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Certification of Trace Element Mass Fractions in Marine Sediment IAEA-475

IAEA
International Atomic Energy Agency

CERTIFICATION OF TRACE ELEMENT MASS FRACTIONS IN MARINE SEDIMENT IAEA-475

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INTERNATIONAL ATOMIC ENERGY AGENCY
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FOREWORD

The protection of the marine environment is a high priority worldwide. The identification of environmental pollution is based on monitoring campaigns that periodically assess the quality of sea water, marine sediments and biota samples. The reliability and comparability of analytical results produced in this context are crucial for the management of the marine environment in general, for example in taking decisions and meaningful actions related to remediation policies. One of the most basic internal methods of quality control is the concurrent processing of a material similar in matrix to the unknown sample and of known composition, notably certified reference materials traceable to the common system of reference. Since the 1960s the IAEA has been providing help to its Member States in the field of data quality and quality assurance. To support Member States in their monitoring activities, the IAEA has produced certified reference materials characterized for trace elements and methylmercury using samples of marine origin biota and sediments.

This publication describes the production of a new certified reference material for trace element mass fractions in a sediment matrix in accordance with the requirements of international guidelines for the production and characterization of certified reference materials. Eight laboratories with demonstrated measurement capabilities participated in the characterization of the sediment sample. This publication replaces the initial certification publication published in 2018 and presents combined results from 2018 and 2022, based on new data that has since become available.

IAEA-475 was produced within the framework of the IAEA's Peaceful Uses Initiative. The sediment matrix used for IAEA-475 comes from Australia's marine coastal zone and will be an appropriate matrix certified reference material for monitoring studies along the Australian coast.

The IAEA is grateful to the Government of Monaco for the support provided to its Marine Environment Laboratories, and to the Government of Australia for funding this project as part of the Peaceful Uses Initiative.

The IAEA officer responsible for this publication was S. Azemard of the Marine Environment Laboratories.

CONTENTS

1. INTRODUCTION

1.1. BACKGROUND

Sound strategies for marine chemical monitoring call for measurement systems capable of producing comparable analytical results with demonstrated quality. Comparability of monitoring data is mandatory for evaluating spatial and temporal contamination trends and is achievable only if reliable measurement results are considered. The use of suitable certified reference materials (CRM) is necessary for the quality assurance of measurement result produced by monitoring laboratories. They are also used for the validation of analytical methods and for the traceability of results. They are valuable tools for the straightforward assessment of the trueness of the method applied. To ensure the reliability of analytical results, quality assurance through the use of CRMs is regarded as a crucial prerequisite in ISO 17025 [1].

1.2. OBJECTIVE

Selection and use of marine matrix CRMs that match the matrix of the analysed sample and the concentration range of analytes of interest in the "real" samples is essential for obtaining high quality measurement results. IAEA-475 was produced to support the accurate and traceable measurements of trace metals in marine sediment from the Pacific coast of Australia and other pristine areas.

IAEA-475 will assist laboratories in validating their analytical methods and controlling the quality of produced analytical results for the determination of trace elements in marine sediment samples [1].

1.3. SCOPE

The scope of this publication is to describe the certification process of trace elements in IAEA- 475. The material was initially certified in 2018 and a certification report was published [2]. Since then, new data is available, and this publication replaces the initial certification report [2], and presents combined results from 2018 and 2022.

1.4. STRUCTURE

This publication is structured in five sections, the first being the introduction. Section 2 provides the methodology used for the preparation of the reference material, including the selection of laboratories for the characterization campaign, and the procedures for the homogeneity, stability, and reference values assignment. Section 3 covers the results and discussions on the determination and justifications of the assigned values and their expanded uncertainties. Section 4 provides the information on metrological traceability and commutability of the new CRM. Section 5 provides the conclusions from the certification campaign of IAEA-475.

2. METHODOLOGY

2.1. PREPARATION OF THE MATERIAL

The base material for IAEA-475 coastal sediment was collected in the vicinity of Townsville, Australia during a sampling campaign organised by the Marine Environment Studies Laboratory (MESL), James Cook University and Australia's Nuclear Science Technology

Organization Australia. Approximately 50 kg of surface sediment sample were collected at 2.2–2.5 m water depth into polyester buckets and transferred at 4ºC to MESL, Monaco.

After freeze drying, the sample was grinded by micronisation. The obtained sample contained 100% of particulates below 20 µm (Figure 1). The bulk material was then sterilised by gamma irradiation (30 kGy).

The homogenization of the sediment sample was performed by dividing the total mass of material into seven lots, each of them 800 g. Each lot was transferred into a clean plastic bottle and mixed with a shaker for 2 hours. Immediately after finalizing the mixing operation, aliquots of about 20 g were packed into pre - cleaned polyethylene bottles with secured screw caps. The process was repeated seven times and bottles were labelled with the respective bottle numbers.
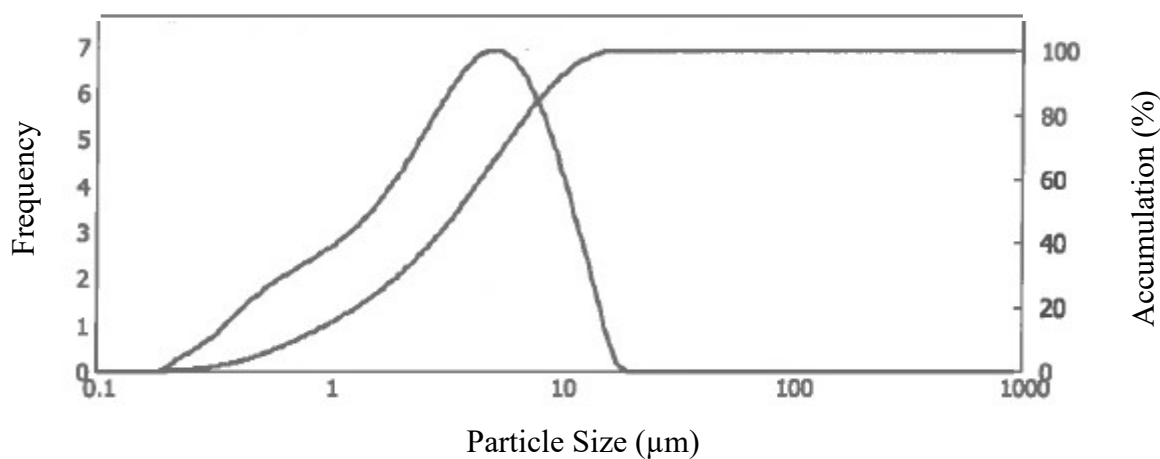

FIG. 1. Grain size distribution of final sample.

2.2. SELECTION OF LABORATORIES

The selection of participants for the characterization study was based on the measurement performances demonstrated by laboratories in the previous three ILCs organized by the IAEA in past years. Only results of laboratories having a quality system in place, using validated methods, applying uncertainty and traceability concepts were used for the calculation of the assigned values and their uncertainties [3].

The list of laboratories participating in the IAEA-475 characterization study is presented at the end of this report.

2.3. ANALYTICAL METHODOLOGIES

For all analytes Ag, Al, As, Cd, Co, Cr, Cu, Fe, Li, Mn, Ni, Pb, Sr, and Zn subsamples of 0.2 g were mineralized with 5 ml of nitric acid and 2 ml of hydrofluoric acid in a microwave oven. Digested solutions were then treated with boric acid to remove excess of hydrofluoric acid and fluorine precipitates. The final measurements were performed by inductively coupled plasma mass spectrometry (ICP-MS), flame atomic absorption spectrometry (FAAS) or inductively coupled plasma optical emission spectrometry (ICP-OES) under repeatability conditions, and in a randomized way.

The determination of the total Hg was done in solid subsamples (50 mg) with an advanced mercury analyser.

Methylmercury determination was performed by gas chromatography coupled to atomic fluorescence spectroscopy after purge and trap. Subsamples of 0.2g were extracted in dichloromethane after leaching in 5ml of H_2SO_4 (5% v/v) /KBr (15% w/v) and 1ml of 1M $CuSO_4$ and back extracted in 2mM thiosulfate solution before derivatization with sodium tetraethyl borate.

The results were corrected for the moisture content determined in each unit by using the procedure describe in Section 2.6.

All methods were previously validated in the IAEA, MESL inorganic chemistry laboratories.

2.4. HOMOGENEITY ASSESSMENT

A key requirement for any reference material certified as a batch is the equivalence between the various CRM units in this batch. In this respect, the important issue is whether the variation between and within units is significant compared to the uncertainty of the certified value. Consequently, ISO 17034 [3] requires reference material producers to quantify the between-unit variation.

2.4.1. Between-unit homogeneity assessment

The between-unit homogeneity was evaluated to ensure that the certified values of the CRM are valid for all produced units, within the stated uncertainty. In total, 14 bottles from the whole batch were selected, using random stratified sampling. Duplicate subsamples from each bottle were analysed for their total element and methyl mercury mass fractions in the IAEA, MESL inorganic chemistry laboratories as described in Section 2.3.

The measurements were performed in one analytical run under repeatability conditions and in a randomized way to be able to separate a potential analytical trend from a bottling trend.

The potential trend in the analytical sequence was tested by regression analyses. As the analytical sequences were randomized (i.e. not correlated with unit number), any significant (95%) trend between run position and mass fraction were corrected using Eq. (1). Correction of trend is expected to reduce analytical variation improving the evaluation of potential between unit inhomogeneity.

$$\text{Corrected result} = \text{Measured Result} - (b \times i) \tag{1}$$

where

b   is the slope of the linear model;
i   is the position of the analysed subsample in the run.

Results (analytical trend corrected if needed) are then tested for processing bottling trend, using regression between unit number and results. Grubbs and Dixon tests were performed to identify potential outlying individual results or unit means.

Quantification of between-unit heterogeneity was done by one way analysis of variance (ANOVA) which can separate the between-unit variation (s_{bb}) from the within-unit variation (s_{wb}). The latter accounts for method repeatability and within unit inhomogeneity.

As a prerequisite for the application of ANOVA for the estimation of uncertainty arising from homogeneity, it was verified whether the individual results and unit means followed a normal distribution, or the data set had a single mode of distribution.

ANOVA allows the calculation of the within-unit standard deviation s_{wb} and the between-units standard deviation s_{bb} from mean squares (MS) of within- and between-groups, and enable the estimation of associated uncertainty components, by applying Eqs (2) and (3).

$$s_{wb} = u_{wb} = \sqrt{MS_{wb}} \tag{2}$$

$$s_{bb} = u_{bb} = \sqrt{\frac{MS_{bb} - MS_{wb}}{n}} \tag{3}$$

where n is the number of replicates sub-samples per bottle.

s_{bb} and s_{wb} are estimates of the standard deviations and are subject to random fluctuations. Therefore, the mean square between groups (MS_{bb}) can be smaller than the mean square within groups (MS_{wb}), resulting in negative arguments under the square root used for the estimation of the between-units variation, whereas the true variation cannot be lower than zero. In this case, u^*_{bb}, the maximum heterogeneity, that could be hidden by method repeatability, was calculated as described by Linsinger et al. [4].

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

where

n is the number of replicates sub-samples per bottle;
ν_{MSwb} is the degrees of freedom of MS_{wb}.

2.4.2. Within-unit homogeneity assessment

ISO Guide 35:2017 [5] recommends specific assessment of within-unit variation when the minimum sample intake is significantly lower than the unit size, which is the case for this study (20g units and sample size 0.2g).

For the within-unit study, 6 subsamples from the same unit were analysed in triplicates for their total element and methylmercury mass fractions as described in Section 2.3. The measurements were performed under repeatability conditions. For Hg, the measurement on a same subsample cannot be repeated as the measurement was performed using a destructive technique. Therefore, the within bottle study was performed by measurements of 10 sub-samples from the same unit.

The potential trend in the analytical sequence was tested by regression analyses as explained in Section 2.4.1, significant trends (95%) between the run position and the mass fraction were corrected using Eq. (1). The results corrected for sequence trend were checked for single outlier and subsample mean outlier applying Grubbs and Dixon tests.

As a prerequisite for the application of ANOVA for the estimation of uncertainty arising from within-unit inhomogeneity, it was verified whether the individual results and unit means followed a normal distribution and if not, whether as a minimum the distribution of the data had a single mode.

The quantification of uncertainty arising from within-unit inhomogeneity was done by one-way ANOVA which in this case can separate within-bottle variation (Eq. (5)) from the measurement variation (Eq. (6)). The between-group mean square (MS_{wb}) represents the within-bottle variance while the within-group mean square (MS_{method}) represents the analytical variation. For total Hg, as only one measurement can be performed per subsample, s_{method} is obtained from validation and is estimated as 2.5%.

$$s_{\mathrm{method}} = \sqrt{MS_{\mathrm{method}}} \tag{5}$$

$$s_{wb} = \sqrt{\frac{MS_{wb} - M_{\ method}}{n}} \tag{6}$$

2.5. STABILITY STUDY

Time, temperature and radiation were regarded as the most important influences on the stability of the materials. The influence of light was minimized by choice of the container and packaging which eliminates most of the incoming light. In addition, materials are stored in the dark, thus practically eliminating the possibility of degradation by radiation. Additionally, the material was sterilized by γ-irradiation treatment to eliminate microbial growth. Therefore, only the influences of time and temperature needed to be investigated.

All the measurements of stability studies were performed under repeatability conditions and in a randomized way in order to separate a potential analytical drift from a trend related to the storage time. As explained in Section 2.4 the correction of trends is expected to improve the sensitivity of the subsequent statistical analysis through a reduction in analytical variation without masking the potential instability. Before applying any statistical analysis, analytical trends (i.e., mass fraction versus run position) significant at at least a 95 % confidence level were corrected using Eq. (1). Results corrected for sequence trend (if necessary) were checked for single outlier and mean outlier by applying Grubbs and Dixon tests.

Evaluation of data was then carried out by performing a linear regression on the determined mass fractions versus time (for long term and short term study).

2.5.1. Short term stability

The short term stability study was carried out using an isochronous design [6]. In this approach, samples were stored for a given time at different temperature conditions. Afterwards, the samples were moved to conditions where further degradation can be assumed to be negligible (reference conditions). At the end of the isochronous storage, the samples were analysed under repeatability conditions. Analysis of the material (after various exposure times and temperatures) under repeatability conditions significantly improves the sensitivity of the stability tests.

In order to evaluate potential degradation of the material during transport, some bottles were stored at (60 ± 3) °C. After 1, 2, 3 and 4 weeks, respectively, two bottles were selected and placed under 'reference' condition (-20 ± 2) °C. Duplicates subsamples from each bottle were analysed for their total element and methyl mercury mass fractions as describe in Section 2.3.

2.5.2. Long term stability

For the evaluation of the influence of long term storage conditions, 10 bottles were stored under 'reference' conditions at (-20 ± 2) °C in the dark since the time of bottling (February 2018). All other produced unit were stored under 'normal' conditions at (+20 ± 5) °C in the dark. After four years, four bottles stored under normal conditions and two bottles stored under reference conditions at the time of bottling were analysed in duplicate for their total element and methyl mercury mass fractions as describe in Section 2.3.

2.6. CHARACTERIZATION

The final characterization was based on results obtained in the IAEA MESL inorganic chemistry laboratories (section 2.3) and the results submitted by selected laboratories with demonstrated measurement capabilities (see Section 2.2).

