



CERTIFICATION REPORT

**The certification of trace elements mass fraction in
electrolytic copper with added impurities:
ERM[®]-EB075A, B and C**



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Abstract

This report describes the production of ERM[®]-EB075A, B and C, a copper material with added impurities certified for the mass fraction of Ag, Al, As, Au, Be, Bi, Cd, Co, Cr, Fe, In, Mg, Mn, Ni, P, Pb, S, Sb, Se, Si, Sn, Te, Ti, Zn and Zr. The material was produced following ISO Guide 34:2009.

A melt was produced with pure copper and alloy to obtain a pure copper material with added impurities. After casting, the material was processed by hot extrusion and cold machining to produce discs of 39 mm diameter (ERM-EB075A), cylinders of 8 mm diameter (ERM-EB075B) and chips of 250 mg (ERM-EB075C).

Between unit-homogeneity was quantified and stability during dispatch and storage were assessed in accordance with ISO Guide 35:2006. Within-unit homogeneity was quantified to determine the minimum sample intake.

The material was characterised by an intercomparison among laboratories of demonstrated competence and adhering to ISO/IEC 17025. Technically invalid results were removed but no outlier was eliminated on statistical grounds only.

Uncertainties of the certified values were calculated in compliance with the Guide to the Expression of Uncertainty in Measurement (GUM) []; the total estimated uncertainty includes uncertainties related to possible inhomogeneity, instability and characterisation.

The material is intended for use in the quality control and assessment of method performance. As any reference material, it can also be used for control charts or validation studies. The certified reference material (CRM) is available in three different formats:

- ERM-EB075A: disc of 39 mm diameter; 30 mm thick; packed in a box
- ERM-EB075B: cylinder of 8 mm diameter; 100 mm length; sealed in a plastic sachet under vacuum
- ERM-EB075C: bottle of 50 g of chips; chip weight of approximately 250 mg

For ERM-EB075A, B and C, the minimum amount of sample to be used is 10 mg for the determination of Ag, Al, As, Be, Bi, Cd, Co, Cr, Fe, Mg, Mn, Ni, P, Pb, S, Sb, Se, Si, Sn, Ti, Zn; 20 mg for Au, In and Te.

The CRM was accepted as European Reference Material (ERM[®]) after peer evaluation by the partners of the European Reference Materials Consortium.



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Disclaimer

Certain commercial equipment, instruments, and materials are identified in this paper to specify adequately the experimental procedure. In no case does such identification imply recommendation or endorsement by the European Commission, nor does it imply that the material or equipment is necessarily the best available for the purpose.

Summary

This report describes the production of ERM®-EB075A, B and C, a copper material with added impurities certified for the mass fraction of Ag, Al, As, Au, Be, Bi, Cd, Co, Cr, Fe, In, Mg, Mn, Ni, P, Pb, S, Sb, Se, Si, Sn, Te, Ti, Zn and Zr. The material was produced following ISO Guide 34:2009 [1].

A melt was produced with pure copper and alloy to obtain a pure copper material with added impurities. After casting, the material was processed by hot extrusion and cold machining to produce discs of 39 mm diameter (ERM-EB075A), cylinders of 8 mm diameter (ERM-EB075B) and chips of 250 mg (ERM-EB075C).

Between-unit homogeneity was quantified and stability during dispatch and storage were assessed in accordance with ISO Guide 35:2006 [2]. Within-unit homogeneity was quantified to determine the minimum sample intake.

The material was characterised by an intercomparison among laboratories of demonstrated competence and adhering to ISO/IEC 17025 [3]. Technically invalid results were removed but no outlier was eliminated on statistical grounds only.

Uncertainties of the certified values were calculated in compliance with the Guide to the Expression of Uncertainty in Measurement (GUM) [4]; the total estimated uncertainty includes uncertainties related to possible inhomogeneity, instability and characterisation.

The material is intended for use in the quality control and assessment of method performance. As any reference material, it can also be used for control charts or validation studies. The certified reference material (CRM) is available in three different formats:

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- ERM-EB075C: bottle of 50 g of chips; chip weight of approximately 250 mg

For ERM-EB075A, B and C, the minimum amount of sample to be used is 10 mg for the determination of Ag, Al, As, Be, Bi, Cd, Co, Cr, Fe, Mg, Mn, Ni, P, Pb, S, Sb, Se, Si, Sn, Ti, Zn; 20 mg for Au, In and Te.

The CRM was accepted as European Reference Material (ERM®) after peer evaluation by the partners of the European Reference Materials Consortium.

The following certified values were assigned for ERM-EB075A, B and C:

	Mass Fraction	
	Certified value ¹⁾ [mg/kg]	Uncertainty ²⁾ [mg/kg]
Ag	10.8	0.6
Al	2.3	0.4
As	3.18	0.10
Au	1.46	0.14
Be	1.08	0.24
Bi	1.79	0.11
Cd	2.69	0.09
Co	2.64	0.08
Cr	1.40	0.07
Fe	9.3	0.4
In	1.83	0.10
Mg	7.0	0.7
Mn	1.35	0.07
Ni	2.18	0.16
P	2.59	0.30
Pb	4.8	0.9
S	25	4
Sb	2.93	0.14
Se	1.69	0.10
Si	2.6	0.4
Sn	2.13	0.11
Te	1.78	0.12
Ti	3.2	0.5
Zn	6.51	0.29

1) Unweighted mean value of the means of accepted sets of data, each set being obtained in a different laboratory and/or with a different method of determination. The certified value and its uncertainty are traceable to the International System of Units (SI).

2) The uncertainty of the certified value is the expanded uncertainty with a coverage factor $k = 2$ corresponding to a level of confidence of about 95 % estimated in accordance with ISO/IEC Guide 98-3, Guide to the Expression of Uncertainty in Measurement (GUM:1995), ISO, 2008.

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Glossary

AC	Alternating current
ASTM International	ASTM International (formerly American Society for Testing and Materials)
ANOVA	Analysis of variance
b	Slope in the equation of linear regression $y = a + bx$
BCR [®]	One of the trademarks of CRMs owned by the European Commission; formerly Community Bureau of Reference
BIPM	Bureau International des Poids et Mesures(International Bureau of Weights and Measures)
CEN	European Committee for Standardization
CI	Confidence interval
CRM	Certified reference material
DC	Direct current
EC	European Commission
EN	European norm (standard)
ERM [®]	Trademark of European Reference Materials
ETV	Electro-thermal vaporisation
EU	European Union
GD	Glow discharge
GUM	Guide to the Expression of Uncertainty in Measurements [4]
ICP	Inductively coupled plasma
ICP-MS	Inductively coupled plasma-mass spectrometry
IGF	Inert gas fusion
INAA	Instrumental neutron activation analysis
IR	Infra-red
IRMM	Institute for Reference Materials and Measurements of the JRC
ISO	International Organization for Standardization
JRC	Joint Research Centre of the European Commission
k	Coverage factor
k_0 -NAA	k_0 -Neutron activation analysis
LA	Laser ablation
LOD	Limit of Detection
MS	Mass spectrometry
MS_{between}	Mean of squares between-unit from an ANOVA
MSDS	Material safety data sheet
MS_{within}	Mean of squares within-unit from an ANOVA
n	Number of replicates per unit
N	Number of samples (units) analysed
n.a.	Not applicable
n.c.	Not calculated
n.d.	Not detectable
NIST	National Institute of Standards and Technology (USA)
OES	Optical emission spectrometry
QC	Quality control
rel	Index denoting relative figures (uncertainties etc.)
RM	Reference material
RSD	Relative standard deviation
s	Standard deviation
s_{bb}	Between-unit standard deviation; an additional index "rel" is added when appropriate
s_{between}	Standard deviation between groups as obtained from ANOVA; an additional index "rel" is added as appropriate

SI	International System of Units
s_{meas}	Standard deviation of measurement data; an additional index "rel" is added as appropriate
s_{ns}	Standard deviation of results of normal stock samples
s_{within}	Standard deviation within groups as obtained from ANOVA; an additional index "rel" is added as appropriate
s_{wb}	Within-unit standard deviation
$t_{\alpha, df}$	Critical t -value for a t -test, with a level of confidence of $1-\alpha$ and df degrees of freedom
u	Standard uncertainty
U	Expanded uncertainty
u_{bb}^*	Standard uncertainty related to a maximum between-unit inhomogeneity that could be hidden by method repeatability; an additional index "rel" is added as appropriate
u_{bb}	Standard uncertainty related to a possible between-unit inhomogeneity; an additional index "rel" is added as appropriate
u_{char}	Standard uncertainty of the material characterisation; an additional index "rel" is added as appropriate
u_{CRM}	Combined standard uncertainty of the certified value; an additional index "rel" is added as appropriate
U_{CRM}	Expanded uncertainty of the certified value; an additional index "rel" is added as appropriate
u_{Δ}	Combined standard uncertainty of measurement result and certified value
u_{meas}	Standard measurement uncertainty
U_{meas}	Expanded measurement uncertainty
u_{rec}	Standard uncertainty related to possible between-unit inhomogeneity modelled as rectangular distribution; an additional index "rel" is added as appropriate
u_{sts}	Standard uncertainty of the short-term stability; an additional index "rel" is added as appropriate
u_t	Standard uncertainty of trueness
VIM	Vacuum induction melting
VIDP	Vacuum induction degassing pouring
\bar{x}	Arithmetic mean
\bar{x}_{ns}	Arithmetic mean of all results of normal stock samples
\bar{x}_{ref}	Arithmetic mean of results of reference samples
α	Significance level
Δ_{meas}	Absolute difference between mean measured value and the certified value
$\nu_{s,meas}$	Degrees of freedom for the determination of the standard deviation s_{meas}
$\nu_{MS_{within}}$	Degrees of freedom of MS_{within}

1 Introduction

1.1 Background

Copper is essential for humans in their daily life and in industry. Copper was one of the first metals ever extracted and used by humans (e.g. coins, ornaments) and one of the first alloying metals with zinc (brass), aluminium and tin (bronze) [5]. Copper is important to the development of human civilisation (e.g. Bronze Age).

Presently, copper exists in various sectors and industries: building construction, power generation and transmission, electronic product manufacturing, production of industrial machinery and the transportation sector [6]. Its relevance is due to its physical (ductility) and chemical properties (excellent thermal and electrical conductivity, corrosion resistance) [7] and its high antimicrobial activity [8].

Copper is traded internationally; production and transformation of copper (from cathode to semi-finished product and final production) are widespread geographically. The demand of refined copper was estimated to be 0.5 million tons in 1900 and was 20 million tons in 2012 [5]. The copper market is an important globalised market, which drives the need for international standardisation.

Depending on the commodity exchange, the chemical composition of electrolytic copper cathode is defined according to three standards: EN 1978:1998 (Cu-CATH-1) [9], GB/T 467-2010 (Cu-CATH-1) [10] and ASTM B115-10 (cathode Grade 1) [11]. These require the determination of 12 to 18 elements which should, alone or in groups of a few elements, not exceed upper limits of a few mg/kg: Ag, As, Bi, Cd, Co, Cr, Fe, Mn, Ni, P, Pb, S, Sb, Se, Si, Sn, Te and Zn [9-11].

The price of copper is established as a function of the impurity levels, the premium paid for very low levels and penalties for high levels.

As the analyses of impurities at these very low levels are subject to many possibilities of error, the industry has a strong need for reliable certified reference materials to ensure the quality and the accuracy of their measurements.

To support the industry, two CRMs for trace elements in copper were produced in 1992 within the scope of the European Commission's BCR-programme. The two CRMs (BCR-074 and BCR-075 [12]) are close to exhaustion and need replacement.

In addition to the elements required by the various standards, eleven elements (Al, Au, Be, H, Hg, In, Mg, O, Ti, W and Zr) were considered to be of interest for the copper industry and quality control laboratories involved in copper trade. These elements were found to be relevant to the certification or as indicative values and were included in the scope of the project.

1.2 Choice of the material

ERM-EB075 is produced from pure copper with added impurities. The addition of impurities permits achieving pre-defined levels of the trace elements mass fraction more easily. These levels of trace elements were chosen to be close to the limits set in the international standards. The levels of trace elements are similar to those of the exhausted CRM for trace elements in electrolytic copper with added impurities: BCR-075 [12].

The material was produced to comply with the various analytical methods used by industry, and in three different formats;

ERM-EB075A: discs of 39 mm diameter were designed for solid sampling techniques (e.g. spark-optical emission spectrometry (spark-OES) or glow discharge – mass spectrometry (GD-MS)),

ERM-EB075B: cylinders of 8 mm diameter were designed for solid sampling techniques (e.g. GD-MS) and analysis after acid dissolution,

ERM-EB075C: chips of approximately 250 mg were designed for solid sampling techniques (e.g. direct current arc optical emission spectrometry (DC-arc-OES)) and analysis after acid dissolution.

1.3 Design of the project

After processing, homogeneity of the materials was evaluated in a dedicated study of each material format using different techniques. The homogeneity results of the three formats were finally pooled to obtain an overall uncertainty. The stability was assessed using a similar existing material. The certification was performed by means of an interlaboratory comparison. The results showed no difference between the three formats, so one single value for each element for all three formats could be assigned.

2 Participants

2.1 Project management and data evaluation

European Commission, Joint Research Centre, Institute for Reference Materials and Measurements (IRMM), Geel, BE
(accredited to ISO Guide 34 for production of certified reference materials, BELAC No. 268-RM)

2.2 Processing

Luvata Pori Oy, Pori, FI

European Commission, Joint Research Centre, Institute for Reference Materials and Measurements (IRMM), Geel, BE
(accredited to ISO Guide 34 for production of certified reference materials, BELAC No. 268-RM)

Wieland-Werke, Ulm, DE

2.3 Homogeneity study

Evans Analytical Group SAS, Tournefeuille, FR

Umicore Analytical competence center, Olen, BE

Umicore Analytical Competence Center, Hanau-Wolfgang, DE

Alfred H Knight International Ltd, St Helens, UK

(Measurements performed under the scope of ISO/IEC 17025 accreditation, UKAS No. 1543)

2.4 Characterisation

Activation Laboratories Ltd., Ancaster, CA

(Measurements performed under the scope of ISO/IEC 17025 accreditation, SCC No. 266)

Aurubis AG, Hamburg, DE

Bundesanstalt für Materialforschung und –prüfung (BAM), Berlin, DE

(Measurements performed under the scope of ISO/IEC 17025 accreditation, DAkkS No. DP-L-11075-14-00)

CCR affinerie, Montreal, CA

Evans Analytical Group LLC, Liverpool, NY, US

Evans Analytical Group SAS, Tournefeuille, FR

Institut "Jozef Stefan" (IJS), Department of Environmental Sciences, Ljubljana, SI
(measurements performed under the scope of ISO/IEC 17025 accreditation, Slovenska Akreditacija-LP090)

Laboratory Testing Inc., Hatfield, US

(measurements performed under the scope of ISO/IEC 17025 accreditation, A2LA No. 0117.05)

Laboratoire national de métrologie et d'essais, Trappes, FR

LCABIE-IPREM - UMR 5254, Pau, FR

National Research Council Canada, Ottawa, CA

(Measurements performed under the scope of ISO/IEC 17025 accreditation, SCC No. 474)

Umicore Analytical Competence Center, Olen, BE

Umicore Analytical Competence Center, Hanau-Wolfgang, DE

Studie centrum voor Kernenergie, SCK, Mol, BE

(Measurements performed under the scope of ISO/IEC 17025 accreditation; BELAC No. 015-TEST)

TU Delft, Delft, NL

(Measurements performed under the scope of ISO/IEC 17025 accreditation; Rva L049)

Ultra Traces Analyses Aquitaine (UT2A), Pau, FR

3 Material processing and process control

3.1 Origin/Purity of the starting material

The starting materials were pure copper (purity >99.999%) from Luvata Pory Oy (Pori,FI), pure metals (Ag, Al, Au, In, Ni, Pb, Sb, Sn and Zn; purity > 99.7%) and copper master alloys (CuAs30, CuBe4, CuBi2, CuCd50, CuCo15, CuCr10, CuFe20, CuMg20, CuMn50, CuP10, CuS20, CuSe37, CuSi10, CuTe50, CuTi30, CuZr50; purity > 99.5%) from Wieland Werke (Ulm, DE). The specification of the startings material complied with international standards (EN 1981: 2003 [13])and was documented in certificates of analysis from producers.

3.2 Processing

The production of ERM-EB075A, B, C was done in 2 steps:

- Melting and casting of copper rods with added impurities,
- Extrusion of copper billets and mechanical processing into final dimensions.

Melting and casting of copper with added impurities and extrusion into rods was realised by Wieland-Werke (Ulm, DE). Machining into the final form was done by Luvata Pori Oy (Pori, FI). The production scheme is detailed in Figure 1.

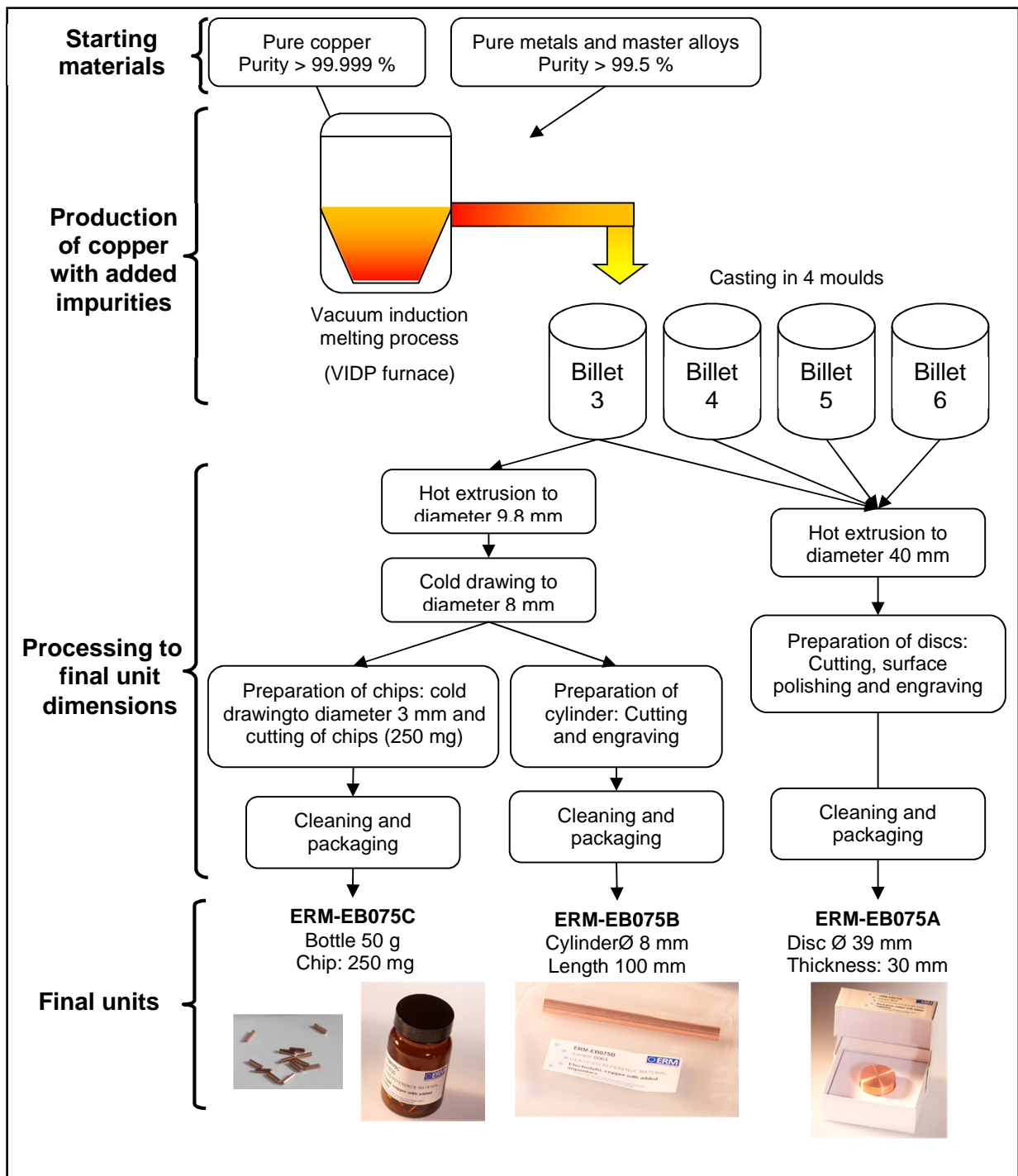


Figure 1: Processing scheme of ERM-EB075A, B and C

3.2.1 Melting and casting of copper billets with added impurities

The first step of the production was obtaining a homogeneous melt of pure copper with added impurities by Wieland-Werke (Ulm, DE). The technique used was vacuum induction melting (VIM), a technique for melting metal via electromagnetic induction under vacuum. An induction furnace containing an electrographite crucible surrounded by an induction coil is located inside a vacuum chamber. The induction furnace is connected to an alternating current power source at a frequency that is precisely correlated to the furnace size and the material being melted.

The advantages of VIM are:

- Homogeneous distribution of chemical elements in the material due to constant stirring of the molten metal during melting and casting;
- Limitation of non-metallic oxide inclusions. Melting under vacuum helps to limit metal reactivity with atmospheric oxygen;
- Precise adjustment of alloy composition, since the temperature, vacuum, gas atmosphere, pressure and material transport (e.g. through stirring of the bath) can be adjusted independently of one another.

The VIM technique produces a highly homogenous melt and allows casting of materials with controlled composition. The crucible used for melting was a dedicated electrographite crucible, which mitigates the risk of metallic contamination.

Approximately 2.2 tonnes of copper were charged into the electrographite crucible in the vacuum induction degassing pouring furnace (VIDP furnace) under vacuum ($10^{-1} - 10^{-2}$ mbar). The molten metal composition was adjusted using the different pure metals and master alloys until the precise melt chemistry was achieved. The manufacturer performed a single analysis to verify the composition before casting. After verification, four copper billets were cast in four different moulds.

3.2.2 Extrusion of copper billets and mechanical processing into final dimensions

The four copper billets were dispatched for the production of ERM-EB075A, B and C by hot extrusion and mechanical machining.

ERM-EB075A: production of discs

Three and a half billets were dedicated to the production of ERM-EB075A (discs with a diameter of 39 mm). Each billet was first cut into two parts, and hot extruded to a final diameter of 40 mm by Wieland-Werke (Ulm, DE). The seven last rods had a length of nine metres; they were labelled and cut into three parts for further processing.

From the 21 last rods (diameter: 40 mm; length: 3 m), 12 rods were selected for final processing by Luvata Pori Oy (Pori, FI). The rods were cut to 30 mm length and polished (on all surfaces), the final diameter after polishing was 39 mm. The discs were engraved mechanically on the curved surface with "75-XXX". The code "75-XXX" corresponds to the material ID (75 stands for ERM-EB075) and the unit number (XXX). Then, the discs were degreased, cleaned with deionised water, dropped into 2-propanol / isopropanol and dried. Finally, the discs were visually checked at IRMM and packed in individual carton boxes.

ERM-EB075B and C: production of cylinders and chips

A half billet was dedicated to the production of ERM-EB075B and C. The half billet was hot extruded to a diameter of 9.8 mm and then processed to 8 mm diameter using cold drawing by Wieland-Werke (Ulm, DE). In total, 68 rods were obtained with a diameter of 8 mm and a length of 3 m.

Forty-four rods were selected after analysis of Ag, Al, As, Au, Be, Bi, Cd, Co, Cr, Fe, Hg, In, Mg, Mn, Ni, P, Pb, S, Sb, Se, Si, Sn, Te, Ti, W, Zn and Zr by GD-MS (one GD-MS analysis per rod). The purpose was to exclude outlying rods from further processing (Section 3.3.1). Eighteen rods were randomly selected for the production of ERM-EB075B and 26 rods for the production of ERM-EB075C.

For ERM-EB075B, the 18 selected rods were cut into cylinders of 100 mm length and engraved mechanically with the code "75-XXX" by Luvata Pori Oy (Pori, FI). Afterwards; the cylinders were degreased, cleaned with deionised water, dropped into 2-propanol / isopropanol and dried. The final batch of cylinders was visually checked at IRMM and packed into plastic sachet sealed under vacuum.

For ERM-EB075C, the 26 selected rods were processed to 3 mm diameter using cold drawing by Luvata Pori Oy (Pori, FI). The material obtained was cut into pieces of 250 mg (with a relative tolerance of 3%). The pieces were grouped per rods (~ 5200 pieces per rod) and followed a cleaning process (degreasing, cleaning with deionised water, rinsing with 2-propanol / isopropanol and drying). Finally, 50 g of chips were placed into cleaned amber glass bottles, flushed with inert gas (Ar) and closed.

The final products were:

ERM-EB075A: 900 units. Each unit is a disc of 39 mm diameter with thickness of 30 mm;

ERM-EB075B: 500 units. Each unit is a cylinder of 8 mm diameter with length of 100 mm;

ERM-EB075C: 675 units. Each unit is a bottle containing 50 g of chips. Each chip has a mass of approximately 250 mg.

3.3 Process control

Several controls of the composition and homogeneity of trace elements in copper in the semi-finished products were realised by the manufacturer during the melting (one composition analysis by GD-MS) and after casting of the four billets (one GD-MS analysis on the top and bottom of each billet). No major element inhomogeneity was observed by the measurements.

Before processing the 8 mm rods into their final formats ERM-EB075B and C, one GD-MS analysis was performed on every 8 mm rod for Ag, Al, As, Au, Be, Bi, Cd, Co, Cr, Fe, Hg, In, Mg, Mn, Ni, P, Pb, S, Sb, Se, Si, Sn, Te, Ti, W, Zn and Zr by GD-MS (data not shown). The objective was to select the required number of 8 mm rods for the production of ERM-EB075B and C and to exclude outlying values and extreme values (highest and lowest values for each element).

Visual control was done for each unit during the packaging. Few units were excluded due to major scratches or unclear engraving.

The segregation between billets is a source of inhomogeneity in metal production; the different processing steps could be another source of difference between the formats A, B and C. It was studied during the process control (data not shown) and it is detailed in Section 4.1.2.

4 Homogeneity

A key requirement for any reference material is the equivalence between the various units. In this respect, it is relevant whether the variation between units is significant compared to the uncertainty of the certified value. It is not relevant if the variation between units is significant compared to the analytical variation of the study performed. Consequently, ISO Guide 34 requires RM producers to quantify the between-unit variation. This aspect is covered in between-unit homogeneity studies.

Production of metallic CRMs requires extensive homogeneity tests since several inhomogeneity sources have to be taken into account.

- Trend or segregation within billet: during the casting, some elements are known to segregate within the billet (e.g. lead in copper). The trend or segregation within billet was tested using ERM-EB075A units from one billet (Section 4.1.2). The outcome of the study is considered to be similar for all billets as they were melted and casted under the same conditions.

- Between-billet homogeneity (Section 4.1.3): the final material is composed of four casted billets that may show differences due to the melting or casting process (i.e. inhomogeneity of the melt, delay in casting) for some elements (see Section 3.3.2). Between-billet homogeneity was studied using a large number of ERM-EB075A units from the four billets.

- Between-unit homogeneity (Section 4.1.4).

- Within-unit inhomogeneity is essential for solid sampling techniques (e.g. GD-MS, spark-OES). These techniques use tiny sample intake and are subject to differences due to the aliquot location (radial homogeneity on discs; Section 4.2.1) and to the sample intake (minimum sample intake).

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

4.1 Between-unit homogeneity

The between-unit homogeneity was evaluated to ensure that the certified values are valid for all units of the material, within the stated uncertainty.

4.1.1 Study setup

The between-unit homogeneity study was performed for each format independently using an appropriate analytical method (Table 2). This approach allows coherence among the intended use, homogeneity estimation and the minimum sample intake.

Within-billet homogeneity and between-billet homogeneity were evaluated using ERM-EB075A units (Table 2). The evaluation of these three studies was necessary to select the most appropriate uncertainty estimation (s_{bb} , u_{bb}^* or u_{rec})

35 units of ERM-EB075A and B and the 29 bottles of ERM-EB075C were selected using a random stratified sampling scheme covering the whole batch for the between-unit homogeneity test. For this, the batch was divided into 29 or 35 groups (with a similar number of units) and one unit was selected randomly from each group. The number of selected units (35 units for ERM-EB075A, 35 units for ERM-EB075B and 29 bottles for ERM-EB075C) corresponds to more than 3.8% of the production of each format (superior to the cubic root of the total number of the produced units). It is considered sufficient to represent a lot/batch consisting of large number of units for which it is impractical to test 8% of the units as recommended in ASTM E826 [14].

Four independent aliquots for Ag, Al, As, Au, Be, Bi, Cd, Co, Cr, Fe, Hg, In, Mg, Mn, Ni, P, Pb, S, Sb, Se, Si, Sn, Te, Ti, W, Zn, Zr and three independent aliquots for O were taken from each selected unit, and analysed by glow discharge mass spectroscopy and by inert gas fusion with IR detection for ERM-EB075A and B. For ERM-EB075C, four independent aliquots for Ag, Al, As, Au, Be, Bi, Cd, Co, Cr, Fe, Hg, In, Mg, Mn, Ni, P, Pb, Sb, Se, Si, Sn, Te, Ti, W, Zn, Zr and three independent aliquots for O and S were taken from each selected unit, and analysed by ICP-MS, ICP-OES (for Al and Si), by inert gas fusion with IR detection (O) and by combustion with IR detection (S). A summary of the study is given in Table 2.

The measurements were performed in a randomised block design because the number of replicates on all units (87 - 140 analyses) could not be included in a single run due to instrumental constraints (drift towards the end of a long run, time of analysis). Improved precision (measured as the within-unit standard deviation) was obtained using several short runs in a randomised block design compared to the one achieved in a single run with 87 to 140 analyses.

The design applied consisted of three to four measurement sequences, each consisting of a single measurement on each of the 29 to 35 units of ERM-EB075. The order of units in each run was randomised individually for each sequence in order to separate a potential analytical drift from a trend in the processing sequence.

Table 2: Summary of the homogeneity study.

CRM format	Within-billet homogeneity study Number of units selected	Between-unit homogeneity study Number of units selected	Elements	Technique	Replicates / Analytical runs
ERM-EB075A	1 billet 9 discs	35 discs (7-10 units per billet)	Ag, Al, As, Au, Be, Bi, Cd, Co, Cr, Fe, Hg, In, Mg, Mn, Ni, P, Pb, S, Sb, Se, Si, Sn, Te, Ti, W, Zn, Zr	GD-MS	4 / 4
			O	IGF-IR	3 / 3
ERM-EB075B	n.a.	35 cylinders	Ag, Al, As, Au, Be, Bi, Cd, Co, Cr, Fe, Hg, In, Mg, Mn, Ni, P, Pb, S, Sb, Se, Si, Sn, Te, Ti, W, Zn, Zr	GD-MS	4 / 4
			O	IGF-IR	3 / 3
ERM-EB075C	n.a.	29 bottles	Ag, As, Au, Be, Bi, Cd, Co, Cr, Fe, Hg, In, Mg, Mn, Ni, P, Pb, Sb, Se, Sn, Te, Ti, W, Zn, Zr	ICP-MS	4 / 4
			Al and Si	ICP-OES	4 / 4
			O	IGF-IR	3 / 3
			S	Combustion-IR	3 / 3

A two-way analysis of variance without replication was used to estimate the within- and between-unit standard deviations independently of a potential analytical sequence effect.

For two elements (Hg and W), the results in all units were reported below the detection limit of the method (0.01 mg/kg). The data treatment was not applied to these two elements.

For Ag, Al, As, Au, Be, Bi, Cd, Co, Cr, Fe, In, Mg, Mn, Ni, O, P, Pb, S, Sb, Se, Si, Sn, Te, Ti, Zn, Zr, the data evaluation was performed in the following order:

1 – Trends in analytical run / correction for significant trends

Regression analyses were done to evaluate potential trends in each analytical run. Some significant (95 % confidence level) trends in the analytical sequence were visible; pointing at a signal drift in the analytical system (Tables 3-5). The correction of biases, even if they are statistically not significant, allows a combination of the smallest uncertainty with the highest probability to cover the true value [11]. Correction of trends is therefore expected to improve

the sensitivity of the subsequent statistical analysis through a reduction in analytical variation without masking potential between-unit heterogeneities. As the analytical sequence and the unit numbers were not correlated, trends significant at 95 % confidence level were corrected as explained in equation 1.

2 – Evaluation of between analytical run effect / Normalisation of dataset (if necessary)

The analytical trend-corrected dataset was evaluated for significant differences between analytical runs (95 % confidence level) using one way ANOVA. Significant differences between analytical runs were observed on 95 % confidence level for all elements in ERM-EB075A and ERM-EB075B and for Ag, Al, As, Au, Co, Cr, Fe, In, Mn, Ni, P, Pb, S, Se, Si, Sn, Te, Ti, Zn, Zr in ERM-EB075C (Tables 3-5). As it is assumed that run effects and unit effects are independent, differences between analytical runs on 95 % confidence level were normalised as explained in equation 2.

$$x_R(r,i) = \frac{x_T(r,i)}{\bar{x}_T(r)} \times \bar{x}_T \quad \text{Equation 2}$$

i	position of the result in the analytical run
r	number of the analytical run from 1 to 4
\bar{x}_T	mean results of all the analytical runs
$\bar{x}_T(r)$	mean results of the analytical run r after correction for the trend in analytical sequence (if necessary)
$x_T(r,i)$	corrected results for analytical trend on the position i in the analytical run r
$x_R(r,i)$	normalised results on the position i in the analytical run r

3 – Statistical evaluation of the datasets

The normalised datasets were tested for consistency using Grubbs outlier tests on a confidence level of 99 % on the individual results and the unit means (Tables 3-5). The unit means for ERM-EB075A, B and C are presented graphically in Annex B.

Some outlying individual results were detected. No outlying means were detected at the 99 % confidence level except for Zr mass fraction in ERM-EB075B and C. Since no technical reason for the outliers could be found, all the data were retained for statistical analysis.

Table 3: Results of the statistical evaluation of the homogeneity study of ERM-EB075A; n = number of outliers; T = number of series with analytical trend.

ERM-EB075A	Analytical trends	Between analytical run difference Tested with one way ANOVA	Outliers at 99% confidence level		Distribution	
			Unit means (n)	Individual results (n)	Unit means	Individual results
Analyte	Significant at 95% confidence level (T)	Significant difference at 95% confidence level (F-test)				
Ag	Yes (4)	Yes	No	No	unimodal	unimodal
Al	Yes (1)	Yes	No	No	multimodal	multimodal
As	Yes (2)	Yes	No	No	unimodal	unimodal
Au	Yes (4)	Yes	No	Yes (1)	unimodal	unimodal
Be	Yes (1)	Yes	No	No	multimodal	multimodal
Bi	Yes (4)	Yes	No	Yes (2)	unimodal	unimodal
Cd	Yes (3)	Yes	No	No	unimodal	unimodal
Co	Yes (1)	Yes	No	Yes (1)	unimodal	unimodal
Cr	Yes (4)	Yes	No	Yes (1)	unimodal	unimodal
Fe	Yes (2)	Yes	No	Yes (1)	unimodal	unimodal
In	Yes (4)	Yes	No	Yes (1)	unimodal	unimodal
Mg	Yes (2)	Yes	No	No	multimodal	multimodal
Mn	Yes (1)	Yes	No	No	multimodal	multimodal
Ni	Yes (1)	Yes	No	No	multimodal	multimodal
O	Yes (1)	Yes	No	No	unimodal	unimodal
P	Yes (1)	Yes	No	Yes (1)	unimodal	unimodal
Pb	Yes (2)	Yes	No	No	multimodal	multimodal
S	Yes (3)	Yes	No	No	unimodal	unimodal
Sb	Yes (4)	Yes	No	Yes (1)	unimodal	unimodal
Se	Yes (4)	Yes	No	Yes (2)	unimodal	unimodal
Si	Yes (1)	Yes	No	No	unimodal	unimodal
Sn	Yes (4)	Yes	No	Yes (2)	unimodal	unimodal
Te	Yes (4)	Yes	No	No	unimodal	unimodal
Ti	Yes (4)	Yes	No	No	multimodal	multimodal
Zn	Yes (1)	Yes	No	No	unimodal	unimodal
Zr	Yes (4)	Yes	No	No	multimodal	multimodal

Table 4: Results of the statistical evaluation of the homogeneity study of ERM-EB075B; n = number of outliers; T = number of series with analytical trend.

ERM-EB075B	Analytical trends	Between analytical run difference Tested with one way ANOVA	Outliers at 99% confidence level		Distribution	
			Unit means (n)	Individual results (n)	Unit means	Individual results
Analyte	Significant at 95% confidence level (T)	Significant difference at 95% confidence level (F-test)				
Ag	Yes (2)	Yes	No	No	unimodal	unimodal
Al	Yes (1)	Yes	No	Yes (1)	unimodal	unimodal
As	Yes (2)	Yes	No	Yes (1)	unimodal	unimodal
Au	Yes (2)	Yes	No	Yes (2)	unimodal	unimodal
Be	Yes (1)	Yes	No	No	unimodal	unimodal
Bi	Yes (2)	Yes	No	No	unimodal	unimodal
Cd	Yes (2)	Yes	No	No	unimodal	unimodal
Co	Yes (3)	Yes	No	No	unimodal	unimodal
Cr	Yes (2)	Yes	No	Yes (1)	unimodal	unimodal
Fe	None	Yes	No	Yes (1)	unimodal	unimodal
In	Yes (2)	Yes	No	No	unimodal	unimodal
Mg	Yes (2)	Yes	No	No	unimodal	unimodal
Mn	Yes (1)	Yes	No	No	unimodal	unimodal
Ni	Yes (2)	Yes	No	No	unimodal	unimodal
O	Yes (1)	Yes	No	No	unimodal	unimodal
P	Yes (2)	Yes	No	No	unimodal	unimodal
Pb	Yes (2)	Yes	No	No	unimodal	unimodal
S	Yes (2)	Yes	No	No	unimodal	unimodal
Sb	Yes (2)	Yes	No	No	unimodal	unimodal
Se	Yes (2)	Yes	No	No	unimodal	unimodal
Si	Yes (3)	Yes	No	Yes (2)	unimodal	unimodal
Sn	Yes (2)	Yes	No	No	unimodal	unimodal
Te	Yes (2)	Yes	No	No	unimodal	unimodal
Ti	Yes (1)	Yes	No	No	unimodal	unimodal
Zn	Yes (1)	Yes	No	No	unimodal	unimodal
Zr	Yes (1)	Yes	Yes (1)	No	unimodal	unimodal

Table 5: Results of the statistical evaluation of the homogeneity study of ERM-EB075C; n = number of outliers; T = number of series with analytical trend.

ERM-EB075C	Analytical trends	Between analytical run difference Tested with one way ANOVA	Outliers at 99% confidence level		Distribution	
			Unit means (n)	Individual results (n)	Unit means	Individual results
Analyte	Significant at 95% confidence level (T)	Significant difference at 95% confidence level (F-test)				
Ag	None	Yes	No	No	unimodal	unimodal
Al	Yes (1)	Yes	No	Yes (1)	unimodal	unimodal
As	None	Yes	No	Yes (1)	unimodal	unimodal
Au	None	Yes	No	No	unimodal	unimodal
Be	None	No	No	No	unimodal	unimodal
Bi	None	No	No	No	unimodal	unimodal
Cd	None	No	No	Yes (2)	unimodal	unimodal
Co	Yes (1)	Yes	No	Yes (1)	unimodal	unimodal
Cr	Yes (1)	Yes	No	Yes (2)	unimodal	unimodal
Fe	Yes (1)	Yes	No	Yes (1)	unimodal	unimodal
In	None	Yes	No	No	unimodal	unimodal
Mg	None	No	No	Yes (1)	unimodal	unimodal
Mn	Yes (1)	Yes	No	Yes (1)	unimodal	unimodal
Ni	Yes (1)	Yes	No	No	unimodal	unimodal
O	None	No	No	Yes (1)	unimodal	unimodal
P	None	Yes	No	No	unimodal	unimodal
Pb	None	Yes	No	No	unimodal	unimodal
S	None	Yes	No	No	unimodal	unimodal
Sb	None	No	No	No	unimodal	unimodal
Se	None	Yes	No	No	unimodal	unimodal
Si	None	Yes	No	No	unimodal	unimodal
Sn	None	Yes	No	No	unimodal	unimodal
Te	None	Yes	No	Yes (1)	unimodal	unimodal
Ti	None	Yes	No	Yes (1)	unimodal	unimodal
Zn	None	Yes	No	No	unimodal	unimodal
Zr	None	Yes	Yes (1)	No	unimodal	unimodal

4.1.2 Evaluation of the trend or segregation within-billet

The normalised datasets of ERM-EB075A study were used to detect significant trends within-billet. Nine units of ERM-EB075A were selected using a random stratified sampling scheme covering a half billet for the within-billet homogeneity test. No outlying means were detected in the datasets used for within billet homogeneity study.

Regression analyses were performed using these nine units to evaluate potential trends within the billet. No trends in the billet were visible on a 95 % confidence level for Ag, Al, As, Au, Be, Bi, Cd, Co, Cr, Fe, Hg, In, Mg, Mn, Ni, O, P, S, Sb, Se, Si, Sn, Te, Ti, W, Zn, Zr. A significant trend within billet was detected for Pb on 95 % confidence level.

When a trend in the billet (segregation) was significant at least at the 95 % confidence level, the uncertainty was assessed in a different way. Here, u_{rec} was estimated using a rectangular distribution between the highest and lowest unit mean. The corrected uncertainty in those cases where there was a significant trend in the filling sequence is given in Equation 3.

$$u_{rec,rel} = \frac{|highest\ result - lowest\ result|}{2 \cdot \sqrt{3} \cdot \bar{y}} \quad \text{Equation 3}$$

\bar{y} mean of all results of the homogeneity study

This applies for Pb for ERM-EB075A, B and C.

4.1.3 Between-billet inhomogeneity quantification for ERM-EB075A

ERM-EB075A was the only format produced using different billets (four billets). As observed during the process control, significant differences for several elements were found between billets. The significance of the between-billet differences was evaluated using the results of the ERM-EB075A homogeneity study.

In the ERM-EB075A homogeneity study, seven to ten units were selected from each of the four billets. Quantification of between billet inhomogeneity was accomplished using the analytical trend corrected dataset by the mean of a two-way ANOVA. A two-way ANOVA can separate the between-run variation (s_R), the between-billet variation ($s_{bb,billet}$) and the within-billet variation ($s_{wb,billet}$). A F-test was used to determine if the variance due to the billets is significant on a 95 % confidence level. For Al, Be, Mg, Mn, Ni, Pb, Ti and Zr, the difference between billets was significant. The data does not follow a uni-modal distribution for analytes Al, Be, Mg, Mn, Ni, Pb, Ti and Zr in ERM-EB075A. Therefore, u_{rec} was estimated using a rectangular distribution between the highest and lowest unit mean [12]. The uncertainty in those cases is given in Equation 3.

$$u_{rec,rel} = \frac{|highest\ result - lowest\ result|}{2 \cdot \sqrt{3} \cdot \bar{y}} \quad \text{Equation 3}$$

\bar{y} mean of all results of the homogeneity study

This applies for Al, Be, Mg, Mn, Ni, Pb, Ti and Zr in ERM-EB075A and the results of these evaluations are listed in Table 6.

4.1.4 Between-unit inhomogeneity quantification

Quantification of between-unit inhomogeneity was accomplished using the analytical trend corrected dataset by two-way ANOVA, which can separate the between-run variation (s_R), the between-unit variation (s_{bb}) and the within-unit variation (s_{wb}). The latter is equivalent to the method repeatability if the individual samples are representative of the whole unit.

Evaluation by ANOVA requires unit means that follow at least a unimodal distribution and unit results that follow unimodal distributions with approximately the same standard deviations. Distribution of the unit means was visually tested using histograms and normal probability plots. Minor deviations from unimodality of the individual values do not significantly affect the estimate of between-unit standard deviations.

Recall that $s_{bb,rel}$ and $s_{wb,rel}$ are estimates of the true standard deviations and, therefore, subject to random fluctuations. Therefore, the mean of squares between groups ($MS_{between}$) can be smaller than the mean of squares within groups (MS_{within}), resulting in negative arguments under the square root used for the estimation of the between-unit variation. Since the true variation cannot be lower than zero, u_{bb}^* , the maximum inhomogeneity that could be hidden by method repeatability, was calculated as described by Linsinger *et al.* [15]. u_{bb}^* is comparable to the limit of detection of an analytical method, yielding the maximum inhomogeneity that might be undetected by the given study setup.

Analysis of variance applied to a randomised block design with one observation per unit per run leads to a between-run mean square MS_R together with a between-unit mean square $MS_{between}$, and a residual mean square MS_{within} . The residual mean square MS_{within} is an unbiased estimate of the repeatability variance s_r^2 . The between-unit standard deviation s_{bb} is calculated as described in equation 5. Method repeatability ($s_{wb,rel}$), between-unit standard deviation ($s_{bb,rel}$) and $u_{bb,rel}^*$ were calculated as:

$$s_{wb,rel} = \frac{\sqrt{MS_{within}}}{\bar{y}} \quad \text{Equation 4}$$

$$s_{bb,rel} = \frac{\sqrt{\frac{MS_{between} - MS_{within}}{n}}}{\bar{y}} \quad \text{Equation 5}$$

$$u_{bb,rel}^* = \frac{\sqrt{\frac{MS_{within}}{n}} \sqrt{\frac{2}{v_{MSwithin}}}}{\bar{y}} \quad \text{Equation 6}$$

MS_{within} mean square within a unit from an ANOVA

$MS_{between}$ mean squares between-unit from an ANOVA

\bar{y} mean of all results of the homogeneity study

n number of analytical runs

$v_{MSwithin}$ degrees of freedom of MS_{within}

For ERM-EB075A, the homogeneity study showed unimodal distribution and no outlying unit means or trends within-billet for Ag, As, Au, Bi, Cd, Co, Cr, Fe, In, O, P, S, Sb, Se, Si, Sn, Te and Zn. Therefore, the between-unit standard deviation can be used as estimate of u_{bb} . As u_{bb}^* sets the limits of the study to detect inhomogeneity, the larger value of s_{bb} and u_{bb}^* is adopted as the uncertainty contribution to account for potential inhomogeneity.

For ERM-EB075B and C, the homogeneity study showed unimodal distribution and no outlying unit means or trends within-billet for Ag, Al, As, Au, Be, Bi, Cd, Co, Cr, Fe, Hg, In, Mg, Mn, Ni, P, S, Sb, Se, Si, Sn, Te, Ti, W, Zn. Therefore, the between-unit standard deviation can be used as estimate of u_{bb} . As u_{bb}^* sets the limits of the study to detect inhomogeneity, the larger value of s_{bb} and u_{bb}^* is adopted as the uncertainty contribution to account for potential inhomogeneity.

