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Certification of Trace Element Mass Fractions in IAEA-457 Marine Sediment Sample

IAEA
International Atomic Energy Agency

CERTIFICATION OF TRACE ELEMENT MASS FRACTIONS IN IAEA-457 MARINE SEDIMENT SAMPLE

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IAEA Analytical Quality in Nuclear Applications No. IAEA/AQ/26

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FOREWORD

The primary goal of the IAEA Environment Laboratories in Monaco (NAEL) is to help Member States understand, monitor and protect the marine environment. The major impact exerted by large coastal cities on marine ecosystems is therefore of great concern to the IAEA and its Environment Laboratories. Given that marine pollution assessments of such impacts depend on accurate knowledge of contaminant concentrations in various environmental compartments, the NAEL has assisted national laboratories and regional laboratory networks through its Reference Products for Environment and Trade programme since the early 1970s.

Quality assurance (QA), quality control (QC) and associated good laboratory practice are essential components of all marine environmental monitoring studies. QC procedures are commonly based on the analysis of certified reference materials and reference samples in order to validate analytical methods used in monitoring studies and to assess reliability and comparability of measurement data. QA can be realized by participation in externally organized laboratory performance studies, also known as interlaboratory comparisons, which compare and evaluate analytical performance and measurement capabilities of participating laboratories. Data that are not based on adequate QA/QC can be erroneous and their misuse can lead to incorrect environmental management decisions.

A marine sediment sample with certified mass fractions for Ag, Al, As, Cd, Cr, Co, Cu, Fe, Hg, Li, Mn, Ni, Pb, Sn, Sr, V and Zn was recently produced by the NAEL in the frame of a project between the IAEA and the Korea Institute of Ocean Science and Technology.

This report describes the sample preparation methodology, the material homogeneity and stability study, the selection of laboratories, the evaluation of results from the certification campaign and the assignment of property values and their associated uncertainty. As a result, reference values for mass fractions and associated expanded uncertainty for 17 trace elements (Ag, Al, As, Cd, Cr, Co, Cu, Fe, Hg, Li, Mn, Ni, Pb, Sn, Sr, V and Zn) in marine sediment were established.

The IAEA is grateful to the participants and the laboratories who took part in this certification exercise and contributed their time and facilities to the present work. Special thanks are given to the Korea Institute of Ocean Science and Technology for providing the bulk sediment sample. The IAEA is also grateful to the Government of Monaco for its support. The IAEA officers responsible for this publication were E. Vasileva and S. Azemard of the IAEA Environment Laboratories in Monaco.

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CONTENTS

1. INTRODUCTION 1
2. METHODOLOGY 2
    2.1. Collection and preparation of the material 2
    2.2. Selection of laboratories for the certification campaign 2
    2.3. Homogeneity testing 3
    2.4. Stability study 3
    2.5. Characterization 3
    2.6. Moisture determination 5
3. RESULTS AND DISCUSSION 5
    3.1. Results of the homogeneity study 5
    3.2. Results of the stability study 7
    3.3. Determination of certified values and uncertainties 9
4. METROLOGICAL TRACEABILITY 19
5. CONCLUSIONS 20

APPENDIX I: DATA REPORT OF RESULTS SORTED BY ELEMENTS 21
APPENDIX II: 29
REFERENCES 32

1. INTRODUCTION

Society's growing interest in environmental issues requires the production of reliable information for policy makers, stakeholders and public in general. This information must be based on accurate and comparable results produced by qualified laboratories. National and international marine monitoring programs have been initiated worldwide to assess the quality of the marine environment. In monitoring program it is considered essential to ensure that the data produced from different laboratories over a number of years can be compared. If results are to be comparable, it is essential that they are based on reliable measurement standards whose values are linked to a stated reference.

The Marine Environmental Studies Laboratory (MESL) of the NAEL has the programmatic responsibility to provide assistance to Member States' laboratories in maintaining and improving the reliability of analytical measurement results, both in trace elements and organic pollutants. This is accomplished through the provision of Certified Reference Materials (CRMs) 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. IAEA's Analytical Quality Control Service (AQCS), now named Reference Products for Environment and Trade represents an important benchmark in upgrading the quality of laboratory performances and assessing the validity of the analytical methods used for marine monitoring studies in the Member States.

Many laboratories around the world are providing monitoring data on trace elements in a variety of marine matrixes such as water, suspended matter, sediments and biota. These laboratories may develop and validate new analytical methods, study the environmental impact of human activities, provide services to other organizations, etc. In all cases scientific conclusions must be based on valid and internationally comparable data in order to provide policy-makers with correct information for their decisions on the state of the environment.

Laboratories need to be able to check the performance of their methods for the determination of trace elements in difficult matrices such as marine sediments. This is also true for standardized methods, the use of which does not guarantee accurate results. It is widely accepted that laboratories need to demonstrate their proficiency in the applicability of standard methods, for example, by using certified reference materials (CRMs).

While there are several CRMs certified for trace elements, the lack of matrix CRMs is still remaining.

The work presented in this report refers exclusively to the certification of the total content of trace elements in marine sediment. This material is a certified reference material (CRM), released in September 2012 by the IAEA.

2. METHODOLOGY

2.1. COLLECTION AND PREPARATION OF THE MATERIAL

A sample of forty four kg of sediment was delivered to the NAEL by the Korean Ocean Research and Development Institute. The freeze-dried material was milled to a powder in a grinder Retsch SM 200 (Retsch, Haan, Germany). The powder was then sieved through a set of sieves (Fritsch, Idar-Oberstein) and the fraction of 26 µm was collected. The sieved material with a particle size of less than 26 µm was further homogenized. The homogeneity was performed by mixing the material in a stainless steel rotating homogenizer Moritz ERM-BB124 (Moritz, Chatou, France) for 14 days at a temperature of 20°C (+/-2°), and relative humidity of 50%. After checking for the homogeneity of the sample material, aliquots of 30 g were packed into pre-cleaned brown borosilicate glass bottles with polyethylene screw caps and then sealed in plastic bags. The sample material was labeled as ***IAEA-457***. The average moisture content of the sample after bottling was determined by drying to a constant weight at 105°C.

2.2. SELECTION OF LABORATORIES FOR THE CERTIFICATION CAMPAIGN

The selection of participants for this certification exercise was based on the measurement performances, demonstrated by laboratories in the previous IAEA certification campaigns and interlaboratory comparisons on marine sediments. Only results from laboratories having a quality system in place, using validated methods, applying uncertainty and traceability concepts and having provided good results in the previous IAEA ILCs were accepted for the calculation of the assigned values and their uncertainties.

Each laboratory received one bottle of sediment sample, accompanied by an information sheet and a reporting form. The participants were requested to analyze Ag, Al, As, Cd, Cr, Co, Cu, Fe, Hg, Li, Mn, Ni, Pb, Sn, Sr, V and Zn, using a validated analytical method. They were asked to report the measurement results (six replicates and average value) along with the expanded uncertainty in addition to the information about the applied quality control procedure. The second request was to report results for the trace elements mass fraction in a certified reference material with a similar matrix to the candidate reference material. The moisture determination method was also prescribed.

The list of laboratories participating in the certification exercise is presented in the list below.

2.3. HOMOGENEITY TESTING

Extensive homogeneity tests were carried out on this material in order to ensure its suitability as a proficiency test reference sample and, to estimate the uncertainty associated with the homogeneity of the sample. The between-bottle homogeneity was tested by the determination of the mass fraction of some typical elements (Al, Cr, Cu, Fe, Hg, Mn, Ni, Pb, and Zn). In total, 10 bottles were selected using random stratified sampling of the whole batch. Care was taken to ensure that the order of measurements did not correspond to the filling sequence of the bottles, which enables the differentiation between potential trend in the filling sequence and analytical drift. Three subsamples from each bottle were analyzed for their total element mass fractions. The within-bottle homogeneity was assessed by 15 replicate determinations of the content of investigated trace elements in one bottle. Subsamples of 0.2 g were mineralized with 5 ml conc. HNO_3 and digested in a microwave oven by adding 2 ml conc. HF according to the protocol described earlier [1]. The final measurements were performed by flame and graphite furnace atomic absorption spectrometry under repeatability conditions, and in a randomized way, in order to be able to separate a potential analytical drift from a trend in the filling sequence. The determination of the total mercury was done in solid subsamples with solid mercury analyzer. All the methods used for the homogeneity studies were previously validated by MESL, IAEA.

2.4. STABILITY STUDY

Three sets of five bottles each were stored in the dark at different temperatures: -20°C, +20°C and +60°C, just after the bottling process and kept at the described conditions over a period of 2 years. One isochronous study over 6 weeks was applied in order to evaluate the short-term stability of the materials during transport and one isochronous study over 9 months to evaluate the stability during storage. The obtained results were compared with the results from samples kept at -20°C during this period (-20°C is considered as the reference temperature). The stability investigation for the evaluation of long-term stability is still on-going.

2.5. CHARACTERIZATION

Characterization refers to the process of determining the reference values. The material was initially analyzed at the NAEL. The final characterization was based on the results delivered by selected laboratories with demonstrated measurement capabilities, based on criteria that comprised both technical and quality management aspects. The characterization of the trace element mass fraction in the sediment sample was based on the application of different analytical techniques as summarized in Figure 1.

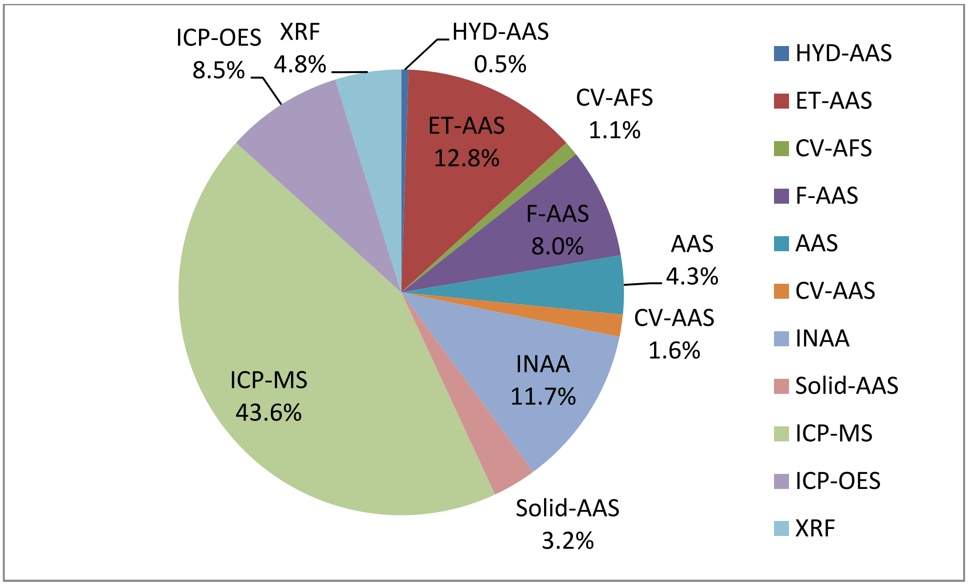

FIG. 1. Analytical methods used for the certification of trace elements in the IAEA-457 sediment sample.