Each laboratory received one bottle of sediment sample, accompanied by an information sheet and one reporting form. Participants were requested to analyse Ag, Al, As, Cd, Co, Cr, Cu, Fe, Hg, MeHg, Li, Mn, Ni, Pb, Sr, and Zn using validated analytical methods. They were requested to report measurement results (three replicates and average value), expanded uncertainty, information on the applied quality control procedures as well as information on the standard calibration solutions used in the measurement step.

In addition, each participant also received one bottle of IAEA-458 (CRM with similar matrix composition) as a blind quality control sample. The reported results for the quality control sample were evaluated against the certified values and associated uncertainties of the CRM IAEA-458 [7], except for Ag and MeHg mass fraction as no certified values were available.

Laboratories were requested to provide results corrected for moisture, as the result for the moisture content in the sample is an operationally dependent parameter, the method for moisture determination being prescribed to all participating laboratories (see Section 2.7).

The results of laboratories not fulfilling the above requirements were excluded from further evaluation. Datasets non-compliant to the preliminary defined criteria or considered as not technically valid are listed in Table 1.

The characterization of the trace elements mass fractions in the sample was based on the application of several analytical techniques. The abbreviations of the analytical techniques, applied in this characterization study are given in Table 2.

TABLE 1. DATASET EXCLUDED FROM FURTHER EVALUATION AFTER TECHNICAL REVIEW

Element	Lab code	Description
As	7	high uncertainty (>20%) when compared to other expert laboratories
As	15	QC result does not agree within uncertainties
Fe	2	high uncertainty (>20%) when compared to other expert laboratories
Hg	2	high uncertainty (>20%) when compared to other expert laboratories
MeHg	13	high uncertainty (>50%) when compared to other expert laboratories
Mn	13 and 15	QC result does not agree within uncertainties
Ni	13	QC result does not agree within uncertainties
Sr	13	QC result does not agree within uncertainties

TABLE 2. ABBREVIATION USED FOR ANALYTICAL TECHNIQUES

Abbreviation	
AAS	Atomic absorption spectrometry
AFS	Atomic fluorescence spectrometry
CV	Cold vapour
ET-AAS	Electro thermal atomic absorption spectrometry
F-AAS	Flame atomic absorption spectrometry
ICP-MS	Inductively coupled plasma mass spectrometry
ID	Isotope dilution
NAA	Neutron activation analysis (or instrumental neutron activation)
ICP-OES	Inductively coupled plasma optical emission spectrometry

2.7. MOISTURE DETERMINATION

The determination of the moisture content of the samples is to some extent an 'operationally defined' parameter. In view of the comparability of results, the protocol for the correction for moisture was developed at the IAEA and prescribed to other participants. Correction for dry mass is obtained by drying a minimum 0.5 g of the material at (105 ± 3) °C until the constant mass is attained (usually 24 hours).

Moisture, determined at MESL (10 subsamples from 5 bottles) was found to be 2.3 ($\pm$ 0.4) % for bottles kept at 20°C.

3. RESULTS AND DISCUSSION

3.1. RESULTS OF HOMOGENEITY STUDY

The results of homogeneity studies for within and between units have been evaluated separately as describe in Section 2.4.

3.1.1. Between-unit homogeneity

A significant analytical sequence trend (99%) was found for As, Li, Mn, Ni, Sr and Zn: the datasets were corrected as explained in Section 2.4.1 using Eq. (1). No processing trends were observed, and all datasets (means and individuals) were found to be normally distributed.

Individual outliers were detected for Mn and Hg (at 99%), but since no technical reasons were found for observed single outliers, and distributions were normal, all reported results were retained for further evaluation. No outlying unit means were detected.

The uncertainty contributions due to the between-unit inhomogeneity were estimated according to ISO Guide 35 [5] as the maximum values obtained with Eqs (3) or (4) and presented in Table 3. As can be seen in Table 3 the between-unit variations for all analytes were sufficiently small to demonstrate the homogeneity of the material for the specified sample masses.

TABLE 3. ESTIMATED CONTRIBUTIONS OF INHOMOGENEITY TO THE COMBINED UNCERTAINTY OF THE CERTIFIED VALUES FOR EACH TRACE ELEMENTS

	s_{bb}	u^*_{bb}	u_{bb}
Ag	0.5%	1.0%	1.0%
Al	1)	1.3%	1.3%
As	1.3%	0.7%	1.3%
Cd	2.0%	2.5%	2.5%
Co	1.2%	0.6%	1.2%
Cr	1)	1.5%	1.5%
Cu	1.5%	1.3%	1.5%
Fe	1)	1.1%	1.1%
Hg	1)	1.9%	1.9%
MeHg	1)	4.9%	4.9%
Li	0.2%	1.7%	1.7%
Mn	2.7%	1.3%	2.7%
Ni	1.4%	1.1%	1.4%
Pb	1)	1.4%	1.4%
Sr	0.2%	1.0%	1.0%
Zn	1.1%	1.3%	1.3%

1) Not defined due to negative argument under the square root

3.1.2. Within-unit homogeneity

A significant analytical sequence trend (99%) was found for As, Cd, Co and Ni: datasets were corrected as explained in Section 2.4.1 using Eq. (1). All datasets (means and individuals) were found to be normally distributed. Individual outliers were detected for Co and Mn (at 95%), but since no technical reasons were found for observed single outliers, all reported results were retained for further evaluation. No outlying subsample means were detected.

The obtained estimate of standard uncertainties arising from within-unit inhomogeneity are presented in Table 4.

3.1.3. Estimates of inhomogeneity uncertainties

The uncertainty u_{hom} associated with inhomogeneity of the material at the prescribed minimum sample size was estimated using Eq. (7) and presented in Table 4.

$$u_{ho} = \sqrt{u_{wb}^2 + u_{bb}^2} \qquad (7)$$

The conclusion from the obtained results was that the sediment sample showed good homogeneity with estimated relative standard uncertainties being less 5% for most analytes. Only Cd and MeHg showed relatively higher values (6% and 7.8%, respectively) mainly linked to their sub ng g^{-1} levels in the sample. Sample masses of 0.05 g for Hg and 0.2 g for other trace elements were set up as a minimum sample intake.

TABLE 4. THE ESTIMATE OF HOMOGENEITY CONTRIBUTIONS TO THE TOTAL UNCERTAINTY FOR THE CERTIFIED TRACE ELEMENTS

Element	$u_{bb,rel}$	$u_{wb,\,rel}$	$u_{hom,\,rel}$
Ag	1.0%	0.6%	1.2%
Al	1.3%	3.3%	3.5%
As	1.3%	0.6%	1.4%
Cd	2.5%	5.4%	6.0%
Co	1.2%	1.2%	1.7%
Cr	1.5%	1.0%	1.8%
Cu	1.5%	2.0%	2.5%
Fe	1.1%	1.3%	1.7%
Hg	1.9%	[1]	1.9%
MeHg	4.9%	7.1%	7.8%
Li	1.7%	0.5%	1.8%
Mn	2.7%	1.2%	3.0%
Ni	1.4%	0.8%	1.6%
Pb	1.4%	1.0%	1.7%
Sr	1.0%	2.0%	2.2%
Zn	1.3%	2.2%	2.6%

[1] negative argument in Eq. 6

3.2. RESULTS FOR STABILITY STUDY

The results of the long term and short term stability studies have been evaluated separately as described in Section 2.5.

3.2.1. Short term stability

A significant analytical sequence trend (99%) was found for Li and Ni: datasets were corrected as explained in Section 2.5.1 using Eq. (1). One individual result was detected at 99% as an outlier for Hg, but as no technical reason was found and results were normally distributed, all data were taken for further evaluation.

Evaluation of data was carried out by performing a linear regression on the determined mass fractions versus time. The tested properties showed no significant trend at +60°C storage temperature within four weeks. No significant slope at a 95% level of confidence was detected for any of the investigated trace elements in the short term stability study. As no degradation was observed under the applied conditions, it was concluded that no special precautions regarding temperature control during shipment are necessary. The uncertainty associated with short term stability was set to 0. Graphical representations of the short term stability study are displayed in Appendix I (Figs 2–17).

3.2.2. Long term stability

A significant analytical sequence trend (99%) was found for all datasets except Hg and MeHg: these were corrected as explained in Section 2.5.1 using Eq. (1). No outliers were detected.

Mass fraction versus storage time at 20°C did not reveal a significant trend at a 95% level of confidence except for MeHg. For all analytes except MeHg, the approach proposed in ISO Guide 35 [5] was followed and an uncertainty contribution related with the stability of the candidate reference material (u_{lt}) was estimated as the standard error of the slope multiplied by the selected shelf life of five years and presented in Table 5.

As shown in Fig. 28 (Appendix II) for MeHg, the observed change of mass fraction over four years is almost 50%, demonstrating a significant lack of stability preventing the assignment of an assigned value for MeHg in the candidate CRM.

Graphical representations of the long term stability study are displayed in Appendix II (Figs 18–33).

3.3. DETERMINATION OF ASSIGNED VALUES AND THEIR UNCERTAINTIES

The characterization campaign resulted in 3–9 measurement results for the selected trace elements. The obtained measurement results were first checked for compliance with the certification requirements, and then for their validity based on technical reasoning. All accepted sets of results were submitted to the following statistical tests: Grubbs and Dixon's test to detect outliers and Kolmogorov-Smirnov's test for normal distribution. No results were detected as outlier and all datasets were normally distributed.

The medians and unweighted mean of the means were calculated and compared. No significant differences were observed, and the reference values obtained with the mean of the mean approach was further used. These values are considered to be the most reliable estimates of the property values of the selected trace elements in IAEA-475.

The uncertainties associated with the assigned values were calculated according to ISO Guide 35 [5]. The relative combined uncertainty of the certified value of the CRM consists of uncertainty related to its characterization (u_{char}), sample heterogeneity (u_{hom}) and long term stability (u_{stab}). These different contributions were combined to estimate the expanded uncertainty.

$$U = k \times \sqrt{u_{char}^2 + u_{stab}^2 + u_{hom}^2}$$ (8)

Where

k: coverage factor equalling 2, representing a level of confidence of about 95%;

u_{hom} was estimated as described in Section 3.1;

u_{stab} was estimated as described in Section 3.2;

u_{char} was estimated as described in ISO Guide 35 [5] using Eq. (9):

$$u_{char} = \frac{s}{\sqrt{p}}$$ (9)

Where: s is the standard deviation of the mean; p is the number of datasets.

Mean values, their relative expanded uncertainties ($k=2$) and uncertainty contributions from the characterization, homogeneity and stability for all trace elements are presented in Table 5.

TABLE 5. MEAN OF THE MEANS AND UNCERTAINTIES

Element	Mean of the means mg kg^{-1}	$u_{char,rel}$	$u_{hom,rel}$	$u_{stab,rel}$	$U_{,rel}$ ($k=2$)
Ag	0.121	14.8%	1.2%	1.6%	29.8%
Al	72.4 ×10³	2.9%	3.5%	3.3%	11.2%
As	12.6	2.5%	1.4%	3.6%	9.2%
Cd	0.066	8.6%	6.0%	4.7%	23.0%
Co	12.36	1.3%	1.7%	2.6%	6.7%
Cr	65.8	1.3%	1.8%	1.7%	5.5%
Cu	27.8	2.2%	2.5%	2.1%	7.8%
Fe	34.2 ×10³	2.2%	1.7%	2.4%	7.4%
Hg	0.030	1.6%	1.9%	3.0%	7.8%
Li	40.6	2.3%	1.8%	2.4%	7.6%
Mn	566	1.4%	3.0%	2.3%	8.1%
Ni	28.5	1.1%	1.6%	2.6%	6.5%
Pb	29.9	1.7%	1.7%	2.9%	7.6%
Sr	249	2.1%	2.2%	0.7%	6.5%
Zn	100.1	1.9%	2.6%	3.1%	9.0%

The results for the mass fractions of the trace elements as reported by the participants in this characterization study, grouped by the applied analytical methods are presented in Appendix III. In all figures the reported results are plotted versus the laboratory code, and the mean of the means value denoted by a bold line, while the dashed lines represent the mean of the means ± its associated expanded uncertainty (k=2) (Eq. (8)). The error bars represent the expanded uncertainties, reported by participants.

As shown on Figs 34–48, methods with different quantification principles as well as methods without a sample preparation step, such as neutron activation or solid sampling AAS, were used for the characterization of the candidate reference material. A good agreement within the stated uncertainty was observed for results obtained with different methods, therefore all of them were considered in the process of calculation of the assigned values. The agreement between results confirms the absence of any significant method bias and demonstrates the identity of the analyte.

Certified values were calculated as the mean of the means of accepted dataset for the elements fulfilling the following criteria: (i) at least 5 results from 2 different methods were available; and (ii) relative expanded uncertainties of the assigned value were less than 15% (k=2).

These criteria were fulfilled for Al, As, Co, Cr, Cu, Fe, Hg, Li, Mn, Ni, Pb, Sr and Zn. The assigned certified values are presented in Table 6, together with their expanded uncertainty (k=2).

TABLE 6. CERTIFIED VALUES FOR TRACE ELEMENT MASS FRACTIONS AND THEIR EXPANDED UNCERTAINTY (K=2) IN IAEA-475

Element	Unit	Certified value[1]	Expanded uncertainty (k=2)[2]
Al	mg kg^{-1}	72.4×10^3	8.1×10^3
As	mg kg^{-1}	12.6	1.2
Co	mg kg^{-1}	12.36	0.83
Cr	mg kg^{-1}	65.8	3.6
Cu	mg kg^{-1}	27.8	2.2
Fe	mg kg^{-1}	34.2×10^3	2.5×10^3
Hg	mg kg^{-1}	29.9×10^{-3}	2.3×10^{-3}
Li	mg kg^{-1}	40.6	3.1
Mn	mg kg^{-1}	566	46
Ni	mg kg^{-1}	28.5	1.9
Pb	mg kg^{-1}	29.9	2.3
Sr	mg kg^{-1}	249	15
Zn	mg kg^{-1}	100.1	9.0

[1] The value is the mean of the means of the accepted sets of data, each set being obtained by different laboratory. The certified values are reported on dry mass basis and are traceable to the SI.
[2] The uncertainty is expressed as an expanded uncertainty with a coverage factor k=2, corresponding to the level of confidence of about 95%, estimated in accordance with the JCGM 100:2008 [8], and ISO Guide 35 [5].

The criteria for certification were not fulfilled for Ag, and Cd, and as a result only information values are provided. The information values for Ag, and Cd, are presented in Table 7.

TABLE 7. INFORMATION VALUES FOR TRACE ELEMENTS MASS FRACTIONS AND THEIR EXPANDED UNCERTAINTY (K=2) IN IAEA-475

Element	Unit	Information value[1]	Expanded uncertainty (k=2)[2]
Ag	mg kg^{-1}	0.121	0.036
Cd	mg kg^{-1}	0.066	0.015

[1] The value is the mean of the means of accepted sets of data.
[2] The uncertainty is expressed as an expanded uncertainty with a coverage factor k=2, corresponding to the level of confidence of about 95%, estimated in accordance with the JCGM 100:2008 [8], and ISO Guide 35 [5].

