IAEA/AQ/31

IAEA Analytical Quality in Nuclear Applications Series No. 31

Certification of Trace Element Mass Fractions in IAEA-458 Marine Sediment Sample

IAEA
International Atomic Energy Agency

CERTIFICATION OF TRACE ELEMENT MASS FRACTIONS IN IAEA-458 MARINE SEDIMENT SAMPLE

The following States are Members of the International Atomic Energy Agency:

AFGHANISTAN
ALBANIA
ALGERIA
ANGOLA
ARGENTINA
ARMENIA
AUSTRALIA
AUSTRIA
AZERBAIJAN
BAHRAIN
BANGLADESH
BELARUS
BELGIUM
BELIZE
BENIN
BOLIVIA
BOSNIA AND HERZEGOVINA
BOTSWANA
BRAZIL
BULGARIA
BURKINA FASO
BURUNDI
CAMBODIA
CAMEROON
CANADA
CENTRAL AFRICAN REPUBLIC
CHAD
CHILE
CHINA
COLOMBIA
CONGO
COSTA RICA
CÔTE D'IVOIRE
CROATIA
CUBA
CYPRUS
CZECH REPUBLIC
DEMOCRATIC REPUBLIC OF THE CONGO
DENMARK
DOMINICA
DOMINICAN REPUBLIC
ECUADOR
EGYPT
EL SALVADOR
ERITREA
ESTONIA
ETHIOPIA
FIJI
FINLAND
FRANCE
GABON
GEORGIA
GERMANY
GHANA
GREECE
GUATEMALA
HAITI
HOLY SEE
HONDURAS
HUNGARY
ICELAND
INDIA
INDONESIA
IRAN, ISLAMIC REPUBLIC OF
IRAQ
IRELAND
ISRAEL
ITALY
JAMAICA
JAPAN
JORDAN
KAZAKHSTAN
KENYA
KOREA, REPUBLIC OF
KUWAIT
KYRGYZSTAN
LAO PEOPLE'S DEMOCRATIC REPUBLIC
LATVIA
LEBANON
LESOTHO
LIBERIA
LIBYA
LIECHTENSTEIN
LITHUANIA
LUXEMBOURG
MADAGASCAR
MALAWI
MALAYSIA
MALI
MALTA
MARSHALL ISLANDS
MAURITANIA
MAURITIUS
MEXICO
MONACO
MONGOLIA
MONTENEGRO
MOROCCO
MOZAMBIQUE
MYANMAR
NAMIBIA
NEPAL
NETHERLANDS
NEW ZEALAND
NICARAGUA
NIGER
NIGERIA
NORWAY
OMAN
PAKISTAN
PALAU
PANAMA
PAPUA NEW GUINEA
PARAGUAY
PERU
PHILIPPINES
POLAND
PORTUGAL
QATAR
REPUBLIC OF MOLDOVA
ROMANIA
RUSSIAN FEDERATION
RWANDA
SAUDI ARABIA
SENEGAL
SERBIA
SEYCHELLES
SIERRA LEONE
SINGAPORE
SLOVAKIA
SLOVENIA
SOUTH AFRICA
SPAIN
SRI LANKA
SUDAN
SWAZILAND
SWEDEN
SWITZERLAND
SYRIAN ARAB REPUBLIC
TAJIKISTAN
THAILAND
THE FORMER YUGOSLAV REPUBLIC OF MACEDONIA
TOGO
TRINIDAD AND TOBAGO
TUNISIA
TURKEY
UGANDA
UKRAINE
UNITED ARAB EMIRATES
UNITED KINGDOM OF GREAT BRITAIN AND NORTHERN IRELAND
UNITED REPUBLIC OF TANZANIA
UNITED STATES OF AMERICA
URUGUAY
UZBEKISTAN
VENEZUELA
VIETNAM
YEMEN
ZAMBIA
ZIMBABWE

The Agency's Statute was approved on 23 October 1956 by the Conference on the Statute of the IAEA held at United Nations Headquarters, New York; it entered into force on 29 July 1957. The Headquarters of the Agency are situated in Vienna. Its principal objective is "to accelerate and enlarge the contribution of atomic energy to peace, health and prosperity throughout the world''.

IAEA Analytical Quality in Nuclear Applications No. IAEA/AQ/31

CERTIFICATION OF TRACE ELEMENT MASS FRACTIONS IN IAEA-458 MARINE SEDIMENT SAMPLE

INTERNATIONAL ATOMIC ENERGY AGENCY
VIENNA, 2013

COPYRIGHT NOTICE

For further information on this publication, please contact:

IAEA Environment Laboratories, Monaco
Marine Environmental Studies Laboratory
International Atomic Energy Agency
4a Quai Antoine 1er, MC 98000
Principality of Monaco

Printed by the IAEA in Austria
September 2013

CERTIFICATION OF TRACE ELEMENT MASS FRACTIONS IN IAEA-458
MARINE SEDIMENT SAMPLE
IAEA, VIENNA, 2013
IAEA/AQ/31
ISSN 2074–7659
© IAEA, 2013
Printed by the IAEA in Austria
September 2013

FOREWORD

The primary goal of the IAEA Environment Laboratories (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 the 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.

This report describes the sample preparation methodology, material homogeneity and stability study, selection of laboratories, evaluation of results from the certification campaign and assignment of property values and their associated uncertainty. As a result, reference values for mass fractions and associated expanded uncertainty for 16 trace elements (Al, As, Cd, Cr, Co, Cu, Fe, Hg, Li, Mn, Ni, Pb, Sr, Sn, V and Zn) in marine sediment were established.

The IAEA is grateful to the participants and the laboratories that took part in this interlaboratory comparison 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.

EDITORIAL NOTE

This publication has been prepared from the original material as submitted by the authors. The views expressed do not necessarily reflect those of the IAEA, the governments of the nominating Member States or the nominating organizations.

This publication has not been edited by the editorial staff of the IAEA. It does not address questions of responsibility, legal or otherwise, for acts or omissions on the part of any person.

The use of particular designations of countries or territories does not imply any judgement by the publisher, the IAEA, as to the legal status of such countries or territories, of their authorities and institutions or of the delimitation of their boundaries.

The mention of names of specific companies or products (whether or not indicated as registered) does not imply any intention to infringe proprietary rights, nor should it be construed as an endorsement or recommendation on the part of the IAEA.

The authors are responsible for having obtained the necessary permission for the IAEA to reproduce, translate or use material from sources already protected by copyrights.

The IAEA has no responsibility for the persistence or accuracy of URLs for external or third party Internet web sites referred to in this book and does not guarantee that any content on such web sites is, or will remain, accurate or appropriate..

CONTENTS

1. INTRODUCTION

Many laboratories are involved in the production of local or regional environmental 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 (CRM) 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 sub-programme '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 is still a noticeable lack of matrix CRMs.

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 April 2013 by the IAEA.

2. METHODOLOGY

2.1. COLLECTION AND PREPARATION OF THE MATERIAL

A sample of thirty four kg of sediment was delivered to the MESL 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 125 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 about 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-458. The average moisture content of the sample after bottling was determined by drying to a constant mass 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 of laboratories having a quality system in place, using validated methods, applying uncertainty and traceability concepts and having provided good results in previous IAEA inter-laboratory comparisons 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. Participants were requested to analyze Al, As, Cd, Cr, Co, Cu, Fe, Hg, Mn, Ni, Pb, Li, Sr, Sn, 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 element mass fractions in a CRM with a similar matrix to the candidate reference material. The moisture determination method was also prescribed.

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

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 in Reference [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 described conditions over a period of 2 years. One isochronous study over 4 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

The 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 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 summarized in Figure 1.

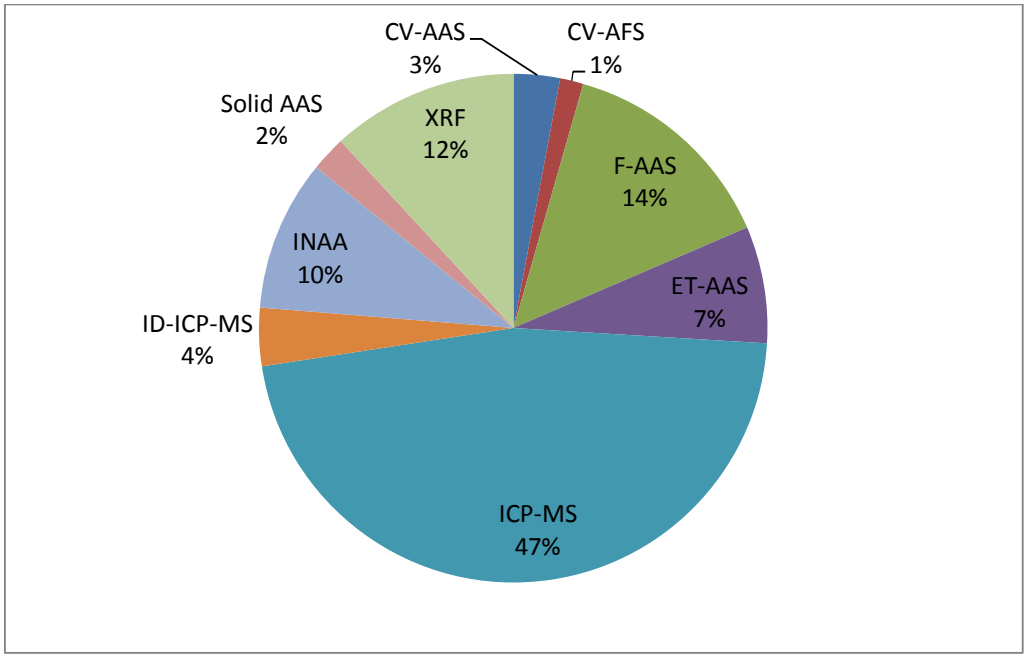

FIG. 1. Analytical methods used for the certification of trace elements in the IAEA-458 sediment sample.