However, a different approach was adopted for Zr in ERM-EB075B and C because one and two outlying unit mean was detected in ERM-EB075B and C respectively. In this case between-unit inhomogeneity was modelled as a rectangular distribution limited by the largest outlying unit mean, and the rectangular standard uncertainty of homogeneity was estimated by:

$$u_{rec} = \frac{|outlier - \bar{y}|}{\sqrt{3} \cdot \bar{y}} \quad \text{Equation 7}$$

\bar{y} mean of all results of the homogeneity study

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

The results of the evaluation of the between-unit variation in ERM-EB075A, B and C are summarised in Tables 6-8. The resulting values from the above equations were converted into relative uncertainties.

Table 6: Results of the homogeneity study of ERM-EB075A

ERM-EB075A	$S_{wb,rel}$ [%]	$S_{bb,rel}$ [%]	$U_{bb,rel}$ [%]	$U_{rec,rel}$ [%]	$U_{bb,rel}$ [%]
Ag	2.36	0.64	0.44	n.a. ²⁾	0.64
Al	5.04	2.05	0.94	4.70	4.70
As	2.94	n.c. ¹⁾	0.55	n.a. ²⁾	0.55
Au	3.35	n.c. ¹⁾	0.63	n.a. ²⁾	0.63
Be	10.80	6.48	2.02	10.23	10.23
Bi	6.21	n.c. ¹⁾	1.16	n.a. ²⁾	1.16
Cd	3.40	1.10	0.64	n.a. ²⁾	1.10
Co	3.11	0.54	0.58	n.a. ²⁾	0.58
Cr	2.58	n.c. ¹⁾	0.48	n.a. ²⁾	0.48
Fe	2.00	0.18	0.37	n.a. ²⁾	0.37
In	6.52	1.05	1.22	n.a. ²⁾	1.22
Mg	5.26	2.18	0.98	3.91	3.91
Mn	1.53	0.33	0.29	1.17	1.17
Ni	3.81	1.68	0.71	3.01	3.01
O	62.84	n.c. ¹⁾	15.02	n.a. ²⁾	15.02
P	6.66	n.c. ¹⁾	1.25	n.a. ²⁾	1.25
Pb	6.35	8.07	1.19	9.09	9.09
S	4.54	n.c. ¹⁾	0.85	n.a. ²⁾	0.85
Sb	4.58	n.c. ¹⁾	0.86	n.a. ²⁾	0.86
Se	3.37	0.94	0.63	n.a. ²⁾	0.94
Si	4.46	n.c. ¹⁾	0.84	n.a. ²⁾	0.84
Sn	4.22	0.91	0.79	n.a. ²⁾	0.91
Te	4.64	1.11	0.87	n.a. ²⁾	1.11
Ti	4.45	4.83	0.83	5.84	5.84
Zn	4.87	n.c. ¹⁾	0.91	n.a. ²⁾	0.91
Zr	6.67	7.02	1.25	7.53	7.53

¹⁾n.c.: cannot be calculated as $MS_{between} < MS_{within}$ ²⁾n.a.: not applicable**Table 7:** Results of the homogeneity study of ERM-EB075B

ERM-EB075B	$S_{wb,rel}$ [%]	$S_{bb,rel}$ [%]	$U_{bb,rel}$ [%]	$U_{rec,rel}$ [%]	$U_{bb,rel}$ [%]
Ag	2.20	n.c. ¹⁾	0.41	n.a. ²⁾	0.41
Al	8.26	n.c. ¹⁾	1.55	n.a. ²⁾	1.55
As	2.61	n.c. ¹⁾	0.49	n.a. ²⁾	0.49
Au	3.22	n.c. ¹⁾	0.60	n.a. ²⁾	0.60
Be	8.58	n.c. ¹⁾	1.61	n.a. ²⁾	1.61
Bi	5.62	n.c. ¹⁾	1.05	n.a. ²⁾	1.05
Cd	3.03	n.c. ¹⁾	0.57	n.a. ²⁾	0.57
Co	2.15	0.46	0.40	n.a. ²⁾	0.46
Cr	3.08	0.35	0.58	n.a. ²⁾	0.58
Fe	2.61	0.27	0.49	n.a. ²⁾	0.49
In	5.90	0.43	1.10	n.a. ²⁾	1.10
Mg	4.35	0.88	0.81	n.a. ²⁾	0.88
Mn	1.25	0.50	0.23	n.a. ²⁾	0.50
Ni	3.01	0.79	0.56	n.a. ²⁾	0.79
O	42.15	28.40	10.08	n.a. ²⁾	28.40
P	5.41	2.19	1.01	n.a. ²⁾	2.19
Pb	5.00	1.21	0.94	3.35	3.35
S	3.47	1.19	0.65	n.a. ²⁾	1.19
Sb	4.12	n.c. ¹⁾	0.77	n.a. ²⁾	0.77
Se	3.52	0.46	0.66	n.a. ²⁾	0.66
Si	10.24	1.45	1.93	n.a. ²⁾	1.93
Sn	3.61	0.12	0.67	n.a. ²⁾	0.67
Te	4.50	n.c. ¹⁾	0.84	n.a. ²⁾	0.84
Ti	4.42	0.69	0.83	n.a. ²⁾	0.83
Zn	3.24	n.c. ¹⁾	0.61	n.a. ²⁾	0.61
Zr	7.30	3.40	1.37	10.95	10.95

¹⁾n.c.: cannot be calculated as $MS_{between} < MS_{within}$ ²⁾n.a.: not applicable

Table 8: Results of the homogeneity study of ERM-EB075C

ERM-EB075C	$S_{wb,rel}$ [%]	$S_{bb,rel}$ [%]	$U_{bb,rel}$ [%]	$U_{rec,rel}$ [%]	$U_{bb,rel}$ [%]
Ag	10.19	n.c. ¹⁾	2.00	n.a. ²⁾	2.00
Al	13.94	n.c. ¹⁾	2.75	n.a. ²⁾	2.75
As	3.90	n.c. ¹⁾	0.77	n.a. ²⁾	0.77
Au	6.17	1.70	1.21	n.a. ²⁾	1.70
Be	10.50	n.c. ¹⁾	2.06	n.a. ²⁾	2.06
Bi	4.65	n.c. ¹⁾	0.91	n.a. ²⁾	0.91
Cd	3.52	0.69	0.69	n.a. ²⁾	0.69
Co	3.30	n.c. ¹⁾	0.65	n.a. ²⁾	0.65
Cr	3.61	n.c. ¹⁾	0.71	n.a. ²⁾	0.71
Fe	3.64	n.c. ¹⁾	0.72	n.a. ²⁾	0.72
In	4.28	n.c. ¹⁾	0.84	n.a. ²⁾	0.84
Mg	7.10	n.c. ¹⁾	1.39	n.a. ²⁾	1.39
Mn	3.39	n.c. ¹⁾	0.67	n.a. ²⁾	0.67
Ni	3.95	0.80	0.78	n.a. ²⁾	0.80
O	47.70	n.c. ¹⁾	11.97	n.a. ²⁾	11.97
P	16.00	n.c. ¹⁾	3.14	n.a. ²⁾	3.14
Pb	4.33	n.c. ¹⁾	0.85	2.34	2.34
S	3.31	0.35	0.83	n.a. ²⁾	0.83
Sb	4.24	0.77	0.83	n.a. ²⁾	0.83
Se	15.47	n.c. ¹⁾	3.04	n.a. ²⁾	3.04
Si	12.98	n.c. ¹⁾	2.55	n.a. ²⁾	2.55
Sn	10.43	n.c. ¹⁾	2.05	n.a. ²⁾	2.05
Te	7.06	n.c. ¹⁾	1.39	n.a. ²⁾	1.39
Ti	4.36	n.c. ¹⁾	0.86	n.a. ²⁾	0.86
Zn	3.45	n.c. ¹⁾	0.68	n.a. ²⁾	0.68
Zr	7.78	1.29	1.53	7.62	7.62

¹⁾n.c.: cannot be calculated as $MS_{between} < MS_{within}$

²⁾n.a.: not applicable

The three formats were produced from the same melt; it was decided to assign one uncertainty contribution to ERM-EB075. It is detailed in Section 4.3.

4.2 Within-unit homogeneity and minimum sample intake

The within-unit homogeneity is closely correlated to the minimum sample intake. Due to this correlation, individual aliquots of a material will not contain the same amount of analyte. The minimum sample intake is the lowest amount of sample that is representative of the whole unit and thus can be used in an analysis. Sample sizes greater than or equal to the minimum sample intake guarantee the certified value within its stated uncertainty.

4.2.1 Radial within-unit homogeneity for ERM-EB075A

For ERM-EB075A, which was suspected of being inhomogeneous across the face of a disk, perhaps due to the migration of certain elements during cooling of casting, face homogeneity was tested using solid sampling techniques that consume microgram quantities of material. A mapping technique was applied in which analytical spots were selected across the face of the discs.

For the radial within unit homogeneity study, one unit of ERM-EB075A was selected randomly and cut into four thinner discs to test four different faces for the radial within-unit homogeneity test. The discs were prepared according the instruction for use, including mechanical cleaning of the surfaces.

For this, the disc face was divided into three areas, central, intermediate and external area (Figure 2B). Three, four and six independent locations were analysed from the central, intermediate and external areas respectively. Analyses were performed by , by spark-OES

for Ag, Al, As, Be, Bi, Cd, Co, Cr, Fe, Mg, Mn, Ni, P, Pb, S, Sb, Se, Si, Sn, Ti, Zn, Zr mass fraction.

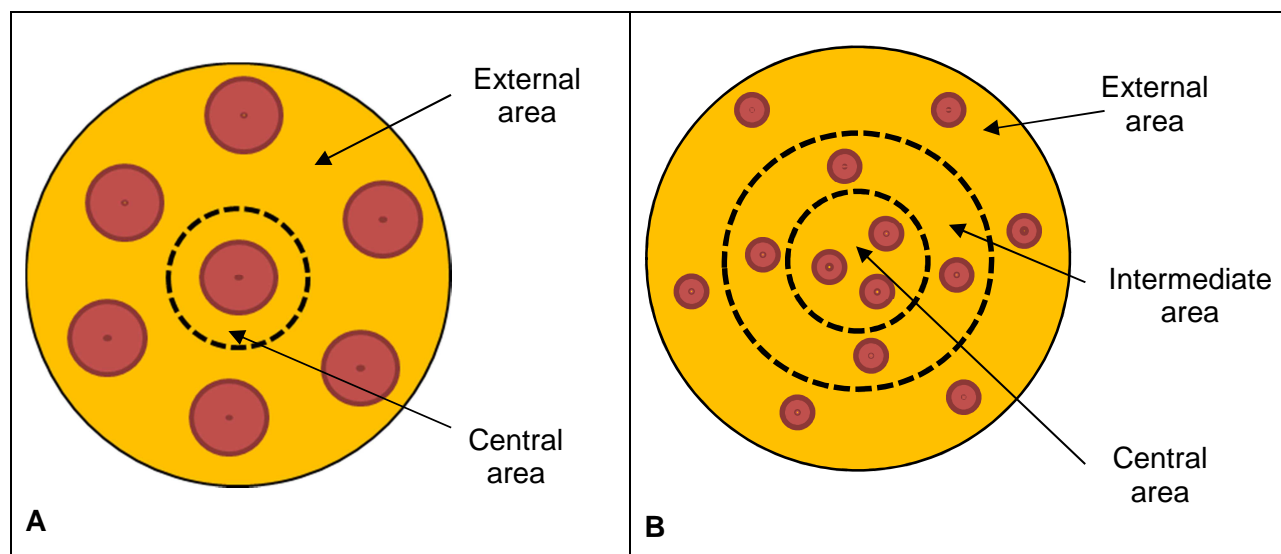


Figure 2: Figure 2A: Localisation of the two different areas for the radial within-unit homogeneity study by GD-MS; Figure 2B: Localisation of the three different areas for radial within-unit homogeneity study by spark-OES. The red points represent the number of spot analysis per area.

For Au, In and Te, the within-unit homogeneity study was performed by GD-MS. The analysis area is larger for GD-MS than for spark-OES. Therefore, it was only possible to perform one analysis in the central area and six analyses in the external area. No analyses were made in the intermediate area (Figure 2A). One and six independent locations from central and external area were analysed by GD-MS for Au, In and Te.

The measurements were performed in a randomised block design because the number of replicates on all units (52 analyses) could not be included in a single run due to instrumental constraints (drift towards the end of a long run). Improved precision (measured as the within-unit standard deviation) was obtained using several short runs in a randomised block design.

In the randomised block design for 13 independent spot analyses on each of the four faces of ERM-EB075A, four measurement sequences (one measurement sequence per face) were planned, with each spot measured once in random order. Runs were randomised individually in a manner to be able to separate a potential analytical drift from any radial trend in the disc.

Regression analysis and F-test (or T-test for Au, In and Te) were used to estimate the radial within-unit homogeneity independently of the analytical sequence effect. The linear regression analysis allows detecting radial, linear inhomogeneity and the F-test allows detecting radial non-linear inhomogeneity.

The data evaluation was performed in the following order:

1 – Trends in analytical run / correction for significant trends

Regression analyses were performed to evaluate potential trends in each analytical run. Some significant (95 % confidence level) trends in the analytical sequence were visible, pointing at a signal drift in the analytical system (Table 1). As the analytical sequence and the unit numbers were not correlated, trends significant on a 95 % confidence level were corrected as given in equation 1.

2 – Evaluation of between analytical run effect / Normalisation of dataset (if necessary)

The analytical trend-corrected dataset was evaluated for significant difference between analytical runs (95 % confidence level) using one way ANOVA. Significant differences between analytical runs were observed on the 95 % confidence level for all elements in ERM-EB075A except As, Sb, Sn, Te, Zn and Zr (Table 10). As it is assumed that run effects and unit effects were independent, differences between analytical runs on 95 % confidence level were corrected as given in equation 2.

3 – Statistical evaluation of the datasets

The normalised datasets were tested for consistency using Grubbs outlier tests on a confidence level of 99 % on the individual results and the unit means (Table 10).

No outlying means were detected on the 99 % confidence level. Central, intermediate and external area mean values are given in Annex B.

Regression analyses were performed to evaluate potential radial linear trends within-unit. No trends within the unit face were visible on a 95 % confidence level for all elements except Be, Fe, Mg, Sb and Se were significant radial trends within unit (decrease or increase from center to external area) were detected (95 % confidence level).

An F-test was used to determine if a significant difference is observed between the three areas on a 95 % confidence level. For Cd, Fe, Mn, Se and Zn, the difference between face areas was significant on a 95% confidence level.

For Au, In and Te, a T-test was used to determine if a significant difference is observed between the central and external areas on a 95 % confidence level. For In, the difference between central and external area was significant on a 95 % confidence level.

For Be, Fe, In, Mn, Mg, Sb, Se and Zn, inhomogeneity was observed along radial axis of ERM-EB075A face. Therefore, u_{rec} was estimated using a rectangular distribution between the highest and lowest face area mean [12]. The uncertainty in those cases is given in:

$$u_{rec,rel} = \frac{|highest\ result - lowest\ result|}{2 \cdot \sqrt{3} \cdot \bar{y}} \quad \text{Equation 3}$$

\bar{y} mean of all results of the homogeneity study

The results of the study is summarised in Table 10.

The radial inhomogeneity was tested, and considered as not significant for Ag, Al, As, Au, Bi, Cd, Co, Cr, Ni, P, Pb, S, Si, Sn, Ti and Zr. Significant radial inhomogeneity was observed for Be, Fe, In, Mg, Mn, Sb, Se, Te and Zn and estimated using rectangular distribution. The uncertainty contribution from radial within-unit uncertainty is below 1 % for all elements which is considered sufficiently small to make the material useful.

Table 10: Results of the statistical evaluation of the radial within-unit homogeneity study of ERM-EB075A; *T* = number of series with analytical trend

ERM-EB075A	Analytical trends	Between analytical run difference	Outliers at 99% confidence level		Trend / inhomogeneity		Inhomogeneity uncertainty
			Unit means	Individual results	Radial trend at 95% confidence level	Between area difference at 95% confidence level	
Analyte	Significant at 95% confidence level (<i>T</i>)	Significant difference at 95% confidence level					$U_{rec,rel}$ [%]
Ag	Yes (1)	Yes	None	None	No	No (F-test)	n.a. ²⁾
Al	Yes (2)	Yes	None	None	No	No (F-test)	n.a. ²⁾
As	No	No	None	None	No	No (F-test)	n.a. ²⁾
Au ¹⁾	Yes (1)	Yes	None	None	n.a. ²⁾	No (T-test)	n.a. ²⁾
Be	No	Yes	None	None	Yes	No (F-test)	0.54
Bi	No	Yes	None	None	No	No (F-test)	n.a. ²⁾
Cd	No	Yes	None	None	No	No (F-test)	n.a. ²⁾
Co	Yes (2)	Yes	None	None	No	No (F-test)	n.a. ²⁾
Cr	Yes (2)	Yes	None	None	No	No (F-test)	n.a. ²⁾
Fe	No	Yes	None	None	Yes	Yes (F-test)	0.70
In ¹⁾	No	Yes	None	None	n.a. ²⁾	Yes (T-test)	0.80
Mg	Yes (1)	Yes	None	None	Yes	No (F-test)	0.68
Mn	No	Yes	None	None	No	Yes (F-test)	0.22
Ni	No	Yes	None	None	No	No (F-test)	n.a. ²⁾
P	No	Yes	None	None	No	No (F-test)	n.a. ²⁾
Pb	Yes (2)	Yes	None	None	No	No (F-test)	n.a. ²⁾
S	No	Yes	None	None	No	No (F-test)	n.a. ²⁾
Sb	No	No	None	None	Yes	No (F-test)	0.84
Se	No	Yes	None	None	Yes	Yes (F-test)	0.93
Si	No	Yes	None	None	No	No (F-test)	n.a. ²⁾
Sn	No	No	None	None	No	No (F-test)	n.a. ²⁾
Te ¹⁾	No	No	None	None	n.a. ²⁾	Yes (T-test)	0.98 ²⁾
Ti	No	Yes	None	None	No	No (F-test)	n.a. ²⁾
Zn	No	No	None	None	No	Yes (F-test)	0.91
Zr	No	No	None	None	No	No (F-test)	n.a. ²⁾

¹⁾ Au, In and Te were tested using GD-MS and only on central and external area

²⁾ n.a.: not applicable

4.2.2 Minimum sample intake estimation

Homogeneity experiments were performed using GD-MS and spark-OES technique for Ag, Al, As, Au, Be, Bi, Cd, Co, Cr, Fe, Hg, In, Mg, Mn, Ni, P, Pb, S, Sb, Se, Si, Sn, Te, Ti, W, Zn, Zr. The sample intake of these methods gives acceptable repeatability, demonstrating that the within-unit inhomogeneity does not contribute to analytical variation at this sample intake.

Quantification of the sample intake for GD-MS and spark-OES is difficult, as both are direct surface sampling and analysis methods. In contrast, the minimum sample intake required for ICP-MS is significantly larger and quantifiable. Based on the acceptable repeatability of the direct surface sampling and analysis methods, the sample intake of those methods meets the minimum sample intake requirements for ERM-EB075A and B.

To estimate the sample intake used for GD-MS and spark-OES, rough estimation was done for each techniques. For spark-OES, a spot analysis of 4 mm diameter, a depth of 0.1 mm and the copper density of 8.96 g/cm³ was assumed. For GD-MS, a spot analysis of 8 mm diameter, a depth of 0.05 mm and the copper density of 8.96 g/cm³ was assumed. Therefore, the sample intake of spark-OES was estimated to approximately 11 mg and 20 mg for GD-MS. Using the rough estimation of GD-MS and spark-OES sample intake, the following minimum sample intakes are derived:

- Sample intake of 10 mg for Ag, Al, As, Be, Bi, Cd, Co, Cr, Fe, Hg, Mg, Mn, Ni, P, Pb, S, Sb, Se, Si, Sn, Ti, W, Zn, Zr

- Sample intake of 20 mg for Au, In and Te.

Results by laser ablation (data not shown) indicate that the material is not homogeneous on the micrometre scale. Results from several points need to be pooled when using laser ablation

4.3 Uncertainty of homogeneity of ERM-EB075A, B and C

The three formats are produced from the same melt; it was decided to assign the same uncertainty contribution to all three formats.

For ERM-EB075A, the final homogeneity uncertainty is the combination of the $u_{bb,rel}$ and the $u_{within-rad,rel}$.

The largest $u_{bb,rel}$ of ERM-EB075A, B and C is adopted as the uncertainty inhomogeneity of ERM-EB075 except when the uncertainty contribution of ERM-EB075C corresponds to $u_{bb,rel}^*$ for the following reason:

The methods used to assess homogeneity (Table 2) were different for ERM-EB075A, B (GD-MS) and ERM-EB075C (ICP-MS/ICP-OES). The GD-MS method used for ERM-EB075A and B provided better repeatability and lower $u_{bb,rel}^*$ than the method used for ERM-EB075C. It should be noticed that in all cases except for Au, Ni, Pb and Zr, the homogeneity uncertainty assigned to ERM-EB075C corresponds to $u_{bb,rel}^*$. In order to avoid overestimation of the homogeneity uncertainty due to the method repeatability, $u_{bb,rel}^*$ of ERM-EB075C was not used for the estimation of $u_{bb,rel}$ of ERM-EB075.

The results of the combined homogeneity uncertainty are given in Table 9.

The uncertainty of homogeneity of the Ag, Al, As, Au, Bi, Cd, Co, Cr, Fe, Hg, In, Mg, Mn, Ni, P, Pb, S, Sb, Se, Si, Sn, Te, Ti, W and Zn mass fractions in ERM-EB075A, B and C is sufficient small to make the material useful. The homogeneity study showed significant inhomogeneity for the Be, O and Zr mass fractions. The homogeneity uncertainty estimated is not sufficient to certify the mass fraction of O. Typical measurement uncertainties of for Be and Zr are higher, so a certified value can be assigned despite the larger uncertainty.

Table 9: Summary of the homogeneity study of ERM-EB075, the largest uncertainty contribution from the three formats ERM-EB075A, B and C is adopted as uncertainty contribution to account for potential inhomogeneity except when the uncertainty contribution of ERM-EB075C corresponds to $u_{bb,rel}^*$

Analyte	ERM-EB075A			Combined $u_{bb,rel}$ [%]	ERM-EB075B		ERM-EB075C		ERM-EB075 $u_{bb,rel}$ [%]
	$u_{bb,rel}$ [%]	$u_{within-rad.,rel}$ [%]			$u_{bb,rel}$ [%]		$u_{bb,rel}$ [%]		
Ag	$u_{rec,rel}$	0.64	n.a. ¹⁾	0.64	$s_{bb,rel}$	0.41	$s_{bb,rel}$	2.00	2.00
Al	$s_{bb,rel}$	4.70	n.a. ¹⁾	4.70	$s_{bb,rel}$	1.55	$u_{rec,rel}$	2.75	4.70
As	$u_{bb,rel}$	0.55	n.a. ¹⁾	0.55	$u_{bb,rel}$	0.49	$u_{bb,rel}$	0.77	0.55
Au	$u_{bb,rel}$	0.63	n.a. ¹⁾	0.63	$u_{bb,rel}$	0.60	$u_{bb,rel}$	1.70	1.70
Be	$s_{bb,rel}$	10.23	0.54	10.25	$u_{bb,rel}$	1.61	$u_{bb,rel}$	2.06	10.25
Bi	$u_{bb,rel}$	1.16	n.a. ¹⁾	1.16	$u_{bb,rel}$	1.05	$u_{bb,rel}$	0.91	1.16
Cd	$u_{bb,rel}$	1.10	n.a. ¹⁾	1.10	$s_{bb,rel}$	0.57	$u_{bb,rel}$	0.69	1.10
Co	$u_{rec,rel}$	0.58	n.a. ¹⁾	0.58	$s_{bb,rel}$	0.46	$u_{bb,rel}$	0.65	0.58
Cr	$u_{rec,rel}$	0.48	n.a. ¹⁾	0.48	$u_{bb,rel}$	0.58	$u_{bb,rel}$	0.71	0.58
Fe	$u_{rec,rel}$	0.37	0.70	0.79	$s_{bb,rel}$	0.49	$u_{rec,rel}$	0.72	0.79
In	$u_{bb,rel}$	1.22	0.80	1.46	$s_{bb,rel}$	1.10	$u_{bb,rel}$	0.84	1.46
Mg	$s_{bb,rel}$	3.91	0.68	3.97	$u_{bb,rel}$	0.88	$u_{bb,rel}$	1.39	3.97
Mn	$s_{bb,rel}$	1.17	0.22	1.20	$u_{bb,rel}$	0.50	$u_{bb,rel}$	0.67	1.20
Ni	$s_{bb,rel}$	3.01	n.a. ¹⁾	3.01	$s_{bb,rel}$	0.79	$u_{bb,rel}$	0.80	3.01
O	$u_{rec,rel}$	15.02	n.a. ¹⁾	15.02	$s_{bb,rel}$	28.40	$s_{bb,rel}$	11.97	28.40
P	$s_{bb,rel}$	1.25	n.a. ¹⁾	1.25	$s_{bb,rel}$	2.19	$s_{bb,rel}$	3.14	2.19
Pb	$u_{rec,rel}$	9.09	n.a. ¹⁾	9.09	$u_{rec,rel}$	3.35	$u_{rec,rel}$	2.34	9.09
S	$s_{bb,rel}$	0.85	n.a. ¹⁾	0.85	$s_{bb,rel}$	1.19	$s_{bb,rel}$	0.83	1.19
Sb	$u_{bb,rel}$	0.86	0.84	1.20	$u_{bb,rel}$	0.77	$u_{bb,rel}$	0.83	1.20
Se	$u_{bb,rel}$	0.94	0.93	1.33	$s_{bb,rel}$	0.66	$u_{bb,rel}$	3.04	1.33
Si	$u_{rec,rel}$	0.84	n.a. ¹⁾	0.84	$u_{rec,rel}$	1.93	$u_{bb,rel}$	2.55	1.93
Sn	$u_{rec,rel}$	0.91	n.a. ¹⁾	0.91	$s_{bb,rel}$	0.67	$u_{bb,rel}$	2.05	0.91
Te	$u_{bb,rel}$	1.11	0.98	1.48	$s_{bb,rel}$	0.84	$s_{bb,rel}$	1.39	1.48
Ti	$s_{bb,rel}$	5.84	n.a. ¹⁾	5.84	$s_{bb,rel}$	0.83	$s_{bb,rel}$	0.86	5.84
Zn	$u_{rec,rel}$	0.91	0.91	1.29	$s_{bb,rel}$	0.61	$u_{bb,rel}$	0.68	1.29
Zr	$s_{bb,rel}$	7.53	n.a. ¹⁾	7.53	$s_{bb,rel}$	10.95	$s_{bb,rel}$	7.62	10.95

¹⁾ n.a.: not applicable

5 Stability

Stability assessment is necessary to establish conditions for storage (long-term stability) as well as conditions for dispatch to the customers (short-term stability). During transport, especially in the summer, temperatures up to 60 °C could be reached and stability under these conditions must be demonstrated if transport at ambient temperature has to be applied.

Metallic copper is stable over time and temperature as archaeological artefacts and antique copper cooking ware demonstrate: they remain unchanged without special precautions for hundreds and thousands of years.

Copper has good chemical resistance to degradation under normal storage and handling conditions. However, copper is subject to surface oxidation over time. It is mandatory to remove the oxide layer that may appear on the surface of the material as explained in the section 9.3 Instructions for use before using ERM-EB075A,B and C.

Concerning the stability of trace elements in electrolytic copper, data from BCR-075 [12] was used. BCR-075 was released in 1992 and used in this investigation as a quality control material for evaluation of the characterisation results. The BCR-075 results reported in this study confirmed that the certified values are taking into consideration the measurement uncertainty and substantiated the stability of trace elements in copper.

The trace element mass fractions in ERM-EB075A, B and C are therefore considered stable under normal dispatch conditions (up to 60 °C).

Based on previous experience, a validity period of the certificate for ERM-EB074A, B and C of 10 years is set.

Recommended storage and transport conditions:

The material can be transported under ambient conditions without special precautions and stored at a temperature not exceeding 18 ± 5 °C. Uncertainties due to storage and shipment conditions were considered negligible regarding the material property.

After the certification campaign, the material will be subjected to IRMM's regular stability monitoring programme to control its further stability.

6 Characterisation

Material characterisation is the process of determining the property values of a reference material. The material characterisation was based on an intercomparison of expert laboratories. The Ag, Al, As, Au, Be, Bi, Cd, Co, Cr, Fe, H, Hg, In, Mg, Mn, Ni, O, P, Pb, S, Sb, Se, Si, Sn, Te, Ti, W, Zn and Zr mass fractions of the ERM-EB075A, B and C were determined in different laboratories that applied different measurement procedures to demonstrate the absence of a measurement bias. This approach aims at randomisation of laboratory bias. The intercomparison of laboratories seeks to reduce the combined uncertainty.

6.1 Selection of participants

Nineteen laboratories were selected based on criteria that comprised both technical competence and quality management aspects. Each participant was required to operate a quality system and to deliver documented evidence of its laboratory proficiency in the field of element measurements in relevant matrices by submitting results for intercomparison exercises or method validation reports. Having a formal accreditation was not mandatory, but meeting the requirements of ISO/IEC 17025 was obligatory. Where the scope of accreditation covers measurements, the accreditation number is stated in the list of participants (Section 2).

6.2 Study setup

Each laboratory received two units of ERM-EB075A, B and C and was requested to provide at least 3 independent results per unit. Several analytical techniques (i.e. GD-MS, spark-OES, DC-arc-OES) were not able to analyse trace elements in all three formats due to instrument constraints (sample size). Each laboratory was asked to provide the results of at least 3 independent measurements on each of the two units for one format analysed for the following elements: Ag, Al, As, Au, Be, Bi, Cd, Co, Cr, Fe, H, Hg, In, Mg, Mn, Ni, O, P, Pb, S, Sb, Se, Si, Sn, Te, Ti, W, Zn and Zr.

The units for material characterisation were selected using a random stratified sampling scheme and covered the whole batch. The sample preparations and measurements had to be spread over at least two days to ensure intermediate precision conditions. An independent calibration was performed for each result.

Each participant received a sample of BCR-075 as a blind quality control (QC) sample except for L13 which used BCR-075 for calibration. In this case, the calibration results were used to support the evaluation of the characterisation results.

Laboratories were also requested to give estimations of the expanded uncertainties of the mean value of their results. No approach for the estimation was prescribed, i.e. top-down and bottom-up were regarded as equally valid procedures. These uncertainties were used to evaluate the dispersion of the laboratory results (Annex E).

The majority of the laboratories reported the same number of measurements for each format. Some techniques were only capable to analyse one or two formats (i.e. GD-MS can analyse ERM-EB075A and B; DC-arc-OES can analyse ERM-EB075C).

6.3 Methods used

A variety of acid digestion (HCl, HNO₃, HF), extraction methods (co-precipitation with yttrium) with different quantification steps (ICP-MS, ICP-OES) as well as methods without sample preparation (combustion with IR quantification, GD-MS, LA-ICP-MS, IGF-IR, INAA or spark-OES) were used to characterise the material. The combination of results from methods based on entirely different principles mitigates undetected method bias.

All methods used during the characterisation study are summarised in Annex C. The laboratory code (e.g. L1) is a random number and does not correspond to the order of laboratories in Section 2. The lab-method code consists of a number assigned to each laboratory (e.g. L01) and abbreviation of the measurement method used (e.g. ICP-MS).

6.4 Evaluation of results

The characterisation campaign resulted in 3-17 datasets per element. All individual results of the participants, grouped per element are displayed in a tabular and/or graphical form in Annex D.

ERM-EB075A, B and C were considered homogeneous. Therefore, the results were pooled per laboratory independently of the format analysed by each laboratory. The characterisation study was performed for each format using the accepted datasets to support this assumption. The mean of laboratory means and the standard deviation of the mean are presented in each format in Annex E, demonstrating the equivalence of the three formats.

6.4.1 Technical evaluation

The obtained data were first checked for compliance with the requested analysis protocol and their validity based on technical reasons. The following criteria were considered during the evaluation:

- appropriate validation of the measurement procedure,
- compliance with the analysis protocol: sample preparations and measurements performed on two days, and the analytical sequence,
- absence of values given as a below limit of detection or below limit of quantification,
- absence of technical problems (i.e. contamination),
- method performance, i.e. agreement of the measurement results with the assigned value of the QC sample. As this test is performed using the respective uncertainties of the certified values and the measurement uncertainties estimated by the laboratory, it should be borne in mind that even national metrology institutes tend to underestimate their measurement uncertainties [16].

Most technical problems were inherent in one laboratory only, except data for Zr: Laboratories not using HF for digestion found considerable lower values for Zr than laboratories using HF. This can be explained by the fact that Zr precipitates in the absence of

HF [17]. Laboratory 9 repeated the analysis using HF and obtained results between 4.3 and 6.4 mg/kg, confirming that this is not a laboratory-dependent effect. Therefore, results from digestions without HF were excluded for Zr.

Based on the above criteria, the following datasets were rejected as not technically valid (Table 11).

Table 11: Datasets that showed non-compliances with the analysis protocol and technical specifications, and action taken.

Analyte	Laboratory code	Description of problem	Action taken
Ag	L12 L1	Deviating result on QC sample Technical problem (contamination)	not used for evaluation
Al	L1, L18	Technical problem (contamination)	not used for evaluation
As	L8, L18	Deviating result on QC sample	not used for evaluation
Au	L9	Technical problem (no use of aqua regia)	not used for evaluation
Bi	L3, L4, L5 and L18	Deviating result on QC sample	not used for evaluation
Cd	L18	Deviating result on QC sample	not used for evaluation
Co	L10, L15, L18	Deviating result on QC sample	not used for evaluation
Cr	L14, L15, L18	Deviating result on QC sample	not used for evaluation
Fe	L7, L8, L15, L21 L9, L10 L1	Deviating result on QC sample Report results below LOD Technical problem (contamination)	not used for evaluation
Mg	L20	Technical problem (blank)	not used for evaluation
Mn	L5, L8, L18 L21	Deviating result on QC sample Report results below LOD	not used for evaluation
Ni	L8, L18	Deviating result on QC sample	not used for evaluation
O	L1 L17	Report results below LOD Dataset not compliant with analysis protocol	not used for evaluation
P	L1, L6, L9	Report results below LOD	not used for evaluation
Pb	L1, L2, L8, L18, L20, L21	Deviating result on QC sample	not used for evaluation
S	L4	Technical problem	not used for evaluation
Sb	L3 L5	Technical problem Deviating result on QC sample	not used for evaluation
Se	L1, L4, L8, L11, L20	Deviating result on QC sample	not used for evaluation
Si	L1, L9	Report results below LOD	not used for evaluation
Sn	L19, L21	Deviating result on QC sample	not used for evaluation
Te	L9 L2	Deviating result on QC sample Report results below LOD	not used for evaluation
Zn	L1, L8, L9, L19, L21	Deviating result on QC sample	not used for evaluation
Zr	L1, L8, L9, L18	Technical problem in digestion (HF not used for digestion)	not used for evaluation

6.4.2 Statistical evaluation

The datasets accepted based on technical reasons were tested for normality of dataset means using kurtosis/skewness tests and normal probability plots. Datasets were further evaluated for outlying means using the Grubbs test and the Cochran test for outlying standard deviations, (both at a 99 % confidence level). Standard deviations within (s_{within}) and between ($s_{between}$) laboratories were calculated using one-way ANOVA. The results of these evaluations are shown in Table 12.

Table 12: Statistical evaluation of the technically accepted datasets for Ag, Al, As, Au, Be, Bi, Cd, Co, Cr, Fe, In, Mg, Mn, Ni, O, P, Pb, S, Sb, Se, Si, Sn, Te, Ti, Zn and Zr. p : number of technically valid datasets

Analyte	p	Outliers		Normally distributed	Statistical parameters			
		Means	Variances		Mean of the laboratory means [mg/kg]	s [mg/kg]	$s_{between}$ [mg/kg]	s_{within} [mg/kg]
Ag	18	No	Yes (L5)	Yes	10.765	0.650	0.672	0.413
Al	11	No	Yes (L4)	Yes	2.304	0.376	0.341	0.314
As	17	No	Yes (L5)	Yes	3.184	0.190	0.178	0.194
Au	11	No	No	Yes	1.425	0.214	0.218	0.099
Be	12	No	Yes (L9)	Yes	1.077	0.145	0.139	0.104
Bi	14	No	Yes (L2)	Yes	1.787	0.191	0.202	0.151
Cd	15	No	Yes (L5)	Yes	2.692	0.121	0.101	0.142
Co	16	No	Yes (L9)	Yes	2.637	0.147	0.145	0.134
Cr	16	No	Yes (L6)	Yes	1.401	0.129	0.125	0.136
Fe	11	No	Yes (L2)	Yes	9.344	0.579	0.558	0.906
In	14	No	Yes (L5)	Yes	1.829	0.151	0.152	0.121
Mg	12	No	Yes (L9)	Yes	7.019	0.707	0.698	0.365
Mn	11	Yes (L11)	Yes (L9)	Yes	1.348	0.094	0.097	0.072
Ni	16	No	Yes (L1)	Yes	2.179	0.153	0.153	0.233
O	3	No	Yes (L9)	Yes	3.367	1.443	1.364	0.874
P	10	No	No	Yes	2.594	0.436	0.417	0.284
Pb	12	Yes (L6)	Yes (L5)	Yes	4.848	0.332	0.345	0.374
S	8	No	Yes (L1)	Yes	25.317	4.494	4.762	2.031
Sb	18	Yes (L11)	Yes (L16)	Yes	2.926	0.244	0.257	0.154
Se	15	No	Yes (L7)	Yes	1.694	0.170	0.162	0.174
Si	6	No	Yes (L6)	Yes	2.589	0.444	0.381	0.437
Sn	15	No	Yes (L1)	Yes	2.131	0.194	0.163	0.255
Te	16	No	Yes (L7)	Yes	1.783	0.204	0.214	0.149
Ti	13	No	Yes (L5)	No	3.185	0.295	0.242	0.242
Zn	15	No	Yes (L4)	Yes	6.513	0.481	0.499	0.481
Zr	8	No	Yes (L5)	Yes	19.537	3.783	3.208	3.154

The laboratory means follow a normal distribution. None of the data contained outlying means except Mn, Pb and Sb. The statistical evaluation flagged at least one laboratory as having an outlying variance for each analyte except for Au and P (Table 12). This reflects the fact that different methods have different intrinsic variability. As all measurement methods were found to be technically sound, all the results were retained.

The statistical evaluation flagged laboratory L6 as outlier for Pb and L11 as outlier for Mn and Sb mass fraction, although outlier tests do not take uncertainty information into consideration. A closer investigation revealed that the difference between the mean Pb, Mn and Sb values of laboratory L6 and L11 and the other dataset were covered by the measurement uncertainty of laboratories L6 and L11. There is, therefore, no evidence that the results of laboratory L6 and L11 deviate from the other results.

For the Al, As, Cr and S mass fractions, a closer investigation revealed that a difference between the assigned value and L20 (for Al and As) and L21 (for Cr and S) mean value, respectively, is not covered by the measurement uncertainty. However, the measurement uncertainties reported by L20 and L21 is considered to be underestimated (two times the standard deviation). Standard deviations among laboratories were larger than the standard deviation within laboratories, showing that two times the standard deviation of replicate measurements is unsuitable as an estimate of measurement uncertainty. Moreover, the L20 and L21 mean value for Al, As, Cr and S were not flagged as outliers by the statistical test. Therefore, it was decided to keep the Al, As, Cr and S uncertainty as calculated in Table 13.

For the Au and Zr mass fractions, a closer investigation revealed that a difference between the assigned value and the L8 mean value is not covered by the measurement uncertainty. However, the measurement uncertainty reported by L8 is considered to be underestimated compared to other uncertainties for the ICP-MS method. Moreover, the L8 mean values for Au and Zr were not flagged as an outlier by the statistical test. Therefore, it was decided to keep the Au and Zr uncertainty as calculated in Table 13.

For In mass fraction, a closer investigation revealed that the measurement uncertainty does not cover the difference between the assigned value and L2 mean value. However, the measurement uncertainty reported by L2 is considered to be underestimated compared to other uncertainties for the ICP method. Moreover, the L2 mean value was not flagged as an outlier by the statistical test. Therefore, it was decided to keep the uncertainty for In as calculated in Table 13.

The datasets therefore are consistent and the mean of laboratory means is considered to be a good estimate of the true value. The uncertainty related to the characterisation (u_{char}) is estimated as the standard error of the mean of laboratory means (Table 13).

Table 13: Uncertainty of characterisation for Ag, Al, As, Au, Be, Bi, Cd, Co, Cr, Fe, In, Mg, Mn, Ni, O, P, Pb, S, Sb, Se, Si, Sn, Te, Ti, Zn and Zr.

Analyte/	ρ	Mean [mg/kg]	s [mg/kg]	u_{char} [mg/kg]	$u_{\text{char,rel}}$ [%]
Ag	18	10.765	0.650	0.153	1.42
Al	11	2.304	0.376	0.113	4.92
As	17	3.184	0.190	0.046	1.45
Au	10	1.455	0.199	0.063	4.32
Be	12	1.077	0.145	0.042	3.88
Bi	14	1.787	0.191	0.051	2.86
Cd	15	2.692	0.121	0.031	1.16
Co	16	2.637	0.147	0.037	1.39
Cr	16	1.401	0.129	0.032	2.30
Fe	11	9.344	0.579	0.175	1.87
In	14	1.829	0.151	0.040	2.21
Mg	12	7.019	0.707	0.204	2.91
Mn	11	1.348	0.094	0.028	2.10
Ni	16	2.179	0.153	0.038	1.76
O	3	3.367	1.443	0.833	24.74
P	10	2.594	0.436	0.138	5.31
Pb	13	4.807	0.351	0.097	2.02
S	8	25.317	4.494	1.589	6.28
Sb	18	2.926	0.244	0.057	1.96
Se	15	1.694	0.170	0.044	2.60
Si	6	2.589	0.444	0.181	6.99
Sn	15	2.131	0.194	0.050	2.35
Te	16	1.783	0.204	0.051	2.86
Ti	13	3.185	0.295	0.082	2.57
Zn	15	6.513	0.481	0.124	1.91
Zr	8	19.537	3.783	1.337	6.85

7 Value Assignment

Certified, indicative and informative values were assigned.

Certified values are values that fulfil the highest standards of accuracy. Procedures at IRMM require generally pooling of not less than 6 datasets to assign certified values. Full uncertainty budgets in accordance with the 'Guide to the Expression of Uncertainty in Measurement' [4] were established.

Indicative values are values where either the uncertainty is deemed too large or where too few independent datasets were available to allow certification. Uncertainties are evaluated according to the same rules as for certified values.

Additional material information refers to values that were obtained in the course of the study. For example, results reported from only one or two laboratories or in cases where individual measurement uncertainty is high, would fall under this category.

7.1 Certified values and their uncertainties

The unweighted mean of the means of the accepted datasets, as shown in Table 12, was assigned as certified value for each parameter.

The assigned uncertainty consists of uncertainties related to characterisation, u_{char} (Section 6), potential between-unit inhomogeneity, u_{bb} (Section 4.1). The uncertainty related to stability during transport/long-term storage was estimated to be negligible. These different contributions were combined to estimate the expanded, relative uncertainty of the certified value ($U_{\text{CRM,rel}}$) with a coverage factor k as:

$$U_{\text{CRM,rel}} = k \cdot \sqrt{u_{\text{char,rel}}^2 + u_{\text{bb,rel}}^2} \quad \text{Equation 8}$$

- $u_{\text{char,rel}}$ was estimated as described in Section 6,
- $u_{\text{bb,rel}}$ was estimated as described in Section 4.1,

Because of the sufficient numbers of the degrees of freedom of the different uncertainty contributions, a coverage factor k of 2 was applied, to obtain the expanded uncertainties.

The certified values and their uncertainties are summarised in Table 14.

Table 14: Certified values and their uncertainties for ERM-EB075A, B and C

Analyte	Certified value [mg/kg] ¹⁾	$u_{\text{char, rel}}$ [%]	$u_{\text{bb, rel}}$ [%]	$U_{\text{CRM, rel}}$ [%]	U_{CRM} [mg/kg] ³⁾
Ag	10.8	1.42	2.00	4.91	0.6
Al	2.3	4.9	4.70	13.62	0.4
As	3.18	1.45	0.55	3.10	0.10
Au	1.46	4.32	1.70	9.29	0.14
Be	1.08	3.88	10.25	21.91	0.24
Bi	1.79	2.86	1.16	6.17	0.11
Cd	2.69	1.16	1.10	3.20	0.09
Co	2.64	1.39	0.58	3.02	0.08
Cr	1.40	2.30	0.58	4.75	0.07
Fe	9.3	1.87	0.79	4.06	0.4
In	1.83	2.21	1.46	5.29	0.10
Mg	7.0	2.91	3.97	9.84	0.7
Mn	1.35	2.10	1.20	4.84	0.07
Ni	2.18	1.76	3.01	6.97	0.16
P	2.59	5.31	2.19	11.49	0.30
Pb	4.8	2.02	9.09	18.62	0.9
S	25	6	1.19	12.78	4
Sb	2.93	1.96	1.20	4.60	0.14
Se	1.69	2.60	1.33	5.83	0.10
Si	2.6	6.99	1.93	14.51	0.4
Sn	2.13	2.35	0.91	5.04	0.11
Te	1.78	2.86	1.48	6.44	0.12
Ti	3.2	2.57	5.84	12.77	0.5
Zn	6.51	1.91	1.29	4.60	0.29

¹⁾rounded certified value

²⁾n.a.: not applicable

³⁾ Expanded ($k = 2$) and rounded uncertainty.

7.2 Indicative values and their uncertainties

Indicative values were assigned for Hg and W and Zr.

For the Hg and W mass fractions, all laboratories reported values below detection limits. Therefore, ERM-EB075 has been considered to contain less than the highest LOD reported by participating laboratories. With a 95 % confidence, the Hg and W mass fraction of the material is below 0.35 mg/kg and 0.1 mg/kg, respectively. The highest LOD reported by participating laboratories is, therefore, a conservative estimate of the indicative value.

For Zr, the fact that all neutron activation results are clustered on the high side of the population indicates that significant method differences might have been found if more datasets had been obtained. For this reason, only indicative values were assigned.

Indicative values may not be used as certified values. The uncertainty budgets were set up as for the certified values and are listed together with the assigned values in Table 15.

Table 15: Indicative values and their uncertainties for ERM-EB075A, B and C

Analyte	Indicative value ¹⁾ [mg/kg]
Hg	< 0.35 ²⁾
W	< 0.1 ²⁾
Zr	20 ± 5

¹⁾ rounded value

²⁾ With a 95 % probability, the indicative value is below this level.

³⁾ Expanded ($k = 2$) and rounded uncertainty.

7.3 Additional material information

The data provided in this section should be regarded as informative only on the general composition of the material and, in any case, cannot be used as certified or indicative value.

Three laboratories performed oxygen analysis on ERM-EB075A, B and C.

Two laboratories performed hydrogen analysis by fusion followed by thermal conductivity detection on 2 units of ERM-EB075A, B and C. The results are given as a range between the lowest highest result.

