

# Certification of Trace Elements and Methyl Mercury Mass Fractions in IAEA-456 Marine Sediment Samples



**IAEA**

International Atomic Energy Agency

CERTIFICATION OF  
TRACE ELEMENTS AND METHYL  
MERCURY MASS FRACTIONS  
IN IAEA-456 MARINE  
SEDIMENT SAMPLES

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CERTIFICATION OF TRACE ELEMENTS AND METHYL MERCURY MASS FRACTIONS IN IAEA-456 MARINE SEDIMENT SAMPLES

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## FOREWORD

The primary goal of the IAEA Environment Laboratories is to assist Member States in the use of both stable and radioisotope analytical techniques to understand, monitor and protect the environment. In this context, the major impact of large coastal cities on marine ecosystems is an issue of prime concern for the IAEA and the IAEA Environment Laboratories. The marine pollution assessments required to understand such impacts depend on accurate knowledge of contaminant concentrations in various environmental compartments. The IAEA Environment Laboratories has been assisting national laboratories and regional laboratory networks since the early 1970s through the provision of a reference material programme for the analysis of radionuclides, trace elements and organic compounds in marine samples.

Quality assurance, quality control and associated good laboratory practice are essential components of all marine environmental monitoring studies. Quality control 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 the reliability and comparability of measurement data. Data that are not based on adequate quality assurance and quality control can be erroneous, and their misuse can lead to poor environmental management decisions.

A marine sediment sample with certified mass amount contents for aluminium, arsenic, cadmium chromium, cobalt, copper, iron, lead, mercury, methyl mercury, manganese, nickel, vanadium and zinc was recently produced by the IAEA Environment Laboratories. This publication presents the sample preparation methodology, including material homogeneity and the 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, certified values for mass fractions and associated expanded uncertainty were established for 13 trace elements and methyl mercury in the marine sediment sample.

The IAEA wishes to thank all the participants and the laboratories who took part in this interlaboratory comparison. The IAEA is also grateful to the Government of Monaco for its support and provision of the raw material. The IAEA officers responsible for this publication were E. Vasileva and S. Azemard of the IAEA Environment Laboratories.

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## **1. INTRODUCTION**

Many laboratories are involved in the production of local or regional environmental monitoring data leading, in many cases, to wider assessments. These laboratories may develop and validate new analytical methods, study the environmental impact of human activities, provide services to other organizations, etc. Due to the needs to base scientific conclusions on valid and internationally comparable data and to provide policy-makers with correct information on the state of the environment, it is indispensable to ensure the quality of the data produced by each laboratory.

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 comparisons. IAEA's subprogram 'Reference Products for Science and Trade' represents an important benchmark in upgrading the quality of laboratory performances and assessing the validity of the analytical methods used for marine monitoring studies in the Member States.

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, there still is a noticeable lack of matrix CRMs.

The work presented in this report refers exclusively to the certification of the total content of trace elements and methyl mercury in marine sediment. This material is a certified reference material (CRM), released in November 2014 by the IAEA.

## **2. METHODOLOGY**

### **2.1. DESCRIPTION OF THE MATERIAL**

The sediment material was collected in New Caledonia, immediately frozen and then freeze dried. The sediment was first sieved at 500  $\mu\text{m}$ , ground, and then sieved at 125  $\mu\text{m}$  followed by mechanical homogenization of the collected fraction.

The sieving cut-off value (125  $\mu\text{m}$ ) was selected to ensure that the physical properties of the material are sufficiently uniform, whilst retaining sufficient material to make an adequate number of units.

Aliquots of about 15 g were packed into amber glass bottles with polyethylene caps and sealed in plastic bags.

## 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 interlaboratory comparisons and certification campaigns on marine sediments. Only results of laboratories having a quality system in place, using validated methods, applying uncertainty and traceability concepts and having provided good results in previous IAEA interlaboratory comparisons were accepted for the calculation of certified values and their uncertainties.

Each laboratory received one bottle of sediment sample, accompanied by an information sheet and a reporting form. Participants were requested to analyze Al, As, Cd, Co, Cr, Cu, Fe, Hg,  $\text{CH}_3\text{Hg}$ , Mn, Ni, Pb, V and Zn, using a validated analytical method. They were asked to report the measurement results (three replicates and average value) along with the expanded uncertainty in addition to the information about applied quality control procedure. The second request was to report results for the trace element mass fractions in a CRM with a matrix similar to the candidate reference material. The moisture determination method was preliminary validated in MESL and detailed description of the method sent to participants in this certification campaign.

## 2.3. HOMOGENEITY TESTING

Extensive homogeneity tests were carried out on this material in order 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 (Cr, Cd, Cu, Hg, Mn, Ni, Pb, Zn and  $\text{CH}_3\text{Hg}$ ). In total, 10 bottles were selected using random stratified sampling from the whole batch. Three subsamples from each bottle were analyzed for their total element mass fractions. Subsamples of 0.2 g were mineralized with 5 ml concentrated  $\text{HNO}_3$  and 2ml HF using a microwave oven according to the protocol described earlier [1]. Cu, Mn and Zn were measured by flame atomic absorption (F-AAS) and Cd, Cr, Ni and Pb by graphite furnace AAS (ET-AAS) Determination of total mercury was done with solid mercury analyzer (Solid-AAS) and methyl mercury was determined by gas chromatography coupled with atomic fluorescence (GC-AFS) after solvent extraction and room temperature derivatization. Special 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. All methods used for the homogeneity studies were previously validated.

#### 2.4. STABILITY STUDY

Two sets of five bottles each were stored in the dark at different temperatures (+20°C and +60°C) just after the bottling process and kept at described conditions over a period of 2 years. In order to evaluate the short-term stability of the material during the transportation one isochronous study over 6 weeks was applied. The results from another study over the period of 24 months were used to evaluate the long term stability and stability during the storage period. After each of above mentioned time intervals two bottles kept at +20°C and another two kept at +60°C were transferred to -20°C temperature, considered as reference temperature for this study. Two independent measurements on each of the bottles transferred to -20°C were performed under repeatability conditions. The obtained results were compared with the results from samples kept at -20°C during the entire period of the stability study. The stability investigation for the further evaluation of long-term stability is still on-going.

Selected elements were measured: Mn, Zn by F-AAS; Cd, Ni, Pb by ET-AAS, Hg by Solid-AAS and CH<sub>3</sub>Hg by GC-AFS.

#### 2.5. CHARACTERIZATION

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

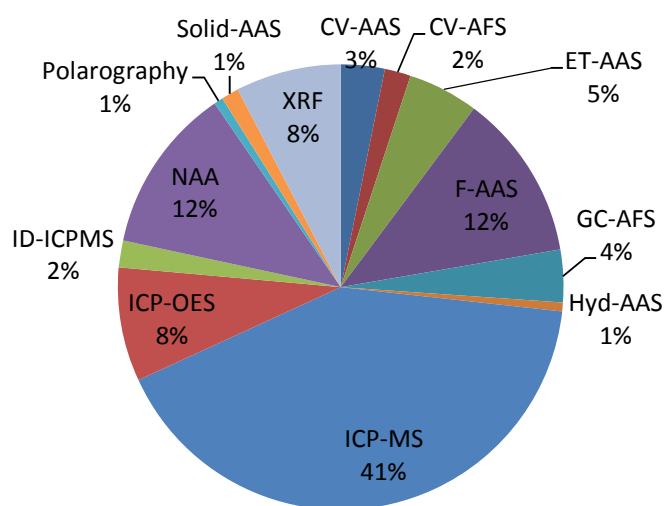


FIG. 1. Analytical methods used for the certification of trace elements in the IAEA-456 sediment sample.

TABLE 1. INSTRUMENTAL TECHNIQUES

Method code	Instrumental technique
F-AAS	Atomic Absorption Spectrometry-Flame
Hyd- AAS	Hydride Generation Atomic Absorption Spectrometry
Solid-AAS	Solid Sampling Atomic Absorption Spectrometry
ICP-MS	Inductively Coupled Plasma-Mass Spectrometry
ID ICP-MS	Isotope Dilution Inductively Coupled Plasma-Mass Spectrometry
AFS	Atomic Fluorescence Spectrometry
CV-AAS	Cold Vapour-Atomic Absorption Spectrometry
ET-AAS	Atomic Absorption Spectrometry-Graphite furnace
NAA	Neutron Activation Analysis
CV-AFS	Cold Vapour-Atomic Fluorescence Spectrometry
XRF	X-Ray Fluorescence
ICP-OES	Inductively Coupled Plasma-Optical Emission Spectrometry
Polarography	Polarography

All participating laboratories have used validated methods. In addition, they have provided measurement results from the analysed 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) [2], including uncertainties coming from 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 IAEA-NAEL and prescribed to other participants. The drying procedure at  $(105 \pm 2)^\circ\text{C}$  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  $105^\circ\text{C}$  was found to be  $(2.1 \pm 0.5)\%$  for bottles kept at  $20^\circ\text{C}$ .

