flame AAS

TABLE I.21. ZINC IN THE IAEA-452 SAMPLE: INFORMATION REPORTED BY PARTICIPANTS PLUS LABORATORY SCORING PROVIDED BY THE ORGANISER (cont.)

Lab code	Mean value	Standard deviation	Z-score	Instrumental technique
80	175.2	4.0	0.44	Flame AAS
81	143	2.082	-1.11	Flame AAS
82	143	6	-1.11	ICP-MS
83	176.4	3.78	0.50	Flame AAS
86	156	1.67	-0.48	ICP-AES
88	160	12	-0.29	ICP-AES
91	126.30	10.27	-1.91	ICP-AES
92	159.0	4.8	-0.34	Polarography
93	412	44	11.86	ETAAS
94	176	2.07	0.48	Flame AAS
95	103.82	4.07	-3.00	Flame AAS
96	156.58	10.55	-0.45	XRF
97	184.0	5.5	0.87	Flame AAS
98	163	6	-0.14	ICP-MS
99	152.9	0.48	-0.63	ICP-AES
100	122	31.72	-2.12	ETAAS
102	166	2.1	0.00	ICP-MS
103	161.3	1.40	-0.23	Flame AAS
104	172.29	6.13	0.30	Flame AAS
105	164.0	2.96	-0.10	ICP-MS
106	131	7	-1.69	ICP-MS
107	163.9	2.1	-0.10	Flame AAS
108	158	9	-0.39	Neutron activation
112	133.3	14.13	-1.58	Flame AAS
113	183	3.81	0.82	Flame AAS
114	147.9674	3.7442	-0.87	Flame AAS
115	170	4.3	0.19	Flame AAS
117	162.58	6.723	-0.16	Flame AAS
118	170	9.8	0.19	Flame AAS
119	165.1	3.60	-0.04	Flame AAS
121	166	3	0.00	ICP-AES
122	134	4	-1.54	Not reported
123	162	13	-0.19	ICP-MS
124	1743.3	46.6	76.01	ICP-AES
125	170.7	2.5	0.23	Neutron activation

TABLE I.21. ZINC IN THE IAEA-452 SAMPLE: INFORMATION REPORTED BY PARTICIPANTS PLUS LABORATORY SCORING PROVIDED BY THE ORGANISER (cont.)

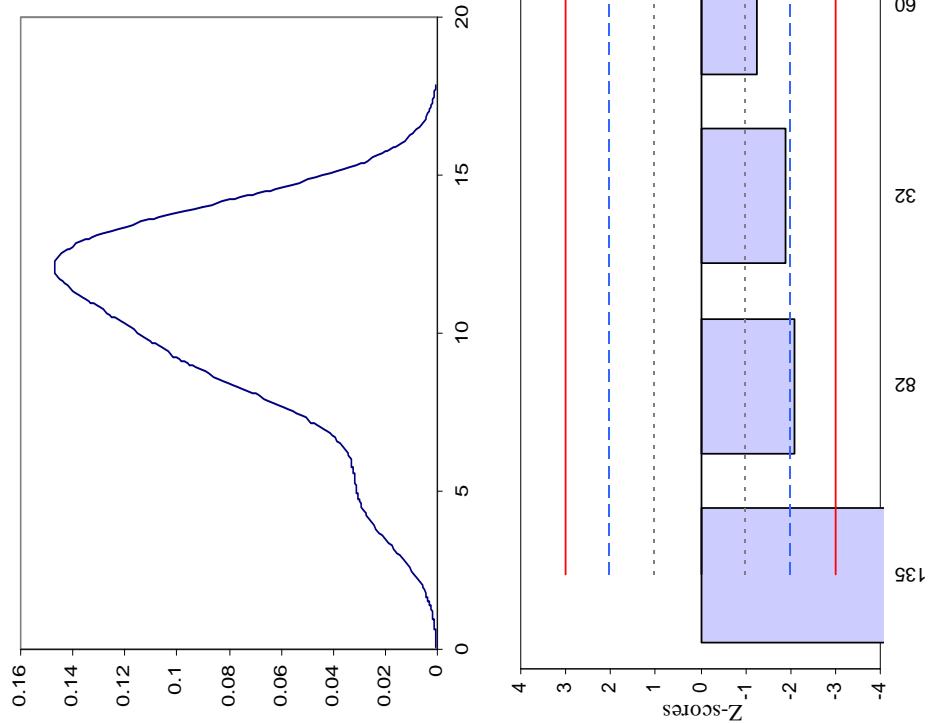
Lab code	Mean value	Standard deviation	Z-score	Instrumental technique
127	176.68	8.77	0.51	ICP-MS
128	0.59	0.009	-7.97	Flame AAS
129	43.50	3.0	-5.90	Neutron activation
130	168	10	0.10	Flame AAS
131	154	6.18	-0.58	Flame AAS
133	173	6.31	0.34	Flame AAS
135	85.4	11.6	-3.88	Neutron activation
136	158.7	6.4	-0.35	Neutron Activation
137	136.64	1.31	-1.41	ICP-AES
138	171.667	1.033	0.27	ICP-MS
139	179.4	10.7	0.65	ICP-AES

APPENDIX II

**GRAPHICAL PRESENTATION OF RESULTS
SORTED BY ELEMENTS**

FIGURE II.1. – FIGURE II.21.

Kernel Density Plot
Used $h_{\text{Opt}} = 1.58103181236442$

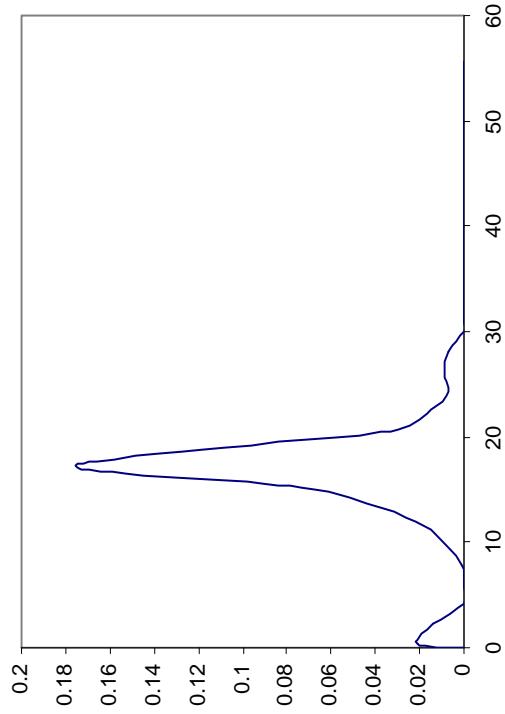


Summary of results

Assigned value	11.80 mg kg ⁻¹
Robust standard deviation	2.1 mg kg ⁻¹
Target standard deviation (fitness for purpose)	1.5 mg kg ⁻¹
Number of participants	9
Number of methods	4
Satisfactory: $ Z \leq 2$	77.8 %
Questionable: $2 < Z < 3$	11.1 %
Unsatisfactory: $ Z \geq 3$	11.1 %

FIG. II.1. Performance evaluation of reported results for Ag in the IAEA-452 sample.

Kernel Density Plot
Used hOpt =0.774678724577148



Summary of results

Assigned value	17.52 mg kg^{-1}
Robust standard deviation	2.3 mg kg^{-1}
Target standard deviation (fitness for purpose)	2.2 mg kg^{-1}
Number of participants	76
Number of methods	6
Satisfactory: $ Z \leq 2$	80.3 %
Questionable: $2 < Z < 3$	6.6 %
Unsatisfactory: $ Z \geq 3$	13.2 %

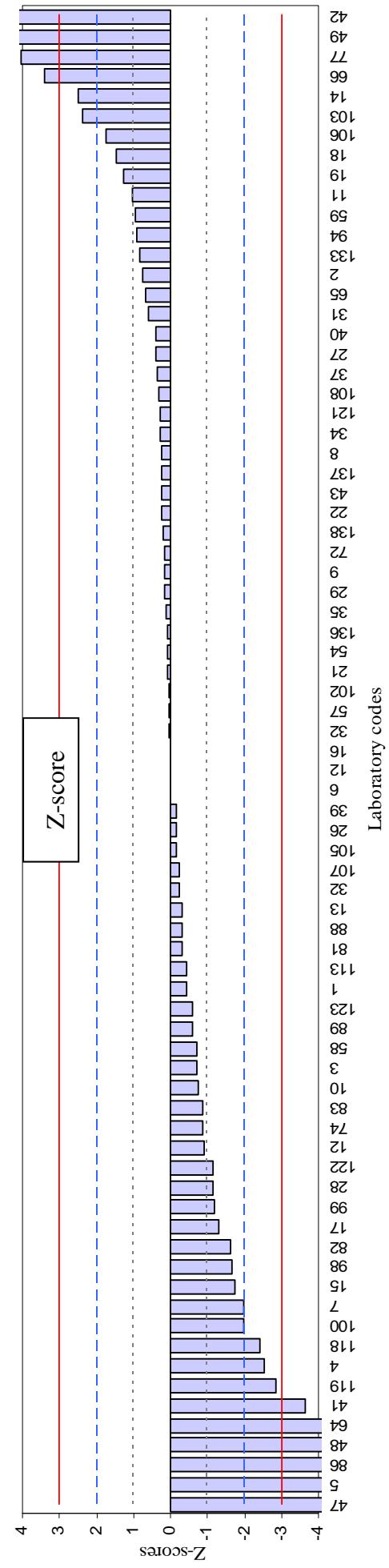
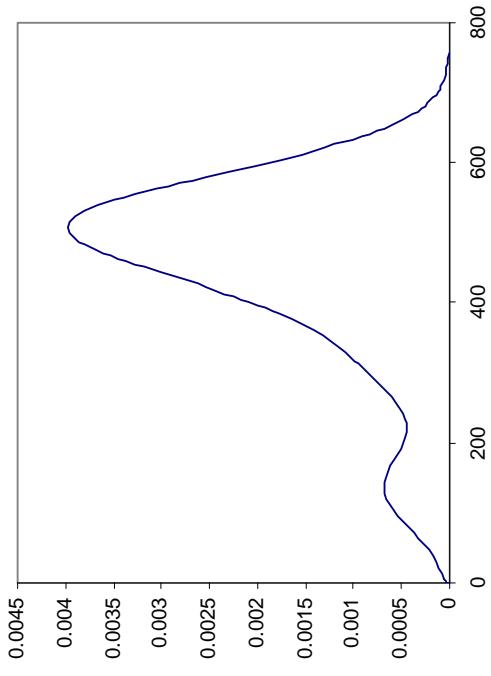


FIG. II.2. Performance evaluation of reported results for As in the IAEA-452 sample.