Table 16: Additional material information for ERM-EB075A, B and C. P: Number of laboratories

Analyte	Mass fraction [mg/kg]	p	Number of measurements	Method
O	1 – 6	3	48 results	See Annex C
H	0.3 - 4	2	36 results	IGF-conductivity

8 Metrological traceability and commutability

8.1 Metrological traceability

Identity

Ag, Al, As, Au, Be, Bi, Cd, Co, Cr, Fe, Hg, In, Mg, Mn, Ni, O, P, Pb, S, Sb, Se, Si, Sn, Te, Ti, W, Zn, Zr are clearly defined analytes. The participants used different methods for the sample preparation, as well as for the final determination, demonstrating absence of measurement bias. The measurand is therefore structurally defined and independent of the measurement method.

Quantity value

Only validated methods were used for the determination of the assigned values. Different calibrants/calibrants of (known purity and) specified traceability of their assigned values were used and all relevant input parameters were calibrated. The individual results are therefore traceable to the SI, as it is also confirmed by the agreement among the technically accepted datasets. As the assigned values are combinations of agreeing results individually traceable to the SI, the assigned quantity values themselves are traceable to the SI as well.

For Hg and W, the absence of the analyte at the level of the indicative value stated in Section 7.2 was reported using validated methods that report results traceable to SI, so the reported LOD are therefore traceable to SI.

8.2 Commutability

Many measurement procedures include one or more steps, which are selecting specific (or specific groups of) analytes from the sample for the subsequent steps of the whole measurement process. Often the complete identity of these 'intermediate analytes' is not fully known or taken into account. Therefore, it is difficult to mimic all the analytically relevant properties of real samples within a CRM. The degree of equivalence in the analytical behaviour of real samples and a CRM with respect to various measurement procedures (methods) is summarised in a concept called 'commutability of a reference material'. There are various definitions expressing this concept. For instance, the CSLI Guideline C-53A [18] recommends the use of the following definition for the term *commutability*:

"The equivalence of the mathematical relationships among the results of different measurement procedures for anRM and for representative samples of the type intended to be measured."

The commutability of a CRM defines its fitness for use and, thus, is a crucial characteristic in case of the application of different measurement methods. When commutability of a CRM is not established in such cases, the results from routinely used methods cannot be legitimately compared with the certified value to determine whether a bias does not exist in calibration, nor can the CRM be used as a calibrant.

It should be borne in mind that the methods used in the characterisation are methods routinely applied for measuring trace elements in electrolytic copper. The agreement of results from different methods demonstrates that the processing did not affect any properties relevant for these methods and that ERM-EB075A, B and C behave like a real sample.

ERM-EB075A, B and C were produced from pure copper with added impurities. Therefore, the analytical behaviour will be the same as for a routine sample of electrolytic copper. For samples other than electrolytic copper, the commutability has to be assessed.

9 Instructions for use

9.1 Safety information

The usual laboratory safety measures apply.

9.2 Storage conditions

The materials shall be stored at 18 °C ± 5 °C in the dark.

Please note that the European Commission cannot be held responsible for changes that happen during storage of the material at the customer's premises, especially of opened units/bottles.

9.3 Preparation and use of the material

For ERM-EB075A and B, the usual mechanical cleaning should be applied prior to the measurement (the CRM and the user's samples should be treated in the same way).

For ERM-EB075C, it is recommended to clean the chips chemically before use to remove any traces of oxidation.

9.4 Minimum sample intake

For ERM-EB075A, B and C, the minimum amount of sample to be used is 10 mg for Ag, Al, As, Be, Bi, Cd, Co, Cr, Fe, Mg, Mn, Ni, P, Pb, S, Sb, Se, Si, Sn, Ti, Zn; 20 mg for Au, In and Te.

9.5 Use of the certified value

The main purpose of these materials is to assess method performance, i.e. for checking the accuracy of analytical results/calibration. As any reference material, it/they can also be used for control charts or validation studies.

Use as a calibrant

It is not recommended to use this matrix material as calibrant. If used, nevertheless, the uncertainty of the certified value will be taken into account in the estimation of the measurement uncertainty.

Comparing an analytical result with the certified value

A result is unbiased if the combined standard uncertainty of measurement and certified value covers the difference between the certified value and the measurement result (see also ERM Application Note 1, www.erm-crm.org[19]).

For assessing the method performance, the measured values of the CRMs are compared with the certified values. The procedure is described here in brief:

- Calculate the absolute difference between mean measured value and the certified value (Δ_{meas}).
- Combine measurement uncertainty (u_{meas}) with the uncertainty of the certified value (u_{CRM}): $u_{\Delta} = \sqrt{u_{\text{meas}}^2 + u_{\text{CRM}}^2}$
- Calculate the expanded uncertainty (U_{Δ}) from the combined uncertainty (u_{Δ}) using an appropriate coverage factor, corresponding to a level of confidence of approximately 95 %
- If $\Delta_{\text{meas}} \leq U_{\Delta}$ no significant difference between the measurement result and the certified value, at a confidence level of about 95 % exists.

Use of quality control charts

The materials can be used for quality control charts. Different CRM units will give the same result as inhomogeneity was included in the uncertainties of the certified values.

10 Acknowledgments

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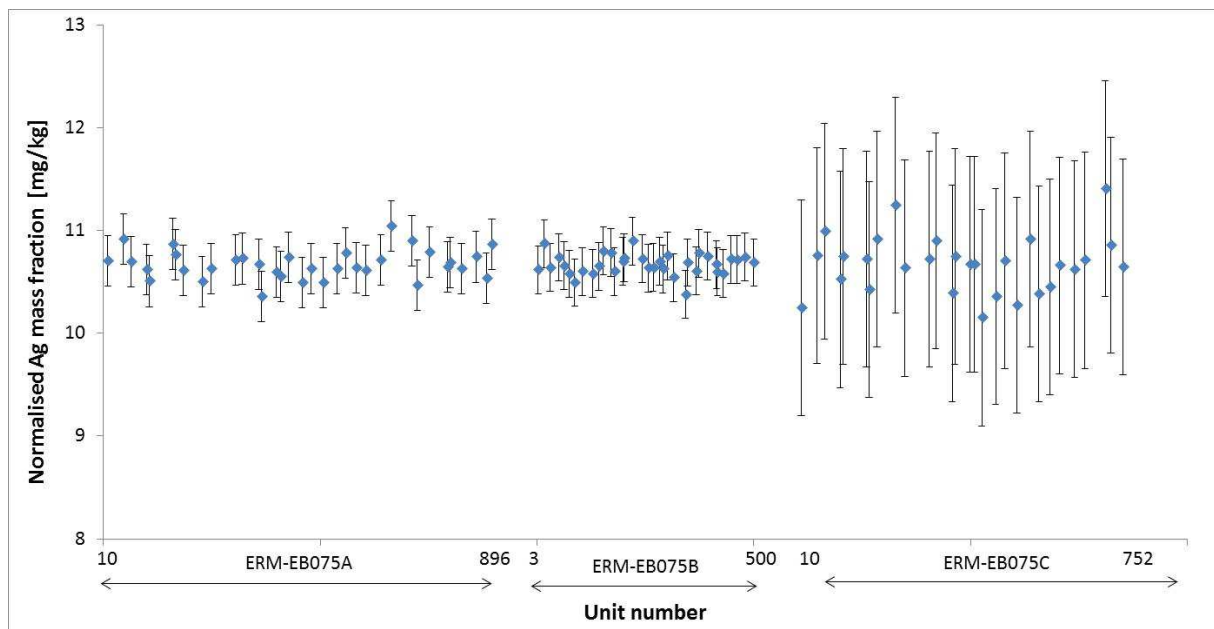
Annexes

Annex A: Results of the between-unit homogeneity measurements

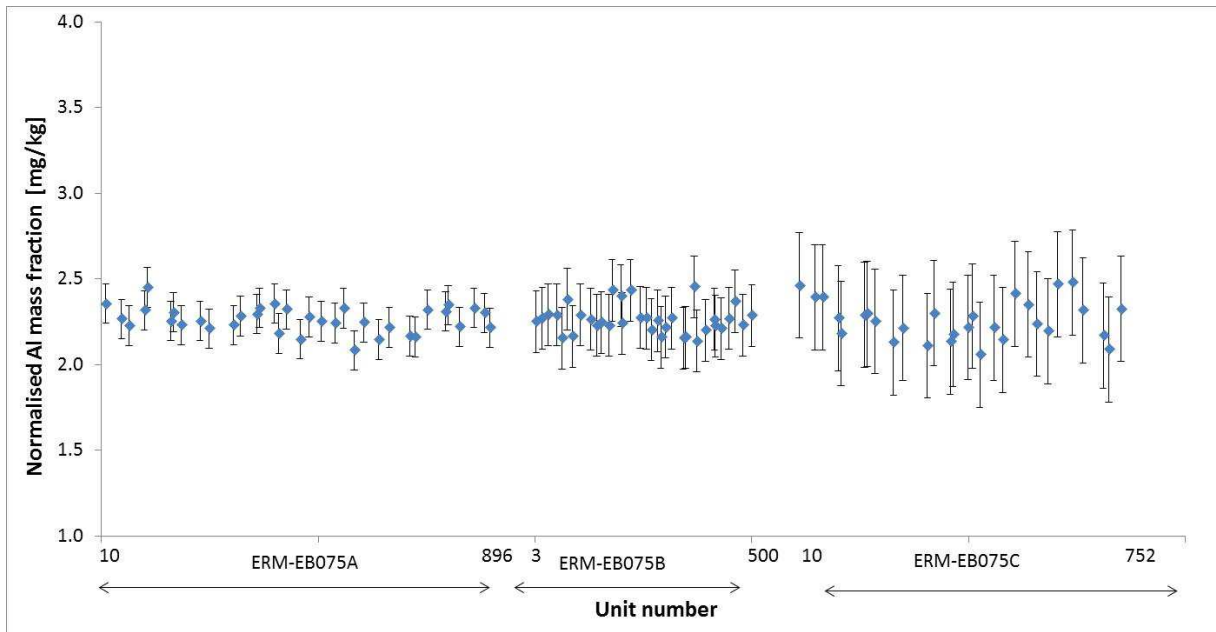
Results of homogeneity studies for the three formats ERM-EB075A, B and C. The studies were not performed using the same technique (Table 2) which may explain the variance difference between the three formats. The unit means are presented after correction for analytical trend and normalisation to mean of the three formats means. The associated uncertainty equals to s_{wb} from ANOVA for all units of each study. The distance between tick,marks is 500 units.

The within-billet homogeneity study was performed using the first nine units of ERM-EB075A.

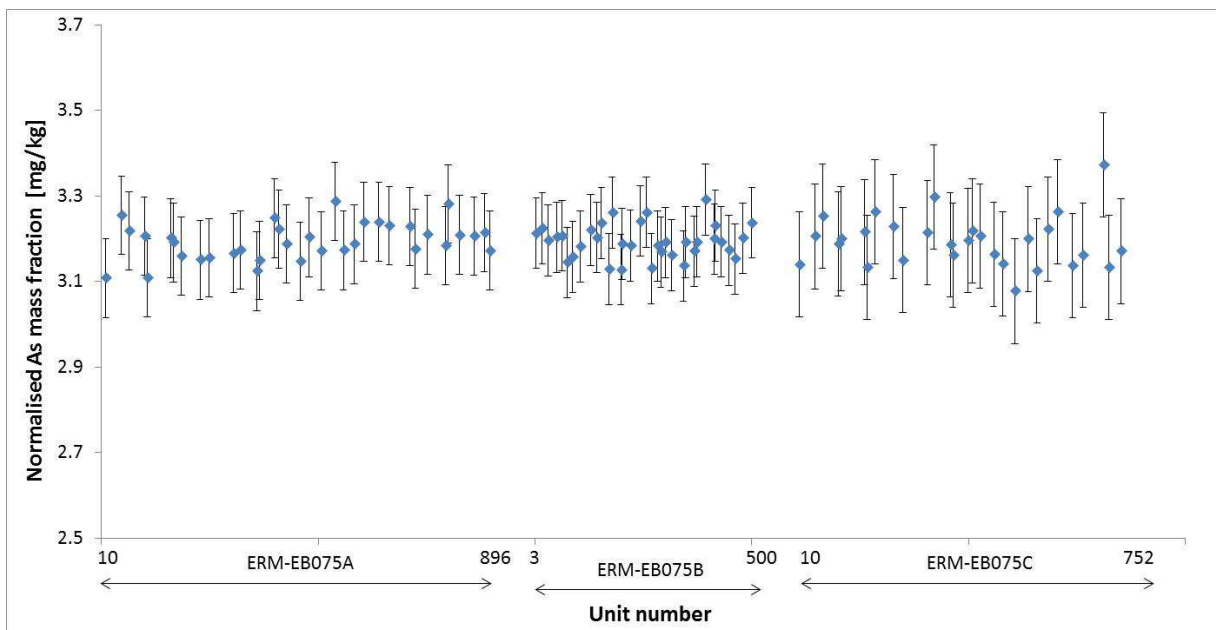
Ag (Silver)



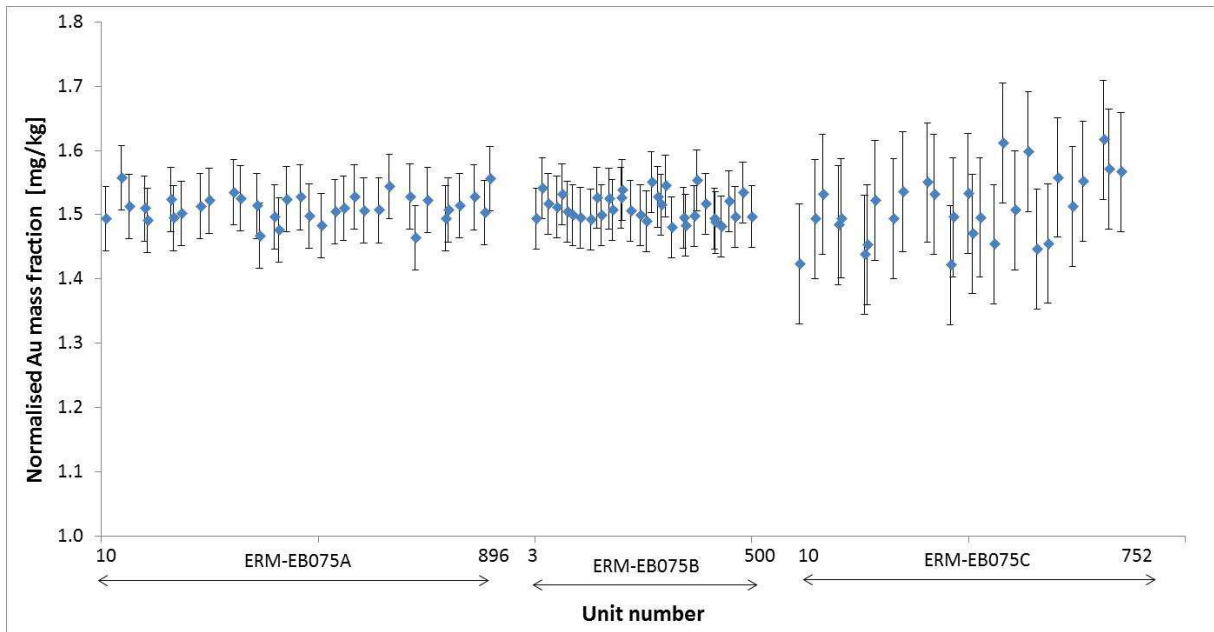
Al (Aluminium)



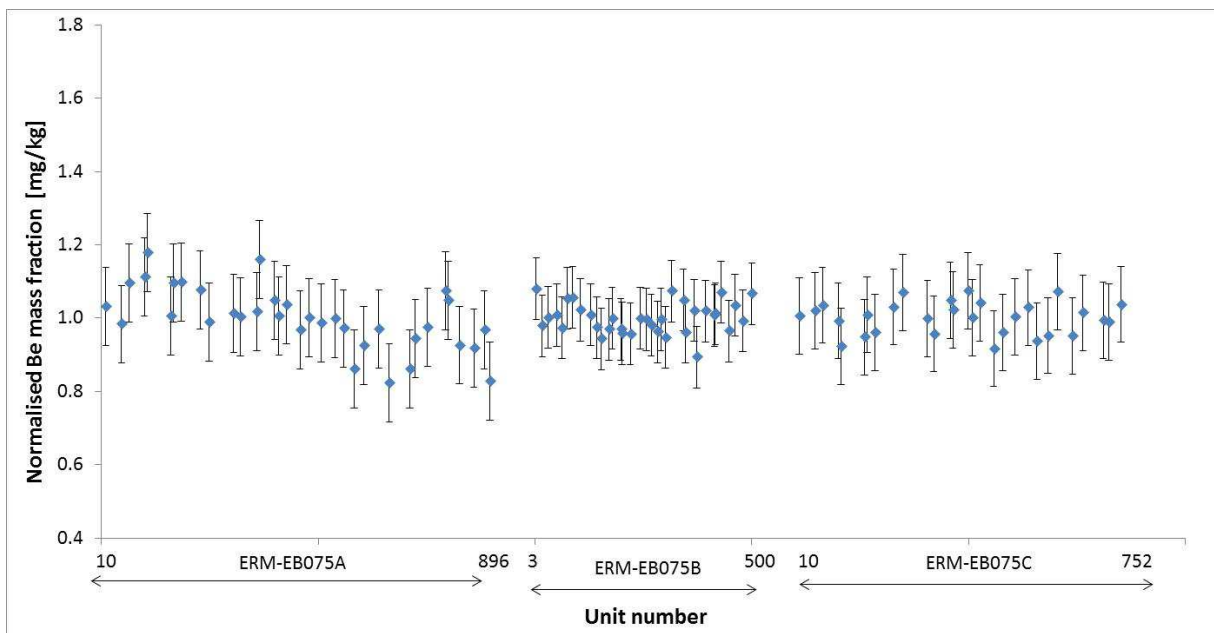
As (Arsenic)



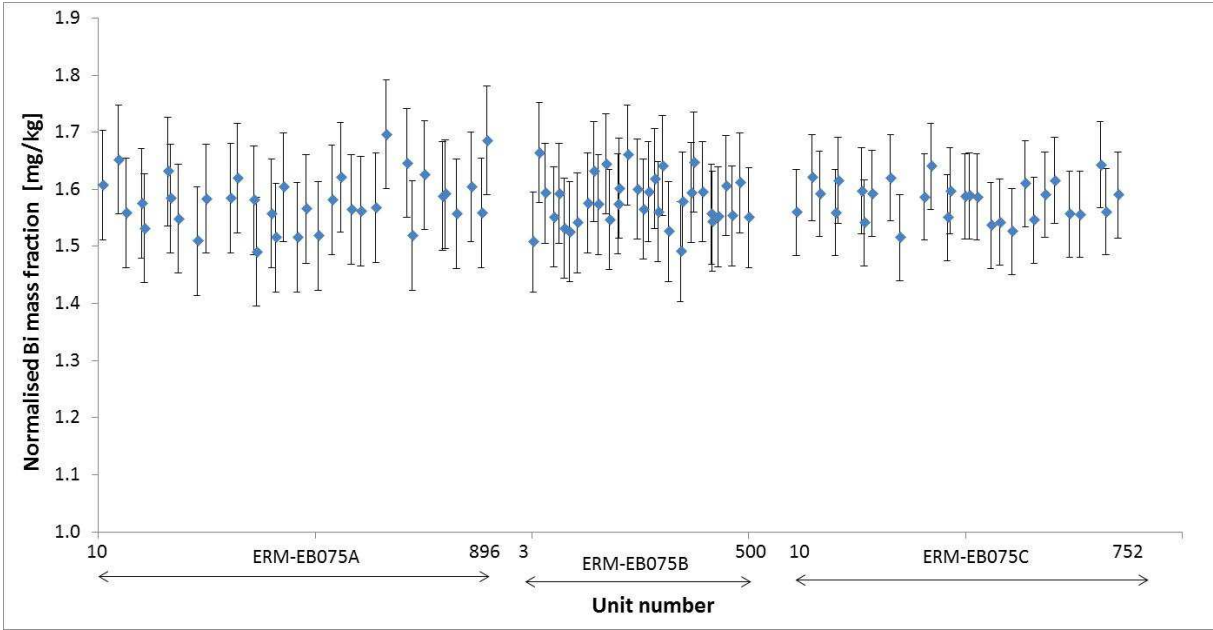
Au (Gold)



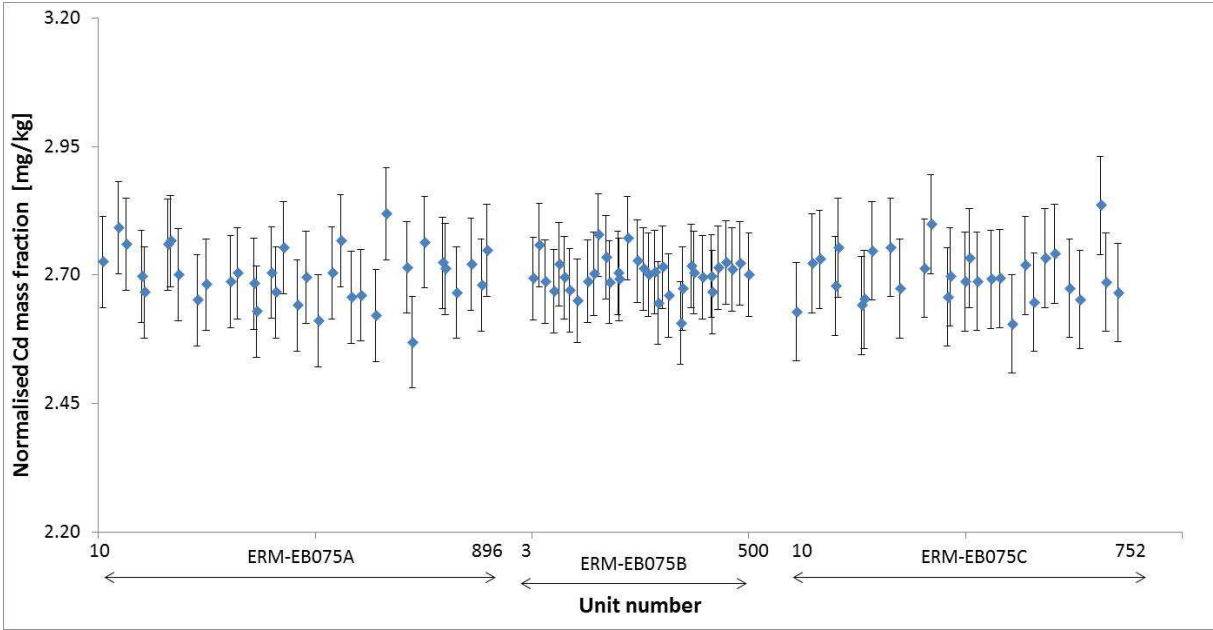
Be (Beryllium)



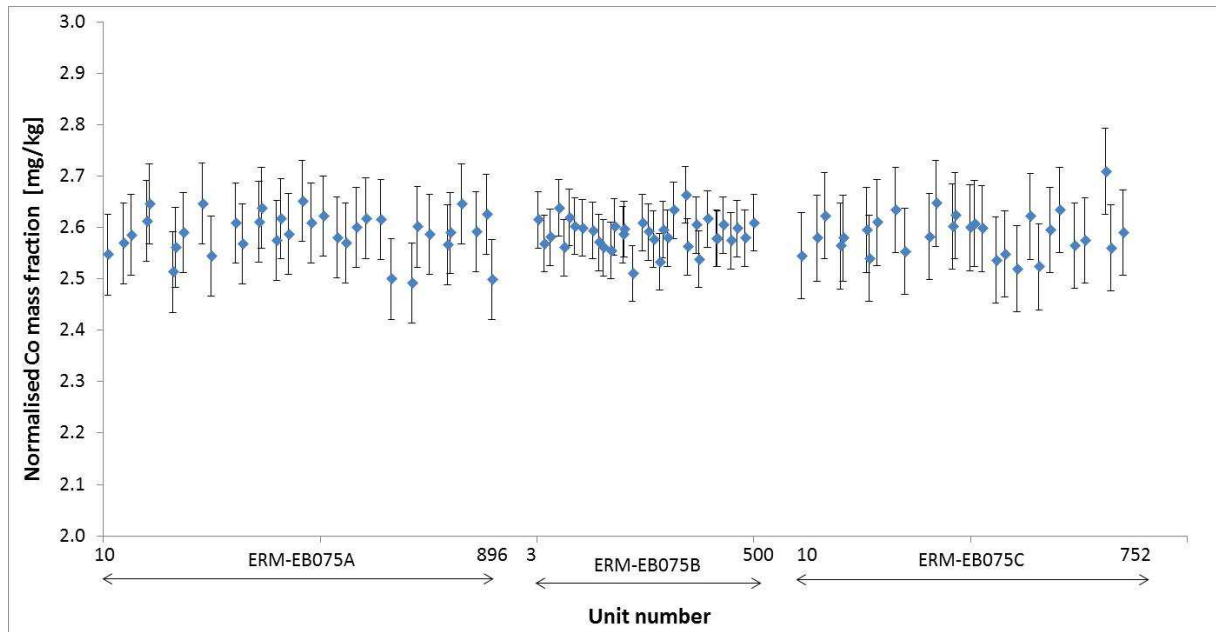
Bi (Bismuth)



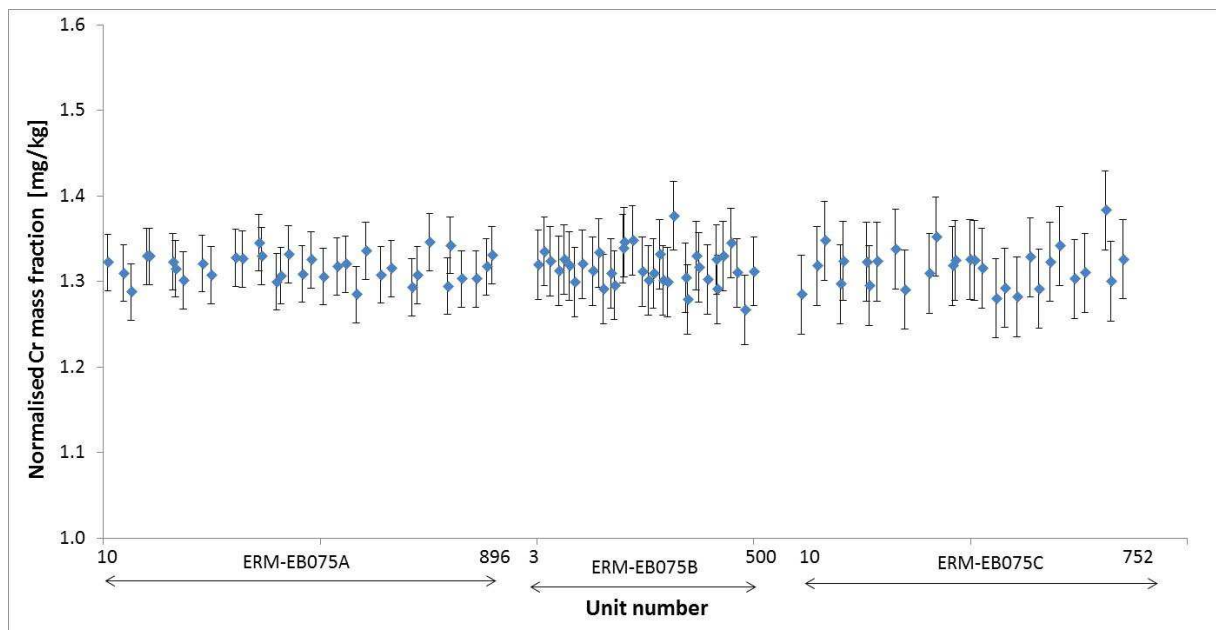
Cd (Cadmium)



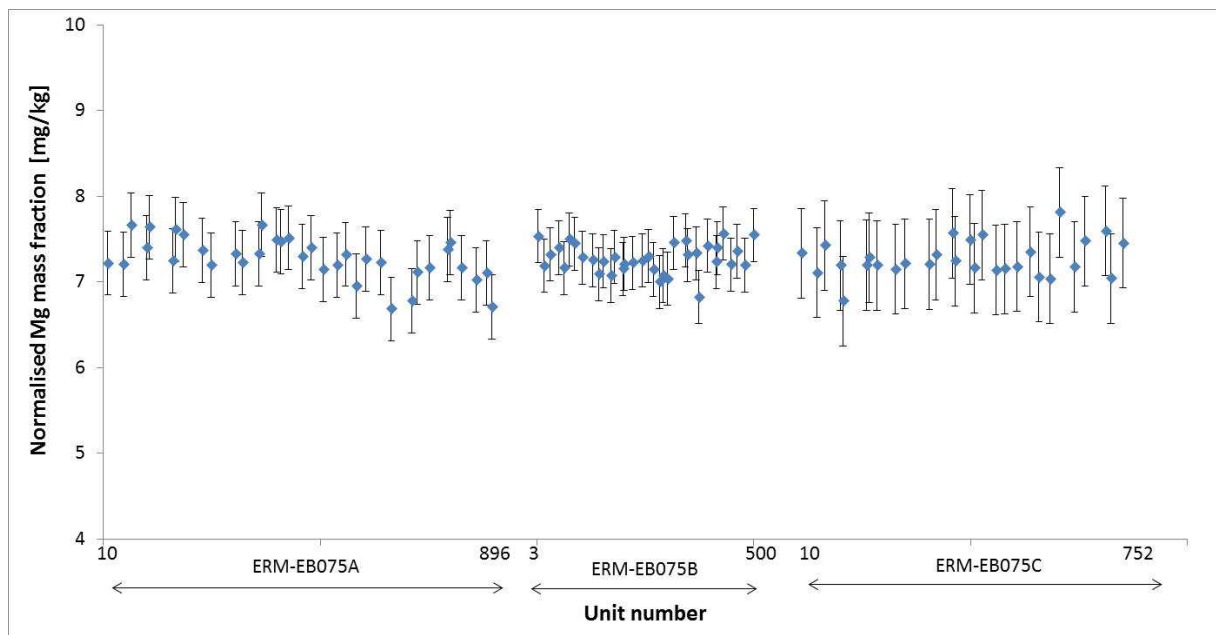
Co (Cobalt)



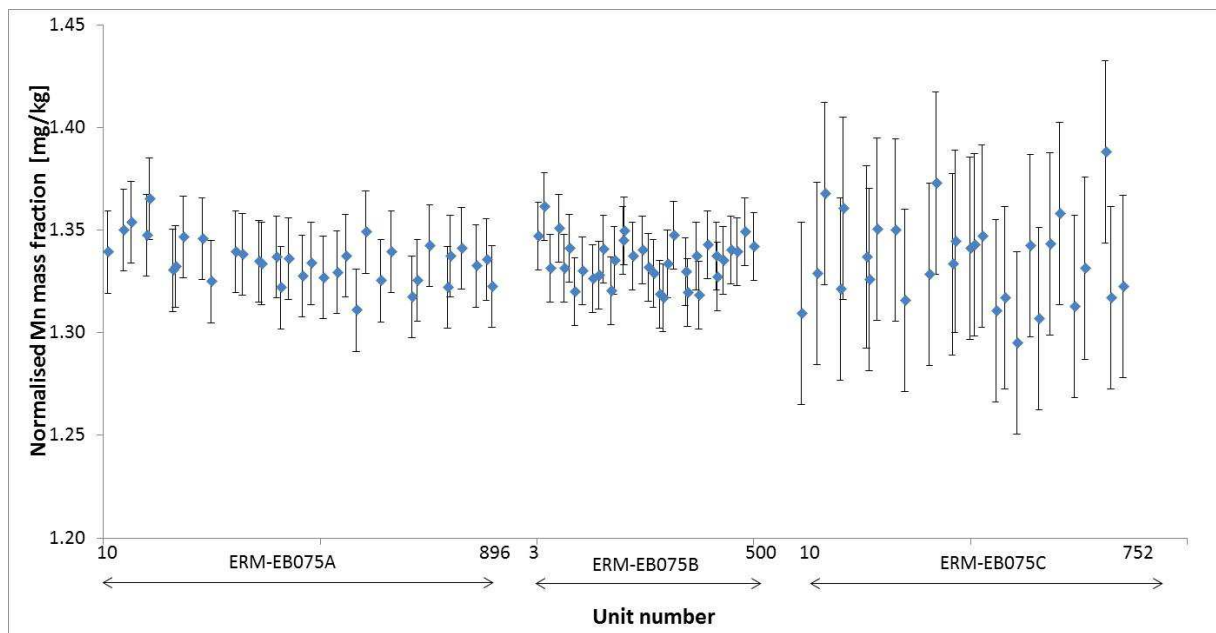
Cr (Chromium)



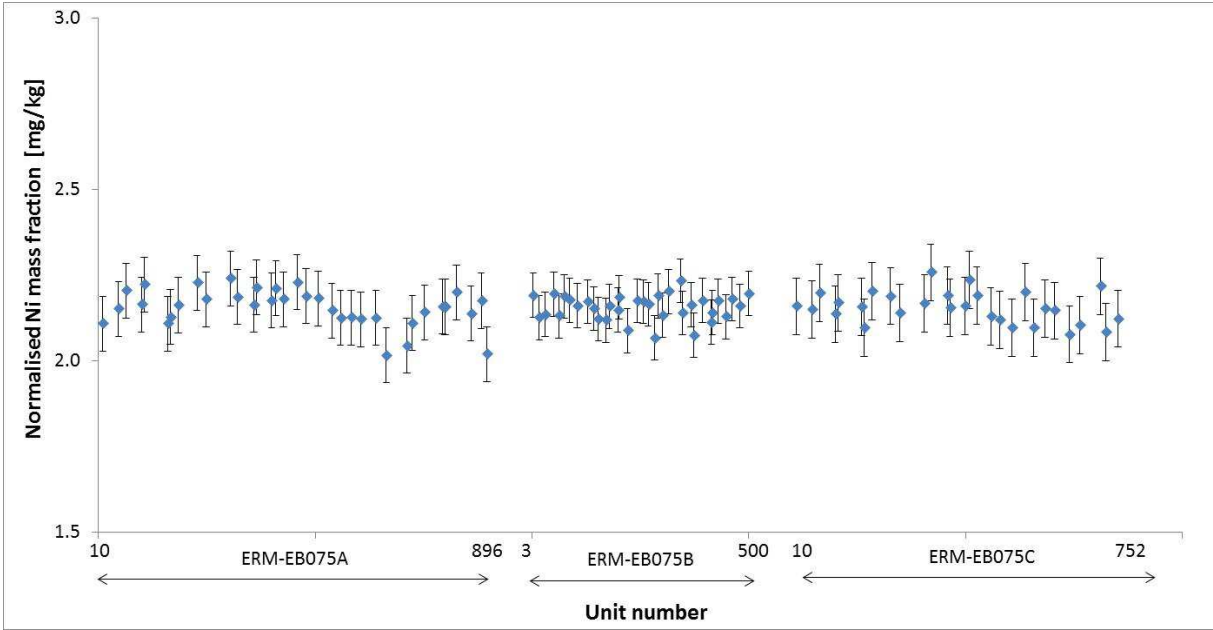
Mg (Magnesium)



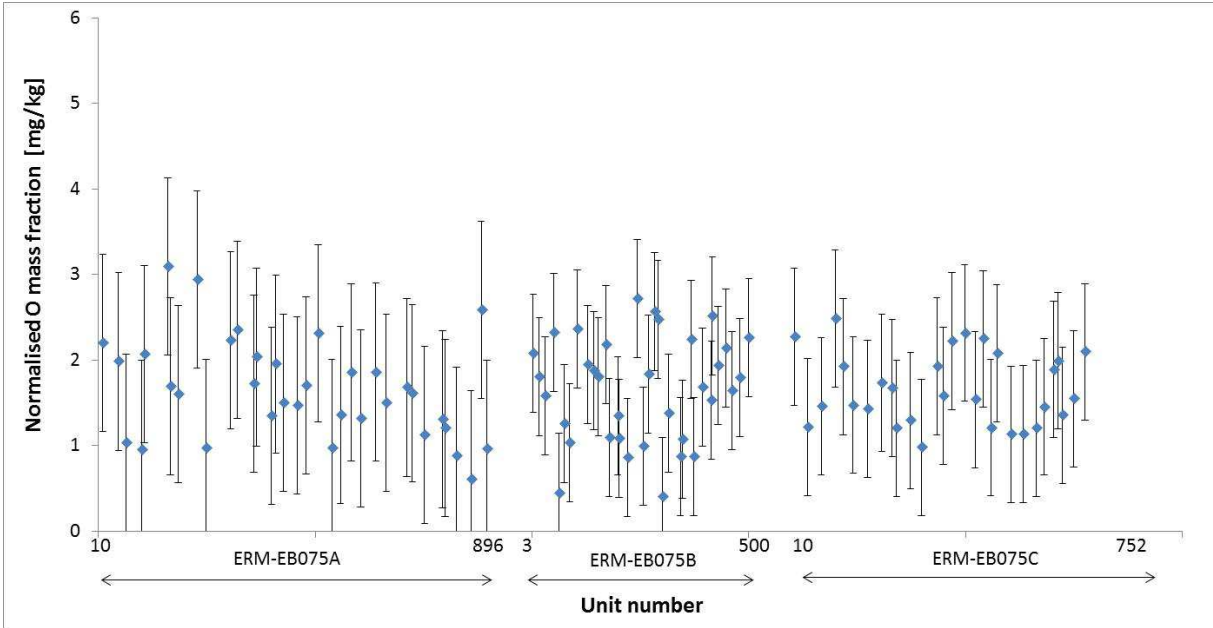
Mn (Manganese)



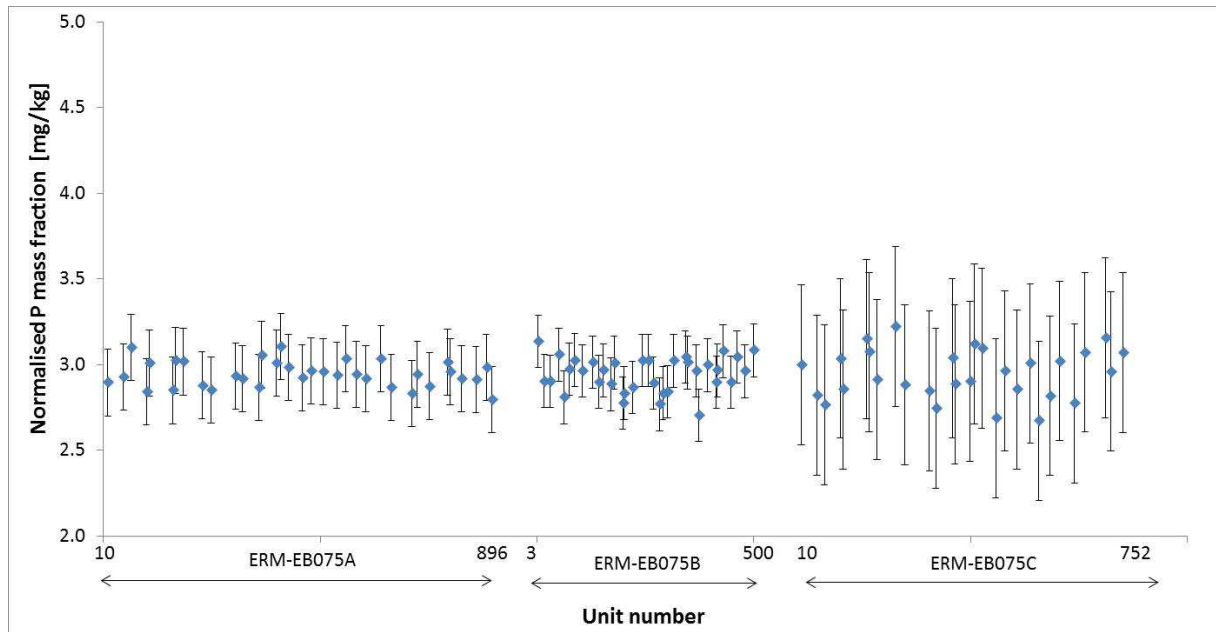
Ni (Nickel)



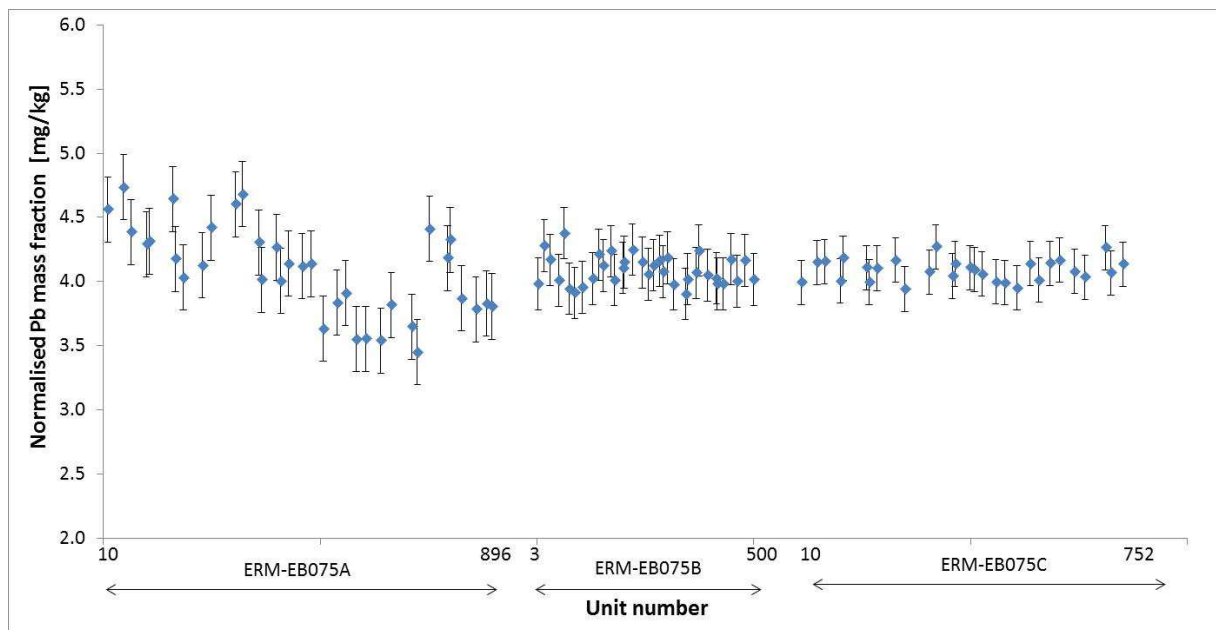
O (Oxygen)



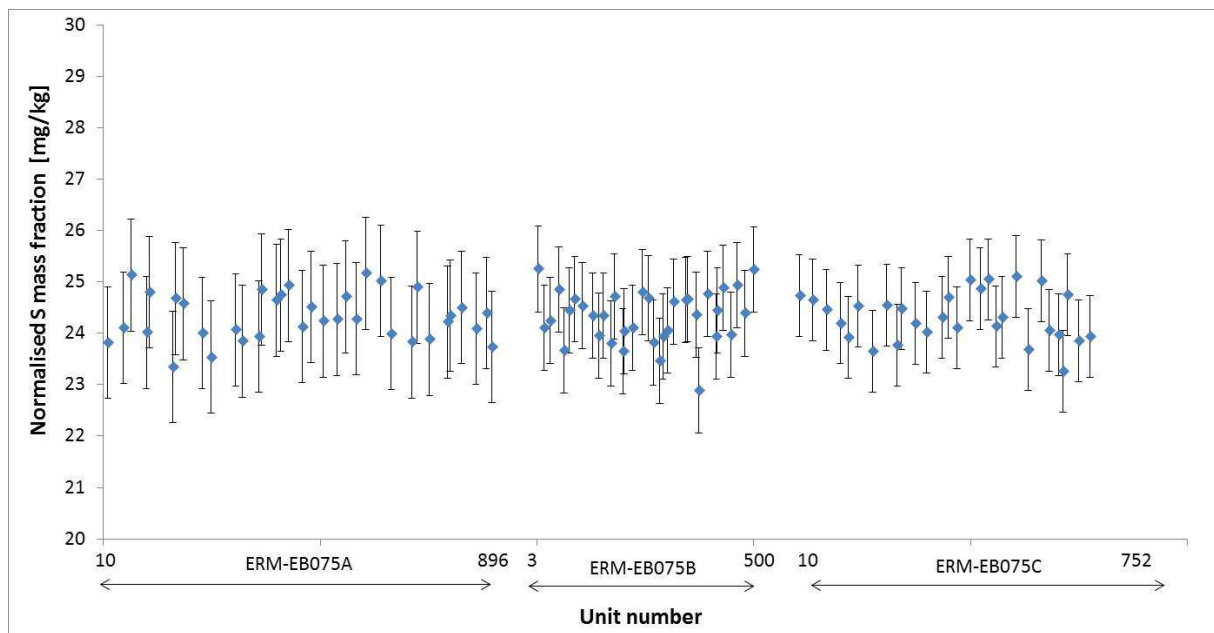
P (Phosphorus)



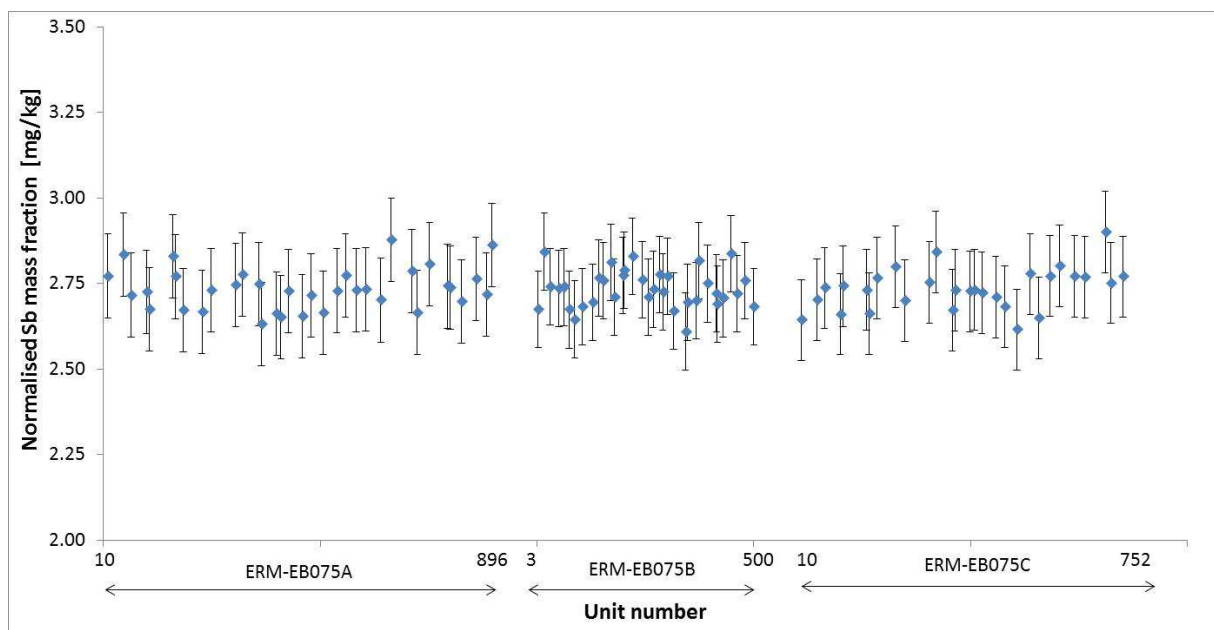
Pb (Lead)



