Certification Report

Certified Reference Material

ERM®-EB317

AlZn6CuMgZr

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Coordinator: Dr. Sebastian Recknagel
BAM Federal Institute for Materials Research and Testing
Division 1.6 “Inorganic Reference Materials“
Richard-Willstätter-Straße 11
12489 Berlin
Tel.: ++49/30/8104 1111
Fax.: ++49/30/8104 1117
Email: sebastian.recknagel@bam.de

Summary

This report describes preparation, analysis and certification of the aluminium alloy reference materials ERM®-EB317.
The certified reference material is available in the form of discs (50 mm diameter and 30 mm height). It is intended for establishing and checking the calibration of optical emission and X-ray spectrometers (excluding micro-analysis) for the analysis of samples of similar matrix composition.

The following mass fractions and uncertainties have been certified:

Element	**Mass fraction** in %	**Uncertainty** in %
Fe	0.112	0.003
Cu	1.77	0.06
Mg	2.39	0.07
Cr	0.141	0.003
Zn	6.93	0.26
Zr	0.130	0.008
	in mg/kg	in mg/kg
Si	271	22
Mn	912	19
Ni	359	14
Ti	952	156
Ag	73	5
Be	10.1	0.8
Bi	41	6
Ga	183	12
In	162	11
Pb	48.1	2.3
Sn	238	18
V	105	7

The following mass fractions and uncertainties are given as indicative values:

Element	**Mass fraction** in mg/kg	**Uncertainty** in mg/kg
B	37	32
Ca	6.0	2.7
P	27	15

This report contains detailed information on the preparation of the CRMs as well as on homogeneity investigations and on the analytical methods used for certification analysis.
The certified values are based on the results of 11 laboratories which participated in the certification interlaboratory comparison.

Content

List of abbreviations
(if not explained elsewhere)

CRM	certified reference material
ERM	European reference material
FAAS	flame atomic absorption spectrometry
ICP-OES	inductively coupled plasma optical emission spectrometry
ICP-MS	inductively coupled plasma mass spectrometry
SOES	spark optical emission spectrometry
XRF	X-ray fluorescence spectrometry
M	mean value
n	number of accepted data sets
INAA	instrumental neutron activation analysis
s	standard deviation of an individual data set
s_M	standard deviation of the mean of means
s_{rel}	relative standard deviation
$\bar{s}_i$	mean standard deviation
M_i	single result
I	ICP-OES (Tables 24 – 44)
I(R)	ICP-OES, revised value (Tables 24 – 44)
IMS	ICP-MS (Tables 4 – 24)
A	FAAS (Tables 4 – 24)
A(R)	FAAS, revised value (Tables 24 – 44)
P	spectrophotometry (Tables 24 – 44)
-s	dissolution in acid
-a	dissolution in base

1. Introduction

In the metal-producing and metal-working industry mainly spark emission spectrometry (SOES) and X-ray fluorescence spectrometry (XRF) are used for reception inspection of raw materials, e.g. scrap, for quality control of end products and production control. These time saving analytical techniques require suitable reference materials for calibration and recalibration. The certified reference material ERM®-EB317 is based on a AlZn6CuMgZr alloy (EN AW-7050).

The idea to produce a reference material of this alloy type was the outcome of discussions within the German Gesellschaft für Bergbau, Metallurgie, Rohstoff- und Umwelttechnik (GDMB), especially the working group „Aluminium" of the Committee of Chemists within GDMB. From this working group the needs were defined, since the members are potential users of the prepared CRMs. Secondly from this group the participating laboratories are recruited. Since all of these laboratories are highly experienced with aluminium alloy analysis and participated in earlier interlaboratory comparisons, there was no preceding round robin test for qualification.

Certification of reference materials is carried out on the basis of the relevant ISO-Guides [1-3], the „Guidelines for the production of BAM Reference Materials" [4] and the "Technical Guidelines for the Production and Acceptance of a European Reference Material" [5].

2. Companies/laboratories involved

<u>Preparation of the material:</u>
- Otto Fuchs KG, Meinerzhagen

<u>Test for homogeneity:</u>

- BAM Bundesanstalt für Materialforschung und -prüfung

<u>Participants in the certification interlaboratory comparison:</u>

- 3A Technology & Management AG, Neuhausen, Switzerland
- Constellium, Centre de Recherches de Voreppe, Voreppe, France
- AMAG Austria Metall AG, Ranshofen, Austria
- BAM Bundesanstalt für Materialforschung und -prüfung, Berlin, Germany
  - Division 1.6 Inorganic Reference Materials
  - Division 1.4 Process Analytical Technology
- HORIBA Jobin Yvon GmbH, Unterhaching, Germany
- Hydro Aluminium Rolled Products GmbH, R&D-Bonn, Germany
- Hydro Aluminium Rolled Products GmbH, Hamburg, Germany
- Hydro Aluminium Gießerei Hannover GmbH, Hannover, Germany
- Institute of Non-Ferrous Metals, Gliwice, Poland
- Otto Fuchs KG, Meinerzhagen, Germany

<u>Statistical evaluation of the data</u>
- BAM Bundesanstalt für Materialforschung und -prüfung, Berlin

3. Candidate material

The candidate material, EN AW-7050, was casted by Otto Fuchs KG, Meinerzhagen, Germany to an ingot with a diameter of 242 mm and a length of 850 mm.
After casting the material was pressed to a rod with a length of 17 m. This rod was sawed into 2 m – pieces. All these steps were performed by Otto Fuchs KG. The 2 m rods were sent to BAM, where the rods were cut into discs with a height of 32 mm.
In total a number of 496 discs was prepared and numbered according to Figure 1.

1 to 59	1 to 58	1 to 57	1 to 58	1 to 58	1 to 57	1 to 57	1 to 59	1 to 33
A	M1	M2	M3	M4	M5	M6	M7	EA

Fig. 1: Preparation of discs from a 17 m - rod.

4. Homogeneity testing

Homogeneity testing of the raw material was performed on some specific discs using spark emission spectrometry (SOES). In addition the whole batch of discs was investigated using X-ray fluorescence spectrometry (XRF). Especially in case of SOES the sample amount investigated is much smaller than the sample intake typically used for wet chemical analysis.

Possible reasons for an inhomogeneous distribution of elements in the raw material may be a change of the composition of the melt during the casting procedure because some elements may volatise or segregate during the solidification of the material. Since the raw material was produced by casting of a rod, concentration gradients can occur over the length of the rod (axial) as well as over the area of the rod (radial, see Figures 2 and 3):

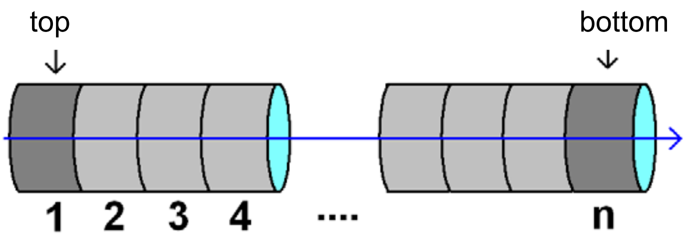

Fig. 2: Axial composition gradient

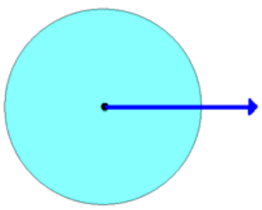

Fig. 3: Radial composition gradient

Therefore it is necessary to investigate the raw material for both axial and radial inhomogeneities. The testing for radial inhomogeneities was performed on three discs (M2-37, M3-2 and M6-52). This investigation was carried out at BAM. To perform this test all three discs were investigated twice putting the sparks on three circles (outer circle: 16 sparks, inner circle: 8 sparks; centre: 1 spark).
For some elements there was an inhomogeneity in the centre of the disc. Therefore the centre of the sample was excluded for analysis. I.e. an area 12 mm in diameter in the centre of the discs should be avoided for optical emission spectrometry.
Tab. 1 shows the discs selected for the axial homogeneity test using SOES (ca. 8 % of the total number of discs according to ASTM standard E 826-90 [6]). All tests were carried out with an OBLF QSL 1500 spectrometer.

Tab. 1: Discs analysed for homogeneity testing of ERM®-EB317

A3	M1-2	M2-2	M3-2	M4-3	M5-2	M6-2	M7-5	EA05
A17	M1-22	M2-19	M3-12	M4-16	M5-14	M6-19	M7-15	EA14
A35	M1-36	M2-37	M3-29	M4-33	M5-38	M6-37	M7-34	EA28
A55	M1-58	M2-56	M3-56	M4-56	M5-55	M6-52	M7-57	

In addition all discs were analysed with XRF using a wavelength dispersive MagiX Pro instrument (Panalytical, Almelo, The Netherlands). Before measuring the disc its surface was milled. One of the discs was used as a drift control sample.

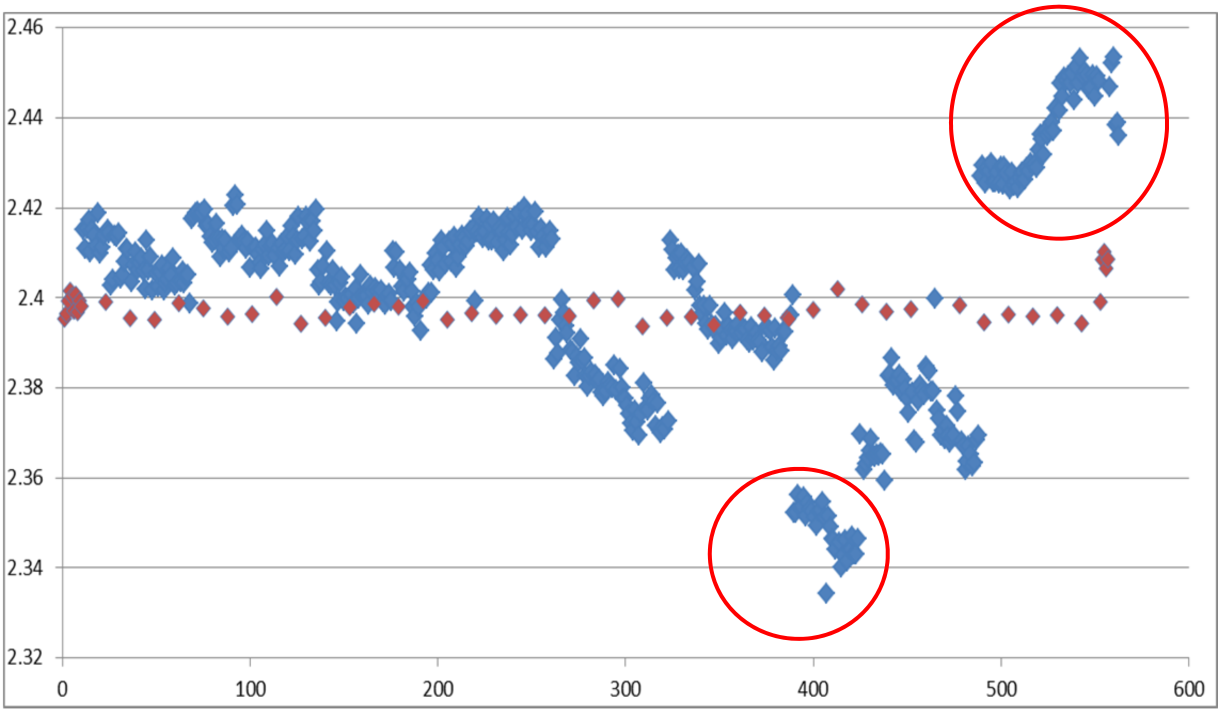

Fig. 4: Determination of Mg in the total batch of ERM®-EB317 (encircled: results for rod sections A and EA, x-axis: number of measurement, y-axis: Mg mass fraction in %, red dots: drift control sample)

This sample was used to determine the spread deriving from the method (10 repetitions). It was also measured after each group of 12 samples to compensate for drifts. For the elements Si, Mg, P, Ni, Cu, Zn, drift compensation was necessary. As a result of XRF-measurements the parts A and EA, i.e. beginning and end of the rod were removed and not used as CRM. Fig. 4 shows the results of Mg-determination. Parts A and EA are encircled. Similar effects could be observed for the elements P and Cu.

All uncertainty contributions due to inhomogeneities are included into the total uncertainty of the resp. certified values.

Since the measured spread of results in all cases contains contributions from both inhomogeneity and the analytical method it is necessary to distinguish between them and to separate the contribution due to the inhomogeneity. This is done by subtracting the variance of the instrumental spread from the variances resulting from inhomogeneities. This approach was used for inhomogeneity investigation over the length of the rods.

Assuming that during the cast process of a rod the solidification of the material proceeds from the outer part of the rod to the center there should be circular areas without any composition gradient. Apart from potential agglomerations of trace elements on crystal boundaries the spread of results measured on a specific circle represents the spread of the method. With this assumption it is possible to calculate the spread of the method and to separate it from the spread due to inhomogeneities of the material.

The lowest standard deviation measured on one specific circle ("inner" or "outer") is taken as a measure for the repeatability of the measure s_{method}.

The number of measurements performed on the three discs chosen for testing radial inhomogeneity was 16 on the outer circle, 8 on the inner circle and one in the center of the area.

The uncertainty contributions resulting from homogeneity testing over and rod length (between) were calculated following the equation:

$$s_{\text{eff}} = \sqrt{s_{\text{sample}}^2 - s_{\text{method}}^2} \qquad (1)$$

In principle it has to be:

$$s_{\text{eff}} = \sqrt{\frac{s_{\text{sample}}^2 - s_{\text{method}}^2}{n_0}} \qquad (1a)$$

here n_0 = 1 as „worst-case-estimation" was used.

In case that the inhomogeneity contributions are in the same order as the precision of the analytical method, it could happen that the term under the square root becomes negative. In this case "s_{eff}" cannot be calculated and an uncertainty contribution u_{bb} taking into account a possibly hidden inhomogeneity u_{bb} is then calculated using the following equation:

$$u_{\text{bb}} = \sqrt{\frac{s_{\text{method}}^2}{p}} \cdot \sqrt[4]{\frac{2}{\nu - 1}} \qquad (2)$$

with p = number of measurements to establish the precision of the method
$\nu - 1$ = degrees of freedom of the calculation of „s^2_{method}”.

Homogeneity testing over the length of the rod was performed twice (SOES and XRF). Inhomogeneity contribution to the total uncertainty was considered only once, i.e. the more precise results were taken into account.