## 3. RESULTS AND DISCUSSION

### 3.1. RESULTS OF THE HOMOGENEITY STUDY

A key requirement for any reference material is the equivalence between the various units. In this respect, it is not relevant whether the variation between units is significant compared to the analytical variation, but whether this variation is significant to the certified uncertainty.

Consequently, ISO Guide 34 [3] requires RM producers to quantify the between-unit variation. This aspect is covered in between-unit homogeneity studies.

The within-unit heterogeneity does not influence the uncertainty of the certified value when the minimum sample intake is respected, but determines the minimum size of an aliquot that is representative for the whole unit. Quantification of within-unit heterogeneity is therefore necessary to determine the minimum sample intake.

For the homogeneity study, 10 samples (about 2% of the total batch) of the sediment were selected using a random sampling and analysed for their trace element 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 bottle means. As presented in table 2, few individual results were detected as outliers at 95% and 99%, and one outlying bottle mean was found for Nickel. Since no technical reasons were identified for outlying results, all data were retained for statistical analysis.

It was checked whether the retained individual results and bottle means follow a normal distribution or are unimodally distributed. The series of results for the investigated trace elements were normally distributed. One way analysis of variance ANOVA [4] 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  $S_{bb}$ :

$$s_{wb} = u_{wb} = \sqrt{MS_{wb}} \quad (1)$$

$$s_{bb} = u_{bb} = \sqrt{\frac{MS_{bb} - MS_{wb}}{n}} \quad (2)$$

For Zn, Ni, Cd, Cr and CH<sub>3</sub>Hg  $MS_{bb}$  (ANOVA mean square between-bottles) were 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 [5]:

$$u^*_{bb} = \frac{s_{wb}}{\sqrt{n}} \sqrt{\frac{2}{v_{MS_{wb}}}} \quad (3)$$

Where:

$n$  is the number of replicate sub-samples per bottle; and  $v_{MS_{wb}}$  is the degrees of freedom of  $MS_{wb}$ .

However, a different approach was adopted for Nickel for which outlying unit average was detected. In this case between-unit inhomogeneity was modelled as a rectangular distribution limited by the largest outlying unit average, and the rectangular standard uncertainty of homogeneity was estimated as given by:

$$u_{rec} = \frac{|\text{outlier} - y|}{\sqrt{3}} \quad (4)$$

$y$  : average of all results of the homogeneity study

It should be mentioned that the outlying unit averages are a result of presence of outlying individual values and do not necessarily reflect the real distribution of these elements in the material.

The heterogeneity could be quantified thanks to the good repeatability of the method used. The between-bottles variations/heterogeneity were between 1 and 3.8%, small enough to ensure the homogeneity of the material. The uncertainty in contributions due to the inhomogeneity were estimated according to ISO Guide 35 [4] as the maximum values obtained with Eq.(2), Eq.(3) or Eq.(4).

TABLE 2. RESULTS FOR INHOMOGENEITY STUDY OF THE IAEA-456 SEDIMENT SAMPLE FOR SELECTED TRACE ELEMENTS

	Individual Outliers		Mean Outliers		$S_{wb}$ %	$S_{bb}$ %	$u^*_{bb}$ %	$u_{rect}$ %	$u_{homo}$ %
	95%	99%	95%	99%					
Cd	2	0	0	0	4.7	<sup>1)</sup>	1.9		1.9
CH <sub>3</sub> Hg	1	0	0	0	6.8	<sup>1)</sup>	3.8		3.8
Cr	0	0	0	0	4.8	<sup>1)</sup>	1.6		1.6
Cu	2	2	0	0	3.5	0.8	1.1		1.1
Hg	3	2	0	0	4.1	1.0	1.3		1.3
Mn	1	0	0	0	2.5	1.0	0.8		1.0
Ni	5	5	1	1	5.1	<sup>1)</sup>	2.0	2.5	2.5
Pb	0	0	0	0	3.6	1.6	1.2		1.6
Zn	2	2	0	0	3.3	<sup>1)</sup>	1.1		1.1

<sup>1)</sup>Not defined due to negative argument under the square root

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 [4], 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.

The results of the evaluation of the between-unit variation are summarized in Table 2. In most cases, the uncertainty contribution for homogeneity was determined by the method repeatability.

### 3.2. RESULTS OF THE STABILITY STUDY

Samples selected for stability study were analyzed and each of the elements was evaluated individually. Regression analyses were performed to evaluate potential trends in the analytical sequence. The results were screened for outliers using the single and double Grubbs test. One outlier was detected at 95% confidence level for Pb and Ni; since no technical reasons were identified for outlying results and data were normally distributed, all data were retained for statistical analysis.

Furthermore, the data were plotted against storage time and linear regression lines of mass concentration versus time were calculated. The slope of the regression lines was tested for statistical significance (loss/increase due to storage conditions).



The test material showed no significant trend to degradation over the time frame at different temperature (+20°C and +60°C). No impact of storage conditions on the stability of the certified properties was detected, neither of storage time nor of temperature (up to +60°C). In all 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 investigated analytes. As no degradation could be observed under any conditions, neither in the short-term (up to 6 weeks) nor in the long term (up to 24 months), it was concluded that no special precautions regarding temperature control during shipment are necessary. The uncertainty of the short-term stability ( $u_{st}$ ) was assumed to be negligible since no degradation is expected to happen during this short time. Nevertheless, 20°C was chosen as storage temperature.

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 [4] 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. [5]. 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 3 years. The results obtained from the stability studies provide evidence to a good stability of all analytes considered.

Figure 2 represent the results on the long term stability (24 months) for CH<sub>3</sub>Hg obtained with isochronous approach.

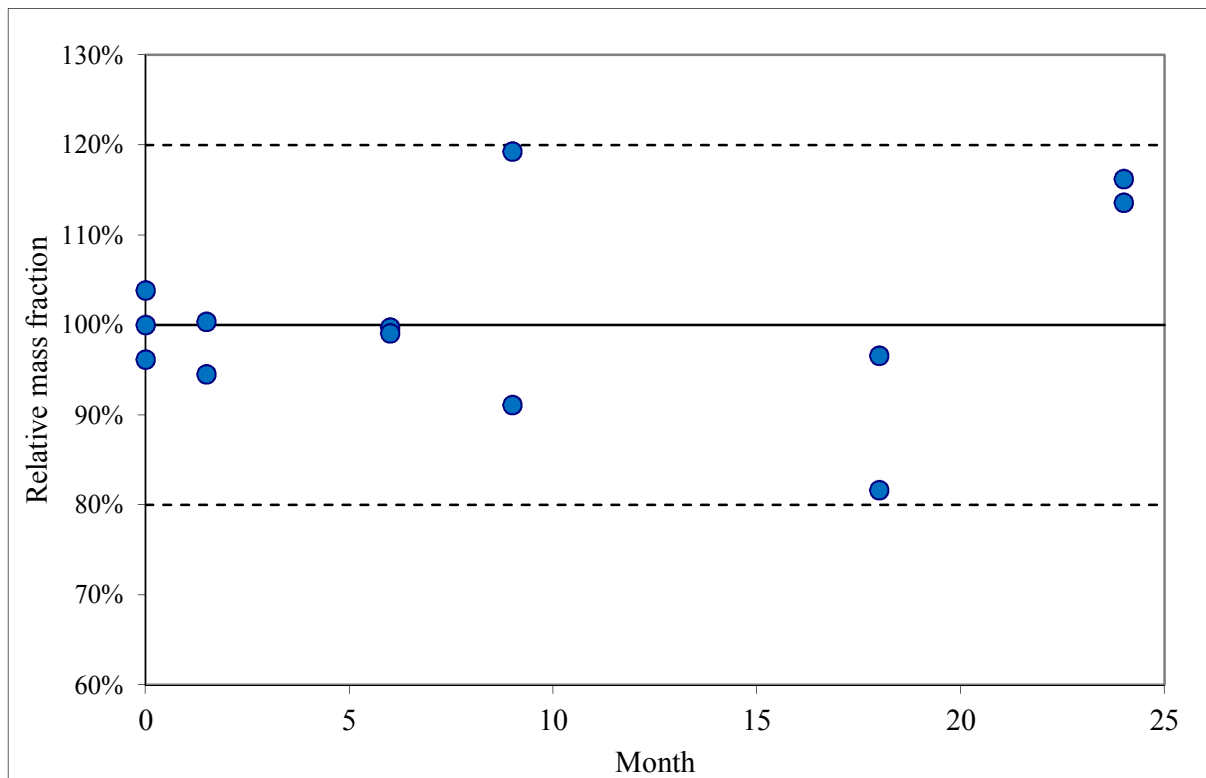


FIG. 2. Results obtained with isochronous approach for the long-term stability study of  $CH_3Hg$  in sediment samples kept at  $20^\circ C$ ; Dots present individual mass fractions measured at each time-point relative to the mean at time zero, against the time that the samples were held at  $20^\circ C$ . Dashed lines represent expanded uncertainty of measurements.