Kernel Density Plot
Used hOpt = 59.0427752371139



Summary of results

Assigned value	500 mg kg^{-1}
Robust standard deviation	95 mg kg^{-1}
Target standard deviation (fitness for purpose)	62 mg kg^{-1}
Number of participants	9
Number of methods	2
Satisfactory: $ Z \leq 2$	77.8 %
Questionable: $2 < Z < 3$	0.0 %
Unsatisfactory: $ Z \geq 3$	22.2 %

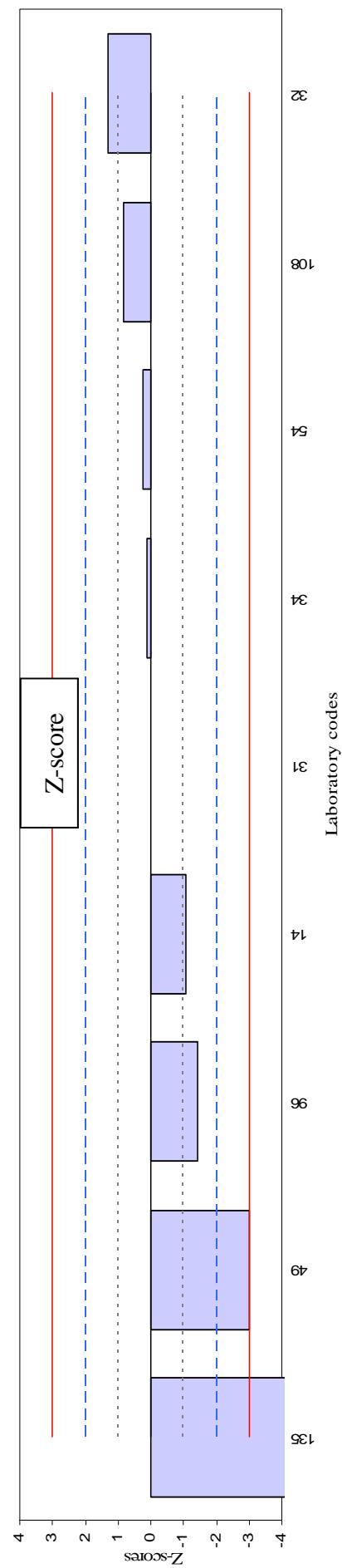
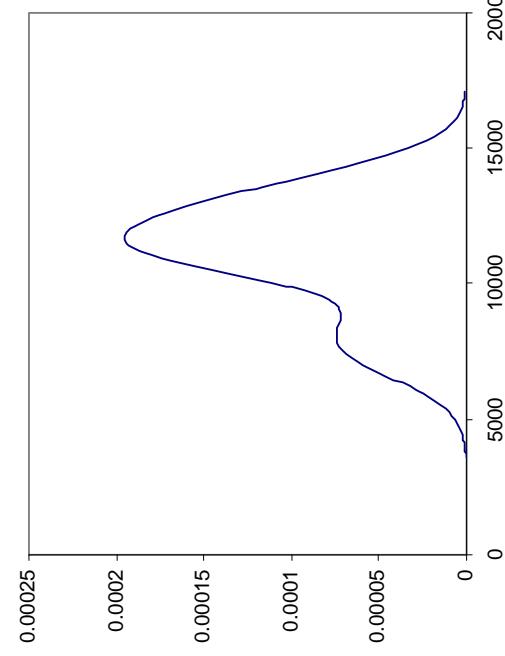


FIG. II.3. Performance evaluation of reported results for Br in the IAEA-452 sample.

Kernel Density Plot
Used hOpt = 1127.49674134481



Summary of results

Assigned value	11305 mg kg ⁻¹
Robust standard deviation	2230 mg kg ⁻¹
Target standard deviation (fitness for purpose)	1410 mg kg ⁻¹
Number of participants	12
Number of methods	4
Satisfactory: $ Z \leq 2$	75.0 %
Questionable: $2 < Z < 3$	16.7 %
Unsatisfactory: $ Z \geq 3$	8.3 %

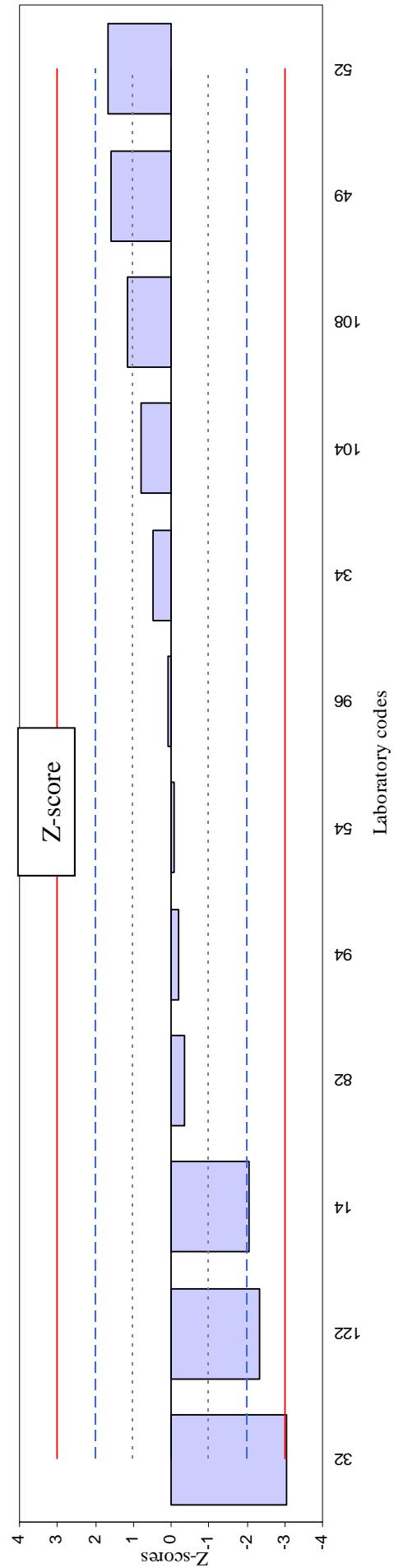
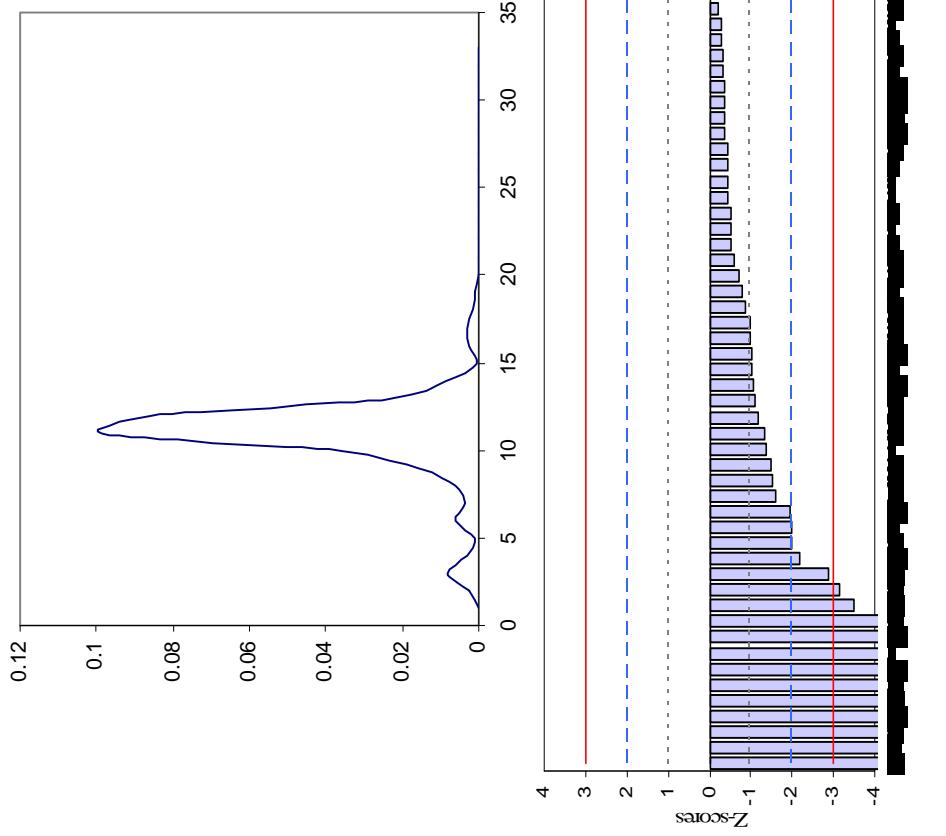


FIG. II.4. Performance evaluation of reported results for Ca in the IAEA-452 sample.

Kernel Density Plot
Used hOpt = 1.27407273814219

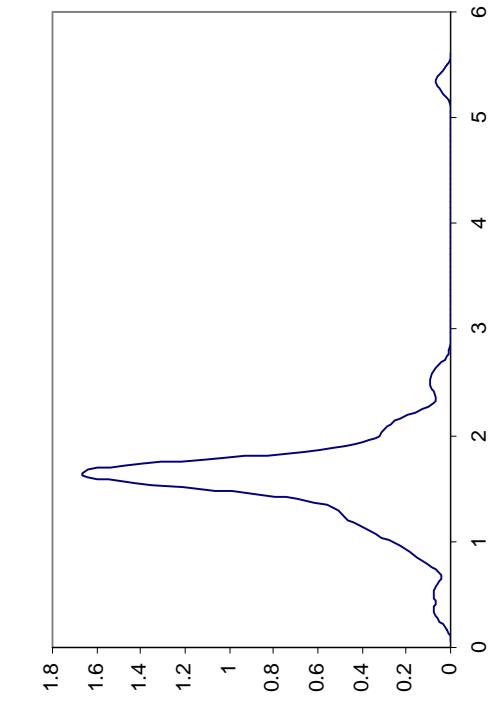


Summary of results

Assigned value	29.60 mg kg ⁻¹
Robust standard deviation	3.7 mg kg ⁻¹
Target standard deviation (fitness for purpose)	3.7 mg kg ⁻¹
Number of participants	121
Number of methods	7
Satisfactory: Z ≤ 2	81.8 %
Questionable: 2 < Z < 3	5.0 %
Unsatisfactory: Z ≥ 3	13.2 %

FIG. II.5. Performance evaluation of reported results for Cd in the IAEA-452 sample.