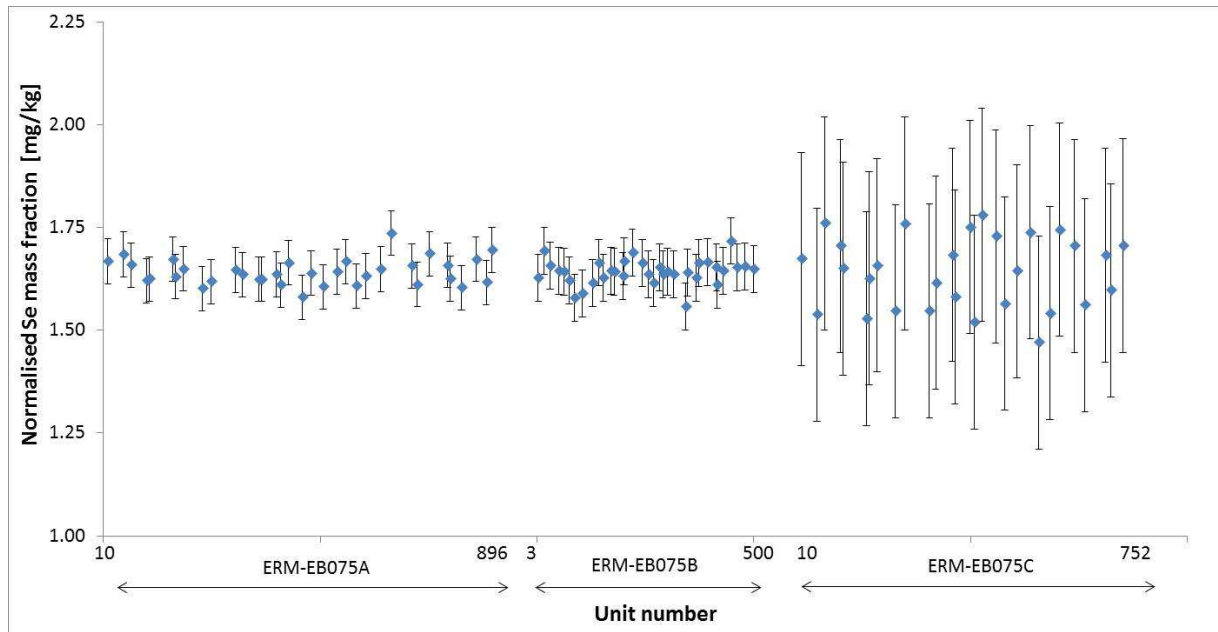
S (Sulfur)



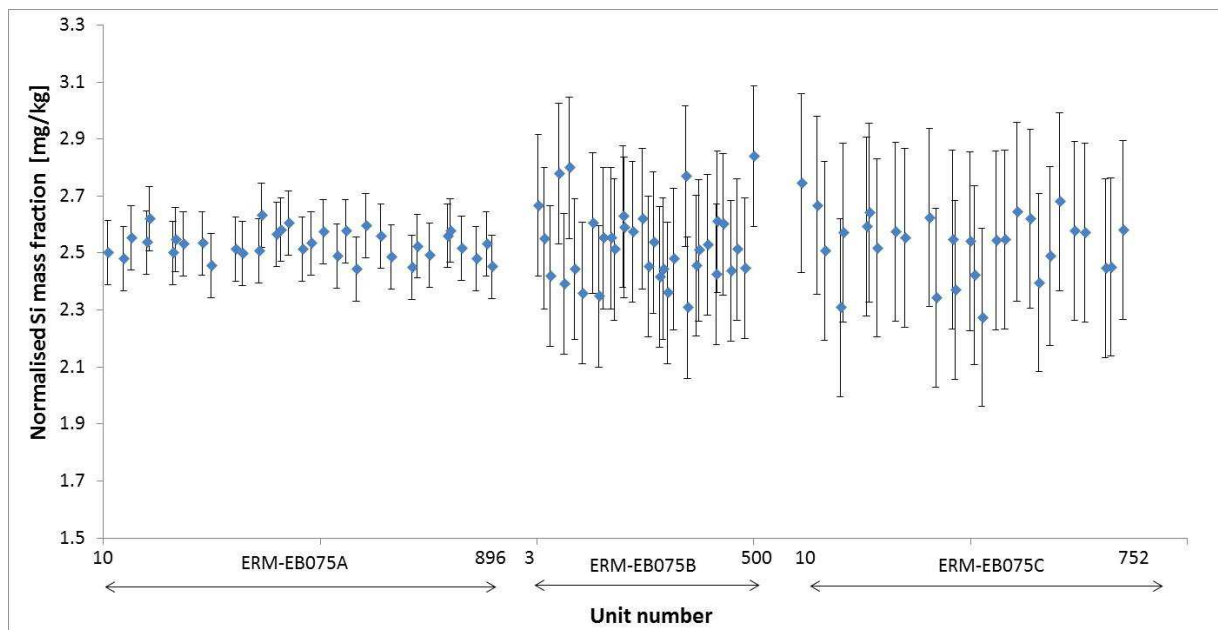
Sb (Antimony)



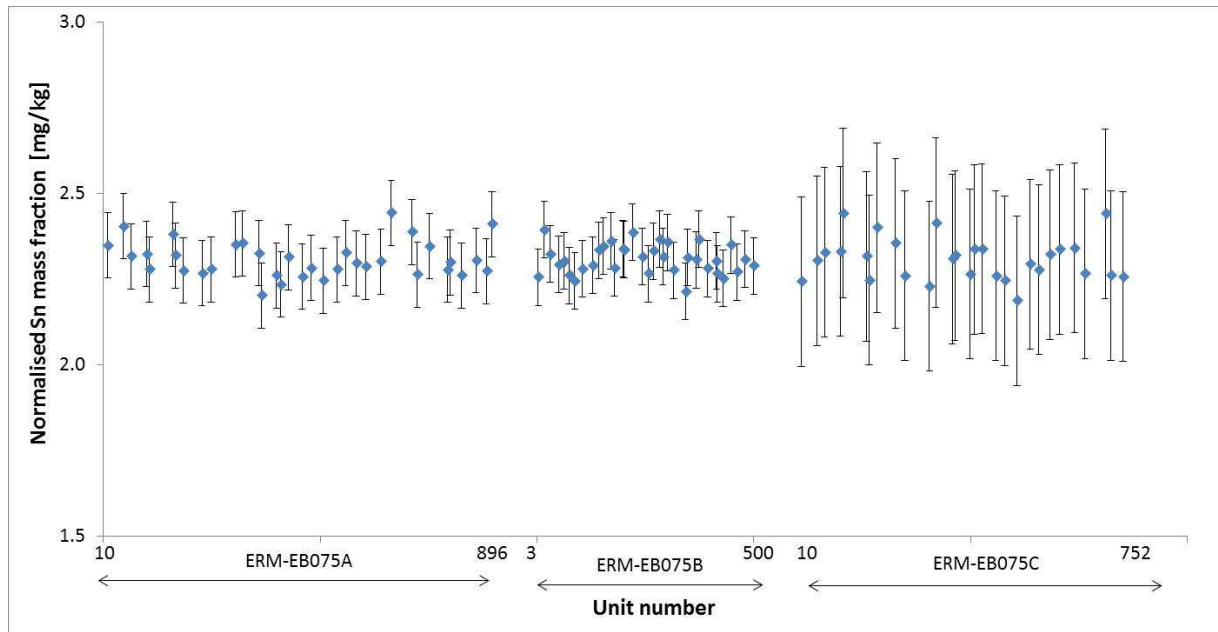
Se (Selenium)



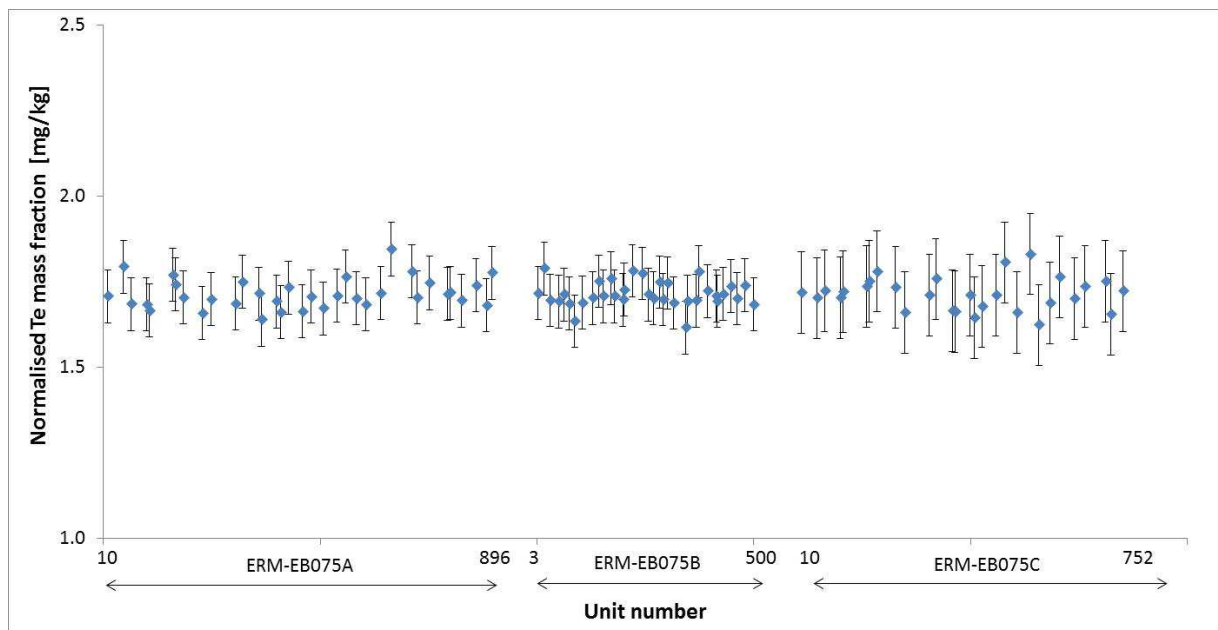
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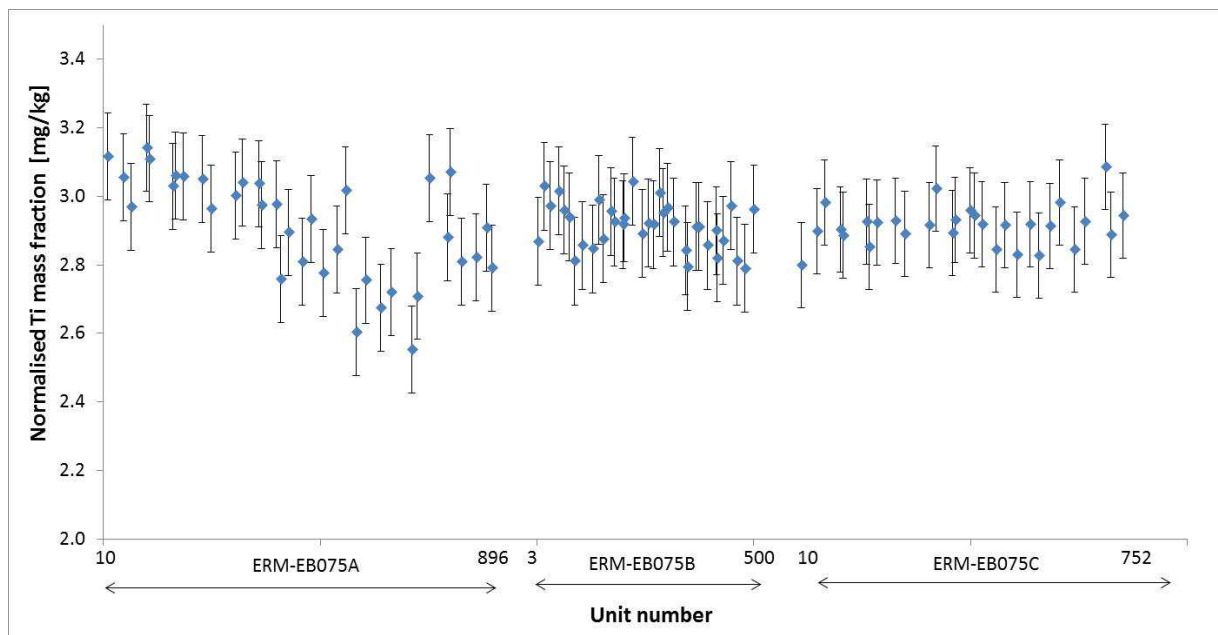
Sn (Tin)



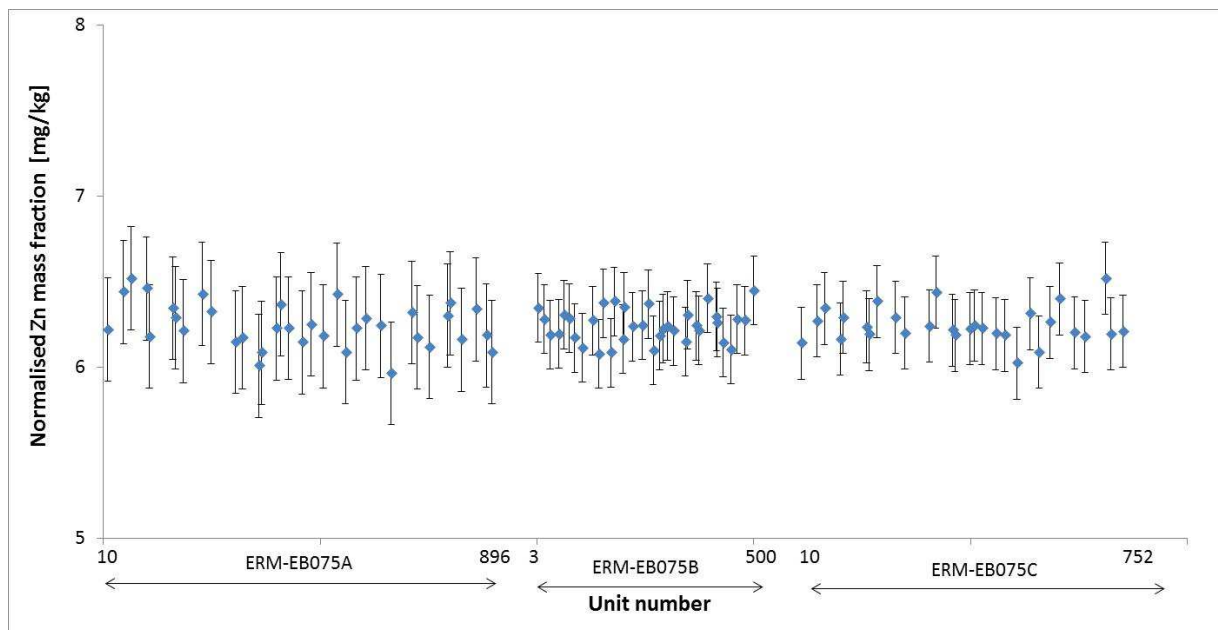
Te (Tellurium)



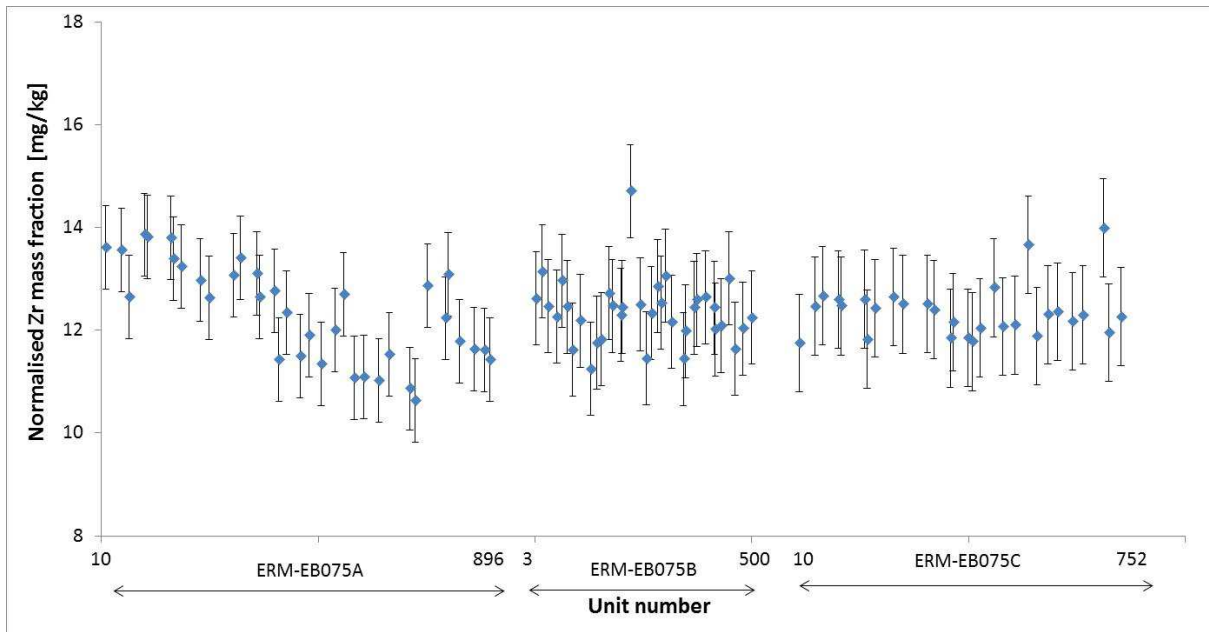
Ti (Titanium)



Zn (Zinc)



Zr (Zirconium)



Annex B: Results of the minimum sample intake measurements

The results presented in the Table correspond to the mean of each area on the four different disc faces analysed of ERM-EB075A. The standard deviation reported is the standard deviation between the results of the four disc faces. The measurements were performed by spark-OES except for Au, In and Te which were tested by GD-MS.

Analyte	Central area		Intermediate area		External area	
	Corrected / Normalised results [mg/kg]	Standard deviation of the measurements [mg/kg]	Corrected / Normalised results [mg/kg]	Standard deviation of the measurements [mg/kg]	Corrected / Normalised results [mg/kg]	Standard deviation of the measurements [mg/kg]
Ag	10.534	0.246	10.691	0.192	10.568	0.116
Al	2.538	0.036	2.558	0.034	2.585	0.038
As	3.528	0.108	3.520	0.048	3.522	0.005
Au ¹⁾	0.995	0.012	n.a.	n.a.	1.001	0.005
Be	1.595	0.028	1.608	0.011	1.625	0.019
Bi	1.668	0.058	1.751	0.105	1.638	0.097
Cd	2.930	0.045	2.983	0.034	2.978	0.027
Co	1.474	0.038	1.498	0.035	1.462	0.034
Cr	2.772	0.120	2.758	0.073	2.697	0.099
Fe	9.390	0.156	9.511	0.053	9.619	0.077
In ¹⁾	0.976	0.019	n.a.	n.a.	1.004	0.011
Mg	7.336	0.174	7.415	0.080	7.512	0.086
Mn	1.396	0.008	1.405	0.003	1.407	0.002
Ni	2.083	0.049	2.063	0.025	2.048	0.036
P	2.735	0.073	2.725	0.039	2.703	0.032
Pb	5.220	0.269	5.502	0.239	5.103	0.266
S	25.319	0.860	25.905	0.697	26.920	0.888
Sb	3.270	0.070	3.321	0.125	3.365	0.047
Se	1.816	0.013	1.848	0.026	1.876	0.023
Si	3.354	0.175	3.535	0.143	3.511	0.160
Sn	2.788	0.194	2.879	0.093	2.895	0.188
Te ¹⁾	0.971	0.039	n.a.	n.a.	1.005	0.014
Ti	3.371	0.108	3.494	0.162	3.487	0.084
Zn	6.229	0.131	6.429	0.058	6.380	0.062
Zr	18.583	0.898	18.893	0.576	19.643	0.825

¹⁾ element analysed by GD-MS, the analysis area did not allow to perform measurements in the intermediate area.

Annex C: Summary of methods used in the characterisation study

Laboratory code: L1

Elements	Technique	Sample mass [g]	Sample preparation	Calibration	Instrumentation
Ag, Al, As, Be, Bi, Cd, Co, Cr, In, Mg, Mn, Ni, Pb, Sn, Te, Ti, Zn, Zr	ICP-MS	1	Hot acid digestion in HNO ₃	external calibration, prepared from certified standard solutions Inorganic Venture	ELAN and Agilent 7700x
Cu	Titration	0.5 - 1	n.a.	Pure Cu shot from Alfa Aesar	Metrohm
Fe, P, Sb	ICP-OES	1	Hot acid digestion in HNO ₃	external calibration, prepared from certified standard solutions Inorganic Venture	Agilent 735-ES
Au and Se	INAA	1	n.a.	external calibration, prepared from certified standard solutions Inorganic Venture	Canberra Ge Detector GC1318
Hg	CV-AAS	1	n.a.	external calibration, prepared from certified standard solutions ISOSpec	CETAC M-7600
O, H	IGF-IR	1	n.a.	RM from LECO and Alfa Aesar	Eltra ONH-2000
S	C-IR	1	n.a.	using metal RM from Alpha	Leco CS-200

Laboratory code: L2

Elements	Technique	Sample mass [g]	Sample preparation	Calibration	Instrumentation
Ag, Al, As, Be, Bi, Cd, Co, Cr, Fe, In, Mg, Mn, Ni, P, Pb, Sb, Se, Sn, Te, Ti, Zn, Zr	ICP-OES	4	Hot acid digestion in HNO ₃ and HF	external calibration, prepared from certified standard solutions	Spectro (Arcos) and Perkin Elmer (5300DV)
Cu	electrogravimetry	2	Dissolution in HNO ₃		
S	spark-OES	n.a.	n.a.	23 copper standards RM and CRM including BAM-M-381-386 and BCR-074 and BCR-075	Spectro Lab M9

Laboratory code: L3

Elements	Technique	Sample mass [g]	Sample preparation	Calibration	Instrumentation
As, Bi, Ni, Pb, Sb, Se, Sn, Te	ETV-ICP-OES	0.02	n.a.	4 copper standards RM and CRM including BCR-074 and BCR-075	Spectro Arcos-EOP

Laboratory code: L4

Elements	Technique	Sample mass [g]	Sample preparation	Calibration	Instrumentation
Ag, Al, As, Be, Bi, Cd, Co, Cr, Fe, In, Mg, Mn, Ni, P, Pb, S, Sb, Se, Sn, Te, Ti, W, Zn, Zr	ICP-MS	1	Hot acid digestion in HNO ₃ and HF	external calibration, prepared from certified standard solutions from Merck, Alfa Aesar and Bernd Kraft	Thermo Element XR

Laboratory code: L5

Elements	Technique	Sample mass [g]	Sample preparation	Calibration	Instrumentation
Ag, Al, As, Be, Bi, Cd, Co, Cr, Fe, In, Mg, Mn, Ni, P, Pb, S, Sb, Se, Si, Sn, Te, Ti, W, Zn, Zr	GD-MS	n.a.	n.a.	Solid calibrants prepared from certified standard solutions from Merck, Alfa Aesar and Bernd Kraft	Thermo Element GD

Laboratory code: L6

Elements	Technique	Sample mass [g]	Sample preparation	Calibration	Instrumentation
As, Be, Bi, Fe, Pb, Sb, Se, Sn, Te	ICP-MS	2.5	Acid digestion/Yttrium collection to remove Cu matrix	High Purity certified stds	Perkin-Elmer Elan DRCII
Ag, Cd, Co, Cr, Mn, Ni, P, Si, Zn	ICP-OES	2.5	Acid digestion	SCP PlasmaCal certified stds	Varian 735-ES

Laboratory code: L7

Elements	Technique	Sample mass [g]	Sample preparation	Calibration	Instrumentation
Ag, As, Bi, Fe, Ni, Pb, S, Sb, Se, Sn, Te, Zn	DC-arc-OES	0.3	n.a.	NIST and Leco for sulfur Copper Spec for all others	Teledyne Leeman Prodigy

Laboratory code: L8

Elements	Technique	Sample mass [g]	Sample preparation	Calibration	Instrumentation
Ag, Al, As, Be, Bi, Cd, Co, Cr, Fe, Hg, In, Mg, Mn, Ni, P, Pb, Sb, Se, Sn, Te, Ti, W, Zn, Zr	ICP-MS	0.1	Acid digestion with HNO ₃	external calibration, prepared from certified standard solutions from Inorganic Venture	Perkin Elmer DRC II

Laboratory code: L9

Elements	Technique	Sample mass [g]	Sample preparation	Calibration	Instrumentation
Ag, Al, As, Be, Bi, Cd, Co, Cr, Fe, Hg, In, Mg, Mn, Ni, P, Pb, Sb, Se, Sn, Te, Ti, W, Zn, Zr	ICP-MS	0.1	Acid digestion with HNO ₃	external calibration, prepared from certified standard solutions from Inorganic Venture	Perkin Elmer Elan 9000
O	IGF-IR	1	n.a.	external calibration with RM from Alpha Resources and LECO	LECO TC-436
H	IGF-conductivity	1	n.a.	external calibration with RM from Alpha Resources and LECO	LECO TC-436
S	C-IR	1	n.a.	external calibration with RM from Alpha Resources and LECO	Analyseur C/S Horiba EMIA 820V

Laboratory code: L10

Elements	Technique	Sample mass [g]	Sample preparation	Calibration	Instrumentation
Ag, As, Au, Co, Cr, Cu, Fe, Hg, In, Sb, Se, Te, Zn	k_0 neutron activation analysis	0.2	n.a.	IRMM-530R	250 kW TRIGA Mark II reactor (GA), HPGe detector with 40 % relative efficiency (Canberra)

Laboratory code: L11

Elements	Technique	Sample mass [g]	Sample preparation	Calibration	Instrumentation
Ag, Al, As, Be, Bi, Cd, Co, Cr, Hg, In, Mg, Mn, Ni, Pb, S, Sb, Se, Sn, Te, Ti, W, Zn, Zr	ICP-MS	0.1	Acid digestion with HNO ₃	external calibration, prepared from certified standard solutions from VHG	THERMO iCAP QC
Au	ICP-MS	0.1	Acid digestion with HNO ₃ and HCl	external calibration, prepared from certified standard solutions from VHG	THERMO iCAP QC
Fe, Mn, P, Si	ICP-OES	0.5	Acid digestion with HNO ₃	external calibration, prepared from certified standard solutions from VHG	SPECTRO ARCOS
O, S, H	E1019	1	n.a.	external calibration with RM from Alpha Resources and LECO	LECO

Laboratory code: L12

Elements	Technique	Sample mass [g]	Sample preparation	Calibration	Instrumentation
Ag, Bi, Cd, Co, In, Pb	ICP-MS	0.25	Acid digestion with HNO ₃	external calibration, prepared from pure metal, KRISS CRM and from certified standard solutions from Perkin Elmer	Perkin-Elmer NEXION 300
Cr, Fe, Ni, P, Zn	ICP-MS	0.05	Acid digestion with HNO ₃	external calibration, prepared from pure metal, KRISS CRM and from certified standard solutions from Perkin Elmer	Thermo Fisher ELEMENT XR magnetic sector field ICP mass spectrometer

Laboratory code: L13

Elements	Technique	Sample mass [g]	Sample preparation	Calibration	Instrumentation
Ag, Al, As, Bi, Cd, Co, Cr, Fe, Mg, Mn, Ni, O, P, Pb, S, Sb, Se, Sn, Te, Ti, Zn	GD-MS	n.a.	n.a.	external calibration using certified reference materials (BCR-075, BCR-022, SRM-494, BAM M382 and BAM M386)	VG9000

Laboratory code: L14

Elements	Technique	Sample mass [g]	Sample preparation	Calibration	Instrumentation
Ag, As, Au, Co, Cr, Cu, Fe, In, Sb, Se, Te, Zn, Zr	k_0 neutron activation analysis	0.2	n.a.	IRMM-530R	Samples irradiated in reactor and measured with HPGe detectors

Laboratory code: L15

Elements	Technique	Sample mass [g]	Sample preparation	Calibration	Instrumentation
Ag, Au, Co, Cr, Fe, Sb, Se, Zn, Zr	Instrumental neutron activation analysis	0.2	n.a.	Calibration using SRM from NIST	Samples irradiated in nuclear reactor Detector: 2 HPGe coaxial

Laboratory code: L16

Elements	Technique	Sample mass [g]	Sample preparation	Calibration	Instrumentation
As, Au, Bi, Cd, Cr, Hg, In, Ni, P, Pb, S, Sb, Se, Sn, Te, Ti, W, Zn	ICP - MS	2	Digestion with HCl and HNO ₃	external calibration, prepared from certified standard solutions from Merck	Thermo iCAP Q
Ag, Al, Be, Co, Fe, Mg, Mn, Si, Zr	ICP-OES	2	Digestion with HCl and HNO ₃	external calibration, prepared from certified standard solutions from Merck	Varian 725ES

Laboratory code: L17

Elements	Technique	Sample mass [g]	Sample preparation	Calibration	Instrumentation
As, Au, Be, Bi, Cd, Co, Cr, Fe, Hg, In, Mg, Mn, Ni, Pb, Sb, Se, Sn, Te, Ti, W, Zn, Zr	ICP - MS	2	Digestion with HCl and HNO ₃	external calibration, prepared from certified standard solutions from Merck	Agilent 7500cx
Ag	ICP - MS	0.5	Digestion with HNO ₃	external calibration, prepared from certified standard solutions from Merck	Agilent 7500cx
Al, P, S, Si	ICP-OES	5	Digestion with HCl and HNO ₃	external calibration, prepared from certified standard solutions from Merck and Alfa Aesar	Varian 730-ES
O	C-IR	1	n.a.	LECO RM	Leco TC600

Laboratory code: L18

Elements	Technique	Sample mass [g]	Sample preparation	Calibration	Instrumentation
Ag, Al, As, Au, Be, Bi, Cd, Co, Cr, Fe, Hg, In, Mg, Mn, Ni, Pb, Sb, Se, Sn, Te, Ti, W, Zn, Zr	ICP-MS	0.2	Hot acid digestion with HNO ₃	external calibration, prepared from certified standard solutions from Inorganic Ventures	Agilent technologies AT 7500 CCT

Laboratory code: L19

Elements	Technique	Sample mass [g]	Sample preparation	Calibration	Instrumentation
Ag, Al, As, Au, Bi, Cd, Co, Cr, Fe, Mn, Ni, Pb, Sb, Se, Sn, Te, Ti, Zn, Zr	LA-ICP-MS	minimum of 10 ablation analysis (diameter 100 µm) on different locations	n.a.	Calibration using CRM from BAM	ICPMS quadrupole DRC2, Perkin Elmer

Laboratory code: L20

Elements	Technique	Sample mass [g]	Sample preparation	Calibration	Instrumentation
As, Au, Be, Bi, Cd, Co, Cr, Fe, In, Mg, Mn, Ni, Pb, Se, Te, Ti, Zr, W	ICP-MS		Dissolution in closed vessel with acid	external calibration, prepared from certified standard solutions for ICP	Agilent ICP-MS 7700
Ag, Al, Sb, Sn, Zn	ICP-MS		Open vessel dissolution with acid	external calibration, prepared from certified standard solutions for ICP	Agilent ICP-MS 7700

Laboratory code: L21

Elements	Technique	Sample mass [g]	Sample preparation	Calibration	Instrumentation
Ag, As, Bi, Cd, Co, Cr, Fe, Mn, Ni, P, Pb, S, Sb, Se, Sn, Te, Zn	Spark-OES	n.a.	Sample cleaned with diluted HNO ₃ and ethanol	external calibration using CRM	Spectrolab M8

Annex D: Results of the characterisation measurements for ERM-EB075A, B and C (data not pooled).

The results reported correspond to the mean of the means of the laboratory for each format (ERM-EB075A, B and C).

Analyte	ERM-EB075A		ERM-EB075B		ERM-EB075C	
	Mean of laboratory mean [mg/kg]	standard deviation of mean [mg/kg]	Mean of laboratory mean [mg/kg]	standard deviation of mean [mg/kg]	Mean of laboratory mean [mg/kg]	standard deviation of mean [mg/kg]
Ag	10.647	0.154	10.876	0.214	10.797	0.186
Al	2.238	0.100	2.159	0.099	2.392	0.153
As	3.220	0.057	3.218	0.056	3.205	0.054
Au	1.406	0.063	1.413	0.079	1.434	0.074
Be	1.058	0.041	1.059	0.043	1.070	0.051
Bi	1.727	0.073	1.732	0.079	1.765	0.044
Cd	2.660	0.024	2.664	0.033	2.707	0.035
Co	2.639	0.040	2.654	0.046	2.593	0.049
Cr	1.382	0.035	1.415	0.038	1.439	0.035
Fe	9.416	0.222	9.644	0.391	8.947	0.114
In	1.822	0.050	1.829	0.046	1.844	0.050
Mg	6.404	0.652	6.340	0.708	6.150	0.861
Mn	1.327	0.030	1.343	0.028	1.342	0.031
Ni	2.173	0.047	2.165	0.046	2.144	0.050
P	2.600	0.122	2.569	0.156	2.673	0.164
Pb	4.836	0.117	4.727	0.160	4.731	0.161
S	25.789	1.683	25.891	2.321	26.321	2.815
Sb	2.914	0.073	2.910	0.071	2.923	0.073
Se	1.726	0.047	1.689	0.058	1.724	0.053
Si	2.643	0.242	2.548	0.167	2.740	0.036
Sn	2.079	0.063	2.137	0.069	2.180	0.032
Te	1.828	0.057	1.828	0.051	1.817	0.051
Ti	3.082	0.067	3.119	0.062	3.248	0.105
Zn	6.630	0.170	6.515	0.163	6.530	0.126
Zr	16.457	1.438	17.311	2.053	17.907	1.336

Annex E: Results of the characterisation measurements (pooled data)

The characterisation study was performed using the results with the number of significant digits provided by each laboratory. In the Figures of Annex E, the results are reported with two significant digits after the coma for formatting reason.

Ag (Silver)

Table E1. Individual results for Ag mass fraction in ERM-EB075 provided by each laboratory. The mean value was calculated as the unweighted mean of all individual results. The expanded uncertainty budget equals to k factor ($k = 2$) multiplied by u provided by the laboratory or is estimated as two times the standard deviation of the measurements (if u is not provided).

Lab code - Method	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Rep 6	Rep 7	Rep 8	Rep 9	Rep 10	Rep 11	Rep 12	Rep 13	Rep 14	Rep 15	Rep 16	Rep 17	Rep 18	Mean	Uncertainty (k=2)	
	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]
L2-ICP-OES	10.60	10.50	10.40	10.60	10.50	10.30	10.20	10.90	10.60	10.50	10.30	10.50	10.40	10.70	10.50	10.50	11.00	10.60	10.53	0.57	
L4-ICP-MS	9.94	11.50	11.40	10.90	11.40	10.80	11.70	10.70	10.70	11.60	11.50	10.90	10.80	10.80	11.60	11.40	10.80	11.00	11.08	0.92	
L5-GD-MS	9.75	9.20	9.51	10.17	10.14	9.12	11.38	10.85	9.28	9.83	9.50	9.52							9.85	3.00	
L6-ICP-OES	10.40	10.20	10.40	10.50	10.40	10.40	10.10	10.30	10.40	10.40	10.40	10.30	10.40	10.40	10.50	10.30	10.40	10.40	10.37	0.19	
L7-DC-arc-OES													12.00	11.00	11.00	10.00	11.00	11.00	11.00	1.26	
L8-ICP-MS	9.80	9.70	9.90	9.70	9.60	9.80	9.80	9.80	9.30	9.90	9.70	9.60	9.80	9.90	9.64	9.80	9.80	9.60	9.73	0.97	
L9-ICP-MS	10.22	10.60	10.40	9.82	9.82	10.60	10.09	9.75	10.30	9.70	11.70	11.40	10.00	10.01	10.70	10.31	10.60	10.70	10.37	2.07	
L10-INAA	11.90	11.90	11.80	12.00	11.80	11.80	11.70	11.70	11.90	11.90	11.80	11.80	11.70	11.90	12.00	11.80	11.90	12.00	11.85	0.80	
L11-ICP-MS	9.82	9.72	9.78	9.77	9.70	9.67	9.70	9.62	9.64	9.80	9.78	9.80	9.72	9.78	9.78	9.67	9.59	9.65	9.72	2.43	
L13-GD-MS	11.17	10.73	11.08	10.09	10.91	11.10	11.42	11.45	11.70	10.77	10.88	11.46							11.06	0.69	
L14-INAA	10.30	10.11	10.46	10.53	10.58	10.73	10.45	10.50	10.12	9.79	10.46	10.47	10.40	10.91	10.57	11.36	10.30	10.98	10.50	0.50	
L15-INAA	10.80	10.70	10.90	11.20	10.80	11.00	11.30	11.20	11.20	11.40	11.10	11.00	12.00	12.10	12.20	12.00	12.30	11.70	11.38	0.20	
L16-ICP-OES	11.43	10.89	11.36	10.96	10.94	11.34	12.25	11.57	11.41	10.96	11.29	10.58	11.00	11.79	10.89	11.58	10.71	11.29	11.24	0.67	
L17-ICP-MS	10.85	10.73	10.87	10.77	10.79	10.87	10.69	10.70	10.97	10.75	10.47	10.88	10.66	10.74	9.64	10.59	11.00	10.45	10.69	0.73	
L18-ICP-MS	11.27	11.43	11.78	11.45	11.47	11.63	12.25	12.04	11.93	12.47	12.33	12.60	11.29	11.04	11.84	11.24	11.33	11.74	11.73	0.92	
L19-LA-ICP-MS	10.55	10.52	10.49	10.97	10.91	10.64													10.68	0.42	
L20-ICP-MS													10.60	10.50	9.64	10.50	10.60	10.60	10.41	0.76	
L21-Spark-OES	10.50	11.30	11.70	10.00	10.80	11.10	12.10	12.40	12.30	11.70	12.80	12.20							11.58	1.70	
<i>Results not used for certification</i>																					
L1-ICP-MS	9.53	8.89	8.77	9.21	9.00	9.44	8.67	9.38	9.45	8.62	8.55	9.12	9.44	9.12	9.00	8.85	9.21	8.99	9.07	0.61	
L2-ICP-MS	10.00	9.06	10.25	10.01	10.11	9.37	9.32	9.59	9.83	9.33	9.40	10.04	10.00	10.03	9.33	9.27	9.85	9.36	9.68	1.56	

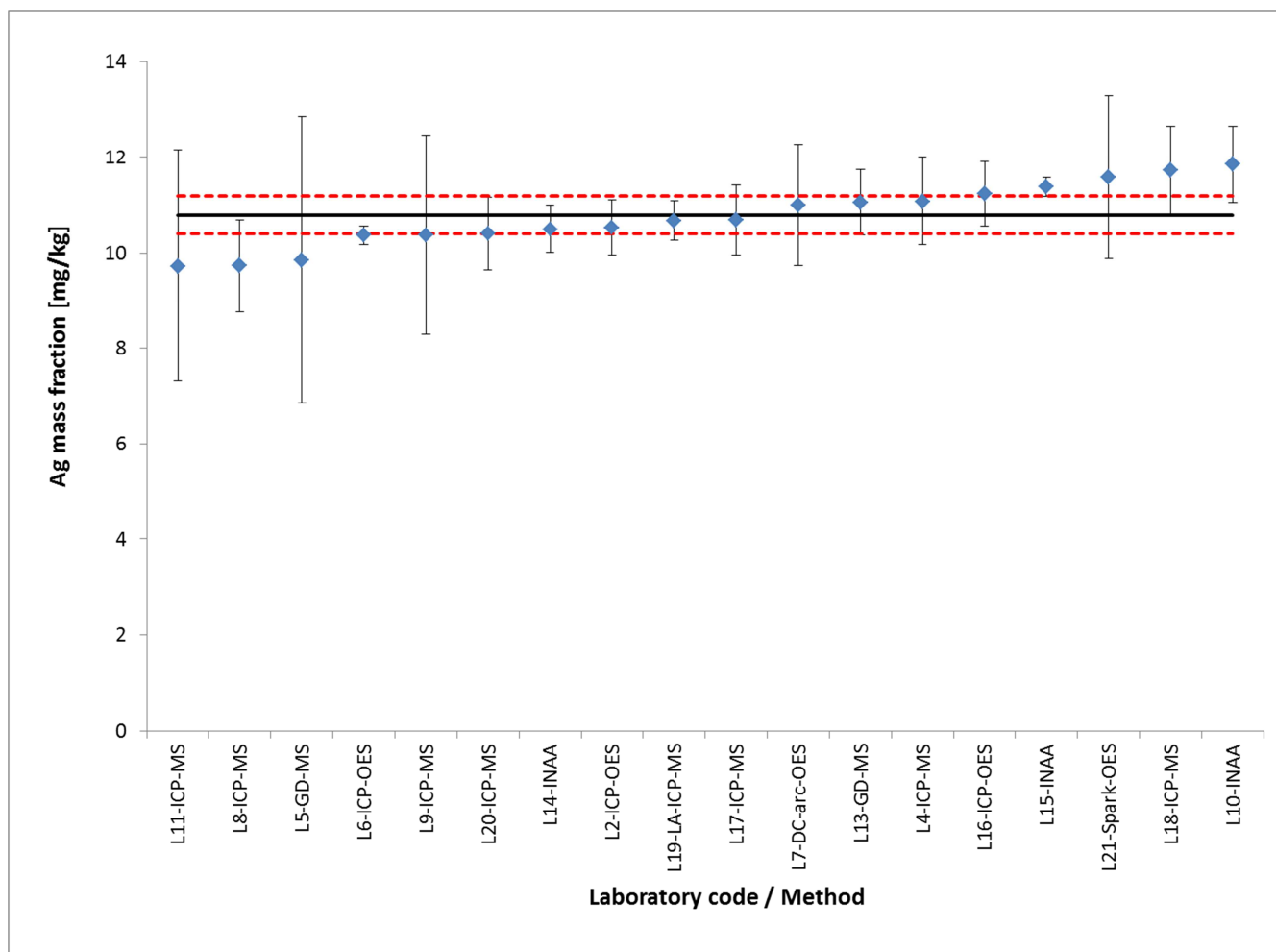


Figure E1. Mean Ag mass fraction in ERM-EB075 reported by participating laboratories. Error bars represent expanded uncertainties. The solid line represents the certified values (the mean of laboratory means), while the broken lines represent the expanded uncertainty of the certified value. Each laboratory is represented by its code.

Al (Aluminium)

Table E2. Individual results for Al mass fraction in ERM-EB075 provided by each laboratory. The mean value was calculated as the unweighted mean of all individual results. The expanded uncertainty budget equals to k factor ($k = 2$) multiplied by u provided by the laboratory or is estimated as two times the standard deviation of the measurements (if u is not provided).

Lab code - Method	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Rep 6	Rep 7	Rep 8	Rep 9	Rep 10	Rep 11	Rep 12	Rep 13	Rep 14	Rep 15	Rep 16	Rep 17	Rep 18	Rep 19	Rep 20	Mean	Uncertainty (k=2)	
	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]
L2-ICP-OES	3.20	3.80	2.70	2.80	2.40	2.60	2.60	2.90	3.00	2.60	2.60	2.90	2.70	3.60	2.90	3.70	2.80	2.60			2.91	0.82	
L4-ICP-MS	3.27	1.37	1.83	2.02	2.17	2.04	1.40	2.30	1.99		1.81	2.36	2.73	1.98	1.85	3.66	2.03	1.97			2.16	1.18	
L5-GD-MS	1.50	1.76	1.98	1.90	2.05	1.61	2.52	2.30	1.72	1.76	1.64	1.46									1.85	1.20	
L8-ICP-MS	1.80	1.80	1.90	1.80	1.90	2.00	1.90	1.80	1.80	1.90	2.00	1.80	1.80	1.90	1.80	1.80	1.80	1.90			1.86	0.19	
L9-ICP-MS	1.72	2.50	2.83	1.70	1.80	2.10	1.59	1.69	1.18	1.64	2.71	2.39	1.63	1.64	2.05	1.48	2.66	2.62			2.00	0.40	
L11-ICP-MS	2.30	2.42	2.33	2.29	2.34	2.32	2.33	2.29	2.43	2.33	2.33	2.26	2.45	2.35	2.40	2.32	2.38	2.41			2.35	0.59	
L13-GD-MS	2.28	2.33	2.39	2.28	2.34	2.37	2.35	2.21	2.25	2.37	2.30	2.38									2.32	0.19	
L16-ICP-OES	2.32	2.35	2.29	2.26	2.30	2.35	2.27	2.23	2.18	2.34	2.26	2.23	2.35	2.31	2.34	2.34	2.27	2.23			2.29	0.05	
L17-ICP-OES	2.30	2.02	2.05	2.33	1.96	2.26	1.99	2.31	2.29	2.08	1.99	2.35	2.34	2.32	2.03	2.04	2.35	2.02			2.17	0.49	
L19-LA-ICP-MS	2.47	2.55	2.72	2.36	2.05	2.51															2.44	0.46	
L20-ICP-MS													3.00	3.00	2.80	3.20	2.80	3.20			3.00	0.36	
<i>Results not used for certification</i>																							
L1-ICP-MS	4.75	5.61	3.79	3.02	3.70	4.15	4.72	6.79	5.25	4.49	4.97	3.51	3.78	6.19	3.72	4.58	3.25	4.80			4.50	2.02	
L18-ICP-MS	13.55	13.33	14.25	14.05	11.65	13.89	12.55	14.42	11.70	16.74	11.20	14.01	10.00	9.71	11.71	12.56	12.53	11.06			12.72	3.50	

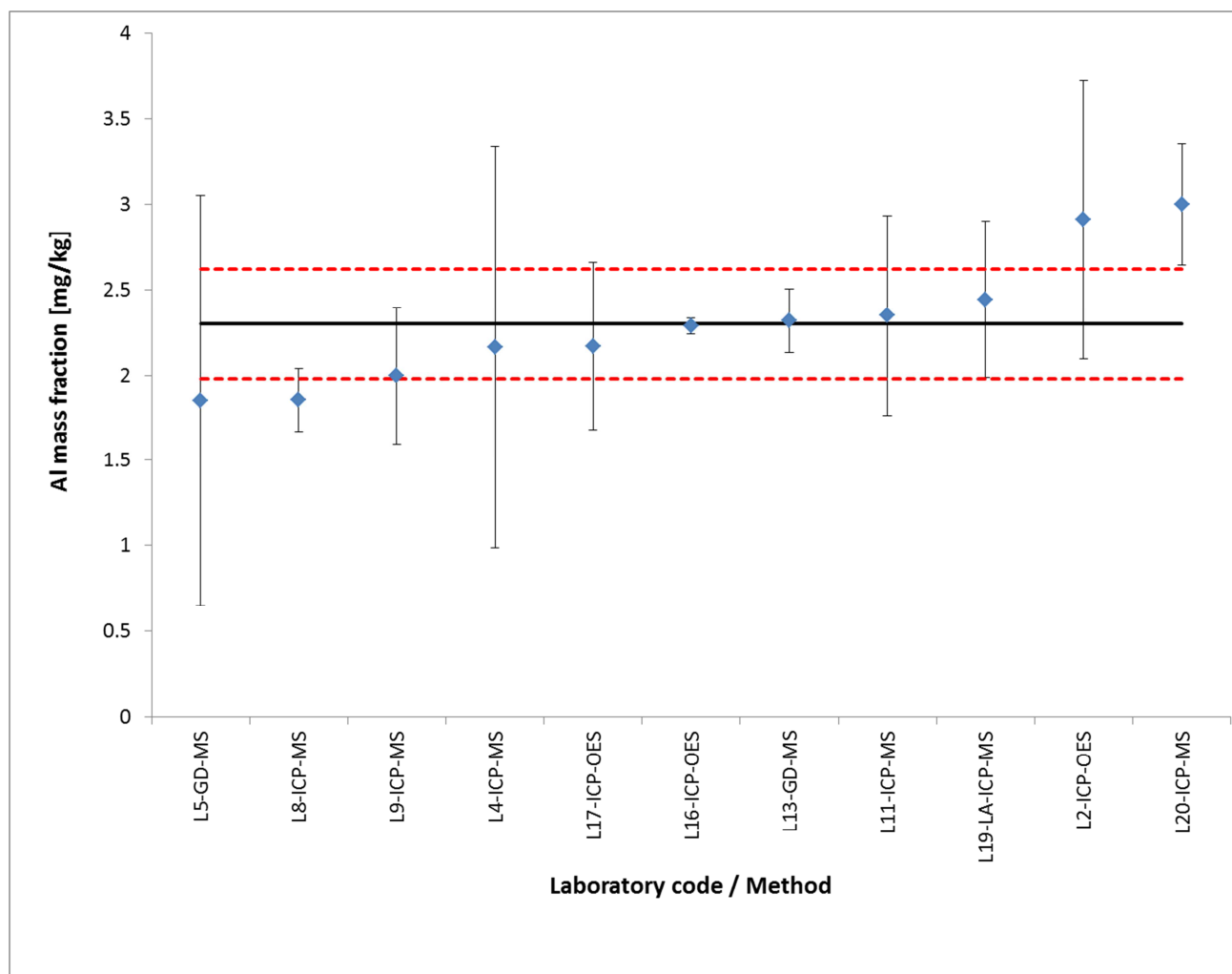


Figure E2. Mean Al mass fraction in ERM-EB075 reported by participating laboratories. Error bars represent expanded uncertainties. The solid line represents the certified values (the mean of laboratory means), while the broken lines represent the expanded uncertainty of the certified value. Each laboratory is represented by its code.