### 3.3. DETERMINATION OF CERTIFIED VALUES AND UNCERTAINTIES

The characterization campaign resulted in 7 to 13 results per element. The obtained data were first checked for compliance with the certification requirements, and then for their validity based on technical reasoning. For Al, Co, Cr, Fe, Mn and Ni only results obtained after total dissolution (i.e. using HF) or using solid sampling techniques were retained. 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 [6] 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 [6].

Briefly, individual results were ranked in increasing order:

$$(x_1, x_2, \dots, x_p)$$

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

$$x^* = \text{median } x_i (1, 2, \dots, p) \quad (5)$$

$$s^* = 1.483 \times \text{median } |x_i - x^*| \quad (1,2,\dots,p) \quad (6)$$

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

$$\delta = 1.5s^* \quad (7)$$

Each  $x_i$  and  $x_i^*$  were calculated where:

$$\text{if } x_i < x^* - \delta; \quad x_i^* = x^* - \delta \quad (8)$$

$$\text{if } x_i > x^* + \delta; \quad x_i^* = x^* + \delta \quad (9)$$

$$\text{Otherwise:} \quad x_i^* = x_i \quad (10)$$

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

$$x^* = \sum x_i^* / p \quad (11)$$

$$s^* = 1,134 \sqrt{(x_i - x^*)^2 / (p-1)} \quad (12)$$

The robust estimates of  $x^*$  and  $s^*$  were calculated by iteration by 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 means. No significant differences were observed and the certified values obtained with the robust means approach were further used. These values are considered to be the most reliable estimates of the property values.

The uncertainties associated with the certified values were calculated according to the ISO Guide 35 [4]. The relative combined uncertainty of the certified value of the CRM consists of uncertainty related to characterization ( $u_{char}$ ), between-bottle inhomogeneity ( $u_{hom}$ ) and long-term stability ( $u_{stab}$ ). These different contributions were combined to estimate the expanded relative uncertainty.

$$U_{CRM,rel} = k \sqrt{u_{char}^2 + u_{hom}^2 + u_{stab}^2} \quad (13)$$

Where  $k$  is a coverage factor ( $k=2$ ), 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 the Table 2;

$u_{stab}$  the stability during storage period was chosen as 1%, which, as described before, ensured the validity of the certificate for at least 3 years;

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

$$u_{char} = 1.25 * \frac{\sqrt{\sum_{i=1}^p u_i^2}}{p} \quad (14)$$

Where:

$u_i$  is the combined uncertainty provided by participating laboratories [6];

$p$  is the number of laboratories.

As shown previously in Figure 1, the methods with different quantification steps (AAS, ET-AAS, AFS and ICP-MS) as well as methods without a sample preparation step such as INAA, Solid Sampling AAS and X ray Fluorescence were used for the 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 participants for trace elements mass fractions grouped by methods are displayed in Figures 3 to 16 and in Tables 5 to 18 (Appendix). The detailed results as reported by participants are shown in Appendix. In all figures, the reported results are plotted versus the certified values, which are denoted by a bold line, while the dashed lines represent the expanded uncertainty ( $k=2$ ) associated with the certified 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 deriving the certified values presented in Table 3.

Additional information for the mass fraction of 8 elements (Ba, Ce, La, Li, Na, Sb, Sc and Sr) classified as informative values is presented in Table 4. Presented results do not fulfil the certification criteria completely (i.e. reported without QC or without stated traceability). Informative values are the best estimate of the true value and they are provided with associated uncertainties. The combined uncertainty of informative values may not include all sources of uncertainty, or may reflect a lack of sufficient statistical agreement among multiple analytical methods.

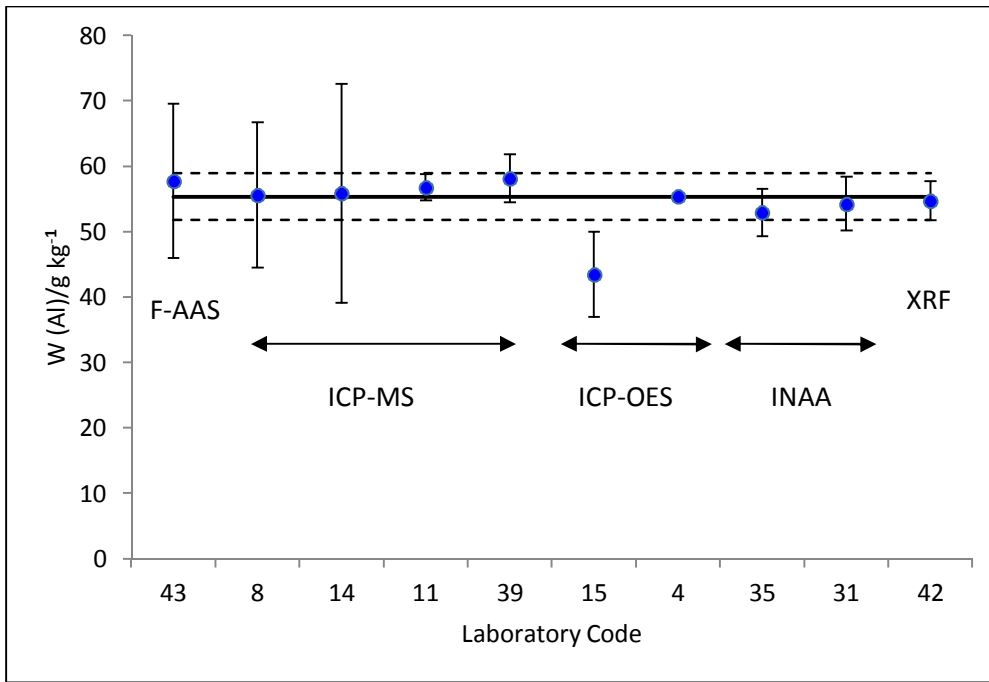


FIG. 3. Laboratory results for aluminium mass fraction ( $\text{g kg}^{-1}$ ) in the IAEA-456 sediment sample. The robust mean (solid line) and corresponding expanded uncertainty ( $k=2$ ) (dashed line) are shown. The error bars correspond to the expanded uncertainty reported by each laboratory.

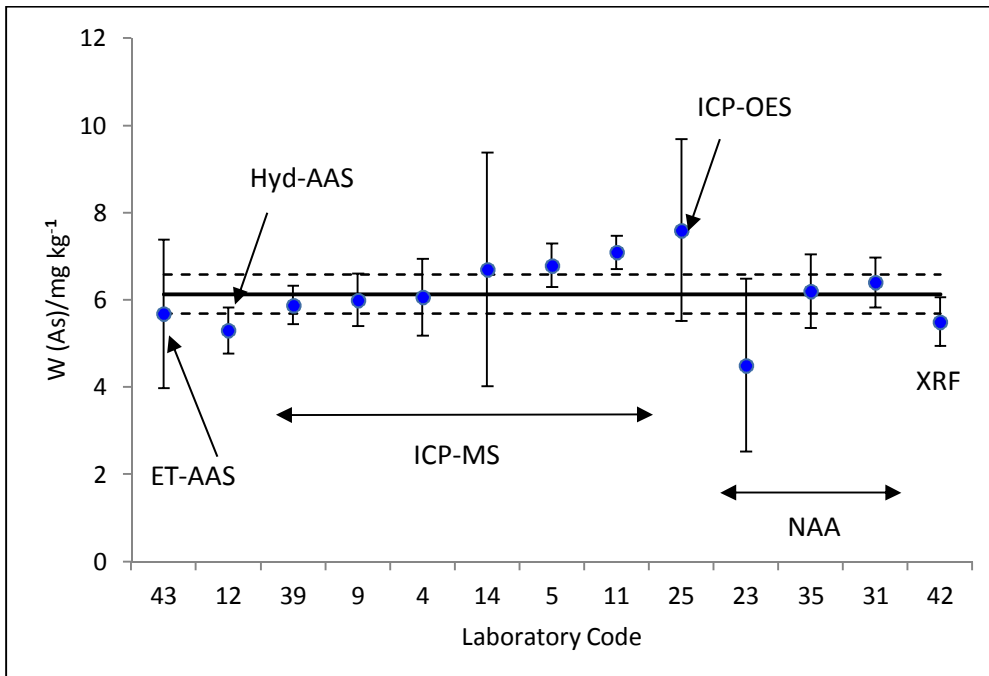


FIG.4 Laboratory results for arsenic mass fraction ( $\text{mg kg}^{-1}$ ) in the IAEA-456 sediment sample. The robust mean (solid line) and corresponding expanded uncertainty ( $k=2$ ) (dashed line) are shown. The error bars correspond to the expanded uncertainty reported by each laboratory.