KernelDensity Plot
Used hOpt = 9.26791751161029E-02



Summary of results

Assigned value	1.62 mg kg⁻¹
Robust standard deviation	0.24 mg kg⁻¹
Target standard deviation (fitness for purpose)	0.20 mg kg⁻¹
Number of participants	65
Number of methods	6
Satisfactory: $ Z \leq 2$	75.4 %
Questionable: $2 < Z < 3$	13.8 %
Unsatisfactory: $ Z \geq 3$	10.8 %

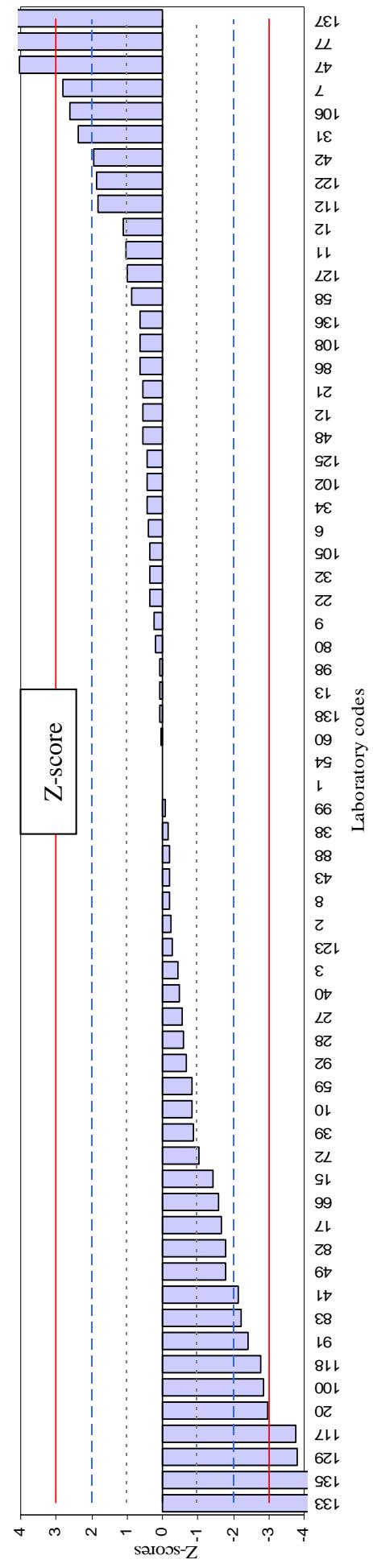


FIG. II.6. Performance evaluation of reported results for Co in the IAEA-452 sample.

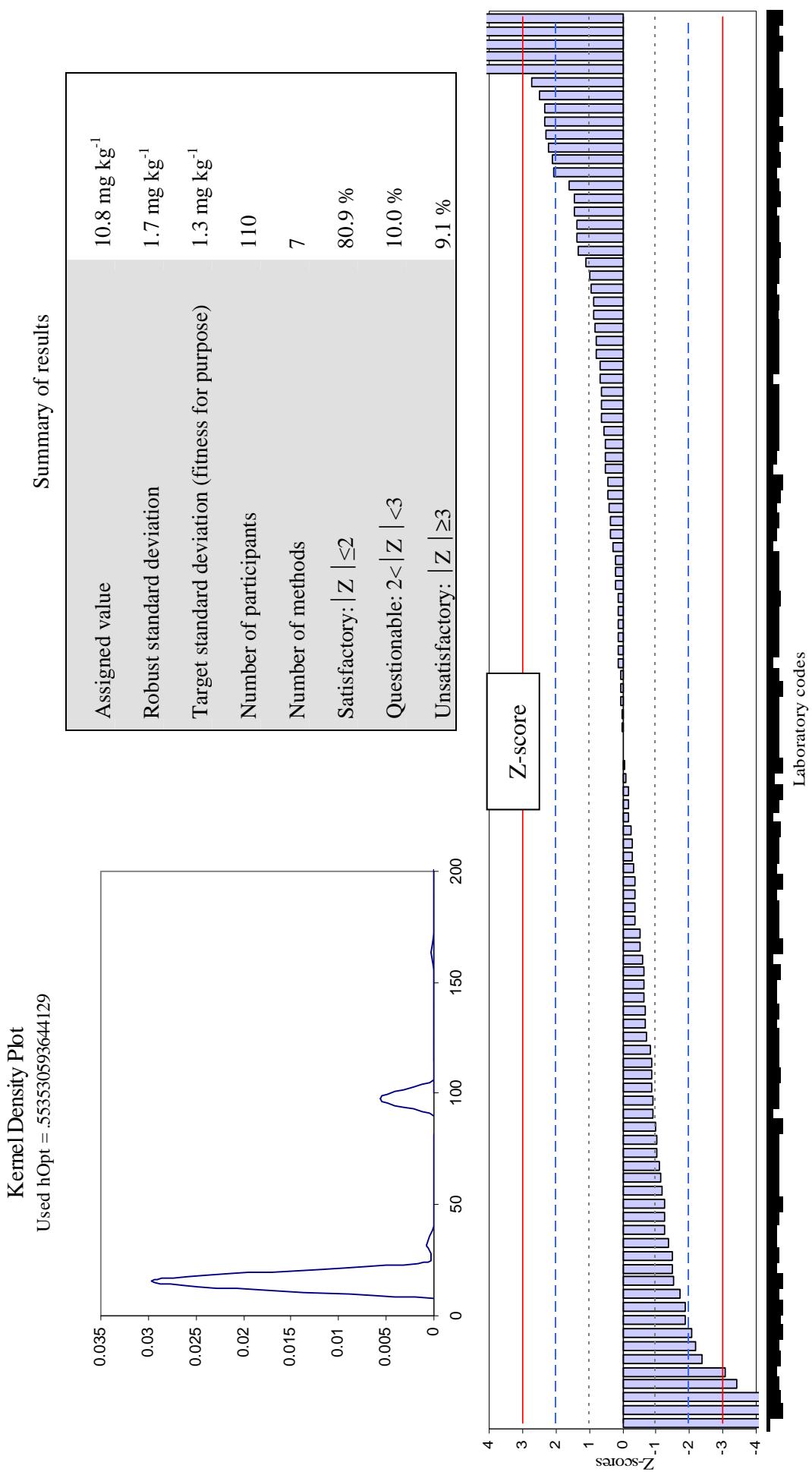
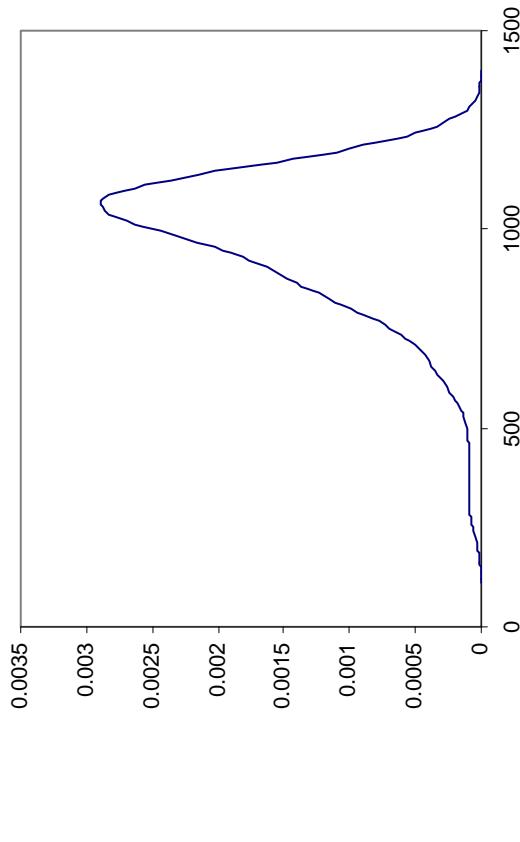


FIG. II.7. Performance evaluation of reported results for Cu in the IAEA-452 sample.

Kernel Density Plot
Used hOpt = 58.6598589865903



Summary of results

	Summary of results
Assigned value	1021 mg kg ⁻¹
Robust standard deviation	134 mg kg ⁻¹
Target standard deviation (fitness for purpose)	128 mg kg ⁻¹
Number of participants	92
Number of methods	6
Satisfactory: Z ≤ 2	89.1 %
Questionable: 2 < Z < 3	5.4 %
Unsatisfactory: Z ≥ 3	5.4 %

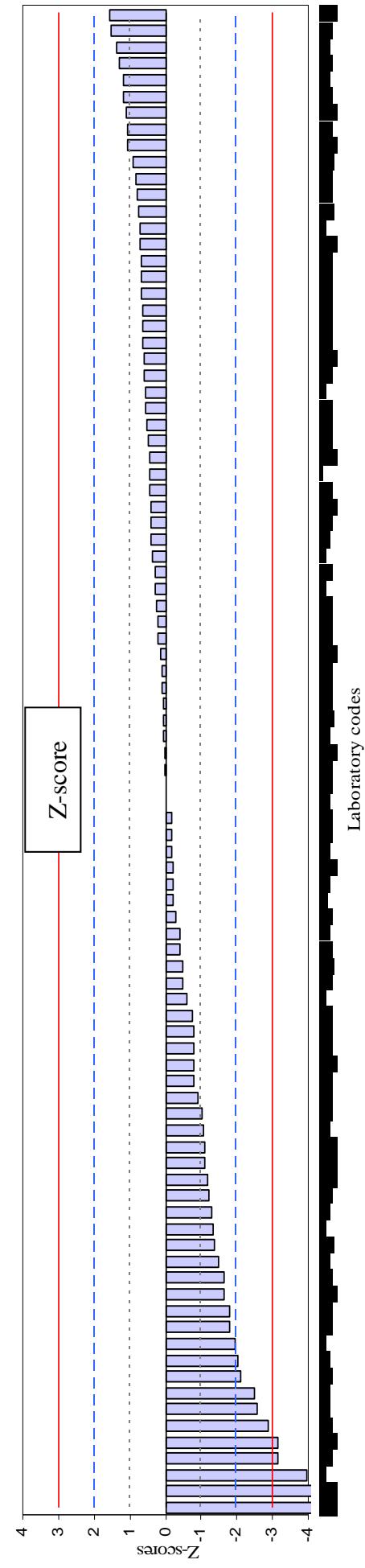
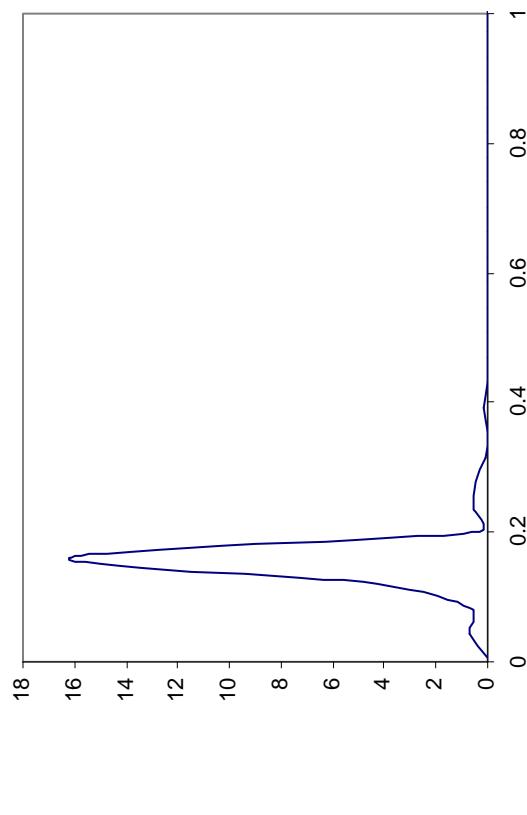


FIG. II.8. Performance evaluation of reported results for Fe in the IAEA-452 sample.