The approach described above was used for the evaluation of the homogeneity measurements over the total batch. In case of the homogeneity investigations over the area (performed on three selected discs) another approach was used.

To calculate the necessary data an unbalanced ANOVA was carried out taking into account that the number of single measurements is different for the center, the inner and the outer circle. For technical reasons, at r_0 (centre) only one measurement per height position is possible. An ANOVA requires minimum two measurements per factor value. Thus, the value for r_0 should be replaced by a dummy. This dummy is defined as follows:

The two values replacing the one measured have a mean equal to the value measured, and a standard deviation equal to the average within-variation. This resembles the situation were one could take two independent measurements at the same place, with values deviating by the average standard deviation (non-destructive testing method). A first guess for the average standard deviation may be calculated from the data for r_in (inner circle) and r_out (outer circle). As results from these calculations an inhomogeneity factor for the radius and one for the height of the disc is obtained. From these values a combined inhomogeneity factor is calculated. This factor is compared with the within standard deviation calculated from the ANOVA-data. The larger factor is used for uncertainty calculation.
The estimates of inhomogeneity contributions u_{bb} potentially hidden by the measurement uncertainty and to be included into the total uncertainty budget were estimated according to ISO Guide 35 [3] as the maximum of the values obtained from Eq. (3) and (4).

$$s_{bb} = \sqrt{\frac{MS_{among} - MS_{within}}{n}} \quad (3)$$

$$s_{bb\,min} = \sqrt{\frac{MS_{within}}{n}} \times \sqrt[4]{\frac{2}{N(n-1)}} \quad (4)$$

with

MS_{among} mean of squared deviations between discs (from 1-way ANOVA)
MS_{within} mean of squared deviations within discs (from 1-way ANOVA)
n number of replicate analyses per disc
N number of discs selected for homogeneity study

To distinguish between the inhomogeneity contributions deriving from radial and axial inhomogeneities the axial part is given as $u_{bb}(1)$, the radial part is given as $u_{bb}(2)$.

5. Characterisation study

5.1 Analytical methods

11 laboratories participated in the certification interlaboratory comparison. For some elements part of the laboratories used more than one analytical method reporting more than

one dataset. 10 laboratories received a randomly chosen disc, the eleventh laboratory received solutions prepared by BAM.
The laboratories were asked to analyse six subsamples. They were free to choose any suitable analytical method for analysis. Table 2 shows the analytical methods used by the participating laboratories.
For all analytical methods where a calibration was necessary this calibration was performed using liquid standard solutions. All participating laboratories were asked to use only standard solutions prepared from pure metals or stoichiometric compounds or well checked commercial calibration solutions.

Table 2: Analytical procedures used by the participating laboratories

Lab-No.	Element.	Sample mass	Sample pretreatment	Analytical method
1	Fe, Cu, Mn, Cr, Ni, Ti, B, Be, Bi, Ca, Ga, In, P, Pb	0.5 g	Dissolution with HCl	ICP-OES, calibration with pure metals or pure substances
	Ag	0.5 g	Dissolution with NaOH	ICP-OES, calibration with pure substance
2	Si, Fe, Cu, Mg, Mn, Cr, Ni, Zn, Ti, Zr, Ga, In, Pb, Sn, V	0.5 g	Dissolution with HCl/H_2O_2	ICP-OES, calibration with commercial solutions (Merck)
	Si	0.5 g	Dissolution with NaOH	Spectrophotometry
	Ti, Ni	1 g	Dissolution with HCl/H_2O_2	Spectrophotometry
	In, Fe, Cr, Zn, Mn, Ag, Ga	15 mg		Instrumental Neutron Activation Analysis
3	Si, Fe, Cu, Mg, Mn, Cr, Ni, Zn, Ti, Zr, Ag, Be, Ga, In, Pb, Sn, V	0.5 g	Dissolution with HCl/H_2O_2	ICP-OES, calibration with commercial mono element solutions
4	Si, Fe, Cu, Mg, Mn, Cr, In, Zn, Ti	0.5 g	Dissolution with NaOH	ICP-OES with matrix matched standards (Al 5N5), calibration with pure metals or pure substances
	Ni, Be, Ca, Ga, V	0.5 g	Dissolution with HNO_3/HF	ICP-OES with matrix matched standards (Al 5N5), calibration with pure metals or pure substances
	P	0.5 g	Dissolution with HCl/HNO_3	ICP-OES with matrix matched standards (Al 5N5), calibration with pure substance
	Ag, B, Bi, Pb, Sn	0.5 g	Dissolution with HNO_3/HF	ICP-MS with matrix matched standards (Al 5N5), calibration with pure metals or pure substances
5	Si, Fe, Cu, Mg, Mn, Cr, Ni, Zn, Ti, Zr, Ag, Be, Bi, Ga, In, Pb, Sn, V	0.5 g	Dissolution with NaOH	ICP-OES
6	Si, Fe, Cu, Mg, Mn, Cr, Ni, Zn, Ti, Zr, Ag, B, Be, Bi, Ga, In, Pb, Sn, V	0.5 g	Dissolution with NaOH	ICP-OES, calibration with pure metals or pure chemicals
	Fe, Cu, Mg, Mn, Cr, Ni, Zn, Ti, Zr, Ag, B, Bi, Ca, Ga, Pb	0.5 g	Dissolution with HCl	ICP-OES, calibration with pure metals or pure chemicals
	P	0.5 g	Dissolution with HNO_3/HF	ICP-OES, calibration with pure chemicals

Table 2 (cont.): Analytical procedures used by the participating laboratories

8	Si, Fe, Cu, Mn, Cr, Ni, Ti, Zr, Ga,	0.3 g	Dissolution with HNO_3	XRF, reconstitution
9	Fe, Mn, Mg, Cr, Ni, Ti, Zr, In, V	1 g	Dissolution with HNO_3/HF	ICP-OES
	Fe, Cu, Mg, Ni, Zn,	1 g	Dissolution with HNO_3/HF	FAAS
10	Si, Fe, Cu, Mn, Mg, Cr, Ni, Zn, Ti, Zr, Be, Sn, V	0.5 g	Dissolution with NaOH	ICP-OES, calibration with pure metals or pure chemicals
	Si	0.5 g	Dissolution with NaOH	Spectrophotometry, calibration with pure chemicals
	Fe, Cu, Mn, Mg, Cr, Ni, Zn, Ti, B, Be, Ca, Ga, Sn, V	1 g	Dissolution with HCl/HNO_3	ICP-OES, calibration with pure metals or pure chemicals
	Ag, Be, Bi, Ga, In, P, Pb, Sn, V	1 g	Dissolution with HNO_3/H_2SO_4	ICP-MS, calibration with pure metals or pure chemicals
11	Fe, Cu, Mn, Cr, Ni, Ti, B, Be, Bi, Ca, Ga, In, P, Pb	0.5 g	Dissolution with HCl	ICP-OES, calibration with pure metals or pure chemicals
	Ag	0.5 g	Dissolution with NaOH	ICP-OES, calibration with pure metals or pure chemicals
12	Bi, In	1 g	Dissolution with HCl	ICP-OES
13	Si	0.5 g	Dissolution with NaOH	ICP-OES, calibration with commercial mono element solutions
	Fe, Cu, Mn, Mg, Cr, Ni, Zn, Ti, Zr, B, Be, Bi, Ga, In, P, Pb, Sn, V	0.5 g	Dissolution with HCl/H_2O_2	ICP-OES, calibration with commercial mono element solutions

5.2 Analytical results and statistical evaluation

The analytical results of the certification interlaboratory comparison are listed in Tables 24 to 44. These tables show the single results (M_i) of each laboratory, the respective laboratory mean (M) together with the relative intralaboratory standard deviation (s_{rel}) and in addition the mean repeatability standard deviation ($\bar{s}_i$) over all laboratories. The continuous line marks the certified value (mean of the laboratories' means), the broken lines mark the standard deviation, calculated from the laboratories' means.
In the related figures for each laboratory its mean value and single standard deviation is given.

The statistical evaluation of the data was performed using the software program SoftCRM 1.2.2. [7]. The following results were received:

Table 3: Outcome of statistical tests of results obtained for Si

Number of data sets	11
Snedecor-F-Test and Bartlett-Test	Pooling not allowed
Dixon (α = 0.05)	---
Dixon (α = 0.01)	---
Nalimov (α = 0.05)	---
Nalimov (α = 0.01)	---
Grubbs (α = 0.05)	---
Grubbs (α = 0.01)	---
Grubbs Pair (α = 0.05)	---
Grubbs Pair (α = 0.01)	---
Cochran	---
Kolmogorov-Smirnov-Lilliefors Test (α = 0.05)	Distribution: normal
Kolmogorov-Smirnov-Lilliefors Test (α = 0.01)	Distribution: normal

Table 4: Outcome of statistical tests of results obtained for Fe

Number of data sets	15
Snedecor-F-Test and Bartlett-Test	Pooling not allowed
Dixon (α = 0.05)	Laboratories 8 and 9/I-s
Dixon (α = 0.01)	---
Nalimov (α = 0.05)	Laboratories 8 and 9/I-s
Nalimov (α = 0.01)	Laboratory 8
Grubbs (α = 0.05)	Laboratory 8
Grubbs (α = 0.01)	---
Grubbs Pair (α = 0.05)	---
Grubbs Pair (α = 0.01)	---
Cochran	---
Kolmogorov-Smirnov-Lilliefors Test (α = 0.05)	Distribution: normal
Kolmogorov-Smirnov-Lilliefors Test (α = 0.01)	Distribution: normal

The outlying values (Lab. 8 and 9/I-s) were not removed.

Table 5: Outcome of statistical tests of results obtained for Cu

Number of data sets	13
Snedecor-F-Test and Bartlett-Test	Pooling not allowed
Dixon (α = 0.05)	---
Dixon (α = 0.01)	---
Nalimov (α = 0.05)	Laboratory 10/I-a
Nalimov (α = 0.01)	---
Grubbs (α = 0.05)	---
Grubbs (α = 0.01)	---
Grubbs Pair (α = 0.05)	---
Grubbs Pair (α = 0.01)	---
Cochran	---
Kolmogorov-Smirnov-Lilliefors Test (α = 0.05)	Distribution: not normal
Kolmogorov-Smirnov-Lilliefors Test (α = 0.01)	Distribution: not normal

The outlying value (Lab. 10/I-a) was not removed.

Table 5: Outcome of statistical tests of results obtained for Mn

Number of data sets	14
Snedecor-F-Test and Bartlett-Test	Pooling not allowed
Dixon (α = 0.05)	---
Dixon (α = 0.01)	---
Nalimov (α = 0.05)	Laboratory 9/A-s
Nalimov (α = 0.01)	Laboratory 9/A-s
Grubbs (α = 0.05)	Laboratory 9/A-s
Grubbs (α = 0.01)	---
Grubbs Pair (α = 0.05)	---
Grubbs Pair (α = 0.01)	---
Cochran	---
Kolmogorov-Smirnov-Lilliefors Test (α = 0.05)	Distribution: normal
Kolmogorov-Smirnov-Lilliefors Test (α = 0.01)	Distribution: normal

The outlying value (Lab. 9/A-s) was removed.

Table 5a: Outcome of statistical tests of results obtained for Mn (after removal of outlier)

Number of data sets	13
Snedecor-F-Test and Bartlett-Test	Pooling not allowed
Dixon (α = 0.05)	---
Dixon (α = 0.01)	---
Nalimov (α = 0.05)	---
Nalimov (α = 0.01)	---
Grubbs (α = 0.05)	---
Grubbs (α = 0.01)	---
Grubbs Pair (α = 0.05)	---
Grubbs Pair (α = 0.01)	---
Cochran	---
Kolmogorov-Smirnov-Lilliefors Test (α = 0.05)	Distribution: normal
Kolmogorov-Smirnov-Lilliefors Test (α = 0.01)	Distribution: normal

Table 6: Outcome of statistical tests of results obtained for Mg

Number of data sets	13
Snedecor-F-Test and Bartlett-Test	Pooling not allowed
Dixon (α = 0.05)	---
Dixon (α = 0.01)	---
Nalimov (α = 0.05)	---
Nalimov (α = 0.01)	---
Grubbs (α = 0.05)	---
Grubbs (α = 0.01)	---
Grubbs Pair (α = 0.05)	---
Grubbs Pair (α = 0.01)	---
Cochran	---
Kolmogorov-Smirnov-Lilliefors Test (α = 0.05)	Distribution: normal
Kolmogorov-Smirnov-Lilliefors Test (α = 0.01)	Distribution: normal

Table 7: Outcome of statistical tests of results obtained for Cr

Number of data sets	14
Snedecor-F-Test and Bartlett-Test	Pooling not allowed
Dixon (α = 0.05)	---
Dixon (α = 0.01)	---
Nalimov (α = 0.05)	Laboratory 8/INAA
Nalimov (α = 0.01)	---
Grubbs (α = 0.05)	---
Grubbs (α = 0.01)	---
Grubbs Pair (α = 0.05)	---
Grubbs Pair (α = 0.01)	---
Cochran	---
Kolmogorov-Smirnov-Lilliefors Test (α = 0.05)	Distribution: normal
Kolmogorov-Smirnov-Lilliefors Test (α = 0.01)	Distribution: normal

The outlying value (Lab. 8/INAA) was not removed.

Table 8: Outcome of statistical tests of results obtained for Ni

Number of data sets	14
Snedecor-F-Test and Bartlett-Test	Pooling not allowed
Dixon (α = 0.05)	Laboratories 8 and 9/I-s
Dixon (α = 0.01)	---
Nalimov (α = 0.05)	Laboratories 8 and 9/I-s
Nalimov (α = 0.01)	---
Grubbs (α = 0.05)	---
Grubbs (α = 0.01)	---
Grubbs Pair (α = 0.05)	---
Grubbs Pair (α = 0.01)	---
Cochran	---
Kolmogorov-Smirnov-Lilliefors Test (α = 0.05)	Distribution: normal
Kolmogorov-Smirnov-Lilliefors Test (α = 0.01)	Distribution: normal

The outlying values (Lab. 8 and 9/I-s) were not removed.