TABLE 1. INSTRUMENTAL TECHNIQUES

Method code	**Instrumental technique**
F-AAS	Atomic Absorption Spectrometry-Flame
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
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) [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 CRM 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 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 2.1% ±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 the 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 Fe and Cr respectively was detected as outliers. These results were excluded as they were outliers 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 the 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}:

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

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

For all elements, except for Pb and Cr, MS_{bb} (ANOVA mean square between-bottles) was smaller than MS_{wb} (ANOVA mean square within-bottles) 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]:

$$u^*_{bb} = \frac{s_{wb}}{\sqrt{n}} \sqrt[4]{\frac{2}{\nu_{MSwb}}} \quad (3)$$

Where:

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

ν_{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 3.7%, 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 sized 0.2 g are presented in Table 2.

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

Element	Al	Fe	Hg	Cr	Cu	Mn	Zn	Pb	Ni
u_{wb}%	2.6	3.1	3.7	3.5	2.1	1.3	1.0	3.3	1.6
u^*_{bb}%	0.9	1.7	1.2	1.5	0.7	0.4	0.3	1.1	0.5

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 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 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 35:2006 [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 the short-term stability (4 weeks) for Cd and Hg obtained with isochronous approach.

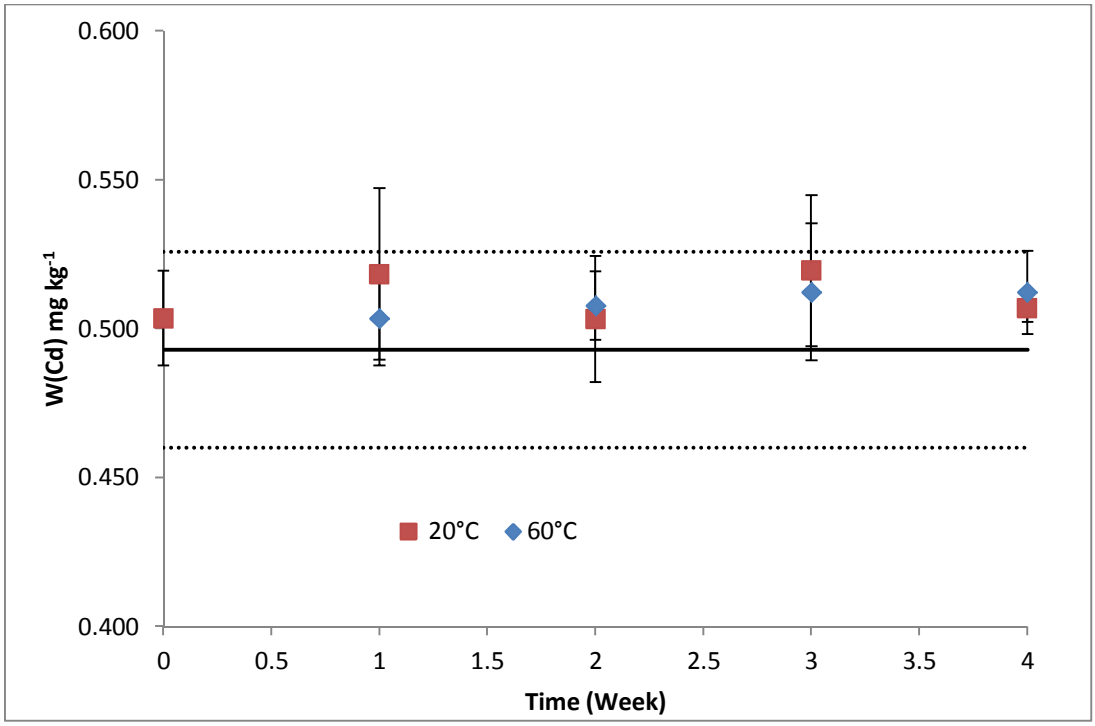

FIG. 1. Results obtained with isochronous approach for short-term stability studies of Cd in the sediment sample kept at 20°C and 60°C respectively.

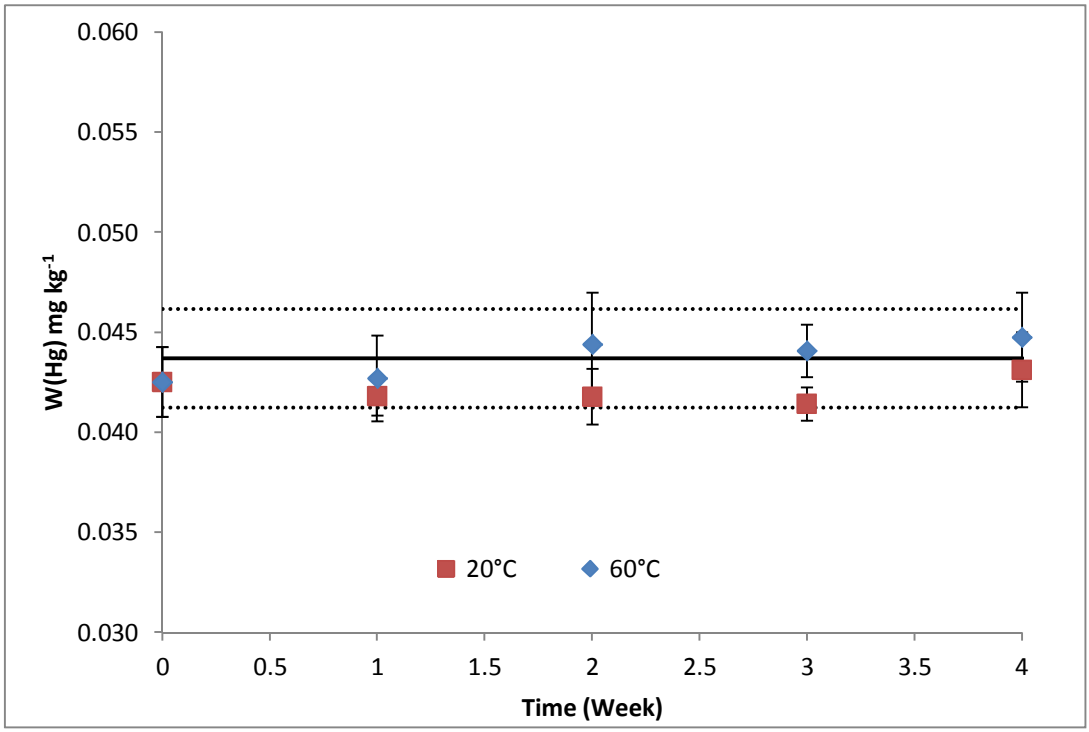

FIG. 2. Results obtained with isochronous approach for the short-term stability study of Hg in sediment samples kept at 20°C and 60°C respectively.

3.3. DETERMINATION OF CERTIFIED VALUES AND UNCERTAINTIES

The characterization campaign resulted from 7 to 16 results per element and 5 results for Li, Sn and Sr. The obtained data were first checked for compliance with the certification requirements, and then for their validity based on technical reasoning. All accepted set 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:

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

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

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

$$\delta = 1.5 s^* \quad (6)$$

Each x_i and x_i* were calculated where:

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

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

Otherwise: $x_i^* = x_i$ (9)

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

$$x^* = \Sigma x_i^* / p \quad (10)$$

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

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 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.

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

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 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 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];

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 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 4–19 and in Tables 4–19 (Appendix I). The detailed results as reported by participants are also 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 expended 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.

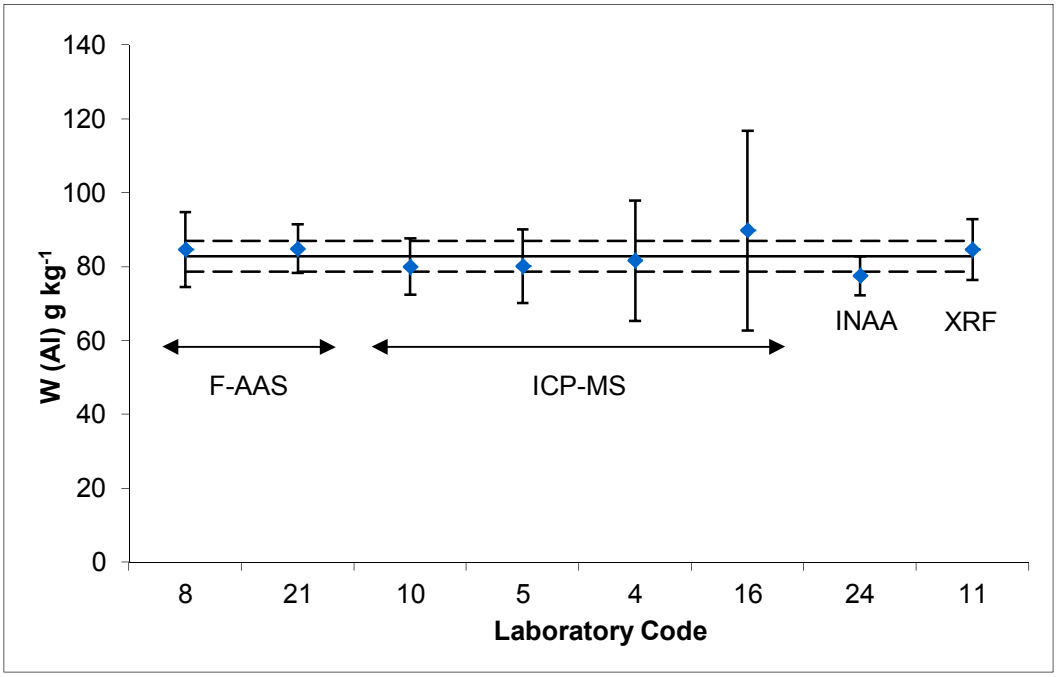

FIG. 3. Laboratory results for aluminum mass fraction (mg·kg^{-1}) in the IAEA-458 sediment sample. The robust mean (solid line) and corresponding expanded uncertainty (k=2) (dashed line) are shown. The error bars correspond to the expanded uncertainty reported by each laboratory.