4. METROLOGICAL TRACEABILITY AND COMMUTABILITY

All standard solutions employed for calibration by laboratories participating in the characterization study were CRM which implies that the mass fractions of the trace elements in the respective standard solutions are traceable to the international system of units (SI), e.g. by comparative measurement against another CRM with established SI traceability. In addition, the IAEA-458 matrix CRM, was used for validation of the methods applied in this study for most of the analytes. Therefore, results used to obtain the certified values are considered as individually traceable to SI, inferring the traceability of certified values.

The trust in the certified values and their trueness are further underpinned by the agreement among the technically accepted datasets obtained with different analytical methods, as shown in Figs 34–48. Results obtained from non-destructive methodologies confirm the absence of bias arising from sample digestion.

The degree of equivalence in the analytical behaviour of real samples and a CRM with respect to various measurement procedures (methods) is summarized in a concept called 'commutability of a reference material'. Various definitions describe this concept, ISO defines it as: "the ability of the reference material, characterized by one measurement procedure (usually a reference procedure) to act as a calibrator or quality control material for a second measurement or testing procedure applied to routine test materials." [5]

The demonstration of commutability of matrix CRMs is not mandatory [5] especially if the matrix and handling are similar to the routine sample. IAEA-475 is a natural marine sediment sample, the analytical behaviour should be the same as for a routine sediment sample. The agreement between results obtained with different analytical methods selected for the IAEA-475 characterization study confirms the absence of any significant method bias and demonstrates commutability of the material for all certified trace elements.

5. CONCLUSIONS

This certification campaign allows assignment of certified values for Al, As, Co, Cr, Cu, Fe, Hg, Li, Mn, Ni, Pb, Sr and Zn with associated uncertainties following ISO guidelines. The certified values are derived from measurement results provided by the laboratories participating in the characterization study. Only validated methods were applied in the characterization of IAEA-475 CRM. As the certified values are combinations of SI traceable individual results, they are also traceable to the international system of units.

RESULTS FROM THE SHORT TERM STABILITY STUDY AT 60°C

Figures 2–17 present unit averages mass fractions per time point. The error bars represent the standard deviation of all results (two bottles per time, analysed in duplicate). In all figures the results are plotted versus storage duration at elevated temperature. In addition, the certified (or informative) value is plotted as a bold line, while the dashed lines represent the certified (or informative) value ± its expanded uncertainty ($k=2$) associated.
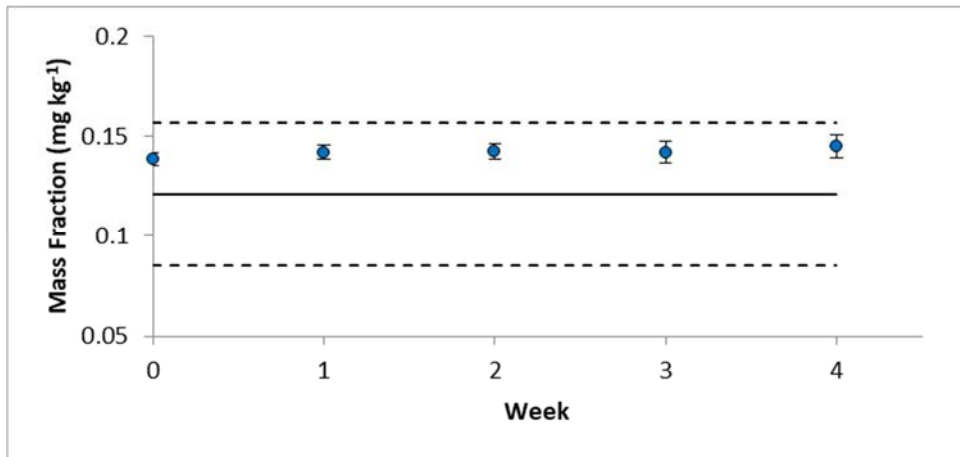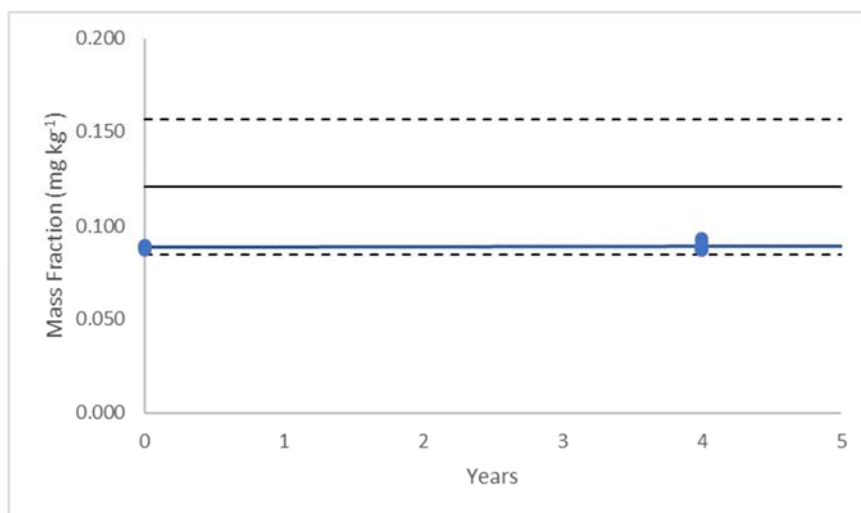