TABLE 1. INSTRUMENTAL TECHNIQUES

Method code	**Instrumental technique**
AAS	Atomic Absorption Spectrometry-Flame
ICP-MS	Inductively Coupled Plasma-Mass Spectrometry
ICP-OES	Inductively Coupled Plasma-Optical Emission Spectrometry
AFS	Atomic Fluorescence Spectrometry
CV-AAS	Cold Vapour-Atomic Absorption Spectrometry
ET AAS	Atomic Absorption Spectrometry-Graphite furnace
AAS-HYD	Atomic Absorption Spectrometry-Hydride Generation
INAA	Neutron Activation Analysis
CV-AFS	Cold Vapour-Atomic Fluorescence Spectrometry
XRF	X-Ray Fluorescence

All the participating laboratories have used validated methods for the determination of trace elements in marine samples. In addition, they provided results from the analyzed CRM with a similar matrix composition, and the information on standard calibration solutions used for every trace metal. The results from the laboratories that did not report any quality assurance information were excluded from further evaluation.

Combined uncertainties were calculated in compliance with the JGCM 100:2008 Evaluation of measurement data - Guide to the expression of uncertainty in measurement (GUM with minor corrections) [2], including uncertainties due to possible heterogeneity and instability.

All participating laboratories claimed traceability of provided results to the International System of Units (SI) via standard calibration solutions and CRMs applied as a part of their analytical procedures.

2.6. MOISTURE DETERMINATION

The determination of the moisture content of the samples is to some extent "operationally defined". In view of the comparability of results, the protocol for the correction of the moisture was developed at the NAEL and prescribed to other participants. The drying procedure at 105°C (+/-2°) was established after experimental evaluation of the sample stability. Correction for dry-mass was obtained from separate portions of the material of a minimum mass of 0.5 g (10 sub samples from 5 bottles). Weighing and repeated drying were performed until constant mass was attained. The moisture determined at 105°C was found to be 2.5% ±0.5 for bottles kept at 20°C.

3. RESULTS AND DISCUSSION

3.1. RESULTS OF THE HOMOGENEITY STUDY

For the homogeneity study, 10 samples (about 2% of the total batch) of sediment were chosen using a random stratified sample picking scheme and analyzed for their trace elements contents in triplicate. The results were combined and evaluated to detect any trends regarding filling or analysis sequence, and to estimate the uncertainty contribution from the possible heterogeneity. Grubbs-tests were performed to identify potentially outlying individual results as well as outlying bottles means. One individual result for Ni, Pb and Zn respectively was detected as outlier. These results were excluded as they were outliers not only at 95% but also at 99% confidence level.

The retained individual results and bottle means were checked whether they follow a normal distribution or are unimodally distributed. The series of results for investigated trace elements were normally distributed. One way analysis of variance ANOVA [3] was then applied to assess between-bottles and within-bottle homogeneities. ANOVA allows the calculation of within unit standard deviation s_{wb} and also between-bottles standard deviation s_{bb}

$$s_{wb} = u_{wb} = \sqrt{MS_{wb}} \tag{1}$$

$$s_{bb} = u_{bb} = \sqrt{\frac{MS_{bb} - MS_{wb}}{n}} \tag{2}$$

For all elements, except for Pb and Cr, MS_{bb} (ANOVA mean square between-bottles) was smaller than MS_{wb} (ANOVA mean square within-bottle) and $S_{bb.}$ could not be calculated. Instead, u^*_{bb} - the heterogeneity that can be hidden by the method repeatability - was calculated, as described by Linsinger et *al*. [4]

$$u^*_{bb} = \frac{s_{wb}}{\sqrt{n}} \sqrt[4]{\frac{2}{v_{MSwb}}} \tag{3}$$

Where:

n is the number of replicate sub-samples per bottle; and

v_{MSwb} is the degrees of freedom of MS_{wb}.

The heterogeneity could be quantified thanks to the good repeatability of the method used. The between-bottles variations/heterogeneity were between 1.3 and 3.3%, small enough to ensure the homogeneity of the material. The uncertainty contributions due to the inhomogeneity were estimated according to ISO Guide 35 [3] as the maximum values obtained with Equation 2 or Equation 3. The results for sample size 0.2 g are presented in Table 2.

TABLE 2. ESTIMATE OF INHOMOGENEITY CONTRIBUTIONS TO THE TOTAL UNCERTAINTY FOR THE CERTIFIED TRACE ELEMENTS

Element	Al	Fe	Cu	Mn	Zn	Pb	Cr	Ni	Hg
u_{wb}%	1.3	2.5	3.3	1.2	1.2	3.2	1.3	1.7	1.4
u_{bb}%						1.5	1.4		
u^*_{bb}%	0.92	1.08	1.06	1.25	1.08			1.6	1.5

The conclusion from the presented results for the tested trace elements was that the homogeneity of the candidate reference material complied with the provisions given by the

ISO Guide 35 at the range of weights used. A minimum sample size of 0.2 g was set, based on the smallest sample size used in the characterization study.

3.2. RESULTS OF THE STABILITY STUDY

The samples selected for the stability study were analyzed and each of the elements was evaluated individually. No outliers were detected at 95% confidence level in any study. The evaluation of the data was carried out further by performing a linear regression on the determined mass fractions versus time.

The test material showed no significant trend of degradation over the timeframe at different temperatures: -20°C, +20°C and +60°C. No significant impact of storage conditions could be detected on the stability of the certified properties, neither due to storage time nor to temperatures (up to +60°C). In any cases, the slope of the linear regression did not significantly differ from zero. No significant slope at 95% level of confidence was detected for any of the investigated analytes in the short-term study. As no degradation could be observed under any conditions either, neither in the short-term nor in the long-term study, it was concluded that no special precautions regarding temperature control during shipment were necessary. The uncertainty of the short-term stability (u_{sts}) was assumed to be negligible since no degradation was expected during this short time.

Failure to detect degradation, however, does not prove stability. The uncertainty of stability u_{stab} describes the potential degradation which still can be reconciled with the data, even if the slope is not statistically and significantly different from zero. Although under these conditions an expansion of the total uncertainty of the certified values is generally not encouraged, in this case the approach of ISO Guide 35 [3] was followed, mainly due to the lack of sound alternatives. An uncertainty contribution related to the stability of the candidate reference material was estimated as uncertainty of the regression line with a slope of 0 multiplied with the chosen shelf-life, as described by Linsinger et *al.* [4]. A factor of 3 was selected, taking into account the minimum shelf-life of 3 years. The stability during the storage period was chosen as 1%, which ensured the validity of the certificate for 10 years. The results obtained from the short-term studies provide evidence to a good stability of all analytes considered.

Figures 2 and 3 represent the results on short-term stability studies (6 weeks) for Cd and Hg obtained with isochronous approach.

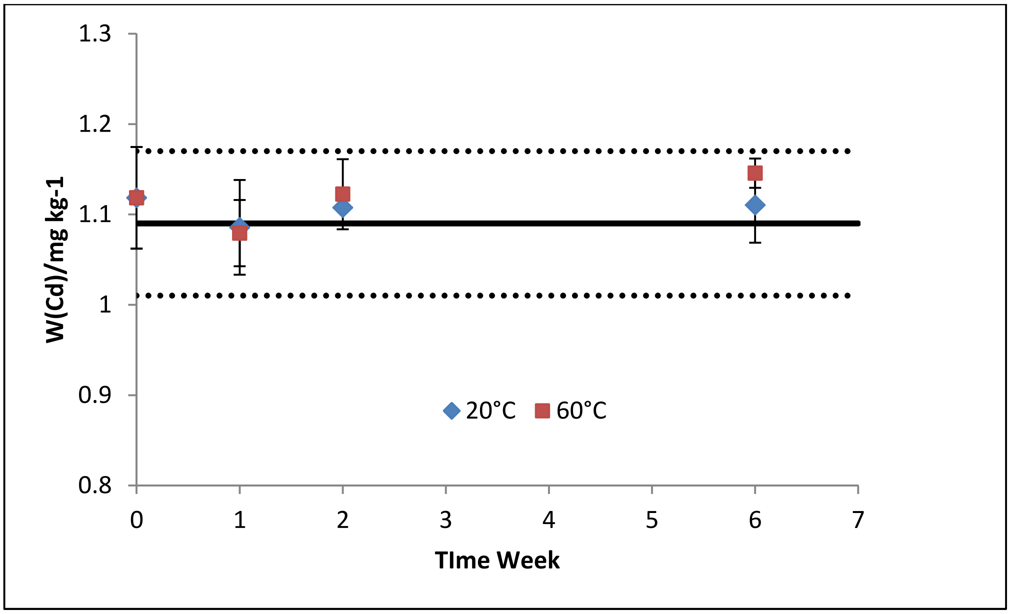

FIG. 2. Results obtained with isochronous approach for the short-term stability studies of Cd in the sediment sample kept at 20°C and 60°C respectively.