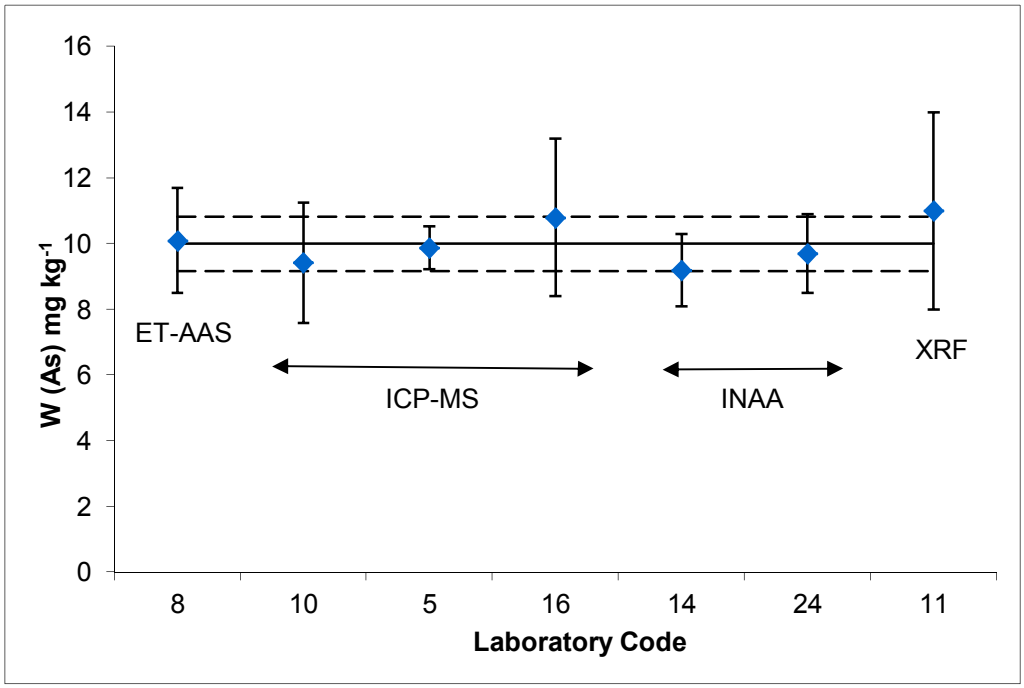

FIG. 4. Laboratory results for arsenic mass fraction (g·kg^{-1}) in the IAEA-458 sediment sample. The robust mean (solid line) and corresponding expanded uncertainty (k=2) (dashed line) are shown. The error bars correspond to the expanded uncertainty reported by each laboratory.

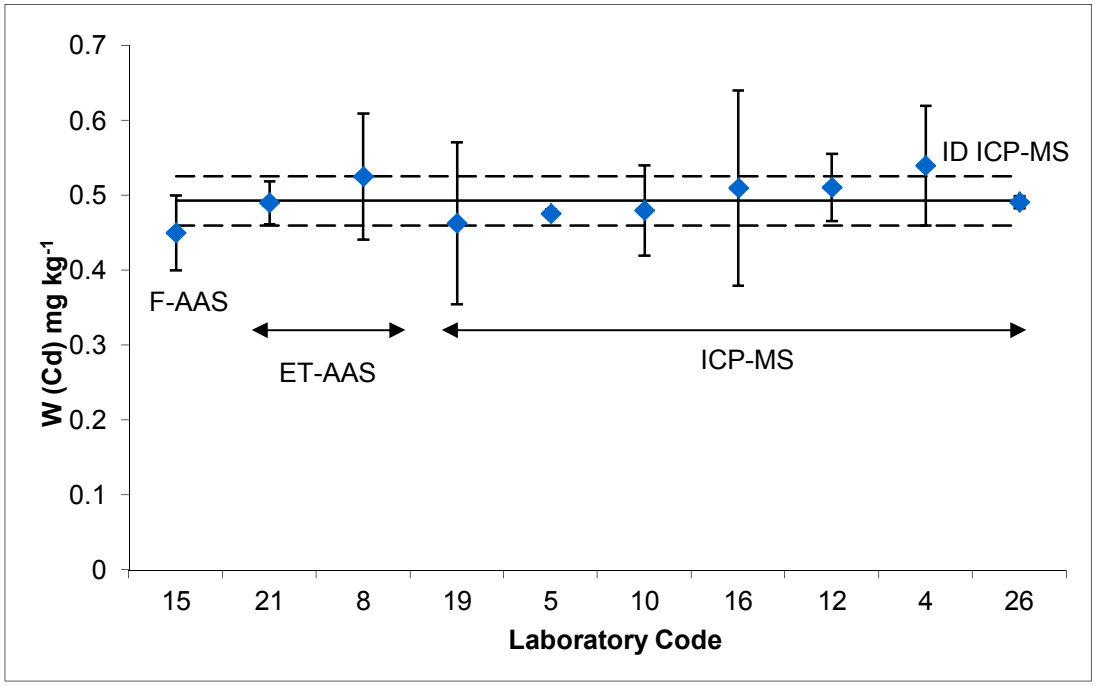

FIG. 5. Laboratory results for cadmium mass fraction (mg·kg^{-1}) in the IAEA-458 sediment sample. The robust mean (solid line) and corresponding expanded uncertainty (k=2) (dashed line) are shown. The error bars correspond to the expanded uncertainty reported by each laboratory.

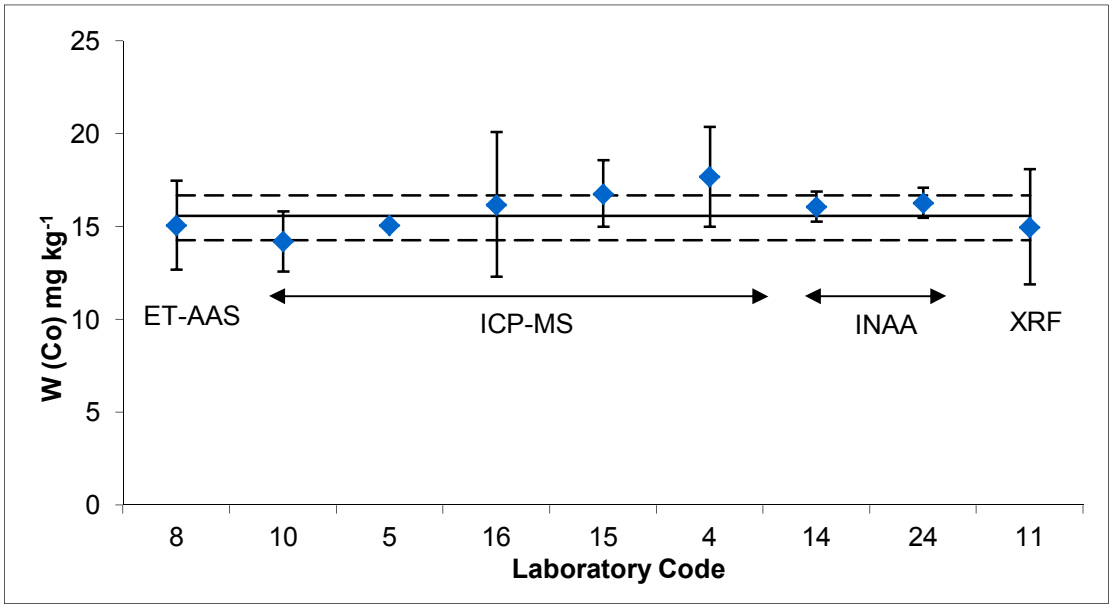

FIG. 6. Laboratory results for cobalt mass fraction (mg·kg^{-1}) in the IAEA-458 sediment sample. The robust mean (solid line) and corresponding expanded uncertainty (k=2) (dashed line) are shown. The error bars correspond to the expanded uncertainty reported by each laboratory.

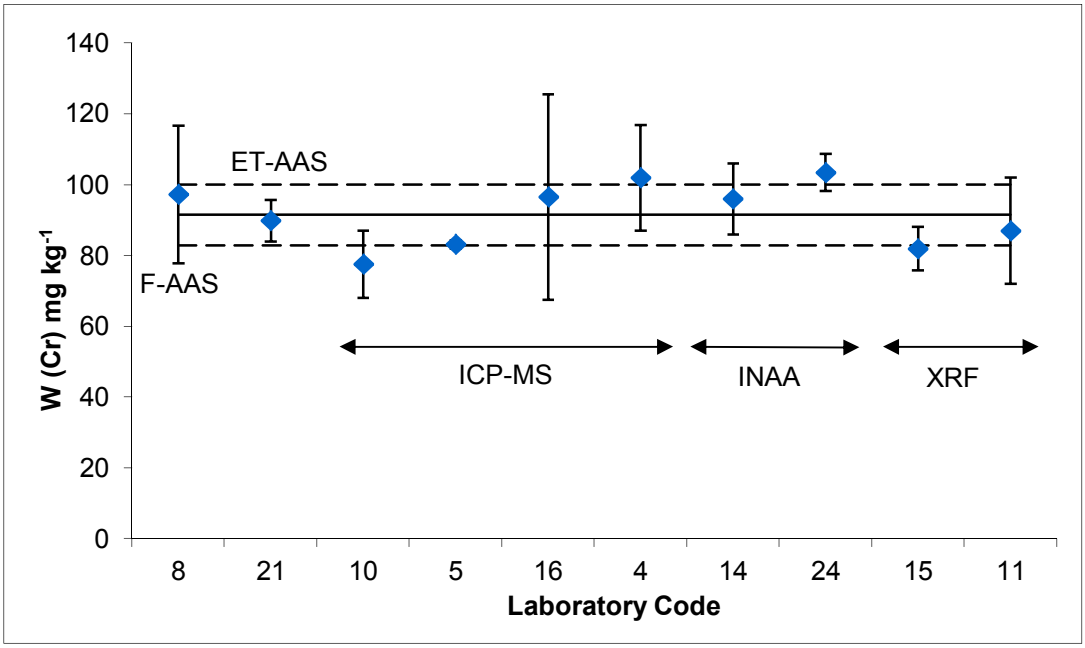

FIG. 7. Laboratory results for chromium mass fraction (mg·kg^{-1}) in the IAEA-458 sediment sample. The robust mean (solid line) and corresponding expanded uncertainty (k=2) (dashed line) are shown. The error bars correspond to the expanded uncertainty reported by each laboratory.