Table 9: Outcome of statistical tests of results obtained for Zn

Number of data sets	13
Snedecor-F-Test and Bartlett-Test	Pooling not allowed
Dixon (α = 0.05)	---
Dixon (α = 0.01)	---
Nalimov (α = 0.05)	Laboratory 8/XRF
Nalimov (α = 0.01)	---
Grubbs (α = 0.05)	---
Grubbs (α = 0.01)	---
Grubbs Pair (α = 0.05)	---
Grubbs Pair (α = 0.01)	---
Cochran	---
Kolmogorov-Smirnov-Lilliefors Test (α = 0.05)	Distribution: normal
Kolmogorov-Smirnov-Lilliefors Test (α = 0.01)	Distribution: normal

The outlying value (Lab. 8/XRF) was not removed.

Table 10: Outcome of statistical tests of results obtained for Ti

Number of data sets	14
Snedecor-F-Test and Bartlett-Test	Pooling not allowed
Dixon (α = 0.05)	---
Dixon (α = 0.01)	---
Nalimov (α = 0.05)	Laboratory 9
Nalimov (α = 0.01)	---
Grubbs (α = 0.05)	---
Grubbs (α = 0.01)	---
Grubbs Pair (α = 0.05)	---
Grubbs Pair (α = 0.01)	---
Cochran	---
Kolmogorov-Smirnov-Lilliefors Test (α = 0.05)	Distribution: normal
Kolmogorov-Smirnov-Lilliefors Test (α = 0.01)	Distribution: normal

The outlying value (Lab. 9) was not removed.

Table 11: Outcome of statistical tests of results obtained for Zr

Number of data sets	11
Snedecor-F-Test and Bartlett-Test	Pooling not allowed
Dixon (α = 0.05)	---
Dixon (α = 0.01)	---
Nalimov (α = 0.05)	Laboratories 8 and 9
Nalimov (α = 0.01)	---
Grubbs (α = 0.05)	---
Grubbs (α = 0.01)	---
Grubbs Pair (α = 0.05)	---
Grubbs Pair (α = 0.01)	---
Cochran	---
Kolmogorov-Smirnov-Lilliefors Test (α = 0.05)	Distribution: normal
Kolmogorov-Smirnov-Lilliefors Test (α = 0.01)	Distribution: normal

The outlying values (Lab. 8 and 9) were not removed.

Table 12: Outcome of statistical tests of results obtained for Ag

Number of data sets	11
Snedecor-F-Test and Bartlett-Test	Pooling not allowed
Dixon (α = 0.05)	---
Dixon (α = 0.01)	---
Nalimov (α = 0.05)	Laboratory 1
Nalimov (α = 0.01)	---
Grubbs (α = 0.05)	---
Grubbs (α = 0.01)	---
Grubbs Pair (α = 0.05)	---
Grubbs Pair (α = 0.01)	---
Cochran	---
Kolmogorov-Smirnov-Lilliefors Test (α = 0.05)	Distribution: not normal
Kolmogorov-Smirnov-Lilliefors Test (α = 0.01)	Distribution: normal

The outlying value (Lab. 1) was not removed.

Table 13: Outcome of statistical tests of results obtained for B

Number of data sets	7
Snedecor-F-Test and Bartlett-Test	Pooling not allowed
Dixon (α = 0.05)	---
Dixon (α = 0.01)	---
Nalimov (α = 0.05)	---
Nalimov (α = 0.01)	---
Grubbs (α = 0.05)	---
Grubbs (α = 0.01)	---
Grubbs Pair (α = 0.05)	---
Grubbs Pair (α = 0.01)	---
Cochran	---
Kolmogorov-Smirnov-Lilliefors Test (α = 0.05)	Distribution: normal
Kolmogorov-Smirnov-Lilliefors Test (α = 0.01)	Distribution: normal

Table 14: Outcome of statistical tests of results obtained for Be

Number of data sets	10
Snedecor-F-Test and Bartlett-Test	Pooling not allowed
Dixon (α = 0.05)	---
Dixon (α = 0.01)	---
Nalimov (α = 0.05)	---
Nalimov (α = 0.01)	---
Grubbs (α = 0.05)	---
Grubbs (α = 0.01)	---
Grubbs Pair (α = 0.05)	---
Grubbs Pair (α = 0.01)	---
Cochran	---
Kolmogorov-Smirnov-Lilliefors Test (α = 0.05)	Distribution: normal
Kolmogorov-Smirnov-Lilliefors Test (α = 0.01)	Distribution: normal

Table 15: Outcome of statistical tests of results obtained for Bi

Number of data sets	8
Snedecor-F-Test and Bartlett-Test	Pooling not allowed
Dixon (α = 0.05)	---
Dixon (α = 0.01)	---
Nalimov (α = 0.05)	---
Nalimov (α = 0.01)	---
Grubbs (α = 0.05)	---
Grubbs (α = 0.01)	---
Grubbs Pair (α = 0.05)	---
Grubbs Pair (α = 0.01)	---
Cochran	---
Kolmogorov-Smirnov-Lilliefors Test (α = 0.05)	Distribution: normal
Kolmogorov-Smirnov-Lilliefors Test (α = 0.01)	Distribution: normal

Table 16: Outcome of statistical tests of results obtained for Ca

Number of data sets	4
Snedecor-F-Test and Bartlett-Test	Pooling not allowed
Dixon (α = 0.05)	---
Dixon (α = 0.01)	---
Nalimov (α = 0.05)	Laboratory 11
Nalimov (α = 0.01)	---
Grubbs (α = 0.05)	---
Grubbs (α = 0.01)	---
Grubbs Pair (α = 0.05)	---
Grubbs Pair (α = 0.01)	---
Cochran	---
Kolmogorov-Smirnov-Lilliefors Test (α = 0.05)	Distribution: normal
Kolmogorov-Smirnov-Lilliefors Test (α = 0.01)	Distribution: normal

The outlying value (Lab. 11) was not removed.

Table 17: Outcome of statistical tests of results obtained for Ga

Number of data sets	13
Snedecor-F-Test and Bartlett-Test	Pooling not allowed
Dixon (α = 0.05)	---
Dixon (α = 0.01)	---
Nalimov (α = 0.05)	---
Nalimov (α = 0.01)	---
Grubbs (α = 0.05)	---
Grubbs (α = 0.01)	---
Grubbs Pair (α = 0.05)	---
Grubbs Pair (α = 0.01)	---
Cochran	---
Kolmogorov-Smirnov-Lilliefors Test (α = 0.05)	Distribution: normal
Kolmogorov-Smirnov-Lilliefors Test (α = 0.01)	Distribution: normal

Table 18: Outcome of statistical tests of results obtained for In

Number of data sets	11
Snedecor-F-Test and Bartlett-Test	Pooling not allowed
Dixon (α = 0.05)	---
Dixon (α = 0.01)	---
Nalimov (α = 0.05)	Laboratory 8
Nalimov (α = 0.01)	---
Grubbs (α = 0.05)	---
Grubbs (α = 0.01)	---
Grubbs Pair (α = 0.05)	---
Grubbs Pair (α = 0.01)	---
Cochran	---
Kolmogorov-Smirnov-Lilliefors Test (α = 0.05)	Distribution: normal
Kolmogorov-Smirnov-Lilliefors Test (α = 0.01)	Distribution: normal

The outlying value (Lab. 8) was not removed.

Table 19: Outcome of statistical tests of results obtained for P

Number of data sets	5
Snedecor-F-Test and Bartlett-Test	Pooling not allowed
Dixon (α = 0.05)	---
Dixon (α = 0.01)	---
Nalimov (α = 0.05)	---
Nalimov (α = 0.01)	---
Grubbs (α = 0.05)	---
Grubbs (α = 0.01)	---
Grubbs Pair (α = 0.05)	---
Grubbs Pair (α = 0.01)	---
Cochran	---
Kolmogorov-Smirnov-Lilliefors Test (α = 0.05)	Distribution: normal
Kolmogorov-Smirnov-Lilliefors Test (α = 0.01)	Distribution: normal

Table 20: Outcome of statistical tests of results obtained for Pb

Number of data sets	10
Snedecor-F-Test and Bartlett-Test	Pooling not allowed
Dixon (α = 0.05)	---
Dixon (α = 0.01)	---
Nalimov (α = 0.05)	Laboratory 5
Nalimov (α = 0.01)	---
Grubbs (α = 0.05)	---
Grubbs (α = 0.01)	---
Grubbs Pair (α = 0.05)	---
Grubbs Pair (α = 0.01)	---
Cochran	---
Kolmogorov-Smirnov-Lilliefors Test (α = 0.05)	Distribution: not normal
Kolmogorov-Smirnov-Lilliefors Test (α = 0.01)	Distribution: normal

The outlying value (Lab. 5) was not removed.

Table 21: Outcome of statistical tests of results obtained for Sn

Number of data sets	11
Snedecor-F-Test and Bartlett-Test	Pooling not allowed
Dixon (α = 0.05)	Laboratory 8
Dixon (α = 0.01)	---
Nalimov (α = 0.05)	Laboratory 8
Nalimov (α = 0.01)	Laboratory 8
Grubbs (α = 0.05)	Laboratory 8
Grubbs (α = 0.01)	---
Grubbs Pair (α = 0.05)	---
Grubbs Pair (α = 0.01)	---
Cochran	---
Kolmogorov-Smirnov-Lilliefors Test (α = 0.05)	Distribution: not normal
Kolmogorov-Smirnov-Lilliefors Test (α = 0.01)	Distribution: normal

The outlying value (Lab. 8) was removed.

Table 21a: Outcome of statistical tests of results obtained for Sn (after removal of outlier)

Number of data sets	10
Snedecor-F-Test and Bartlett-Test	Pooling not allowed
Dixon (α = 0.05)	---
Dixon (α = 0.01)	---
Nalimov (α = 0.05)	Laboratory 3
Nalimov (α = 0.01)	---
Grubbs (α = 0.05)	---
Grubbs (α = 0.01)	---
Grubbs Pair (α = 0.05)	---
Grubbs Pair (α = 0.01)	---
Cochran	---
Kolmogorov-Smirnov-Lilliefors Test (α = 0.05)	Distribution: normal
Kolmogorov-Smirnov-Lilliefors Test (α = 0.01)	Distribution: normal

The outlying value (Lab. 3) was not removed.

Table 22: Outcome of statistical tests of results obtained for V

Number of data sets	11
Snedecor-F-Test and Bartlett-Test	Pooling not allowed
Dixon (α = 0.05)	---
Dixon (α = 0.01)	---
Nalimov (α = 0.05)	---
Nalimov (α = 0.01)	---
Grubbs (α = 0.05)	---
Grubbs (α = 0.01)	---
Grubbs Pair (α = 0.05)	---
Grubbs Pair (α = 0.01)	---
Cochran	---
Kolmogorov-Smirnov-Lilliefors Test (α = 0.05)	Distribution: normal
Kolmogorov-Smirnov-Lilliefors Test (α = 0.01)	Distribution: normal

The certified mass fractions of all elements were calculated as mean of the accepted data sets. These values are given in Table 23.
The respective combined uncertainties were calculated from the contribution resulting from the certification interlaboratory comparison (u_{ilc}) and the uncertainty contributions from possible inhomogeneity of the material using Equation (5).

$$u_{\text{combined}} = \sqrt{u_{ilc}^2 + u_{bb}^2(1) + u_{bb}^2(2)} \qquad (5)$$

with

$u_{\text{ilc}} = \sqrt{\dfrac{s_{\text{M}}^2}{n}}$: uncertainty contribution resulting from interlaboratory comparison

$u_{\text{bb}}(1)$ = inhomogeneity contribution due to a possible composition gradient over the length of the rod

$u_{\text{bb}}(2)$ = inhomogeneity contribution due to a possible composition gradient from the outer part of the disc to its centre

Table 23: Uncertainty calculation

	M	***n***	s_M	u_{bb} (1)	u_{bb} (2)	$u_{combined}$	*U*
Fe	0.1115	15	0.0033	0.00103	0.00051	0.00144	0.00286
Cu	1.77	13	0.015	0.00599	0.0264	0.0274	0.0548
Mg	2.387	13	0.067	0.0161	0.023	0.0337	0.0674
Cr	0.1414	14	0.0016	0.00042	0.00105	0.00121	0.00242
Zn	6.928	13	0.127	0.0214	0.1199	0.127	0.254
Zr	0.1305	11	0.0066	0.00062	0.00291	0.00358	0.00716
Si	271	11	23	5.4	5.9	10.59	21.18
Mn	912	13	12	3.3	8.0	9.23	18.45
Ni	359	15	15	2	5.5	6.99	13.98
Ti	952	14	39	73	25	77.8	155.6
Ag	72.5	11	7.8	0.3	0.57	2.44	4.88
Be	10.08	10	1.9	0.2	2.7	2.79	5.58
Ga	182.6	13	16	3.1	2.36	5.91	11.82
In	161.8	12	7.5	0.6	4.6	5.11	10.21
Pb	48.1	10	2.9	0.3	0.57	1.12	2.25
Sn	238	10	8	0.3	8.4	8.78	17.6
V	104.9	11	2.9	1.8	2.73	3.39	6.77
B	37.2	7	6.66	14.9	4.57	15.79	31.6
Ca	6.05	4	1.25	0.5	0.4	0.90	2.69
P	26.7	5	7.4	1.8	2.9	4.73	14.2

Note: For lead the same behaviour as for Ag was assumed

The expanded uncertainties *U* were calculated by multiplication of $u_{combined}$ with a coverage factor of k = 2 (k = 3 for Ca and P) using Equation 6.

$$U = k \cdot u_{\text{combined}} \qquad (6)$$

The calculated mass fractions and their respective expanded uncertainties are given on Page 3 of this report.