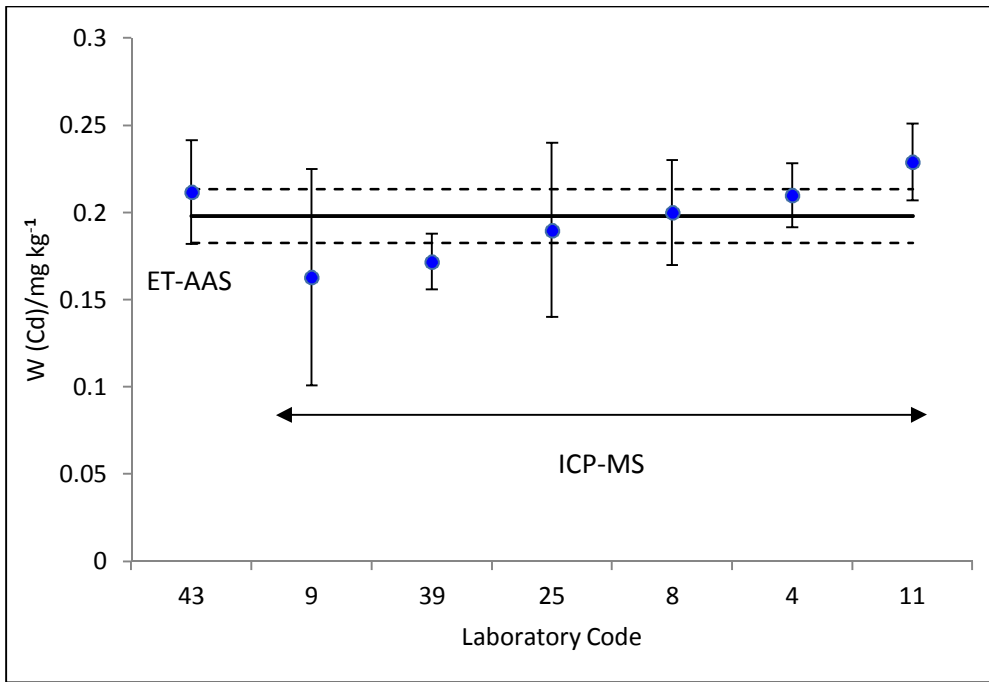


FIG. 5. Laboratory results for cadmium mass fraction ( $\text{mg kg}^{-1}$ ) in the IAEA-456 sediment sample. The robust mean (solid line) and corresponding expanded uncertainty ( $k=2$ ) (dashed line) are shown. The error bars correspond to the expanded uncertainty reported by each laboratory.

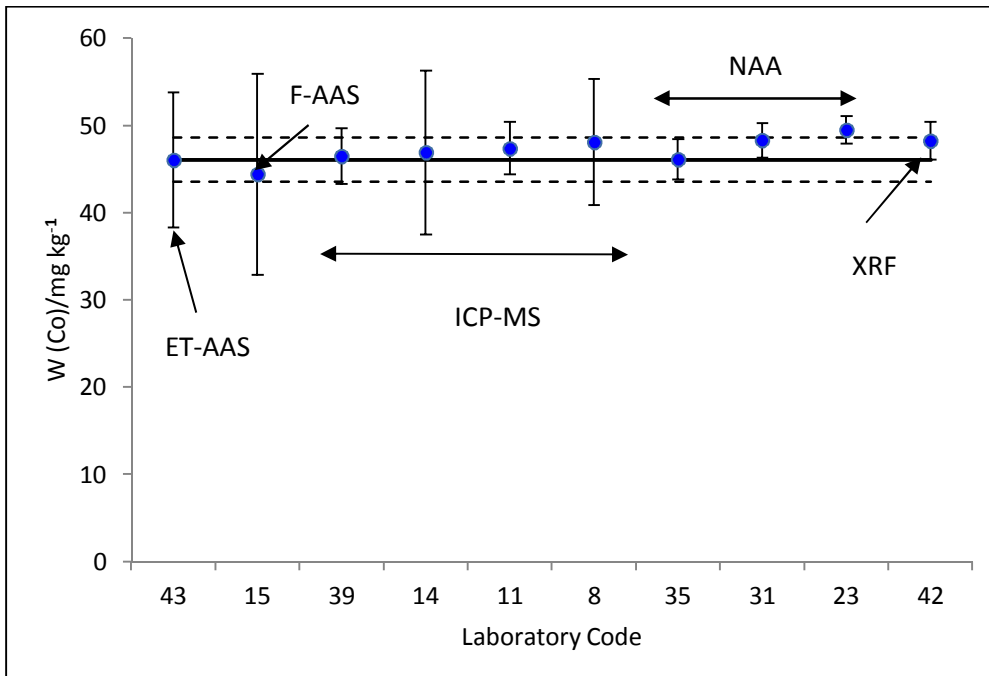


FIG. 6. Laboratory results for cobalt mass fraction ( $\text{mg kg}^{-1}$ ) in the IAEA-456 sediment sample. The robust mean (solid line) and corresponding expanded uncertainty ( $k=2$ ) (dashed line) are shown. The error bars correspond to the expanded uncertainty reported by each laboratory.

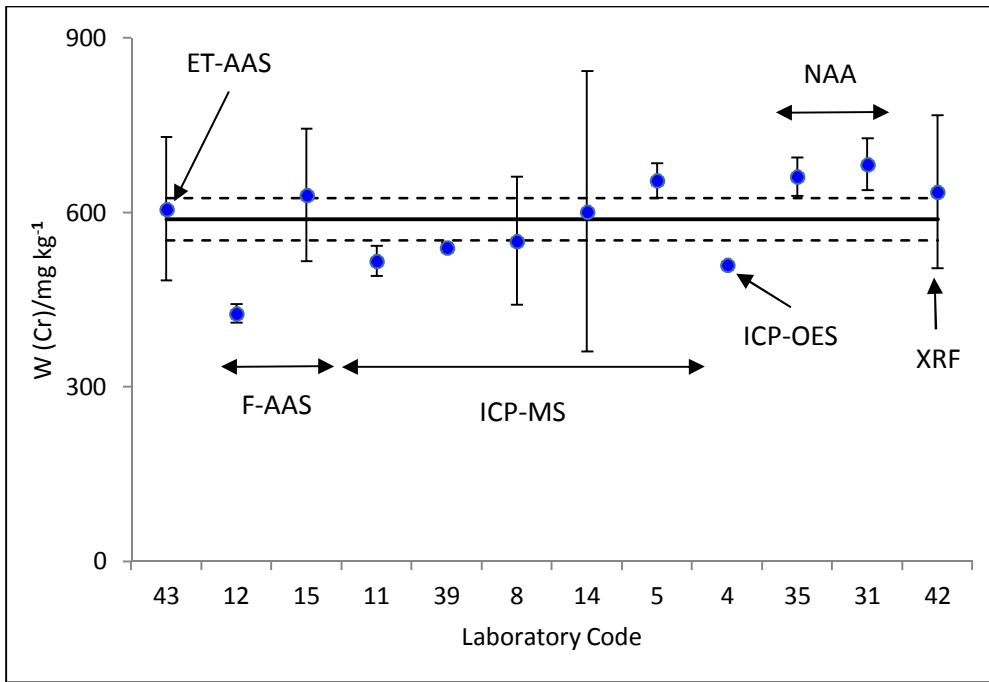


FIG. 7. Laboratory results for chromium mass fraction ( $\text{mg kg}^{-1}$ ) in the IAEA-456 sediment sample. The robust mean (solid line) and corresponding expanded uncertainty ( $k=2$ ) (dashed line) are shown. The error bars correspond to the expanded uncertainty reported by each laboratory.

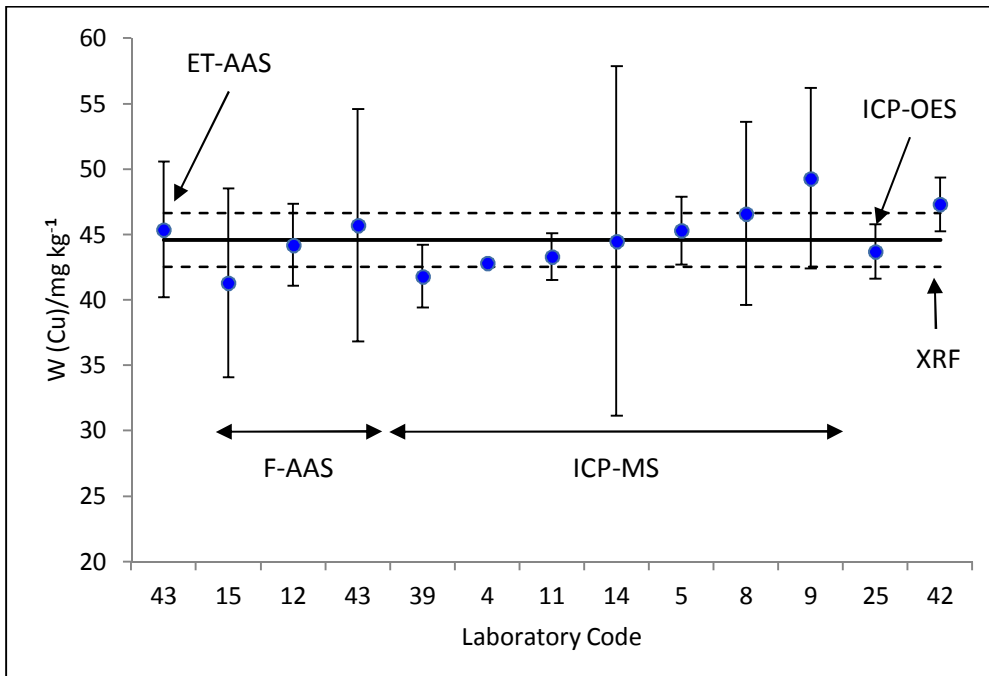


FIG. 8. Laboratory results for copper mass fraction ( $\text{mg kg}^{-1}$ ) in the IAEA-456 sediment sample. The robust mean (solid line) and corresponding expanded uncertainty ( $k=2$ ) (dashed line) are shown. The error bars correspond to the expanded uncertainty reported by each laboratory.