Kernel Density Plot
Used hOpt = 7.0316574065453E-03



Summary of results

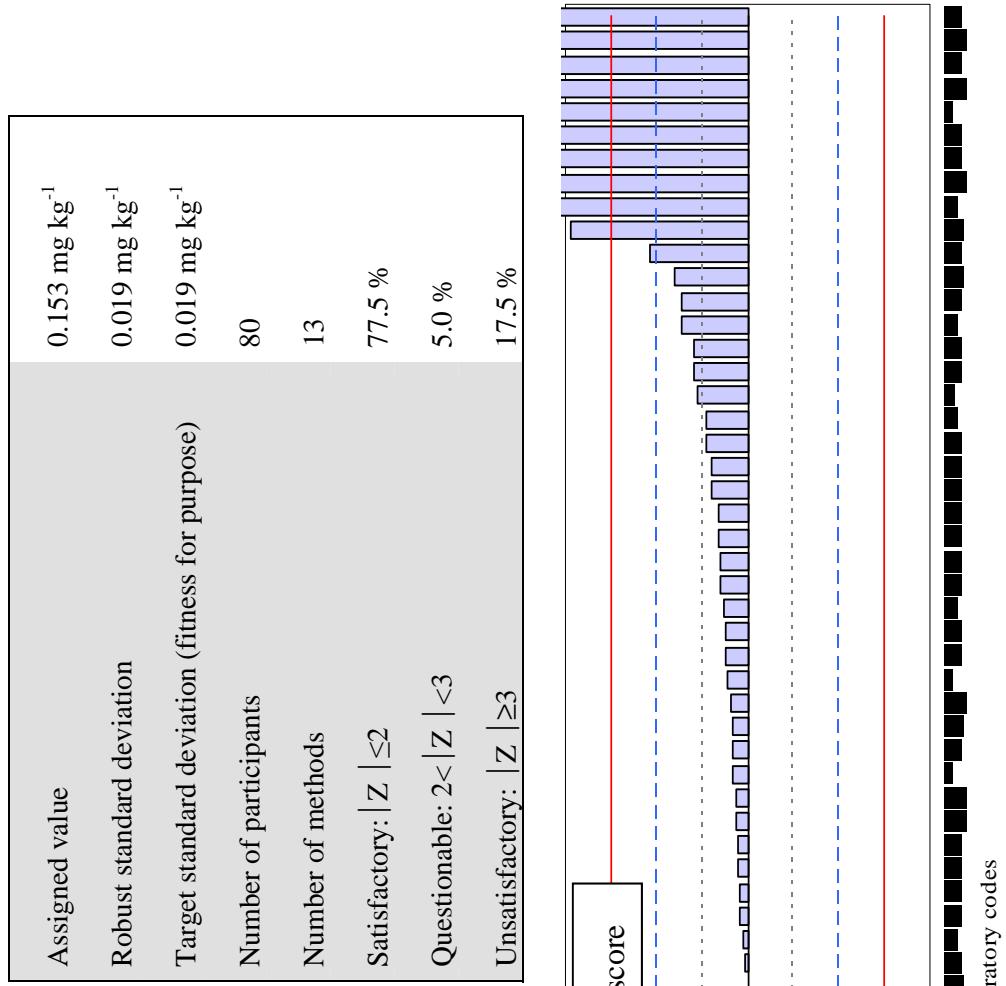
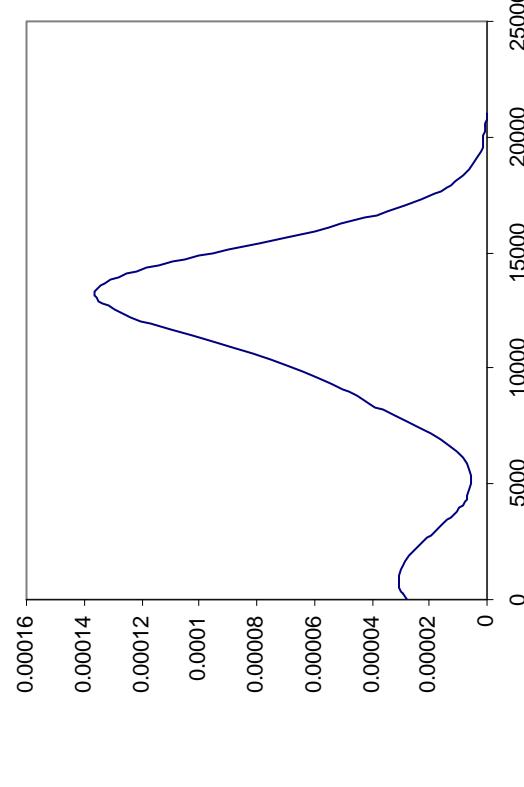


FIG. II.9. Performance evaluation of reported results for Hg in the IAEA-452 sample.

Kernel Density Plot
Used hOpt = 1769.84382978643



Summary of results

Assigned value	13140 mg kg^{-1}
Robust standard deviation	1160 mg kg^{-1}
Target standard deviation (fitness for purpose)	1640 mg kg^{-1}
Number of participants	13
Number of methods	4
Satisfactory: $ Z \leq 2$	69.2%
Questionable: $2 < Z < 3$	15.4 %
Unsatisfactory: $ Z \geq 3$	15.4 %

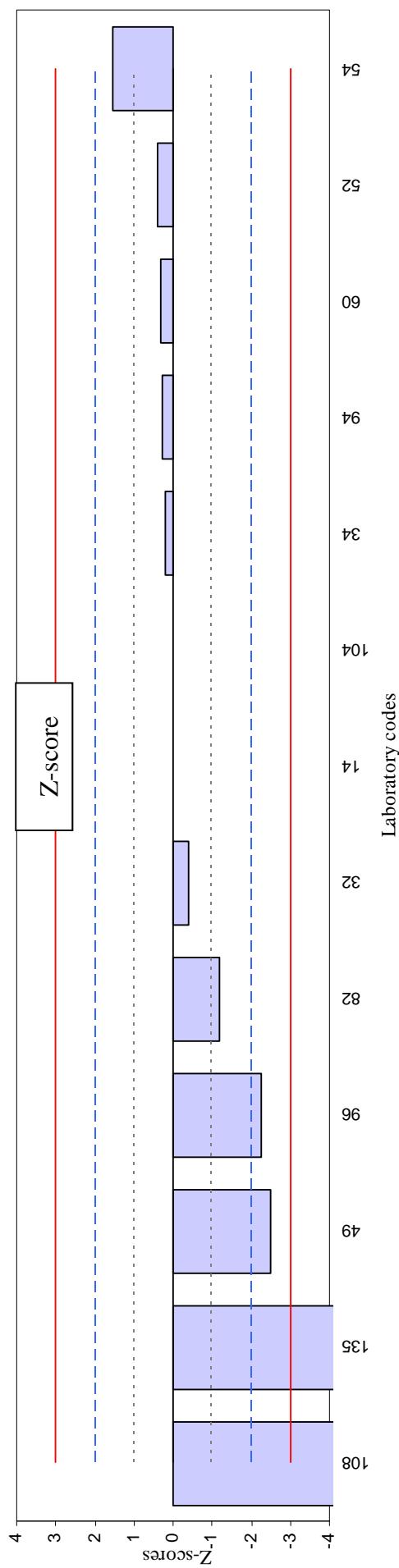
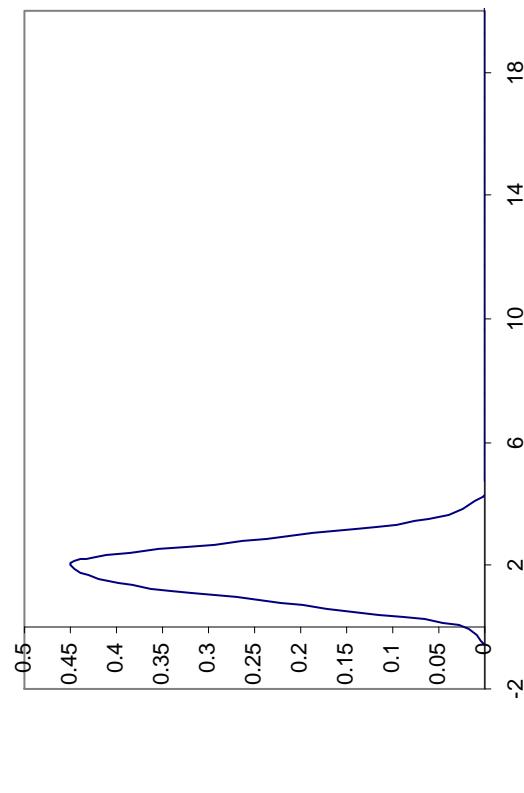


FIG. II.10. Performance evaluation of reported results for K in the IAEA-452 sample.