Lab./Meth.	6/I-a	5/I-a	4/I-a	8/XRF	2/I-s(R)	1/I-a	3/I-s(R)	2/P	10/P	13/I-a	10/I-a		N
M_i [%]	0.0234	0.0250	0.0252	0.0292	0.0254	0.0264	0.0287	0.0289	0.0285	0.0300	0.0301		
	0.0235	0.0250	0.0249	0.0254	0.0259	0.0268	0.0287	0.0297	0.0297	0.0310	0.0317		11
	0.0220	0.0240	0.0249	0.0234	0.0251	0.0266	0.0282	0.0291	0.0296	0.0290	0.0293		
	0.0234	0.0250	0.0252	0.0225	0.0258	0.0263	0.0279	0.0287	0.0304	0.0290	0.0296		
	0.0242	0.0270	0.0252		0.0282	0.0265		0.0283	0.0284	0.0310	0.0318		
	0.0244	0.0240	0.0253			0.0265		0.0285	0.0279	0.0290	0.0290		
M [%]	**0.0235**	**0.0250**	**0.0251**	**0.0251**	**0.0261**	**0.0265**	**0.0284**	**0.0289**	**0.0291**	**0.0298**	**0.0303**		**0.0271**
s_M [%]	0.0008	0.0011	0.0001	0.0030	0.0012	0.0001	0.0004	0.0005	0.0010	0.0010	0.0012		0.0023
$\bar{s}_i$ [%]													0.0012
s_{rel}	0.03597	0.04382	0.00518	0.11841	0.04707	0.00563	0.01391	0.01706	0.03308	0.03296	0.04026		0.08488

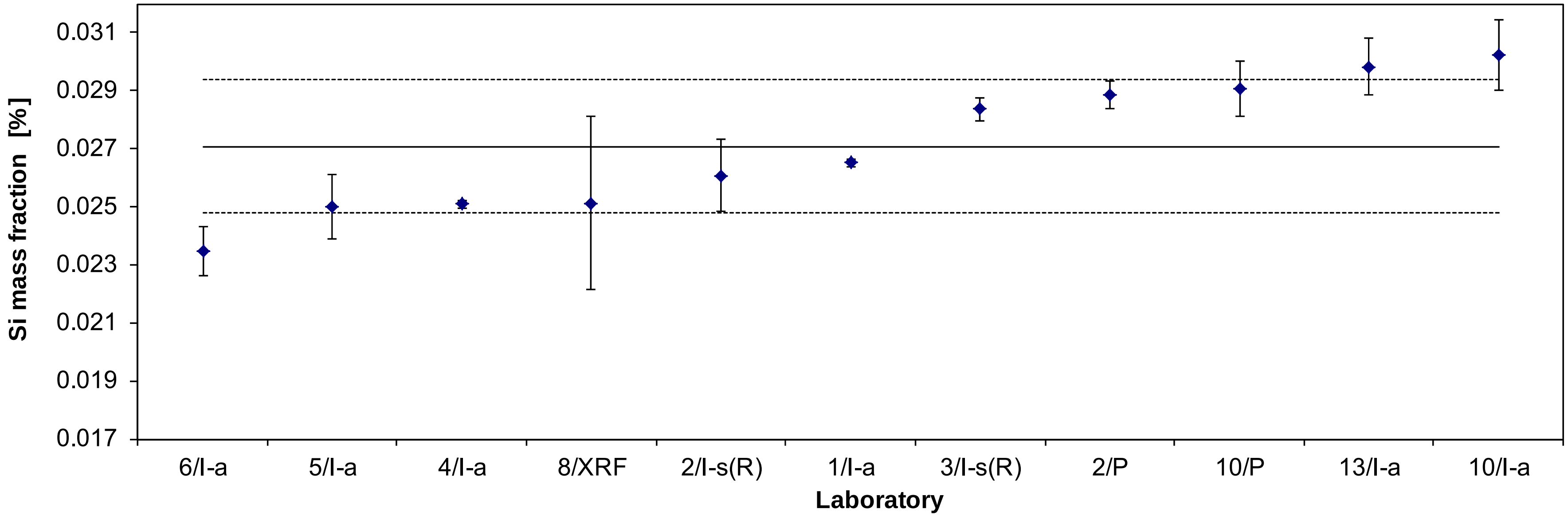

Table 24: Results for Si

Lab./Meth.	9/I-s(R)	9/A-s(R)	6/I-s	11/I	6/I-a	3/I-s	4/I-a	10/I-s	2/I-s	1/I-a	8/INAA	5/I-a	13/I-s	10/I-a	8/XRF		N
M_i [%]	0.1047	0.1062	0.1088	0.1107	0.1100	0.1107	0.1118	0.1111	0.1146	0.113	0.1137	0.1163	0.114	0.1149	0.1209		
	0.1050	0.1061	0.1106	0.1108	0.1110	0.1095	0.1115	0.1104	0.1124	0.113	0.1114	0.1106	0.112	0.1164	0.1193		15
	0.1054	0.1070	0.1103	0.1089	0.1110	0.1103	0.1109	0.1121	0.1114	0.112	0.1170	0.1145	0.113	0.1131	0.1227		
				0.1093		0.1115	0.1112	0.1115	0.1113	0.112	0.1106	0.1110	0.113	0.1138	0.1156		
				0.1075		0.1140	0.1112	0.1119	0.1112	0.113	0.1107	0.1112	0.114	0.1132			
				0.1130		0.1108	0.1116	0.1117	0.1126	0.112	0.1105	0.1121	0.114	0.1131			
***M* [%]**	**0.1050**	**0.1064**	**0.1099**	**0.1100**	**0.1107**	**0.1111**	**0.1114**	**0.1115**	**0.1123**	**0.1123**	**0.1123**	**0.1126**	**0.1133**	**0.1141**	**0.1196**		**0.1115**
s_M [%]	0.0004	0.0005	0.0010	0.0019	0.0006	0.0016	0.0003	0.0006	0.0013	0.0006	0.0026	0.0023	0.0008	0.0013	0.0030		0.0033
$\bar{s}_i$ [%]																	0.0015
s_{rel}	0.00334	0.00463	0.00877	0.01728	0.00522	0.01396	0.00301	0.00555	0.01154	0.00514	0.02306	0.02028	0.00720	0.01165	0.02542		0.02955

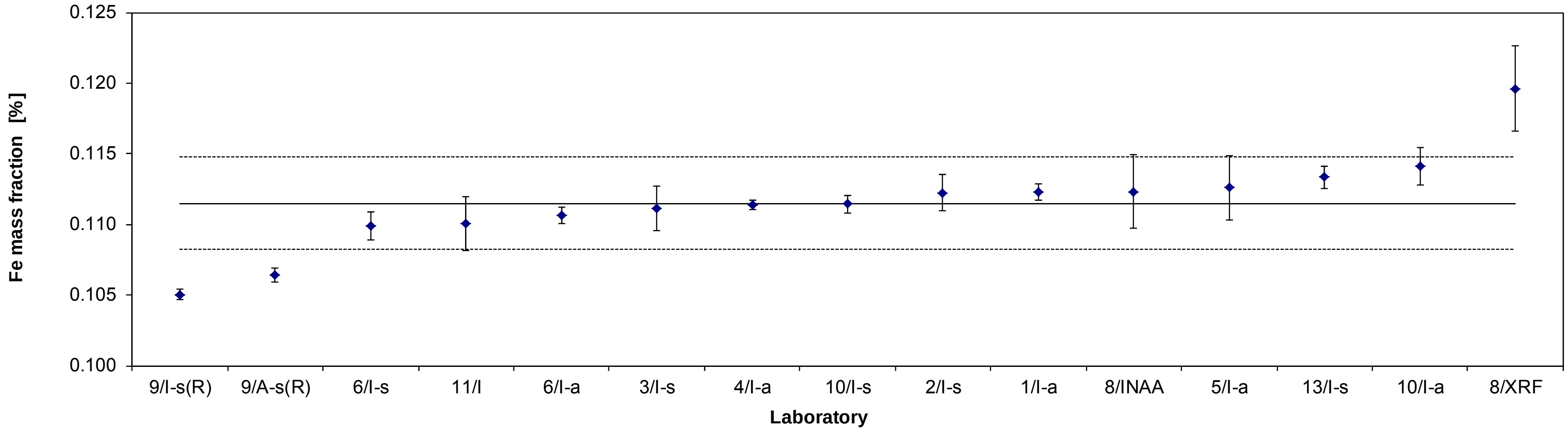

Table 25: Results for Fe

Lab./Meth.	11/I-s	5/I-a	6/I-s	13/I-s	10/I-s	4/I-a	2/I-s	6/I-a	3/I-s	1/I-a	9/A	8/XRF	10/I-a		N
M_i [%]	1.728	1.770	1.750	1.760	1.762	1.7652	1.774	1.760	1.759	1.774	1.7600	1.806	1.822		
	1.715	1.763	1.760	1.770	1.751	1.7620	1.750	1.770	1.755	1.788	1.7770	1.782	1.821		13
	1.737	1.747	1.770	1.760	1.761	1.7652	1.705	1.770	1.772	1.786	1.7890	1.861	1.793		
	1.756	1.766		1.760	1.769	1.7598	1.821		1.767	1.769	1.7980	1.714	1.795		
	1.765	1.763		1.750	1.763	1.7653	1.778		1.768	1.779	1.7880		1.785		
	1.836	1.749		1.760	1.760	1.7615	1.758		1.780	1.768	1.8000		1.791		
***M* [%]**	**1.7560**	**1.7594**	**1.7600**	**1.7600**	**1.7610**	**1.7632**	**1.7643**	**1.7667**	**1.7669**	**1.7773**	**1.7853**	**1.7906**	**1.8012**		**1.7701**
s_M [%]	0.0434	0.0093	0.0100	0.0063	0.0058	0.0024	0.0381	0.0058	0.0088	0.0085	0.0149	0.0611	0.0161		0.0141
$\bar{s}_i$ [%]															0.0248
s_{rel}	0.02470	0.00526	0.00568	0.00359	0.00331	0.00135	0.02159	0.00327	0.00499	0.00477	0.00834	0.03414	0.00894		0.00795

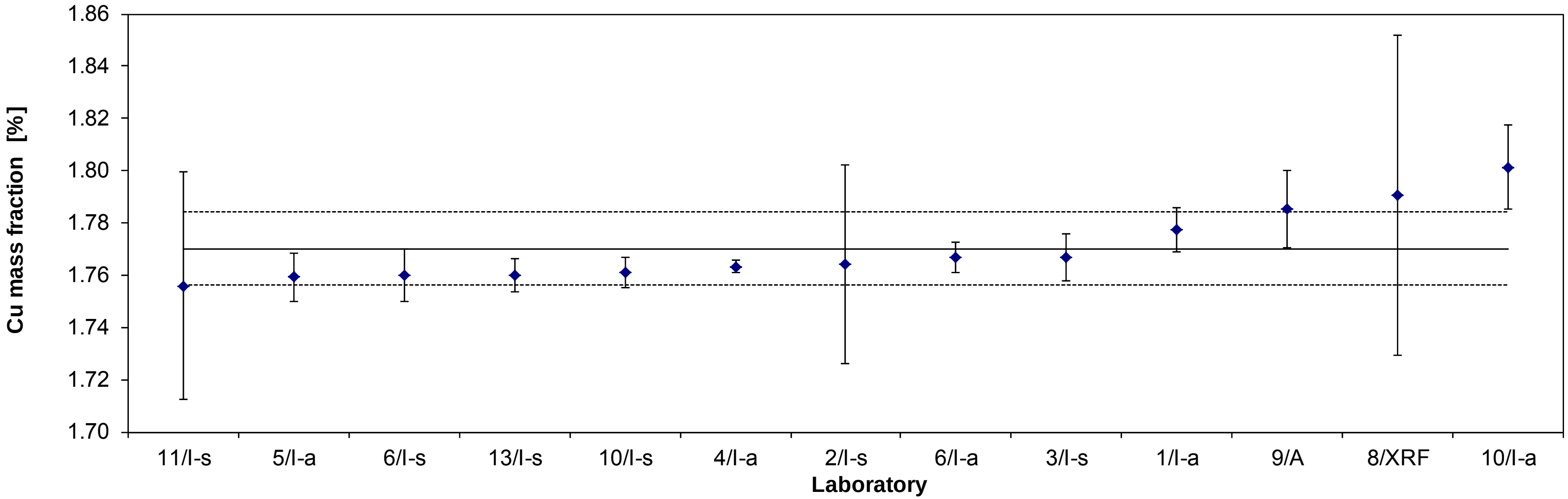

Table 26: Results for Cu

Lab./Meth.	9/A-s	9/I-s	5/I-a	6/I-a	6/I-s	11/I-s	1/I-a	3/I-s	2/I-s	10/I-s	4/I-a	10/I-a	8/INAA		N
M_i [%]	0.0868	0.0907	0.0896	0.0897	0.0910	0.0893	0.0906	0.0909	0.0917	0.0915	0.0915	0.0937	0.0933		
	0.0875	0.0904	0.0904	0.0908	0.0910	0.0898	0.0908	0.0907	0.0916	0.0913	0.0914	0.0938	0.0920		13
	0.0861	0.0892	0.0889	0.0911	0.0900	0.0905	0.0908	0.0900	0.0913	0.0915	0.0917	0.0921	0.0937		
	0.0861	0.0877	0.0896			0.0906	0.0909	0.0918	0.0916	0.0914	0.0916	0.0927	0.0937		
	0.0864	0.0891	0.0896			0.0880	0.0905	0.0913	0.0911	0.0919	0.0919	0.0918	0.0956		
	0.0868	0.0889	0.0892			0.0961	0.0906	0.0913	0.0910	0.0921	0.0917	0.0918	0.0912		
***M* [%]**	**0.0866**	**0.0893**	**0.0895**	**0.0905**	**0.0907**	**0.0907**	**0.0907**	**0.0910**	**0.0914**	**0.0916**	**0.0916**	**0.0927**	**0.0933**		**0.0911**
s_M [%]	0.0005	0.0011	0.0005	0.0007	0.0006	0.0028	0.0002	0.0006	0.0003	0.0003	0.0002	0.0009	0.0015		0.0011
$\bar{s}_i$ [%]															0.0011
s_{rel}	0.00617	0.01219	0.00537	0.00814	0.00637	0.03089	0.00171	0.00671	0.00320	0.00341	0.00181	0.00986	0.01640		0.01241