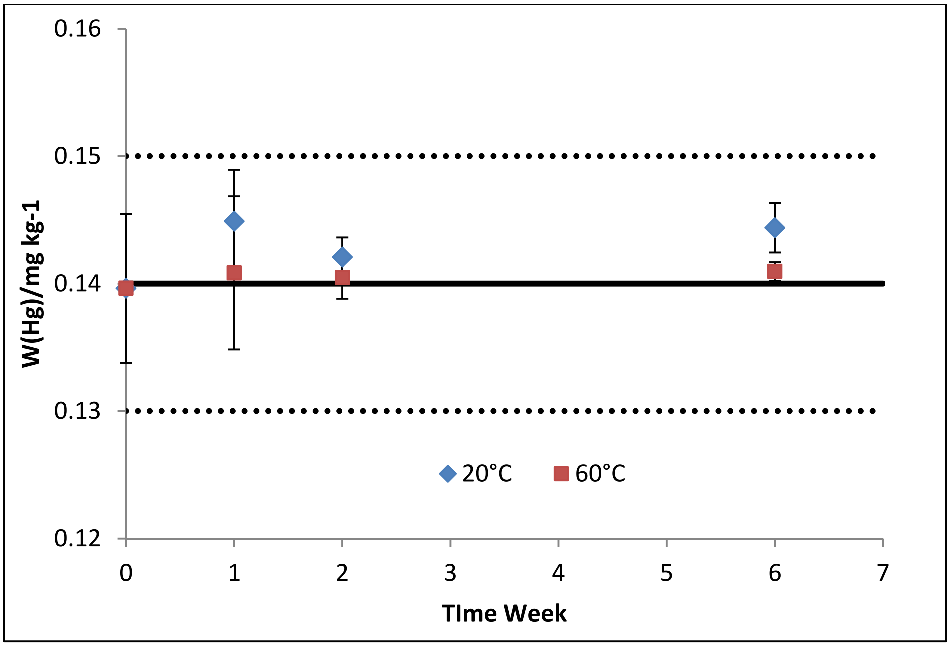

FIG. 3. Results obtained with isochronous approach for the short-term stability studies of Hg in the sediment sample kept at 20°C and 60°C respectively.

3.3. DETERMINATION OF CERTIFIED VALUES AND UNCERTAINTIES

The characterization campaign resulted in 6-18 results per element and 5 results for Li and Sn. The obtained data were first checked for compliance with the certification requirements, and then for their validity based on technical reasoning. All accepted sets of results were submitted to the following statistical tests: Grubbs test to detect single and double outliers, Dixon's test to detect outlying laboratory means and Kolmogorov-Smirnov's test for normal distribution.

Robust statistics as described in ISO 13528 [5] were used for the determination of the assigned values. The robust mean and robust standard deviations were calculated as per Algorithm A, i.e. as described in ISO 13528, Annex C.21 [5].

Briefly, individual results were ranked in increasing order:

$$(x_1, x_2, x_i, \ldots x_p)$$

Initial values of the robust average x* and robust standard deviation s* were calculated as:

$$x^* = \text{median } x_i \ (1,2,\ldots.,p) \tag{4}$$

$$s^* = 1.483 \text{ x median } |x_i - x^*| \ (1,2,\ldots.,p) \tag{5}$$

The initial values x* and s* were updated by calculating:

$$\delta = 1.5s^* \tag{6}$$

Each x_i and x_i^* were calculated where:

$$\text{if } x_i < x^* - \delta; \quad x_i^* = x^* - \delta \tag{7}$$

$$\text{if } x_i > x^* - \delta; \quad x_i^* = x^* + \delta \tag{8}$$

Otherwise: $$x_i^* = x_i \tag{9}$$

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

$$x^* = \Sigma\, x_i^*/p \tag{10}$$

$$s^* = 1{,}134 \sqrt{(x_i - x^*)^2/(p-1)} \tag{11}$$

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

The medians and unweighted mean of the means were also calculated and compared with the respective robust mean. No significant differences were observed and the reference values obtained with the robust mean approach were further used. These values are considered to be the most reliable estimates of the property values.

The uncertainties associated with the reference values were calculated according to the ISO Guide 35 [3]. The relative combined uncertainty of the certified value of the CRM consists of uncertainty related to characterization u_{char}, between-bottle heterogeneity (u_{bb}) and long-term stability (u_{stab}). These different contributions were combined to estimate the expanded relative uncertainty.

$$U^2_{CRM,rel} = 2\sqrt{x^2_{char} + x^2_{\mathrm{hom}} + x^2_{stab}} \qquad (12)$$

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

u_{hom} was estimated as a larger value of the standard deviation between-bottles (u_{bb}) or the maximum heterogeneity potentially hidden by the method repeatability (u_{bb}*) as seen in Table 2;

u_{stab} the stability during storage period was chosen as 1%, which, as described before, ensured the validity of the certificate for 10 years;

u_{char} was estimated using combined uncertainty reported by the individual laboratories results.

$$u_{char} = \frac{\sqrt{\sum_{i=1}^{p} u_i^2}}{p} \qquad (13)$$

Where:

u_i is the combined uncertainty provided by participating laboratories [5]; and p is the number of laboratories.

As shown previously in Figure 1, the methods with different quantification steps (AAS, GF-AAS, AFS, ICP-OES, ICP-MS) as well as methods without sample preparation step such as INAA and X-ray Fluorescence were used for characterization of the material. The agreement between results confirms the absence of any significant method bias and demonstrates the identity of the analyte..

The results provided by the participants for trace elements mass fractions grouped by methods are displayed in Figures 4–20 and in Tables 4–20 (Appendix). The detailed results as reported by participants are shown in Appendix I. In all figures, the reported results are plotted versus the assigned (reference) values, which are denoted by a bold line, while the dashed lines represent the expanded uncertainty ($k=2$) associated with the assigned (reference) value. The error bars represent the expanded uncertainty as reported by participants.

A good agreement within the stated uncertainty was observed for results obtained with different methods. Therefore, all of them were considered in the deriving of reference values.

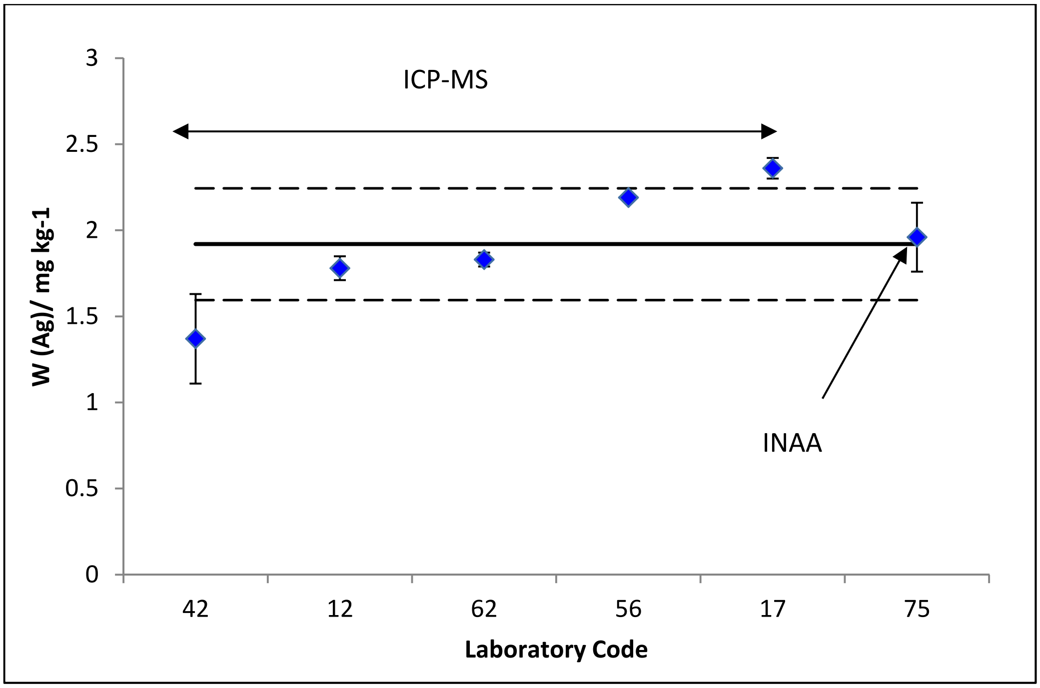

FIG. 4. Laboratory results for silver mass fraction (mg kg^{-1}) in the IAEA-457 sediment sample.

The robust mean (solid line) and corresponding expanded uncertainty (k=2) (dashed line) are shown. The error bars correspond to the expanded uncertainty reported by each laboratory.

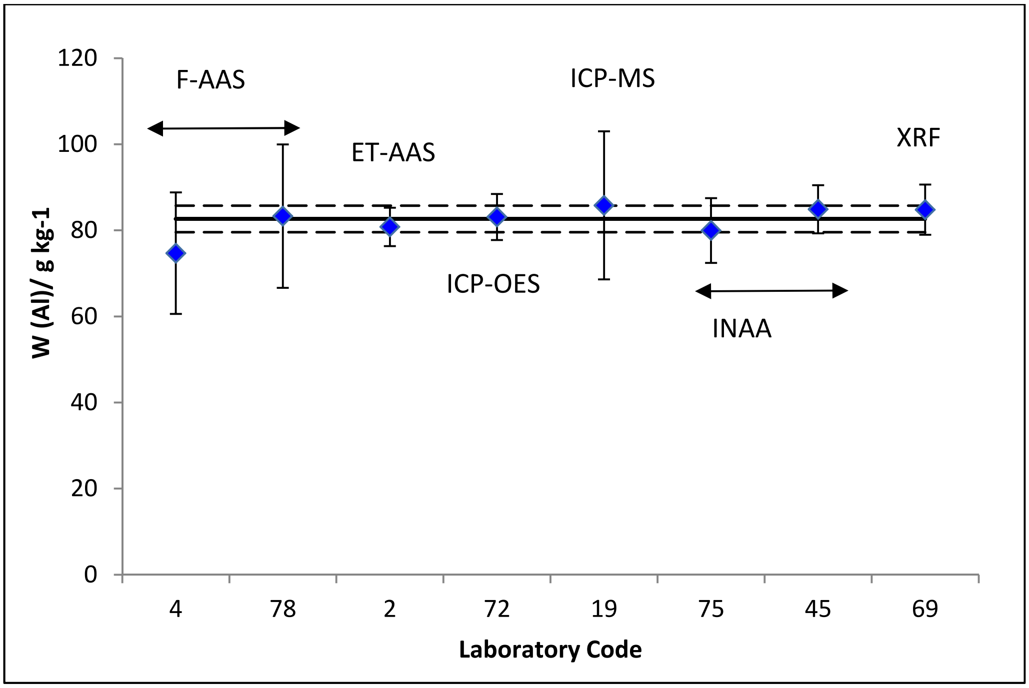

FIG. 5. Laboratory results for aluminum mass fraction (g kg^{-1}) in the IAEA-457 sediment sample.
The robust mean (solid line) and corresponding expanded uncertainty (k=2) (dashed line) are shown. The error bars correspond to the expanded uncertainty reported by each laboratory.