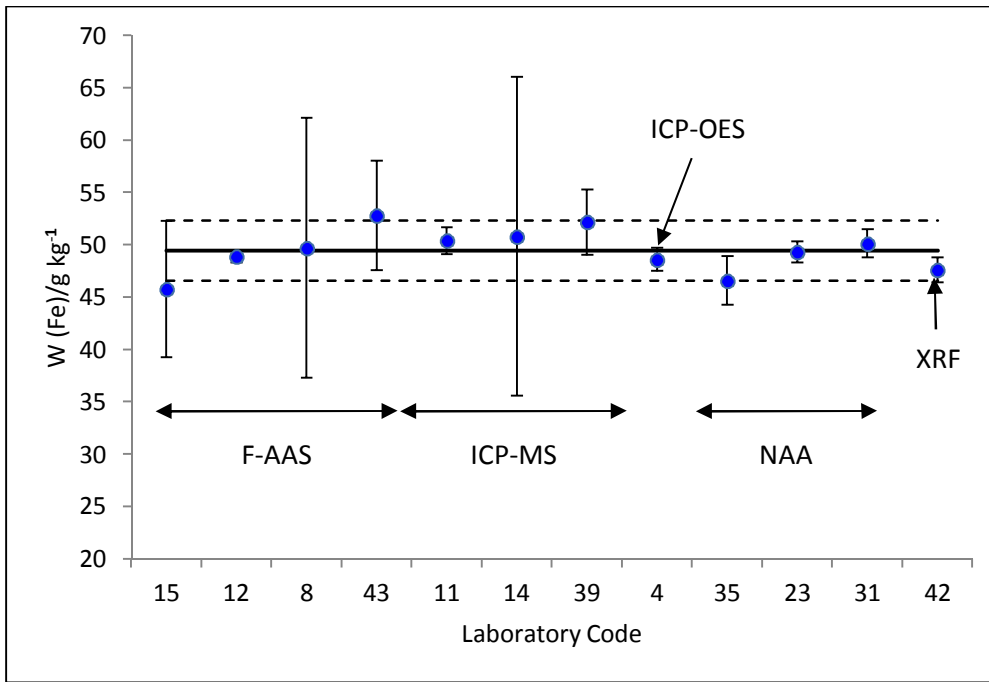


FIG. 9. Laboratory results for iron mass fraction ( $\text{g kg}^{-1}$ ) in the IAEA-456 sediment sample. The robust mean (solid line) and corresponding expanded uncertainty ( $k=2$ ) (dashed line) are shown. The error bars correspond to the expanded uncertainty reported by each laboratory.

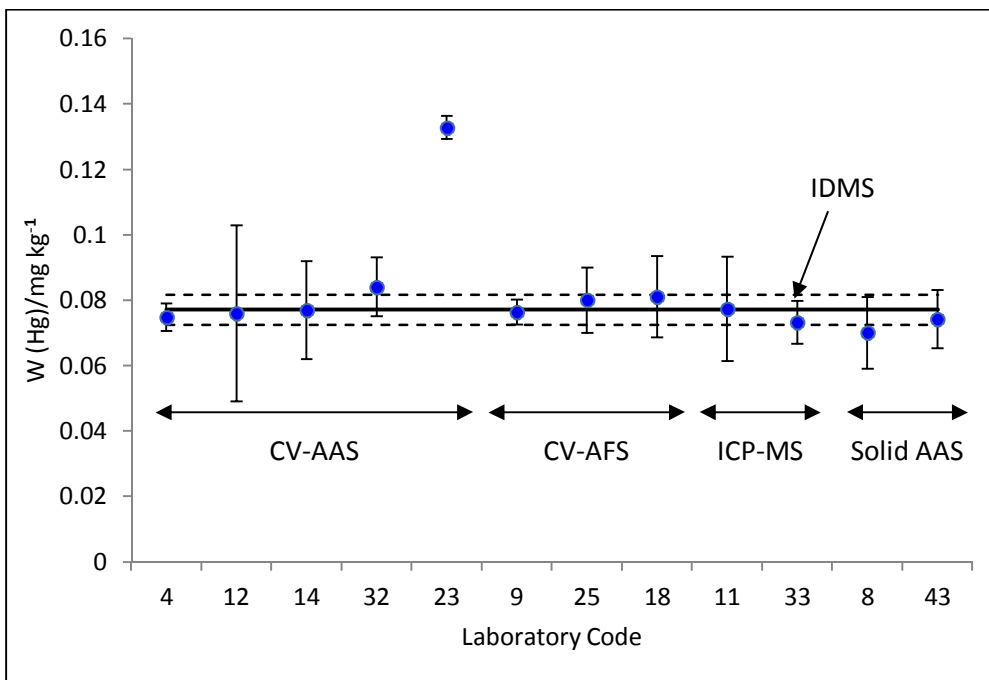


FIG. 10. Laboratory results for mercury mass fraction ( $\text{mg kg}^{-1}$ ) in the IAEA-456 sediment sample. The robust mean (solid line) and corresponding expanded uncertainty ( $k=2$ ) (dashed line) are shown. The error bars correspond to the expanded uncertainty reported by each laboratory.



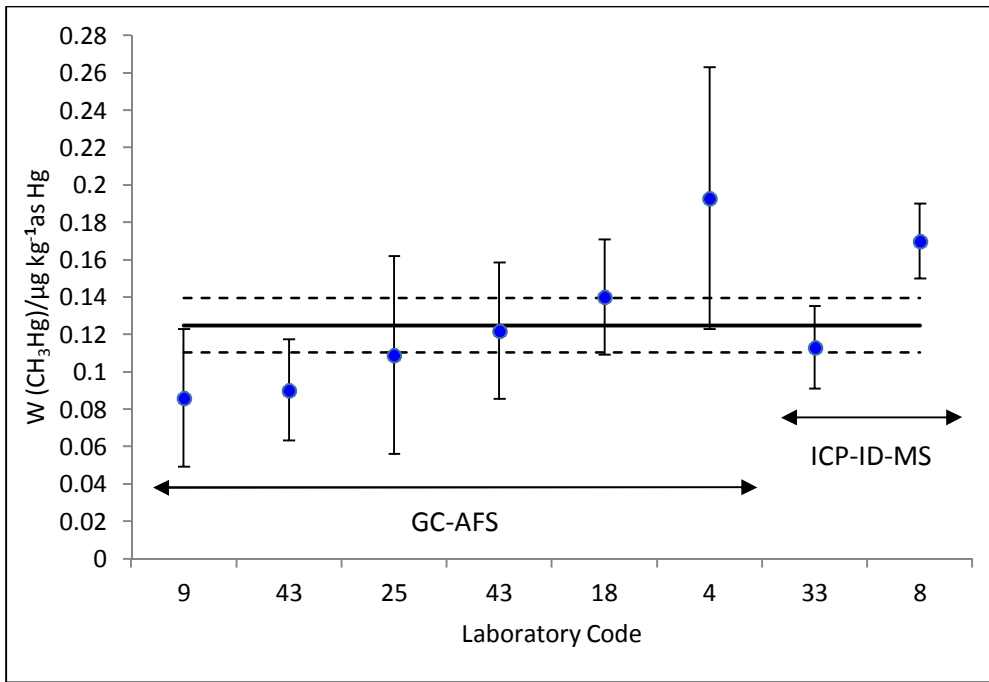


FIG. 11. Laboratory results for methyl mercury mass fraction ( $\mu\text{g kg}^{-1}$  as Hg) in the IAEA-456 sediment sample. The robust mean (solid line) and corresponding expanded uncertainty ( $k=2$ ) (dashed line) are shown. The error bars correspond to the expanded uncertainty reported by each laboratory.