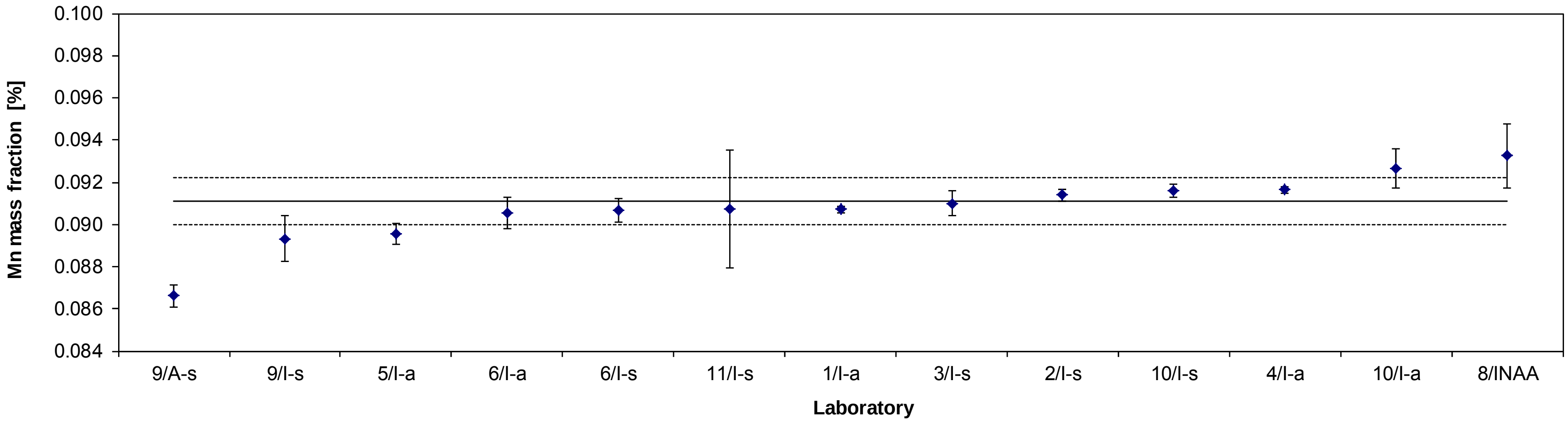

Table 27: Results for Mn

Lab./Meth.	13/I-s	9/A(R)	6/I-s	6/I-a	3/I-s	4/I-a	10/I-s	9/I-s	1/I-a	2/I-s	10/I-a	5/I-a	8/XRF		N
M_i [%]	2.280	2.298	2.320	2.330	2.363	2.3814	2.399	2.403	2.411	2.419	2.420	2.455	2.515		
	2.270	2.291	2.340	2.360	2.357	2.3847	2.384	2.404	2.413	2.419	2.448	2.452	2.505		13
	2.280	2.298	2.340	2.350	2.340	2.3799	2.428	2.401	2.403	2.363	2.429	2.431	2.527		
	2.260				2.378	2.3822	2.414	2.406	2.423	2.413	2.432	2.437	2.515		
	2.340				2.369	2.3797	2.419	2.419	2.408	2.458	2.418	2.456			
	2.160				2.380	2.3822	2.414	2.437	2.415	2.434	2.423	2.447			
M [%]	**2.2650**	**2.2957**	**2.3333**	**2.3467**	**2.3644**	**2.3817**	**2.4097**	**2.4117**	**2.4122**	**2.4177**	**2.4283**	**2.4463**	**2.5155**		**2.3868**
s_M [%]	0.0586	0.0040	0.0115	0.0153	0.0147	0.0019	0.0157	0.0140	0.0068	0.0313	0.0110	0.0105	0.0092		0.0663
$\bar{s}_i$ [%]															0.0212
s_{rel}	0.02586	0.00176	0.00495	0.00651	0.00622	0.00078	0.00652	0.00579	0.00280	0.01296	0.00453	0.00430	0.00365		0.02777

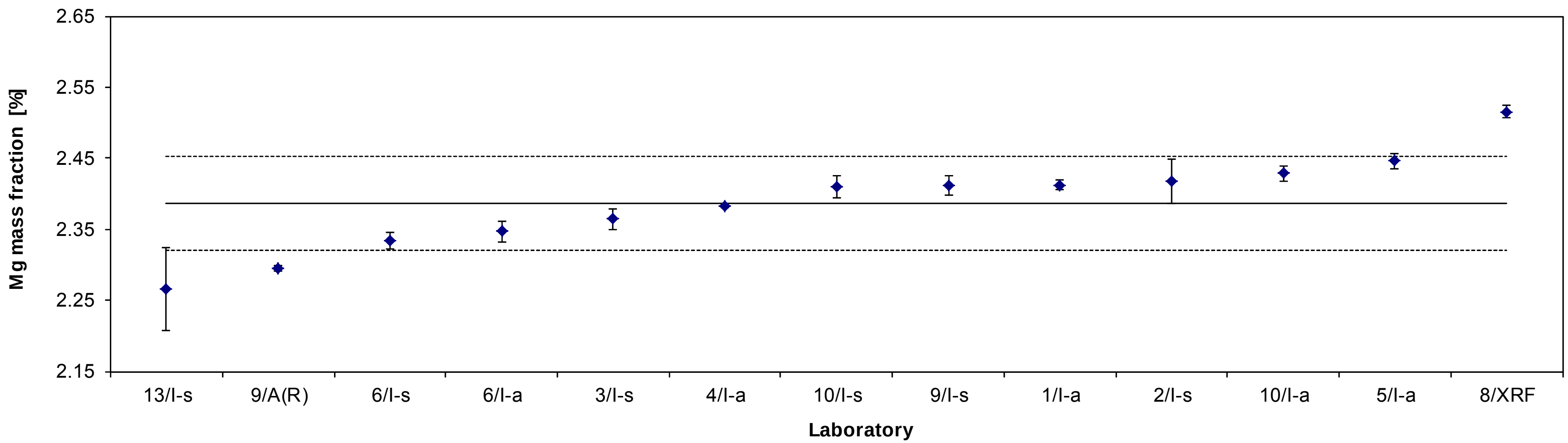

Table 28: Results for Mg

Lab./Meth.	8/INAA	10/I-s	6/I-a	9/I-s	5/I-a	4/I-a	8/XRF	11/I	1/I-a	2/I-s	3/I-s	13/I-s	6/I-a	10/I-a		N
M_i [%]	0.1376	0.1397	0.1406	0.1419	0.1432	0.1405	0.141	0.1399	0.1412	0.1420	0.1421	0.142	0.1440	0.1452		
	0.1374	0.1380	0.1411	0.1412	0.1415	0.1403	0.144	0.1417	0.1425	0.1415	0.1418	0.143	0.1440	0.1460		14
	0.1372	0.1405	0.1401	0.1406	0.1400	0.1405	0.134	0.1393	0.1417	0.1418	0.1411	0.143	0.1440	0.1438		
	0.1407	0.1397		0.1407	0.1386	0.1417	0.147	0.1397	0.1410	0.1425	0.1437	0.142		0.1433		
	0.1375	0.1403		0.1391	0.1426	0.1414		0.1398	0.1411	0.1406	0.1430	0.144		0.1432		
	0.1378	0.1400		0.1404	0.1392	0.1414		0.1474	0.1405	0.1411	0.1429	0.146		0.1433		
M [%]	**0.1380**	**0.1397**	**0.1406**	**0.1407**	**0.1408**	**0.1410**	**0.1413**	**0.1413**	**0.1413**	**0.1416**	**0.1424**	**0.1433**	**0.1440**	**0.1441**		**0.1414**
s_M [%]	0.0013	0.0009	0.0005	0.0009	0.0019	0.0006	0.0057	0.0031	0.0007	0.0007	0.0010	0.0015	0.0000	0.0012		0.0016
$\bar{s}_i$ [%]																0.0020
s_{rel}	0.00957	0.00639	0.00356	0.00662	0.01344	0.00426	0.04051	0.02196	0.00487	0.00476	0.00673	0.01050	0.00000	0.00821		0.01160

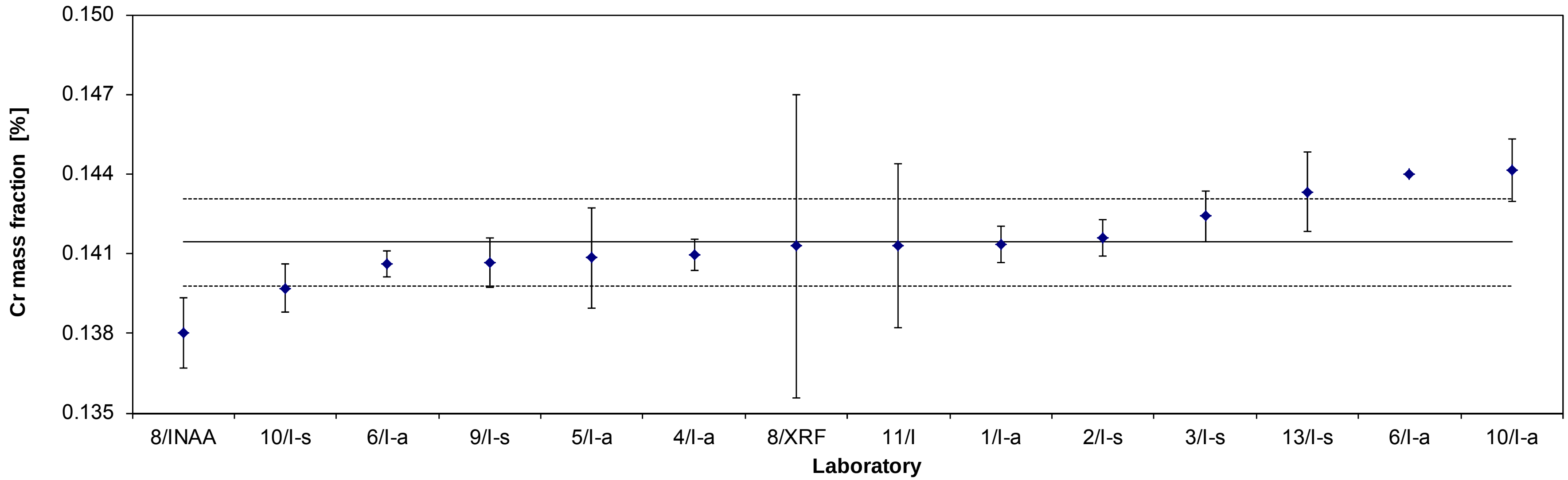

Table 29: Results for Cr

Lab./Meth.	9/I-s	9/A-s	4/I-s	6/I-a	11/I-s	10/I-s	13/I-s	6/I-s	1/I-a	2/I-s	10/I-a	3/I-s	2/P	5/I-a	8/XRF		N
M_i [%]	0.0334	0.0333	0.0351	0.0360	0.0352	0.0358	0.0360	0.0359	0.0360	0.0364	0.0364	0.0364	0.0357	0.0368	0.0391		
	0.0326	0.0339	0.0351	0.0350	0.0354	0.0357	0.0360	0.0360	0.0366	0.0385	0.0370	0.0364	0.0357	0.0375	0.0382		15
	0.0333	0.0341	0.0350	0.0360	0.0343	0.0360	0.0360	0.0362	0.0363	0.0356	0.0363	0.0363	0.0381	0.0372	0.0396		
	0.0329	0.0336	0.0350		0.0374	0.0359	0.0360		0.0363	0.0358	0.0364	0.0365	0.0367	0.0371	0.0393		
	0.0325	0.0340	0.0351		0.0358	0.0360	0.0360		0.0359	0.0355	0.0363	0.0365	0.0366	0.0367			
	0.0326	0.0328	0.0352		0.0369	0.0361	0.0360		0.0363	0.0367	0.0362	0.0369		0.0364			
M [%]	**0.0329**	**0.0336**	**0.0351**	**0.0357**	**0.0358**	**0.0359**	**0.0360**	**0.0360**	**0.0362**	**0.0364**	**0.0364**	**0.0365**	**0.0366**	**0.0369**	**0.0391**		**0.0359**
s_M [%]	0.0004	0.0005	0.0001	0.0006	0.0011	0.0001	0.0000	0.0002	0.0003	0.0011	0.0003	0.0002	0.0010	0.0004	0.0006		0.0014
$\bar{s}_i$ [%]																	0.0006
s_{rel}	0.01168	0.01474	0.00189	0.01619	0.03190	0.00410	0.00000	0.00424	0.00691	0.03084	0.00789	0.00571	0.02691	0.01085	0.01610		0.03901

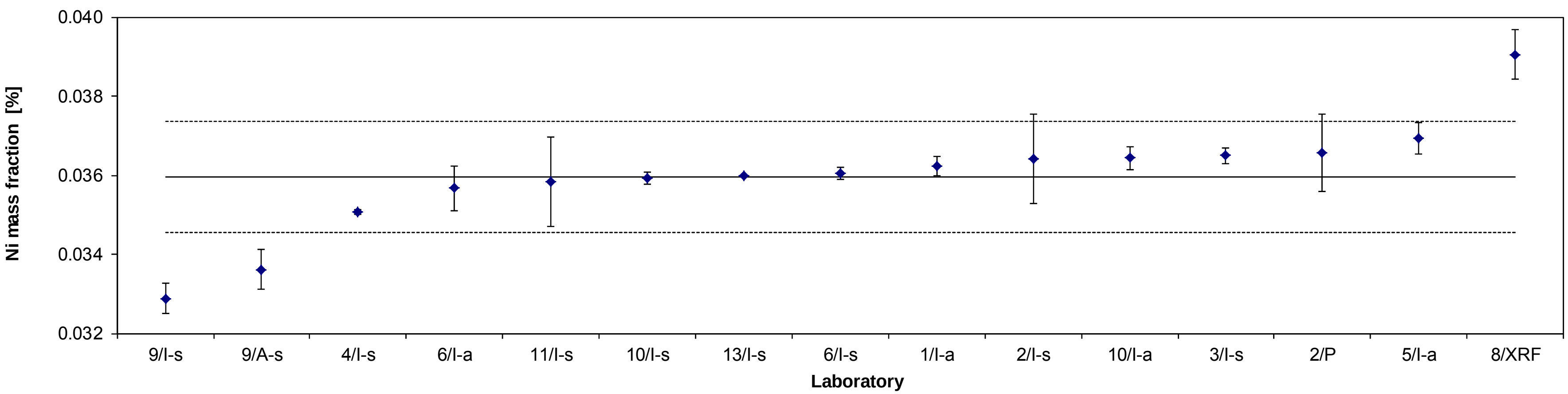

Table 30: Results for Ni

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Lab./Meth.	8/INAA	9/A-s	13/I-s	2/I-s	5/I-a	10/I-a	1/I-a	10/I-s	4/I-a	6/I-s	3/I-s	6/I-a	8/XRF		N
M_i [%]	6.7420	6.7700	6.8190	6.890	6.897	6.9120	6.910	6.9930	6.9682	6.910	6.985	7.060	7.189		
	6.7490	6.7890	6.7830	6.834	6.943	6.8870	6.925	6.9730	6.9381	6.940	6.973	7.100	7.127		13
	6.7290	6.6910	6.8110	6.645	6.879	7.0720	6.930	6.9450	6.9503	7.010	6.908	7.060	7.283		
	6.8590	6.7930	6.9270	6.880	6.884	6.8360	6.960	6.9460	6.9338		7.041		7.282		
	6.6720	6.6990	6.8280	6.964	6.895	6.8580	6.945	6.8933	6.9541		7.007				
	6.6970	6.8000	6.8360	6.860	6.884	6.9640	6.960	6.9233	6.9653		7.026				
***M* [%]**	**6.7413**	**6.7570**	**6.8340**	**6.8455**	**6.8969**	**6.9215**	**6.9383**	**6.9456**	**6.9516**	**6.9533**	**6.9899**	**7.0733**	**7.2203**		**6.9284**
s_M [%]	0.0645	0.0491	0.0491	0.1075	0.0236	0.0861	0.0202	0.0353	0.0139	0.0513	0.0473	0.0231	0.0762		0.1267
$\bar{s}_i$ [%]															0.0565
s_{rel}	0.00957	0.00727	0.00718	0.01570	0.00342	0.01244	0.00291	0.00508	0.00200	0.00738	0.00677	0.00326	0.01055		0.01829