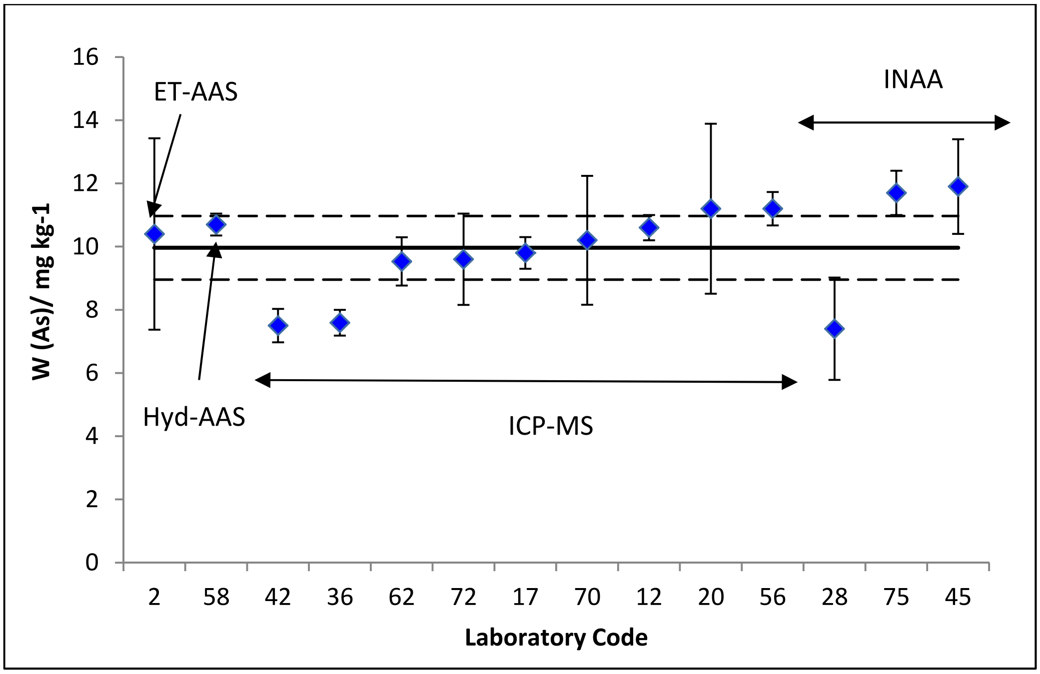

FIG. 6. Laboratory results for arsenic mass fraction (mg kg^{-1}) in the IAEA-457 sediment sample.
The robust mean (solid line) and corresponding expanded uncertainty (k=2) (dashed line) are shown. The error bars correspond to the expanded uncertainty reported by each laboratory.

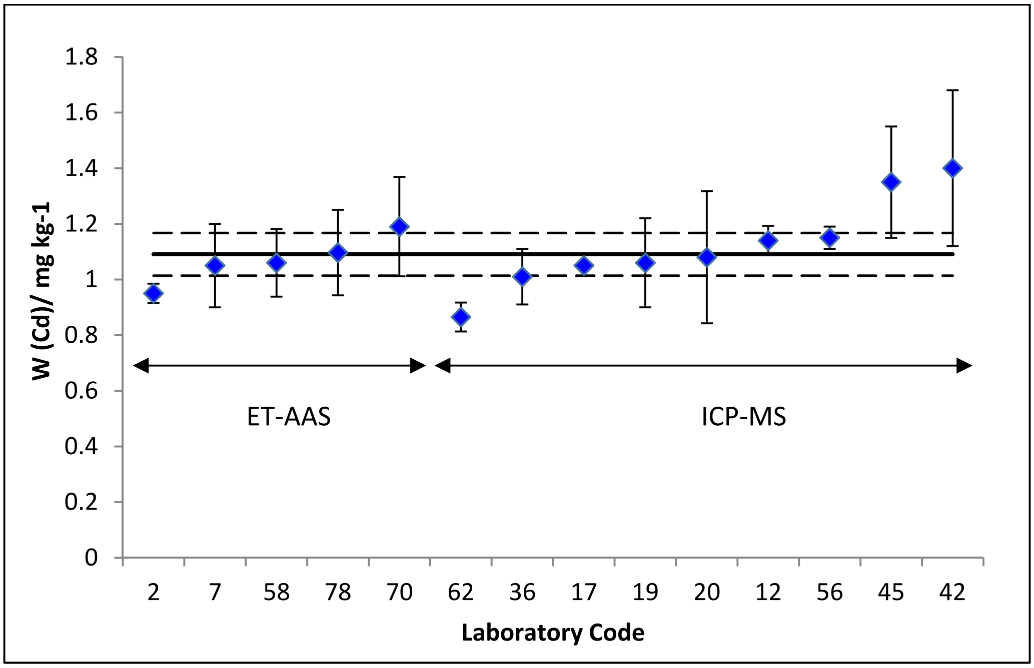

FIG. 7. Laboratory results for cadmium mass fraction (mg kg^{-1}) in the IAEA-457 sediment sample.
The robust mean (solid line) and corresponding expanded uncertainty (k=2) (dashed line) are shown. The error bars correspond to the expanded uncertainty reported by each laboratory.

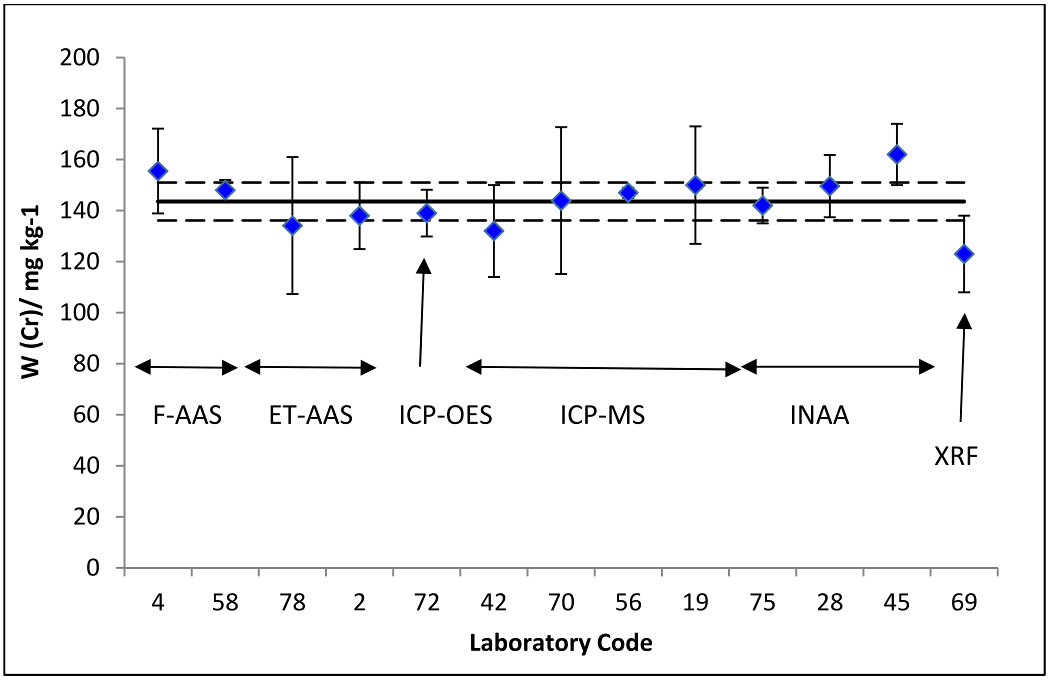

FIG. 8. Laboratory results for chromium mass fraction (mg kg^{-1}) in the IAEA-457 sediment sample.
The robust mean (solid line) and corresponding expanded uncertainty (k=2) (dashed line) are shown. The error bars correspond to the expanded uncertainty reported by each laboratory.

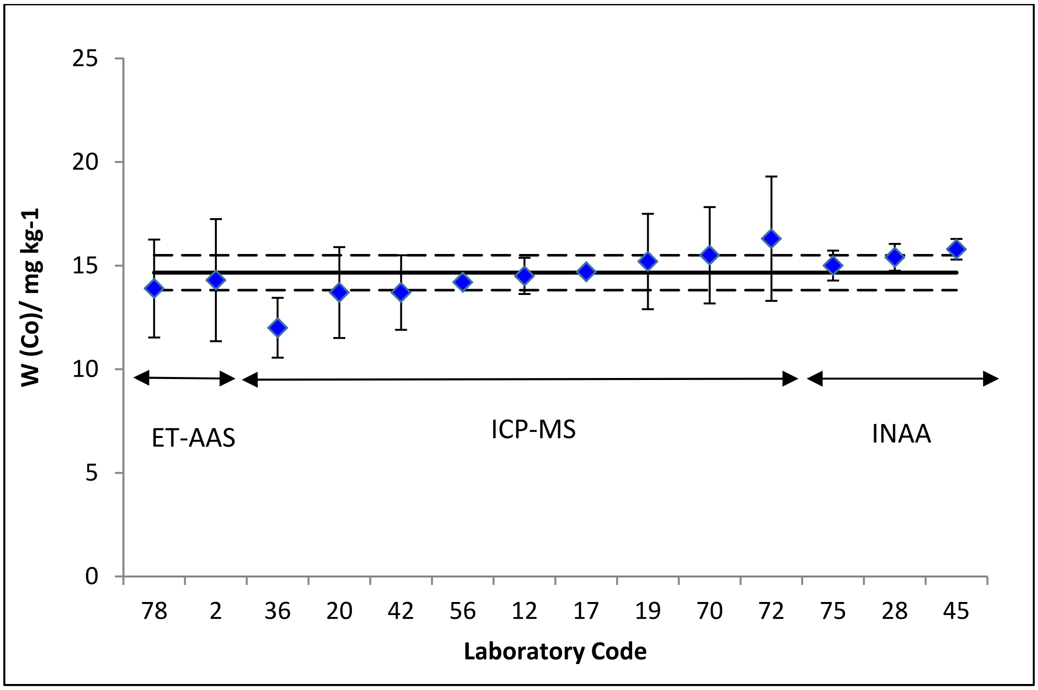

FIG. 9. Laboratory results for cobalt mass fraction (mg kg^{-1}) in the IAEA-457 sediment sample.
The robust mean (solid line) and corresponding expanded uncertainty (k=2) (dashed line) are shown. The error bars correspond to the expanded uncertainty reported by each laboratory.