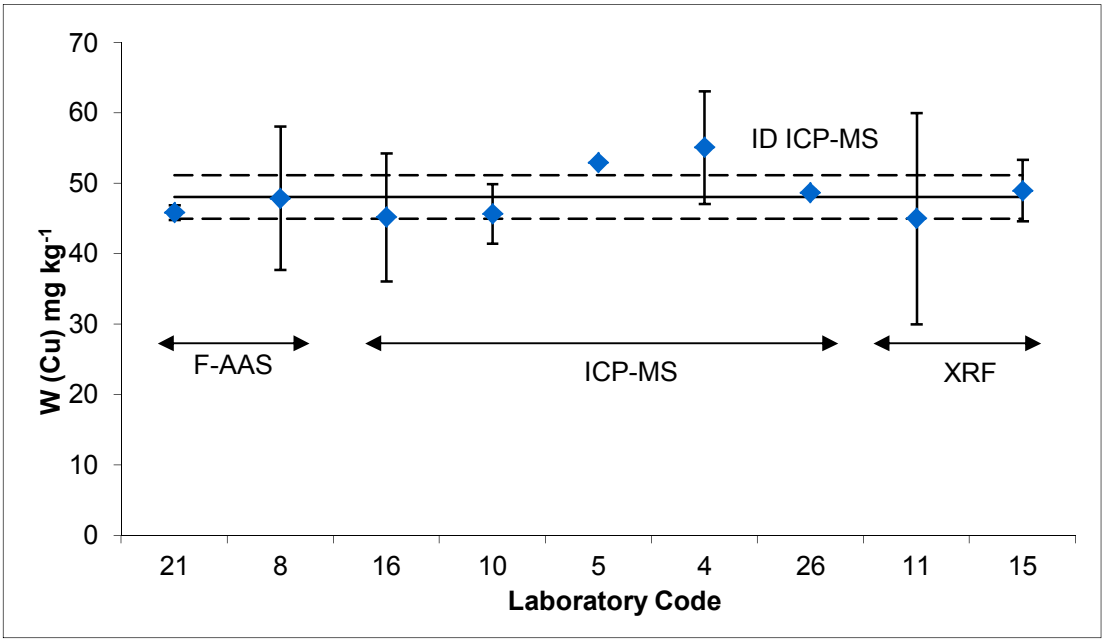

FIG. 8. Laboratory results for copper mass fraction (mg·kg^{-1}) in the IAEA-458 sediment sample. The robust mean (solid line) and corresponding expanded uncertainty (k=2) (dashed line) are shown. The error bars correspond to the expanded uncertainty reported by each laboratory.

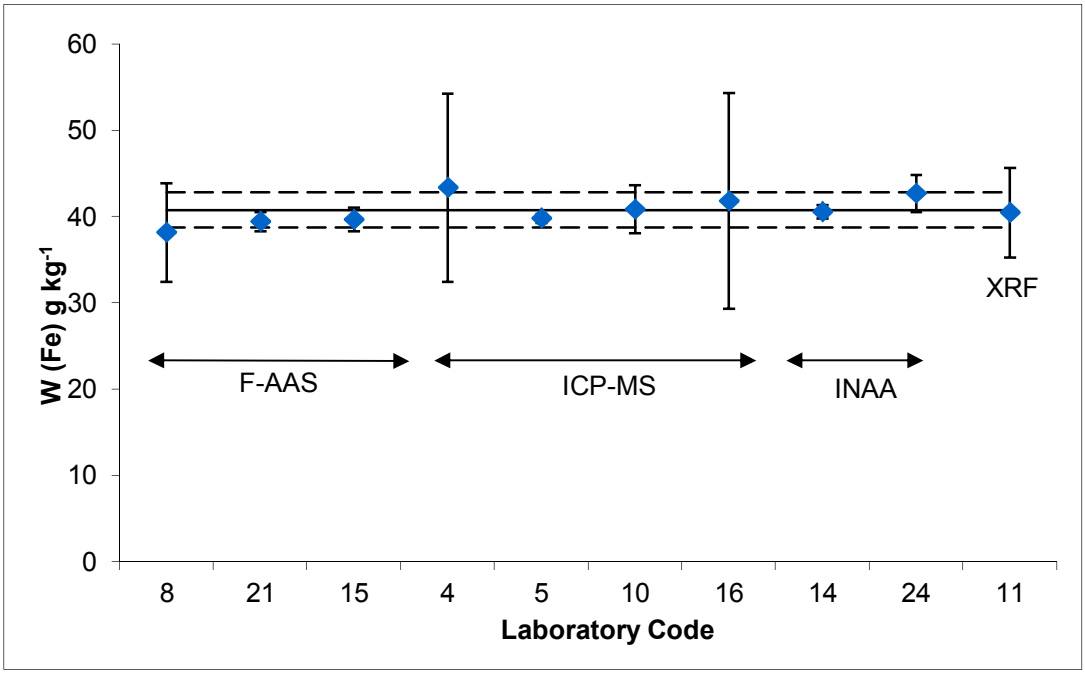

FIG. 9. Laboratory results for iron mass fraction ($g \cdot kg^{-1}$) in the IAEA-458 sediment sample. The robust mean (solid line) and corresponding expanded uncertainty (k=2) (dashed line) are shown. The error bars correspond to the expanded uncertainty reported by each laboratory.

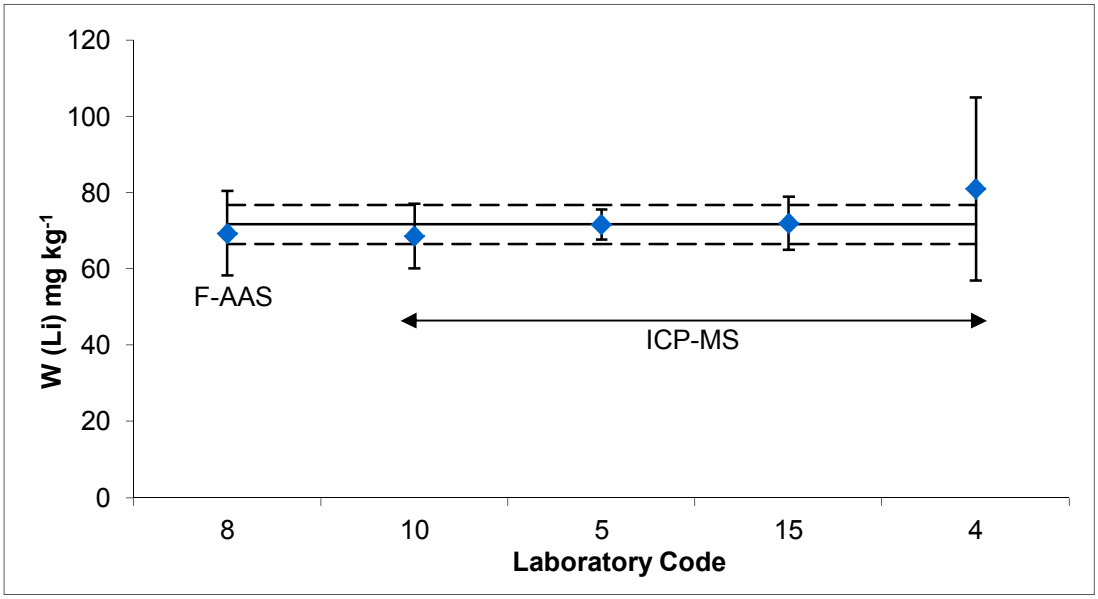

FIG. 10. Laboratory results for lithium mass fraction ($mg \cdot kg^{-1}$) in the IAEA-458 sediment sample. The robust mean (solid line) and corresponding expanded uncertainty (k=2) (dashed line) are shown. The error bars correspond to the expanded uncertainty reported by each laboratory.

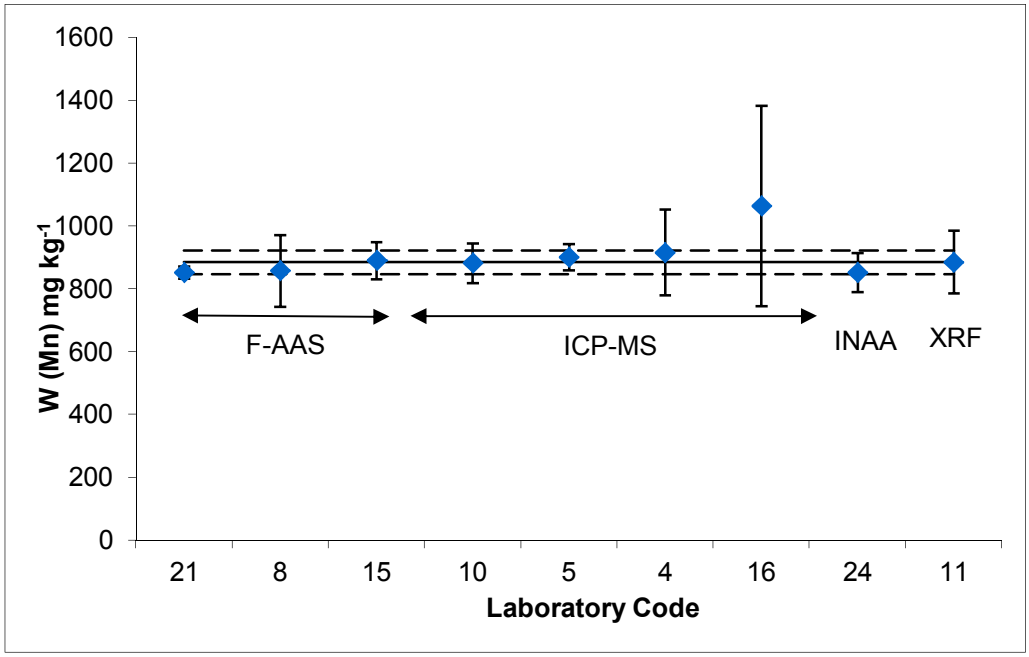

FIG. 11. Laboratory results for manganese mass fraction (mg·kg^{-1}) in the AEA-458 sediment sample. The robust mean (solid line) and corresponding expanded uncertainty (k=2) (dashed line) are shown. The error bars correspond to the expanded uncertainty reported by each laboratory.