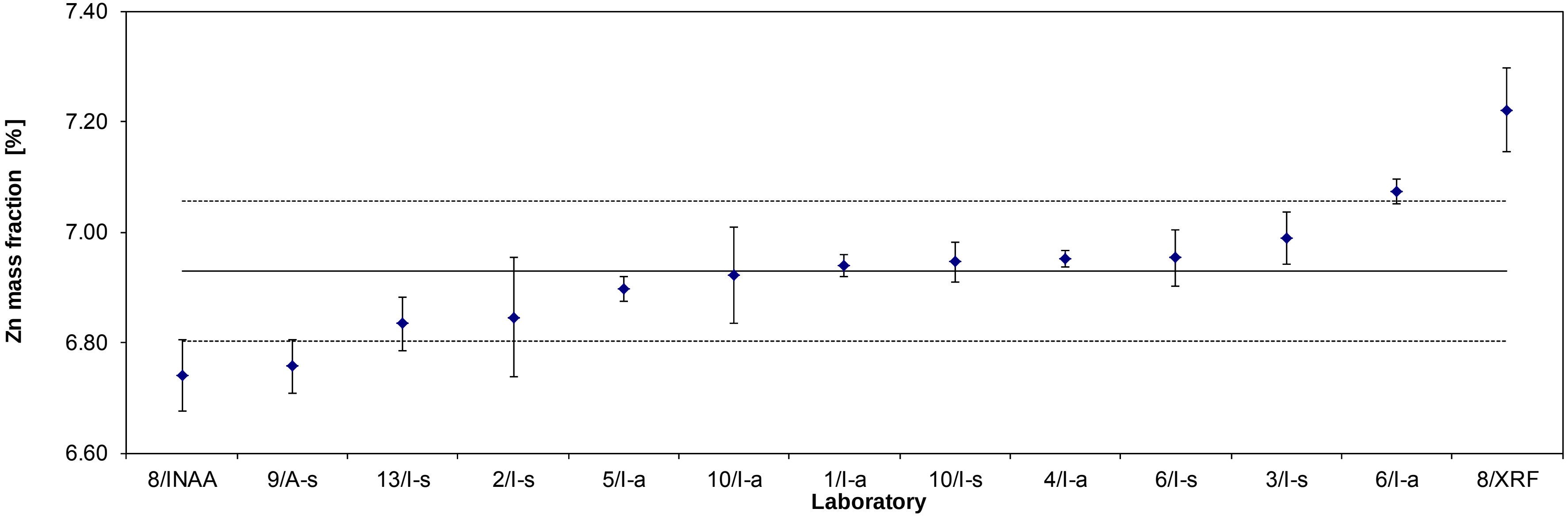

Table 31: Results for Zn

Lab./Meth.	6/I-a	6/I-s	2/I-s	2/P	13/I-s	11/I-s	3/I-s	10/I-s	5/I-a	10/I-a	8/XRF	4/I-a	1/I-a(R)	9/I(R)		N
M_i [%]	0.0890	0.0903	0.0916	0.091	0.094	0.0942	0.0939	0.0947	0.0963	0.0969	0.0971	0.0984	0.1008	0.1012		
	0.0890	0.0906	0.0912	0.090	0.093	0.0951	0.0946	0.0959	0.0960	0.0977	0.0985	0.0985	0.1005	0.1029		14
	0.0890	0.0908	0.0900	0.095	0.092	0.0909	0.0940	0.0960	0.0951	0.0959	0.0938	0.0989	0.1012	0.1031		
			0.0916	0.095	0.094	0.0921	0.0949	0.0954	0.0947	0.0957	0.0970	0.0984	0.1024			
			0.0903	0.095	0.095	0.0942	0.0945	0.0949	0.0952	0.0953		0.0982	0.1031			
			0.0905	0.092	0.095	0.0983	0.0955	0.0946	0.0966	0.0961		0.0987	0.1029			
***M* [%]**	**0.0890**	**0.0906**	**0.0909**	**0.0930**	**0.0938**	**0.0941**	**0.0946**	**0.0953**	**0.0957**	**0.0963**	**0.0966**	**0.0985**	**0.1018**	**0.1024**		**0.0952**
s_M [%]	0.0000	0.0003	0.0007	0.0022	0.0012	0.0026	0.0006	0.0006	0.0008	0.0009	0.0020	0.0003	0.0011	0.0010		0.0039
$\bar{s}_i$ [%]																0.0013
s_{rel}	0.00000	0.00278	0.00761	0.02405	0.01246	0.02725	0.00625	0.00639	0.00812	0.00914	0.02076	0.00263	0.01103	0.01020		0.04090

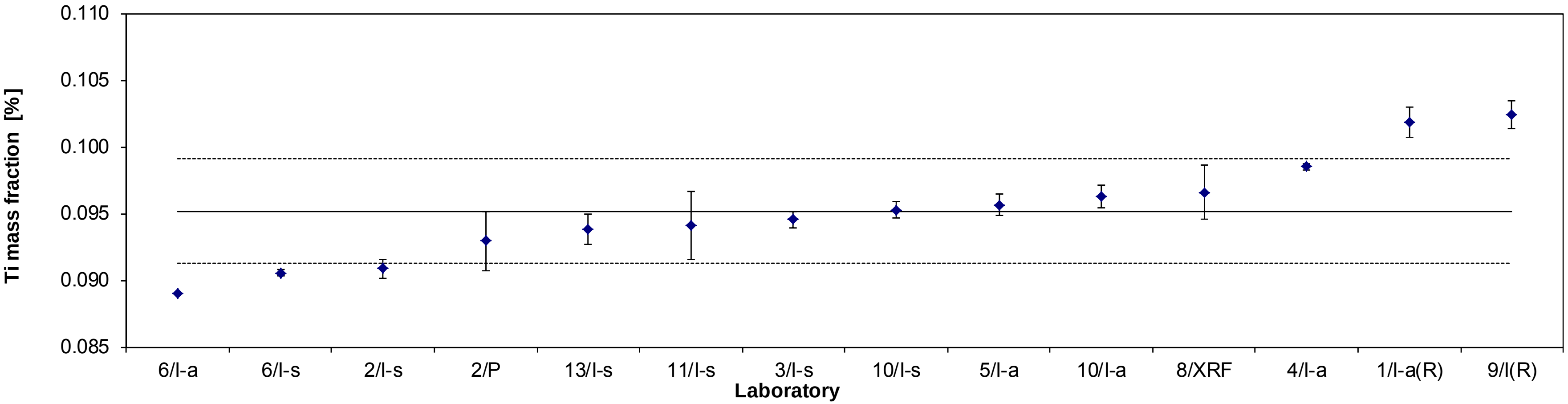

Table 32: Results for Ti

Lab./Meth.	9/I	3/I-s(R)	2/I-s	5/I-a	1/I-a	4/I-s	6/I-s	6/I-a	10/I-s	13/I-s	8/XRF		N
M_i [%]	0.1185	0.1189	0.1256	0.1277	0.1299	0.1314	0.1312	0.1320	0.1339	0.1360	0.143		
	0.1194	0.1276	0.1256	0.1286	0.1301	0.1311	0.1311	0.1330	0.1341	0.1360	0.142		11
	0.1184	0.1210	0.1259	0.1284	0.1303	0.1307	0.1311	0.1310	0.1345	0.1360	0.145		
	0.1179	0.1252	0.1279	0.1275	0.1303	0.1303			0.1338	0.1350	0.145		
	0.1180	0.1272	0.1258	0.1286	0.1305	0.1303			0.1346	0.1360			
	0.1184	0.1288	0.1244	0.1276	0.1304	0.1301			0.1341	0.1370			
***M* [%]**	**0.1184**	**0.1248**	**0.1259**	**0.1281**	**0.1303**	**0.1306**	**0.1311**	**0.1320**	**0.1342**	**0.1360**	**0.1438**		**0.1305**
s_M [%]	0.0005	0.0040	0.0011	0.0005	0.0002	0.0005	0.0001	0.0010	0.0003	0.0006	0.0014		0.0065
$\bar{s}_i$ [%]													0.0014
s_{rel}	0.00449	0.03192	0.00901	0.00410	0.00166	0.00385	0.00044	0.00758	0.00239	0.00465	0.00985		0.05013

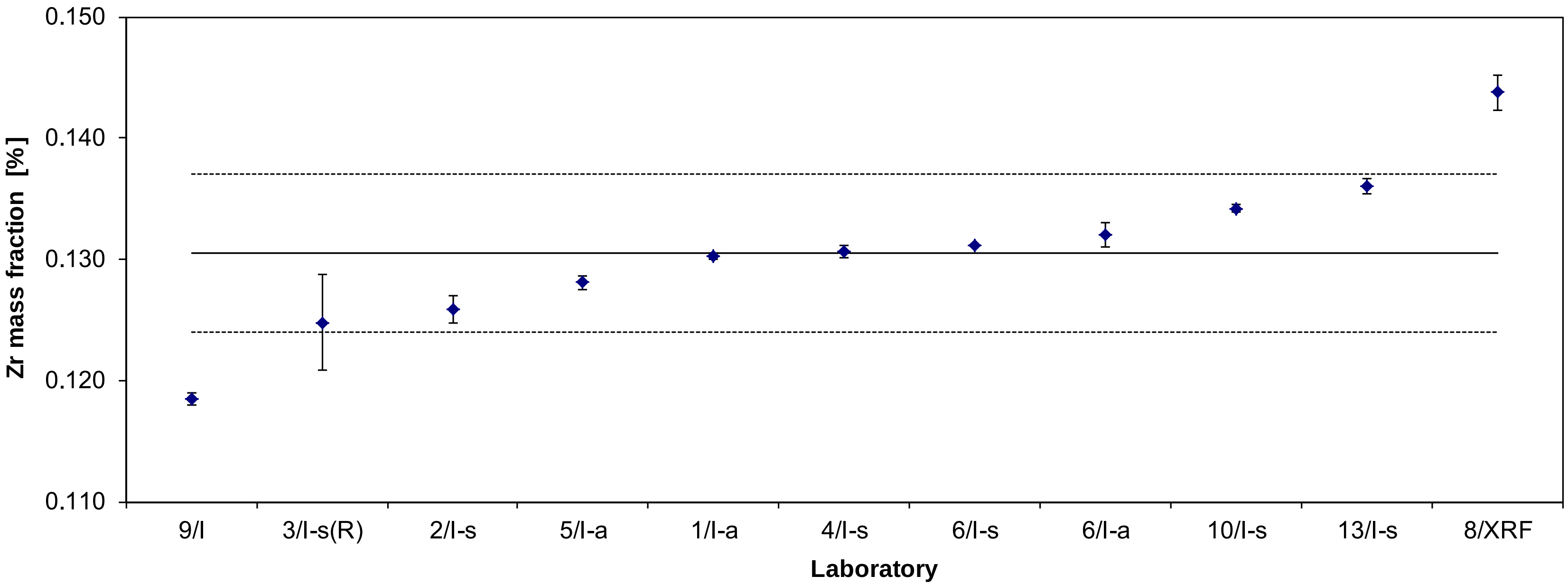

Table 33: Results for Zr

Lab./Meth.	8/INAA	10/I-s	4/IMS-s	10/IMS	6/I-a	11/I	3/-s	6/I-s	2/I-s	5/I-a(R)	1/I-a		N
M_i [mg/kg]	63.8	67.4	66.7	68.4	67.0	68.0	72.2	73.0	79.1	84.3	85.6		
	64.6	67.2	67.0	66.9	68.0	68.0	72.1	75.0	79.2	83.7	88.8		11
	64.1	66.2	67.1	68.0	68.0	68.0	72.3	74.0	78.9	84.4	89.0		
	65.5	65.6	66.5	65.4		69.0	72.7		79.3	85.3	91.0		
	63.4	66.6	66.5	67.2		69.0	72.8		78.2	83.6	85.4		
	63.8	66.3	66.4	68.5		68.0	72.6		78.2	85.6	83.6		
M [mg/kg]	**64.20**	**66.57**	**66.70**	**67.40**	**67.67**	**68.33**	**72.46**	**74.00**	**78.82**	**84.48**	**87.23**		**72.53**
s_M [mg/kg]	0.751	0.660	0.288	1.159	0.577	0.516	0.271	1.000	0.496	0.818	2.793		7.792
$\bar{s}_i$ [mg/kg]													1.0792
s_{rel}	0.012	0.010	0.004	0.017	0.009	0.008	0.004	0.014	0.006	0.010	0.032		0.107