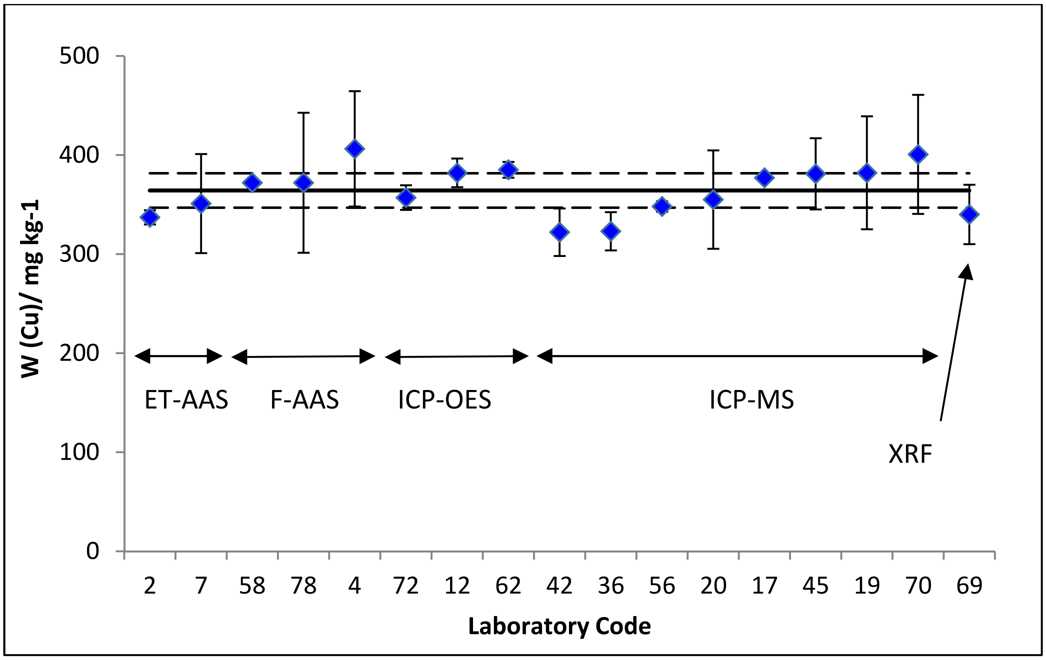

FIG. 10. Laboratory results for copper mass fraction (mg kg^{-1}) in the IAEA-457 sediment sample.
The robust mean (solid line) and corresponding expanded uncertainty (k=2) (dashed line) are shown. The error bars correspond to the expanded uncertainty reported by each laboratory.

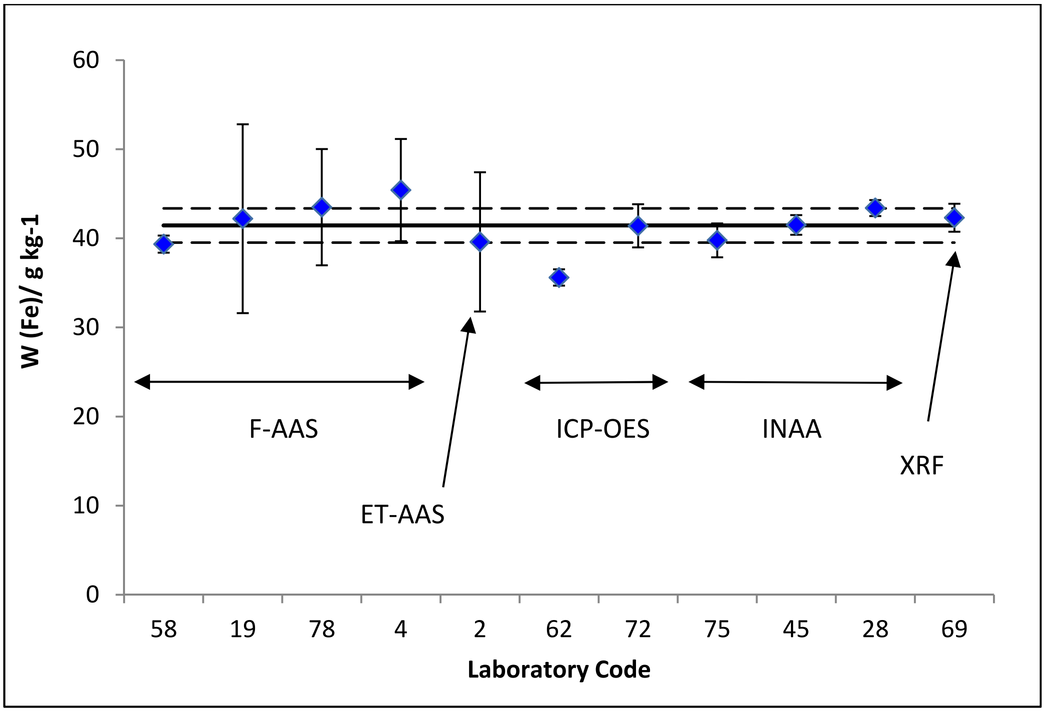

FIG. 11. Laboratory results for iron mass fraction (g kg^{-1}) in the IAEA-457 sediment sample.

The robust mean (solid line) and corresponding expanded uncertainty (k=2) (dashed line) are shown. The error bars correspond to the expanded uncertainty reported by each laboratory.

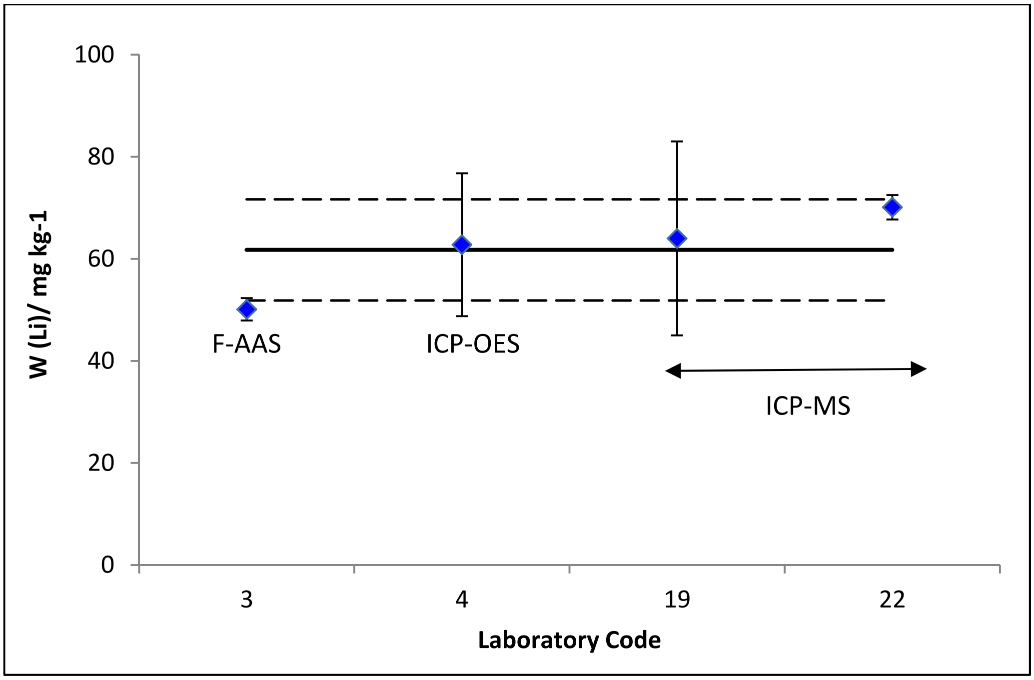

FIG. 12. Laboratory results for lithium mass fraction (mg kg^{-1}*) in the IAEA-457 sediment sample.*
The robust mean (solid line) and corresponding expanded uncertainty (k=2) (dashed line) are shown. The error bars correspond to the expanded uncertainty reported by each laboratory.

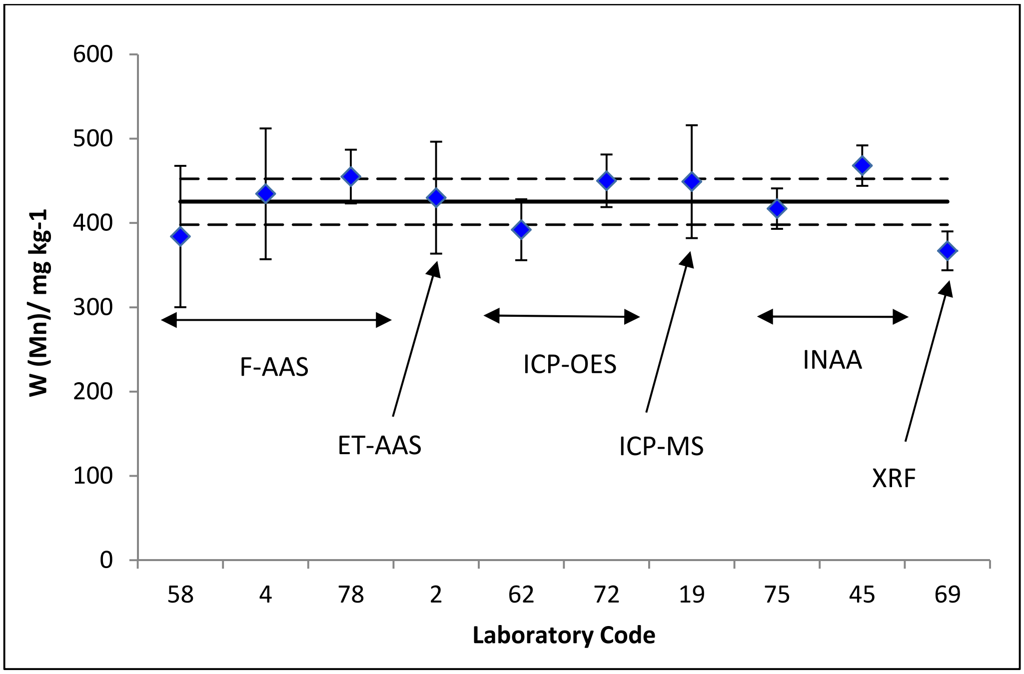

FIG. 13. Laboratory results for manganese mass fraction (mg kg^{-1}*) in the IAEA-457 sediment sample.*
The robust mean (solid line) and corresponding expanded uncertainty (k=2) (dashed line) are shown. The error bars correspond to the expanded uncertainty reported by each laboratory.