As (Arsenic)

Table E3. Individual results for As mass fraction in ERM-EB075 provided by each laboratory. The mean value was calculated as the unweighted mean of all individual results. The expanded uncertainty budget equals to k factor ($k = 2$) multiplied by u provided by the laboratory or is estimated as two times the standard deviation of the measurements (if u is not provided).

Lab code - Method	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Rep 6	Rep 7	Rep 8	Rep 9	Rep 10	Rep 11	Rep 12	Rep 13	Rep 14	Rep 15	Rep 16	Rep 17	Rep 18	Rep 19	Rep 20	Mean	Uncertainty ($k=2$)	
	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]
L1-ICP-MS	3.44	3.27	3.43	3.17	2.43	3.38	3.25	3.61	3.81	3.22	3.25	3.18	2.86	3.33	3.34	3.09	3.26	3.43			3.27	0.58	
L2-ICP-OES	3.20	3.30	3.20	3.30	3.30	3.40	3.30	3.20	3.30	3.30	3.20	3.20	3.20	3.30	3.30	3.10	3.30	3.20			3.26	0.17	
L3-ETV-ICP-OES	3.12	3.10	3.14	3.10	3.09	3.07	3.10	3.09	3.07	3.10	3.13	3.07	3.08	3.08	3.08	3.00	2.98	2.97	3.03	3.08	3.08	0.12	
L4-ICP-MS	2.97	3.09	3.02	3.00	3.06	2.96	3.17	2.95	2.92	3.12	3.00	2.98	2.99	2.97	3.11	3.03	3.02	3.13			3.03	0.14	
L5-GD-MS	2.82	3.65	3.71	2.89	2.84	3.73	2.86	2.83	3.65	2.84	3.83	3.84									3.29	1.20	
L6-ICP-MS	3.20	3.10	3.20	3.10	3.30	3.20	3.30	3.10	3.10	3.30	3.20	3.20	3.10	3.00	3.20	3.20	3.10	3.20			3.17	0.17	
L7-DC-arc-OES													3.10	3.10	3.10	3.10	3.20	3.10			3.12	0.08	
L9-ICP-MS	3.09	3.10	3.00	3.08	2.97	3.10	3.04	2.99	3.00	3.07	3.10	3.00	3.18	3.21	3.30	3.30	3.10	3.20			3.10	0.62	
L10-INAA	3.55	4.18	3.68	3.85	3.58	3.59	3.41	3.58	3.70	3.69	3.68	3.68	3.44	3.60	3.77	3.66	3.51	3.60			3.65	0.72	
L11-ICP-MS	3.56	3.37	3.49	3.41	3.33	3.41	3.39	3.36	3.42	3.36	3.51	3.40	3.36	3.41	3.28	3.42	3.41	3.38			3.40	0.85	
L13-GD-MS	3.23	3.23	3.47	3.38	3.40	3.47	3.22	3.19	3.31	3.00	3.20	3.26									3.28	0.34	
L14-INAA	3.13	3.11	3.71	3.31	3.20	2.88	2.90	2.99	3.04	2.36	3.27	2.69	3.04	3.46	3.27	3.50	3.30	3.26			3.13	0.32	
L16-ICP-MS	3.28	3.39	3.40	2.83	3.51	3.22	3.33	3.51	3.11	3.40	3.38	3.36	3.61	3.47	3.25	3.64	3.30	3.22			3.35	0.27	
L17-ICP-MS	3.07	3.22	3.16	2.97	3.26	2.83	3.22	3.04	2.96	3.24	3.27	3.03	3.06	2.95	3.29	3.26	2.95	3.24			3.11	0.29	
L19-LA-ICP-MS	3.27	3.33	3.13	3.04	3.14	2.90															3.13	0.31	
L20-ICP-MS													2.90	2.80	2.60	2.90	2.80	2.90			2.82	0.23	
L21-Spark-OES	3.00	3.40	2.80	2.60	2.70	2.50	3.10	2.80	3.00	3.50	2.80	3.10									2.94	0.61	
<i>Results not used for certification</i>																							
L8-ICP-MS	2.20	2.10	2.20	2.30	2.20	2.10	2.10	2.00	2.20	2.30	2.20	2.10	2.10	2.20	2.30	2.20	2.20	2.00			2.17	0.22	
L18-ICP-MS	4.49	4.38	4.52	4.77	4.51	4.62	4.45	4.24	4.40	4.27	4.21	4.27	4.28	4.17	4.40	3.99	4.21	4.09			4.35	0.39	

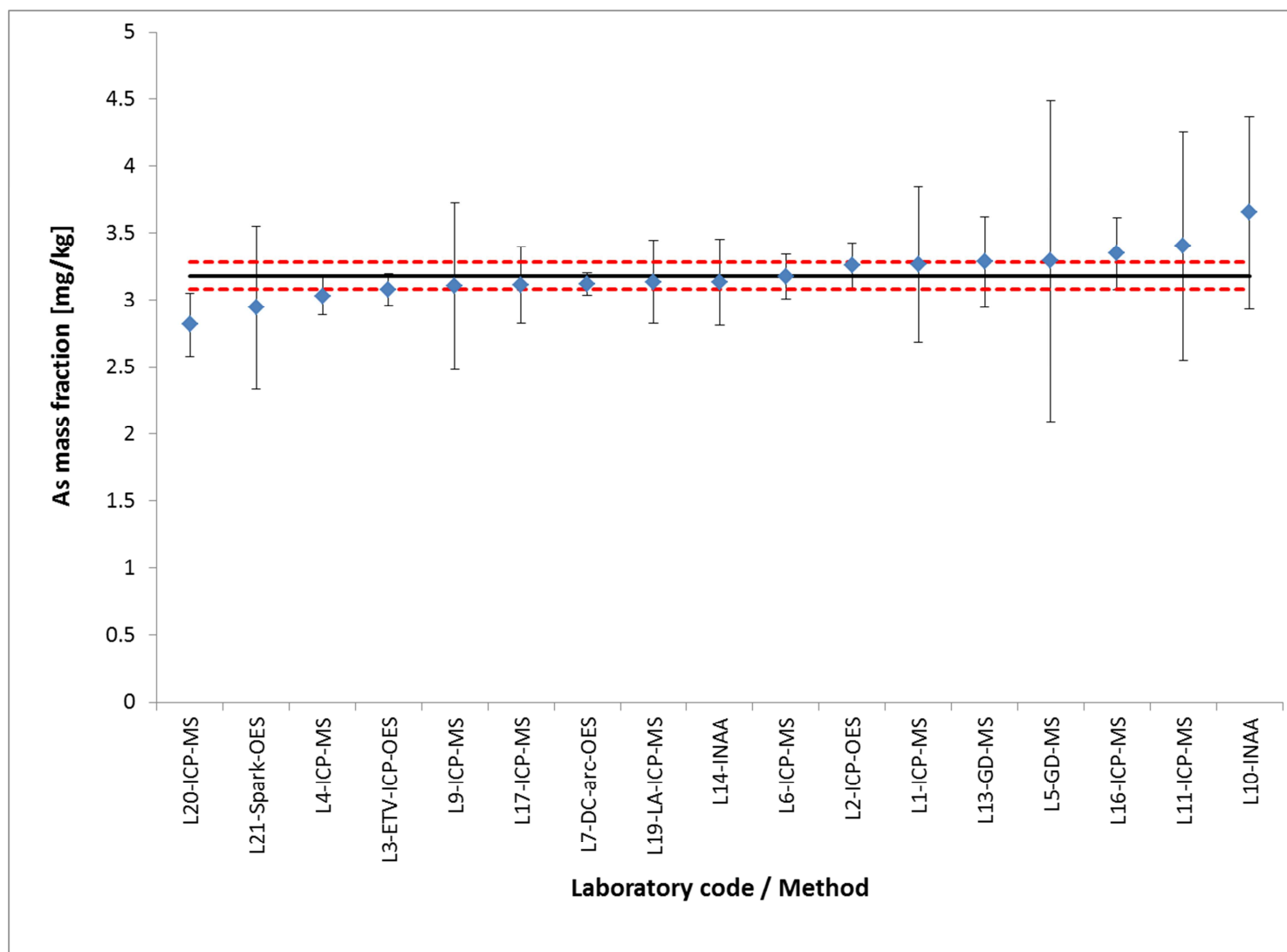


Figure E3. Mean As mass fraction in ERM-EB075 reported by participating laboratories. Error bars represent expanded uncertainties. The solid line represents the certified values (the mean of laboratory means), while the broken lines represent the expanded uncertainty of the certified value. Each laboratory is represented by its code.

Au (Gold)

Table E4. Individual results for Au mass fraction in ERM-EB075 provided by each laboratory. The mean value was calculated as the unweighted mean of all individual results. The expanded uncertainty budget equals to k factor ($k = 2$) multiplied by u provided by the laboratory or is estimated as two times the standard deviation of the measurements (if u is not provided).

Lab code - Method	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Rep 6	Rep 7	Rep 8	Rep 9	Rep 10	Rep 11	Rep 12	Rep 13	Rep 14	Rep 15	Rep 16	Rep 17	Rep 18	Rep 19	Rep 20	Mean	Uncertainty ($k=2$)	
	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]
L1-INAA	1.24	1.30	1.21	1.13	1.18	1.30	1.30	1.26	1.20	1.28	1.30	1.14	1.39	1.33	1.49	1.50	1.35	1.47			1.30	0.22	
L8-ICP-MS	1.80	1.80	1.80	1.70	1.90	1.80	1.70	1.90	1.70	1.80	1.70	1.80	1.60	1.80	1.90	1.90	1.60	1.90			1.78	0.18	
L10-INAA	1.45	1.48	1.45	1.48	1.45	1.45	1.45	1.45	1.47	1.47	1.46	1.46	1.45	1.48	1.48	1.47	1.49	1.49			1.47	0.10	
L11-ICP-MS	1.48	1.61	1.52	1.52	1.48	1.52	1.45	1.56	1.51	1.53	1.51	1.55	1.55	1.43	1.50	1.48	1.48	1.47			1.51	0.38	
L14-INAA	1.40	1.37	1.42	1.39	1.40	1.38	1.42	1.39	1.40	1.41	1.43	1.38	1.36	1.42	1.40	1.42	1.33	1.42			1.40	0.07	
L15-INAA	1.38	1.39	1.37	1.41	1.39	1.39	1.42	1.40	1.40	1.43	1.42	1.39	1.48	1.48	1.46	1.45	1.48	1.45			1.42	0.02	
L16-ICP-MS	1.04	1.35	1.35	0.99	1.27	0.97	1.25	0.91	0.83	1.07	1.08	1.35	1.02	1.11	1.04	1.31	1.04	1.27			1.13	0.34	
L17-ICP-MS	1.25	1.51	1.19	1.40	1.29	1.38	1.29	1.09	1.40	1.29	1.22	1.40	1.39	1.08	1.26	1.22	1.38	1.26			1.29	0.43	
L18-ICP-MS	1.71	1.64	1.71	1.29	1.68	1.56	1.98	1.95	1.64	1.90	1.81	1.80	1.76	1.77	1.74	1.78	1.81	1.69			1.73	0.31	
L20-ICP-MS													1.57	1.50	1.53	1.52	1.53	1.49			1.52	0.06	
<i>Results not used for certification</i>																							
L9-ICP-MS	1.10	1.10	1.10	1.25	1.29	1.22	1.15	1.19	1.01	1.07	1.13	1.15	1.02	0.98	1.07	0.91	1.23	1.19			1.12	0.22	

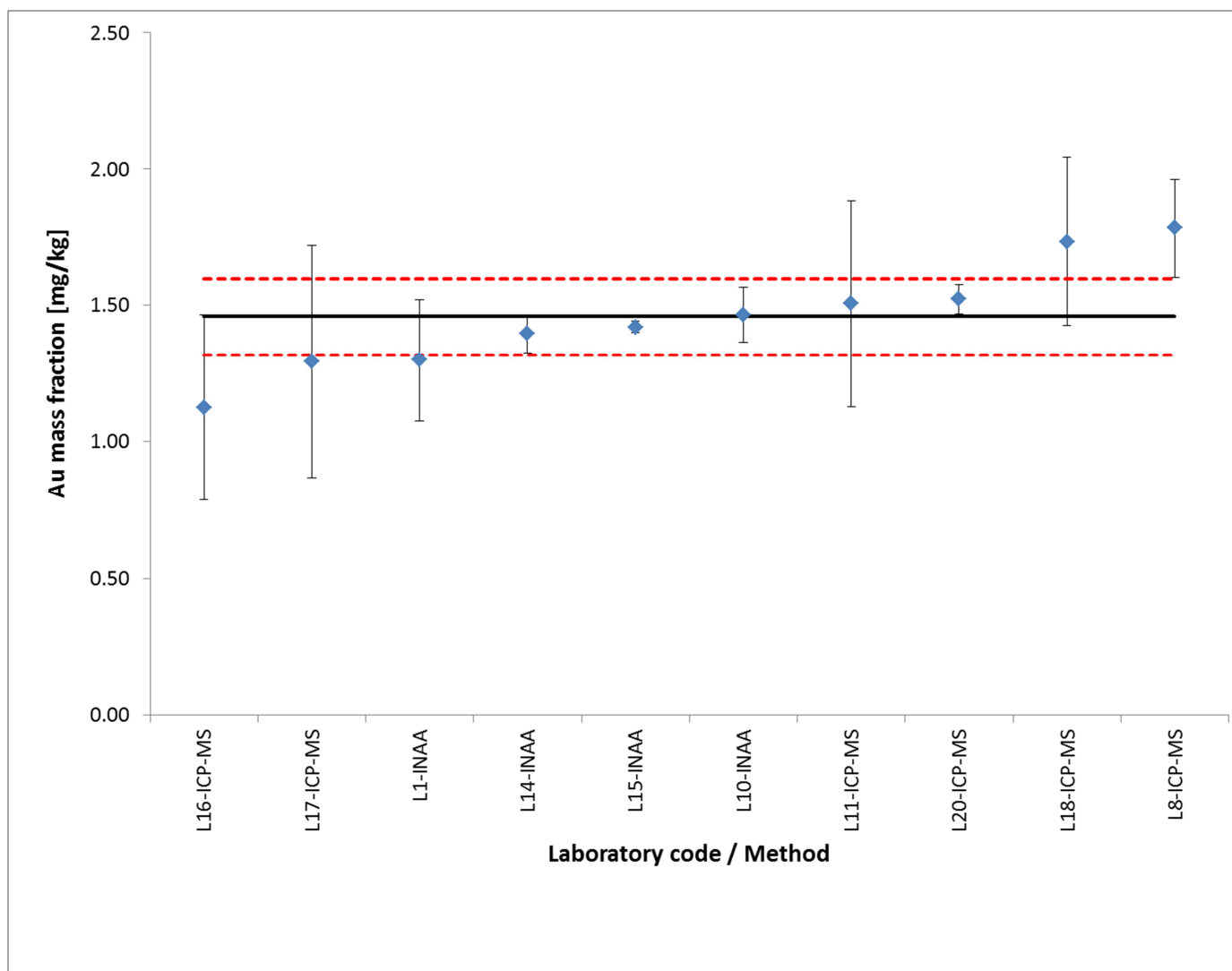


Figure E4. Mean Au mass fraction in ERM-EB075 reported by participating laboratories. Error bars represent expanded uncertainties. The solid line represents the certified values (the mean of laboratory means), while the broken lines represent the expanded uncertainty of the certified value. Each laboratory is represented by its code.

Be (Beryllium)

Table E5. Individual results for Be mass fraction in ERM-EB075 provided by each laboratory. The mean value was calculated as the unweighted mean of all individual results. The expanded uncertainty budget equals to k factor ($k = 2$) multiplied by u provided by the laboratory or is estimated as two times the standard deviation of the measurements (if u is not provided).

Lab code - Method	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Rep 6	Rep 7	Rep 8	Rep 9	Rep 10	Rep 11	Rep 12	Rep 13	Rep 14	Rep 15	Rep 16	Rep 17	Rep 18	Rep 19	Rep 20	Mean	Uncertainty (k=2)	
	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]
L1-ICP-MS	1.12	1.26	1.32	1.12	1.14	1.07	1.14	1.08	1.02	1.18	1.17	1.00	1.18	1.10	1.23	1.24	1.08	1.29			1.15	0.18	
L2-ICP-OES	1.30	1.20	1.20	1.20	1.10	1.20	1.20	1.30	1.30	1.20	1.20	1.30	1.30	1.30	1.30	1.30	1.30	1.30			1.25	0.12	
L4-ICP-MS	1.02	1.08	1.06	1.06	1.13	1.05	1.17	1.09	1.11	1.16	1.13	1.11	1.09	1.12	1.10	1.15	1.07	1.16			1.10	0.08	
L5-GD-MS	1.37	1.00	1.06	1.20	1.12	1.17	1.12	1.09	1.13	1.30	1.24	1.34									1.18	0.52	
L6-ICP-MS	1.29	1.16	1.24	1.12	1.15	1.15	1.28	1.23	1.25	1.26	1.21	1.29	1.33	1.20	1.19	1.23	1.25	1.25			1.23	0.11	
L8-ICP-MS	0.82	0.83	0.84	0.82	0.84	0.85	0.83	0.82	0.81	0.84	0.83	0.81	0.82	0.83	0.82	0.83	0.82	0.83			0.83	0.08	
L9-ICP-MS	1.23	0.94	1.10	1.26	1.25	1.06	1.11	1.11	0.91	1.01	0.92	0.96	0.91	0.97	0.89	0.91	0.84	1.01			1.02	0.20	
L11-ICP-MS	0.84	0.81	0.85	0.83	0.84	0.86	0.83	0.91	0.90	0.92	0.85	0.87	0.87	0.83	0.83	0.87	0.91	0.88			0.86	0.22	
L16-ICP-OES	0.99	1.01	1.02	0.89	0.92	0.89	1.01	0.98	0.98	1.03	1.00	0.97	1.04	1.06	1.05	1.41	1.05	1.07			1.02	0.14	
L17-ICP-MS	0.84	1.45	1.08	0.69	1.05	0.68	1.24	0.86	0.75	1.33	1.12	0.87	0.81	0.75	1.19	1.12	0.82	1.11			0.99	0.49	
L18-ICP-MS	1.09	1.14	1.17	1.04	1.24	1.07	1.10	0.96	0.92	1.05	0.98	0.90	0.98	0.97	0.97	0.97	0.97	0.95			1.03	0.19	
L20-ICP-MS													1.30	1.20	1.30	1.20	1.30	1.30			1.27	0.10	

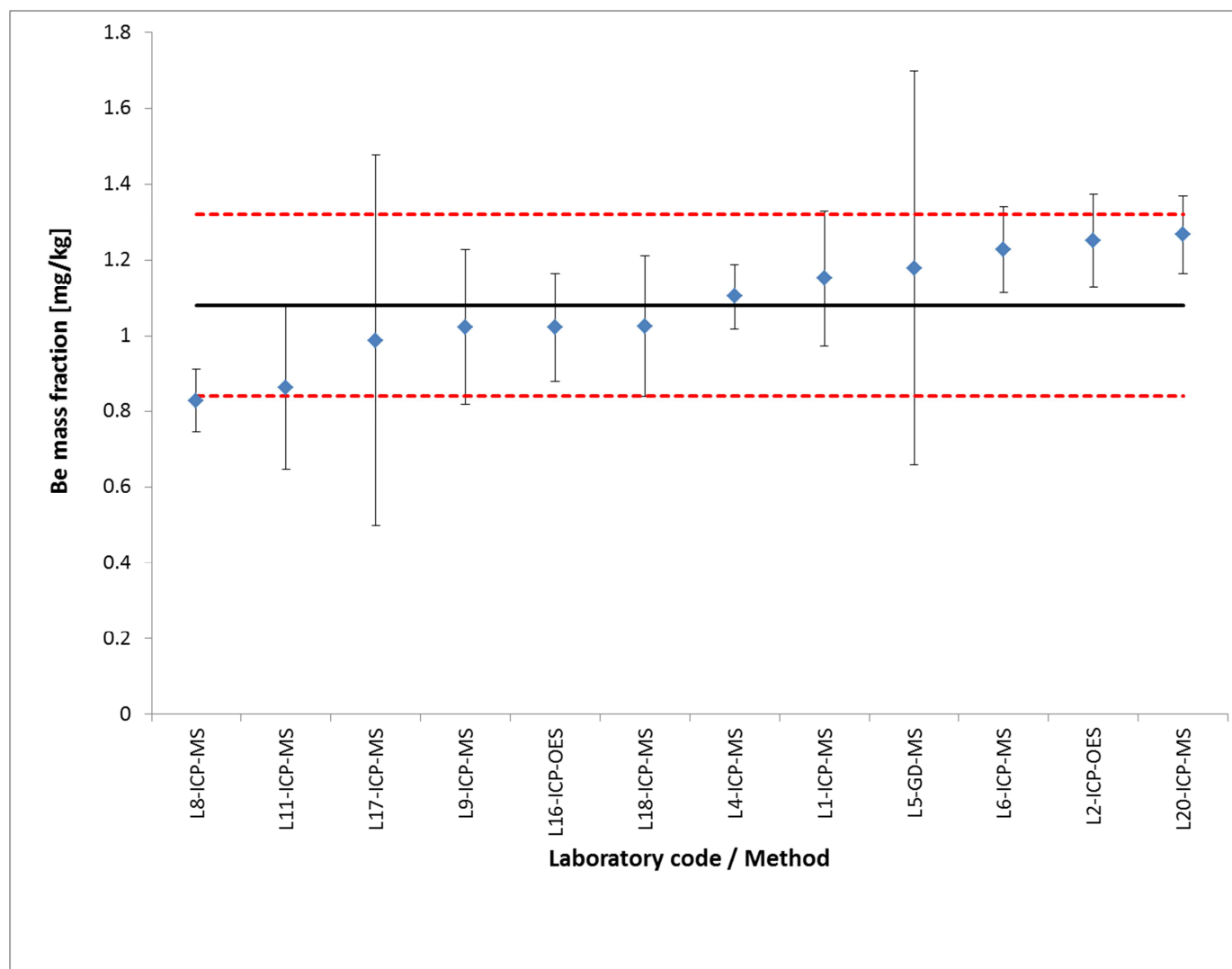


Figure E5. Mean Be mass fraction in ERM-EB075 reported by participating laboratories. Error bars represent expanded uncertainties. The solid line represents the certified values (the mean of laboratory means), while the broken lines represent the expanded uncertainty of the certified value. Each laboratory is represented by its code.

Bi (Bismuth)

Table E6. Individual results for Bi mass fraction in ERM-EB075 provided by each laboratory. The mean value was calculated as the unweighted mean of all individual results. The expanded uncertainty budget equals to k factor ($k = 2$) multiplied by u provided by the laboratory or is estimated as two times the standard deviation of the measurements (if u is not provided).

Lab code - Method	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Rep 6	Rep 7	Rep 8	Rep 9	Rep 10	Rep 11	Rep 12	Rep 13	Rep 14	Rep 15	Rep 16	Rep 17	Rep 18	Rep 19	Rep 20	Mean	Uncertainty (k=2)	
	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]
L1-ICP-MS	1.87	1.60	1.61	1.77	1.64	1.86	1.76	1.85	1.92	1.70	1.74	1.85	1.80	1.84	1.68	1.66	1.87	1.69			1.76	0.20	
L2-ICP-OES	1.80	0.90	1.40	1.40	1.60	1.70	0.90	1.60	1.70	1.00	1.30	1.70	1.80	1.60	1.50	1.50	1.40	1.40			1.46	0.56	
L6-ICP-MS	1.79	1.68	1.76	1.77	1.78	1.81	1.81	1.72	1.74	1.77	1.76	1.80	1.77	1.72	1.71	1.74	1.79	1.74			1.76	0.07	
L7-DC-arc-OES													1.85	1.76	1.81	1.76	1.89	1.76			1.81	0.11	
L8-ICP-MS	1.80	1.80	1.70	1.70	1.90	1.70	1.70	1.90	1.80	1.80	1.80	1.80	1.90	1.80	1.80	1.80	1.70	1.90			1.79	0.18	
L9-ICP-MS	2.33	2.10	2.30	2.29	2.28	2.10	2.30	2.29	2.20	2.88	2.10	2.20	2.07	2.23	2.20	2.33	1.90	2.10			2.23	0.45	
L11-ICP-MS	1.89	1.90	1.86	1.83	1.87	1.83	1.87	1.85	1.87	1.89	1.87	1.90	1.85	1.88	1.85	1.86	1.90	1.88			1.87	0.47	
L12-ICP-MS	1.62	1.72	1.63	1.56	1.71	1.74	1.81	1.66	1.60	1.64	1.69	1.61	1.63	1.57	1.72	1.66	1.60	1.70			1.66	0.27	
L13-GD-MS	2.29	1.86	1.98	1.75	1.94	2.00	1.98	2.22	2.04	1.86	1.80	2.01									1.98	0.25	
L16-ICP-MS	1.32	1.56	1.33	1.39	1.32	1.40	1.43	1.41	1.91	1.53	1.59	1.41	1.76	1.51	1.88	1.61	1.74	1.56			1.54	0.25	
L17-ICP-MS	1.67	1.68	1.67	1.69	1.71	1.70	1.67	1.61	1.67	1.69	1.67	1.67	1.66	1.67	1.67	1.67	1.66	1.68			1.67	0.35	
L19-LA-ICP-MS	1.72	1.68	1.71	1.78	1.75	1.72															1.73	0.07	
L20-ICP-MS													1.90	2.00	1.90	2.00	1.90	1.90			1.93	0.10	
L21-Spark-OES	1.90	2.00	1.90	2.20	1.80	2.00	1.50	1.50	1.90	2.40	1.40	1.50									1.83	0.62	
<i>Results not used for certification</i>																							
L3-ETV-ICP-OES	1.51	1.51	1.50	1.46	1.49	1.47	1.53	1.43	1.45	1.50	1.56	1.49	1.53	1.52	1.53	1.42	1.43	1.40	1.49	1.47	1.49	0.11	
L4-ICP-MS	1.62	1.64	1.62	1.59	1.64	1.59	1.63	1.58	1.59	1.61	1.63	1.60	1.59	1.58	1.62	1.60	1.60	1.62			1.61	0.04	
L5-GD-MS	1.04	1.42	1.56	1.26	1.38	1.28	1.88	1.78	1.50	1.26	1.46	1.31									1.43	0.60	
L18-ICP-MS	1.33	1.15	1.24	1.04	1.33	1.26	1.24	1.31	1.17	1.29	1.33	1.34	1.54	1.52	1.58	1.64	1.65	1.60			1.36	0.36	

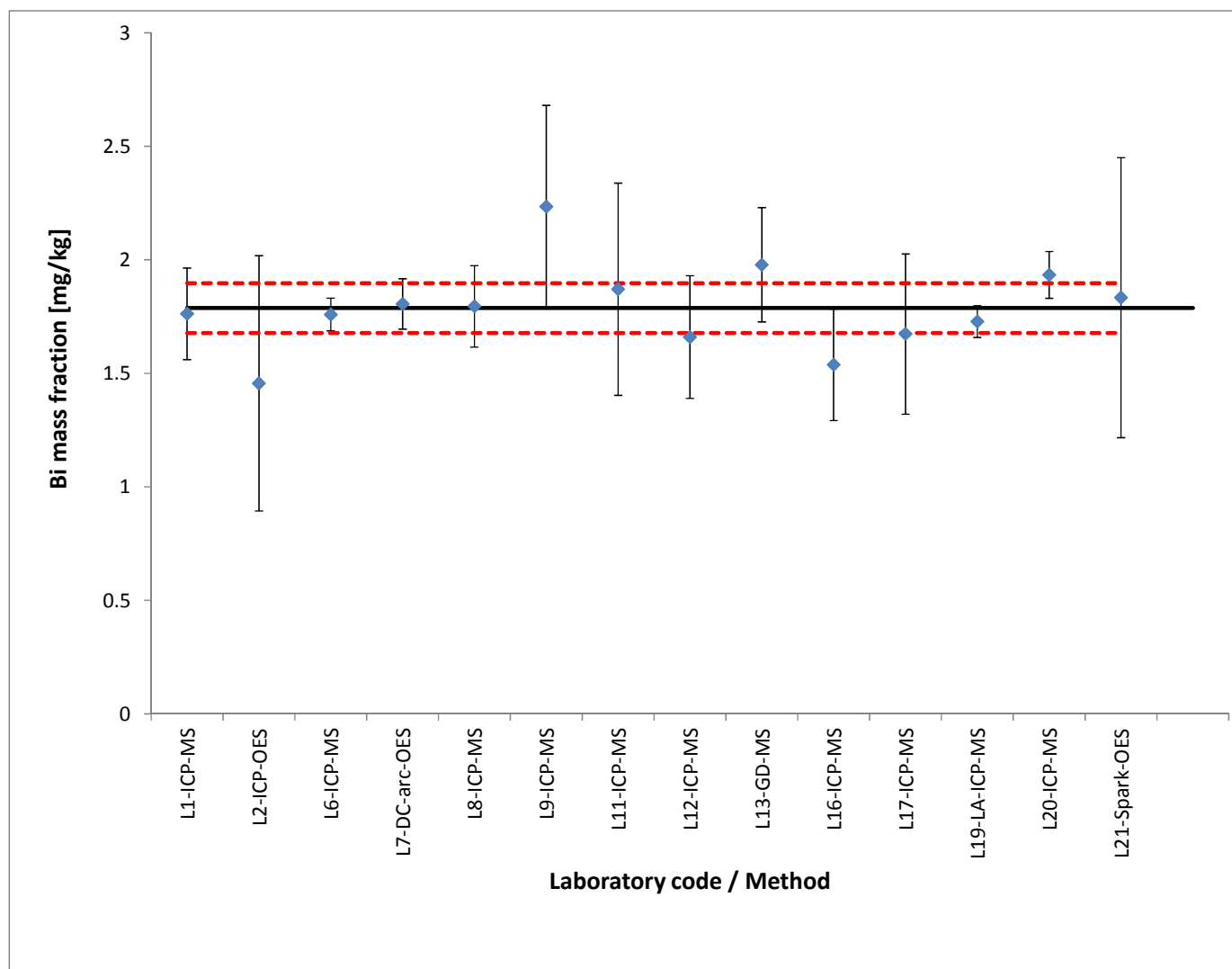


Figure E6. Mean Bi mass fraction in ERM-EB075 reported by participating laboratories. Error bars represent expanded uncertainties. The solid line represents the certified values (the mean of laboratory means), while the broken lines represent the expanded uncertainty of the certified value. Each laboratory is represented by its code.

Cd (Cadmium)

Table E7. Individual results for Cd mass fraction in ERM-EB075 provided by each laboratory. The mean value was calculated as the unweighted mean of all individual results. The expanded uncertainty budget equals to k factor ($k = 2$) multiplied by u provided by the laboratory or is estimated as two times the standard deviation of the measurements (if u is not provided).

Lab code - Method	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Rep 6	Rep 7	Rep 8	Rep 9	Rep 10	Rep 11	Rep 12	Rep 13	Rep 14	Rep 15	Rep 16	Rep 17	Rep 18	Rep 19	Rep 20	Mean	Uncertainty (k=2)	
	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]
L1-ICP-MS	2.58	2.42	2.58	2.57	2.52	2.58	2.59	2.54	2.64	2.44	2.50	2.47	2.56	2.61	2.60	2.50	2.53	2.61			2.55	0.12	
L2-ICP-OES	2.80	2.60	2.70	2.70	2.60	2.70	2.60	2.80	2.70	2.70	2.70	2.80	2.80	2.70	2.70	2.80	2.80	2.80			2.72	0.17	
L4-ICP-MS	2.65	2.85	2.87	2.71	2.84	2.72	2.90	2.63	2.64	2.87	2.82	2.71	2.66	2.66	2.84	2.84	2.67	2.82			2.76	0.19	
L5-GD-MS	2.09	2.94	3.03	2.27	2.29	2.84	2.62	2.52	2.94	2.21	2.98	2.91									2.64	1.20	
L6-ICP-OES	2.50	2.50	2.60	2.40	2.70	2.60	2.70	2.50	2.50	2.50	2.60	2.50	2.40	2.60	2.60	2.70	2.40	2.60			2.55	0.20	
L8-ICP-MS	2.60	2.50	2.60	2.50	2.60	2.60	2.60	2.60	2.20	2.50	2.40	2.70	2.70	2.60	2.70	2.50	2.40	2.40			2.54	0.25	
L9-ICP-MS	2.49	2.89	3.00	2.37	2.36	2.71	2.52	2.56	2.93	2.48	2.93	2.79	2.57	2.51	2.87	2.46	2.91	2.95			2.68	0.54	
L11-ICP-MS	2.70	2.66	2.75	2.72	2.75	2.69	2.68	2.69	2.72	2.67	2.66	2.64	2.62	2.66	2.70	2.66	2.65	2.83			2.69	0.67	
L12-ICP-MS	2.75	2.51	2.76	2.73	2.43	2.48	2.47	2.77	2.77	2.48	2.51	2.73	2.70	2.73	2.57	2.51	2.72	2.57			2.62	0.43	
L13-GD-MS	3.10	2.61	2.87	2.46	2.64	2.72	2.83	3.02	2.92	2.81	2.80	2.76									2.79	0.35	
L16-ICP-MS	2.71	2.75	2.59	2.52	2.64	2.59	2.71	2.72	2.57	2.72	2.67	2.75	3.04	2.68	2.68	2.95	2.61	2.68			2.70	0.16	
L17-ICP-MS	2.81	2.87	2.78	2.80	2.82	2.71	2.84	2.76	2.83	2.88	2.82	2.84	2.84	2.82	2.83	2.82	2.82	2.82			2.82	0.13	
L19-LA-ICP-MS	2.79	2.77	2.72	2.91	2.84	2.69															2.79	0.16	
L20-ICP-MS													3.20	2.90	2.80	2.80	2.90	3.20			2.97	0.37	
L21-Spark-OES	2.80	2.40	2.70	2.70	2.50	2.70	2.60	2.40	2.60	2.50	2.50	2.40									2.57	0.27	
<i>Results not used for certification</i>																							
L18-ICP-MS	2.48	2.48	2.37	2.33	2.39	2.46	2.42	2.48	2.38	2.32	2.47	2.49	2.62	2.54	2.42	2.60	2.63	2.53			2.47	0.19	

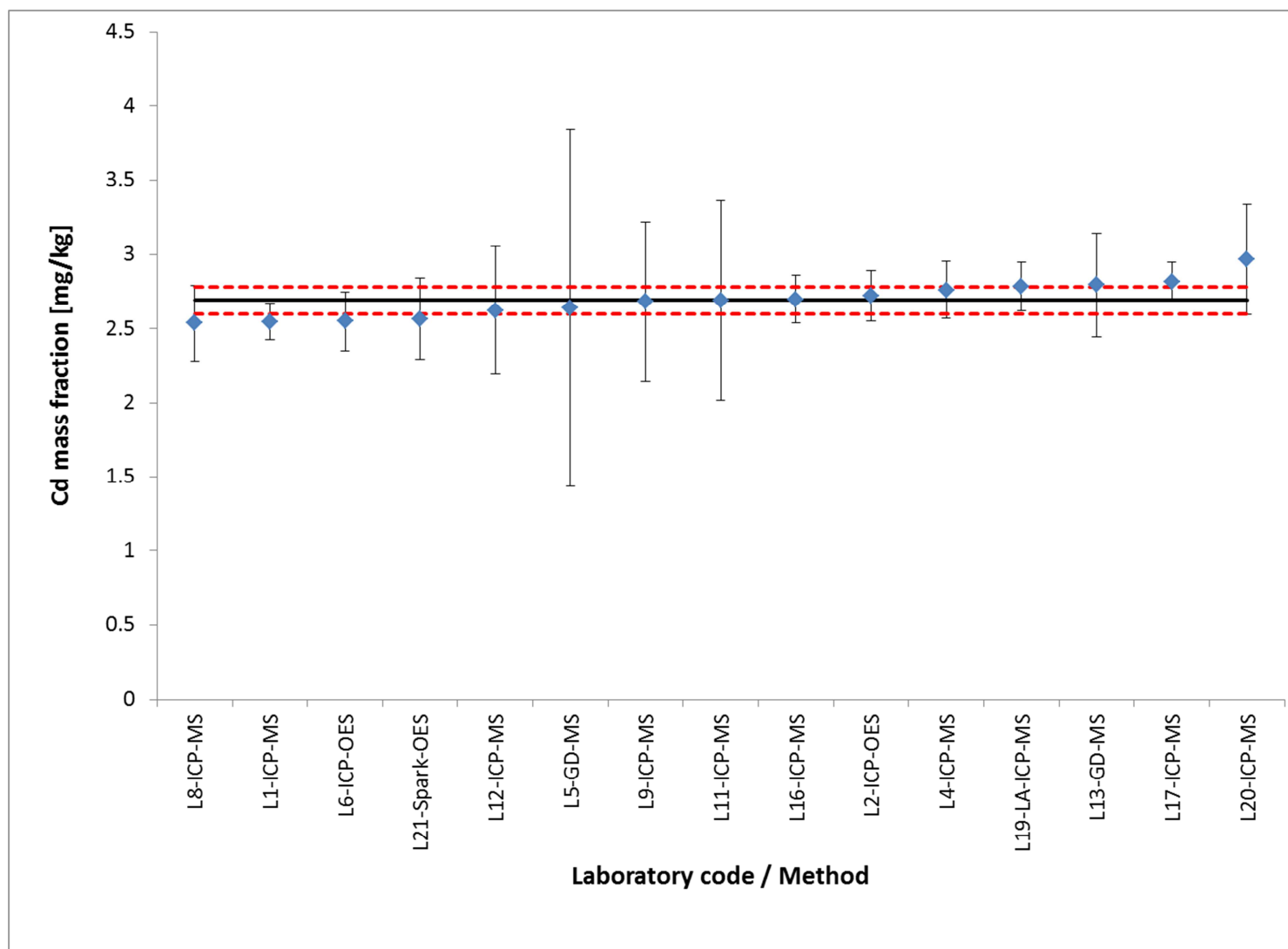


Figure E7. Mean Cd mass fraction in ERM-EB075 reported by participating laboratories. Error bars represent expanded uncertainties. The solid line represents the certified values (the mean of laboratory means), while the broken lines represent the expanded uncertainty of the certified value. Each laboratory is represented by its code.

Co (Cobalt)

Table E8. Individual results for Co mass fraction in ERM-EB075 provided by each laboratory. The mean value was calculated as the unweighted mean of all individual results. The expanded uncertainty budget equals to k factor ($k = 2$) multiplied by u provided by the laboratory or is estimated as two times the standard deviation of the measurements (if u is not provided).

Lab code - Method	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Rep 6	Rep 7	Rep 8	Rep 9	Rep 10	Rep 11	Rep 12	Rep 13	Rep 14	Rep 15	Rep 16	Rep 17	Rep 18	Rep 19	Rep 20	Mean	Uncertainty ($k=2$)	
	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]
L1-ICP-MS	2.68	2.73	2.75	2.66	2.79	2.74	2.66	2.56	2.57	2.68	2.82	2.39	2.64	2.65	2.78	2.74	2.71	2.79			2.68	0.21	
L2-ICP-OES	2.80	2.70	2.80	2.70	2.60	2.80	2.70	2.80	2.70	3.10	2.90	3.20	2.60	2.50	2.60	2.70	2.50	2.70			2.74	0.25	
L4-ICP-MS	2.60	2.64	2.62	2.57	2.59	2.57	2.64	2.56	2.62	2.63	2.60	2.58	2.59	2.56	2.58	2.56	2.56	2.55			2.59	0.06	
L5-GD-MS	2.86	2.92	2.94	2.87	2.93	2.89	2.85	2.91	2.87	2.96	2.84	2.85									2.89	0.32	
L6-ICP-OES	2.50	2.50	2.40	2.50	2.50	2.60	2.50	2.50	2.50	2.50	2.60	2.50	2.50	2.40	2.50	2.60	2.40	2.40			2.49	0.13	
L8-ICP-MS	2.60	2.40	2.40	2.40	2.40	2.50	2.50	2.60	2.20	2.50	2.60	2.40	2.50	2.50	2.50	2.40	2.20	2.30			2.44	0.24	
L9-ICP-MS	2.72	2.33	2.28	3.02	3.29	2.40	2.76	2.97	2.49	2.88	2.41	2.09	2.94	2.92	2.39	2.91	2.51	2.64			2.66	0.53	
L11-ICP-MS	2.54	2.42	2.42	2.51	2.47	2.44	2.45	2.41	2.44	2.42	2.41	2.50	2.33	2.36	2.33	2.33	2.32	2.41			2.42	0.60	
L12-ICP-MS	2.31	2.45	2.32	2.29	2.49	2.44	2.45	2.32	2.32	2.45	2.47	2.31	2.31	2.26	2.53	2.48	2.31	2.56			2.39	0.36	
L13-GD-MS	2.70	2.55	2.68	2.37	2.66	2.62	2.53	2.72	2.71	2.53	2.54	2.69									2.61	0.28	
L14-INAA	2.82	2.63	2.85	2.85	2.85	2.87	2.85	2.85	2.70	2.73	2.87	2.86	2.81	2.91	2.82	2.98	2.76	2.91			2.83	0.15	
L16-ICP-MS	2.61	2.45	2.56	2.49	2.43	2.46	2.77	2.90	2.48	2.66	2.62	2.57	2.62	2.52	2.63	2.61	2.48	2.43			2.57	0.21	
L17-ICP-MS	2.63	2.80	2.80	2.50	2.80	2.39	2.84	2.62	2.64	2.82	2.80	2.60	2.61	2.53	2.82	2.84	2.53	2.87			2.69	0.13	
L19-LA-ICP-MS	2.79	2.75	2.72	2.77	2.76	2.74															2.76	0.05	
L20-ICP-MS													2.70	2.60	2.50	2.60	2.70	2.80			2.65	0.21	
L21-Spark-OES	2.80	2.70	2.70	2.80	2.60	2.60	2.60	2.90	2.80	2.90	2.90	2.90									2.77	0.25	
<i>Results not used for certification</i>																							
L10-INAA	3.05	3.08	3.04	3.11	3.02	3.04	3.08	3.03	3.08	3.07	3.05	3.04	3.02	3.05	3.09	3.00	3.07	3.10			3.06	0.24	
L15-INAA	2.57	2.72	2.57	2.37	2.51	2.47	2.50	2.46	2.49	2.54	2.52	2.45	2.65	2.66	2.65	2.66	2.64	2.66			2.56	0.05	
L18-ICP-MS	2.27	2.19	2.25	2.49	2.27	2.35	2.17	2.23	2.22	2.18	2.04	2.14	2.19	2.09	2.06	2.07	2.06	2.00			2.18	0.24	

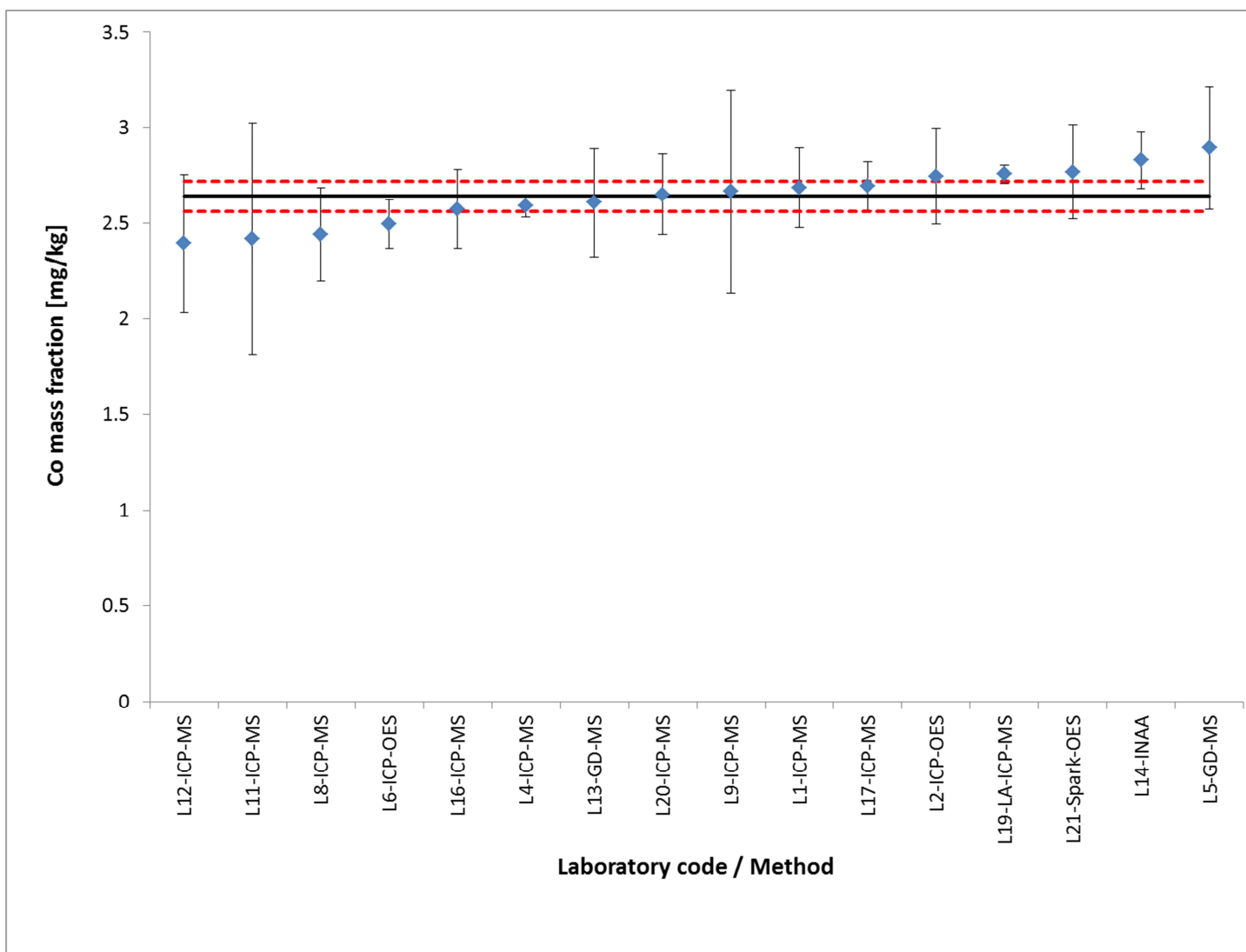


Figure E8. Mean Co mass fraction in ERM-EB075 reported by participating laboratories. Error bars represent expanded uncertainties. The solid line represents the certified values (the mean of laboratory means), while the broken lines represent the expanded uncertainty of the certified value. Each laboratory is represented by its code.