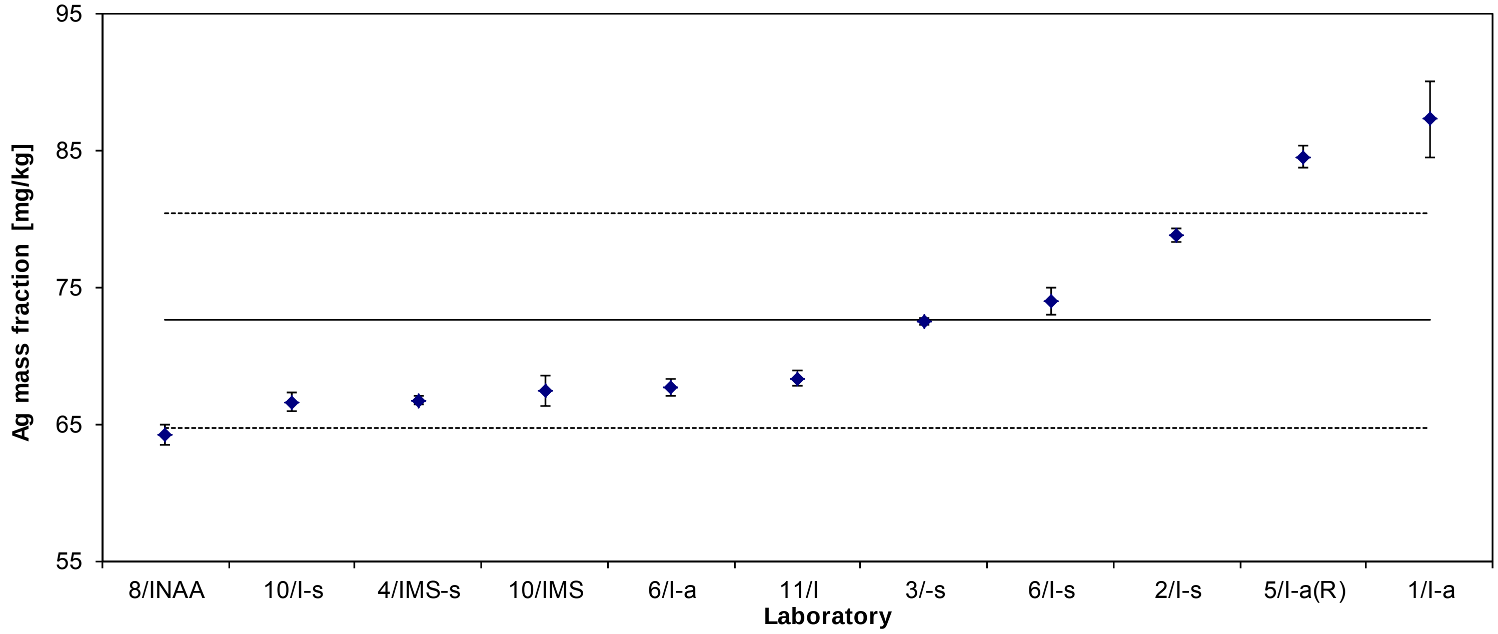

Table 34: Results for Ag

Lab./Meth.	10/I-s	10/I-a	10/IMS	11/I	4/I-s	6/I-a	1/I-a	3/I-s	13/I-s	5/I-a		N
M_i [mg/kg]	9.3	9.4	9.7	9.7	9.8	10.00	10.3	10.4	11.0	11.0		
	9.3	10.0	9.7	9.8	9.9	10.05	10.4	10.5	11.0	11.3		10
	9.4	9.4	9.6	9.7	9.8	10.10	10.3	10.5	11.0	11.0		
	9.3	9.6	9.5	9.7	9.8		10.4	10.6	11.0	11.1		
	9.5	9.3	9.3	9.1	9.7		10.4	10.6	11.0	11.1		
	9.4	9.4	9.5	9.4	9.7		10.4	10.6	11.0	11.1		
M [mg/kg]	**9.37**	**9.52**	**9.56**	**9.57**	**9.79**	**10.05**	**10.36**	**10.54**	**11.00**	**11.10**		**10.08**
s_M [mg/kg]	0.064	0.256	0.144	0.266	0.054	0.050	0.063	0.072	0.000	0.110		0.635
$\bar{s}_i$ [mg/kg]												0.1369
s_{rel}	0.007	0.027	0.015	0.028	0.005	0.005	0.006	0.007	0.000	0.010		0.063

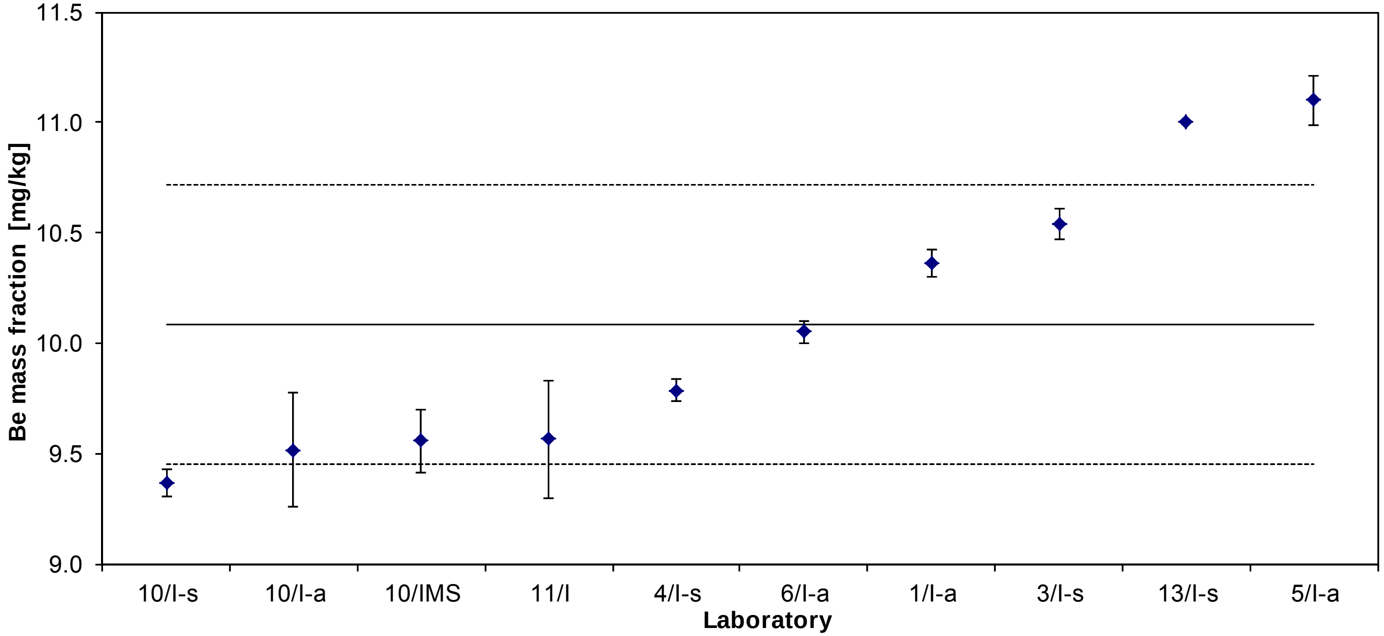

Table 35: Results for Be

Lab./Meth.	10/IMS	13/I-s	12/I-s	4/IMS-s	6/I-a	5/I-a	11/I	1/I-s		N
M_i [mg/kg]	38.1	39.0	41.7	40.3	41.8	40.5	41	42.4		
	37.9	39.0	38.0	40.6	40.8	41.8	41	48.3		8
	38.0	39.0	38.3	40.4	41.6	38.4	46	43.5		
	37.8	38.0		40.2		43.0	44	47.4		
	37.3	39.0		39.7		44.9	42	36.5		
	37.6	39.0		39.7		40.3	43	40.3		
***M* [mg/kg]**	**37.79**	**38.83**	**39.33**	**40.14**	**41.40**	**41.48**	**42.83**	**43.07**		**40.61**
s_M [mg/kg]	0.281	0.408	2.055	0.351	0.529	2.278	1.941	4.418		1.903
$\bar{s}_i$ [mg/kg]										2.0415
s_{rel}	0.007	0.011	0.052	0.009	0.013	0.055	0.045	0.103		0.047

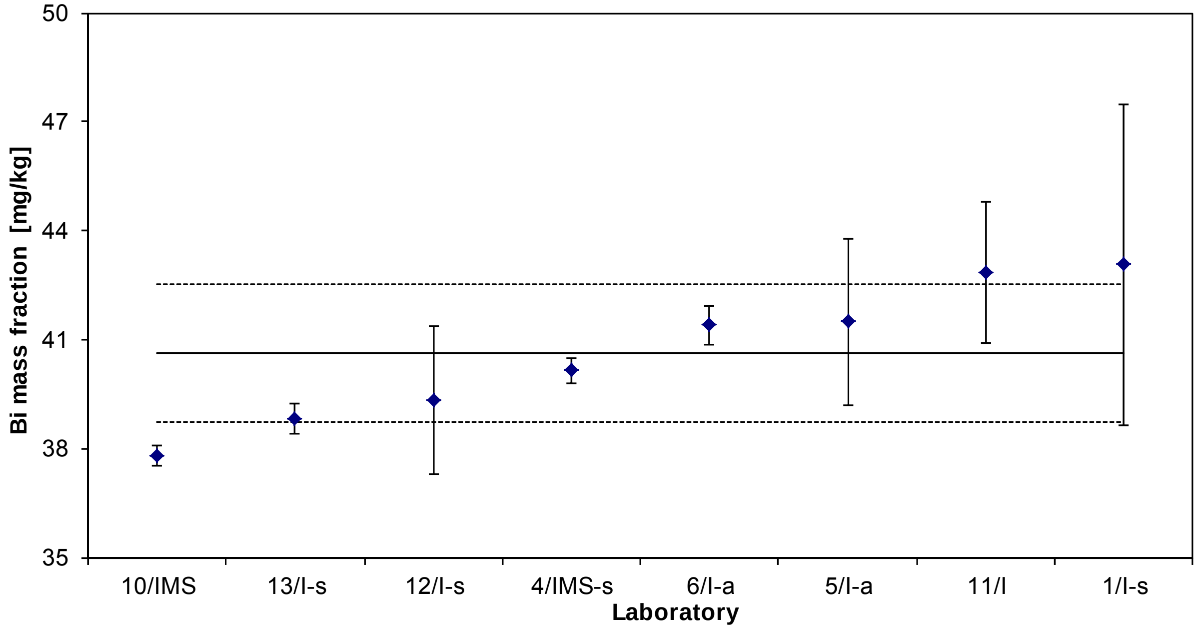

Table 36: Results for Bi

Lab./Meth.	8/INAA	1/I-a	4/I-s	6/I-a	6/I-s	13/I-s	3/I-s	10/IMS	11/I	8/XRF	10/I-s	5/I-a(R)	2/I-s		N
M_i [mg/kg]	155.6	161.3	168.0	170.0	171.0	180.0	186.9	190.0	183.0	188.0	191.5	210.1	209.8		
	159.2	161.7	168.4	172.0	171.0	180.0	184.9	188.7	185.0	187.0	193.4	207.6	210.5		13
	160.6	162.5	168.2	172.0	172.0	180.0	184.6	188.7	184.0	191.0	192.3	210.4	209.1		
	160.5	162.7	168.3			180.0	185.9	185.3	187.0	195.0	191.5	210.2	210.4		
	158.1	164.3	169.3			180.0	184.7	182.7	188.0		193.0	208.2	208.5		
	158.9	163.8	169.5			180.0	187.3	185.6	196.0		192.9	208.7	208.9		
***M* [mg/kg]**	**158.82**	**162.72**	**168.62**	**171.33**	**171.33**	**180.00**	**185.71**	**186.83**	**187.17**	**190.25**	**192.43**	**209.20**	**209.53**		**182.61**
s_M [mg/kg]	1.845	1.163	0.626	1.155	0.577	0.000	1.177	2.755	4.708	3.594	0.804	1.188	0.826		15.952
$\bar{s}_i$ [mg/kg]															2.0309
s_{rel}	0.012	0.007	0.004	0.007	0.003	0.000	0.006	0.015	0.025	0.019	0.004	0.006	0.004		0.087

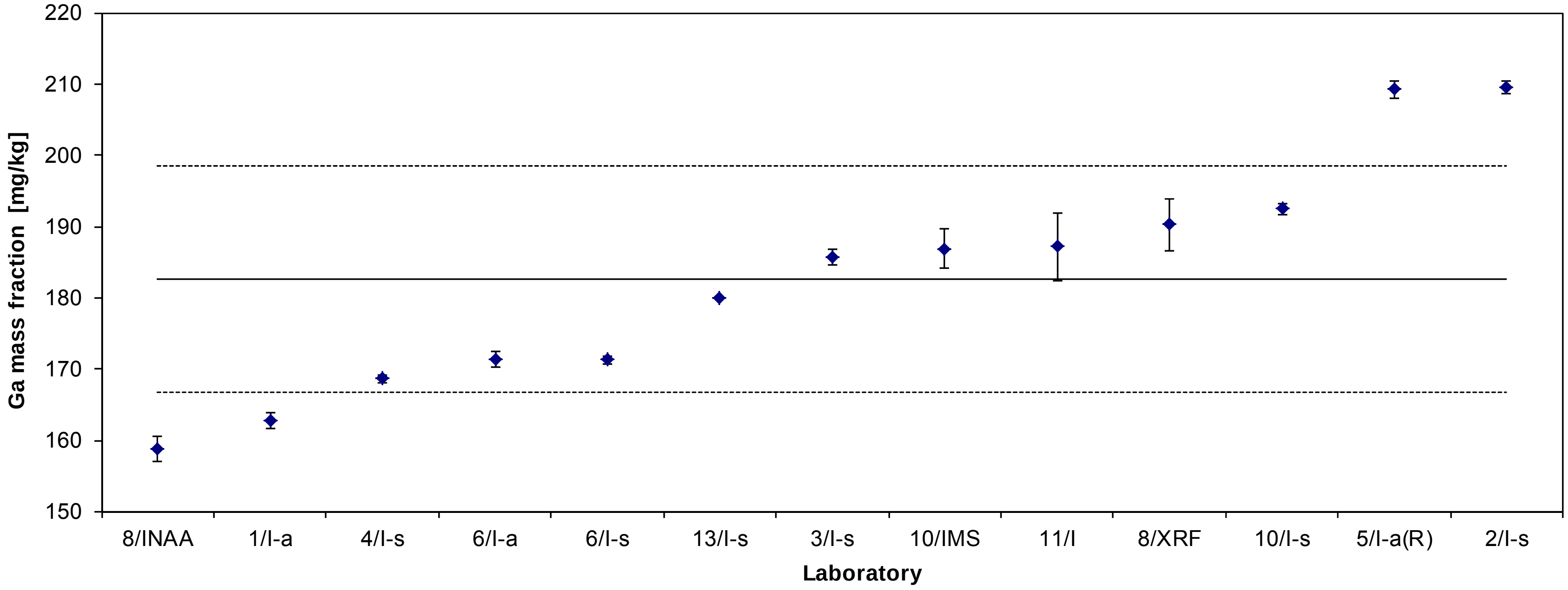

Table 37: Results for Ga

Lab./Meth.	8/INAA-1	8/INAA-1	11/I	10/IMS	3/I-s	6/I-a	12/I-s	5/I-a	4/I-s(R)	13/I-s	9/I-s	8/XRF		N
M_i [mg/kg]	151.4	149.1	158.0	159.3	158.47	159.0	161.2	159.7	163.3	170.0	160.0	[195]		
	148.1	152.6	158.0	159.8	162.60	162.0	159.8	162.8	162.4	170.0	174.0	179		12
	156.4	154.0	152.0	159.9	160.70	159.0	159.1	161.0	163.0	170.0	175.0	174		
	149.1	153.5	156.0	158.9	159.29			159.0	161.0	170.0	170.0	176		
	156.4	153.5	155.0	155.5	158.95			162.0		170.0	177.0			
	151.4	153.4	161.0	158.1	158.23			157.8		170.0	176.0			
M [mg/kg]	**152.13**	**152.68**	**156.67**	**158.58**	**159.71**	**160.00**	**160.05**	**160.38**	**162.43**	**170.00**	**172.00**	**176.33**		**161.75**
s_M [mg/kg]	3.548	1.813	3.077	1.647	1.662	1.732	1.072	1.891	1.021	0.000	6.356	2.517		7.440
$\bar{s}_i$ [mg/kg]														2.6821
s_{rel}	0.023	0.012	0.020	0.010	0.010	0.011	0.007	0.012	0.006	0.000	0.037	0.014		0.046