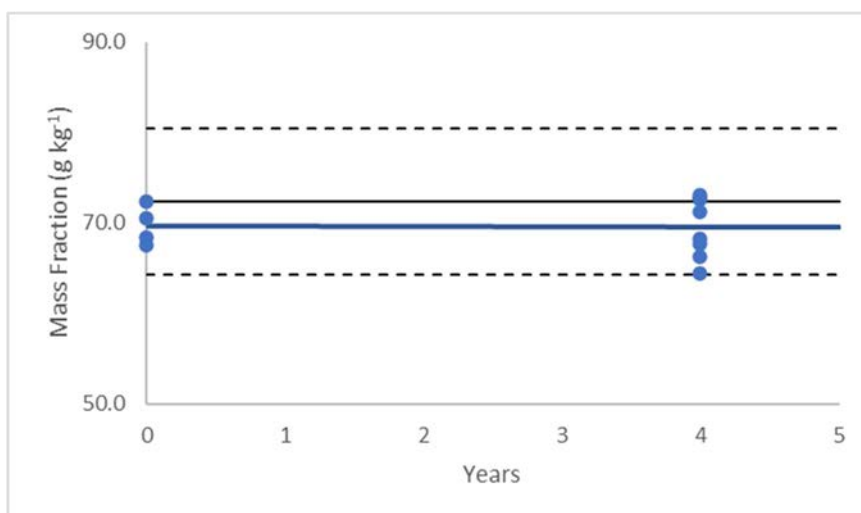*FIG. 2. Results of short term stability study for silver.*
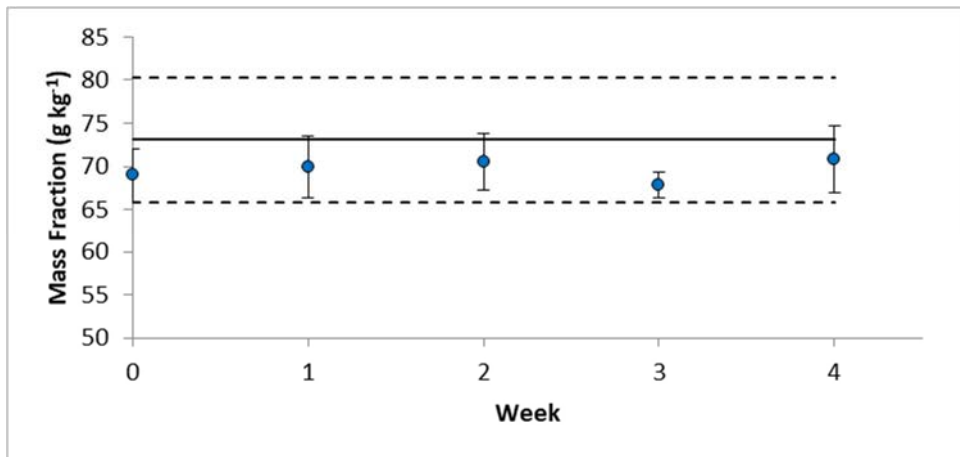
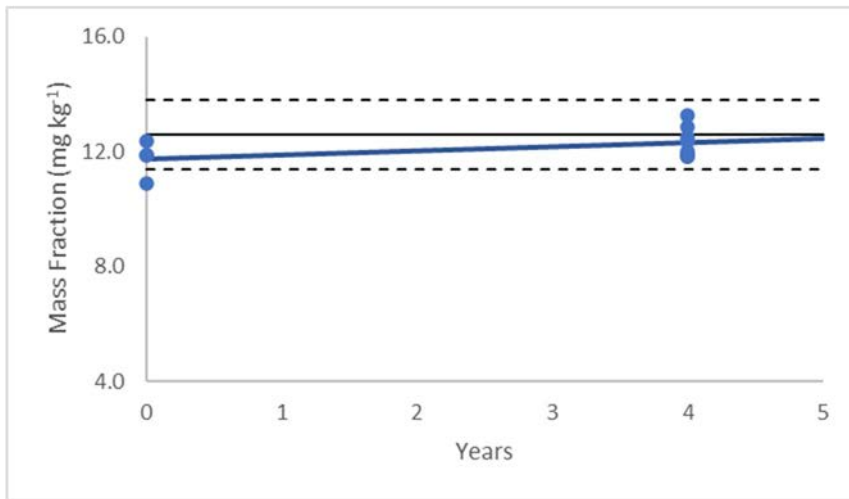*FIG. 3. Results of short term stability study for aluminium.*
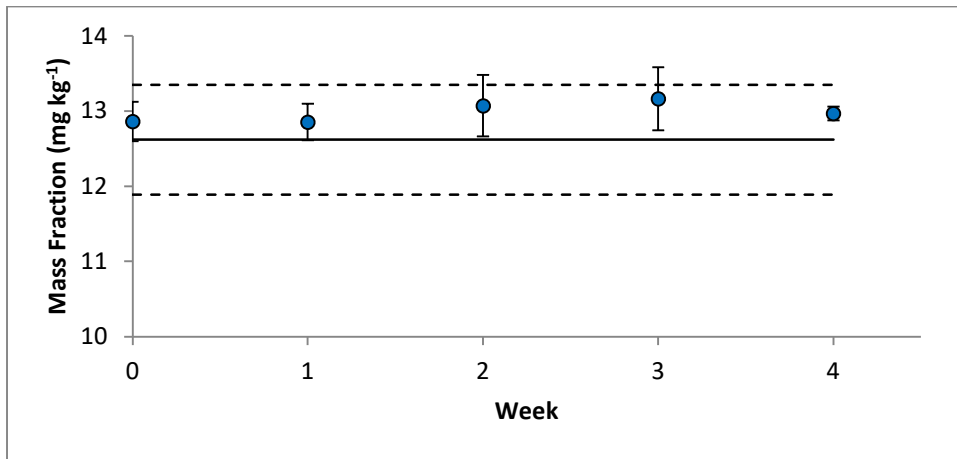
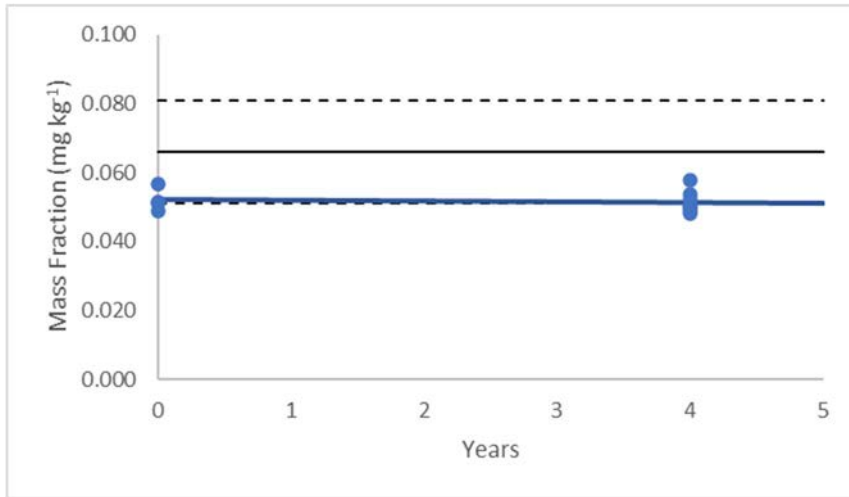*FIG. 4. Results of short term stability study for arsenic.*
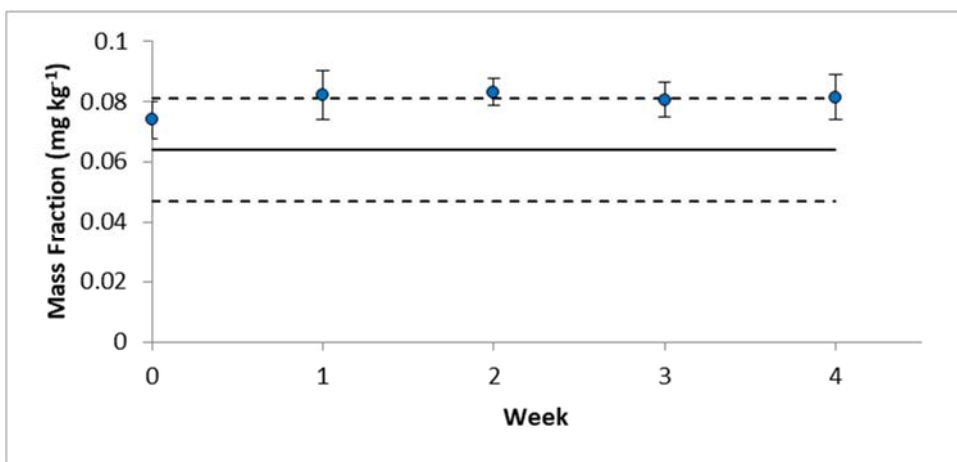
FIG. 5. Results of short term stability study for cadmium.
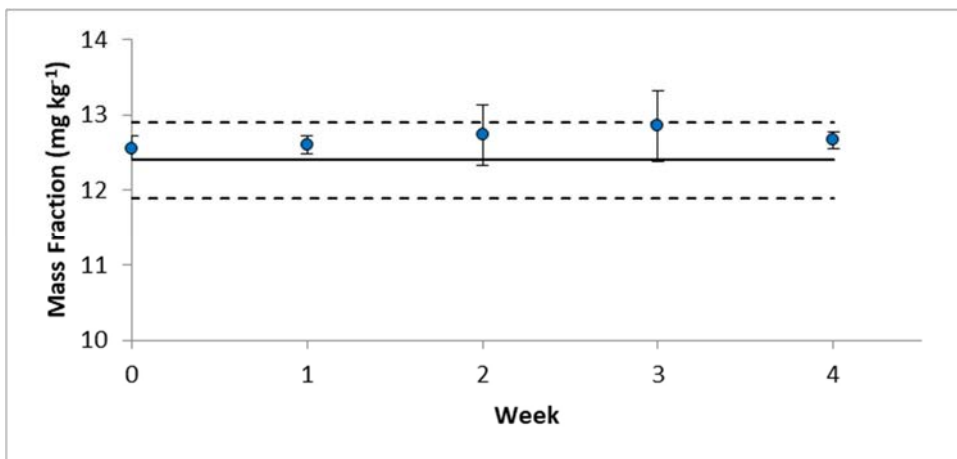
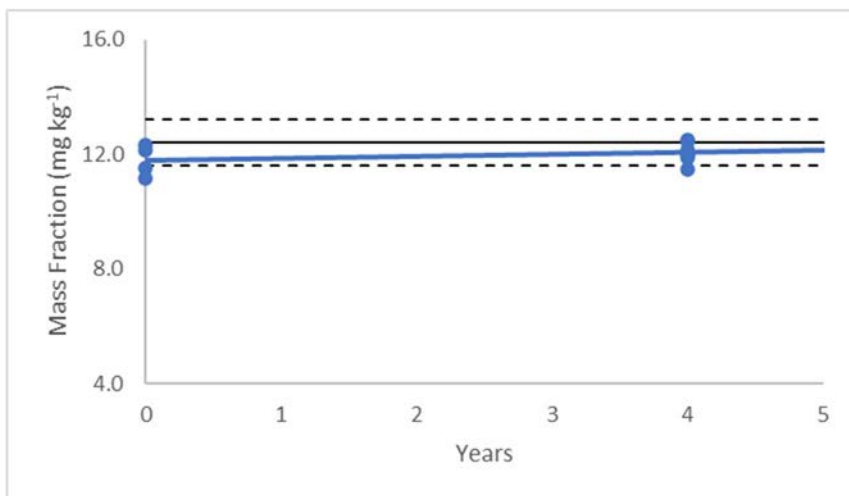
FIG. 6. Results of short term stability study for cobalt.
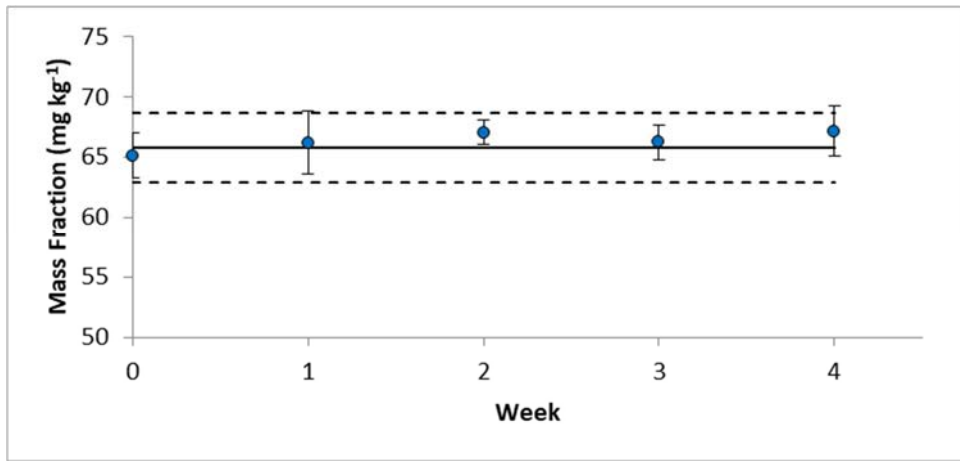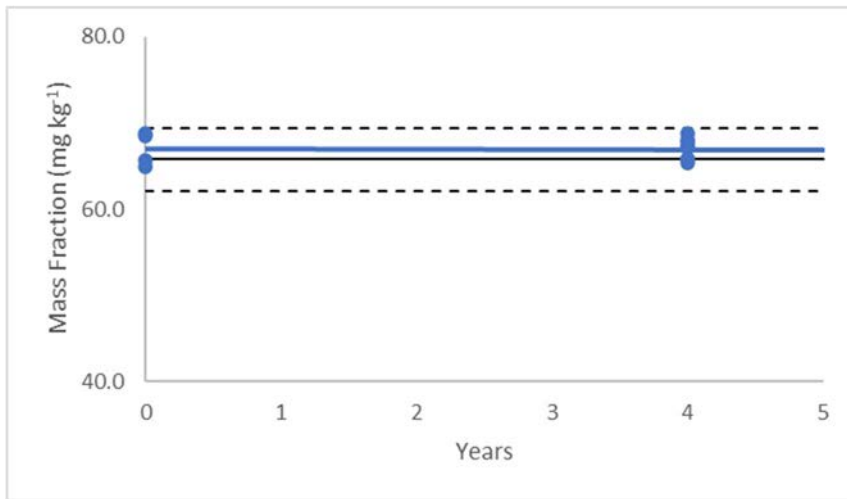
FIG. 7. Results of short term stability study for chromium.
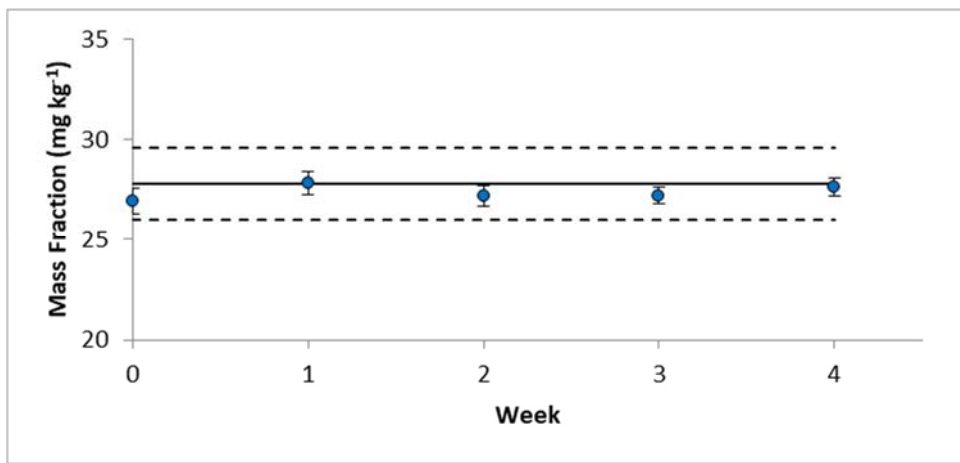
FIG. 8. Results of short term stability study for copper.
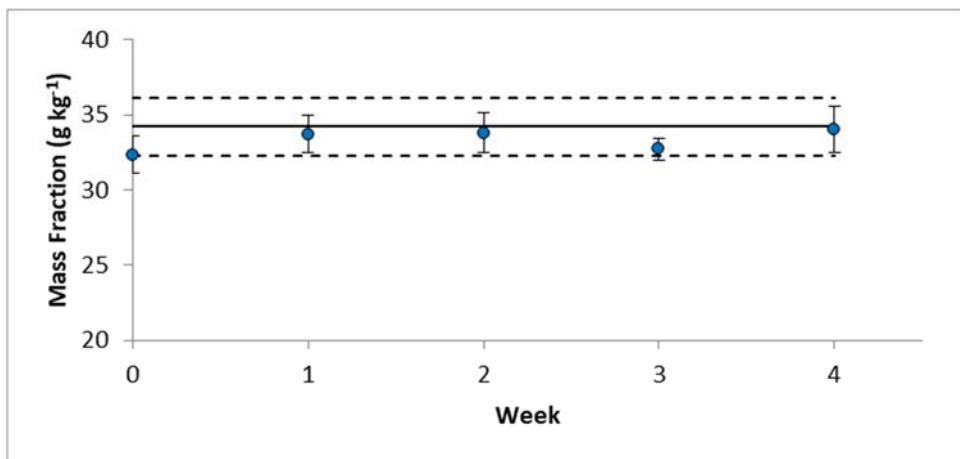
FIG. 9. Results of short term stability study for iron.
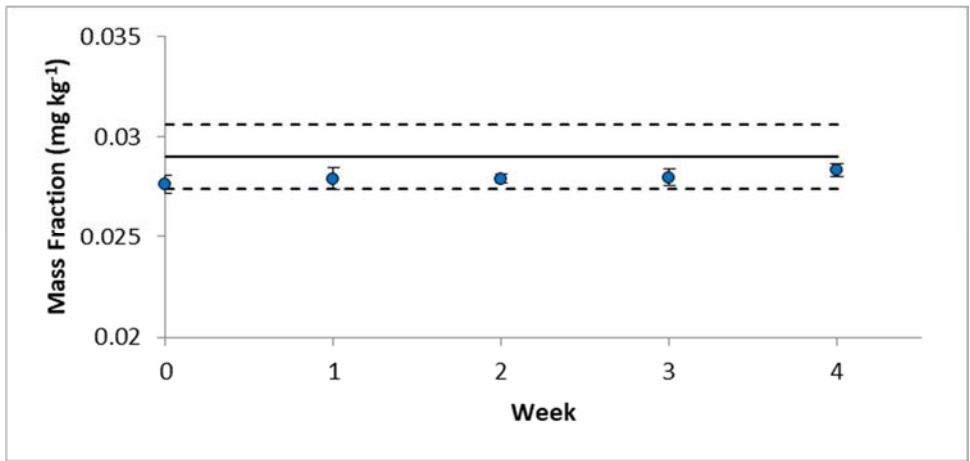
FIG. 10. Results of short term stability study for mercury.
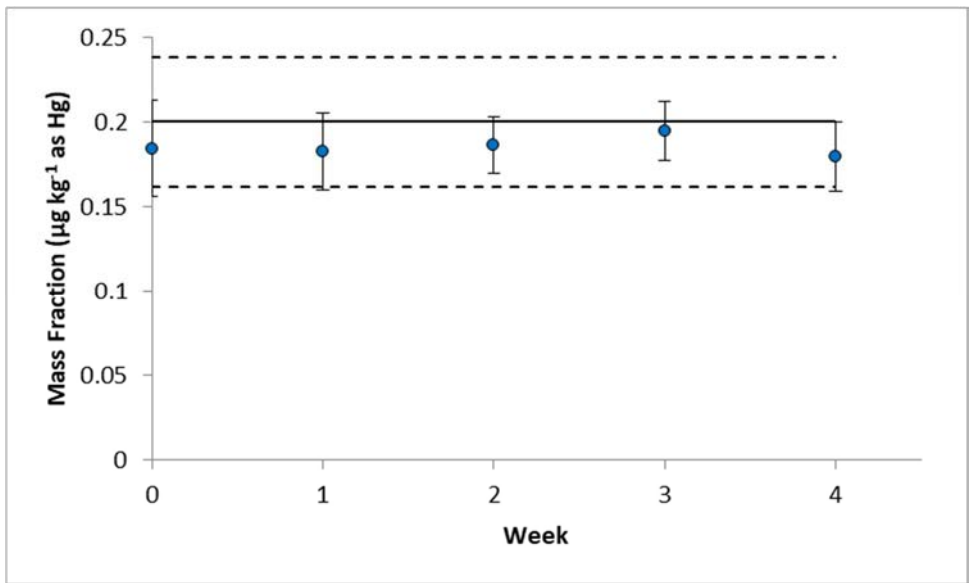
FIG. 11. Results of short term stability study for methylmercury.
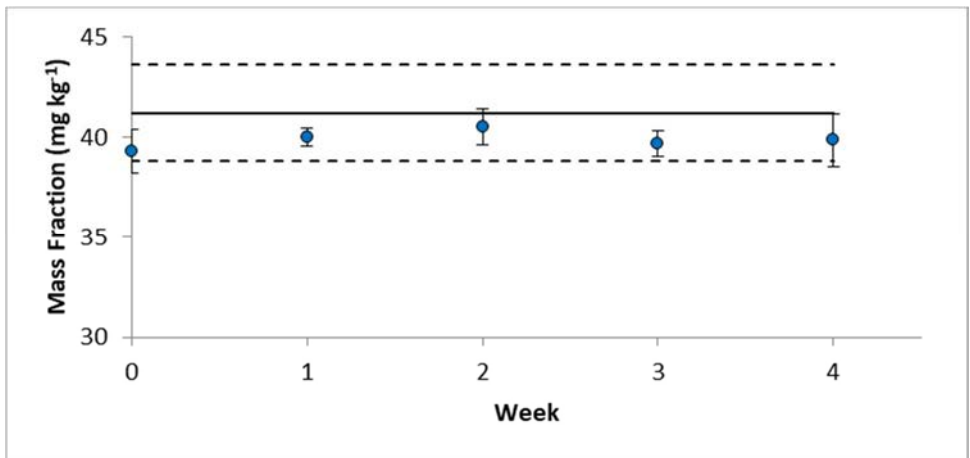
FIG. 12. Results of short term stability study for lithium.