Cr (Chromium)

Table E9. Individual results for Cr mass fraction in ERM-EB075 provided by each laboratory. The mean value was calculated as the unweighted mean of all individual results. The expanded uncertainty budget equals to k factor ($k = 2$) multiplied by u provided by the laboratory or is estimated as two times the standard deviation of the measurements (if u is not provided).

Lab code - Method	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Rep 6	Rep 7	Rep 8	Rep 9	Rep 10	Rep 11	Rep 12	Rep 13	Rep 14	Rep 15	Rep 16	Rep 17	Rep 18	Rep 19	Rep 20	Mean	Uncertainty ($k=2$)	
	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]
L1-ICP-MS	1.58	1.53	1.69	1.50	1.67	1.59	1.38	1.64	1.95	1.61	1.56	1.69	1.48	1.54	1.46	1.34	1.55	1.57			1.58	0.27	
L2-ICP-OES	1.50	1.40	1.40	1.40	1.30	1.40	1.30	1.40	1.30	1.40	1.40	1.40	1.40	1.40	1.40	1.50	1.40	1.50			1.40	0.10	
L4-ICP-MS	1.42	1.44	1.40	1.41	1.41	1.41	1.41	1.40	1.41	0.00	1.40	1.42	1.50	1.42	1.41	1.43	1.41	1.45			1.34	0.05	
L5-GD-MS	1.45	1.25	1.24	1.44	1.56	1.39	1.68	1.71	1.29	1.52	1.21	1.23									1.41	0.56	
L6-ICP-OES	1.30	1.40	1.50	1.10	2.00	1.30	2.10	1.10	1.40	1.50	1.50	1.10	1.20	1.30	1.60	2.10	1.10	1.50			1.45	0.65	
L8-ICP-MS	1.40	1.50	1.40	1.50	1.50	1.60	1.50	1.40	1.40	1.40	1.50	1.50	1.40	1.60	1.50	1.40	1.40	1.50			1.47	0.15	
L9-ICP-MS	1.18	1.52	1.45	1.30	1.27	1.53	1.08	1.16	1.48	1.25	1.51	1.52	1.32	1.38	1.54	1.39	1.47	1.51			1.38	0.28	
L10-INAA	1.51	1.40	1.51	1.41	1.49	1.43	2.22	1.36	1.86	1.47	1.43	1.44	1.42	1.39	1.36	1.50	1.42	1.57			1.51	0.20	
L11-ICP-MS	1.15	1.11	1.14	1.12	1.14	1.10	1.11	1.16	1.15	1.11	1.11	1.20	1.04	1.19	1.05	1.07	1.10	1.12			1.12	0.28	
L12-ICP-MS	1.57	1.34	1.46	1.44	1.44	1.48	1.58	1.53	1.48	1.50	1.57	1.49	1.57	1.56	1.51	1.47	1.51	1.53			1.50	0.11	
L13-GD-MS	1.36	1.25	1.29	1.19	1.30	1.28	1.25	1.33	1.34	1.23	1.23	1.33									1.28	0.14	
L16-ICP-MS	1.37	1.60	1.50	1.31	1.45	1.31	1.47	1.40	1.38	1.37	1.45	1.62	1.33	1.39	1.37	1.43	1.39	1.45			1.42	0.11	
L17-ICP-MS	1.43	1.56	1.57	1.52	1.44	1.41	1.44	1.53	1.53	1.58	1.58	1.30	1.50	1.48	1.55	1.77	1.57	1.76			1.53	0.31	
L19-LA-ICP-MS	1.32	1.28	1.30	1.36	1.33	1.36															1.33	0.07	
L20-ICP-MS													1.50	1.50	1.40	1.50	1.50	1.50			1.48	0.08	
L21-Spark-OES	1.10	1.10	1.10	1.10	1.10	1.00	1.20	1.20	1.10	1.20	1.20	1.30									1.14	0.16	
<i>Results not used for certification</i>																							
L14-INAA	1.55	1.41	1.52	1.65	1.59	1.64	1.52	1.57	1.35	1.56	1.48	1.57	1.58	1.55	1.51	1.40	1.47	1.48			1.52	0.27	
L15-INAA	1.36	1.34	1.35	1.59	1.59	1.34	1.43	1.38	1.55	1.58	1.54	2.34	1.64	1.57	1.34	1.52	1.59	1.46			1.53	0.05	
L18-ICP-MS	1.64	1.74	1.65	1.92	1.55	1.78	1.60	1.47	1.65	1.56	1.62	1.43	1.31	1.26	1.35	1.34	1.27	1.39			1.53	0.38	

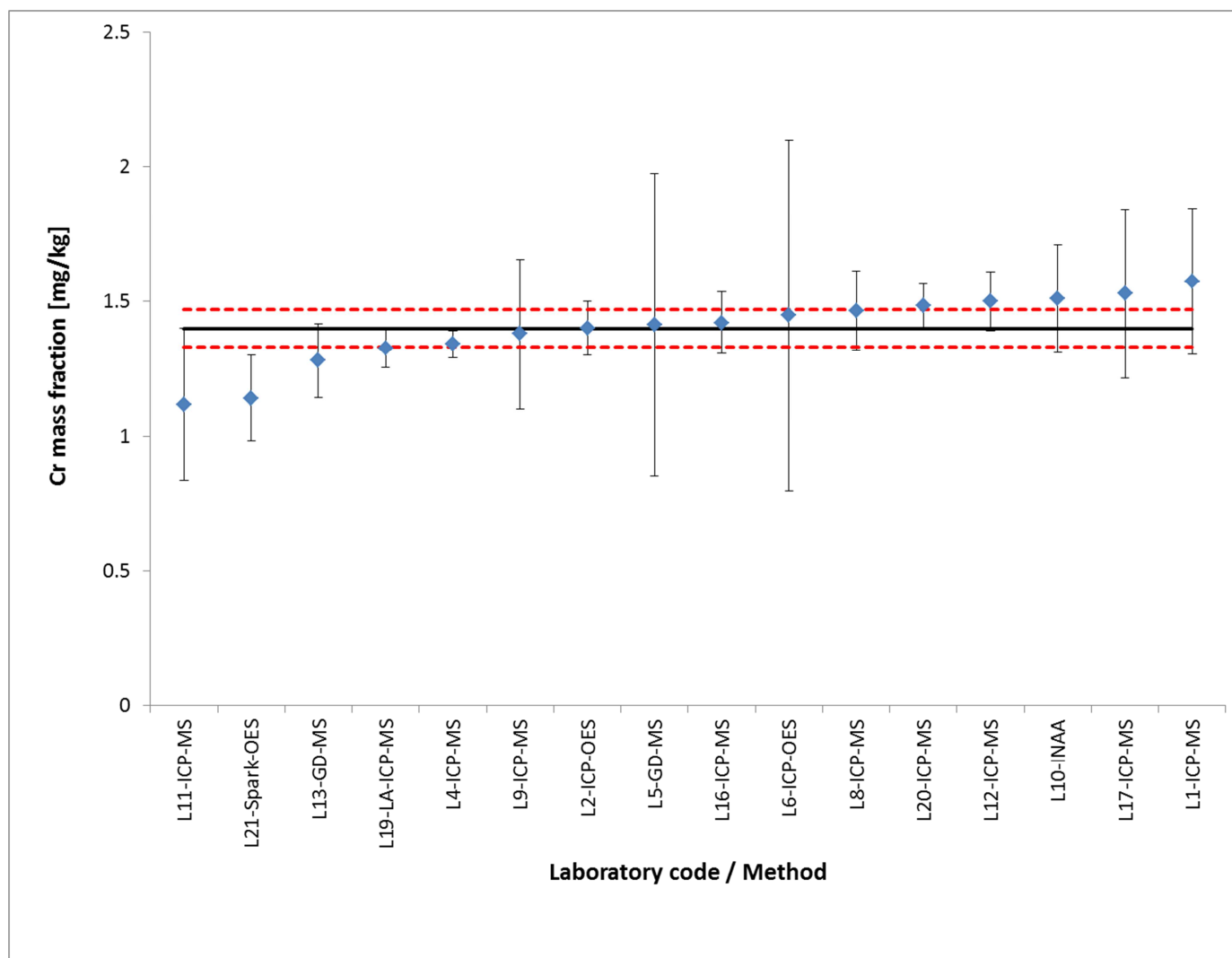


Figure E9. Mean Cr mass fraction in ERM-EB075 reported by participating laboratories. Error bars represent expanded uncertainties. The solid line represents the certified values (the mean of laboratory means), while the broken lines represent the expanded uncertainty of the certified value. Each laboratory is represented by its code.

Fe (Iron)

Table E10. Individual results for Fe mass fraction in ERM-EB075 provided by each laboratory. The mean value was calculated as the unweighted mean of all individual results. The expanded uncertainty budget equals to k factor ($k = 2$) multiplied by u provided by the laboratory or is estimated as two times the standard deviation of the measurements (if u is not provided).

Lab code - Method	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Rep 6	Rep 7	Rep 8	Rep 9	Rep 10	Rep 11	Rep 12	Rep 13	Rep 14	Rep 15	Rep 16	Rep 17	Rep 18	Rep 19	Rep 20	Mean	Uncertainty ($k=2$)	
	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]
L2-ICP-OES	11.30	8.60	9.20	11.80	8.80	10.50	10.80	11.80	13.20	13.60	10.20	15.10	8.90	8.40	8.10	8.60	7.90	8.40			10.29	4.10	
L5-GD-MS	9.52	10.50	10.68	9.73	9.97	10.37	10.06	10.00	10.04	9.77	9.81	9.76									10.02	2.40	
L6-ICP-MS	8.90	9.90	9.80	8.90	10.30	9.10	9.90	8.30	8.30	9.60	9.30	8.50	8.60	8.30	9.40	9.10	8.60	9.10			9.11	1.23	
L11-ICP-OES	9.20	9.10	9.10	9.00	9.10	8.90	9.10	8.80	8.90	9.10	9.10	9.10	8.80	9.00	9.00	8.90	8.80	8.90			8.99	2.25	
L12-ICP-MS	9.30	11.00	9.90	9.20	9.30	8.90	10.50	11.00	9.90	10.00	8.60	10.00	10.00	11.90	7.40	9.00	8.00	8.60			9.58	2.20	
L13-GD-MS	9.10	8.11	8.69	7.65	8.17	8.64	8.32	9.41	9.24	8.46	8.31	9.12									8.60	0.76	
L14-INAA	10.27	9.77	11.18	11.23	10.23	11.23	9.81	10.95	9.83	9.08	9.61	10.51	8.91	10.37	9.00	9.25		10.43			10.10	2.90	
L16-ICP-OES	8.77	8.87	8.75	9.84	8.53	8.92	8.70	8.94	9.11	8.75	8.56	9.43	8.77	8.85	8.65	8.99	8.67	8.31			8.86	0.53	
L17-ICP-MS	8.55	9.00	9.04	8.36	8.86	8.24	9.03	8.50	8.38	8.98	9.07	8.53	8.55	8.71	8.92	8.98	8.31	9.00			8.72	0.65	
L18-ICP-MS													8.66	8.29	9.78	8.17	8.39	9.87			8.86	1.53	
L19-LA-ICP-MS	9.16	8.98	9.27	9.12	9.19	9.34															9.18	0.25	
L20-ICP-MS													9.20	9.50	9.20	9.00	9.50	9.60			9.33	0.47	
<i>Results not used for certification</i>																							
L1-ICP-OES	10.58	10.41	9.50	11.91	13.67	10.18	18.52	18.73	29.37	16.79	15.02	15.64	9.92	10.44	9.74	10.14	15.13	10.05			13.65	10.08	
L8-ICP-MS	8.70	8.90	8.80	8.60	8.70	8.60	8.60	8.00	8.30	8.70	8.60	8.80	8.30	8.80	8.30	8.50	8.60	8.60			8.58	0.86	
L7-DC-arc-OES													26.00	27.00	31.00	32.00	29.00	29.00			29.00	4.56	
L15-INAA	12.40	6.03	9.91	8.91	38.90	6.06	12.50	12.60	15.10	29.20	11.60	199	15.60	6.98	ND	15.50	5.68	11.70			24.57	4.00	
L21-Spark-OES	7.70	7.60	7.70	7.80	7.90	7.80	7.50	8.40	8.20	7.80	8.30	7.80									7.88	0.56	
L9-ICP-MS	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10					
L10-INAA	< 9	< 8	< 9	< 9	< 7	< 10	21.10	< 8	19.00	< 7	< 8	< 9	< 8	< 9	< 15	< 7	< 9	< 9					

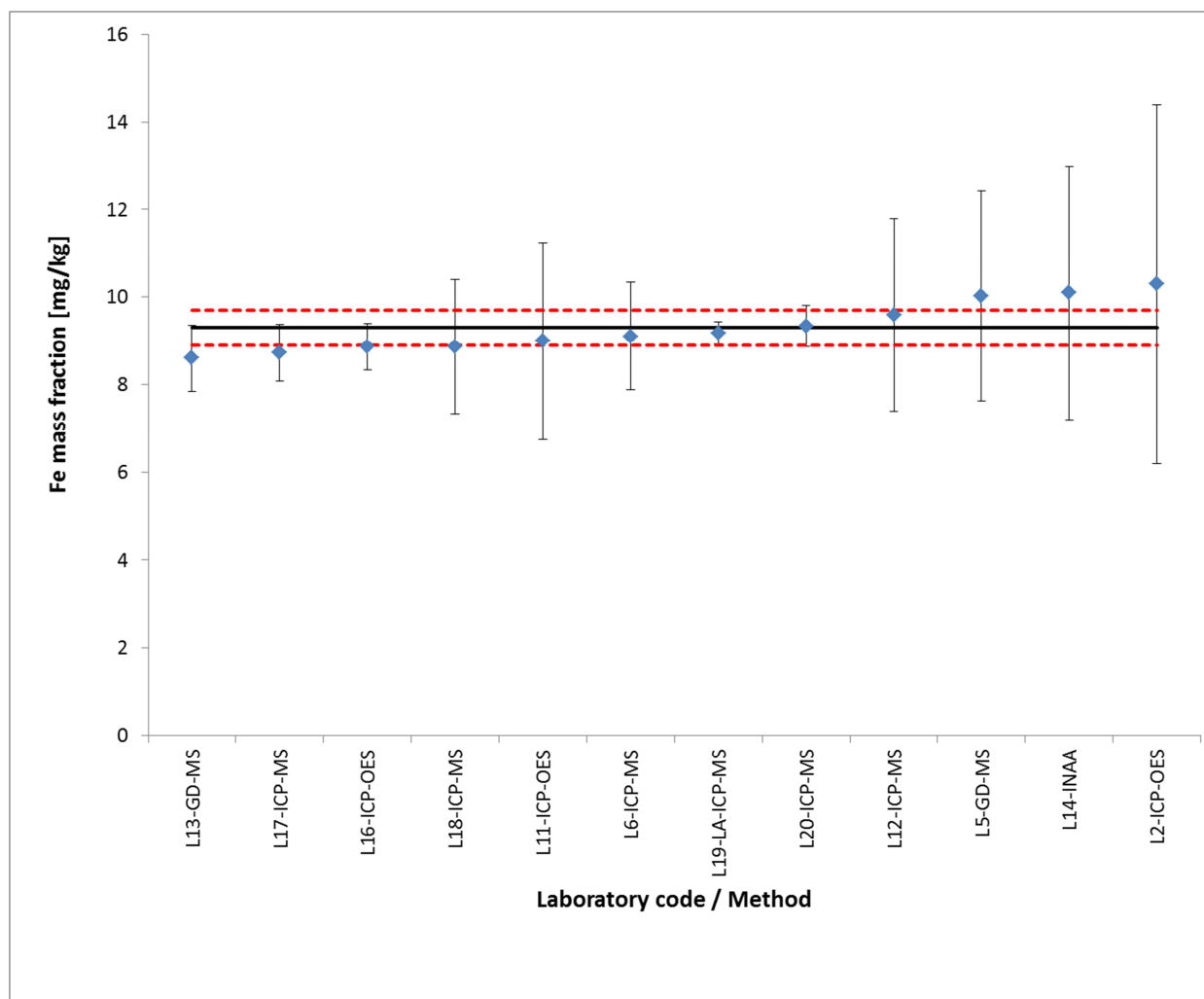


Figure E10. Mean Fe mass fraction in ERM-EB075 reported by participating laboratories. Error bars represent expanded uncertainties. The solid line represents the certified values (the mean of laboratory means), while the broken lines represent the expanded uncertainty of the certified value. Each laboratory is represented by its code.

In (Indium)

Table E11. Individual results for In mass fraction in ERM-EB075 provided by each laboratory. The mean value was calculated as the unweighted mean of all individual results. The expanded uncertainty budget equals to k factor ($k = 2$) multiplied by u provided by the laboratory or is estimated as two times the standard deviation of the measurements (if u is not provided).

Lab code - Method	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Rep 6	Rep 7	Rep 8	Rep 9	Rep 10	Rep 11	Rep 12	Rep 13	Rep 14	Rep 15	Rep 16	Rep 17	Rep 18	Rep 19	Rep 20	Mean	Uncertainty ($k=2$)	
	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]
L1-ICP-MS	1.83	1.80	1.79	1.78	1.78	1.86	1.78	1.76	1.77	1.78	1.79	1.69	1.81	1.82	1.82	1.80	1.84	1.88			1.80	0.08	
L2-ICP-OES	1.50	1.60	1.60	1.50	1.60	1.60	1.50	1.60	1.50	1.50	1.50	1.60	1.50	1.50	1.60	1.60	1.50	1.60			1.55	0.10	
L4-ICP-MS	1.70	1.77	1.77	1.72	1.78	1.70	1.81	1.70	1.69	1.80	1.78	1.71	1.70	1.70	1.78	1.76	1.71	1.78			1.74	0.08	
L5-GD-MS	0.99	1.97	2.25	1.32	1.46	1.67	2.17	1.94	1.98	1.28	1.90	1.65									1.72	0.80	
L8-ICP-MS	1.80	1.90	1.90	1.90	2.00	1.80	1.80	1.90	1.70	1.90	1.80	1.70	1.90	1.90	1.90	1.80	1.90	1.80			1.85	0.19	
L9-ICP-MS	1.81	1.74	1.75	1.80	1.75	1.72	1.88	1.88	1.66	1.76	1.75	1.73	1.72	1.72	1.63	1.72	1.74	1.70			1.75	0.35	
L10-INAA	2.31	2.18	1.82	2.30	1.96	2.16	2.26	1.83	1.98	1.96	1.87	1.89	1.97	1.94	1.99	1.94	1.88	2.06			2.02	0.52	
L11-ICP-MS	1.82	1.82	1.86	1.88	1.84	1.87	1.89	1.86	1.88	1.86	1.86	1.85	1.81	1.86	1.84	1.86	1.87	1.94			1.86	0.47	
L12-ICP-MS	1.67	1.55	1.66	1.68	1.61	1.55	1.53	1.69	1.73	1.61	1.54	1.65	1.66	1.78	1.59	1.54	1.65	1.56			1.63	0.27	
L14-INAA	1.92	1.89	1.87	1.91	1.90	1.88	1.87	1.83	1.86	1.93	1.89	1.82	2.09	1.88	2.03	1.97	1.95	1.99			1.92	0.25	
L16-ICP-MS	2.13	2.18	2.01	1.41	2.05	2.07	2.11	2.12	2.06	2.13	2.07	2.21	2.29	2.09	2.07	2.31	2.03	2.12			2.08	0.33	
L17-ICP-MS	2.03	2.01	2.01	2.05	2.04	2.00	2.02	1.99	2.05	2.04	2.00	2.05	2.03	2.03	2.03	2.00	2.03	2.00			2.02	0.21	
L18-ICP-MS	1.77	1.89	1.83	1.79	1.84	1.80	1.84	1.80	1.77	1.79	1.85	1.78	1.79	1.74	1.90	1.62	1.78	1.82			1.80	0.12	
L20-ICP-MS													1.90	1.90	1.90	1.90	1.90	1.80			1.88	0.08	

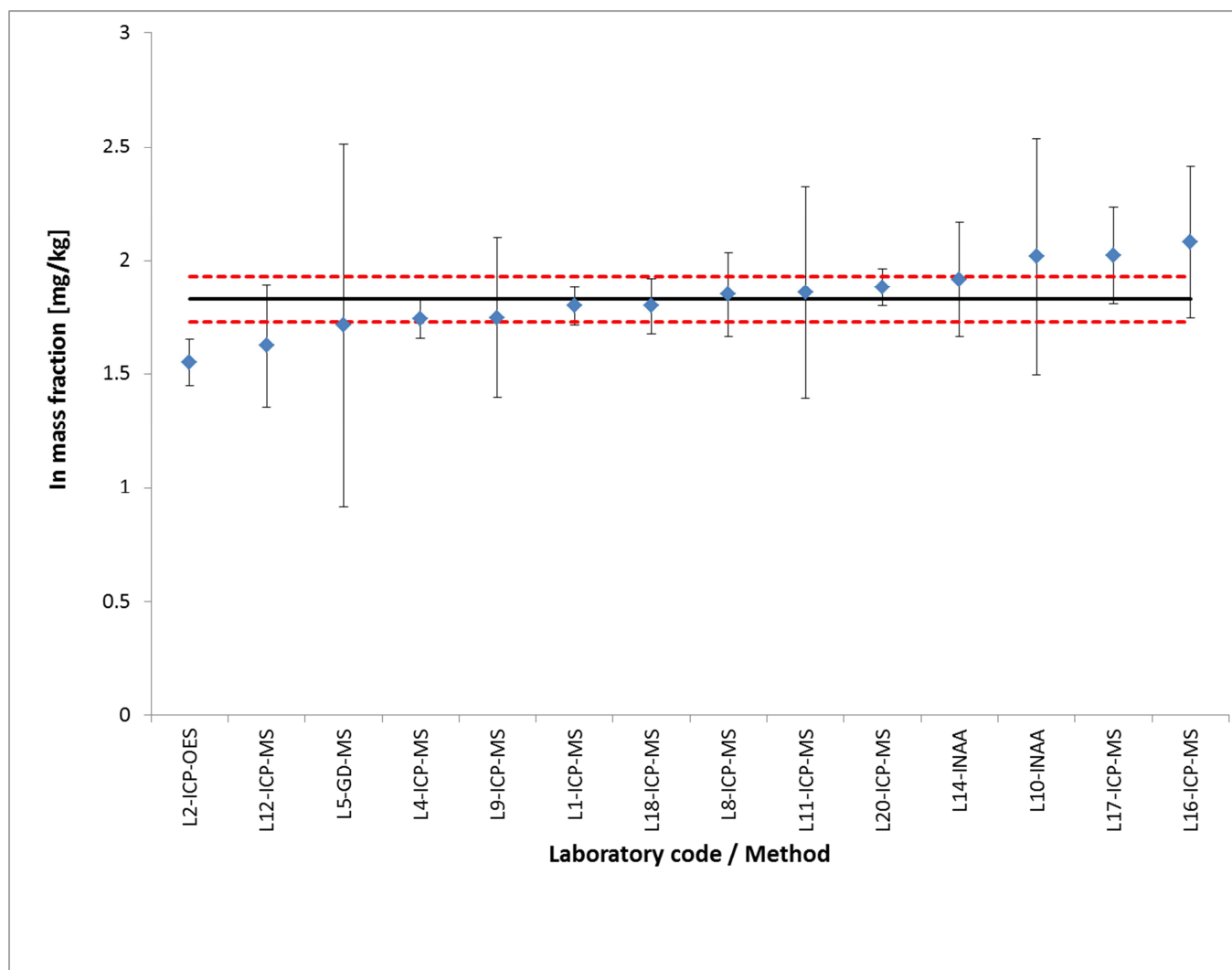


Figure E11. Mean In mass fraction in ERM-EB075 reported by participating laboratories. Error bars represent expanded uncertainties. The solid line represents the certified values (the mean of laboratory means), while the broken lines represent the expanded uncertainty of the certified value. Each laboratory is represented by its code.

Mg (Magnesium)

Table E12. Individual results for Mg mass fraction in ERM-EB075 provided by each laboratory. The mean value was calculated as the unweighted mean of all individual results. The expanded uncertainty budget equals to k factor ($k = 2$) multiplied by u provided by the laboratory or is estimated as two times the standard deviation of the measurements (if u is not provided).

Lab code - Method	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Rep 6	Rep 7	Rep 8	Rep 9	Rep 10	Rep 11	Rep 12	Rep 13	Rep 14	Rep 15	Rep 16	Rep 17	Rep 18	Rep 19	Rep 20	Mean	Uncertainty ($k=2$)	
	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]
L1-ICP-MS	8.30	8.00	8.08	7.57	8.08	8.79	8.50	8.69	9.87	8.09	8.68	7.85	7.90	8.10	8.86	8.26	8.53	8.43			8.36	1.03	
L2-ICP-OES	7.00	6.90	7.00	6.80	6.50	7.00	6.80	7.00	6.90	6.50	6.90	6.90	7.00	6.90	7.00	7.20	6.80	7.20			6.91	0.37	
L4-ICP-MS	7.44	7.25	6.93	7.26	7.24	6.89	7.20	6.98	7.18	0.00	7.57	7.13	7.82	7.10	7.14	7.09	6.99	7.37			6.81	0.48	
L5-GD-MS	6.08	5.16	5.27	6.09	5.81	5.50	5.57	5.52	5.15	6.02	5.56	5.81									5.63	1.20	
L8-ICP-MS	6.00	6.00	5.80	6.20	6.10	6.10	6.20	6.20	5.80	5.90	6.10	6.20	5.90	5.80	6.00	6.20	6.10	5.80			6.02	0.60	
L9-ICP-MS	7.20	7.30	8.10	6.80	6.50	6.80	6.64	6.62	6.85	6.41	7.45	7.34	6.14	6.09	6.78	6.08	6.85	6.96			6.83	1.37	
L11-ICP-MS	7.31	7.22	7.05	7.12	7.12	7.10	7.33	7.16	7.24	6.92	7.28	7.12	7.15	6.95	7.10	7.10	7.21	7.26			7.15	1.79	
L13-GD-MS	7.32	7.47	7.48	6.57	7.36	7.18	7.38	7.41	7.40	7.51	7.41	7.38									7.32	0.57	
L16-ICP-MS	6.81	7.22	6.97	6.10	6.65	6.14	7.12	7.19	6.68	6.47	7.11	7.58	6.53	6.72	6.44	6.89	6.69	6.93			6.79	0.41	
L17-ICP-MS	7.37	7.44	7.41	7.06	7.04	7.20	7.44	7.30	7.25	7.51	7.66	7.48	7.30	7.28	7.48	7.50	7.37	7.31			7.36	0.91	
L18-ICP-MS	8.34	8.33	8.24	8.47	8.02	7.91	7.51	8.28	7.64	7.34	7.39	7.64	7.41	7.03	7.21	6.14	7.44	7.25			7.64	1.17	
L19-LA-ICP-MS	8.02	6.99	7.09	6.59	6.16	7.14															7.00	1.25	
<i>Results not used for certification</i>																							
L20-ICP-MS													8.30	8.40	9.30	8.00	8.30	8.20			8.42	0.91	

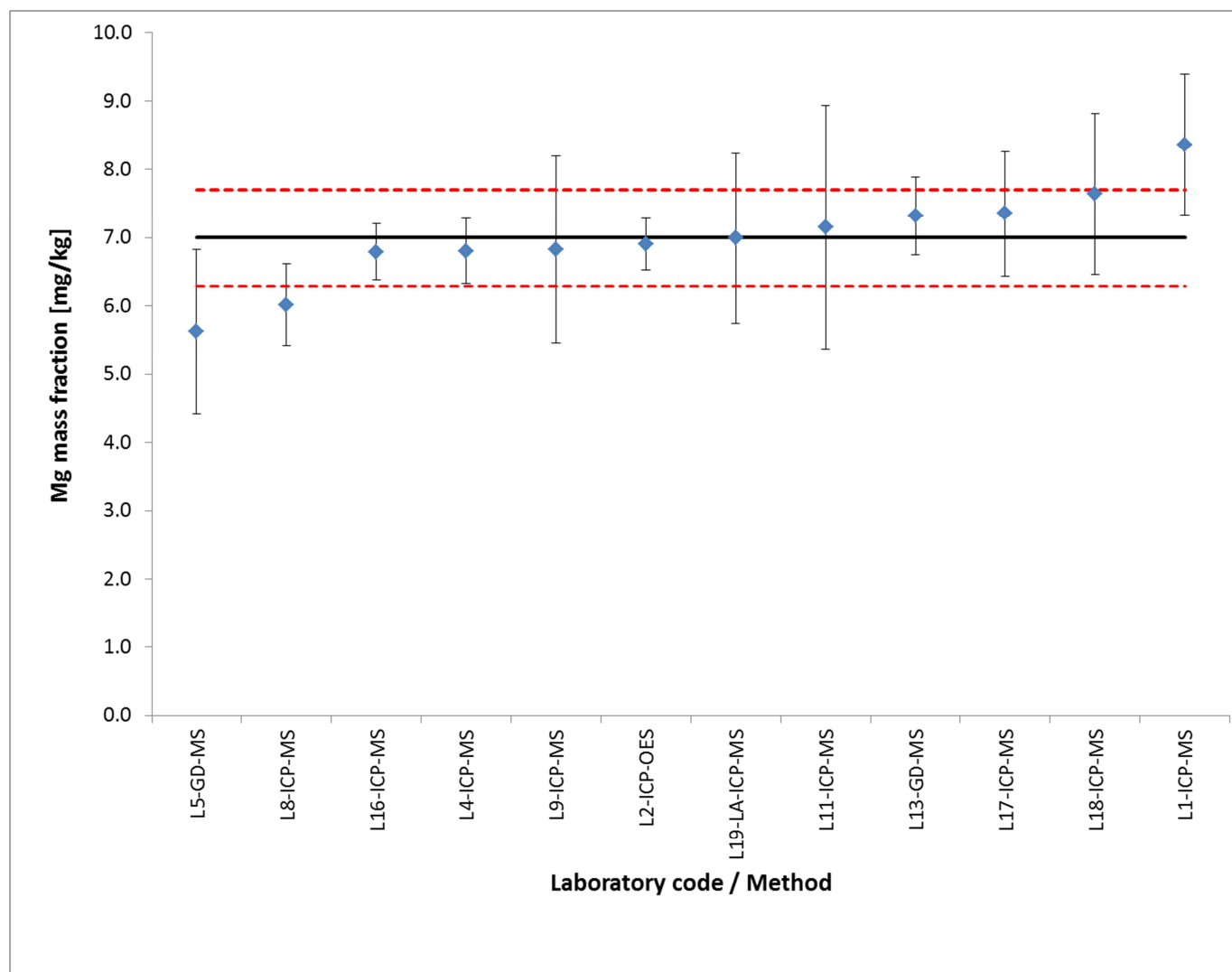


Figure E12. Mean Mg mass fraction in ERM-EB075 reported by participating laboratories. Error bars represent expanded uncertainties. The solid line represents the certified values (the mean of laboratory means), while the broken lines represent the expanded uncertainty of the certified value. Each laboratory is represented by its code.

Mn (Manganese)

Table E13. Individual results for Mn mass fraction in ERM-EB075 provided by each laboratory. The mean value was calculated as the unweighted mean of all individual results. The expanded uncertainty budget equals to k factor ($k = 2$) multiplied by u provided by the laboratory or is estimated as two times the standard deviation of the measurements (if u is not provided).

Lab code - Method	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Rep 6	Rep 7	Rep 8	Rep 9	Rep 10	Rep 11	Rep 12	Rep 13	Rep 14	Rep 15	Rep 16	Rep 17	Rep 18	Rep 19	Rep 20	Mean	Uncertainty (k=2)	
	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]
L1-ICP-MS	1.32	1.34	1.36	1.29	1.39	1.32	1.33	1.32	1.34	1.34	1.35	1.20	1.31	1.29	1.35	1.32	1.32	1.41			1.33	0.09	
L2-ICP-OES	1.40	1.30	1.40	1.30	1.40	1.20	1.40	1.30	1.30	1.30	1.30	1.30	1.30	1.30	1.30	1.40	1.20	1.40			1.32	0.19	
L4-ICP-MS	1.36	1.36	1.24	1.30	1.24	1.32	1.37	1.32	1.35	1.56	1.36	1.35	1.36	1.33	1.41	1.21	1.36	1.26			1.34	0.15	
L6-ICP-OES	1.30	1.30	1.30	1.30	1.30	1.30	1.30	1.30	1.30	1.30	1.30	1.30	1.30	1.30	1.30	1.30	1.30	1.30			1.30	0.00	
L9-ICP-MS	1.21	0.96	1.00	1.25	1.28	1.10	1.25	1.36	1.07	1.34	1.10	1.48	1.41	1.37	1.10	1.37	1.07	1.14			1.21	0.24	
L11-ICP-OES	1.60	1.60	1.60	1.50	1.60	1.50	1.60	1.50	1.50	1.60	1.60	1.60	1.60	1.60	1.60	1.60	1.60	1.60			1.58	0.39	
L13-GD-MS	1.50	1.29	1.35	1.21	1.34	1.31	1.31	1.47	1.52	1.30	1.29	1.46									1.36	0.13	
L16-ICP-OES	1.29	1.29	1.28	1.33	1.29	1.30	1.29	1.30	1.29	1.31	1.28	1.30	1.29	1.28	1.30	1.29	1.29	1.30			1.29	0.03	
L17-ICP-MS	1.33	1.34	1.34	1.28	1.35	1.22	1.35	1.32	1.28	1.37	1.36	1.31	1.32	1.26	1.35	1.35	1.27	1.34			1.32	0.15	
L19-LA-ICP-MS	1.35	1.31	1.33	1.40	1.29	1.27															1.32	0.09	
L20-ICP-MS													1.50	1.50	1.30	1.50	1.40	1.50			1.45	0.17	
<i>Results not used for certification</i>																							
L5-GD-MS	1.07	1.17	1.22	1.15	1.17	1.16	1.22	1.20	1.14	1.13	1.13	1.12									1.16	0.28	
L8-ICP-MS	1.20	1.20	1.20	1.20	1.20	1.30	1.30	1.30	1.20	1.30	1.20	1.30	1.20	1.20	1.20	1.30	1.20	1.20			1.23	0.12	
L18-ICP-MS	1.34	1.44	1.39	1.42	1.24	1.35	1.28	1.26	1.30	1.29	1.22	1.24	1.27	1.20	1.32	1.21	1.21	1.28			1.29	0.14	
L21-Spark-OES	<1.5	<1.5	<1.5	<1.5	<1.5	<1.5	<1.5	<1.5	<1.5	<1.5	<1.5	<1.5											

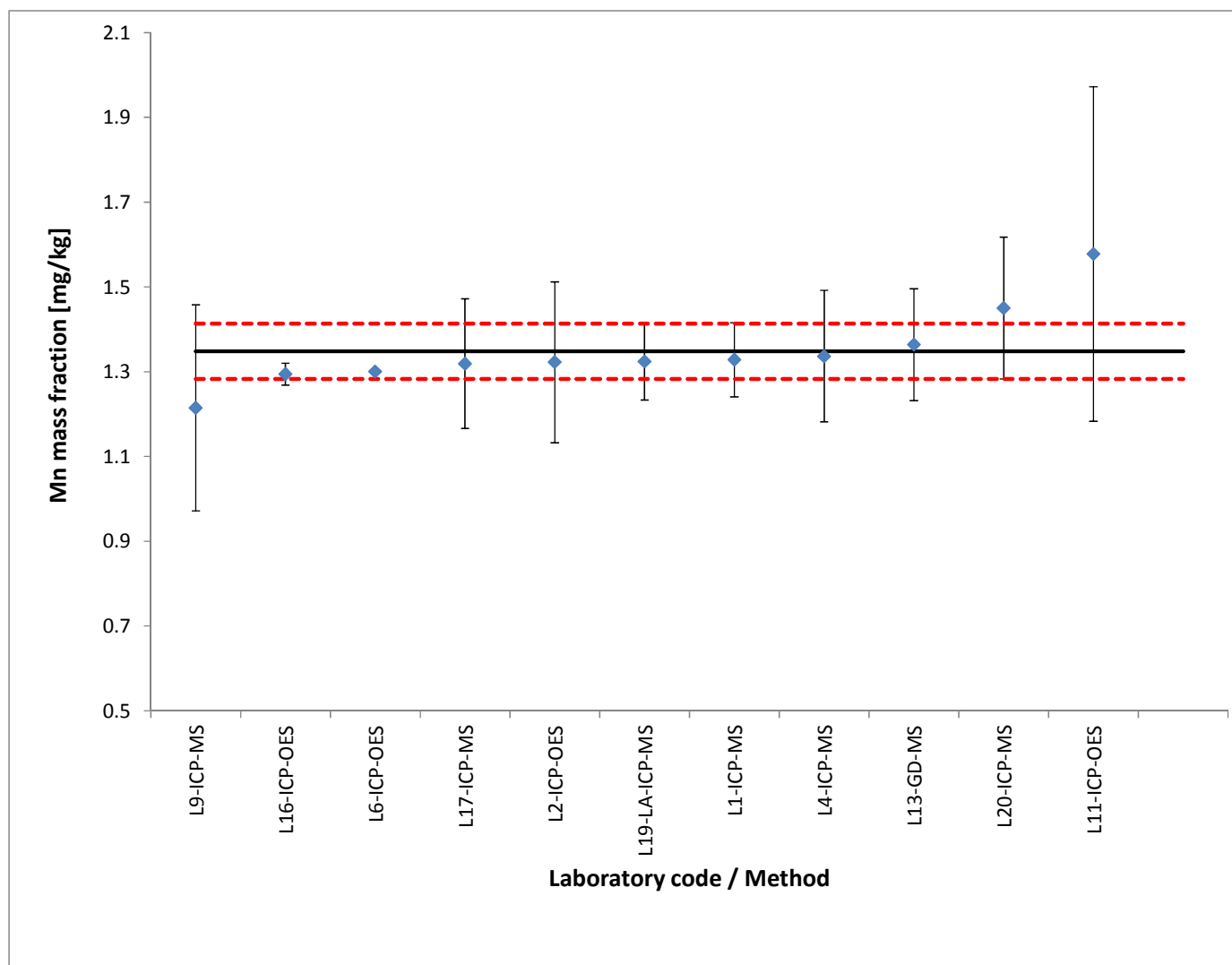


Figure E13. Mean Mn mass fraction in ERM-EB075 reported by participating laboratories. Error bars represent expanded uncertainties. The solid line represents the certified values (the mean of laboratory means), while the broken lines represent the expanded uncertainty of the certified value. Each laboratory is represented by its code.

Ni (Nickel)

Table E14. Individual results for Ni mass fraction in ERM-EB075 provided by each laboratory. The mean value was calculated as the unweighted mean of all individual results. The expanded uncertainty budget equals to k factor ($k = 2$) multiplied by u provided by the laboratory or is estimated as two times the standard deviation of the measurements (if u is not provided).

Lab code - Method	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Rep 6	Rep 7	Rep 8	Rep 9	Rep 10	Rep 11	Rep 12	Rep 13	Rep 14	Rep 15	Rep 16	Rep 17	Rep 18	Rep 19	Rep 20	Mean	Uncertainty ($k=2$)	
	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]
L1-ICP-MS	2.43	2.20	2.18	2.23	2.26	2.27	2.16	2.13	2.16	2.21	2.20	1.91	2.17	2.14	2.25	2.33	0.00	2.30			2.09	0.22	
L2-ICP-OES	2.00	2.00	2.10	2.00	2.30	1.90	2.00	2.00	2.00	2.10	2.20	2.00	2.00	2.00	2.10	2.30	1.90	2.00			2.05	0.23	
L3-ETV-ICP-OES	2.43	2.33	2.59	2.39	2.38	2.27	2.40	2.18	2.26	2.36	2.42	2.33	2.41	2.43	2.54	2.15	2.20	2.14	2.41	2.32	2.35	0.29	
L4-ICP-MS	2.13	2.31	2.22	2.64	2.16	2.00	2.54	2.01	1.98	2.61	2.13		2.11	2.07			2.07	2.18			2.21	0.44	
L5-GD-MS	2.49	2.35	2.30	2.31	2.30	2.44	2.13	2.21	2.33	2.38	2.33	2.47									2.34	0.52	
L6-ICP-OES	2.10	2.40	2.20	2.20	2.80	2.10	2.60	2.00	2.30	2.50	2.50	2.00	1.90	2.00	2.70	2.70	1.90	2.50			2.30	0.59	
L7-DC-arc-OES													2.30	2.10	2.30	2.40	2.40	2.10			2.27	0.27	
L9-ICP-MS	1.96	2.39	2.53	2.18	3.31	2.69	2.03	2.08	2.97	2.04	2.46	2.45	2.06	2.22	2.35	2.77	2.25	2.43			2.40	0.48	
L11-ICP-MS	2.14	1.82	1.82	2.03	1.86	1.81	1.85	1.85	1.80	1.79	1.75	1.76	1.67	1.71	1.69	1.66	1.78	1.73			1.81	0.45	
L12-ICP-MS	2.40	1.80	2.00	2.10	2.00	1.90	1.80	2.10	2.10	2.10	2.30	2.20	1.90	2.20	2.20	2.10	2.20	1.90			2.07	0.30	
L13-GD-MS	2.22	1.97	2.07	1.83	1.96	1.87	1.94	2.15	2.12	1.90	1.91	2.13									2.00	0.15	
L16-ICP-MS	2.17	2.52	2.40	2.00	2.26	1.97	2.34	2.21	2.14	2.25	2.37	2.24	2.06	2.13	2.13	2.93	2.14	2.29			2.25	0.27	
L17-ICP-MS	2.14	2.05	2.02	2.02	1.98	1.96	2.06	2.22	2.13	2.09	2.08	2.15	2.18	2.05	2.12	2.04	2.06	2.05			2.08	0.34	
L19-LA-ICP-MS	2.26	2.24	2.21	2.23	2.17	2.24															2.23	0.06	
L20-ICP-MS													2.20	2.30	2.10	2.20	2.30	2.30			2.23	0.16	
L21-Spark-OES	2.10	2.10	2.20	2.10	2.00	2.20	2.20	2.30	2.20	2.40	2.30	2.30									2.20	0.23	
<i>Results not used for certification</i>																							
L8-ICP-MS	2.00	1.90	1.90	1.90	1.90	2.00	2.00	1.90	1.80	1.90	1.80	1.90	1.90	1.90	1.80	2.00	2.00	1.90			1.91	0.19	
L18-ICP-MS	2.04	2.24	2.14	2.08	2.11	2.06	2.18	1.96	1.96	2.07	2.05	1.98	1.85	1.73	1.98	1.75	1.78	1.90			1.99	0.29	

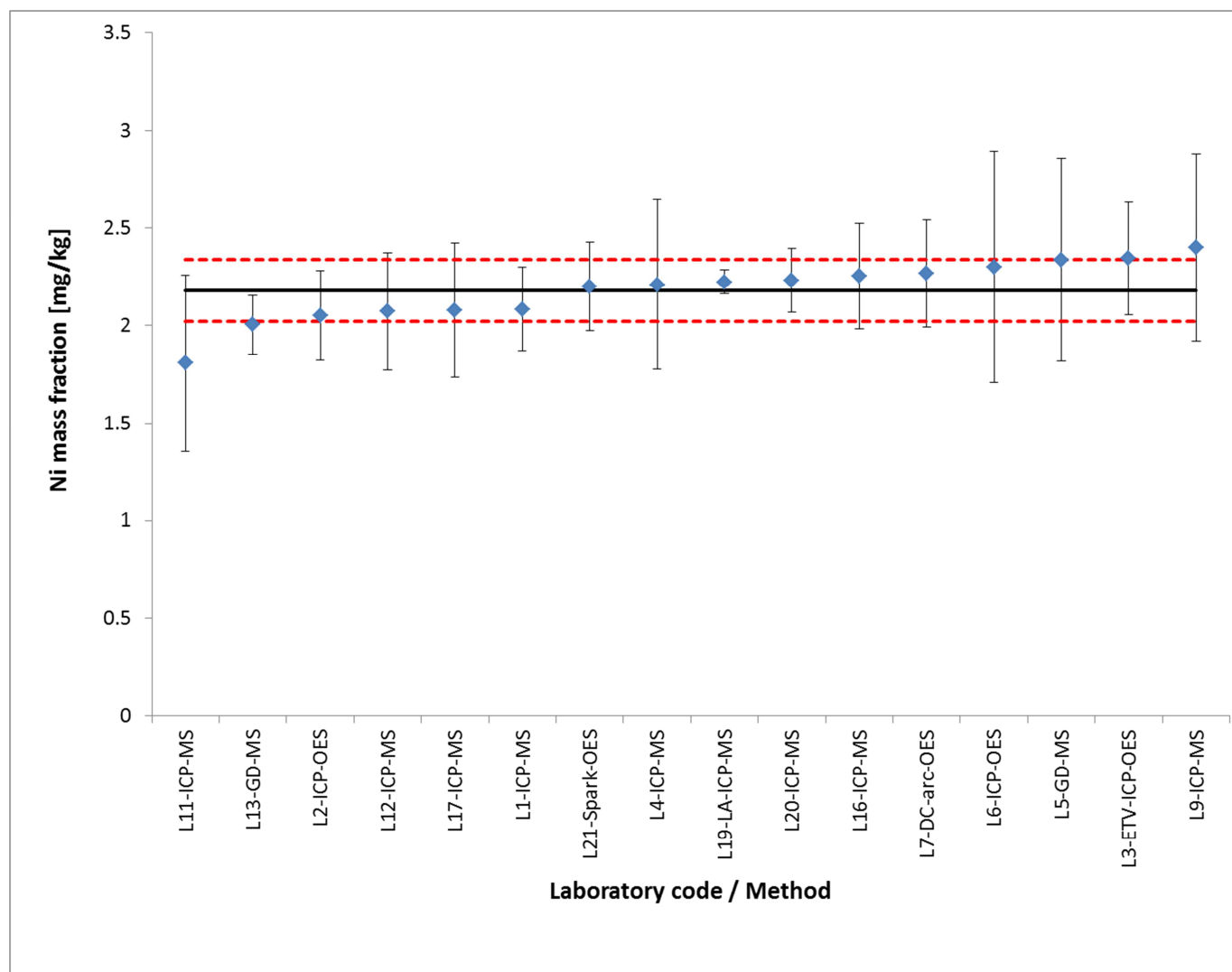


Figure E14. Mean Ni mass fraction in ERM-EB075 reported by participating laboratories. Error bars represent expanded uncertainties. The solid line represents the certified values (the mean of laboratory means), while the broken lines represent the expanded uncertainty of the certified value. Each laboratory is represented by its code.