Kernel Density Plot
Used hOpt = .412562722413259



Summary of results

Assigned value	2.01 mg kg ⁻¹
Robust standard deviation	0.80 mg kg ⁻¹
Target standard deviation (fitness for purpose)	0.25 mg kg ⁻¹
Number of participants	23
Number of methods	4
Satisfactory: Z ≤ 2	43.5 %
Questionable: 2 < Z < 3	26.1 %
Unsatisfactory: Z ≥ 3	30.4 %

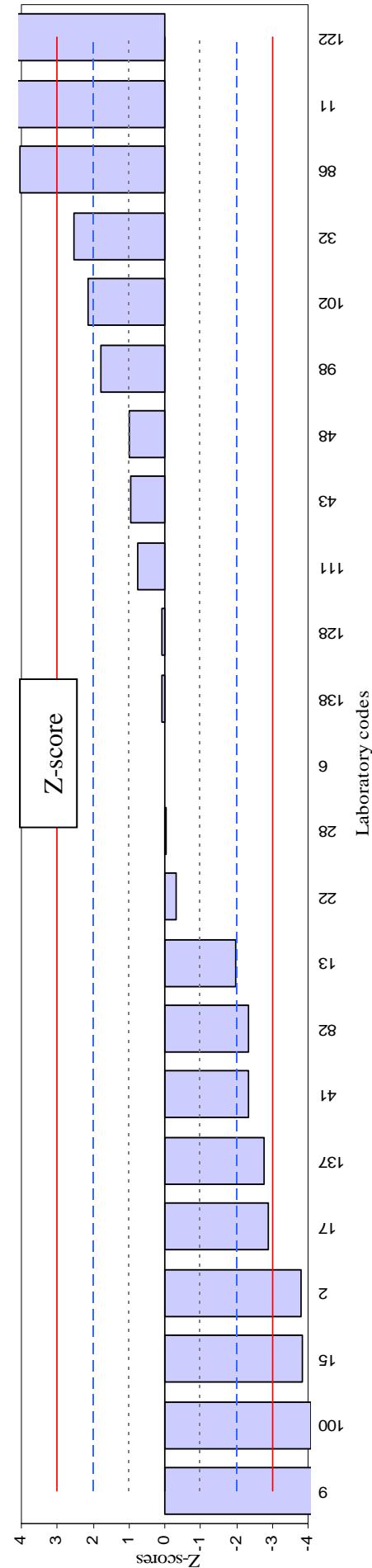
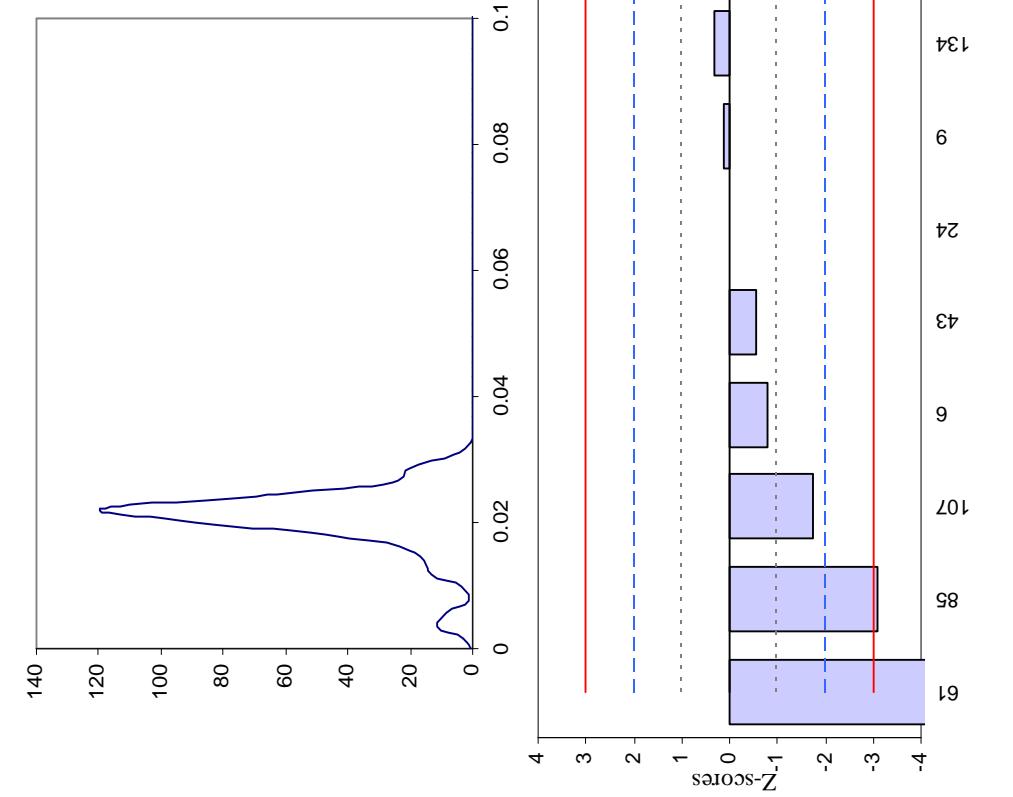


FIG. II.11. Performance evaluation of reported results for Li in the IAEA-452 sample.

Kernel Density Plot
Used hOpt = .001604800196658



Summary of results

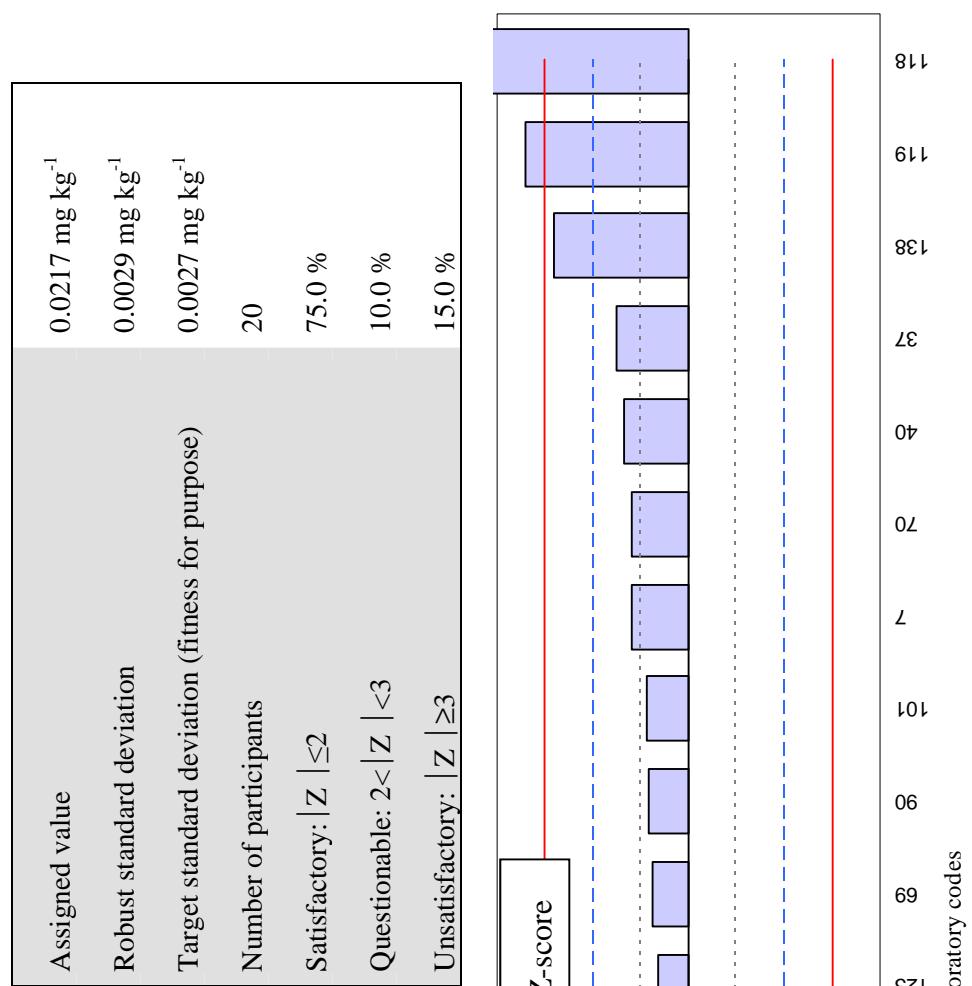
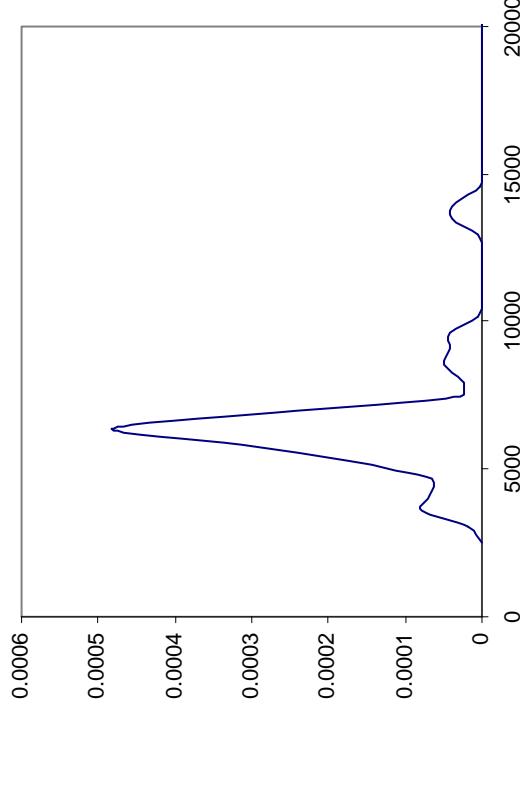


FIG. II.12. Performance evaluation of reported results for MeHg in the IAEA-452 sample.