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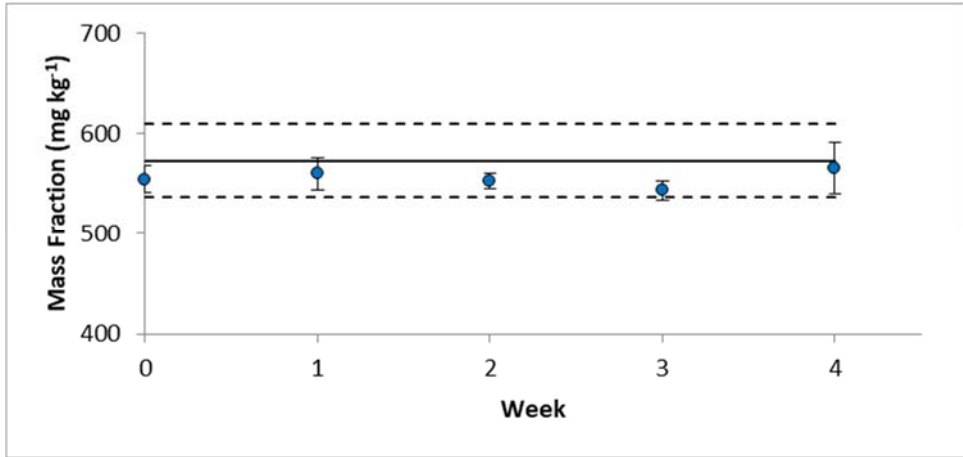
FIG. 13. Results of short term stability study for manganese.
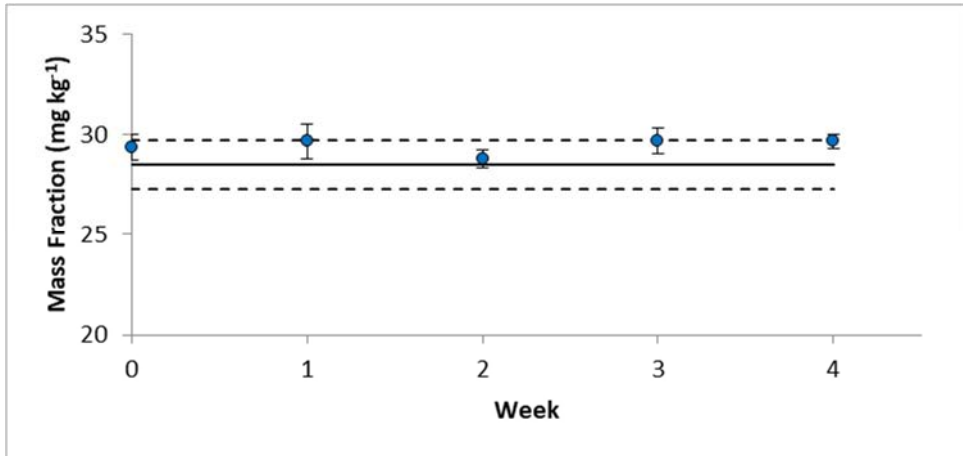
FIG. 14. Results of short term stability study for nickel.
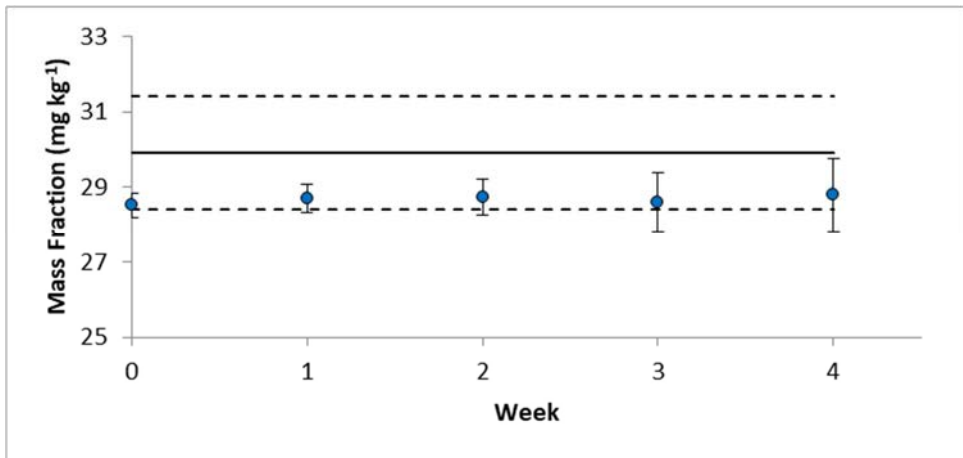
FIG. 15. Results of short term stability study for lead.
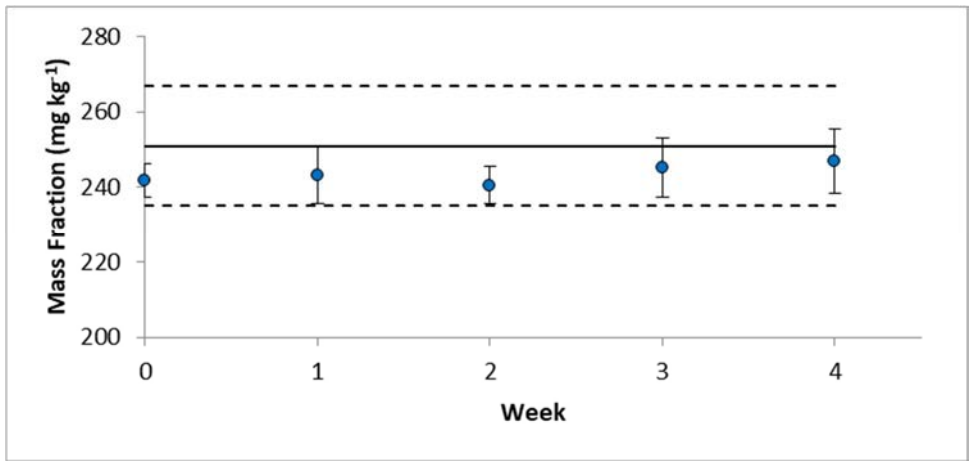
FIG. 16. Results of short term stability study for strontium.
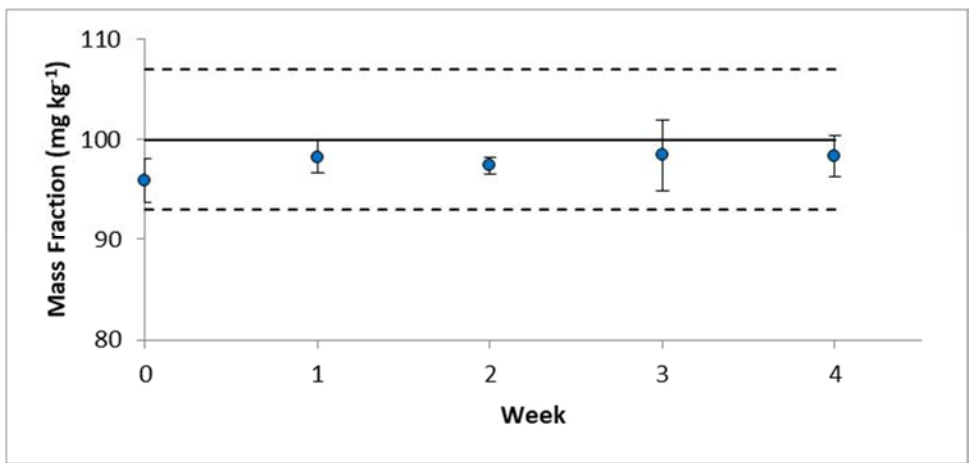
FIG. 17. Results of short term stability study for zinc.

APPENDIX II

RESULTS FROM THE LONG TERM STABILITY STUDY AT 20°C

Figures 18–33 present individual mass fractions per time point. In all figures, the results are plotted versus storage duration. In addition, the certified (or informative) value is plotted as a bold line, while the dashed lines represent the certified (or informative) value ± its expanded uncertainty (*k*=2) associated. The blue line represents the linear regression of mass fraction versus years.

FIG. 18. Results of long term stability study for silver.

FIG. 19. Results of long term stability study for aluminium.

FIG. 20. Results of long term stability study for arsenic.

FIG. 21. Results of long term stability study for cadmium.

FIG. 22. Results of long term stability study for cobalt.

FIG. 23. Results of long term stability study for chromium.
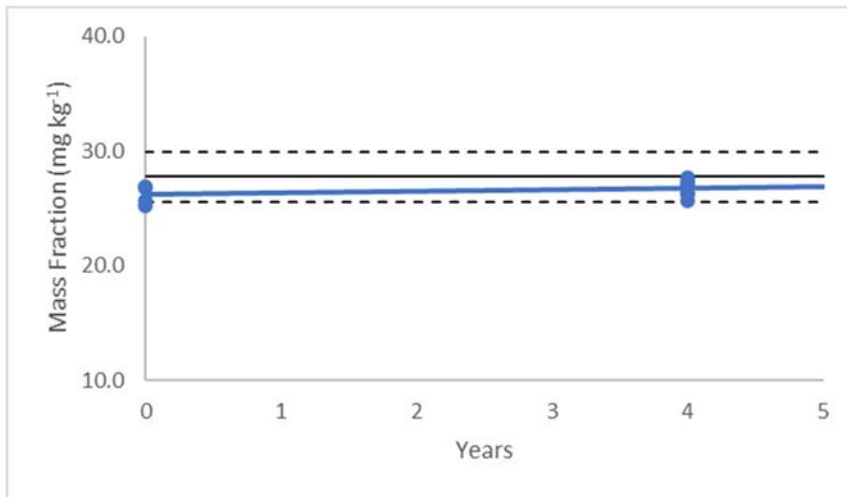

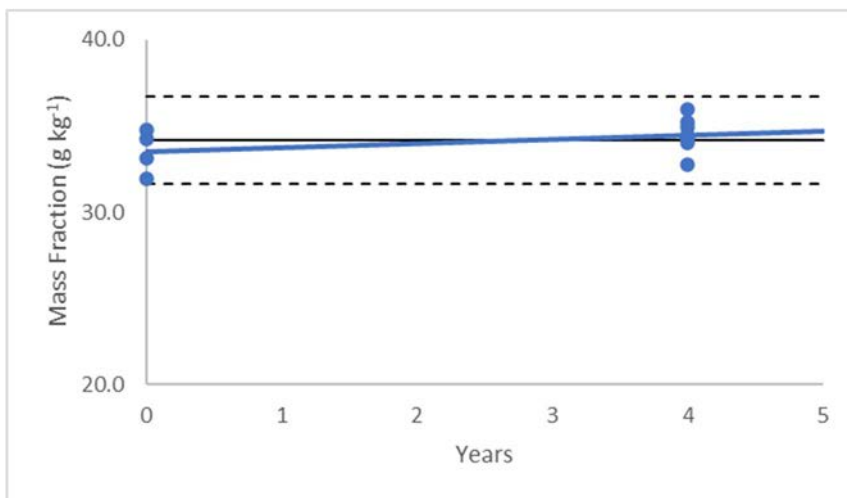*FIG. 24. Results of long term stability study for copper.*

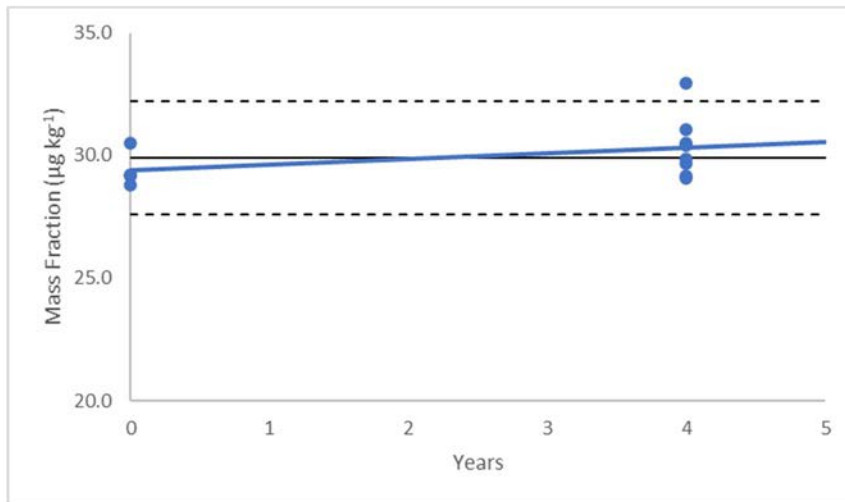*FIG. 25. Results of long term stability study for iron.*

FIG. 26. Results of long term stability study for mercury.
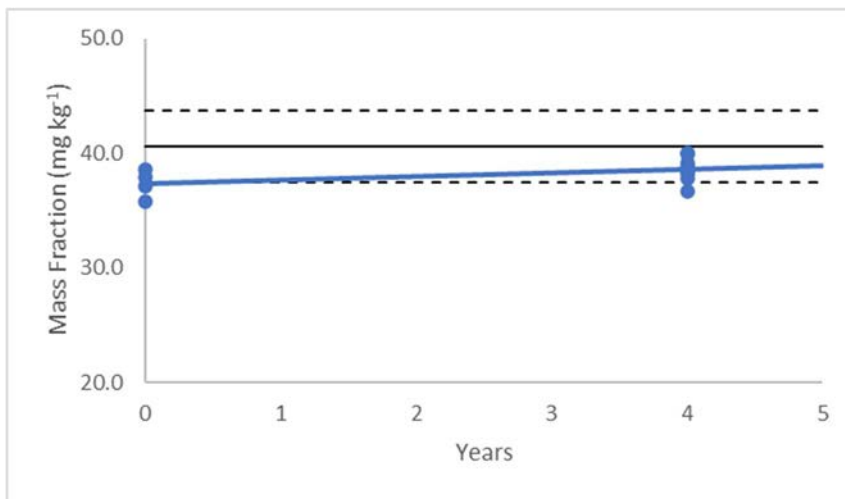

FIG. 27. Results of long term stability study for lithium.
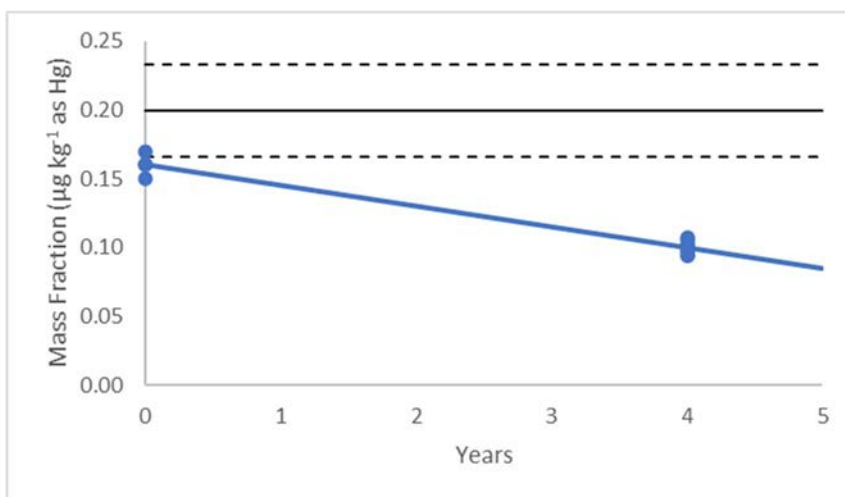

FIG. 28. Results of long term stability study for methylmercury.
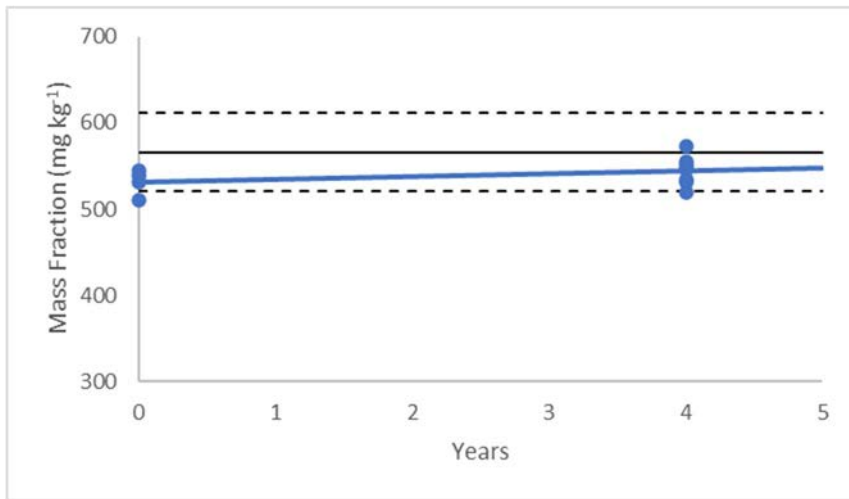
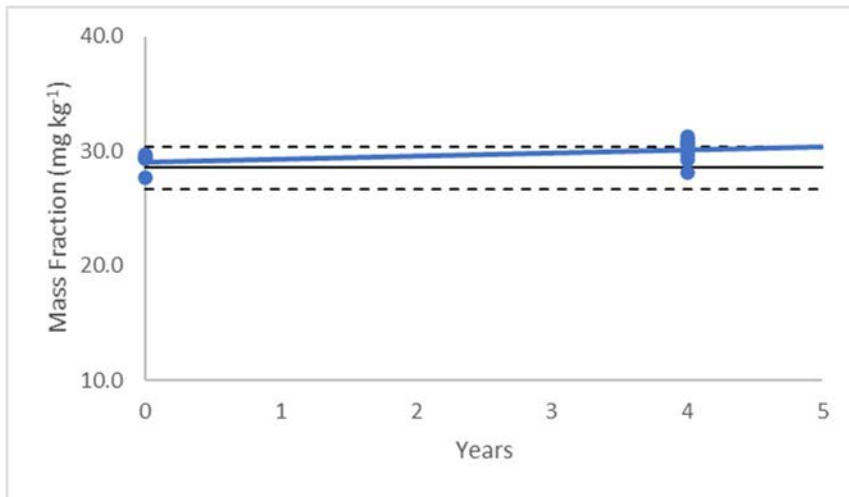*FIG. 29. Results of long term stability study for manganese.*