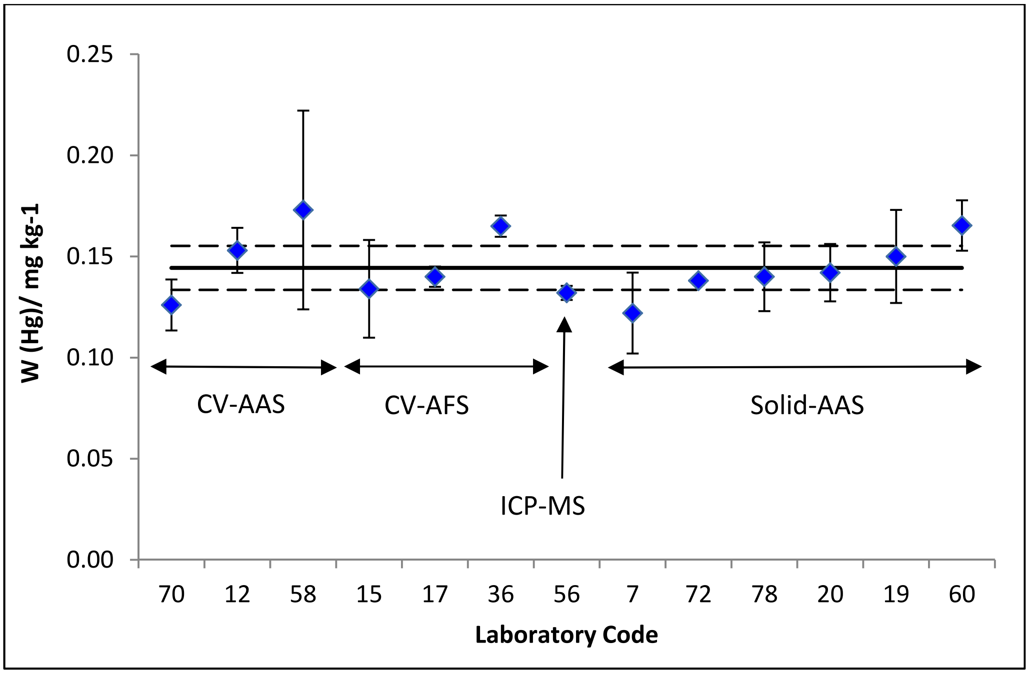

FIG. 14. Laboratory results for mercury mass fraction (mg kg^{-1}) in the IAEA-457 sediment sample.
The robust mean (solid line) and corresponding expanded uncertainty (k=2) (dashed line) are shown. The error bars correspond to the expanded uncertainty reported by each laboratory.

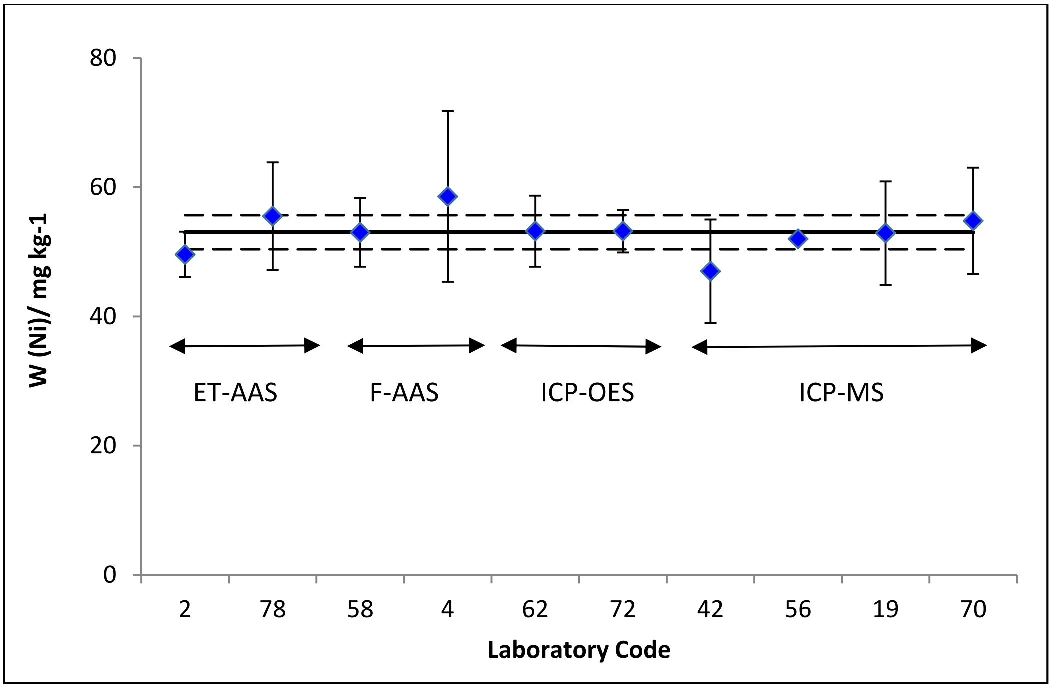

FIG. 15. Laboratory results for nickel mass fraction (mg kg^{-1}) in the IAEA-457 sediment sample.
The robust mean (solid line) and corresponding expanded uncertainty (k=2) (dashed line) are shown. The error bars correspond to the expanded uncertainty reported by each laboratory.

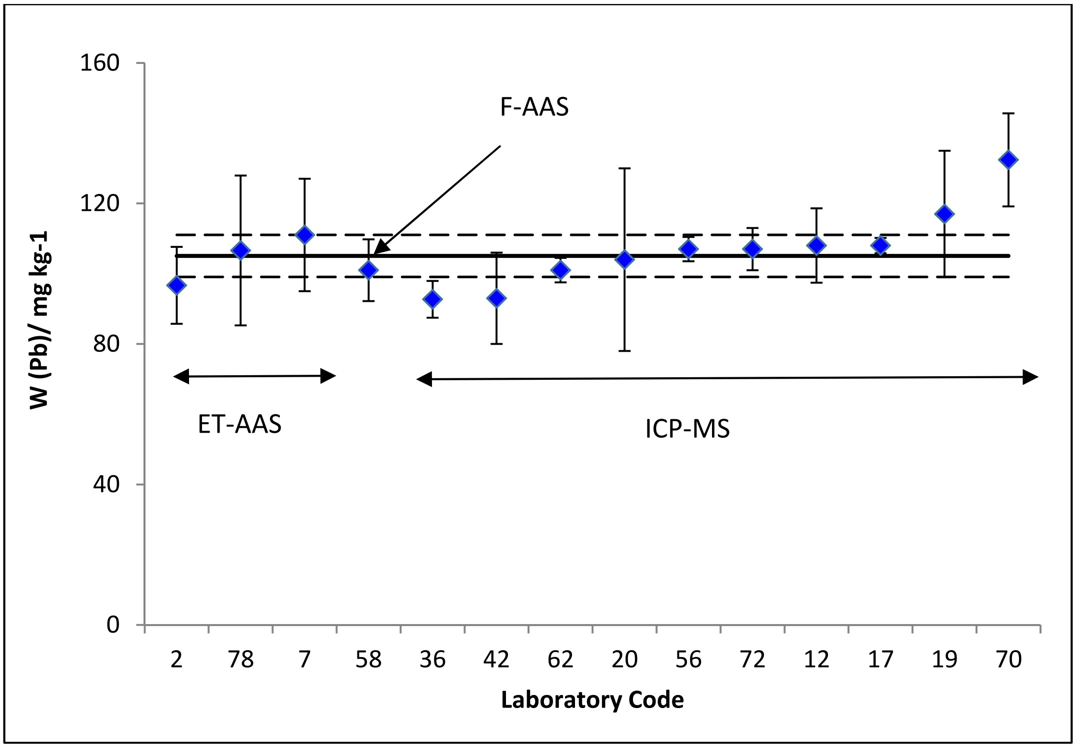

FIG. 16. Laboratory results for lead mass fraction (mg kg^{-1}) in the IAEA-457 sediment sample.

The robust mean (solid line) and corresponding expanded uncertainty (k=2) (dashed line) are shown. The error bars correspond to the expanded uncertainty reported by each laboratory.

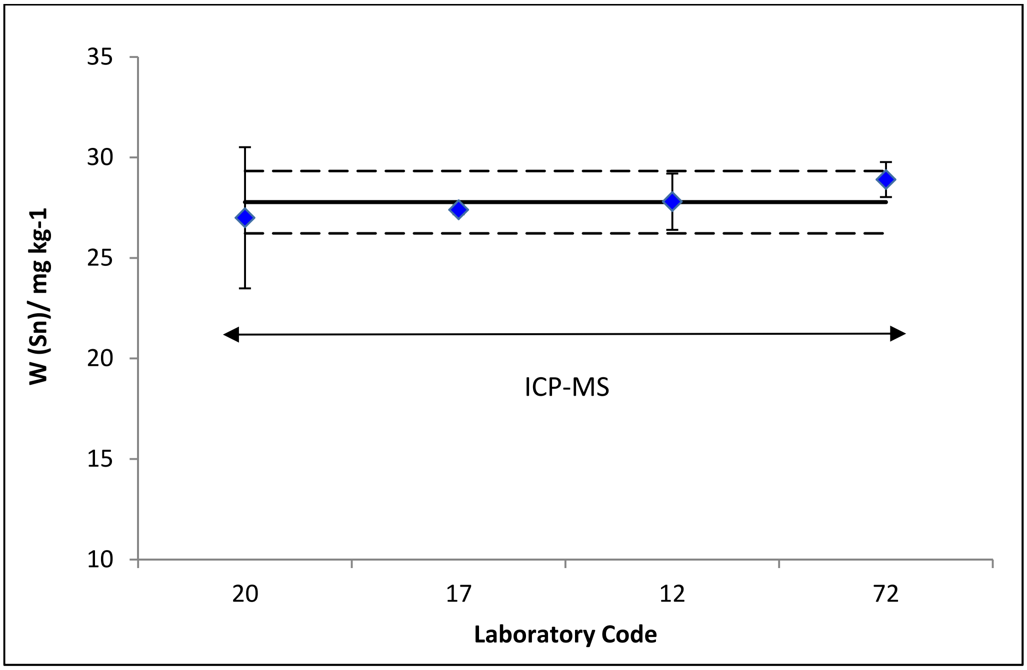

FIG. 17. Laboratory results for tin mass fraction (mg kg^{-1}) in the IAEA-457 sediment sample.