O (Oxygen)

Table E15. Individual results for O mass fraction in ERM-EB075 provided by each laboratory. The mean value was calculated as the unweighted mean of all individual results. The expanded uncertainty budget equals to k factor ($k = 2$) multiplied by u provided by the laboratory or is estimated as two times the standard deviation of the measurements (if u is not provided).

Lab code - Method	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Rep 6	Rep 7	Rep 8	Rep 9	Rep 10	Rep 11	Rep 12	Rep 13	Rep 14	Rep 15	Rep 16	Rep 17	Rep 18	Rep 19	Rep 20	Mean	Uncertainty ($k=2$)	
	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]
L9-IGF-IR	3.60	3.50	2.20	4.20	3.10	3.50	1.20	2.50	2.10	3.00	1.50	2.40	4.40	3.70	2.40	5.50	5.30	5.10			3.29	0.66	
L11-IGF-IR	2.76	2.77	2.63	2.05	2.22	2.30	1.49	2.10	2.20	2.01	2.34	2.11	1.27	1.07	1.15	1.52	1.72	1.65			1.96	0.49	
L13-GD-MS	4.46	4.84	4.03	4.70	4.74	4.44	5.42	4.97	5.50	5.68	4.83	4.57									4.85	0.82	
<i>Results not used for certification</i>																							
L1	< 10	< 10	11.00	12.00	< 10	14.00	27.00	24.00	124	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10			35.33	87.87	
L17-IGF-IR				3.50		2.00		2.30	1.80			0.80	3.80	3.80			1.00				2.38	n.d.	

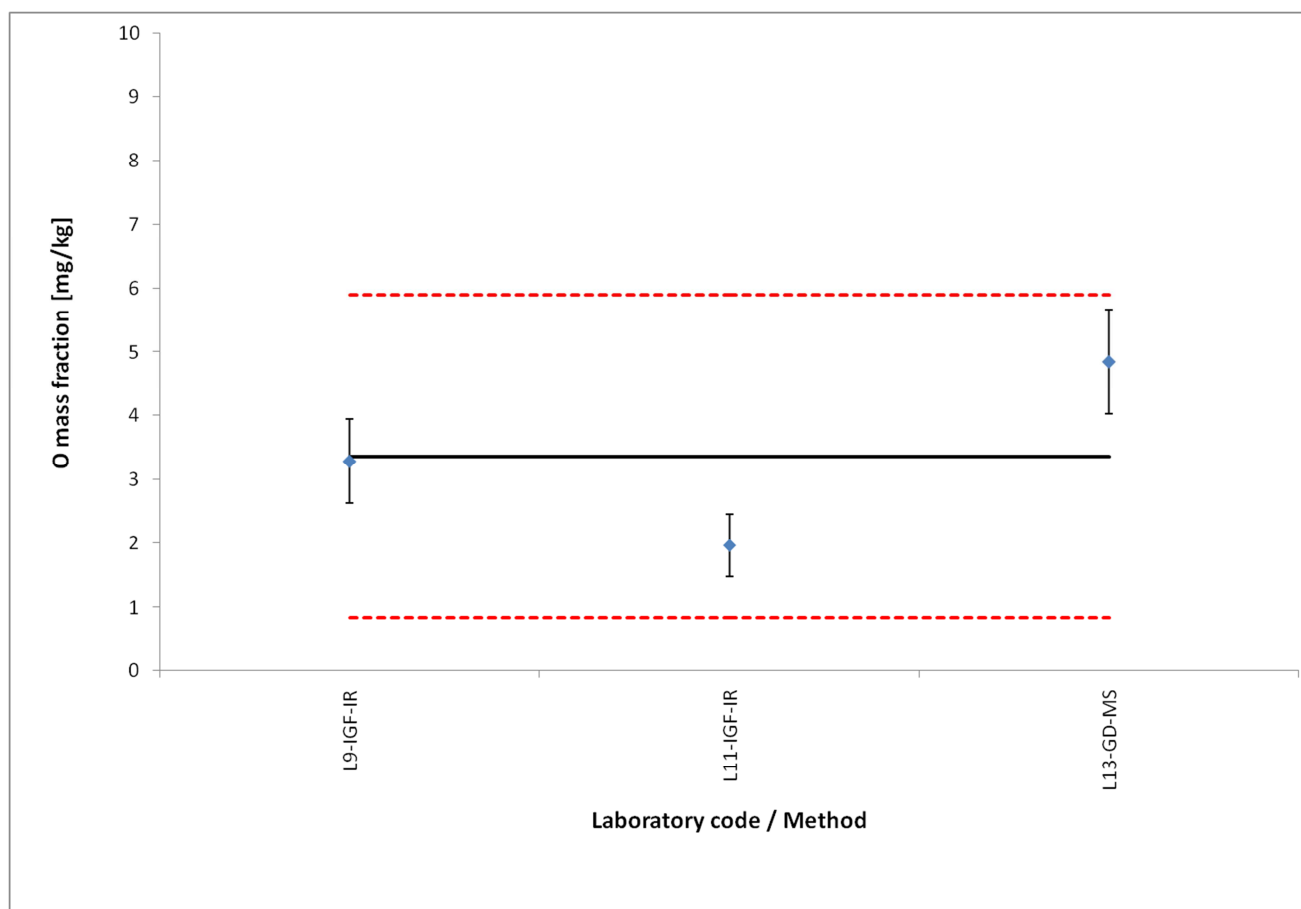


Figure E15. Mean O mass fraction in ERM-EB075 reported by participating laboratories. Error bars represent expanded uncertainties. The solid line represents the certified values (the mean of laboratory means), while the broken lines represent the expanded uncertainty of the certified value. Each laboratory is represented by its code.

P (Phosphorus)

Table E16. Individual results for P mass fraction in ERM-EB075 provided by each laboratory. The mean value was calculated as the unweighted mean of all individual results. The expanded uncertainty budget equals to k factor ($k = 2$) multiplied by u provided by the laboratory or is estimated as two times the standard deviation of the measurements (if u is not provided).

Lab code - Method	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Rep 6	Rep 7	Rep 8	Rep 9	Rep 10	Rep 11	Rep 12	Rep 13	Rep 14	Rep 15	Rep 16	Rep 17	Rep 18	Rep 19	Rep 20	Mean	Uncertainty (k=2)	
	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]
L2-ICP-OES	2.80	2.80	2.90	2.80	2.80	2.80	2.70	2.70	2.80	2.80	2.80	2.80	2.80	2.80	2.80	2.80	2.80	2.70			2.79	0.10	
L4-ICP-MS	2.44	2.60	2.57	2.72	2.77	2.88	2.31	1.67	2.73	2.05	2.99	2.05	2.84	2.75	2.19	2.40	3.06	2.58			2.53	0.73	
L5-GD-MS	2.77	1.34	1.32	2.39	2.13	1.59	1.61	1.69	1.30	2.20	1.50	1.71									1.80	1.20	
L8-ICP-MS	2.40	2.40	2.60	2.50	2.40	2.40	2.50	2.30	2.50	2.60	2.20	2.40	2.30	2.50	2.50	2.40	2.50	2.40			2.43	0.24	
L11-ICP-MS	2.80	2.50	2.70	2.90	2.70	2.80	2.60	2.70	2.90	2.60	2.80	2.80	2.80	2.70	2.80	2.70	2.90	2.90			2.76	0.69	
L12-ICP-MS	2.70	2.70	3.10	2.90	3.30	3.10	3.30	3.60	3.30	3.70	3.00	3.00	3.10	3.90	3.20	3.30	3.40	3.30			3.22	0.40	
L13-GD-MS	2.25	2.35	2.37	3.34	3.32	3.52	2.49	2.30	2.38	2.49	2.62	2.39									2.65	0.44	
L16-ICP-MS	1.91	2.05	2.07	0.88	2.65	1.90	2.05	2.43	1.93	2.11	2.09	1.79	1.65	2.06	2.10	1.98	1.87	1.91			1.97	0.47	
L17-ICP-OES	2.68	2.87	2.92	2.98	2.82	2.80	2.92	2.82	2.89	2.85	2.76	2.88	2.64	2.77	2.76	2.83	2.72	2.86			2.82	0.41	
L21-Spark-OES	2.60	3.30	2.70	2.50	3.10	2.80	2.90	3.10	3.00	3.40	3.10	3.20									2.98	0.56	
L2-ICP-OES	2.80	2.80	2.90	2.80	2.80	2.80	2.70	2.70	2.80	2.80	2.80	2.80	2.80	2.80	2.80	2.80	2.80	2.70			2.79	0.10	
L4-ICP-MS	2.44	2.60	2.57	2.72	2.77	2.88	2.31	1.67	2.73	2.05	2.99	2.05	2.84	2.75	2.19	2.40	3.06	2.58			2.53	0.73	
<i>Results not used for certification</i>																							
L1-ICP-OES	< 10	< 10	< 10	< 10	< 10	< 10	< 10	10.00	16.00	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10					
L6-ICP-OES	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5					
L9-ICP-MS	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10					

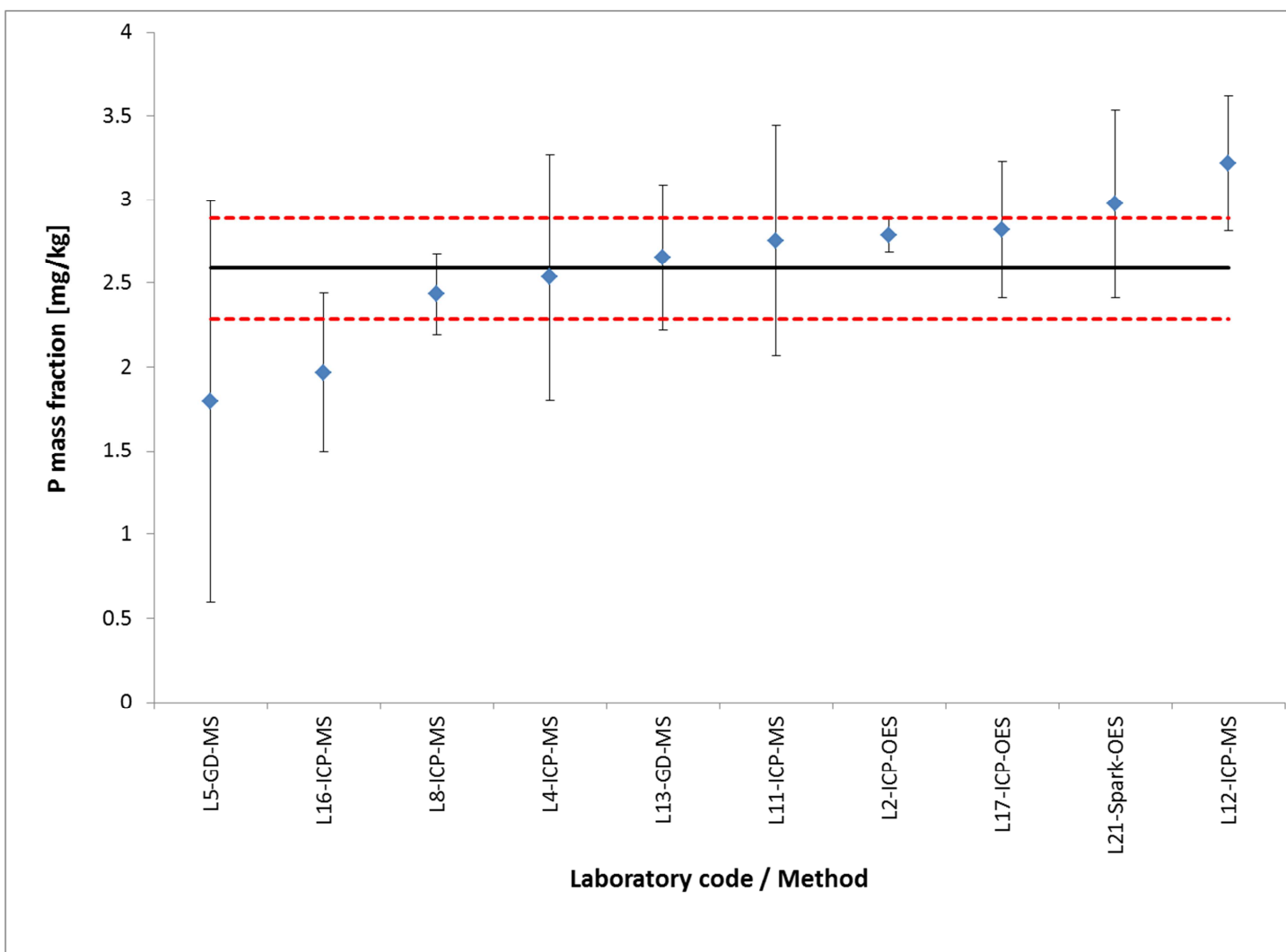


Figure E16. Mean P mass fraction in ERM-EB075 reported by participating laboratories. Error bars represent expanded uncertainties. The solid line represents the certified values (the mean of laboratory means), while the broken lines represent the expanded uncertainty of the certified value. Each laboratory is represented by its code.

Pb (Lead)

Table E17. Individual results for Pb mass fraction in ERM-EB075 provided by each laboratory. The mean value was calculated as the unweighted mean of all individual results. The expanded uncertainty budget equals to k factor ($k = 2$) multiplied by u provided by the laboratory or is estimated as two times the standard deviation of the measurements (if u is not provided).

Lab code - Method	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Rep 6	Rep 7	Rep 8	Rep 9	Rep 10	Rep 11	Rep 12	Rep 13	Rep 14	Rep 15	Rep 16	Rep 17	Rep 18	Rep 19	Rep 20	Mean	Uncertainty ($k=2$)	
	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]
L3-ETV-ICP-OES	5.12	5.08	5.06	5.04	5.01	4.83	4.91	4.91	4.81	4.88	5.30	5.13	5.17	5.17	5.12	4.92	4.83	4.82	4.89	4.97	5.01	0.34	
L4-ICP-MS	5.25	5.34	5.31	4.67	4.80	4.67	4.53	4.37	4.44	4.52	4.58	4.44	4.51	4.57	4.56	4.54	4.48	4.57			4.68	0.61	
L5-GD-MS	3.10	5.38	5.98	3.73	4.25	4.29	5.60	5.33	5.01	3.66	4.72	4.09									4.60	1.40	
L6-ICP-MS	6.10	5.80	6.10	5.40	5.50	5.50	5.90	5.90	5.60	5.80	5.70	5.70	5.70	5.50	5.50	5.70	5.80	5.80			5.72	0.40	
L7-DC-arc-OES													5.10	4.80	4.70	4.70	5.20	5.40			4.98	0.59	
L9-ICP-MS	4.92	5.40	5.55	5.13	5.34	5.40	4.59	4.67	5.18	4.70	5.22	5.29	4.75	5.09	5.28	5.25	4.79	5.06			5.09	1.02	
L11-ICP-MS	4.91	4.84	4.92	4.85	4.78	4.81	4.50	4.44	4.54	4.52	4.46	4.73	4.52	4.49	4.51	4.53	4.72	4.77			4.66	1.16	
L12-ICP-MS	4.86	4.65	4.78	3.98	4.51	4.17	4.36	4.23	4.25	4.38	4.35	4.08	4.75	4.53	4.60	4.42	4.70	4.32			4.44	0.67	
L13-GD-MS	6.19	4.94	5.40	3.81	4.07	4.01	4.96	5.44	5.14	4.24	4.11	5.27									4.80	0.71	
L16-ICP-MS	4.91	5.19	5.26	4.22	4.32	4.14	4.94	4.85	4.78	4.58	4.83	4.21	4.60	4.86	4.82	4.79	4.88	4.79			4.72	0.28	
L17-ICP-MS	5.09	5.16	5.18	4.18	4.22	4.19	4.70	4.56	4.67	4.72	4.64	4.68	4.62	4.61	4.66	4.67	4.64	4.89			4.67	0.91	
L19-LA-ICP-MS	4.91	4.74	4.81	5.02	4.73	4.74															4.83	0.24	
<i>Results not used for certification</i>																							
L1-ICP-MS	5.64	4.97	4.95	4.60	4.27	4.98	4.73	5.03	5.25	4.57	4.63	4.92	4.94	4.97	4.51	4.43	4.98	4.55			4.83	0.65	
L2-ICP-OES	4.60	4.30	4.20	3.40	3.50	3.40	3.80	4.10	3.80	4.00	3.90	4.00	3.90	3.80	3.70	3.80	3.80	3.70			3.87	0.61	
L8-ICP-MS	4.60	4.50	4.50	4.50	4.40	4.60	4.50	4.70	4.60	4.40	4.50	4.50	4.70	4.70	4.70	4.60	4.50	4.60			4.56	0.46	
L18-ICP-MS	4.67	4.75	4.52	4.20	4.37	4.28	4.45	4.40	4.35	4.25	4.17	4.33	4.11	4.00	4.20	4.14	4.11	4.35			4.31	0.39	
L20-ICP-MS													7.70	4.20	8.50	6.00	8.00	6.50			6.82	3.18	
L21-Spark-OES	5.80	6.20	6.70	5.70	6.10	5.70	5.70	5.90	5.80	6.10	5.10	5.60									5.87	0.78	

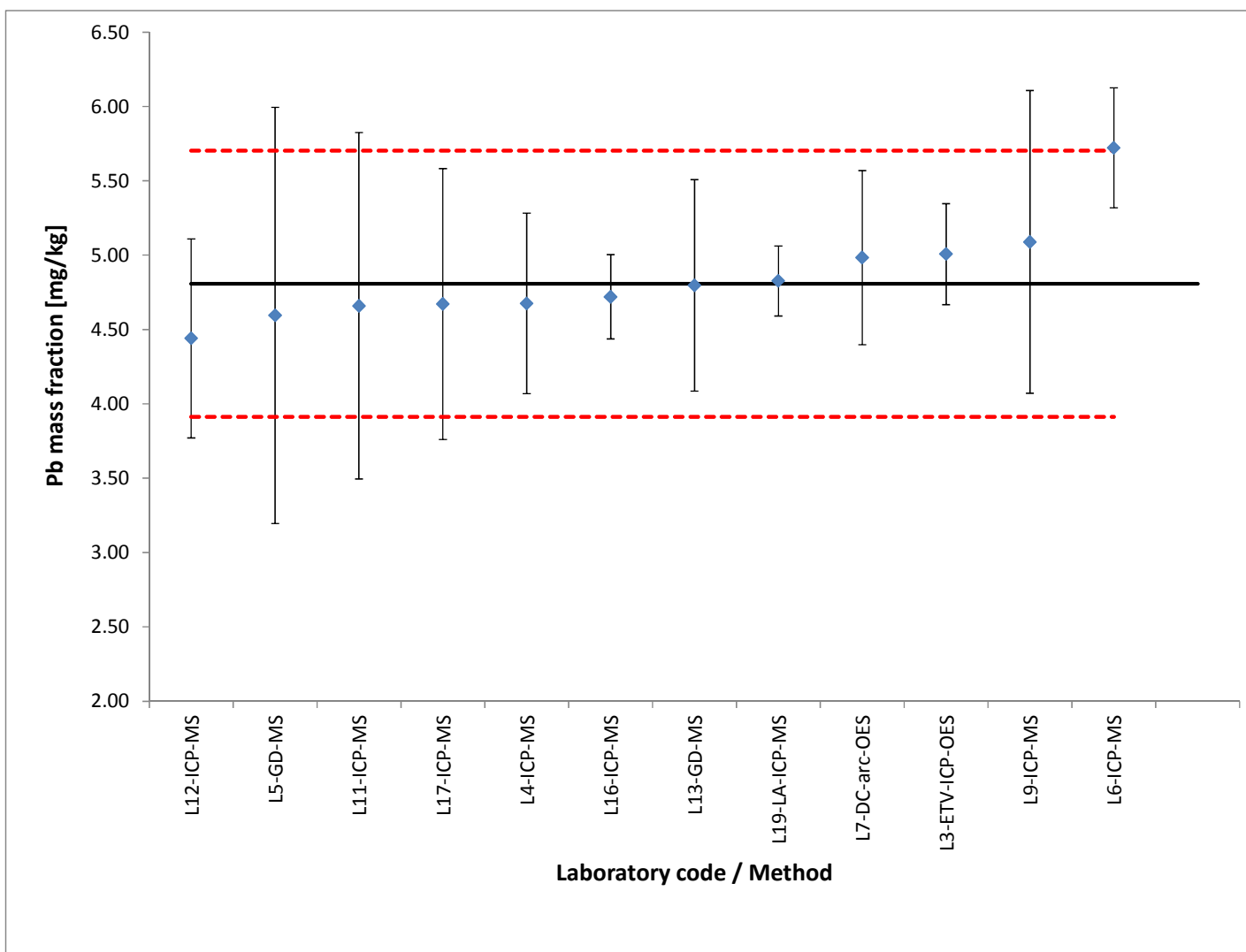


Figure E17. Mean Pb mass fraction in ERM-EB075 reported by participating laboratories. Error bars represent expanded uncertainties. The solid line represents the certified values (the mean of laboratory means), while the broken lines represent the expanded uncertainty of the certified value. Each laboratory is represented by its code.

S (Sulphur)

Table E18. Individual results for S mass fraction in ERM-EB075 provided by each laboratory. The mean value was calculated as the unweighted mean of all individual results. The expanded uncertainty budget equals to k factor ($k = 2$) multiplied by u provided by the laboratory or is estimated as two times the standard deviation of the measurements (if u is not provided).

Lab code - Method	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Rep 6	Rep 7	Rep 8	Rep 9	Rep 10	Rep 11	Rep 12	Rep 13	Rep 14	Rep 15	Rep 16	Rep 17	Rep 18	Rep 19	Rep 20	Mean	Uncertainty (k=2)	
	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]
L1-C-IR	20.00	19.00	19.00	20.00	17.00	25.00	20.00	22.00	21.00	15.00	20.00	22.00	12.00	18.00	18.00	17.00	23.00	17.00			19.17	6.07	
L2-spark-OES	25.97	25.23	25.07	24.30	26.27	25.50	25.57	25.50														25.43	1.19
L5-GD-MS	26.50	19.20	19.10	24.80	23.20	21.60	20.40	20.60	19.20	24.10	20.90	22.20										21.82	9.60
L7-DC-arc-OES													28.00	29.00	28.00	28.00	29.00	23.00				27.50	4.52
L9-IGF-IR	28.00	25.00	25.00	28.00	27.00	27.00	28.00	30.00	28.00	29.00	28.00	30.00	33.00	29.00	31.00	30.00	31.00	29.00				28.67	5.73
L11-IGF-IR	19.88	19.64	19.93	20.42	21.07	21.14	21.02	21.36	20.17	20.92	20.43	20.36	21.46	20.22	21.17	21.12	20.61	21.21				20.67	5.17
L13-GD-MS	25.61	24.23	28.55	29.04	29.59	30.36	25.26	25.35	25.80	26.51	24.71	26.44										26.79	5.16
L21-Spark-OES	31.00	33.10	32.30	30.50	32.10	33.00	33.30	31.80	35.00	35.40	31.60	30.90										32.50	3.09
<i>Results not used for certification</i>																							
L4-ICP-MS	26.30	10.00	15.00	14.00	23.50	11.30	23.70	29.40	33.60	18.50	13.60	27.80	17.00	14.60	23.20	18.70	26.30	17.30				20.21	13.51

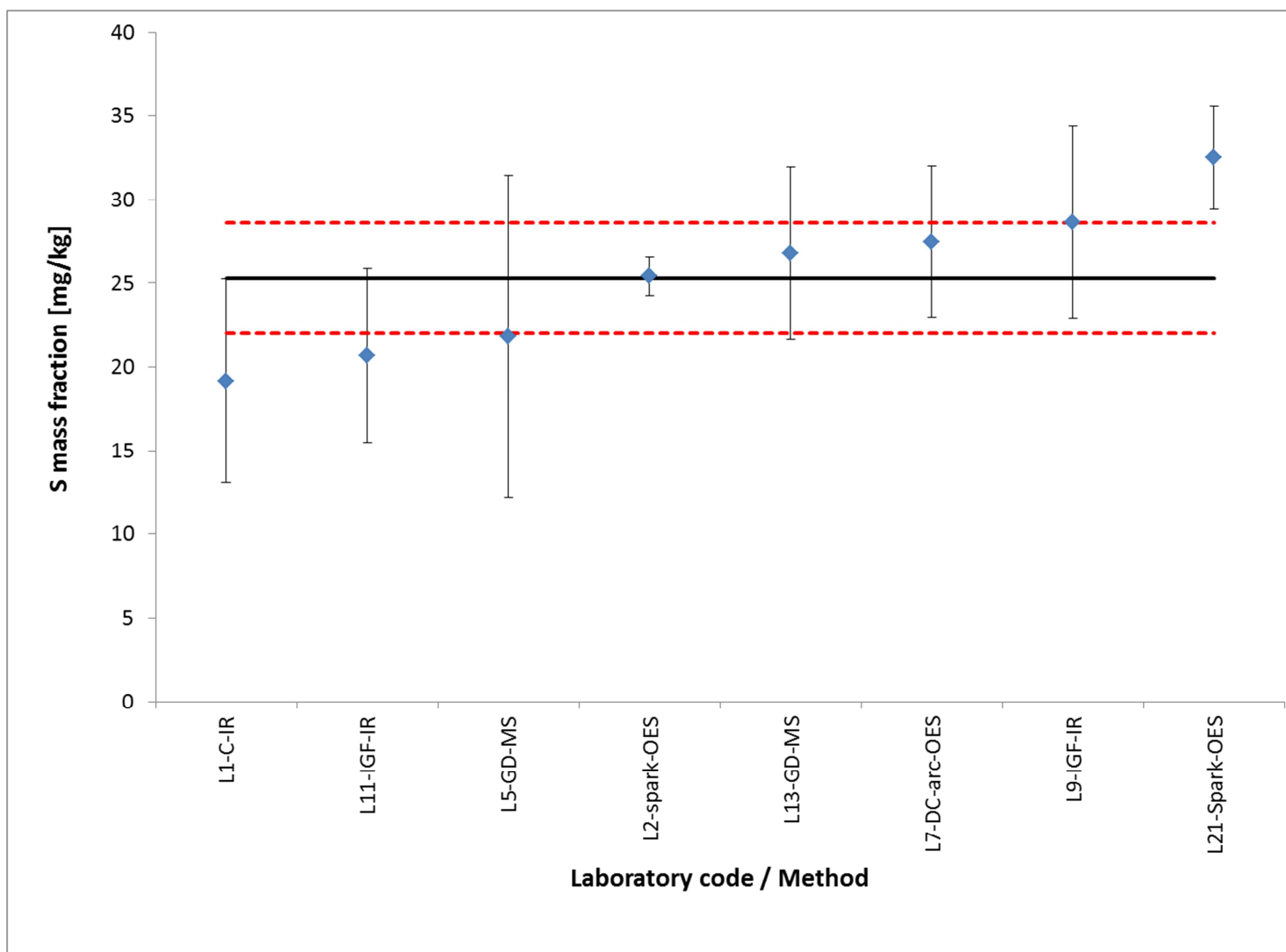


Figure E18. Mean S mass fraction in ERM-EB075 reported by participating laboratories. Error bars represent expanded uncertainties. The solid line represents the certified values (the mean of laboratory means), while the broken lines represent the expanded uncertainty of the certified value. Each laboratory is represented by its code.

Sb (Antimony)

Table E19. Individual results for Sb mass fraction in ERM-EB075 provided by each laboratory. The mean value was calculated as the unweighted mean of all individual results. The expanded uncertainty budget equals to k factor ($k = 2$) multiplied by u provided by the laboratory or is estimated as two times the standard deviation of the measurements (if u is not provided).

Lab code - Method	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Rep 6	Rep 7	Rep 8	Rep 9	Rep 10	Rep 11	Rep 12	Rep 13	Rep 14	Rep 15	Rep 16	Rep 17	Rep 18	Rep 19	Rep 20	Mean	Uncertainty ($k=2$)	
	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]
L1-ICP-MS	2.30	3.01	2.88	2.69	2.52	3.04	3.01	2.93	2.65	3.02	2.92	2.93	2.84	2.99	3.01	2.94	2.99	3.00			2.87	0.40	
L2-ICP-OES	2.70	2.60	2.70	2.70	2.80	2.90	2.70	2.80	2.80	2.50	2.60	2.80	2.70	2.70	2.70	2.70	2.90	2.70			2.72	0.25	
L4-ICP-MS	2.83	2.99	2.96	2.90	3.02	2.86	3.10	2.79	2.80	3.02	2.94	2.84	2.87	2.84	3.05	2.96	2.87	3.02			2.93	0.19	
L6-ICP-MS	3.10	2.80	2.90	3.00	3.00	3.00	3.10	2.90	2.90	3.00	3.00	3.10	3.10	2.90	2.90	2.90	3.00	2.90			2.97	0.18	
L7-DC-arc-OES													3.20	2.90	3.20	2.80	3.00	3.00			3.02	0.32	
L8-ICP-MS	2.70	2.70	2.70	2.60	2.50	2.60	2.70	2.70	2.50	2.70	2.80	2.70	2.80	2.70	2.80	2.60	2.60	2.70			2.67	0.27	
L9-ICP-MS	2.41	2.78	2.77	2.40	2.60	2.82	2.72	2.70	2.77	2.44	2.88	2.81	2.59	2.46	2.70	2.64	2.69	2.67			2.66	0.53	
L10-INAA	3.12	3.16	3.15	3.21	3.14	3.13	3.12	3.12	3.18	3.16	3.13	3.15	3.14	3.15	3.19	3.13	3.19	3.19			3.15	0.24	
L11-ICP-MS	2.23	2.29	2.15	2.16	2.26	2.11	2.28	2.15	2.34	2.22	2.21	2.34	2.31	2.30	2.17	2.19	2.32	2.23			2.24	0.56	
L13-GD-MS	3.38	3.04	3.02	3.06	3.29	3.28	2.97	3.36	3.15	2.98	2.96	3.29									3.15	0.25	
L14-INAA	2.80	2.71	2.88	2.86	2.79	2.83	2.86	2.84	2.74	2.67	2.88	2.83	2.82	2.92	2.84	2.87	2.78	2.94			2.83	0.25	
L15-INAA	2.95	2.96	2.92	3.08	3.03	3.04	3.12	3.05	3.10	3.14	3.11	3.11	3.27	3.26	3.25	3.19	3.28	3.18			3.11	0.05	
L16-ICP-MS	2.96	3.67	3.48	2.90	3.42	2.88	3.30	3.04	3.02	3.29	3.41	2.29	2.91	2.99	3.03	3.28	3.05	3.33			3.12	0.44	
L17-ICP-MS	3.28	3.27	3.21	3.32	3.39	3.25	3.33	3.21	3.30	3.35	3.26	3.32	3.27	3.13	3.31	3.21	3.26	3.25			3.27	0.38	
L18-ICP-MS	2.91	3.10	3.04	2.98	2.95	3.02	2.95	2.84	2.84	2.98	2.85	2.85	3.29	3.18	3.24	2.88	2.95	3.12			3.00	0.28	
L19-LA-ICP-MS	2.82	2.82	2.82	3.03	2.95	2.81															2.87	0.18	
L20-ICP-MS													2.90	3.10	3.20	2.80	3.10	2.90			3.00	0.31	
L21-Spark-OES	2.90	3.50	2.90	2.70	3.20	3.40	2.30	3.40	2.90	3.40	3.40	2.90									3.08	0.73	
<i>Results not used for certification</i>																							
L3-ETV-ICP-OES	2.32	2.27	2.46	2.38	2.30	2.14	2.03	2.39	2.27	2.14	2.30	2.42	2.29	2.29	2.28	2.26	2.17	2.27	2.14	2.24	2.28	0.28	
L5-GD-MS	2.00	2.68	2.85	2.31	2.46	2.48	3.05	2.93	2.75	2.31	2.71	2.54									2.59	0.94	

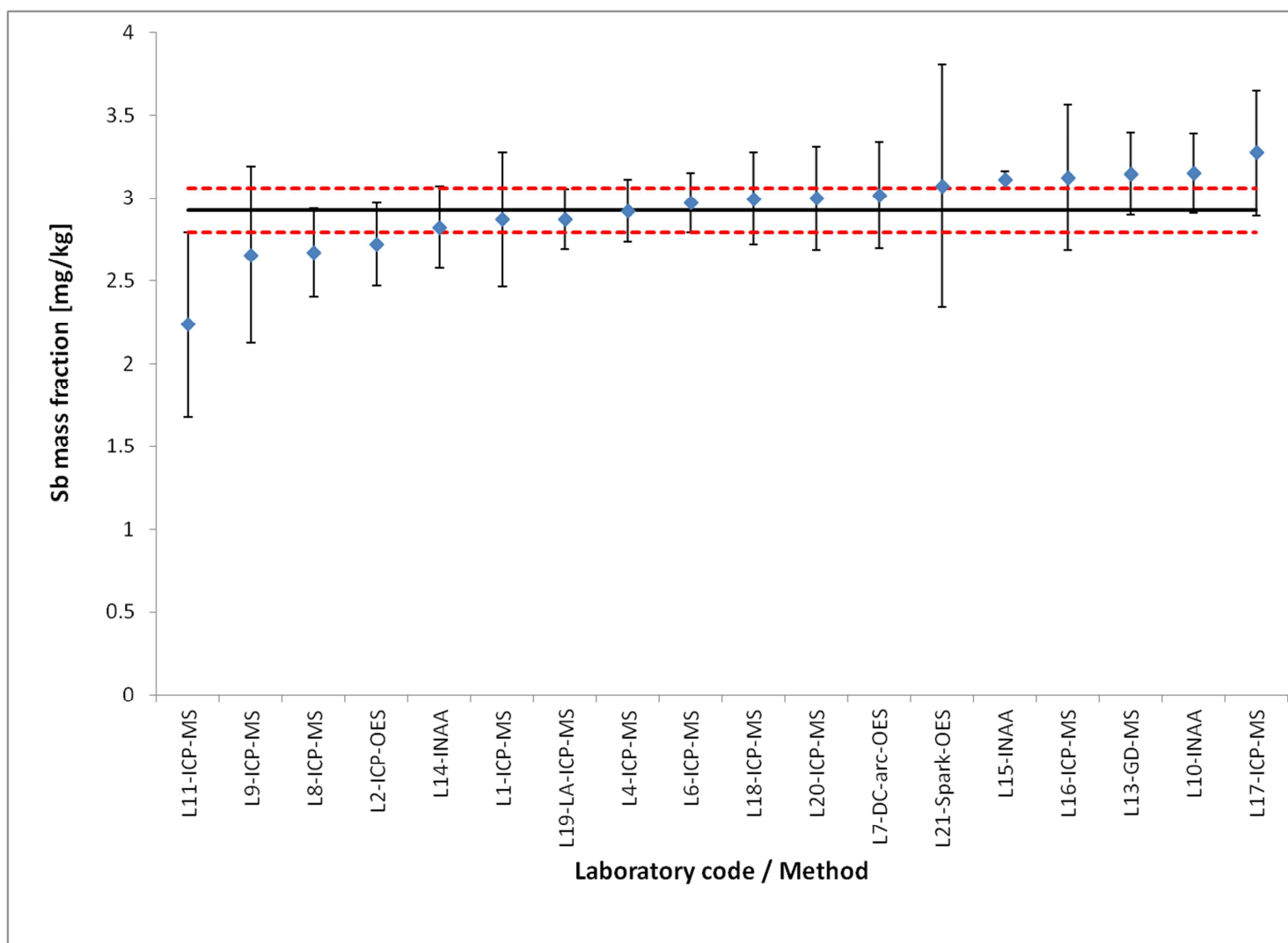


Figure E19. Mean Sb mass fraction in ERM-EB075 reported by participating laboratories. Error bars represent expanded uncertainties. The solid line represents the certified values (the mean of laboratory means), while the broken lines represent the expanded uncertainty of the certified value. Each laboratory is represented by its code.

Se (Selenium)

Table E20. Individual results for Se mass fraction in ERM-EB075 provided by each laboratory. The mean value was calculated as the unweighted mean of all individual results. The expanded uncertainty budget equals to k factor ($k = 2$) multiplied by u provided by the laboratory or is estimated as two times the standard deviation of the measurements (if u is not provided).

Lab code - Method	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Rep 6	Rep 7	Rep 8	Rep 9	Rep 10	Rep 11	Rep 12	Rep 13	Rep 14	Rep 15	Rep 16	Rep 17	Rep 18	Rep 19	Rep 20	Mean	Uncertainty ($k=2$)	
	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]
L2-ICP-OES	1.70	1.50	1.50	2.00	2.00	1.70	1.20	1.70	1.70	1.50	1.30	1.60	2.00	1.90	1.80	1.90	1.60	1.80			1.69	0.46	
L3-ETV-ICP-OES	1.46	1.44	1.93	1.71	1.68	1.88	1.86	2.02	1.99	2.11	1.18	1.27	1.36	1.37	1.50	1.65	1.60	1.59	1.59	1.84	1.64	0.50	
L5-GD-MS	1.38	1.85	1.96	1.51	1.52	1.80	1.72	1.68	1.89	1.39	1.85	1.86									1.70	0.70	
L6-ICP-MS	1.90	1.80	1.90	1.80	2.10	1.90	1.90	1.80	1.80	1.90	1.80	1.90	1.90	1.80	1.80	1.80	1.80	1.90			1.86	0.16	
L7-DC-arc-OES													2.50	1.50	1.50	1.20	1.00	1.00			1.45	1.12	
L9-ICP-MS	1.32	1.48	1.46	1.25	1.27	1.52	1.29	1.23	1.37	1.27	1.42	1.38	1.40	1.34	1.63	1.37	1.61	1.56			1.40	0.28	
L10-INAA	1.97	2.06	1.87	2.05	1.85	2.07	1.85	1.98	2.07	1.90	2.08	1.99	2.09	1.81	2.14	1.83	2.08	2.15			1.99	0.38	
L13-GD-MS	1.68	1.82	1.87	1.91	1.92	2.05	1.96	1.70	1.75	1.92	1.95	1.83									1.86	0.17	
L14-INAA	1.87	1.82	1.79	1.85	1.87	1.93	1.85	1.89	1.94	1.77	1.86	1.86	1.86	1.77	1.87	1.94	1.84	1.91			1.86	0.24	
L15-INAA	1.65	1.59	1.66	1.75	1.83	1.62	1.78	1.84	1.74	1.67	1.64	1.63	1.83	1.84	1.78	1.75	2.00	1.79			1.74	0.07	
L16-ICP-MS	1.61	1.65	1.63	1.62	1.42	1.66	1.59	1.51	1.81	1.47	1.68	1.54	1.59	1.77	1.65	1.59	1.69	1.52			1.61	0.35	
L17-ICP-MS	1.58	1.83	1.51	1.73	1.69	1.66	1.75	1.69	1.66	1.68	1.60	1.71	1.64	1.45	1.58	1.59	1.61	1.58			1.64	0.29	
L18-ICP-MS	1.91	1.81	1.78	2.02	1.88	1.95	1.69	1.80	1.78	1.78	1.85	1.85	1.82	1.75	1.82	1.75	1.85	1.74			1.82	0.16	
L19-LA-ICP-MS	1.77	1.92	1.91	1.60	1.45	1.35															1.67	0.47	
L21-Spark-OES	1.50	1.80	1.80	1.70	1.00	1.40	1.50	1.30	1.40	1.20	1.50	1.40									1.46	0.47	
<i>Results not used for certification</i>																							
L1-INAA	0.93	2.01	1.90	1.20	1.68	1.19	1.98	1.03	1.23	1.70	2.14	1.07	1.51	1.41	2.24	1.86	1.73	2.75			1.64	0.98	
L4-ICP-MS	1.35	1.32	1.15	1.56	1.29	1.05	1.97	1.39	1.09	1.81	1.44	1.37	1.09	1.35	1.59	1.61	1.31	1.57			1.41	0.49	
L8-ICP-MS	0.88	0.89	0.90	0.85	0.86	0.88	0.91	0.88	0.87	0.86	0.87	0.88	0.85	0.91	0.90	0.87	0.86	0.86			0.88	0.09	
L11-ICP-MS	1.25	1.41	1.39	1.38	1.24	1.34	1.35	1.46	1.41	1.35	1.39	1.50	1.45	1.41	1.53	1.36	1.42	1.40			1.39	0.35	
L20-ICP-MS													2.00	2.50	3.00	3.00	2.60	2.30			2.57	0.79	

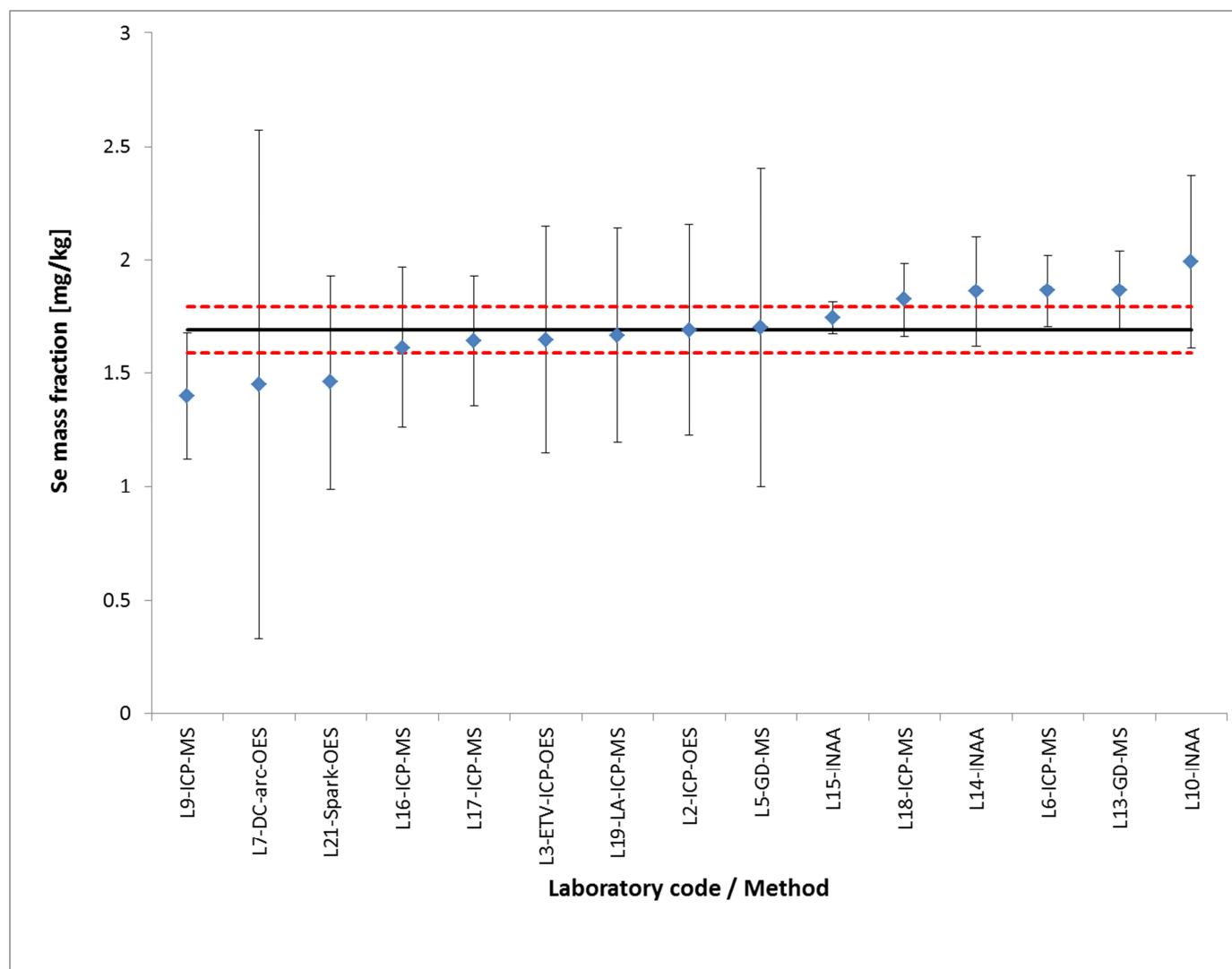


Figure E20. Mean Se mass fraction in ERM-EB075 reported by participating laboratories. Error bars represent expanded uncertainties. The solid line represents the certified values (the mean of laboratory means), while the broken lines represent the expanded uncertainty of the certified value. Each laboratory is represented by its code.

Si (Silicon)

Table E21. Individual results for Si mass fraction in ERM-EB075 provided by each laboratory. The mean value was calculated as the unweighted mean of all individual results. The expanded uncertainty budget equals to k factor ($k = 2$) multiplied by u provided by the laboratory or is estimated as two times the standard deviation of the measurements (if u is not provided).