FIG. 30. Results of long term stability study for nickel.
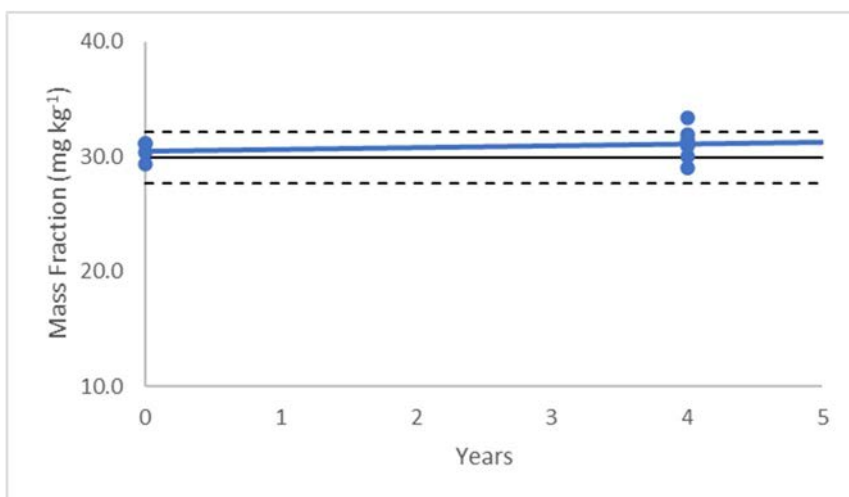

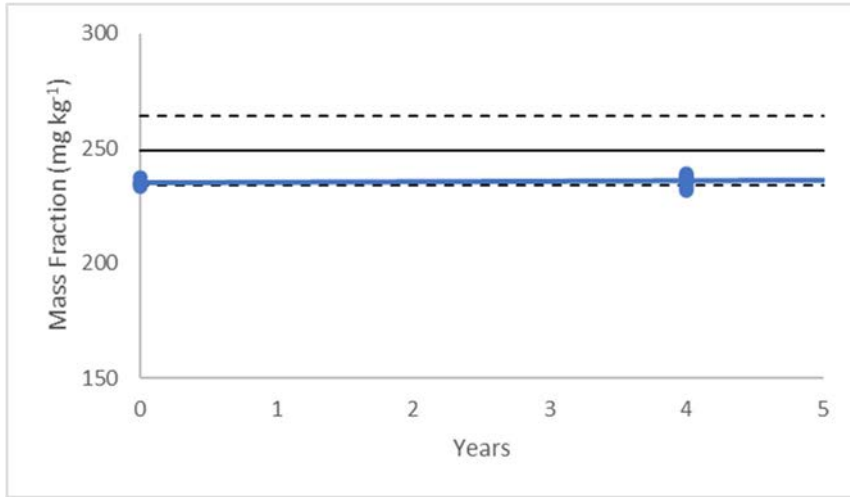
FIG. 31. Results of long term stability study for lead.

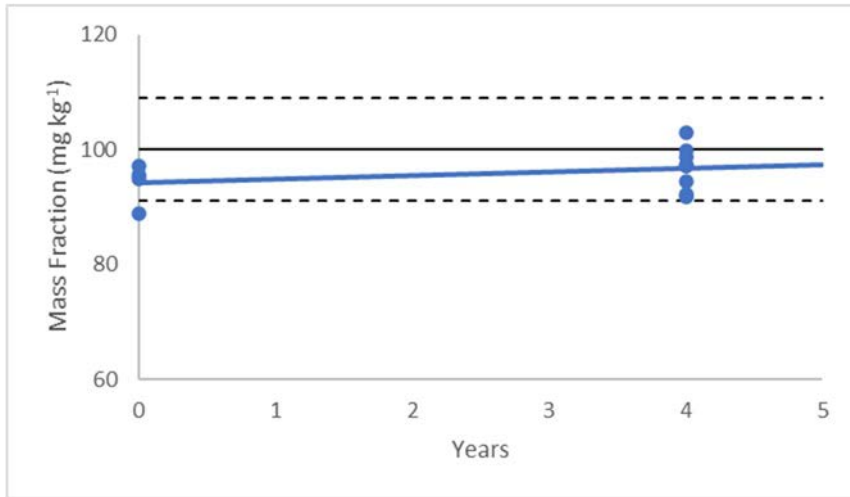*FIG. 32. Results of long term stability study for strontium.*

FIG. 33. Results of long term stability study for zinc.

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APPENDIX III

RESULTS OF THE CHARACTERIZATION MEASUREMENTS

The results reported by participants together with their expanded uncertainty and measurement techniques are presented in Tables 8–22. Figures 34–48 provide graphical presentation of the individual results and their expanded uncertainties as well as the assigned value for the respective trace element and its expanded uncertainty ($k=2$).

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

Laboratory code	Method	Mean	Expanded uncertainty (U)
7	ICP-MS	0.085	0.020
13	ICP-MS	0.135	0.035
IAEA-1	ICP-MS	0.142	0.022

FIG. 34. *Laboratory results for silver mass fraction (mg kg^{-1}) in IAEA-475.*
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TABLE 9. ALUMINIUM: RESULTS AS REPORTED BY PARTICIPANTS (g kg^{-1})

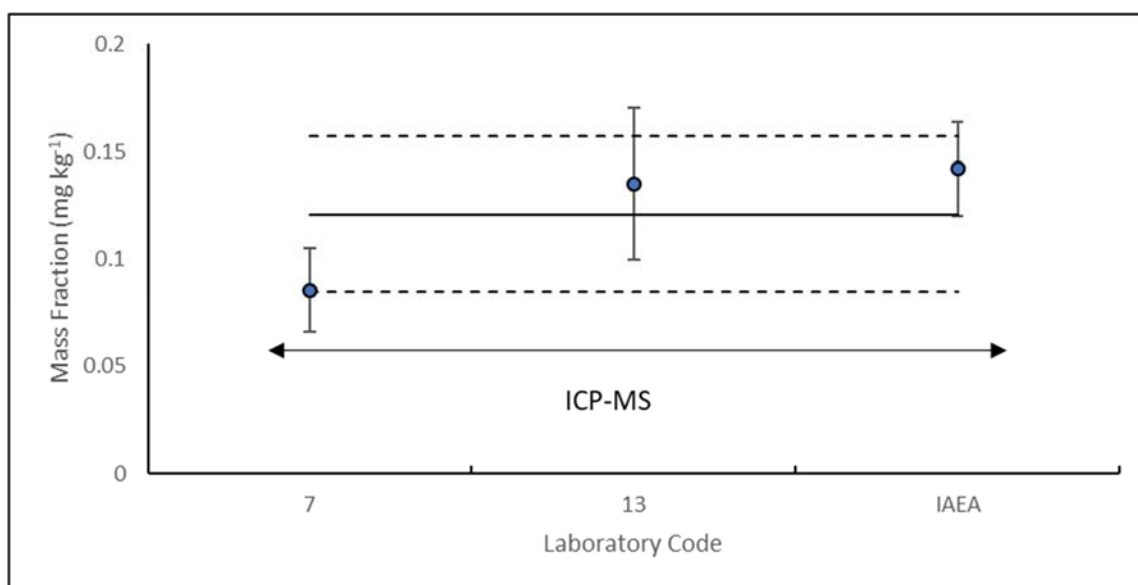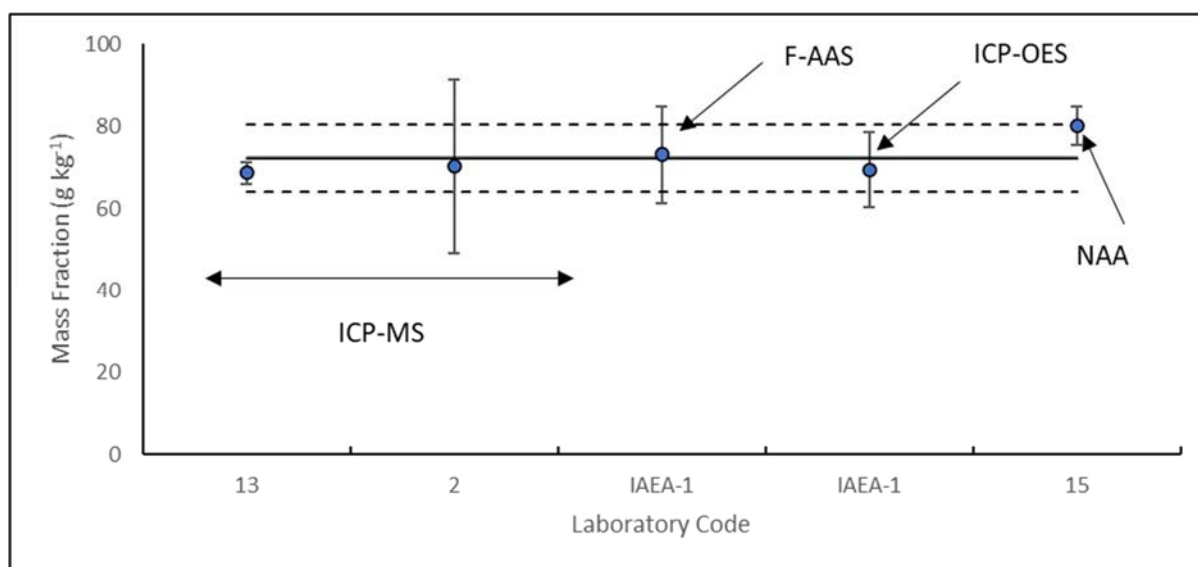| Laboratory code	Method	Mean	Expanded uncertainty (U)
13	ICP-MS	68.8	2.6
2	ICP-MS	70.3	21.1
IAEA-1	Flame AAS	73.2	11.7
IAEA-1	ICP-OES	69.6	9.0
15	Neutron activation	80.2	4.7

FIG. 35. Laboratory results for aluminium mass fraction (mg kg^{-1}) in IAEA-475.

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

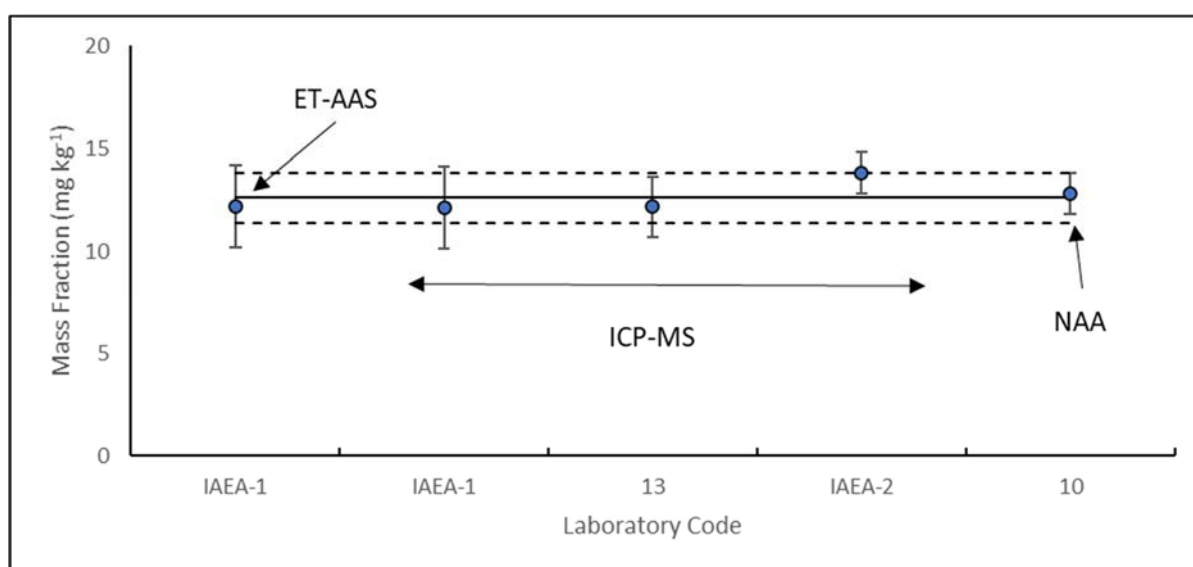| Laboratory code	Method	Mean	Expanded uncertainty (U)
IAEA-1	Graphite furnace AAS	12.2	2.0
IAEA-1	ICP-MS	12.1	2.0
13	ICP-MS	12.2	1.4
IAEA-2	ICP-MS	13.8	1.0
10	Neutron activation (k$_0$-INAA)	12.8	1.0
Results not used in certification			
7	ICP-MS	14.7	3.2
15	Neutron activation	12.9	0.8

FIG. 36. Laboratory results for arsenic mass fraction (mg kg^{-1}) in IAEA-475.

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

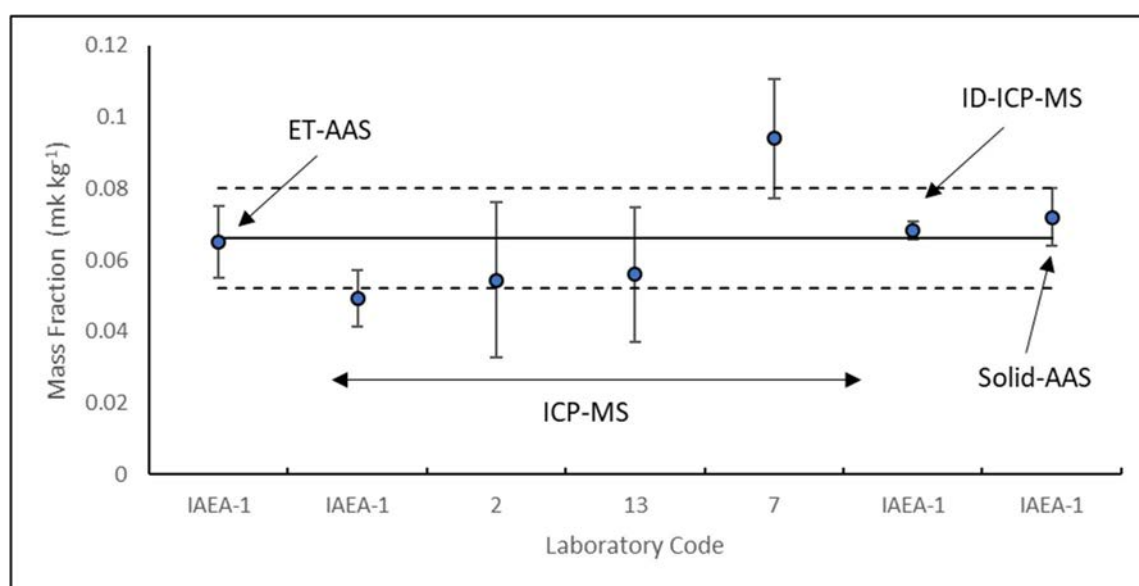| Laboratory code	Method	Mean	Expanded uncertainty (U)
IAEA-1	Graphite furnace AAS	0.065	0.010
IAEA-1	ICP-MS	0.049	0.008
2	ICP-MS	0.054	0.022
13	ICP-MS	0.056	0.019
7	ICP-MS	0.094	0.017
IAEA-1	Isotope dilution ICP-MS	0.068	0.002
IAEA-1	Solid-AAS	0.072	0.008

FIG. 37. Laboratory results for cadmium mass fraction (mg kg^{-1}) in IAEA-475.