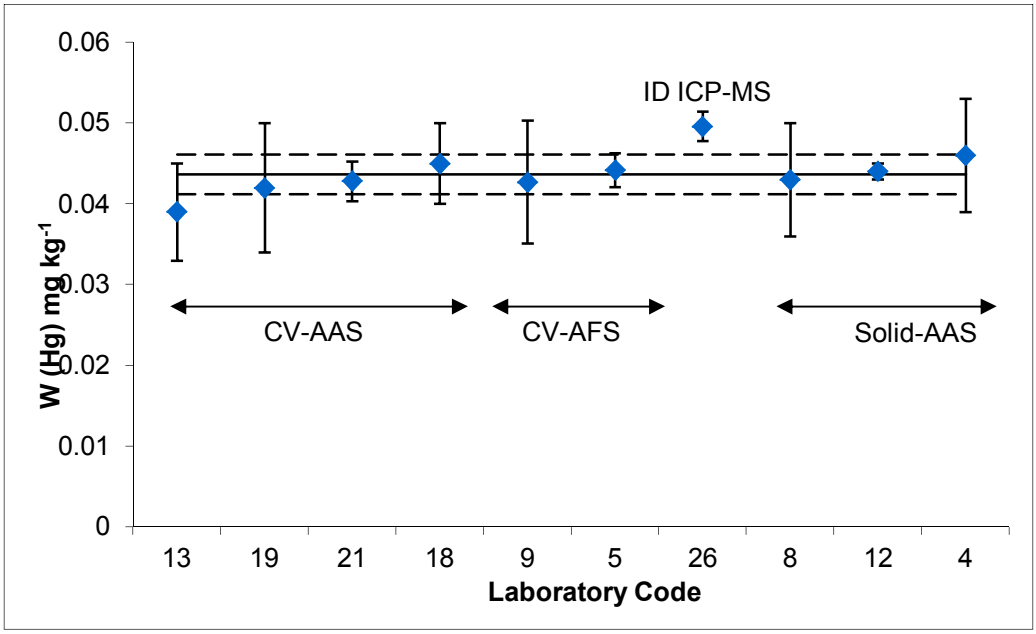

FIG. 12. Laboratory results for mercury mass fraction (mg·kg^{-1}) in the IAEA-458 sediment sample. The robust mean (solid line) and corresponding expanded uncertainty (k=2) (dashed line) are shown. The error bars correspond to the expanded uncertainty reported by each laboratory.

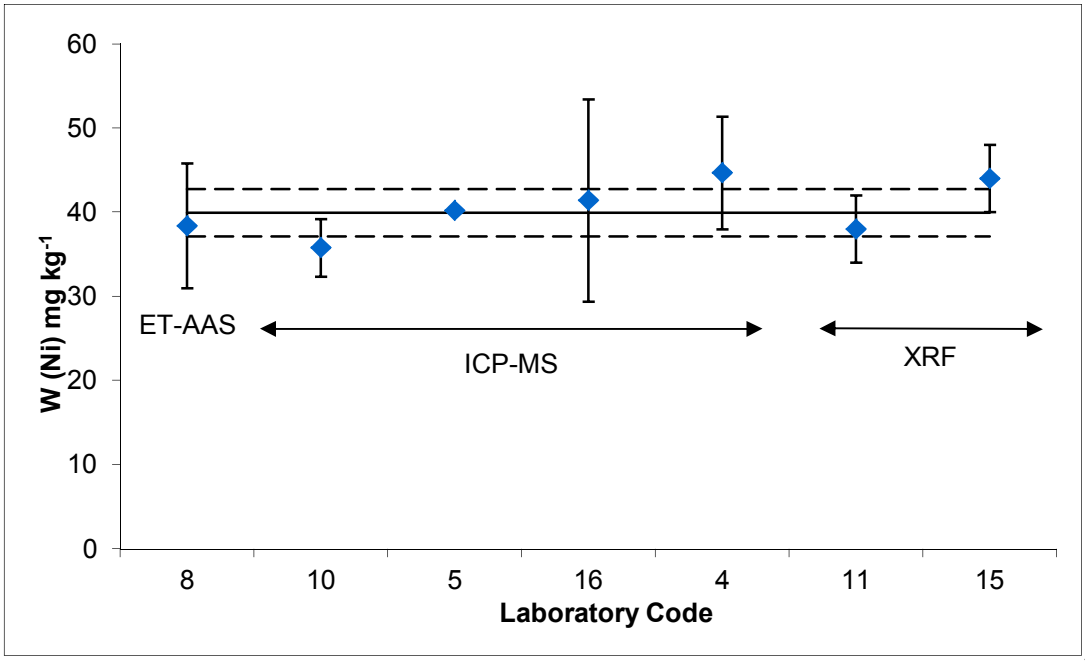

FIG. 13. Laboratory results for nickel mass fraction (mg·kg^{-1}) in the IAEA-458 sediment sample. The robust mean (solid line) and corresponding expanded uncertainty (k=2) (dashed line) are shown. The error bars correspond to the expanded uncertainty reported by each laboratory.

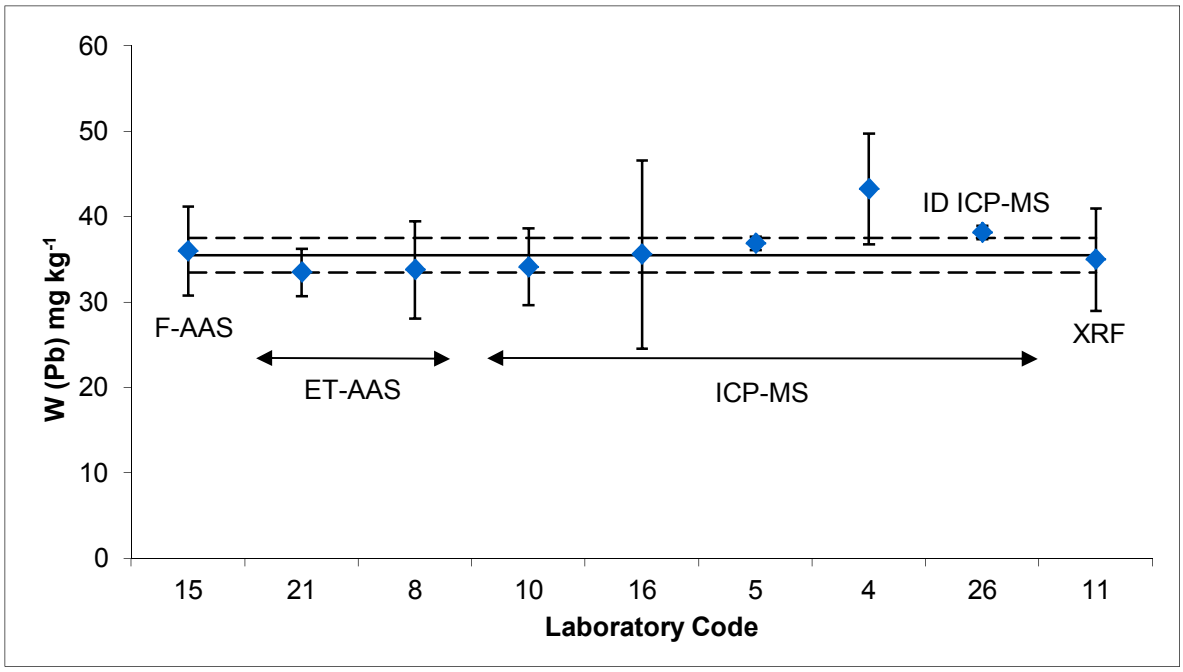

FIG. 14. Laboratory results for lead mass fraction (mg·kg^{-1}) in the IAEA-458 sediment sample. The robust mean (solid line) and corresponding expanded uncertainty (k=2) (dashed line) are shown. The error bars correspond to the expanded uncertainty reported by each laboratory.

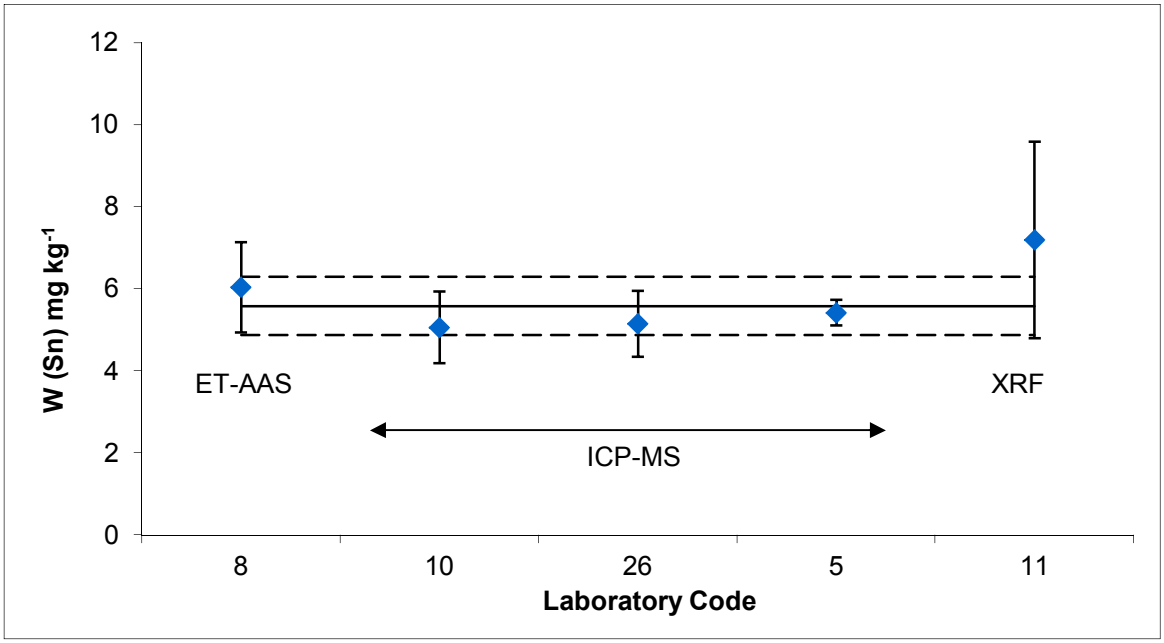

FIG. 15. Laboratory results for tin mass fraction (mg·kg^{-1}) in the IAEA-458 sediment sample. The robust mean (solid line) and corresponding expanded uncertainty (k=2) (dashed line) are shown. The error bars correspond to the expanded uncertainty reported by each laboratory