Lab code - Method	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Rep 6	Rep 7	Rep 8	Rep 9	Rep 10	Rep 11	Rep 12	Rep 13	Rep 14	Rep 15	Rep 16	Rep 17	Rep 18	Rep 19	Rep 20	Mean	Uncertainty (k=2)	
	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]
L5-GD-MS	2.00	1.52	1.54	2.01	1.94	1.70	1.81	1.77	1.55	1.92	1.60	1.67										1.75	0.64
L6-ICP-OES	5.20	3.80	3.30	4.00	2.00	3.20	2.70	3.20	3.00	2.20	2.30	3.10	3.70	3.80	2.40	2.20	2.90	2.10				3.06	1.66
L8-ICP-MS	2.80	2.70	2.80	2.80	2.60	0.27	2.70	2.60	2.60	2.90	2.80	2.50	2.90	2.70	2.60	2.80	2.60	2.60				2.57	0.26
L11-ICP-OES	2.80	2.60	2.80	2.70	2.90	2.60	3.00	2.60	2.90	2.60	2.70	2.70	2.80	2.80	2.70	2.60	2.80	2.80				2.74	0.69
L16-ICP-MS	2.73	2.53	2.64	2.66	2.56	2.71	2.64	2.51	2.58	2.71	2.54	2.72	2.67	2.54	2.58	2.72	2.56	2.70				2.63	0.16
L17-ICP-OES	2.76	2.75	2.83	2.85	2.79	2.77	2.74	2.75	2.78	2.78	2.77	2.79	2.81	2.74	2.80	2.79	2.73	2.73				2.78	0.25
<i>Results not used for certification</i>																							
L1-ICP-OES	< 60	< 60	< 60	< 60	< 60	< 60	< 60	< 60	< 60	< 60	< 60	< 60	< 60	< 60	< 60	< 60	< 60	< 60					
L9-ICP-MS	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5					

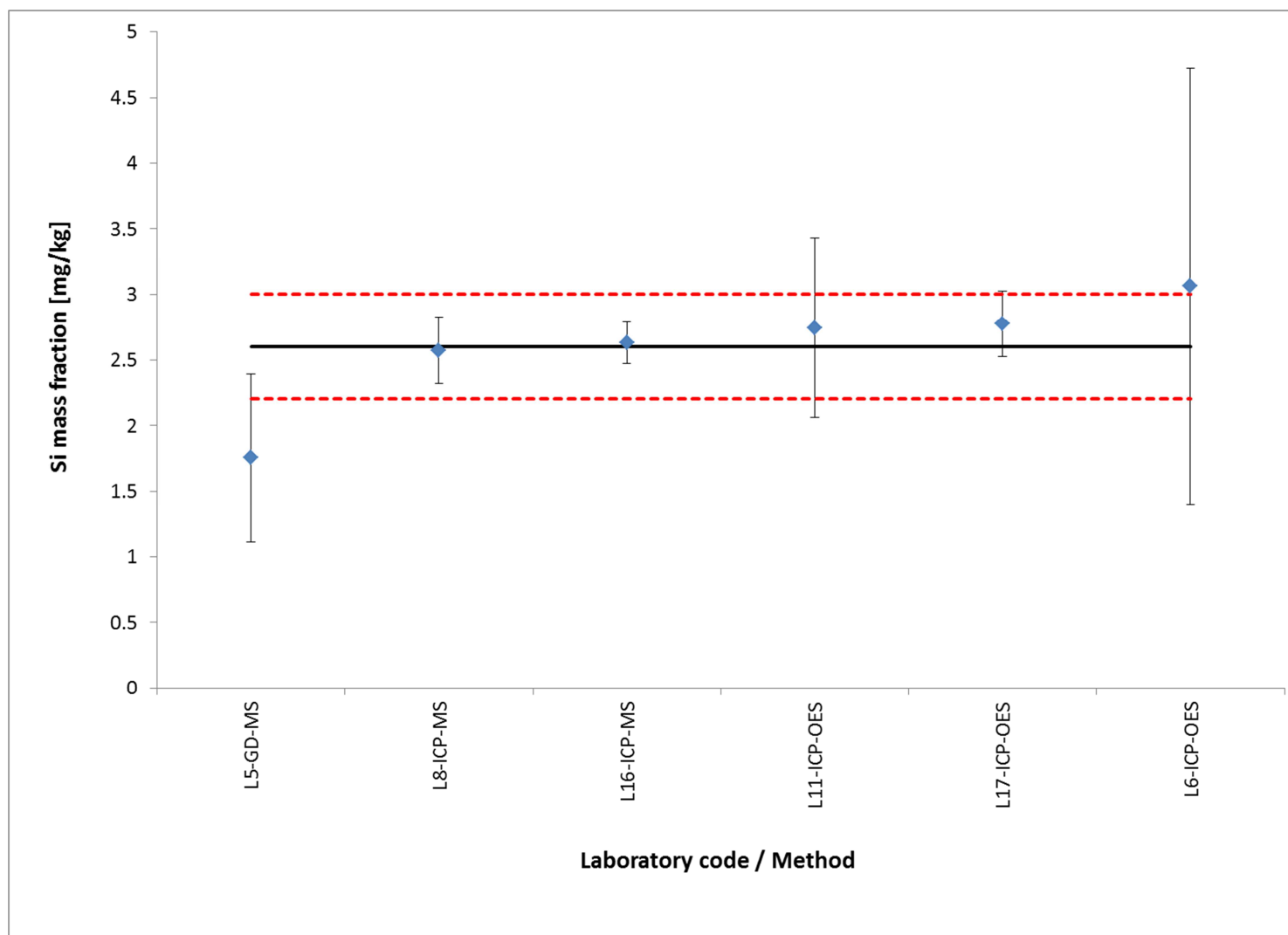


Figure E21. Mean Si mass fraction in ERM-EB075 reported by participating laboratories. Error bars represent expanded uncertainties. The solid line represents the certified values (the mean of laboratory means), while the broken lines represent the expanded uncertainty of the certified value. Each laboratory is represented by its code.

Sn (Tin)

Table E22. Individual results for Sn mass fraction in ERM-EB075 provided by each laboratory. The mean value was calculated as the unweighted mean of all individual results. The expanded uncertainty budget equals to k factor ($k = 2$) multiplied by u provided by the laboratory or is estimated as two times the standard deviation of the measurements (if u is not provided).

Lab code - Method	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Rep 6	Rep 7	Rep 8	Rep 9	Rep 10	Rep 11	Rep 12	Rep 13	Rep 14	Rep 15	Rep 16	Rep 17	Rep 18	Rep 19	Rep 20	Mean	Uncertainty (k=2)	
	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]
L1-ICP-MS	2.14	2.53	2.01	0.58	1.86	2.37	2.20	2.11		2.15	2.92	3.02	1.38	2.92	2.29	2.07	3.00	2.07			2.21	1.22	
L2-ICP-OES	2.40	1.80	2.00	2.10	2.00	2.00	1.70	2.10	2.10	1.90	2.00	2.10	2.20	2.30	2.00	2.10	2.10	2.20			2.06	0.33	
L3-ETV-ICP-OES	1.98	1.98	2.02	1.96	1.96	1.92	1.99	1.90	1.91	1.95	2.05	1.98	2.00	1.99	2.02	1.86	1.87	1.84	1.92	1.92	1.95	0.16	
L4-ICP-MS	2.19	2.32	2.29	2.21	2.27	2.23	2.30	2.16	2.17	2.31	2.53	2.20	2.24	2.24	2.33	2.27	2.21	2.32			2.27	0.17	
L5-GD-MS	1.31	1.57	1.68	1.57	1.70	1.42	2.11	2.04	1.60	1.60	1.54	1.42									1.63	1.00	
L6-ICP-MS	2.30	2.10	2.20	2.30	2.30	2.30	2.30	2.20	2.20	2.20	2.20	2.30	2.30	2.20	2.20	2.20	2.30	2.20			2.24	0.12	
L7-DC-arc-OES													2.40	2.30	2.40	2.20	2.60	2.50			2.40	0.28	
L8-ICP-MS	2.00	2.20	2.20	2.20	2.20	2.10	2.20	2.20	2.00	2.60	2.50	2.50	2.20	2.10	2.20	2.20	2.20	2.30			2.23	0.22	
L9-ICP-MS	1.43	2.95	2.48	1.38	1.28	2.52	1.10	1.04	2.28	1.28	2.41	2.32	1.62	1.93	2.46	1.72	2.27	2.40			1.94	0.39	
L11-ICP-MS	2.08	2.06	2.02	2.04	2.07	1.98	2.02	2.08	1.96	2.12	1.96	2.05	2.13	2.08	2.04	2.02	2.05	2.04			2.04	0.51	
L13-GD-MS	2.61	2.36	2.39	2.22	2.40	2.41	2.32	2.60	2.31	2.39	2.37	2.29									2.39	0.27	
L16-ICP-MS	2.13	2.16	2.15	1.65	2.23	2.10	2.15	2.22	2.11	2.19	2.17	2.14	2.07	2.24	2.08	2.31	2.08	2.09			2.13	0.17	
L17-ICP-MS	2.24	2.23	2.22	2.23	2.30	2.20	2.21	2.14	2.22	2.27	2.20	2.24	2.21	2.21	2.24	2.21	2.21	2.20			2.22	0.33	
L18-ICP-MS	2.07	2.09	2.13	1.91	2.00	2.05	2.04	2.09	2.03	2.16	2.10	2.11	2.21	2.16	2.23	2.15	2.24	2.26			2.11	0.18	
L20-ICP-MS													2.10	2.20	2.10	2.20	2.10	2.20			2.15	0.11	
<i>Results not used for certification</i>																							
L19-LA-ICP-MS	2.99	2.89	2.96	3.08	3.00	3.34															3.04	0.32	
L21-Spark-OES	< 2	< 2	< 2	< 2	< 2	< 2	< 2	< 2	< 2	< 2	< 2	< 2											

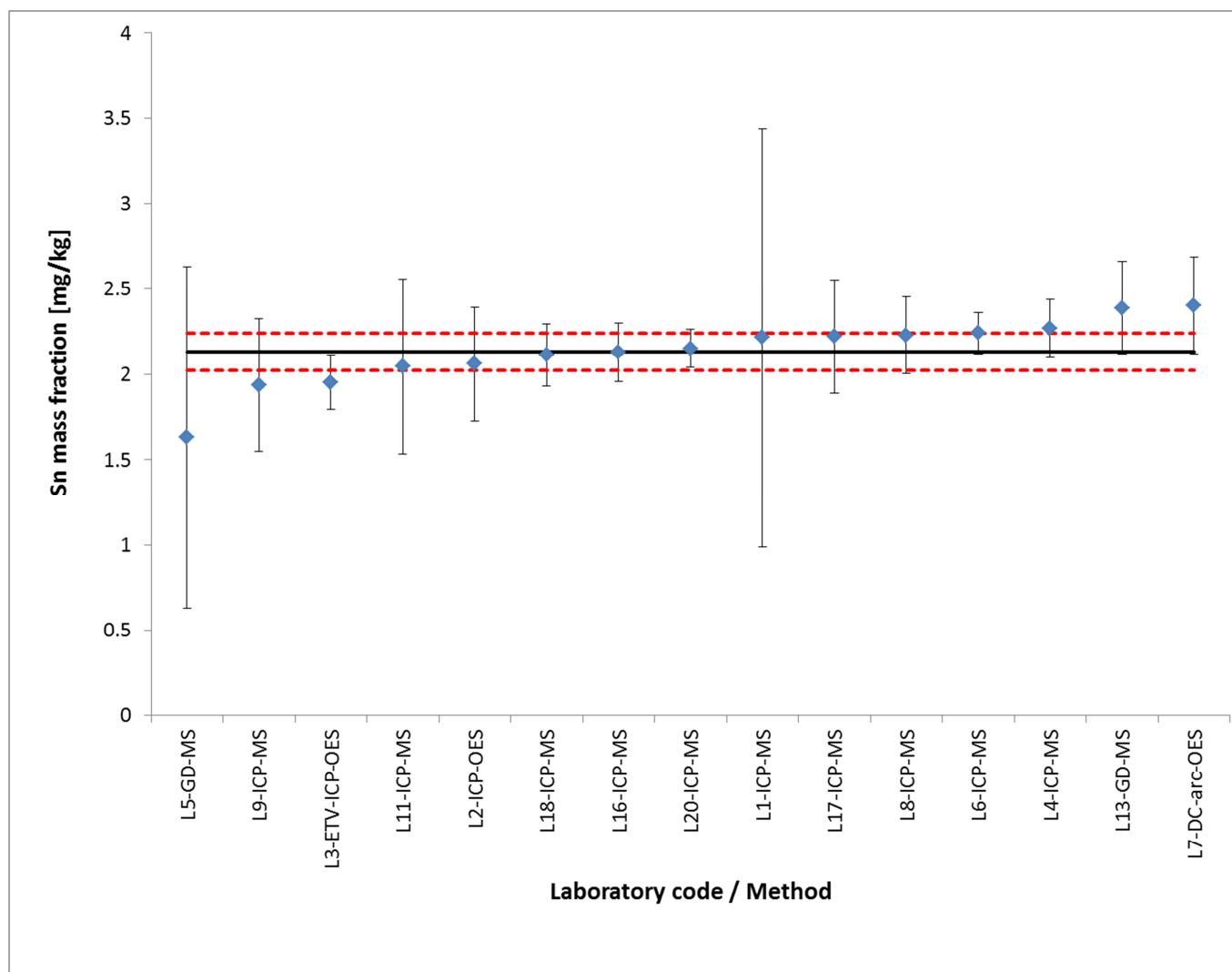


Figure E22. Mean Sn mass fraction in ERM-EB075 reported by participating laboratories. Error bars represent expanded uncertainties. The solid line represents the certified values (the mean of laboratory means), while the broken lines represent the expanded uncertainty of the certified value. Each laboratory is represented by its code.

Te (Tellurium)

Table E23. Individual results for Te mass fraction in ERM-EB075 provided by each laboratory. The mean value was calculated as the unweighted mean of all individual results. The expanded uncertainty budget equals to k factor ($k = 2$) multiplied by u provided by the laboratory or is estimated as two times the standard deviation of the measurements (if u is not provided).

Lab code - Method	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Rep 6	Rep 7	Rep 8	Rep 9	Rep 10	Rep 11	Rep 12	Rep 13	Rep 14	Rep 15	Rep 16	Rep 17	Rep 18	Rep 19	Rep 20	Mean	Uncertainty (k=2)	
	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]
L1-ICP-MS	1.75	1.67	1.60	1.71	1.22	1.89	1.63	1.89	1.87	1.69	1.53	1.78	1.96	1.82	1.71	1.64	1.95	1.62			1.72	0.35	
L3-ETV-ICP-OES	1.35	1.31	1.61	1.46	1.46	1.73	1.68	1.80	1.87	1.82	1.10	1.22	1.28	1.32	1.40	1.56	1.59	1.60	1.70	2.39	1.51	0.74	
L4-ICP-MS	1.81	1.76	1.73	1.82	1.79	1.76	1.73	1.74	1.78	1.77	1.73	1.76	1.76	1.71	1.78	1.75	1.77	1.78			1.76	0.06	
L5-GD-MS	1.36	1.60	1.69	1.50	1.52	1.52	1.84	1.78	1.64	1.47	1.67	1.61									1.60	0.38	
L6-ICP-MS	1.90	1.70	1.80	1.80	1.80	1.80	1.80	1.80	1.80	1.80	1.80	1.90	1.90	1.80	1.80	1.80	1.80	1.80			1.81	0.09	
L7-DC-arc-OES													2.20	1.80	1.60	1.30	1.80	1.60			1.72	0.60	
L8-ICP-MS	1.60	1.60	1.70	1.60	1.60	1.70	1.70	1.70	1.40	1.60	1.60	1.50	1.80	1.70	1.70	1.60	1.70	1.50			1.63	0.16	
L10-INAA	1.82	1.97	1.92	1.81	1.89	1.94	1.82	1.88	1.90	1.81	1.99	1.73	1.91	1.90	1.69	1.54	1.84	1.67			1.84	0.32	
L11-ICP-MS	2.01	2.18	2.14	2.21	1.99	2.20	2.22	2.23	2.06	2.23	2.12	2.16	2.17	2.25	2.21	2.26	2.20	2.22			2.17	0.54	
L13-GD-MS	1.80	1.94	1.88	2.39	2.17	2.46	1.92	1.88	1.83	1.89	1.79	1.89									1.99	0.23	
L14-INAA	2.05	1.96	2.07	1.98	2.06	2.07	1.99	2.05	1.96	2.02	2.18	2.07	2.06	1.88	2.29	2.14	2.04	2.04			2.05	0.35	
L16-ICP-MS	1.14	1.56	1.19	1.23	1.23	1.21	1.27	1.18	1.76	1.42	1.45	1.23	1.47	1.36	1.68	1.45	1.56	1.38			1.38	0.35	
L17-ICP-MS	1.91	1.84	1.81	1.90	1.91	1.89	1.79	1.83	1.89	1.83	1.77	1.90	1.86	1.92	1.79	1.84	1.91	1.85			1.86	0.32	
L18-ICP-MS	2.01	2.07	2.24	2.08	1.98	2.05	1.88	2.08	2.07	1.93	2.07	1.99	1.99	2.03	1.97	1.86	2.04	2.19			2.03	0.19	
L19-LA-ICP-MS	1.870	1.883	1.808	1.542	1.513	1.462															1.68	0.39	
L20-ICP-MS													1.60	1.80	1.70	1.80	1.80	1.80			1.75	0.17	
<i>Results not used for certification</i>																							
L2-ICP-OES	<2	<2	<2	<2	<2	<2	<2	<2	<2	<2	<2	<2	<2	<2	<2	<2	<2	<2			#DIV/0!	#DIV/0!	
L9-ICP-MS	2.58	2.71	3.23	2.64	2.43	2.70	2.57	2.50	2.95	2.92	2.76	2.77	2.51	2.45	2.90	2.50	2.68	2.70			2.69	0.54	

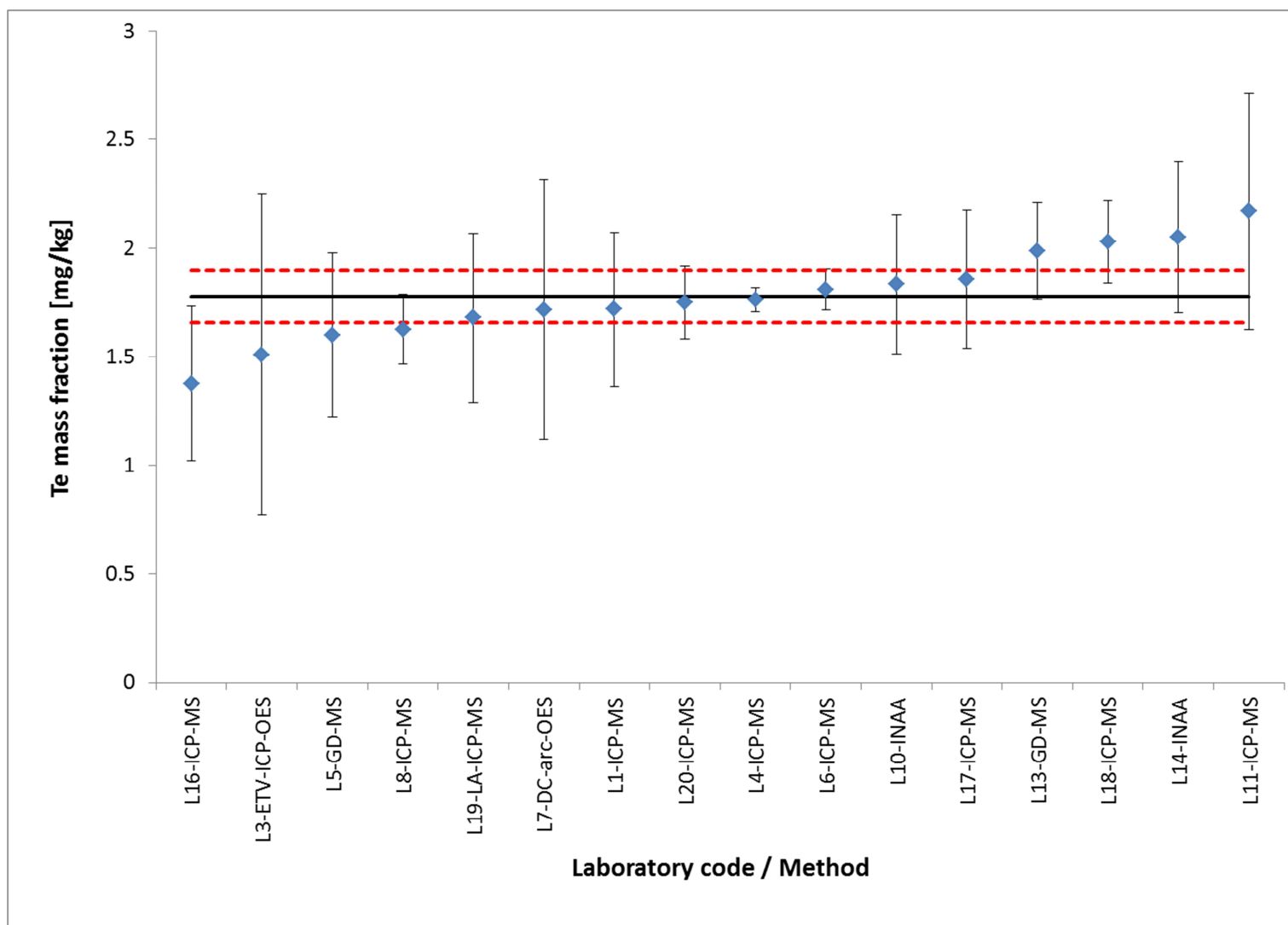


Figure E23. Mean Te mass fraction in ERM-EB075 reported by participating laboratories. Error bars represent expanded uncertainties. The solid line represents the certified values (the mean of laboratory means), while the broken lines represent the expanded uncertainty of the certified value. Each laboratory is represented by its code.

Ti (Titanium)

Table E24. Individual results for Ti mass fraction in ERM-EB075 provided by each laboratory. The mean value was calculated as the unweighted mean of all individual results. The expanded uncertainty budget equals to k factor ($k = 2$) multiplied by u provided by the laboratory or is estimated as two times the standard deviation of the measurements (if u is not provided).

Lab code - Method	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Rep 6	Rep 7	Rep 8	Rep 9	Rep 10	Rep 11	Rep 12	Rep 13	Rep 14	Rep 15	Rep 16	Rep 17	Rep 18	Rep 19	Rep 20	Mean	Uncertainty ($k=2$)	
	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]
L1-ICP-MS	2.98	3.34	3.20	2.82	2.91	2.81	3.18	2.97	2.68	3.13	3.16	2.78	3.16	2.97	3.41	3.26	3.07	3.37			3.07	0.43	
L2-ICP-OES	3.30	3.30	3.40	3.10	3.10	3.20	3.30	3.30	3.30	3.40	3.40	3.30	3.30	3.40	3.40	3.40	3.40	3.40			3.32	0.20	
L4-ICP-MS	3.18	3.20	3.04	3.35	3.38	3.35	3.12	3.31	3.20	3.80	3.16	3.20	3.35	3.19	3.40	3.33	3.26	3.15			3.28	0.33	
L5-GD-MS	1.75	3.62	4.06	2.37	2.78	2.92	3.79	3.50	3.82	2.67	3.36	2.76									3.12	1.00	
L8-ICP-MS	2.70	2.40	2.30	2.90	2.70	2.70	2.70	2.50	2.40	2.60	2.70	2.90	2.50	2.50	2.40	2.50	2.80	2.80			2.61	0.26	
L9-ICP-MS	3.25	3.15	3.31	3.16	3.10	3.21	3.28	3.31	3.21	3.16	3.08	3.25	3.38	3.30	3.19	3.25	3.24	3.46			3.24	0.65	
L11-ICP-MS	3.25	3.16	3.24	3.21	3.16	3.12	3.19	3.08	3.24	3.21	3.20	3.21	3.23	3.26	3.30	3.21	3.19	3.15			3.20	0.80	
L13-GD-MS	3.01	2.99	3.27	3.05	2.95	3.22	2.78	2.95	2.93	3.08	3.03	3.41									3.06	0.25	
L16-ICP-MS	2.93	3.05	3.06	2.67	2.76	2.62	3.12	3.11	3.04	2.94	2.78	2.91	3.01	3.15	3.13	3.04	3.16	3.02			2.97	0.12	
L17-ICP-MS	3.15	3.11	3.12	2.81	2.72	2.81	3.12	3.20	3.21	3.24	3.08	3.32	3.24	3.18	3.19	3.00	3.26	3.13			3.11	0.96	
L18-ICP-MS	3.65	3.42	3.41	3.87	3.05	3.62	3.00	3.02	3.05	3.69	3.12	2.92	3.42	3.28	3.45	3.07	3.29	3.28			3.31	0.55	
L19-LA-ICP-MS	3.46	3.45	3.16	2.97	2.91	3.17															3.19	0.46	
L20-ICP-MS													3.90	3.60	4.20	4.50	3.90	3.60			3.95	0.70	

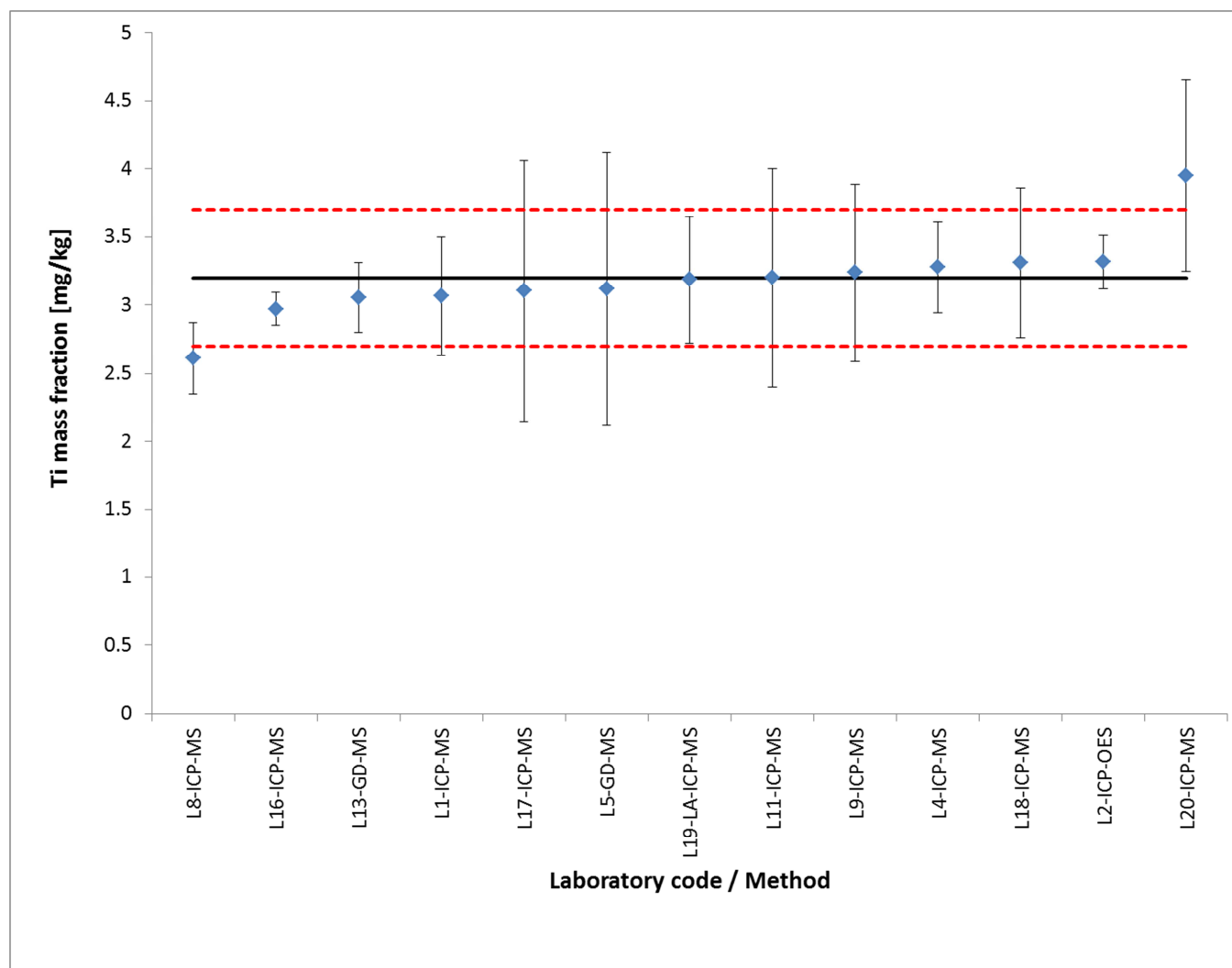


Figure E24. Mean Ti mass fraction in ERM-EB075 reported by participating laboratories. Error bars represent expanded uncertainties. The solid line represents the certified values (the mean of laboratory means), while the broken lines represent the expanded uncertainty of the certified value. Each laboratory is represented by its code.

Zn (Zinc)

Table E25. Individual results for Zn mass fraction in ERM-EB075 provided by each laboratory. The mean value was calculated as the unweighted mean of all individual results. The expanded uncertainty budget equals to k factor ($k = 2$) multiplied by u provided by the laboratory or is estimated as two times the standard deviation of the measurements (if u is not provided).

Lab code - Method	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Rep 6	Rep 7	Rep 8	Rep 9	Rep 10	Rep 11	Rep 12	Rep 13	Rep 14	Rep 15	Rep 16	Rep 17	Rep 18	Rep 19	Rep 20	Mean	Uncertainty ($k=2$)	
	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]
L2-ICP-OES	6.80	6.60	3.50	6.40	5.80	6.70	6.20	6.90	6.40	6.10	6.60	6.30	6.30	6.20	6.20	6.60	5.80	6.50			6.22	1.49	
L4-ICP-MS	7.28	6.69	6.98	5.12	6.96	5.08	7.23	5.01	5.25	6.76	6.89	5.18	5.35	6.75	6.74	6.60	5.04	7.57			6.25	1.87	
L5-GD-MS	6.07	6.96	6.98	5.85	5.75	6.76	5.80	5.60	6.48	5.79	6.75	6.88									6.31	1.40	
L6-ICP-OES	7.90	7.50	7.40	6.60	7.10	6.30	7.20	7.00	6.70	6.60	6.60	6.30	6.70	6.80	6.90	6.80	6.90	6.30			6.87	0.87	
L7-DC-arc-OES													6.20	6.20	6.90	6.70	7.90	7.20			6.85	1.29	
L10-INAA	7.35	7.41	7.37	7.50	7.18	6.81	8.26	7.37	8.21	6.85	7.39	7.03	6.92	7.10	6.84	7.43	7.53	7.43			7.33	1.14	
L11-ICP-MS	6.37	6.37	6.47	6.30	6.30	6.30	6.36	6.23	6.34	6.28	6.25	6.29	6.40	6.05	6.16	6.28	6.31	6.35			6.30	1.58	
L12-ICP-MS	6.20	6.10	5.50	6.40	6.70	5.70	5.50	5.40	5.90	6.50	6.10	5.50	5.50	5.40	5.40	6.50	6.50	6.10			5.94	0.50	
L13-GD-MS	6.21	5.67	6.12	5.53	6.21	6.36	6.14	6.34	6.42	6.09	6.09	6.04									6.10	0.60	
L14-INAA													6.68	6.81	6.55	6.91	6.35	6.76			6.67	0.52	
L15-INAA	7.17	6.91	6.67	7.01	6.97	7.26	7.13	6.91	6.88	7.14	7.05	7.22	6.91	7.40	7.40	7.60	6.99	7.13			7.10	0.50	
L16-ICP-MS	5.83	5.46	5.83	5.68	5.42	5.77	6.46	5.98	6.28	5.43	5.68	6.15	5.85	6.14	5.78	5.59	5.89	5.63			5.83	0.35	
L17-ICP-MS	6.29	6.63	6.56	5.98	6.57	5.65	6.71	6.23	6.24	6.67	6.43	6.19	6.27	6.55	6.50	6.60	6.08	6.52			6.37	0.82	
L18-ICP-MS	8.26	7.65	7.89	7.63	7.21	6.62	7.51	7.48	7.40	7.44	7.59	7.47	7.11	6.79	7.68	6.89	6.78	6.35			7.32	0.97	
L20-ICP-MS													6.30	6.20	6.00	6.30	6.30	6.40			6.25	0.28	
<i>Results not used for certification</i>																							
L1-ICP-MS	9.72	8.19	8.57	8.58	8.25	8.93	10.11	0.00	0.00	9.70	10.78	11.86	8.81	8.69	9.86	8.65	9.61	8.32			8.26	6.31	
L8-ICP-MS	7.60	7.70	7.70	7.50	7.40	7.60	7.90	7.30	7.60	7.50	7.50	7.70	7.80	7.40	7.60	7.40	7.60	7.60			7.58	0.76	
L9-ICP-MS	7.32	8.90	9.13	8.19	9.32	8.92	7.95	8.34	9.23	8.07	9.14	8.99	8.24	8.56	8.70	8.91	8.27	8.40			8.59	1.72	
L19-LA-ICP-MS	7.05	7.50	7.23	6.81	7.30	7.03															7.15	0.48	
L21-Spark-OES	7.00	7.30	7.10	6.60	7.90	6.70	4.40	6.80	5.10	4.60	6.80	6.20									6.38	2.20	

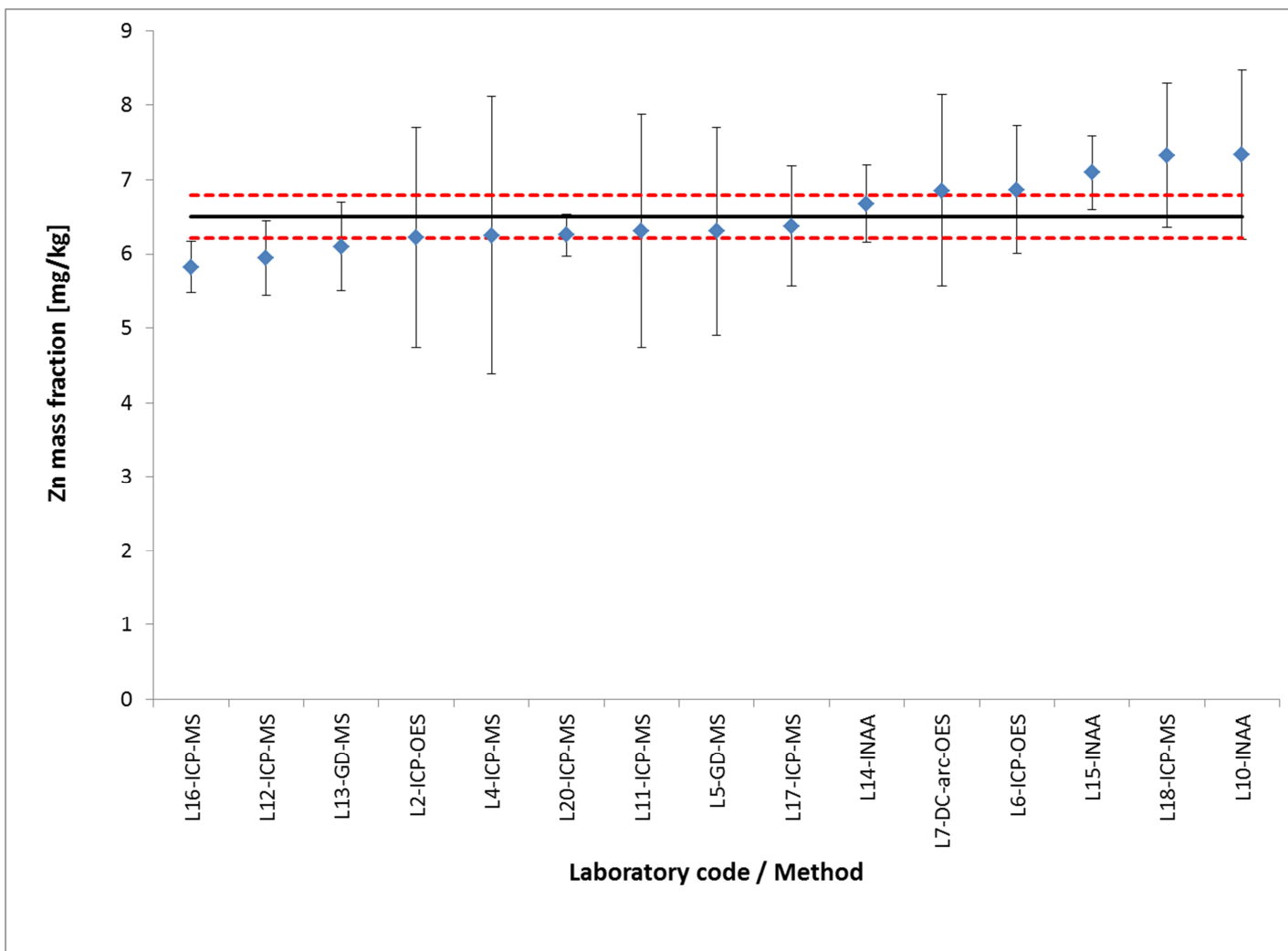


Figure E25. Mean Zn mass fraction in ERM-EB075 reported by participating laboratories. Error bars represent expanded uncertainties. The solid line represents the certified values (the mean of laboratory means), while the broken lines represent the expanded uncertainty of the certified value. Each laboratory is represented by its code.

Zr (Zirconium)

Table E26. Individual results for Zr mass fraction in ERM-EB075 provided by each laboratory. The mean value was calculated as the unweighted mean of all individual results. The expanded uncertainty budget equals to k factor ($k = 2$) multiplied by u provided by the laboratory or is estimated as two times the standard deviation of the measurements (if u is not provided).

Lab code - Method	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Rep 6	Rep 7	Rep 8	Rep 9	Rep 10	Rep 11	Rep 12	Rep 13	Rep 14	Rep 15	Rep 16	Rep 17	Rep 18	Rep 19	Rep 20	Mean	Uncertainty ($k=2$)	
	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]
L2-ICP-OES	18.60	18.10	18.10	15.90	16.20	15.90	17.30	17.50	17.20	17.40	17.40	18.10	18.50	17.90	18.20	18.40	18.10	18.80			17.64	1.77	
L4-ICP-MS	19.70	20.40	20.20	20.80	20.50	20.20	20.90	20.20	20.10	20.90	20.70	20.40	20.70	20.40	21.00	20.70	20.50	20.70			20.50	0.67	
L5-GD-MS	8.14	18.80	22.70	11.50	14.10	13.70	26.80	24.80	20.80	14.50	18.20	14.00									17.34	7.80	
L10-INAA	21.60	21.00	22.30	19.10	20.30	18.50	19.60	20.80	21.10	22.80	21.60	21.80	23.60	24.50	21.60	22.50	22.30	23.60			21.59	3.40	
L14-INAA	22.99	24.92	23.24	24.27	23.00	21.40	21.61	23.67	19.13	18.71	23.14	19.33	21.78	18.56	19.59	17.75	19.81	19.94			21.27	3.80	
L15-INAA	30.20	24.80	24.90	23.70	18.20	19.40	21.90	39.40	25.40	26.30	27.30	30.00	16.70	28.50	24.30	20.40	30.50	20.40			25.13	4.00	
L19-LA-ICP-MS	15.62	15.56	12.95	8.69	12.94	8.41															12.36	6.36	
L20-ICP-MS													21.50	20.10	19.60	19.80	21.40	20.40			20.47	1.62	
<i>Results not used for certification</i>																							
L1-ICP-MS	6.91	8.66	9.13	0.51	8.63	8.45	13.25	12.94	17.07	13.45	13.56	14.29	5.61	7.51	11.41	8.97	10.69	9.27			10.02	7.64	
L8-ICP-MS	10.00	11.00	11.00	10.00	11.00	11.00	11.00	11.00	11.00	10.00	11.00	10.00	11.00	11.00	11.00	10.00	10.00	11.00			10.67	1.07	
L9-ICP-MS	10.70	11.30	11.50	11.14	10.97	11.70	11.96	12.05	11.88	11.54	11.88	11.79	11.34	11.68	12.08	11.44	12.09	12.77			11.66	2.33	
L11-ICP-MS	14.79	14.74	14.86	15.21	14.90	14.60	15.35	15.05	15.26	14.81	14.82	15.05	14.66	15.03	14.96	15.05	14.97	15.24			14.97	3.74	
L16-ICP-OES	12.04	11.14	12.43	9.74	9.70	9.49	14.24	12.92	12.97	12.58	12.25	12.81	14.91	13.99	13.14	13.09	13.34	12.95			12.43	0.87	
L18-ICP-MS	11.60	11.90	12.65	12.22	12.85	12.46	5.62	4.85	4.47	4.67	4.70	4.37	13.37	12.87	13.29	12.47	13.08	13.60			10.06	7.76	

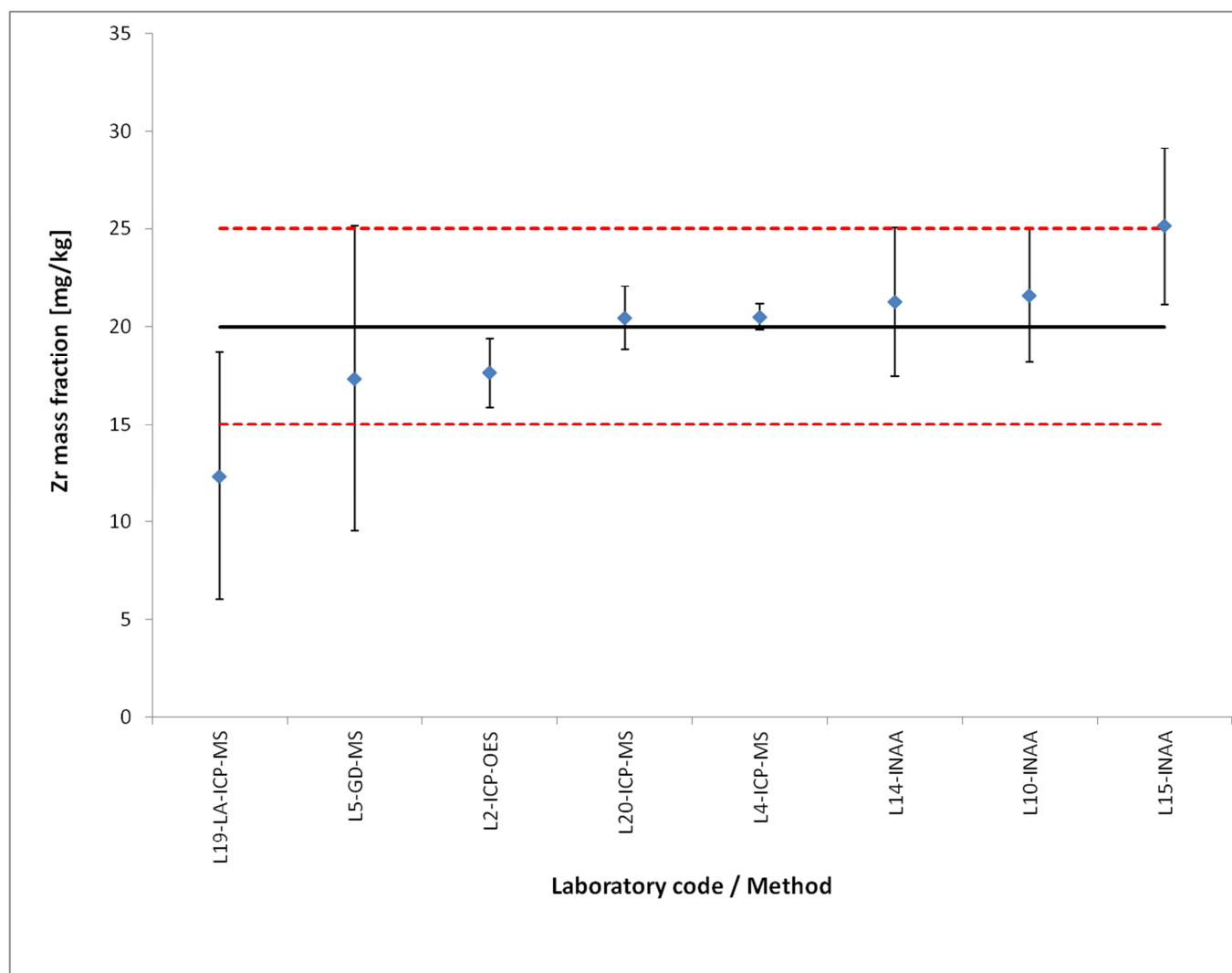


Figure E26. Mean Zr mass fraction in ERM-EB075 reported by participating laboratories. Error bars represent expanded uncertainties. The solid line represents the certified values (the mean of laboratory means), while the broken lines represent the expanded uncertainty of the certified value. Each laboratory is represented by its code.

Hg (Mercury)

Table E27. Individual results for Hg mass fraction in ERM-EB075 provided by each laboratory. The mean value was calculated as the unweighted mean of all individual results. The expanded uncertainty budget equals to k factor ($k = 2$) multiplied by u provided by the laboratory or is estimated as two times the standard deviation of the measurements (if u is not provided).

Lab code - Method	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Rep 6	Rep 7	Rep 8	Rep 9	Rep 10	Rep 11	Rep 12	Rep 13	Rep 14	Rep 15	Rep 16	Rep 17	Rep 18
	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]
L1-CV-AAS	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005
L8-ICP-MS	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
L9-ICP-MS	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
L10-INAA	< 0.25	< 0.28	< 0.28	< 0.06	< 0.24	< 0.05	< 0.28	< 0.30	< 0.30	< 0.07	< 0.06	< 0.28	< 0.06	< 0.30	< 0.25	< 0.25	< 0.27	< 0.35
L11-ICP-MS	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
L16-ICP-MS	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
L18-ICP-MS	< 0.04	< 0.04	< 0.04	< 0.04	< 0.04	< 0.04	< 0.04	< 0.04	< 0.04	< 0.04	< 0.04	< 0.04	< 0.04	< 0.04	< 0.04	< 0.04	< 0.04	< 0.04

W (Tungsten)

Table E28. Individual results for W mass fraction in ERM-EB075 provided by each laboratory. The mean value was calculated as the unweighted mean of all individual results. The expanded uncertainty budget equals to k factor ($k = 2$) multiplied by u provided by the laboratory or is estimated as two times the standard deviation of the measurements (if u is not provided).

Lab code - Method	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Rep 6	Rep 7	Rep 8	Rep 9	Rep 10	Rep 11	Rep 12	Rep 13	Rep 14	Rep 15	Rep 16	Rep 17	Rep 18	Mean	Uncertainty (k=2)
	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]	[mg/kg]
L1-ICP-MS	< 0.08	< 0.08	< 0.08	< 0.08	< 0.08	< 0.08	< 0.08	< 0.08	0.08	< 0.08	< 0.08	< 0.08	< 0.08	< 0.08	< 0.08	< 0.08	0.09	< 0.08		
L4-ICP-MS	0.0140	0.0080	0.0110	0.0060	0.0080	0.0060	0.0070	0.0040	0.0070	0.0070	0.0060	0.0060	0.0200	0.0200	0.0180	0.0210	0.0100	0.0160	0.011	0.005
L8-ICP-MS	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01		
L9-ICP-MS	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25	< 0.25		
L11-ICP-MS	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1		
L16-ICP-MS	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1		
L18-ICP-MS	< 0.04	< 0.04	< 0.04	< 0.04	< 0.04	< 0.04	< 0.04	< 0.04	< 0.04	< 0.04	< 0.04	< 0.04	< 0.04	< 0.04	< 0.04	< 0.04	< 0.04	< 0.04		

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