The robust mean (solid line) and corresponding expanded uncertainty (k=2) (dashed line) are shown. The error bars correspond to the expanded uncertainty reported by each laboratory.

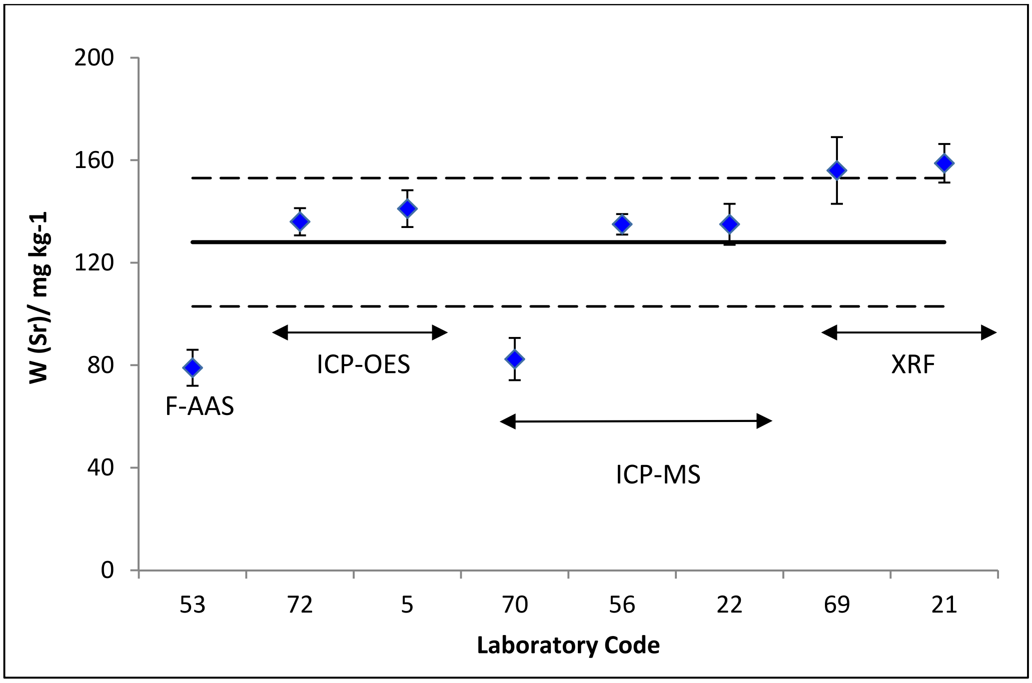

FIG. 18. Laboratory results for strontium mass fraction (mg kg^{-1}) in the IAEA-457 sediment sample.
The robust mean (solid line) and corresponding expanded uncertainty (k=2) (dashed line) are shown. The error bars correspond to the expanded uncertainty reported by each laboratory.

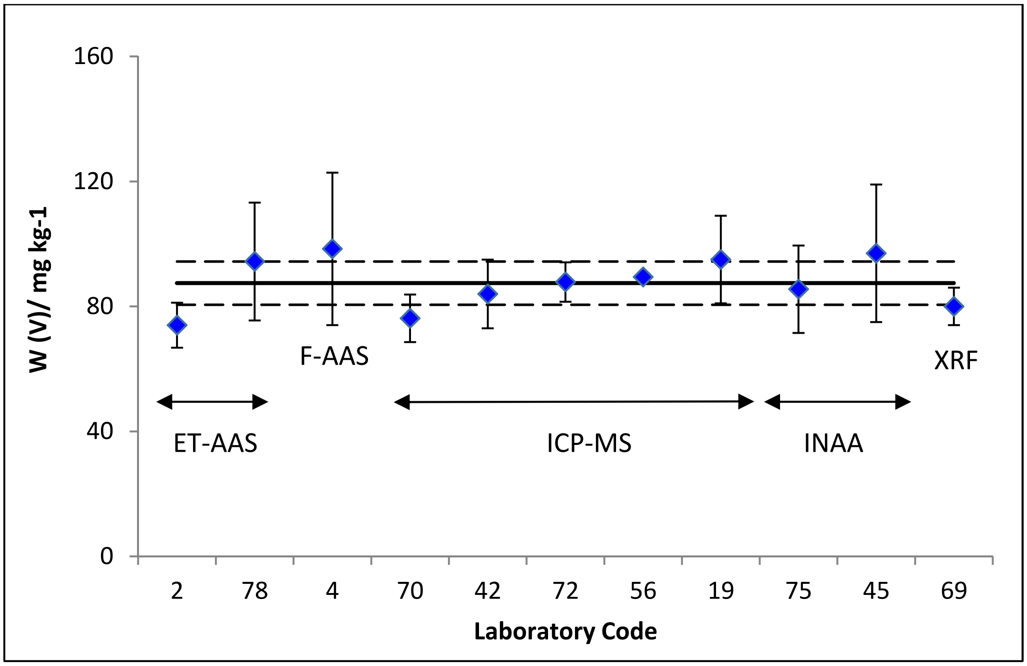

FIG. 19. Laboratory results for vanadium mass fraction (mg kg^{-1}) in the IAEA-457 sediment sample.
The robust mean (solid line) and corresponding expanded uncertainty (k=2) (dashed line) are shown. The error bars correspond to the expanded uncertainty reported by each laboratory.

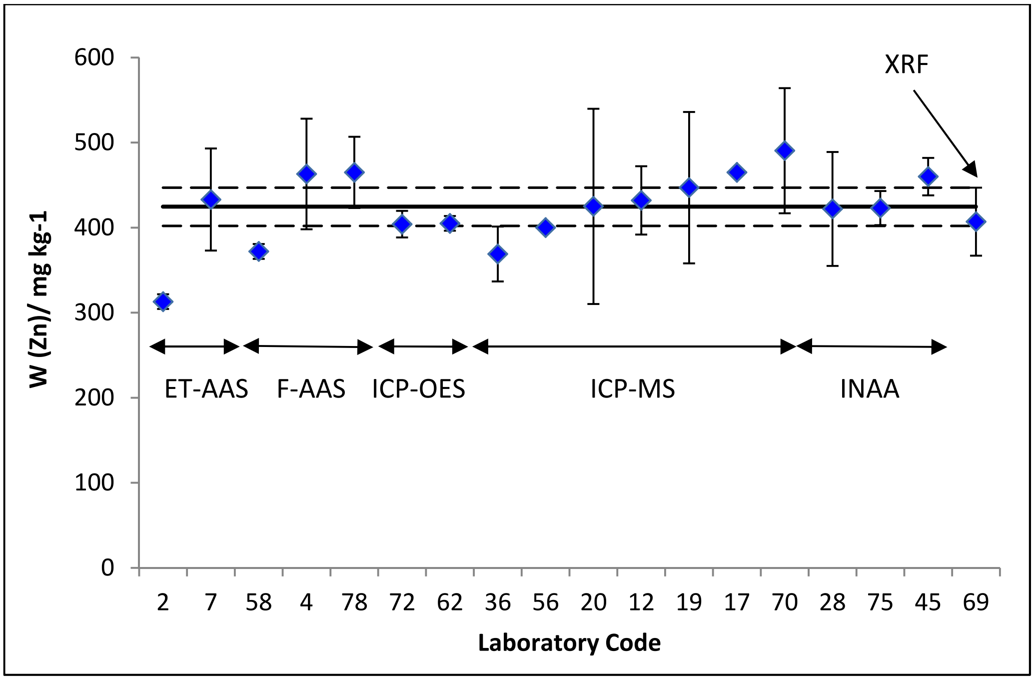

FIG. 20. Laboratory results for zinc mass fraction (mg kg^{-1}) in the IAEA-457 sediment sample.

The robust mean (solid line) and corresponding expanded uncertainty (k=2) (dashed line) are shown. The error bars correspond to the expanded uncertainty reported by each laboratory.

4. METROLOGICAL TRACEABILITY

If the results obtained from different laboratories are to be compared, it is essential that all results are based on reliable measurement standards whose values are linked to a stated reference.

Only validated methods applied within stated scope were used by participating laboratories in this certification exercise. Matrix CRMs with stated SI traceability purchased from NIST, EC JRC IRMM, NRC-CNRC were used for validation of the applied in this study methods [1].

Pure metal standard solutions (CRMs) with stated purity were employed for calibration by all the laboratories participating in this certification campaign. As stated in the respective certificates of all CRM producers, the mass fractions of the trace element in the respective standard solutions were measured against another CRM (i.e. NIST, BAM or EMPA) with demonstrated SI traceability, followed by gravimetric preparation using balances calibrated with SI-traceable weights. Consequently, the value calculated by this unbroken chain of comparison is traceable to the reference to which the starting material is compared to.

In addition, the agreement between the results confirms the absence of any significant method bias and demonstrates the identity of the analytes.

5. CONCLUSIONS

This exercise allows assignment of reference values for Ag, Al, As, Cd, Cr, Co, Cu, Fe, Hg, Li, Mn, Ni, Pb, Sn, Sr, V and Zn with associated uncertainties following ISO guidelines. The certified values are derived from measurement results provided by the laboratories participating in this certification campaign. Only validated methods were applied in the certification of the sediment sample. As the certified values are a combination of SI traceable individual results, themselves traceable to SI, the produced sediment CRM is suitable for the purposes of environment laboratories quality control, and can be used as a sample for the proficiency tests and interlaboratory comparisons. As any certified reference material, it can be used for validation studies.

The reference values for Ag, Al, As, Cd, Cr, Co, Cu, Fe, Hg, Li, Mn, Ni, Pb, Sn, Sr, V and Zn are presented in Table 3, together with their expanded uncertainty (*k=2*).