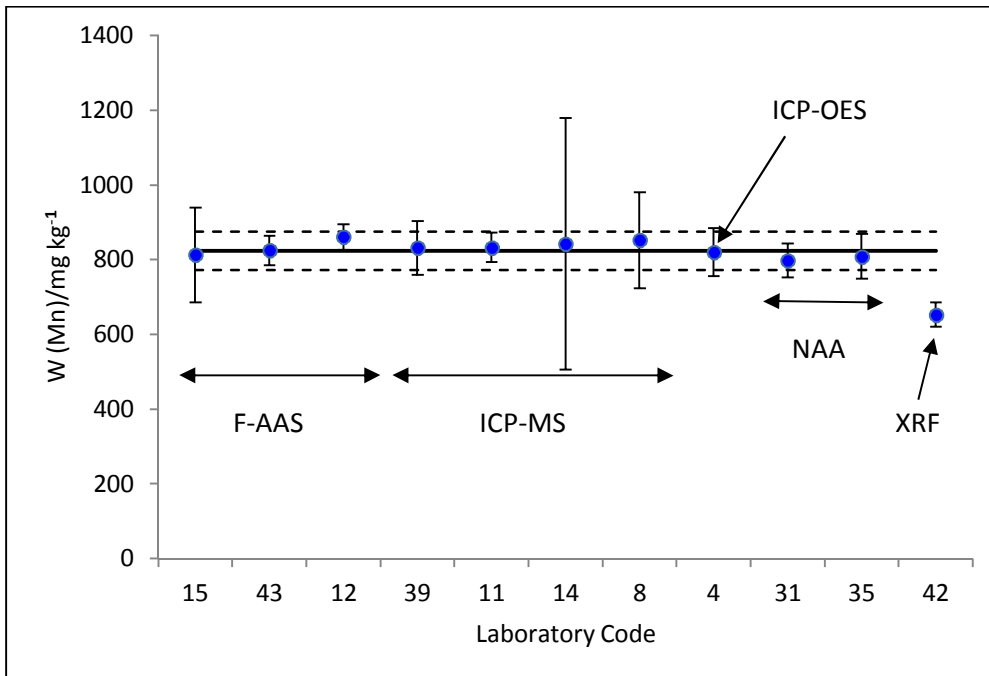


FIG. 12. Laboratory results for manganese mass fraction ( $\text{mg kg}^{-1}$ ) in the IAEA-456 sediment sample. The robust mean (solid line) and corresponding expanded uncertainty ( $k=2$ ) (dashed line) are shown. The error bars correspond to the expanded uncertainty reported by each laboratory.

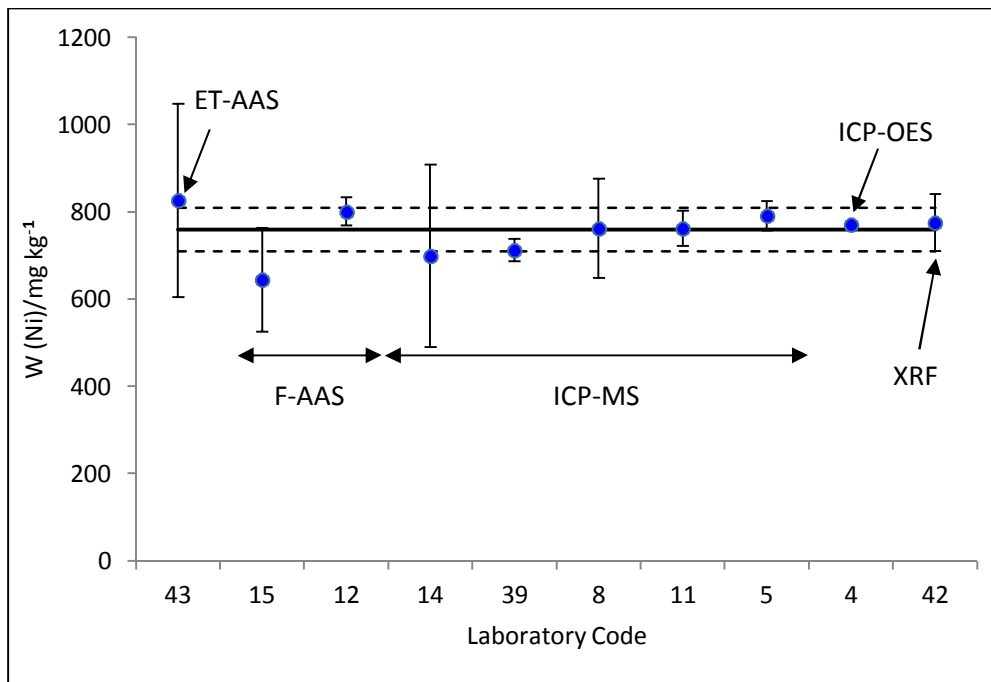


FIG. 13. Laboratory results for nickel mass fraction ( $\text{mg kg}^{-1}$ ) in the IAEA-456 sediment sample. The robust mean (solid line) and corresponding expanded uncertainty ( $k=2$ ) (dashed line) are shown. The error bars correspond to the expanded uncertainty reported by each laboratory.

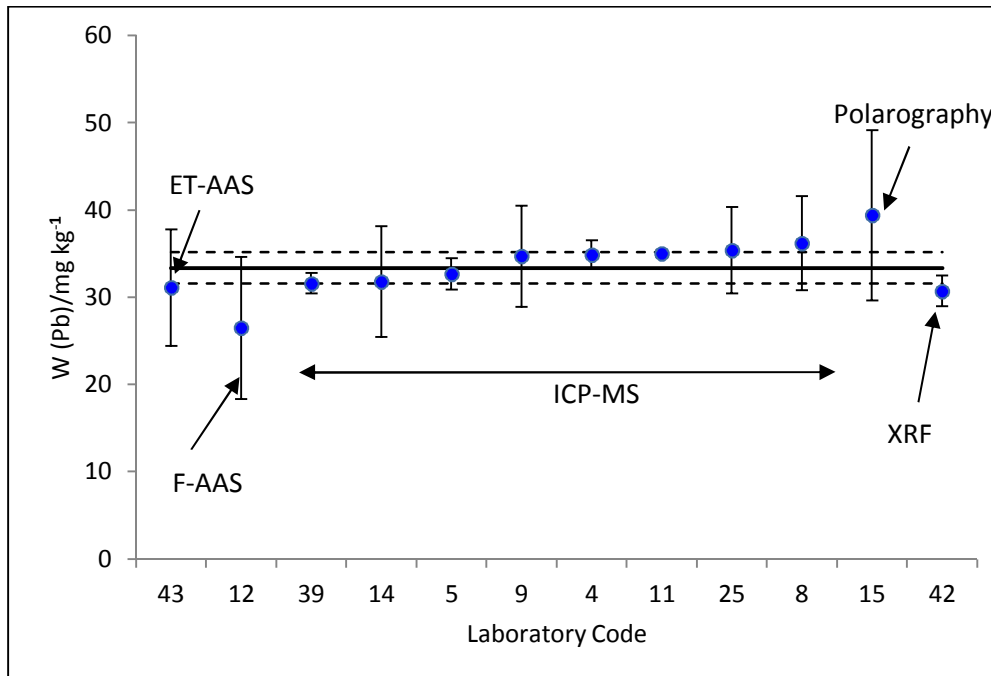


FIG. 14. Laboratory results for lead mass fraction ( $\text{mg kg}^{-1}$ ) in the IAEA-456 sediment sample. The robust mean (solid line) and corresponding expanded uncertainty ( $k=2$ ) (dashed line) are shown. The error bars correspond to the expanded uncertainty reported by each laboratory.

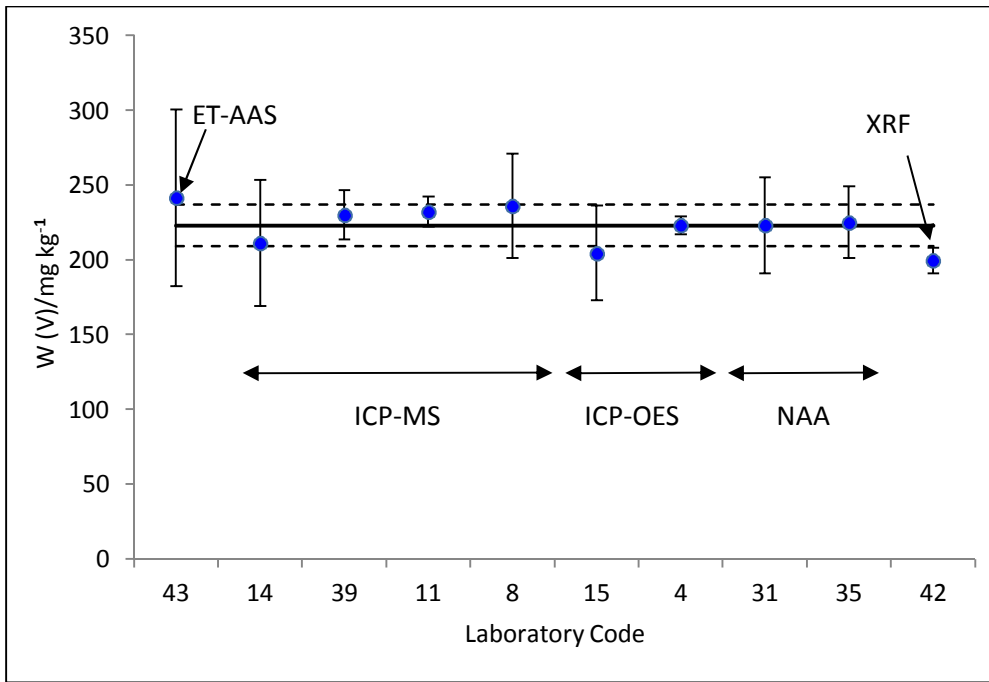


FIG. 15. Laboratory results for vanadium mass fraction ( $\text{mg kg}^{-1}$ ) in the IAEA-456 sediment sample. The robust mean (solid line) and corresponding expanded uncertainty ( $k=2$ ) (dashed line) are shown. The error bars correspond to the expanded uncertainty reported by each laboratory.