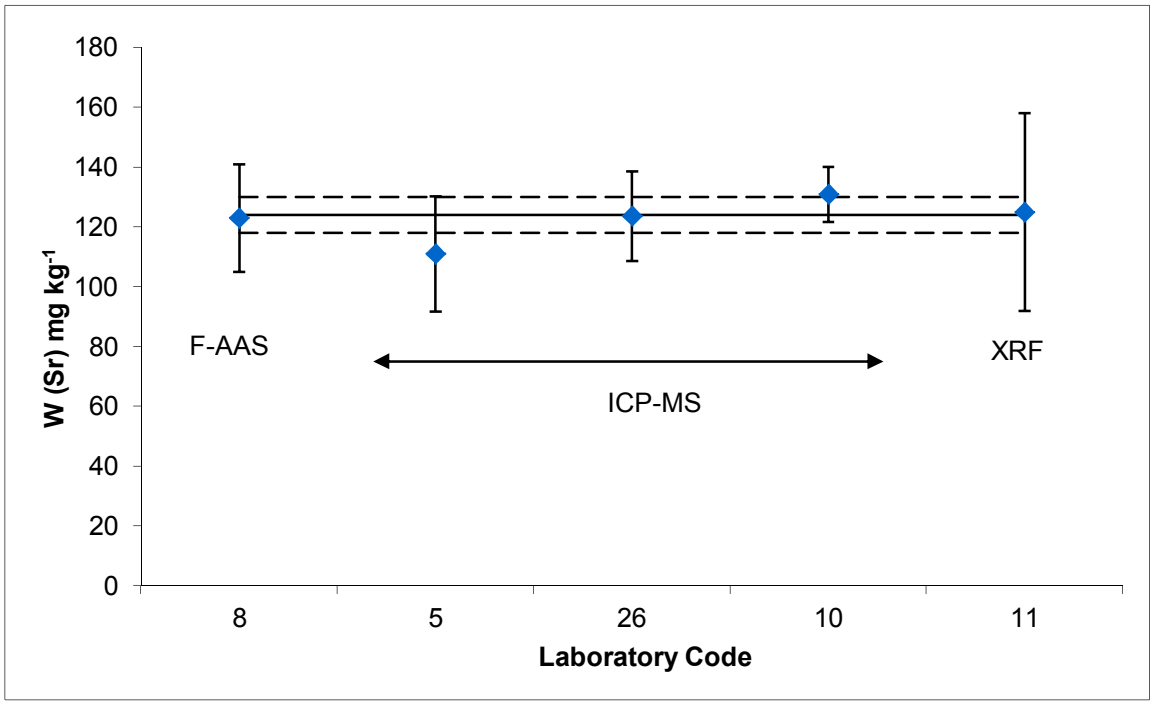

FIG. 16. Laboratory results for strontium mass fraction (mg·kg^{-1}) in the IAEA-458 sediment sample. The robust mean (solid line) and corresponding expanded uncertainty (k=2) (dashed line) are shown. The error bars correspond to the expanded uncertainty reported by each laboratory.

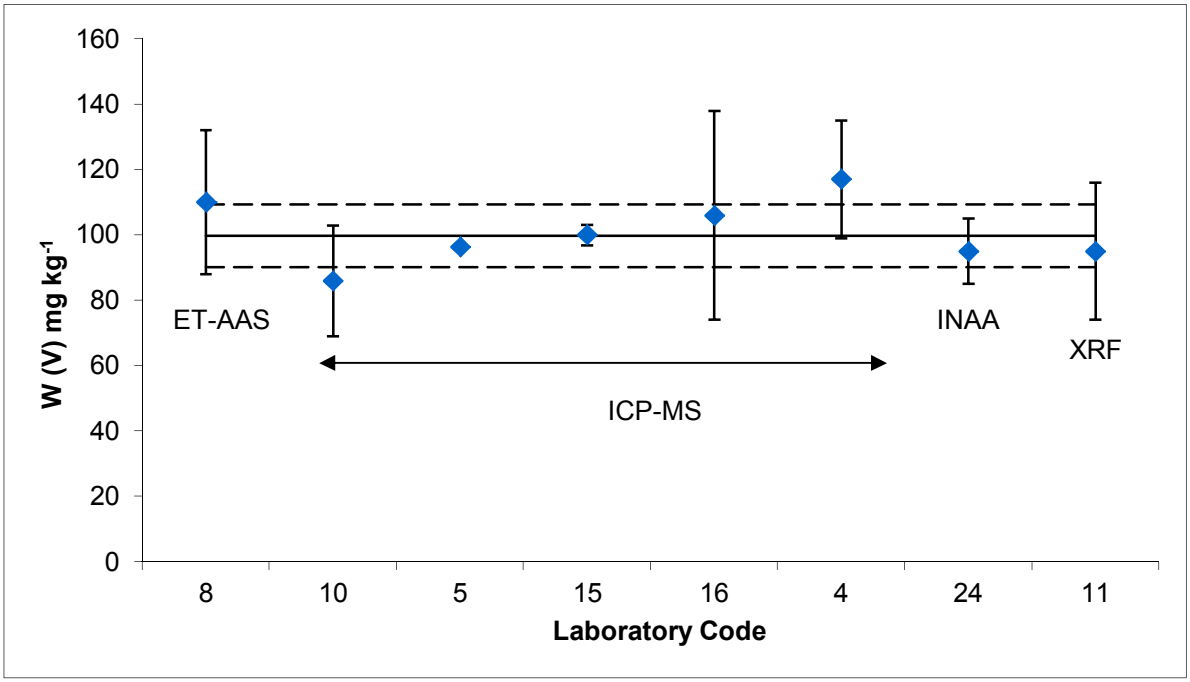

FIG. 17. Laboratory results for vanadium mass fraction (mg·kg^{-1}) in the IAEA-458 sediment sample. The robust mean (solid line) and corresponding expanded uncertainty (k=2) (dashed line) are shown. The error bars correspond to the expanded uncertainty reported by each laboratory.

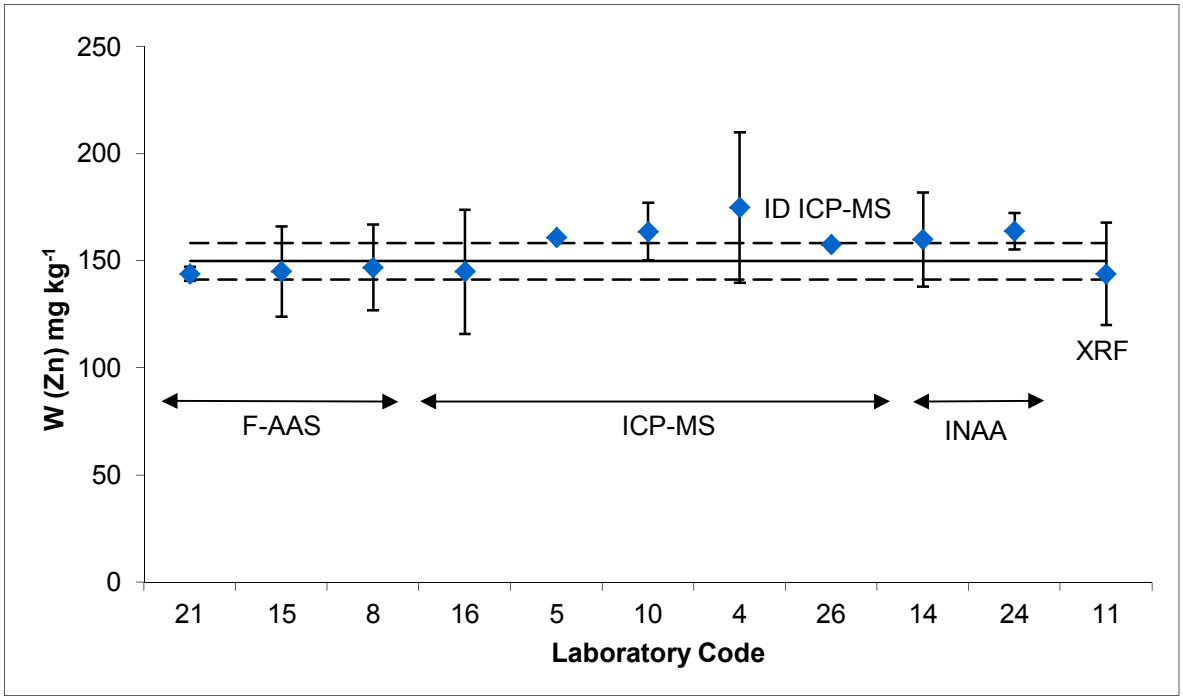

FIG. 18. Laboratory results for zinc mass fraction (mg·kg^{-1}) in the IAEA-458 sediment sample. The robust mean (solid line) and corresponding expanded uncertainty (k=2) (dashed line) are shown. The error bars correspond to the expanded uncertainty reported by each laboratory.

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 whose values 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, NRC-CNRC were used for validation of the methods applied in this study methods [1].

Pure metal standard solutions (CRMs) with stated purity were employed for calibration from 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 (in $mg \cdot kg^{-1}$or $g \cdot kg^{-1}$) is traceable to the reference unit to which the starting material is compared to.

Commutability is a property of a 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 [6].

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 a similar analytical behaviour for given method as 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.

The agreement between results obtained with different analytical methods, selected for the IAEA-458 characterization study, confirms the absence of any significant method bias and demonstrates commutability of the material for all certified trace elements and for the analytical methods applied for characterization of the IAEA-458 sediment sample. In addition, the agreement between the results confirms the identity of the analytes.

5. CONCLUSIONS

This exercise allows assignment of reference values for 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 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 the proficiency tests and inter-laboratory comparisons. As any certified reference material, it can be used for validation studies.

The reference values for 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 ($k=2$). The obtained mass fraction values for Ag were limited in number and inconsistent; therefore those values cannot be certified and included in the reference sheet.