TABLE 3. REFERENCE VALUES FOR TRACE ELEMENTS MASS FRACTIONS AND THEIR EXPANDED UNCERTAINTY (*k=2*) IN THE IAEA-457 SEDIMENT SAMPLE

Element	Reference value[1] mg kg^{-1}	Expanded uncertainty [2] mg kg^{-1}	Number of accepted datasets
Ag	1.85	0.39	6
Al	82660	3430	8
As	10.2	1.0	14
Cd	1.09	0.08	14
Co	14.7	1	14
Cr	144	8	13
Cu	365	19	17
Fe	41450	2240	11
Hg	0.143	0.012	13
Li	64.2	5.5	5
Mn	427	30	10
Ni	53.31	2.7	10
Pb	105	7	14
Sn	27.40	0.75	5
Sr	137	10	9
V	87.4	8.1	11
Zn	425	25.8	18

[1] *The value is the robust mean of accepted sets of data, each set being obtained by different laboratory. The certified values are reported in dry mass basis and are traceable to the SI.*

[2] *Expanded uncertainty with a coverage factor k=2 according to the guide to the Expression of Uncertainty of Measurement (GUM), corresponding to the level of confidence of about 95%*

APPENDIX I

DATA REPORT OF RESULTS SORTED BY ELEMENTS

TABLE 4. SILVER: RESULTS AS REPORTED BY PARTICIPANTS (mg kg^{-1})

Lab code	Mean	Combined uncertainty (u)	Expanded uncertainty (U)
12	1.78	0.03	0.07
17	2.36	0.03	0.06
42	1.37	0.13	0.26
56	2.19	0.01	0.02
62	1.83	0.02	0.04
75	1.96	0.052	0.2

TABLE 5. ALUMINIUM: RESULTS AS REPORTED BY PARTICIPANTS (g kg^{-1})

Lab code	Mean	Combined uncertainty (u)	Expanded uncertainty (U)
2	80.8	2.23	4.47
4	74.701	7.067	14.134
19	85.8	8.6	17.2
45	84.9	2.8	5.6
69	84.8	2.98	5.84
72	83.1	2.67	5.33
75	79.97	0.000047	7.517
78	83.3	8.3	16.7

TABLE 6. ARSENIC: RESULTS AS REPORTED BY PARTICIPANTS (mg kg^{-1})

Lab code	Mean	Combined uncertainty (u)	Expanded uncertainty (U)
2	10.4	1.51	3.03
12	10.6	0.20	0.4
17	9.8	0.25	0.5
20	11.2	1.344	2.688
28	7.4	0.81	1.62
36	7.59	0.20	0.41
42	7.5	0.27	0.53
45	11.9	0.75	1.5
56	11.2	0.26	0.53
58	10.7	0.17	0.35
62	9.53	0.38	0.76
70	10.2	1.02	2.04
72	9.6	0.72	1.44
75	11.7	0.03	0.7

TABLE 7. CADMIUM: RESULTS AS REPORTED BY PARTICIPANTS (mg kg^{-1})

Lab code	Mean	Combined uncertainty (u)	Expanded uncertainty (U)
2	0.95	0.02	0.03
7	1.05	0.075	0.15
12	1.14	0.03	0.05
17	1.05	0.005	0.011
19	1.06	0.08	0.16
20	1.08	0.1188	0.2376
36	1.01	0.05	0.10
42	1.4	0.14	0.28
45	1.35	0.1	0.2
56	1.15	0.02	0.04
58	1.06	0.06	0.12
62	0.865	0.03	0.05
70	1.19	0.08925	0.1785
78	1.10	0.08	0.15

TABLE 8. CHROMIUM: RESULTS AS REPORTED BY PARTICIPANTS (mg kg^{-1})

Lab code	Mean	Combined uncertainty (u)	Expanded uncertainty (U)
2	138	6.56	13.11
4	155.5	8.3	16.6
19	150	11	23
28	149.6	6.1	12.2
42	132	9	18
45	162	6.1	12
56	147	1	2
58	148	2	4
69	123	8	15
70	143.9	14.39	28.78
72	139	4.58	9.17
75	142	0.024	7
78	134.1	13.4	26.8

TABLE 9. COLBAT: RESULTS AS REPORTED BY PARTICIPANTS (mg kg^{-1})

Lab code	Mean	Combined uncertainty (u)	Expanded uncertainty (U)
2	14.3	1.47	2.95
12	14.5	0.44	0.87
17	14.7	0.04	0.08
19	15.2	1.1	2.3
20	13.7	1.096	2.192
28	15.4	0.32	0.65
36	12	0.72	1.44
42	13.7	0.9	1.8
45	15.79	0.25	0.50
56	14.2	0.1	0.2
70	15.5	1.1625	2.325
72	16.3	1.5	3
75	15	0.024	0.72
78	13.89	1.2	2.4

TABLE 10. COPPER: RESULTS AS REPORTED BY PARTICIPANTS (mg kg^{-1})

Lab code	Mean	Combined uncertainty (u)	Expanded uncertainty (U)
2	337	3.61	7.21
4	406.2	29.1	58.2
7	351	25	50
12	382	7.21	14.42
17	377	0.5	1
19	382	29	57
20	355	24.85	49.7
36	323	9.64	19.29
42	322	12	24
45	381	18	36
56	348	2.65	5.29
58	372	1.73	3.46
62	385	4	8
69	340	15	30
70	400.6	30.045	60.09
72	357	6.24	12.49
78	372	35.3	70.7

TABLE 11. IRON: RESULTS AS REPORTED BY PARTICIPANTS (g kg^{-1})

Lab code	Mean	Combined uncertainty (u)	Expanded uncertainty (U)
2	39.6	3.91	7.81
4	45.416	2.8677	5.7354
19	42.2	5.3	10.6
28	43.4	0.4	0.9
45	41.5	0.56	1.1
58	39.346	0.48	0.96
62	35.6	0.46	0.92
69	42.3	0.8	1.57
72	41.4	1.21	2.42
75	39.77	0.000024	1.91
78	43.5	3.3	6.5

TABLE 12. MERCURY: RESULTS AS REPORTED BY PARTICIPANTS (mg kg^{-1})

Lab code	Mean	Combined uncertainty (u)	Expanded uncertainty (U)
7	0.122	0.01	0.02
12	0.153	0.01	0.01
15	0.134	0.01206	0.02412
17	0.14	0.002	0.005
19	0.15	0.011	0.023
20	0.142	0.0071	0.0142
36	0.165	0.00	0.01
56	0.132	0.00	0.00
58	0.173	0.02	0.05
60	0.16528	0.01	0.01
70	0.126	0.0063	0.0126
72	0.138	0.001	0.002
78	0.14	0.0084	0.017

TABLE 13. LITHIUM: RESULTS AS REPORTED BY PARTICIPANTS (mg kg^{-1})

Lab code	Mean	Combined uncertainty (u)	Expanded uncertainty (U)
3	50.11	1.1	2.2
4	62.77	7	14
19	64	10	19
22	70.1	1.2	2.4
78	66.2	4.0	7.9

TABLE 14. MANGANESE: RESULTS AS REPORTED BY PARTICIPANTS (mg kg^{-1})

Lab code	Mean	Combined uncertainty (u)	Expanded uncertainty (U)
2	430	33.15	66.30
4	434.6	38.8	77.6
19	449	34	67
45	468	12	24
58	384	41.9	83.81
62	392	18.03	36.06
69	367	12	23
72	450	15.62	31.24
75	417	0.029	24
78	455	15.9	31.9

TABLE 15. NICKEL: RESULTS AS REPORTED BY PARTICIPANTS (mg kg^{-1})

Lab code	Mean	Combined uncertainty (u)	Expanded uncertainty (U)
2	49.6	1.76	3.52
4	58.56	6.6	13.2
19	52.9	4	8
42	47	4	8
56	52	0.2	0.4
58	53	2.65	5.29
62	53.2	2.75	5.5
70	54.8	4.11	8.22
72	53.2	1.65	3.29
78	55.52	4.2	8.3

TABLE 16. LEAD: RESULTS AS REPORTED BY PARTICIPANTS (mg kg^{-1})

Lab code	Mean	Combined uncertainty (u)	Expanded uncertainty (U)
2	96.7	5.47	10.95
7	111	8	16
12	108	5.29	10.58
17	108	1.1	2.2
19	117	9	18
20	104	13	26
36	92.7	2.62	5.25
42	93	7	13
56	107	1.73	3.46
58	101	4.39	8.78
62	101	1.73	3.46
70	132.4	6.62	13.24
72	107	3	6
78	106.6	10.7	21.3

TABLE 17. TIN: RESULTS AS REPORTED BY PARTICIPANTS (mg kg^{-1})

Lab code	Mean	Combined uncertainty (u)	Expanded uncertainty (U)
12	27.8	0.70	1.40
17	27.4	0.11	0.22
20	27	1.755	3.51
72	28.9	0.44	0.87
78	24.7	1.5	3

TABLE 18. STRONTIUM: RESULTS AS REPORTED BY PARTICIPANTS (mg kg^{-1})

Lab code	Mean	Combined uncertainty (u)	Expanded uncertainty (U)
5	141.1	3.6	7.2
21	158.8	2.93	7.53
22	135	4	8
53	79	0.04367	7
56	135	2	4
69	156	7	13
70	82.4	4.12	8.24
72	136	2.65	5.29
78	140	8.4	16.8

TABLE 19. VANADIUM: RESULTS AS REPORTED BY PARTICIPANTS (mg kg^{-1})

Lab code	Mean	Combined uncertainty (u)	Expanded uncertainty (U)
2	74	3.61	7.21
4	98.43	12.2	24.4
19	95	7	14
42	84	6	11
45	97	11	22
56	89.4	0.10	0.20
69	80	3	6
70	76.2	3.81	7.62
72	87.8	3.14	6.29
75	85.5	0.08	14.0
78	94.37	9.4	18.9

TABLE 20. ZINC: RESULTS AS REPORTED BY PARTICIPANTS (mg kg^{-1})

Lab code	Mean	Combined uncertainty (u)	Expanded uncertainty (U)
2	313	4.36	8.72
4	463	32.5	65
7	433	30	60
12	432	20.07	40.15
17	465	0.7	1.4
19	447	45	89
20	425	57.375	114.75
28	422	33	67
36	369	16.09	32.19
45	460	11	22
56	400	2	4
58	372	4.36	8.72
62	405	4.36	8.72
69	407	20	40
70	490.5	36.7875	73.575
72	404	7.81	15.62
75	423	0.024	20
78	464.9	20.9	41.8

APPENDIX II

LIST OF PARTICIPATING LABORATORIES IN THE CERTIFICATION OF IAEA-457 CANDIDATE REFERENCE MATERIAL

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No. 22

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