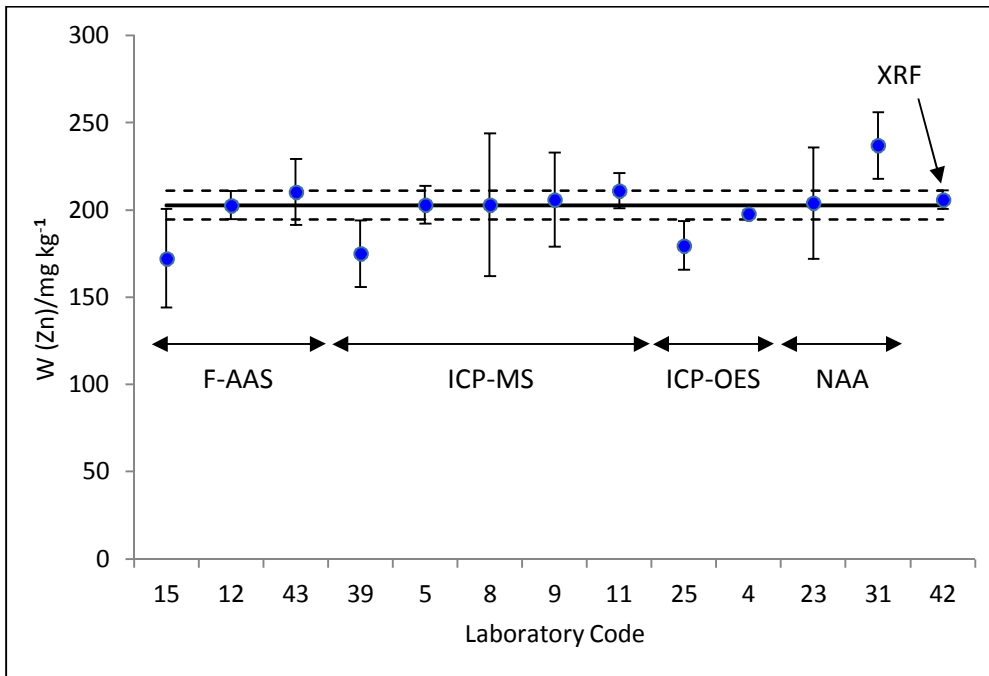


FIG. 16. Laboratory results for zinc mass fraction ( $\text{mg kg}^{-1}$ ) in the IAEA-456 sediment sample. The robust mean (solid line) and corresponding expanded uncertainty ( $k=2$ ) (dashed line) are shown. The error bars correspond to the expanded uncertainty reported by each laboratory.

#### 4. METROLOGICAL TRACEABILITY AND COMMUTABILITY

If the results obtained by different laboratories are to be compared, it is essential that all results are based on reliable measurement standards, the values of which are linked to the same 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, IAEA and NRC-CNRC were used for validation of the methods applied in this study.

Pure metal standard solutions (CRMs) with stated purity were employed for calibration from all 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 (in  $\mu\text{g kg}^{-1}$ ,  $\text{mg kg}^{-1}$  or  $\text{g kg}^{-1}$ ) is traceable to the SI system of units.

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

Commutability is a property of an RM, demonstrated by the closeness of agreement between the relation among the measurement results for a stated quantity in this material, obtained according to two given measurement procedures, and the relation obtained among the measurement results for other specified materials [7].

The appropriate characterization of CRMs, especially those materials intended to be used with routine measurement procedures, must carefully address fitness-for-use for all methods for which the material is intended to be used. Commutability is a critical requirement to avoid introducing unintended, and sometimes undetected, bias results when using a CRM.

Commutable CRMs should exhibit an analytical behaviour for a given method similar to a real laboratory sample. However, CRMs might show behaviour different from that of real samples, in particular during digestion, due to their small particle size in contrast to the possible larger particle size for real laboratory samples. IAEA-456 is a natural marine sediment material. The analytical behaviour will be the same as for a routine sample of dried marine sediments.

The agreement between results obtained with 13 different analytical methods selected for the IAEA-456 characterization study confirms the absence of any significant method bias and demonstrates commutability of the material for all certified trace elements in the IAEA-456 sediment sample.

## 5. CONCLUSIONS

This exercise allows assignment of certified values for Al, As, Cd, Cr, Co, Cu, Fe, Hg, CH<sub>3</sub>Hg, Mn, Ni, Pb, 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 combinations of SI traceable individual results, they are themselves traceable to SI. The produced sediment sample is suitable for the purposes of environment laboratories' quality control, and can be used as a sample for proficiency tests and interlaboratory comparisons. As any certified reference material, it can be used for validation studies.

The certified values for Al, As, Cd, Cr, Co, Cu, Fe, Hg, CH<sub>3</sub>Hg, Mn, Ni, Pb, V and Zn are presented in Table 3, together with their expanded uncertainty ( $k=2$ ). Information values for 8 elements are presented in Table 4.

TABLE 3. CERTIFIED VALUES FOR TRACE ELEMENT MASS FRACTIONS AND THEIR EXPANDED UNCERTAINTY ( $k=2$ ) IN THE IAEA-456 SEDIMENT SAMPLE

Element	Unit	Certified value <sup>1</sup>	U ( $k=2$ )	N° of data
Al	g kg <sup>-1</sup>	55.4	4.0	10
As	mg kg <sup>-1</sup>	6.14	0.52	13
Cd	mg kg <sup>-1</sup>	0.198	0.02	7
Co	mg kg <sup>-1</sup>	47.2	2.5	10
Cr	mg kg <sup>-1</sup>	589	42	12
Cu	mg kg <sup>-1</sup>	44.6	2.4	13
Fe	g kg <sup>-1</sup>	49.5	3.2	12
Hg	mg kg <sup>-1</sup>	0.077	0.005	12
CH <sub>3</sub> Hg	µg kg <sup>-1</sup> as Hg	0.125	0.019	8
Mn	mg kg <sup>-1</sup>	825	52	11
Ni	mg kg <sup>-1</sup>	760	56	10
Pb	mg kg <sup>-1</sup>	33.4	2.1	12
V	mg kg <sup>-1</sup>	223	16	10
Zn	mg kg <sup>-1</sup>	203	9	13

<sup>1</sup> The value is the robust mean of accepted sets of data, each set being obtained by a different laboratory. The certified values are reported on dry mass basis and are traceable to the SI.

<sup>2</sup> Expanded uncertainty with a coverage factor  $k=2$  according to the Evaluation of measurement data – Guide to the expression of uncertainty in measurement JCGM100:2008 [2], corresponding to the level of confidence of about 95%.

TABLE 4. INFORMATION VALUES FOR TRACE ELEMENT MASS FRACTIONS AND THEIR EXPANDED UNCERTAINTY ( $k=2$ ) IN THE IAEA-456 SEDIMENT SAMPLE

Element	Unit	Informative value <sup>1</sup>	U ( $k=2$ ) <sup>2</sup>	N° of data	N° of method
Ba	mg kg <sup>-1</sup>	283	18	5	3
Ce	mg kg <sup>-1</sup>	15.1	3.0	5	3
La	mg kg <sup>-1</sup>	6.6	0.3	4	1
Li	mg kg <sup>-1</sup>	19.8	2.0	4	3
Na	g kg <sup>-1</sup>	29.0	2.0	6	3
Sb	mg kg <sup>-1</sup>	0.61	0.09	4	2
Sc	mg kg <sup>-1</sup>	33.3	1.4	4	1
Sr	mg kg <sup>-1</sup>	273	12	5	3

<sup>1</sup> The value is the robust mean of accepted sets of data, each set being obtained by a different laboratory. The information values are reported in dry mass basis and are traceable to the SI.

<sup>2</sup> Expanded uncertainty with a coverage factor  $k=2$  according to the Evaluation of measurement data – Guide to the expression of uncertainty in measurement JCGM100:2008 [2], corresponding to the level of confidence of about 95%.