TABLE 3. REFERENCE VALUES FOR TRACE ELEMENTS MASS FRACTIONS AND THEIR EXPANDED UNCERTAINTY (*k=2*) IN THE IAEA-458 SEDIMENT SAMPLE

Element	Unit	Reference value [1]	Expended uncertainty [2]
Al	$g \cdot kg^{-1}$	82.8	4.2
As	$mg \cdot kg^{-1}$	10.0	0.8
Cd	$mg \cdot kg^{-1}$	0.493	0.033
Co	$mg \cdot kg^{-1}$	15.6	1.2
Cr	$mg \cdot kg^{-1}$	91.5	8.6
Cu	$mg \cdot kg^{-1}$	48.1	3.1
Fe	$mg \cdot kg^{-1}$	40.7	2.0
Hg	$mg \cdot kg^{-1}$	0.0437	0.0025
Li	$mg \cdot kg^{-1}$	71.7	5.15
Mn	$mg \cdot kg^{-1}$	886	38
Ni	$mg \cdot kg^{-1}$	40.0	2.8
Pb	$mg \cdot kg^{-1}$	35.5	2.0
Sn	$mg \cdot kg^{-1}$	5.58	0.71
Sr	$mg \cdot kg^{-1}$	124	6
V	$mg \cdot kg^{-1}$	99.8	10
Zn	$mg \cdot kg^{-1}$	154	7

[1] *The value is the robust mean of accepted sets of data, each set being obtained by a 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. ALUMINIUM: RESULTS AS REPORTED BY PARTICIPANTS ($g \cdot kg^{-1}$)

Lab. code	Reported mean	Combined uncertainty	Expanded uncertainty
4	81.7	8.2	16.3
5	80.23	5.02	10.0
8	84.7	5.1	10.2
10	80.05	3.83	7.65
11	84.7	4.1	8.20
16	89.84	2.98	27.0
21	84.93	3.31	6.54
24	77.56	3.10	5.27

TABLE 5. ARSENIC: RESULTS AS REPORTED BY PARTICIPANTS ($mg \cdot kg^{-1}$)

Lab. code	Reported mean	Combined uncertainty	Expanded uncertainty
5	9.88	0.33	0.66
8	10.1	0.8	1.6
10	9.42	0.92	1.84
11	11	1.5	3
14	9.2	0.5	1.1
16	10.8	0.2	2.4
24	9.7	0.6	1.2

TABLE 6. CADMIUM: RESULTS AS REPORTED BY PARTICIPANTS ($mg \cdot kg^{-1}$)

Lab. code	Reported mean	Combined uncertainty	Expanded uncertainty
4	0.540	0.040	0.080
5	0.476	0.002	0.005
8	0.525	0.042	0.084
10	0.480	0.030	0.060
12	0.511	0.023	0.045
15	0.450	0.025	0.050
16	0.510	0.030	0.130
19	0.463	0.054	0.108
21	0.490	0.014	0.028
26	0.491	0.004	0.008

TABLE 7. COBALT: RESULTS AS REPORTED BY PARTICIPANTS ($mg \cdot kg^{-1}$)

Lab. code	Reported mean	Combined uncertainty	Expanded uncertainty
4	17.7	1.3	2.7
5	15.1	0.1	0.1
8	15.1	1.2	2.4
10	14.2	0.8	1.6
11	15.0	1.6	3.1
14	16.1	0.4	0.8
15	16.8	0.9	1.8
16	16.2	0.2	3.9
24	16.3	0.4	0.8

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

Lab. code	Reported mean	Combined uncertainty	Expanded uncertainty
4	102.0	8.0	15.0
5	83.2	0.6	1.2
8	97.3	9.7	19.4
10	77.6	4.8	9.5
11	87.0	8.0	15.0
14	96.0	5.0	10.0
15	82.0	3.1	6.1
16	96.6	1.0	29.0
21	89.9	3.0	5.8
24	103.5	2.6	5.2

TABLE 9. COPPER: RESULTS AS REPORTED BY PARTICIPANTS ($mg \cdot kg^{-1}$)

Lab. code	Reported mean	Combined uncertainty	Expanded uncertainty
4	55.1	4.0	8.0
5	53.0	0.2	0.3
8	47.9	5.1	10.2
10	45.7	2.1	4.2
11	45.0	7.0	15.0
15	49.0	2.2	4.4
16	45.2	0.1	9.1
21	45.9	0.5	1.1
26	48.7	0.2	0.4

TABLE 10. IRON: RESULTS AS REPORTED BY PARTICIPANTS ($mg \cdot kg^{-1}$)

Lab. code	Reported mean	Combined uncertainty	Expanded uncertainty
4	43.4	5.4	10.9
5	39.8	0.3	0.5
8	38.2	2.7	5.7
10	40.9	1.4	2.8
11	40.5	2.6	5.2
14	40.6	0.4	0.8
15	39.7	0.7	1.4
16	41.9	0.3	12.5
21	39.5	0.6	1.1
24	42.7	0.0	2.1

TABLE 11. MERCURY: RESULTS AS REPORTED BY PARTICIPANTS ($mg \cdot kg^{-1}$)

Lab. code	Reported mean	Combined uncertainty	Expanded uncertainty
4	0.0460	0.0030	0.0070
5	0.0442	0.0010	0.0021
8	0.0430	0.0035	0.0070
9	0.0427	0.0038	0.0076
12	0.0440	0.0006	0.0010
13	0.0390	0.0030	0.0060
18	0.0450	0.0025	0.0050
19	0.0420	0.0040	0.0080
21	0.0428	0.0012	0.0024
26	0.0496	0.0009	0.0018

TABLE 12. LITHIUM: RESULTS AS REPORTED BY PARTICIPANTS ($mg \cdot kg^{-1}$)

Lab. code	Reported mean	Combined uncertainty	Expanded uncertainty
4	81.0	12.1	24.2
5	71.7	2.0	4.0
8	69.4	5.5	11.1
10	68.6	4.2	8.5
15	72.0	3.6	7.0

TABLE 13. MANGANESE: RESULTS AS REPORTED BY PARTICIPANTS ($mg \cdot kg^{-1}$)

Lab. code	Reported mean	Combined uncertainty	Expanded uncertainty
4	916	69	137
5	901	21	42
8	858	57	114
10	882	32	64
11	885	50	100
15	890	30	59
16	1064	15	319
21	852	10	20
24	852	32	63

TABLE 14. NICKEL: RESULTS AS REPORTED BY PARTICIPANTS ($mg \cdot kg^{-1}$)

Lab. code	Reported mean	Combined uncertainty	Expanded uncertainty
4	44.7	3.4	6.7
5	40.2	0.2	0.4
8	38.4	3.7	7.4
10	35.77	1.7	3.4
11	38	2	4
15	44	2	4
16	41.4	0.7	12

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

Lab. code	Reported mean	Combined uncertainty	Expanded uncertainty
4	43.3	3.2	6.5
5	36.9	0.4	0.8
8	33.8	2.8	5.7
10	34.2	2.3	4.5
11	35.0	3.0	6.0
15	36.0	2.7	5.2
16	35.6	0.6	11.0
21	33.5	1.4	2.8
26	38.2	0.4	0.8

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

Lab. code	Reported mean	Combined uncertainty	Expanded uncertainty
5	5.42	0.16	0.32
8	6.04	0.54	1.10
10	5.06	0.43	0.87
11	7.20	1.20	2.40
26	5.15	0.40	0.80

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

Lab. code	Reported mean	Combined uncertainty	Expanded uncertainty
5	111	10	19
8	123	9	18
10	131	5	9
11	125	17	33
26	124	7.5	15

TABLE 18. VANADIUM: RESULTS AS REPORTED BY PARTICIPANTS ($mg \cdot kg^{-1}$)

Lab. code	Reported mean	Combined uncertainty	Expanded uncertainty
4	117	9	18
5	96.4	0.2	0.5
8	110	11	22
10	85.9	8.5	16.9
11	95	11	21
15	100	2	3
16	106	1	32
24	95	5	10

TABLE 19. ZINC: RESULTS AS REPORTED BY PARTICIPANTS ($mg \cdot kg^{-1}$)

Lab. code	Reported mean	Combined uncertainty	Expanded uncertainty
4	175	18	35
5	161	1	1
8	147	10	20
10	164	7	13
11	144	12	24
14	160	11	22
15	145	11	21
16	145	1	29
21	144	2	3
24	164	4	9
26	158	0.2	0.3

APPENDIX II

LIST OF PARTICIPATING LABORATORIES

AUSTRIA

Katona, R.	International Atomic Energy Agency Environment Laboratories Terrestrial Environment Laboratory Vienna International Centre A-1400 Vienna, Austria

BRAZIL

Favaro, D. Vasconcellos, M.	IPEN-CNEN (LAN-CRPQ) Av Professor Lineu Prestes, 2242 Cidade Universitaria São Paulo CEP 05508-000

CANADA

Flett, R. Gilbert, D. Wei, X.	Flett Research Ltd. 440 Desalaberry Ave Winnipeg Manitoba R2L 0Y7
Franco, H.	ALS Environmental (Vancouver) 8081 Lougheed Highway Suite 100 Burnaby, BC V5A 1W9

DENMARK

Larsen, M.	National Environmental Research Institute Frederiksborgvej 399 P.O. Box 358 DK-4000 Roskilde

FRANCE

Auger, P.	IFREMER - Centre de Nantes Rue de l'Ile d'Yeu B.P. 21105 FR-44311 Nantes Cedex 03
Coquery, M.	Irstea, Laboratoire d'Analyses Physico-Chimiques des Mmilieux Aquatiques (LAMA) UR Milieux Aquatiques, Ecologie et Pollutions 3bis quai Chauveau, CP 220 FR-69336 Lyon cedex 09

GREECE

Karageorgis A P. — Hellenic Centre for Marine Research
46,7 Km Athens, Sounion
19013 Anavissos

ISRAEL

Herut, B.
Shefer, E.
Kress, N. — Israel Oceanographic & Limnological Research
Tel Shikmona
P.O. Box 8030
31080 Haifa

LITHUANIA

Garnaga, G. — Environment Protection Agency
Marine Research Departmentt
Hydrochemistry section
Taikos pr.26, LT-91222
Klaipeda

MONACO

Vassileva, E.
Azemard, S. — International Atomic Energy Agency
Environment Laboratories
Marine Environmental Studies Laboratory
4 Quai Antoine 1er
98000 Monaco

PERU

Bedregal, P.
Torres, B.
Mendoza, P.
Ubillús, M. — Instituto Peruano de Energia Nuclear (IPEN)
Unit of Nuclear Analitycal Techniques
Av Canada 1470
Lima 41

RUSSIAN FEDERATION

Gladilovich, D.B. — LUMEX-MARKETING LLC
70, Bld 2 Obukhovskoy Oborony pr.
P.O. Box 1234
192029 St Petersburg

Olga, O. — Geoligical Institute of Russian Academy of Sciences
Pyzhevsky Line 7
119017 Moscow

UNITED STATES OF AMERICA

Mc Farland, F.
Carter, A.
Creswell, J.