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

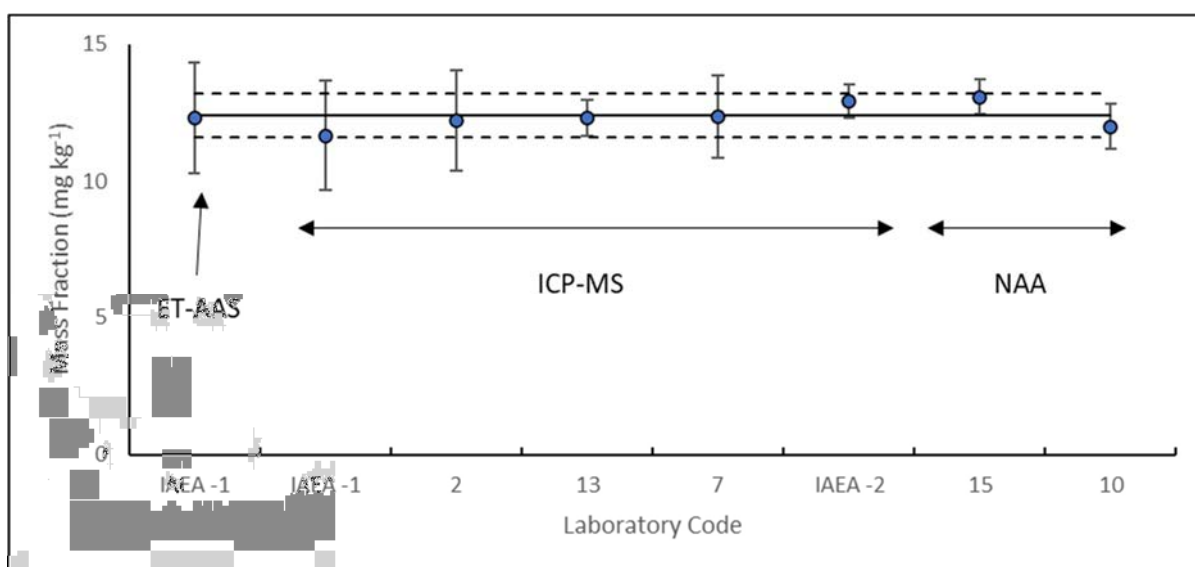| Laboratory code	Method	Mean	Expanded uncertainty (U)
IAEA -1	Graphite furnace AAS	12.3	2.0
IAEA -1	ICP-MS	11.7	2.0
2	ICP-MS	12.2	1.8
13	ICP-MS	12.3	0.6
7	ICP-MS	12.4	1.5
IAEA -2	ICP-MS	12.9	0.6
15	Neutron activation	13.1	0.6
10	Neutron activation (k$_0$-INAA)	12.0	0.8

FIG. 38. Laboratory results for cobalt mass fraction (mg kg $^{-1}$) in IAEA-475.

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

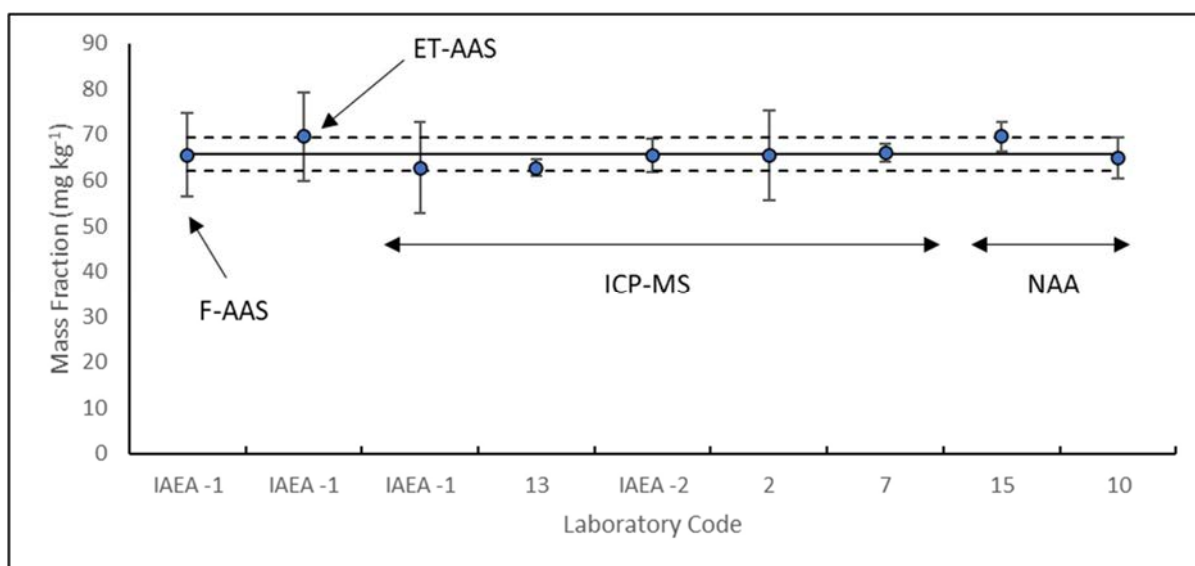| Laboratory code	Method	Mean	Expanded uncertainty (U)
IAEA -1	Flame AAS	65.6	9.2
IAEA -1	Graphite furnace AAS	69.6	9.7
IAEA -1	ICP-MS	62.7	10.0
13	ICP-MS	62.7	1.8
IAEA -2	ICP-MS	65.4	3.7
2	ICP-MS	65.5	9.8
7	ICP-MS	66.0	2.0
15	Neutron activation	69.6	3.3
10	Neutron activation (k$_0$-INAA)	64.9	4.6

FIG. 39. Laboratory results for chromium mass fraction (mg kg^{-1}) in IAEA-475.

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

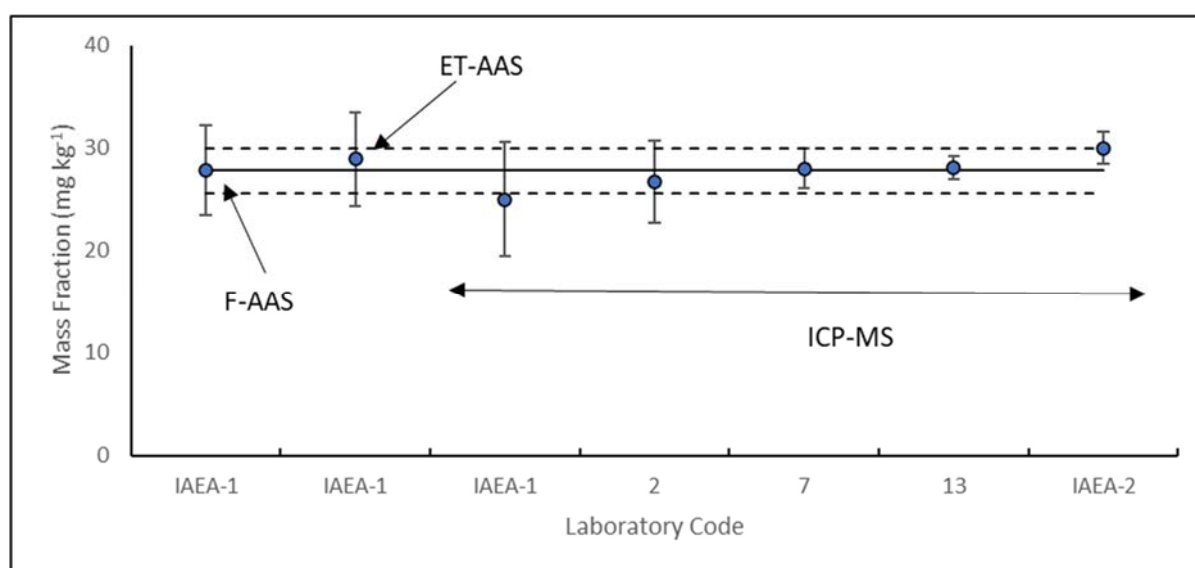| Laboratory code	Method	Mean	Expanded uncertainty (U)
IAEA-1	Flame AAS	27.8	4.4
IAEA-1	Graphite furnace AAS	28.9	4.6
IAEA-1	ICP-MS	25.0	5.6
2	ICP-MS	26.7	4.0
7	ICP-MS	28.0	2.0
13	ICP-MS	28.1	1.1
IAEA-2	ICP-MS	30.0	1.6

FIG. 40. Laboratory results for copper mass fraction (mg kg^{-1}) in IAEA-475.

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

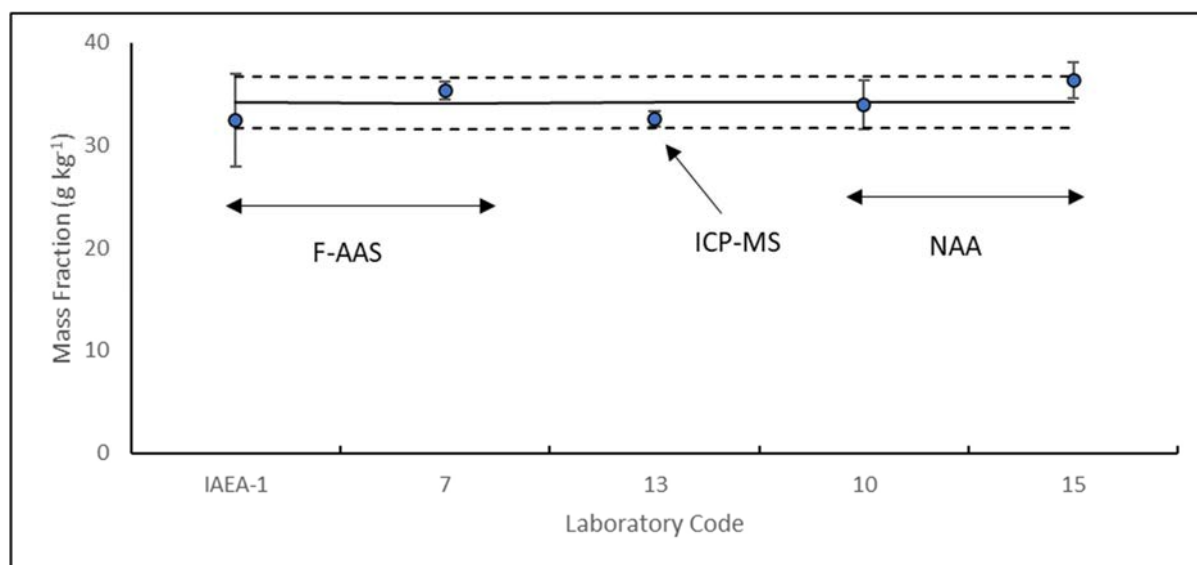| Laboratory code	Method	Mean	Expanded uncertainty (U)
IAEA-1	Flame AAS	32.5	4.5
7	Flame AAS	35.4	0.8
13	ICP-MS	32.6	0.8
10	Neutron activation (k$_0$-INAA)	33.9	2.4
15	Neutron activation	36.4	1.7
Results not used in certification			
2	ICP-MS	35.0	10.5

FIG. 41. Laboratory results for iron mass fraction (g kg^{-1}) in IAEA-475.

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

Laboratory code	Method	Mean	Expanded uncertainty (U)
10	Cold vapour AAS	0.0288	0.0026
7	Cold vapour AAS	0.0310	0.0078
13	Cold vapour AFS	0.0303	0.0033
8	Solid Hg analyser	0.0278	0.0047
IAEA-1	Solid Hg analyser	0.0300	0.0040
IAEA-1	Solid Hg analyser	0.0288	0.0040
IAEA-1	Cold vapour ICP-MS	0.0321	0.0026
IAEA-1	Cold vapour ID ICP-MS	0.0302	0.0013
Results not used in certification			
2	Solid Hg analyser	0.0283	0.0085

FIG. 42. Laboratory results for mercury mass fraction (mg kg^{-1}) in IAEA-475.

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

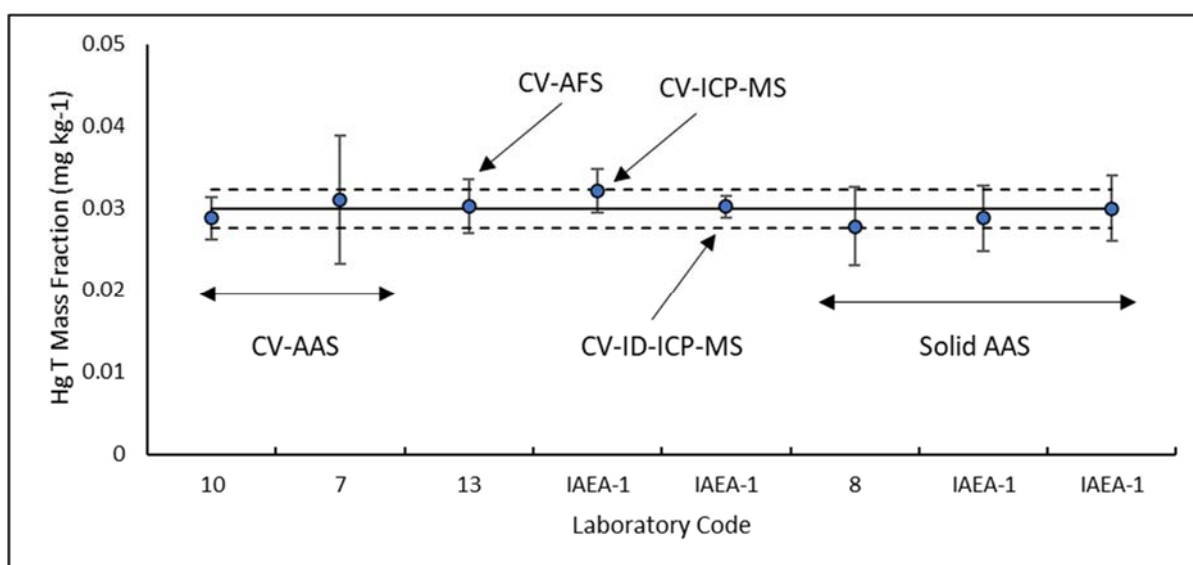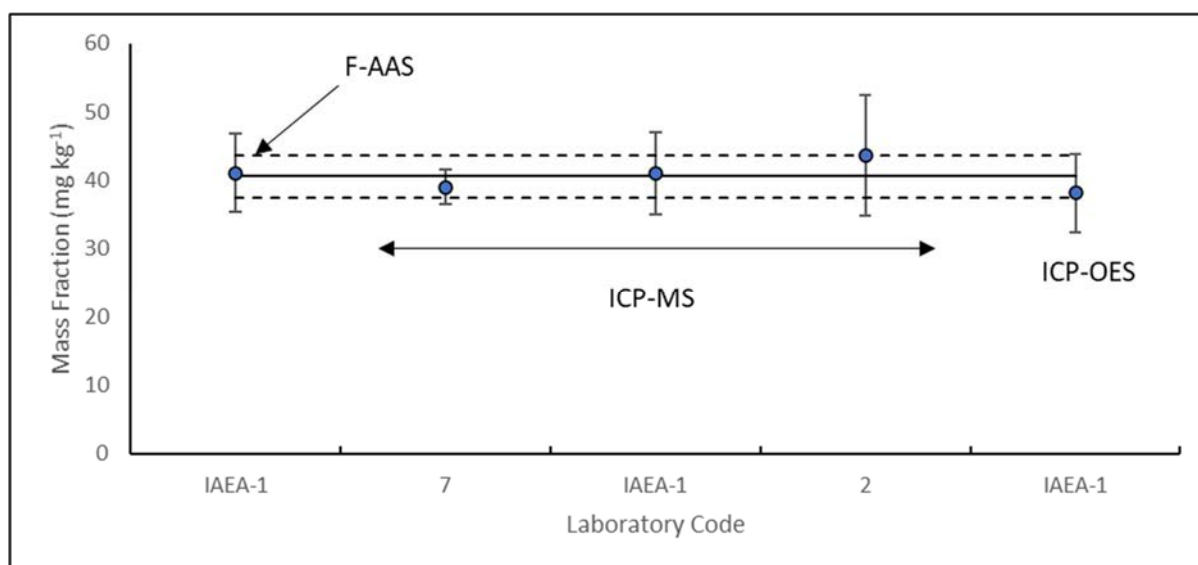| Laboratory code	Method	Mean	Expanded uncertainty (U)
IAEA-1	ICP-OES	38.2	5.7
7	ICP-MS	39.0	2.6
IAEA-1	ICP-MS	41.0	6.0
IAEA-1	Flame AAS	41.1	5.7
2	ICP-MS	43.7	8.7

FIG. 43. Laboratory results for lithium mass fraction (mg kg^{-1}) in IAEA-475.