Kernel Density Plot

Used hOpt = 373.373472005282



Summary of results

	Assigned value	Robust standard deviation	Target standard deviation (fitness for purpose)	Number of participants	Number of methods	Satisfactory: $ Z \leq 2$	Questionable: $2 < Z < 3$	Unsatisfactory: $ Z \geq 3$
	6380 mg kg^{-1}	840 mg kg^{-1}	797 mg kg^{-1}	22	5	72.7 %	9.1 %	18.2 %

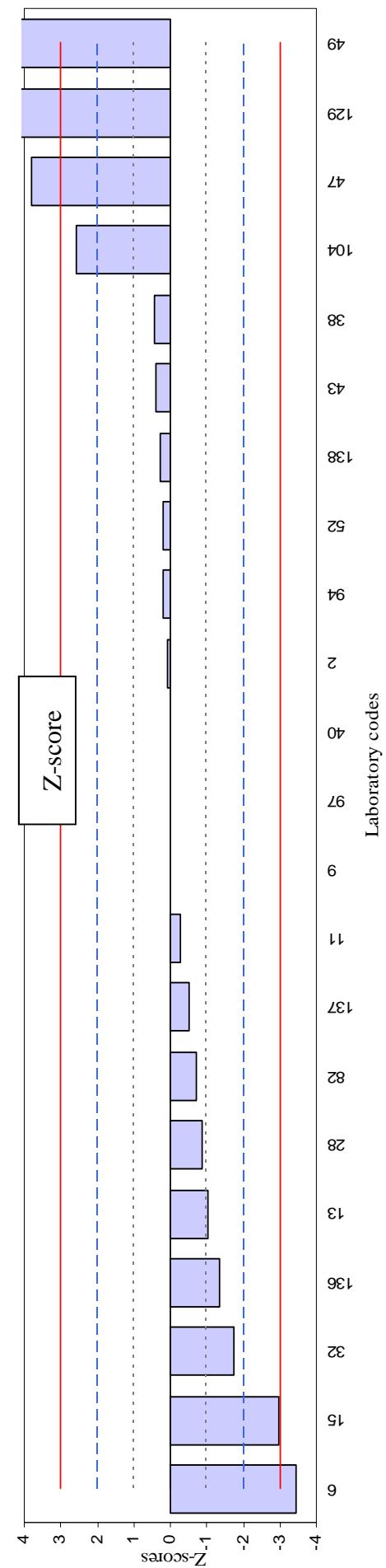
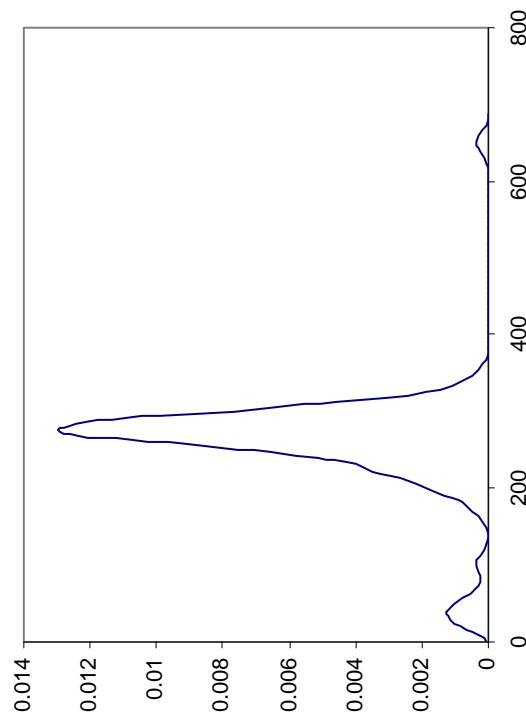


FIG. II.13. Performance evaluation of reported results for Mg in the IAEA-452 sample.

Kernel Density Plot
Used hOpt = 12.6763975989778



Summary of results

Assigned value	273 mg kg^{-1}
Robust standard deviation	30 mg kg^{-1}
Target standard deviation (fitness for purpose)	34 mg kg^{-1}
Number of participants	86
Number of methods	6
Satisfactory: $ Z \leq 2$	86.0 %
Questionable: $2 < Z < 3$	5.8 %
Unsatisfactory: $ Z \geq 3$	8.1 %

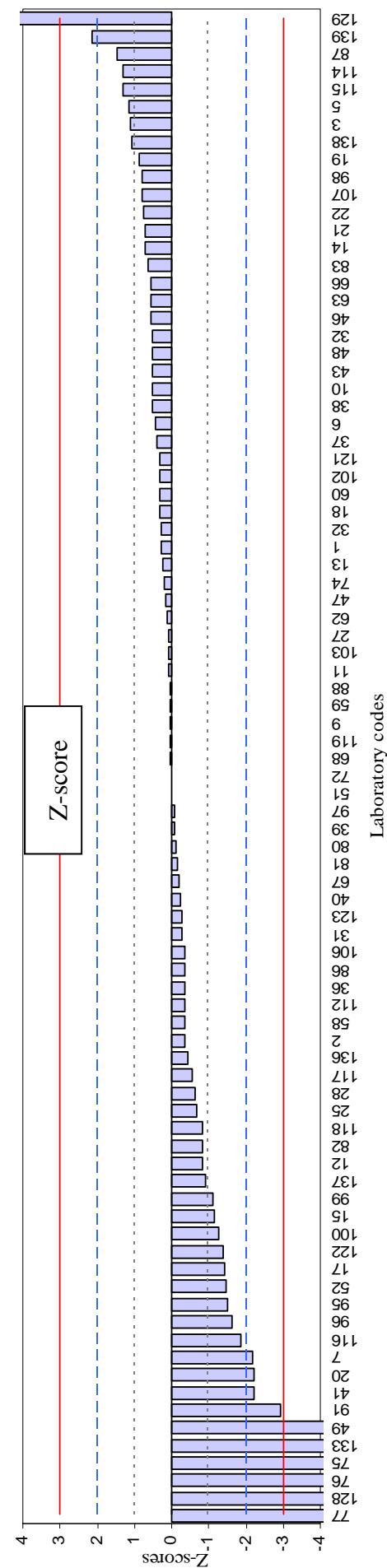
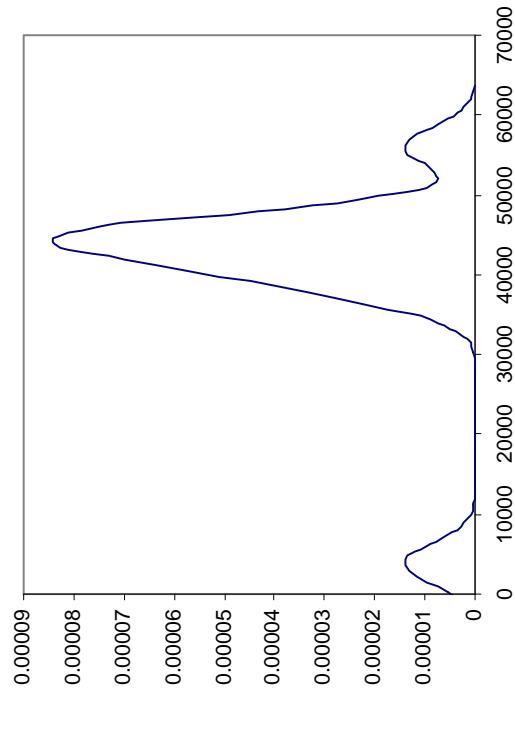


FIG. II.14. Performance evaluation of reported results for Mn in the IAEA-452 sample.

Kernel Density Plot

Used hOpt = 2571.57312275121



Summary of results

Assigned value	43959 mg kg^{-1}
Robust standard deviation	4132 mg kg^{-1}
Target standard deviation (fitness for purpose)	5994 mg kg^{-1}
Number of participants	11
Number of methods	3
Satisfactory: $ Z \leq 2$	81.8 %
Questionable: $2 < Z < 3$	9.1 %
Unsatisfactory: $ Z \geq 3$	9.1 %

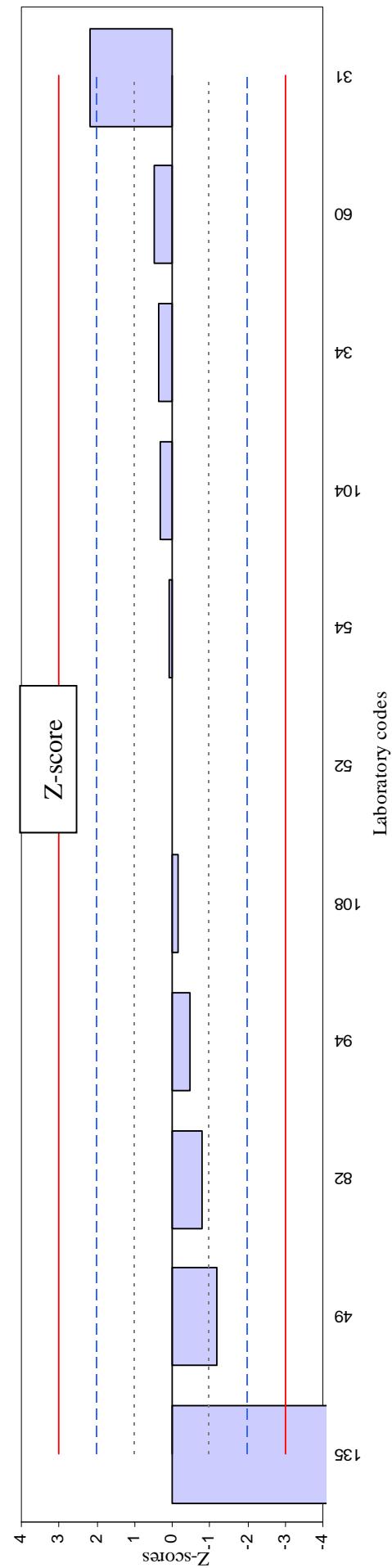
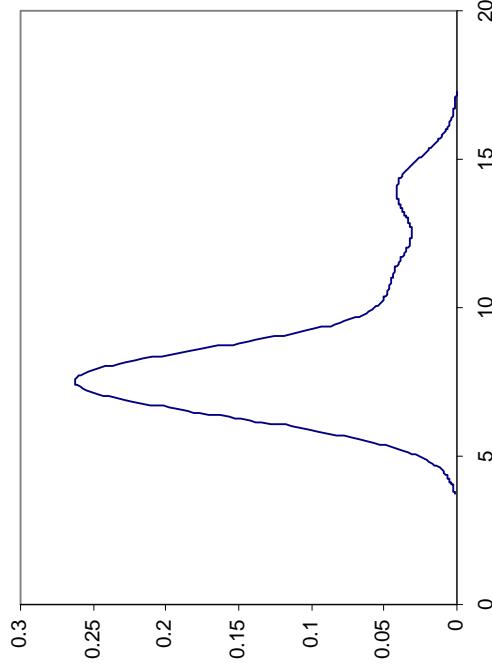


FIG. II.15. Performance evaluation of reported results for Na in the IAEA-452 sample.