Brooks Rand Laboratories
3958 6th Ave. N.W.
Seattle, Washington 98107

6. 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,100 (2008).

[3] INTERNATIONAL ORGANIZATION FOR STANDARDIZATION, ISO Guide 35:2006, Reference Materials – General and Statistical Principles for Certification, ISO, Geneva (2006).

[4] LINSINGER, T., PAUWELS, J., VAN DER VEEN, A., SCHIMMEL, H., LAMBERTY, A., Homogeneity and stability of reference materials, Accredit. Qual. Assurance, 6 1 (2001) 20–25.

[5] INTERNATIONAL ORGANIZATION FOR STANDARDIZATION, ISO 3528:2005, Statistical methods for use in proficiency testing by inter-laboratory comparisons, ISO, Geneva (2005).

[6] INTERNATIONAL ORGANIZATION FOR STANDARDIZATION, ISO Guide 99: International Vocabulary of Metrology–Basic and general concepts and associated terms (VIM), ISO, Geneva (2007).

IAEA
International Atomic Energy Agency

Where to order IAEA publications

In the following countries IAEA publications may be purchased from the sources listed below, or from major local booksellers. Payment may be made in local currency or with UNESCO coupons.

AUSTRALIA
DA Information Services, 648 Whitehorse Road, MITCHAM 3132
Telephone: +61 3 9210 7777 • Fax: +61 3 9210 7788
Email: service@dadirect.com.au • Web site: http://www.dadirect.com.au

BELGIUM
Jean de Lannoy, avenue du Roi 202, B-1190 Brussels
Telephone: +32 2 538 43 08 • Fax: +32 2 538 08 41
Email: jean.de.lannoy@infoboard.be • Web site: http://www.jean-de-lannoy.be

CANADA
Bernan Associates, 4501 Forbes Blvd, Suite 200, Lanham, MD 20706-4346, USA
Telephone: 1-800-865-3457 • Fax: 1-800-865-3450
Email: customercare@bernan.com • Web site: http://www.bernan.com

Renouf Publishing Company Ltd., 1-5369 Canotek Rd., Ottawa, Ontario, K1J 9J3
Telephone: +613 745 2665 • Fax: +613 745 7660
Email: order.dept@renoufbooks.com • Web site: http://www.renoufbooks.com

CHINA
IAEA Publications in Chinese: China Nuclear Energy Industry Corporation, Translation Section, P.O. Box 2103, Beijing

CZECH REPUBLIC
Suweco CZ, S.R.O., Klecakova 347, 180 21 Praha 9
Telephone: +420 26603 5364 • Fax: +420 28482 1646
Email: nakup@suweco.cz • Web site: http://www.suweco.cz

FINLAND
Akateeminen Kirjakauppa, PO BOX 128 (Keskuskatu 1), FIN-00101 Helsinki
Telephone: +358 9 121 41 • Fax: +358 9 121 4450
Email: akatilaus@akateeminen.com • Web site: http://www.akateeminen.com

FRANCE
Form-Edit, 5, rue Janssen, P.O. Box 25, F-75921 Paris Cedex 19
Telephone: +33 1 42 01 49 49 • Fax: +33 1 42 01 90 90
Email: formedit@formedit.fr • Web site: http://www. formedit.fr

Lavoisier SAS, 145 rue de Provigny, 94236 Cachan Cedex
Telephone: + 33 1 47 40 67 02 • Fax +33 1 47 40 67 02
Email: romuald.verrier@lavoisier.fr • Web site: http://www.lavoisier.fr

GERMANY
UNO-Verlag, Vertriebs- und Verlags GmbH, Am Hofgarten 10, D-53113 Bonn
Telephone: + 49 228 94 90 20 • Fax: +49 228 94 90 20 or +49 228 94 90 222
Email: bestellung@uno-verlag.de • Web site: http://www.uno-verlag.de

HUNGARY
Librotrade Ltd., Book Import, P.O. Box 126, H-1656 Budapest
Telephone: +36 1 257 7777 • Fax: +36 1 257 7472 • Email: books@librotrade.hu

INDIA
Allied Publishers Group, 1st Floor, Dubash House, 15, J. N. Heredia Marg, Ballard Estate, Mumbai 400 001,
Telephone: +91 22 22617926/27 • Fax: +91 22 22617928
Email: alliedpl@vsnl.com • Web site: http://www.alliedpublishers.com

Bookwell, 2/72, Nirankari Colony, Delhi 110009
Telephone: +91 11 23268786, +91 11 23257264 • Fax: +91 11 23281315
Email: bookwell@vsnl.net

ITALY
Libreria Scientifica Dott. Lucio di Biasio “AEIOU”, Via Coronelli 6, I-20146 Milan
Telephone: +39 02 48 95 45 52 or 48 95 45 62 • Fax: +39 02 48 95 45 48
Email: info@libreriaaeiou.eu • Website: www.libreriaaeiou.eu

JAPAN
Maruzen Company Ltd, 1-9-18, Kaigan, Minato-ku, Tokyo, 105-0022
Telephone: +81 3 6367 6079 • Fax: +81 3 6367 6207
Email: journal@maruzen.co.jp • Web site: http://www.maruzen.co.jp

REPUBLIC OF KOREA
KINS Inc., Information Business Dept. Samho Bldg. 2nd Floor, 275-1 Yang Jae-dong SeoCho-G, Seoul 137-130
Telephone: +02 589 1740 • Fax: +02 589 1746 • Web site: http://www.kins.re.kr

NETHERLANDS
De Lindeboom Internationale Publicaties B.V., M.A. de Ruyterstraat 20A, NL-7482 BZ Haaksbergen
Telephone: +31 (0) 53 5740004 • Fax: +31 (0) 53 5729296
Email: books@delindeboom.com • Web site: http://www.delindeboom.com

Martinus Nijhoff International, Koraalrood 50, P.O. Box 1853, 2700 CZ Zoetermeer
Telephone: +31 793 684 400 • Fax: +31 793 615 698
Email: info@nijhoff.nl • Web site: http://www.nijhoff.nl

Swets and Zeitlinger b.v., P.O. Box 830, 2160 SZ Lisse
Telephone: +31 252 435 111 • Fax: +31 252 415 888
Email: infoho@swets.nl • Web site: http://www.swets.nl

NEW ZEALAND
DA Information Services, 648 Whitehorse Road, MITCHAM 3132, Australia
Telephone: +61 3 9210 7777 • Fax: +61 3 9210 7788
Email: service@dadirect.com.au • Web site: http://www.dadirect.com.au

SLOVENIA
Cankarjeva Zalozba d.d., Kopitarjeva 2, SI-1512 Ljubljana
Telephone: +386 1 432 31 44 • Fax: +386 1 230 14 35
Email: import.books@cankarjeva-z.si • Web site: http://www.cankarjeva-z.si/uvoz

SPAIN
Díaz de Santos, S.A., c/ Juan Bravo, 3A, E-28006 Madrid
Telephone: +34 91 781 94 80 • Fax: +34 91 575 55 63
Email: compras@diazdesantos.es, carmela@diazdesantos.es, barcelona@diazdesantos.es, julio@diazdesantos.es
Web site: http://www.diazdesantos.es

UNITED KINGDOM
The Stationery Office Ltd, International Sales Agency, PO Box 29, Norwich, NR3 1 GN
Telephone (orders): +44 870 600 5552 • (enquiries): +44 207 873 8372 • Fax: +44 207 873 8203
Email (orders): book.orders@tso.co.uk • (enquiries): book.enquiries@tso.co.uk • Web site: http://www.tso.co.uk

On-line orders
DELTA Int. Book Wholesalers Ltd., 39 Alexandra Road, Addlestone, Surrey, KT15 2PQ
Email: info@profbooks.com • Web site: http://www.profbooks.com

Books on the Environment
Earthprint Ltd., P.O. Box 119, Stevenage SG1 4TP
Telephone: +44 1438748111 • Fax: +44 1438748844
Email: orders@earthprint.com • Web site: http://www.earthprint.com

UNITED NATIONS
Dept. I004, Room DC2-0853, First Avenue at 46th Street, New York, N.Y. 10017, USA
(UN) Telephone: +800 253-9646 or +212 963-8302 • Fax: +212 963-3489
Email: publications@un.org • Web site: http://www.un.org

UNITED STATES OF AMERICA
Bernan Associates, 4501 Forbes Blvd., Suite 200, Lanham, MD 20706-4346
Telephone: 1-800-865-3457 • Fax: 1-800-865-3450
Email: customercare@bernan.com · Web site: http://www.bernan.com

Renouf Publishing Company Ltd., 812 Proctor Ave., Ogdensburg, NY, 13669
Telephone: +888 551 7470 (toll-free) • Fax: +888 568 8546 (toll-free)
Email: order.dept@renoufbooks.com • Web site: http://www.renoufbooks.com

Orders and requests for information may also be addressed directly to:

Marketing and Sales Unit, International Atomic Energy Agency
Vienna International Centre, PO Box 100, 1400 Vienna, Austria
Telephone: +43 1 2600 22529 (or 22530) • Fax: +43 1 2600 29302
Email: sales.publications@iaea.org • Web site: http://www.iaea.org/books

13-32831

INTERNATIONAL ATOMIC ENERGY AGENCY
VIENNA
ISSN 2074– 7659