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

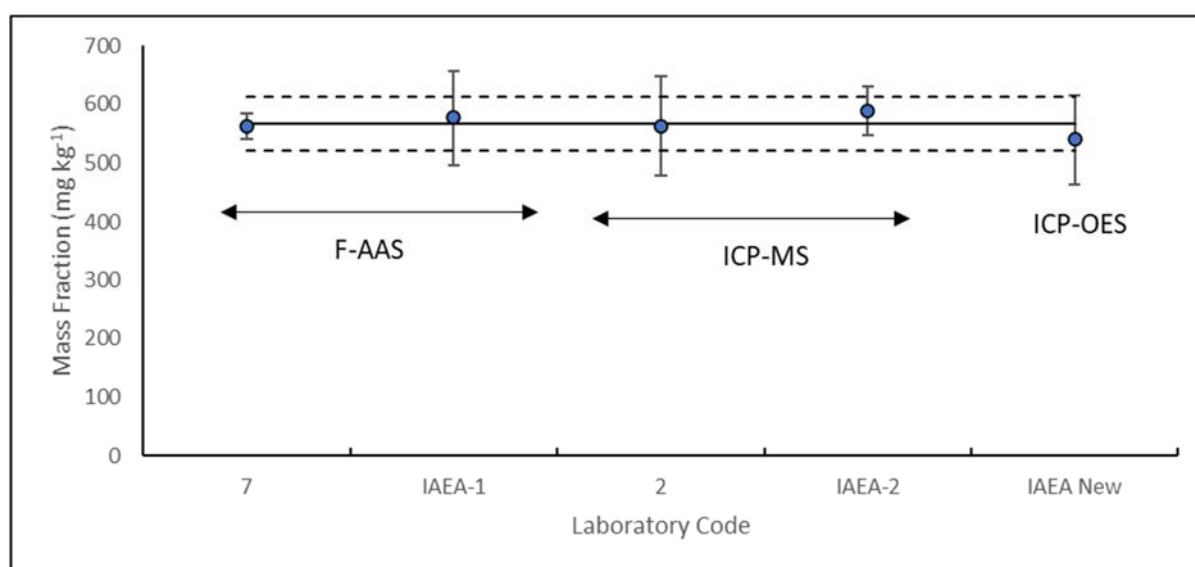| Laboratory code	Method	Mean	Expanded uncertainty (U)
7	Flame AAS	562	21
IAEA-1	Flame AAS	577	80
2	ICP-MS	563	84
IAEA-2	ICP-MS	589	42
IAEA-1	ICP-OES	540	76
Results not used in certification			
13	ICP-MS	555	36
15	Neutron activation	563	32

FIG. 44. Laboratory results for manganese mass fraction (mg kg^{-1}) in IAEA-475.

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

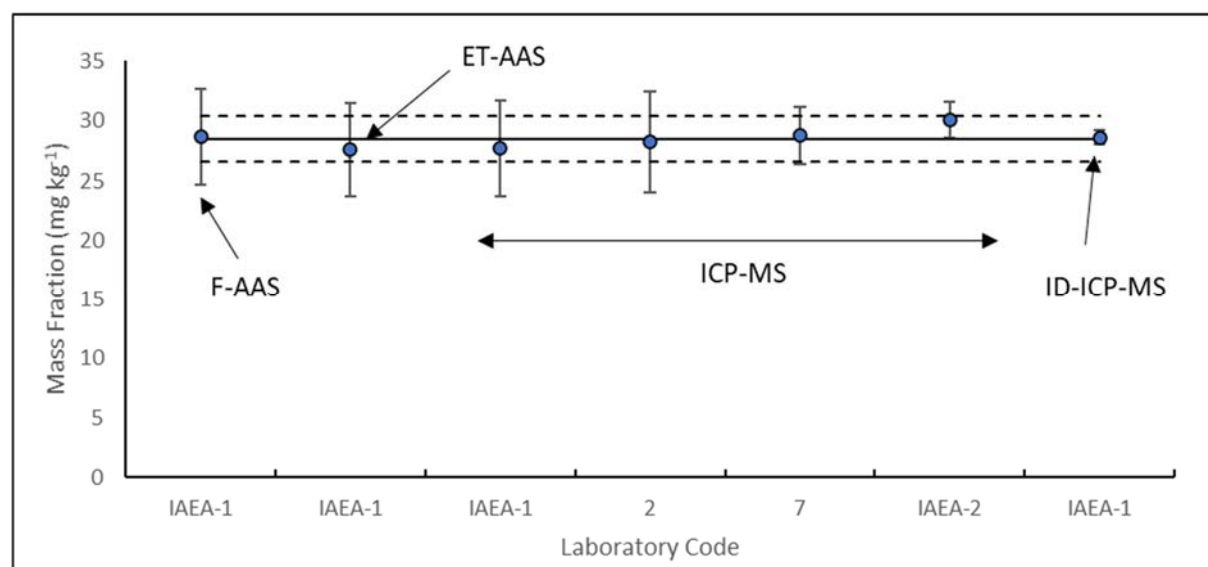| Laboratory code	Method	Mean	Expanded uncertainty (U)
IAEA-1	Flame AAS	28.7	4.0
IAEA-1	Graphite furnace AAS	27.6	3.9
IAEA-1	ICP-MS	27.7	4.0
2	ICP-MS	28.2	4.2
7	ICP-MS	28.8	2.4
IAEA-2	ICP-MS	30.1	1.5
IAEA-1	Isotope dilution ICP-MS	28.6	0.6
Results not used in certification			
13	ICP-MS	24.7	2.2

FIG. 45. Laboratory results for nickel mass fraction (mg kg^{-1}) in IAEA-475.

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

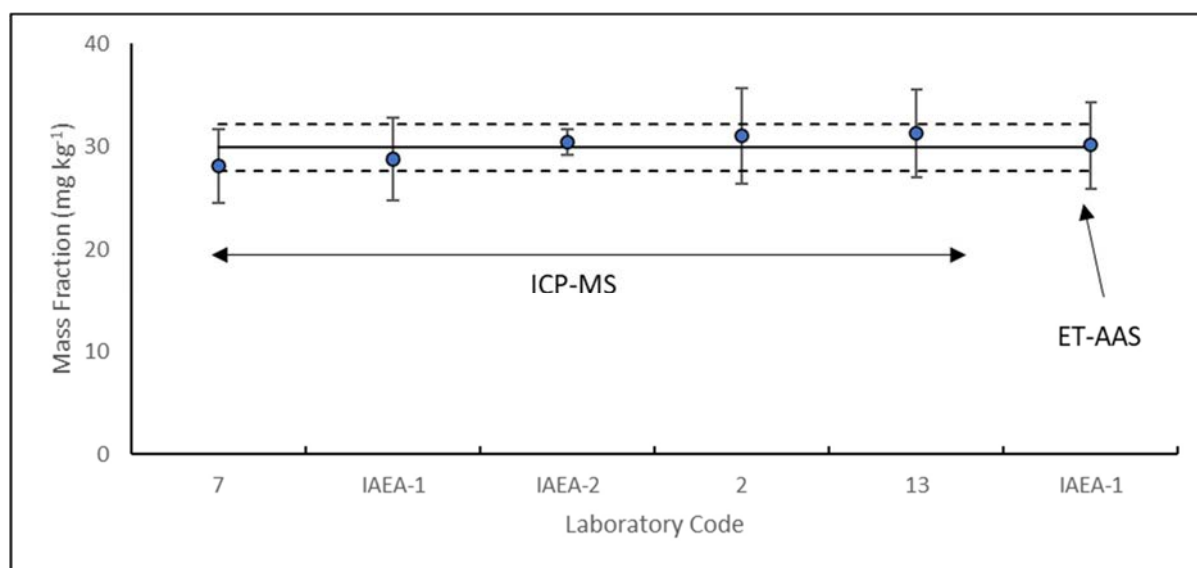| Laboratory code	Method	Mean	Expanded uncertainty (U)
7	ICP-MS	28.1	3.6
IAEA-1	ICP-MS	28.7	4.0
IAEA-2	ICP-MS	30.4	1.3
2	ICP-MS	31.0	4.7
13	ICP-MS	31.3	4.3
IAEA-1	Graphite furnace AAS	30.1	4.2

FIG. 46. Laboratory results for lead mass fraction (mg kg^{-1}) in IAEA-475.

TABLE 21. STRONTIUM: RESULTS AS REPORTED BY PARTICIPANTS (mg kg^{-1})

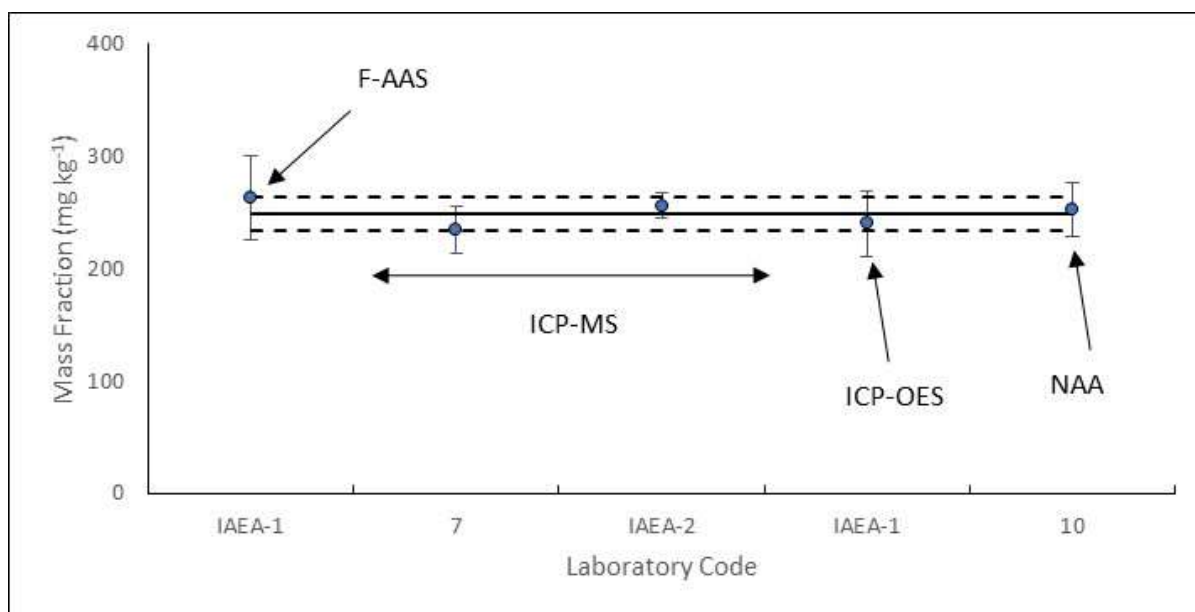| Laboratory code	Method	Mean	Expanded uncertainty (U)
IAEA-1	Flame AAS	263	37
7	ICP-MS	235	21
IAEA-2	ICP-MS	256	11
IAEA-1	ICP-OES	240	29
10	Neutron activation (k$_0$-INAA)	252	24
Results not used in certification			
13	ICP-MS	252	7

FIG. 47. Laboratory results for strontium mass fraction (mg kg^{-1}) in IAEA-475.

TABLE 22. ZINC: RESULTS AS REPORTED BY PARTICIPANTS (mg kg^{-1})

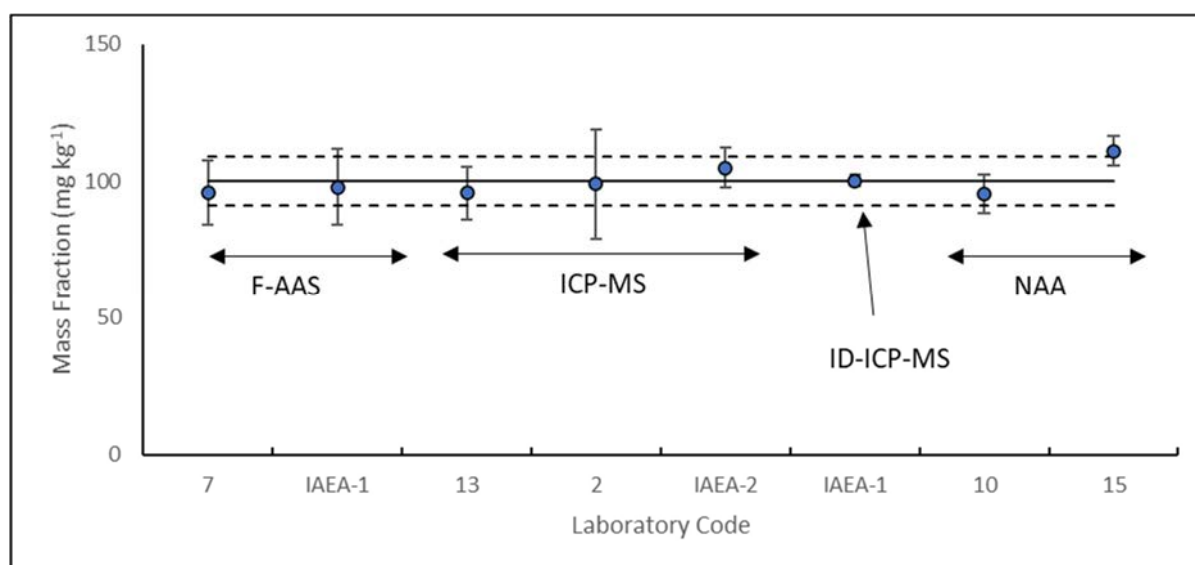| Laboratory code	Method	Mean	Expanded uncertainty (U)
7	Flame AAS	96.0	11.8
IAEA-1	Flame AAS	97.9	13.7
13	ICP-MS	95.7	9.5
2	ICP-MS	99.0	19.8
IAEA-2	ICP-MS	105	7
IAEA-1	Isotope dilution ICP-MS	100	2
10	Neutron activation (k$_0$-INAA)	95.6	7.0
15	Neutron activation	111	5

FIG. 48. Laboratory results for zinc mass fraction (mg kg^{-1}) in IAEA-475.

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