Kernel Density Plot
Used hOpt = 1.08849690515001



Summary of results

Assigned value	7.85 mg kg^{-1}
Robust standard deviation	1.1 mg kg^{-1}
Target standard deviation (fitness for purpose)	0.98 mg kg^{-1}
Number of participants	9
Number of methods	3
Satisfactory: $ Z \leq 2$	77.8 %
Questionable: $2 < Z < 3$	0.0 %
Unsatisfactory: $ Z \geq 3$	22.2 %

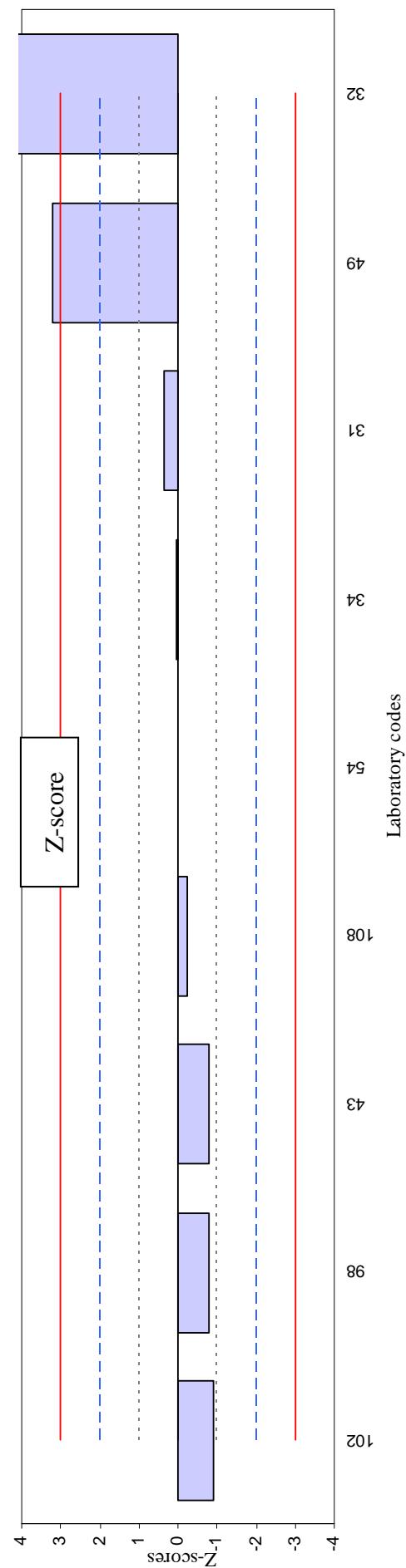


FIG. II.16. Performance evaluation of reported results for Rb in the IAEA-452 sample.

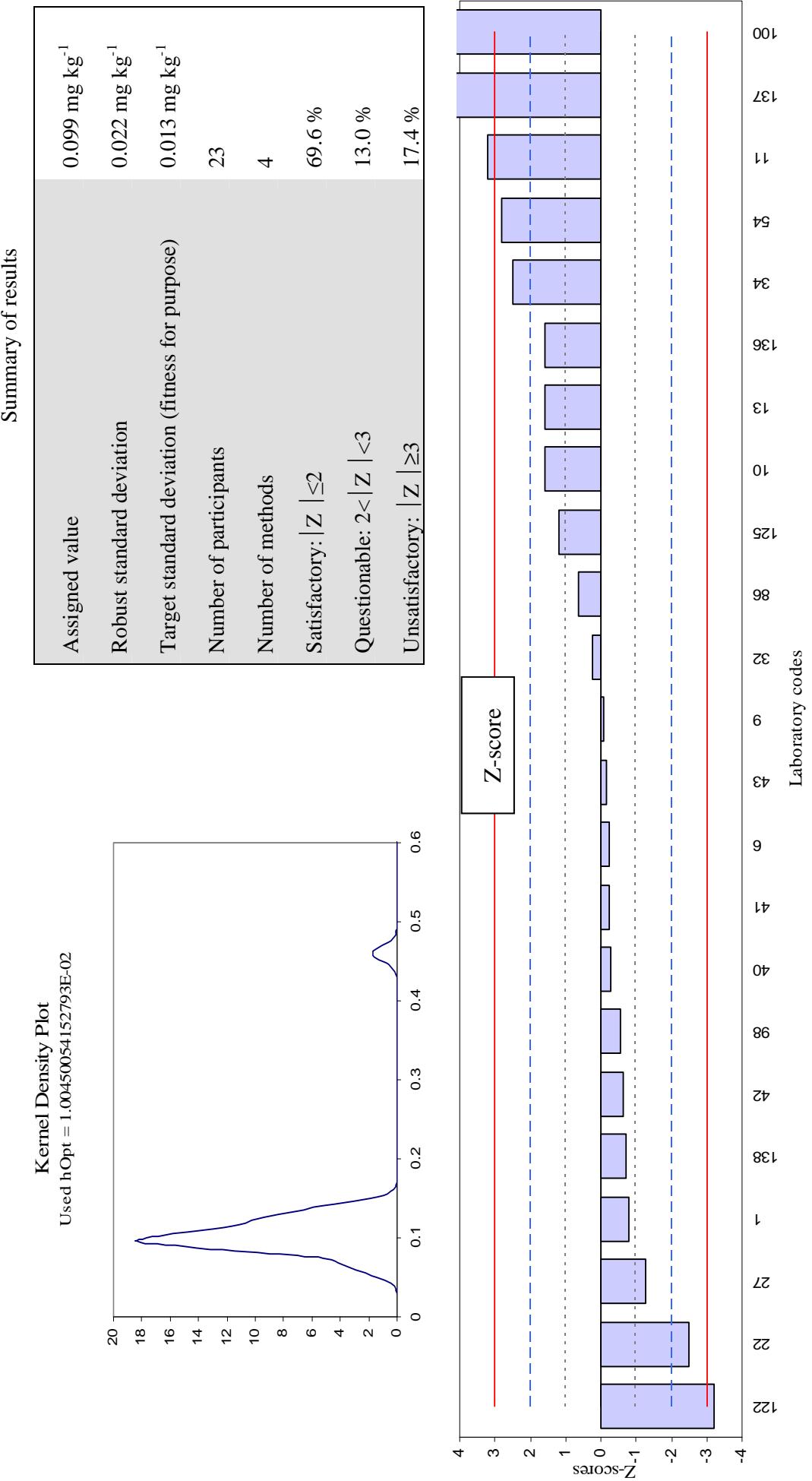


FIG. II.17. Performance evaluation of reported results for Sb in the IAEA-452 sample.

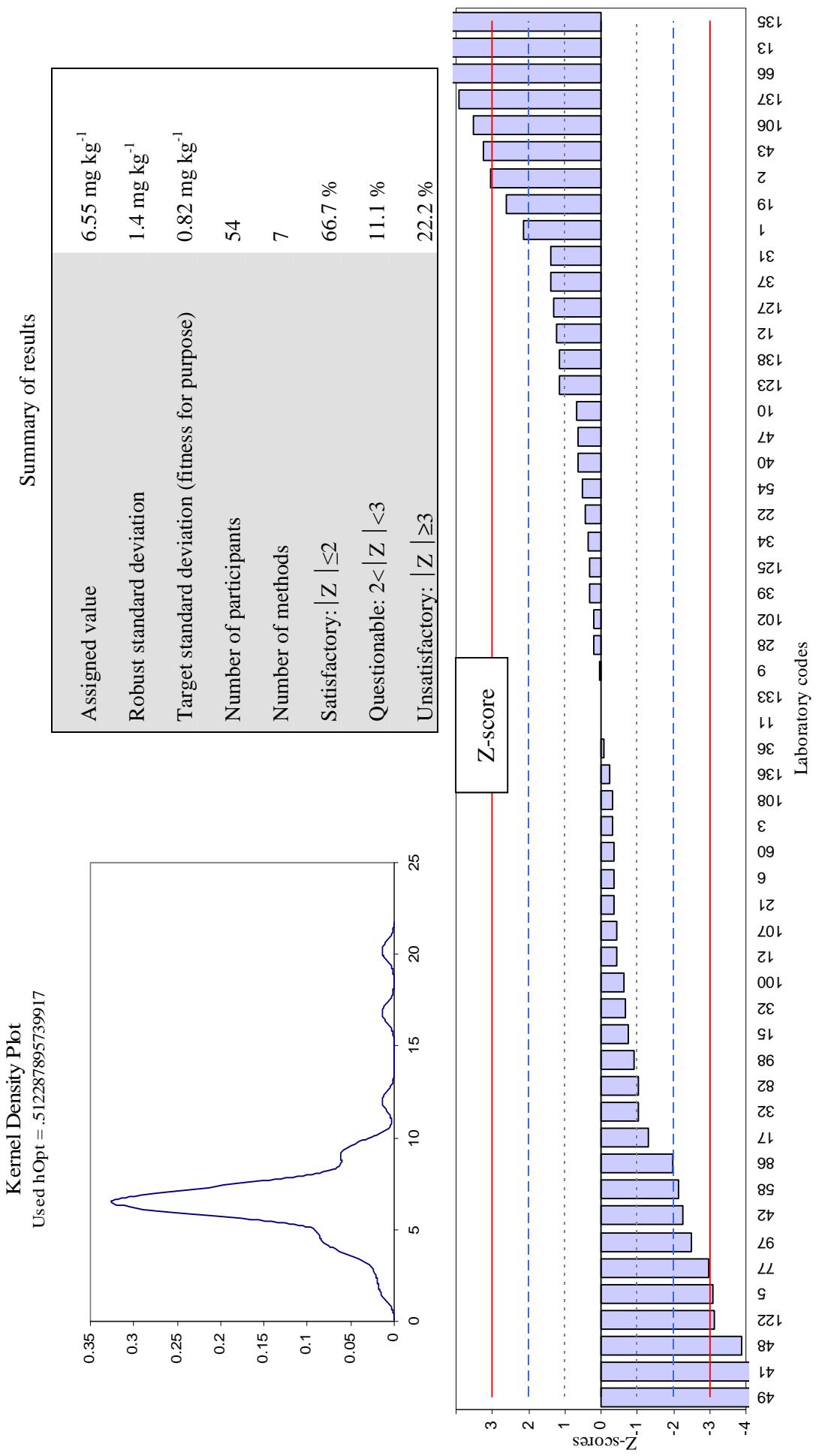


FIG. II.18. Performance evaluation of reported results for Se in the IAEA-452 sample.