## APPENDIX

### DATA REPORT OF RESULTS SORTED BY ELEMENTS

TABLE 5. ALUMINIUM: RESULTS AS REPORTED BY PARTICIPANTS (g kg<sup>-1</sup>)

Lab code	Mean	Combined uncertainty (u)	Expanded uncertainty (U)
4	55.4	0.1	0.2
8	55.6	5.6	11.1
11	56.8	1.0	2.0
14	55.9	8.4	16.8
15	43.5	3.3	6.5
31	54.3	2.1	4.1
35	52.9	1.8	3.6
39	58.2	1.8	3.7
42	54.7	1.5	3.0
43	57.7	5.9	11.8

TABLE 6. ARSENIC: RESULTS AS REPORTED BY PARTICIPANTS (mg kg<sup>-1</sup>)

Lab code	Mean	Combined uncertainty (u)	Expanded uncertainty (U)
4	6.06	0.44	0.88
5	6.79	0.25	0.50
9	6.00	0.14	0.60
11	7.09	0.19	0.38
12	5.30	0.26	0.53
14	6.70	1.34	2.68
23	4.50	0.99	1.98
25	7.60	1.04	2.09
31	6.40	0.28	0.57
35	6.20	0.42	0.84
39	5.88	0.22	0.44
42	5.50	0.28	0.56
43	5.68	0.85	1.70

TABLE 7. CADMIUM: RESULTS AS REPORTED BY PARTICIPANTS (mg kg<sup>-1</sup>)

Lab code	Mean	Combined uncertainty (u)	Expanded uncertainty (U)
4	0.210	0.009	0.018
8	0.200	0.020	0.030
9	0.163	0.015	0.062
11	0.229	0.011	0.022
25	0.190	0.030	0.050
39	0.172	0.008	0.016
43	0.212	0.015	0.030

TABLE 8. COBALT: RESULTS AS REPORTED BY PARTICIPANTS (mg kg<sup>-1</sup>)

Lab code	Mean	Combined uncertainty (u)	Expanded uncertainty (U)
4	38.0	0.6	1.1
8	48.1	3.6	7.2
9	40.3	0.5	2.0
11	47.4	1.5	3.0
14	46.9	4.7	9.4
15	44.4	5.8	11.5
23	49.5	0.8	1.6
25	35.6	1.4	2.9
31	48.3	1.0	2.0
35	46.1	1.2	2.3
39	46.5	1.6	3.2
42	48.3	1.1	2.2
43	46.0	3.9	7.8

TABLE 9. CHROMIUM: RESULTS AS REPORTED BY PARTICIPANTS (mg kg<sup>-1</sup>)

Lab code	Mean	Combined uncertainty (u)	Expanded uncertainty (U)
4	510	3	5
5	655	15	30
8	551	55	110
11	517	13	26
12	427	8	16
14	602	120	241
15	630	57	114
31	683	22	45
35	662	17	33
39	540	4	8
42	636	66	132
43	606	62	123



TABLE 10. COPPER: RESULTS AS REPORTED BY PARTICIPANTS (mg kg<sup>-1</sup>)

Lab code	Mean	Combined uncertainty (u)	Expanded uncertainty (U)
4	42.8	0.1	0.2
5	45.3	1.3	2.6
8	46.6	3.5	7.0
9	49.3	1.6	6.9
11	43.3	0.9	1.8
12	44.2	1.6	3.1
14	44.5	6.7	13.4
15	41.3	3.6	7.2
25	43.7	1.0	2.1
39	41.8	1.2	2.4
42	47.3	1.0	2.0
43	45.4	2.6	5.2
43	45.7	4.4	8.9

TABLE 11. IRON: RESULTS AS REPORTED BY PARTICIPANTS (g kg<sup>-1</sup>)

Lab code	Mean	Combined uncertainty (u)	Expanded uncertainty (U)
4	48.6	0.6	1.1
8	49.7	6.2	12.4
11	50.4	0.6	1.3
12	48.9	0.3	0.5
14	50.8	7.6	15.2
15	45.7	3.3	6.5
23	49.3	0.5	1.0
31	50.1	0.7	1.4
35	46.6	1.2	2.3
39	52.2	1.6	3.1
42	47.6	0.6	1.2
43	52.8	2.6	5.2

TABLE 12. MERCURY: RESULTS AS REPORTED BY PARTICIPANTS (mg kg<sup>-1</sup>)

Lab code	Mean	Combined uncertainty (u)	Expanded uncertainty (U)
4	0.075	0.002	0.004
8	0.070	0.005	0.011
9	0.076	0.001	0.004
11	0.077	0.008	0.016
12	0.076	0.013	0.027
14	0.077	0.008	0.015
18	0.081	0.006	0.012
23	0.133	0.002	0.004
25	0.080	0.010	0.010
32	0.084	0.005	0.009
33	0.073	0.003	0.007
43	0.074	0.004	0.009

TABLE 13. METHYLMERCURY: RESULTS AS REPORTED BY PARTICIPANTS (µg kg<sup>-1</sup> as Hg)

Lab code	Mean	Combined uncertainty (u)	Expanded uncertainty (U)
4	0.193	0.035	0.070
8	0.170	0.010	0.020
9	0.086	0.009	0.037
18	0.140	0.015	0.031
25	0.109	0.027	0.053
33	0.113	0.011	0.022
43	0.090	0.014	0.027
43	0.122	0.018	0.037

TABLE 14. MANGANESE: RESULTS AS REPORTED BY PARTICIPANTS (mg kg<sup>-1</sup>)

Lab code	Mean	Combined uncertainty (u)	Expanded uncertainty (U)
4	820	32	64
8	852	64	128
11	833	20	40
12	861	17	34
14	843	169	337
15	813	63	127
31	798	22	45
35	809	30	60
39	832	36	72
42	653	16	33
43	825	20	39

TABLE 15. NICKEL: RESULTS AS REPORTED BY PARTICIPANTS (mg kg<sup>-1</sup>)

Lab code	Mean	Combined uncertainty (u)	Expanded uncertainty (U)
4	771	4	7
5	791	17	34
8	762	57	114
11	762	20	40
12	801	16	33
14	699	105	210
15	644	60	119
39	712	13	26
42	775	33	65
43	826	111	222

TABLE 16. LEAD: RESULTS AS REPORTED BY PARTICIPANTS (mg kg<sup>-1</sup>)

Lab code	Mean	Combined uncertainty (u)	Expanded uncertainty (U)
4	34.9	0.8	1.6
5	32.7	0.9	1.8
8	36.2	2.7	5.4
9	34.7	1.3	5.8
11	35.0	0.2	0.4
12	26.5	4.1	8.1
14	31.8	3.2	6.4
15	39.4	4.9	9.8
25	35.4	2.5	5.0
39	31.6	0.6	1.2
42	30.7	0.9	1.8
43	31.1	3.3	6.7

TABLE 17. VANADIUM: RESULTS AS REPORTED BY PARTICIPANTS (mg kg<sup>-1</sup>)

Lab code	Mean	Combined uncertainty (u)	Expanded uncertainty (U)
4	223	3	6
8	236	18	35
11	232	5	10
14	211	21	42
15	205	16	32
31	223	16	32
35	225	12	24
39	230	8	16
42	199	4	9
43	241	30	59

TABLE 18. ZINC RESULTS AS REPORTED BY PARTICIPANTS (mg kg<sup>-1</sup>)

Lab code	Mean	Combined uncertainty (u)	Expanded uncertainty (U)
4	198	1	2
5	203	6	11
8	203	20	41
9	206	6	27
11	211	5	10
12	203	4	8
15	172	14	28
23	204	16	32
25	180	7	14
31	237	10	19
39	175	10	19
42	206	3	5
43	210	9	19



## REFERENCES

- [1] INTERNATIONAL ATOMIC ENERGY AGENCY, Worldwide Laboratory Comparison on the Determination of Trace Elements in IAEA-452 Biota Sample, IAEA Analytical Quality in Nuclear Applications Series No. 23, IAEA/AQ/23, IAEA, Vienna (2012).
- [2] JOINT COMMITTEE FOR GUIDES IN METROLOGY (JGCM), Evaluation of measurement data – Guide to the expression of uncertainty in measurement, JGCM 100:2008 (GUM with minor corrections), (2008).  
[http://www.bipm.org/utis/common/documents/jcgm/JCGM\\_100\\_2008\\_E.pdf](http://www.bipm.org/utis/common/documents/jcgm/JCGM_100_2008_E.pdf)
- [3] INTERNATIONAL ORGANIZATION FOR STANDARDIZATION, ISO Guide 34:2009, General requirements for the competence of reference material producers ISO, Geneva (2009).
- [4] INTERNATIONAL ORGANIZATION FOR STANDARDIZATION, ISO Guide 35:2006, Reference Materials – General and Statistical Principles for Certification, ISO, Geneva (2006).
- [5] LINSINGER, T., PAUWELS, J., VAN DER VEEN, A., SCHIMMEL, H., LAMBERTY, A., Homogeneity and stability of reference materials, *Accredit. Qual. Assur.* **61**, (2001) 20–25.
- [6] INTERNATIONAL ORGANIZATION FOR STANDARDIZATION, ISO 13528:2005, Statistical methods for use in proficiency testing by interlaboratory comparisons, ISO, Geneva (2005).
- [7] JOINT COMMITTEE FOR GUIDES IN METROLOGY (JGCM), International Vocabulary of Metrology – Basic and General Concepts and Associated Terms, VIM3, JGCM 200:2012 (JCGM 200:2008 with minor corrections), (2012).

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