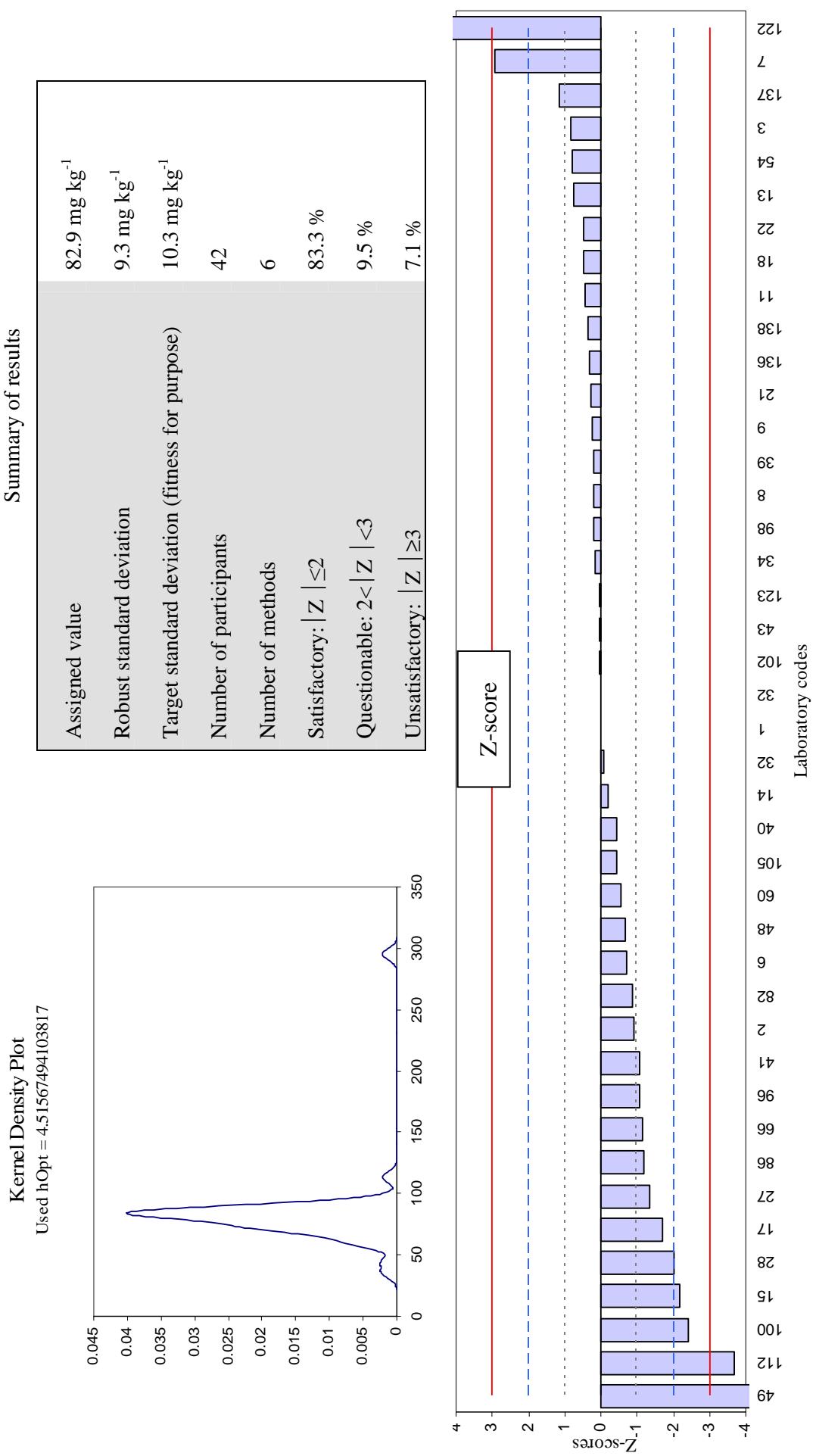
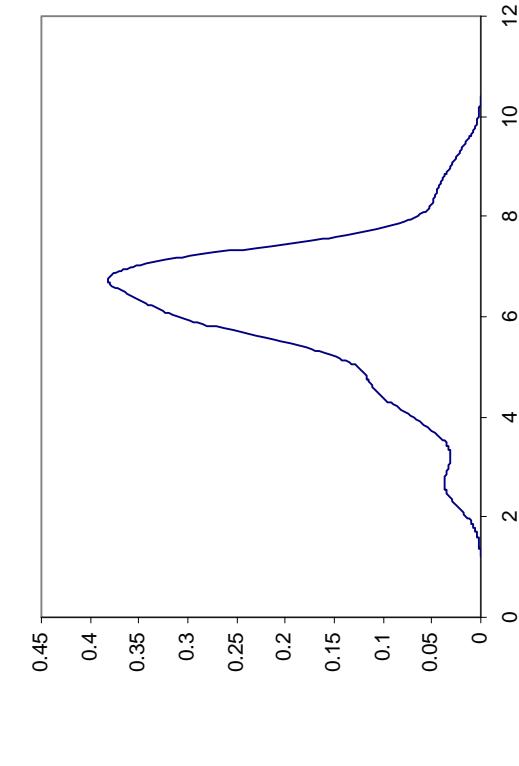


FIG. II.19. Performance evaluation of reported results for Sr in the IAEA-452 sample.

Kernel Density Plot
Used hOpt = .437399135250428



Summary of results

Assigned value	6.36 mg kg ⁻¹
Robust standard deviation	0.98 mg kg ⁻¹
Target standard deviation (fitness for purpose)	0.79 mg kg ⁻¹
Number of participants	48
Number of methods	6
Satisfactory: Z ≤ 2	79.2 %
Questionable: 2 < Z < 3	12.5 %
Unsatisfactory: Z ≥ 3	8.3 %

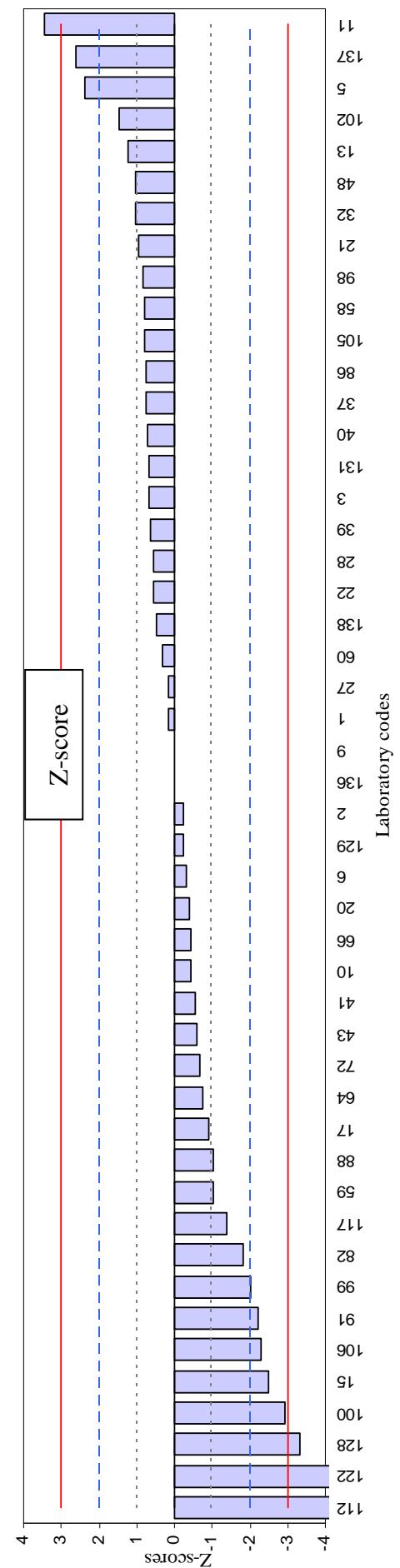
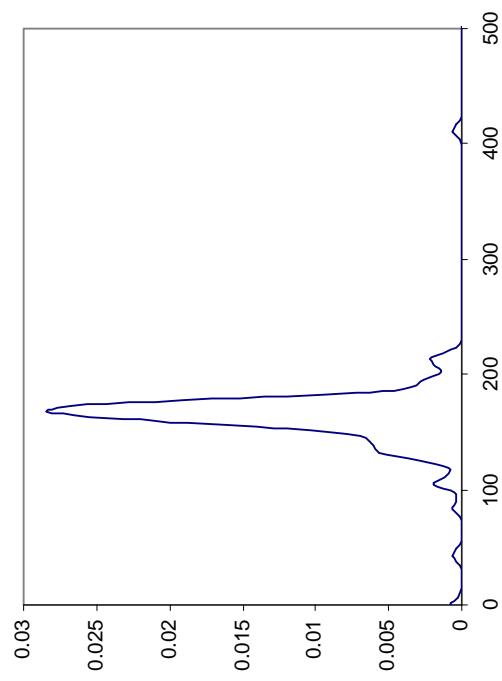


FIG. II.20. Performance evaluation of reported results for V in the IAEA-452 sample.

Kernel Density Plot
Used hOpt = 4.74267076402317



Summary of results

Assigned value	166 mg kg^{-1}
Robust standard deviation	14 mg kg^{-1}
Target standard deviation (fitness for purpose)	21 mg kg^{-1}
Number of participants	115
Number of methods	7
Satisfactory: $ Z \leq 2$	89.6 %
Questionable: $2 < Z < 3$	6.1 %
Unsatisfactory: $ Z \geq 3$	4.3 %

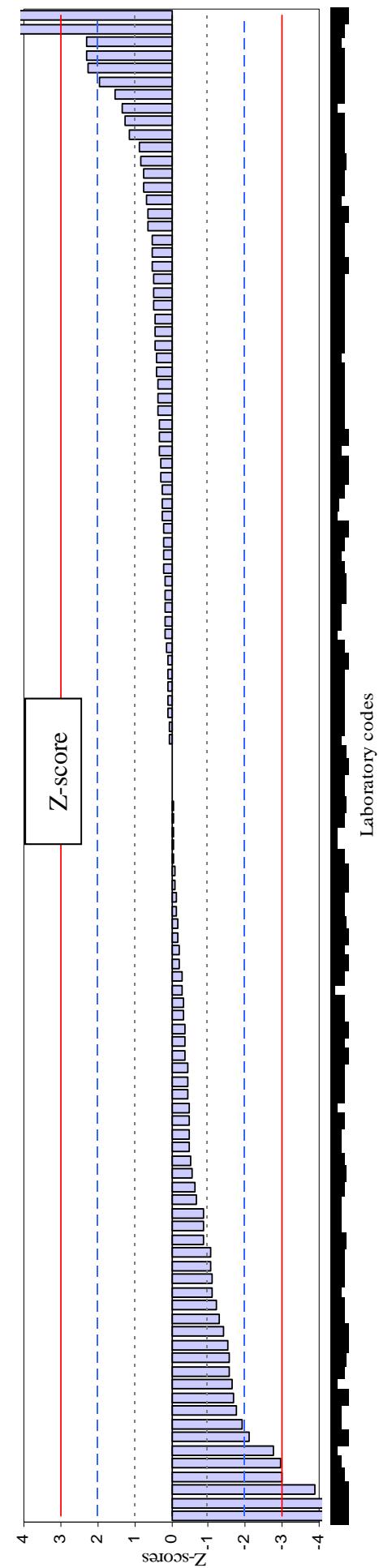


FIG. II.21. Performance evaluation of reported results for Zn in the IAEA-452 sample.

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