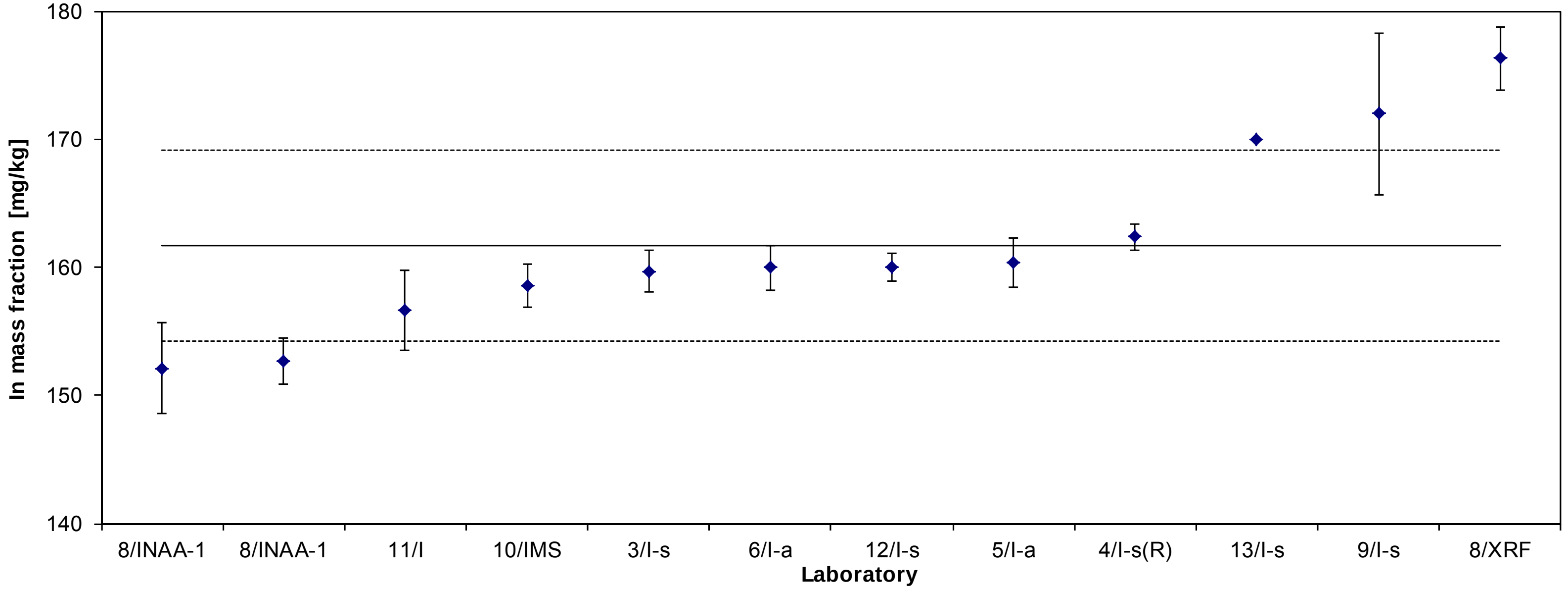

Table 38: Results for In

Lab./Meth.	3/I-s	13/I-s	6/I-a	10/IMS	6/I-s	11/I-s	4/IMS-s	1/I-a	2/I-s(R)	5/I-a		N
M_i [mg/kg]	46.4	45.0	45.0	46.1	47.0	44.0	48.7	50.2	47.9	56.9		
	46.3	45.0	45.0	46.5	46.0	46.0	48.6	50.6	51.9	52.4		10
	44.1	45.0	47.0	45.9	46.0	45.0	48.3	47.8	53.2	53.7		
	44.4	45.0		46.0		52.0	48.6	51.0	51.1	55.2		
	46.7	46.0		45.4		46.0	48.3	50.7	49.7	50.1		
	45.8	47.0		45.6		47.0	48.3	49.3		54.6		
M [mg/kg]	**45.62**	**45.50**	**45.67**	**45.92**	**46.33**	**46.67**	**48.47**	**49.93**	**50.76**	**53.82**		**47.87**
s_M [mg/kg]	1.106	0.837	1.155	0.382	0.577	2.805	0.182	1.199	2.042	2.361		2.818
$\bar{s}_i$ [mg/kg]												1.5105
s_{rel}	0.024	0.018	0.025	0.008	0.012	0.060	0.004	0.024	0.040	0.044		0.059

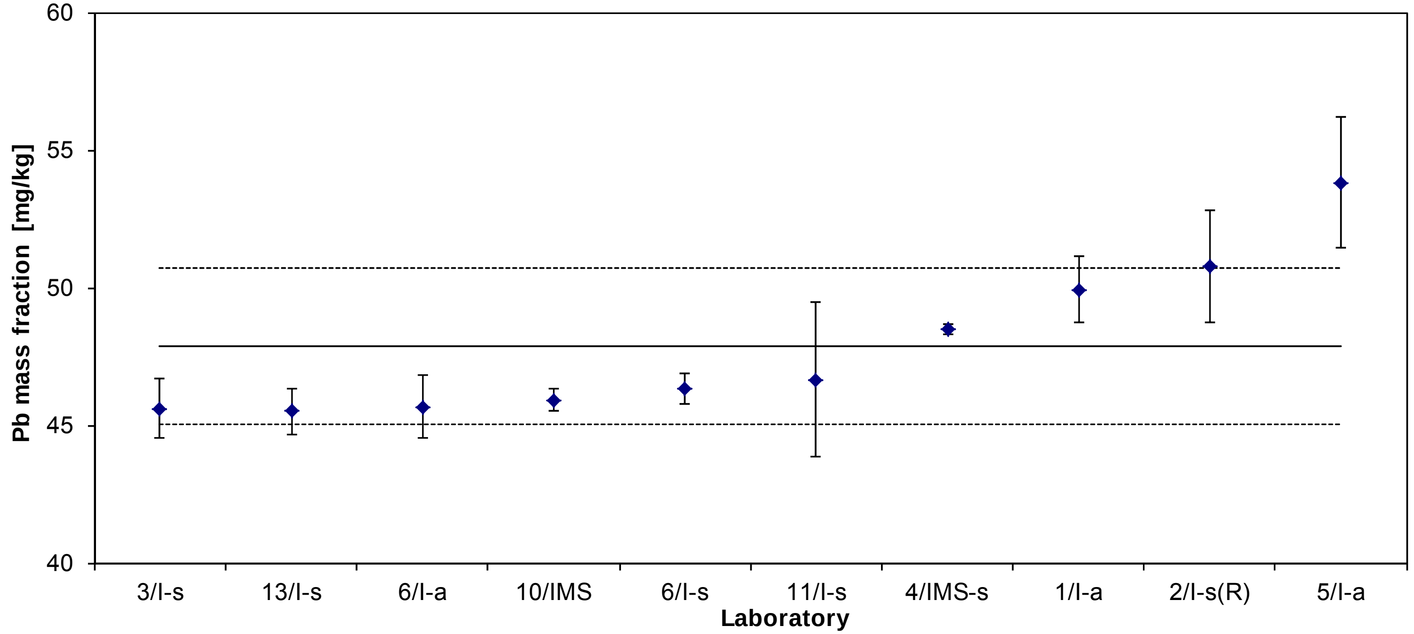

Table 39: Results for Pb

Lab./Meth.	13/I-s	1/I-a	4/IMS-s	6/I-a	10/IMS	2/I-s	10/I-s	5/I-a(R)	10/I-a	3/I-s(R)	8/XRF		N
M_i [mg/kg]	220.0	229.5	234.2	232.0	238.1	235.7	239.9	240.4	241.0	235.9	270		
	230.0	231.4	231.4	234.0	236.0	234.4	242.6	240.6	243.0	257.7	266		10
	220.0	230.5	232.2	234.0	233.2	238.4	240.7	243.1	244.0	243.1	269		
	220.0	224.3	233.5		230.9	238.0	238.0	244.4	249.0	250.4	272		
	230.0	227.8	232.6		238.0	235.4	242.5	240.0	243.0	257.5			
	230.0	226.9	234.2		230.5	235.7	242.2	241.4	241.0	259.4			
***M* [mg/kg]**	**225.00**	**228.40**	**233.00**	**233.33**	**234.45**	**236.27**	**240.98**	**241.65**	**243.50**	**250.67**	**269.25**		**236.73**
s_M [mg/kg]	5.477	2.609	1.134	1.155	3.409	1.577	1.819	1.739	2.950	9.461	2.500		7.604
$\bar{s}_i$ [mg/kg]													3.8651
s_{rel}	0.024	0.011	0.005	0.005	0.015	0.007	0.008	0.007	0.012	0.038	0.009		0.032

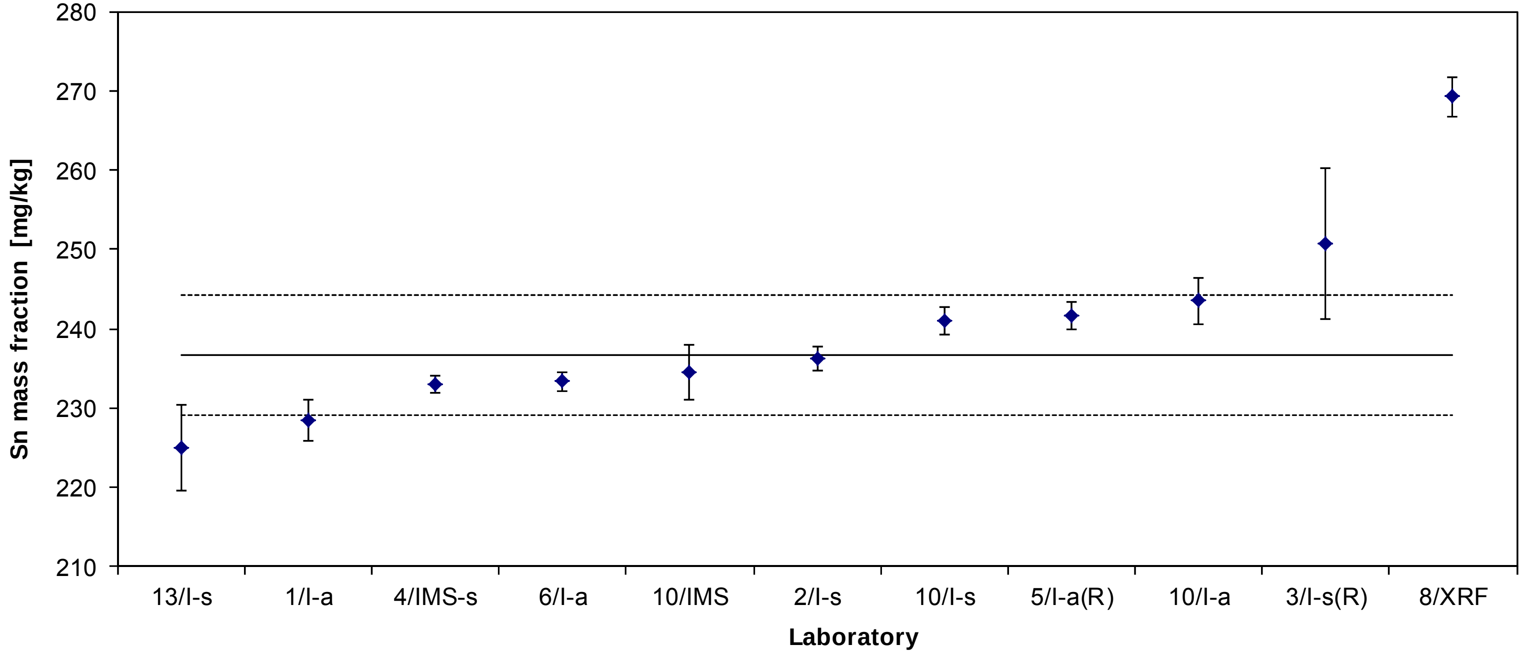

Table 40: Results for Sn

Lab./Meth.	9/I-s	10/IMS	10/I-s	4/I-s	6/I-a	2/I-s	10/I-a	1/I-a	3/I-s(R)	5/I-a(R)	13/I-s		N
M_i [mg/kg]	99.3	101.7	103.7	103.6	104.0	104.9	105.0	105.1	103.03	109.0	110.00		
	101.3	101.1	103.8	103.2	105.0	104.6	104.0	104.7	108.09	108.5	110.00		11
	102.2	101.2	103.5	104.5	105.0	104.7	104.0	103.3	104.04	110.4	110.00		
	100.1	100.9	102.5	104.4		105.4	106.0	106.3	107.38	110.4	110.00		
	101.6	100.0	103.6	104.2		104.3	106.0	105.0	106.98	108.6	110.00		
	100.9	100.6	104.1	104.0		104.6		105.7	107.65	109.6	110.00		
M [mg/kg]	**100.90**	**100.92**	**103.53**	**103.97**	**104.67**	**104.75**	**105.00**	**105.02**	**106.20**	**109.42**	**110.00**		**104.94**
s_M [mg/kg]	1.053	0.578	0.547	0.494	0.577	0.373	1.000	1.017	2.116	0.854	0.000		2.877
$\bar{s}_i$ [mg/kg]													0.9390
s_{rel}	0.010	0.006	0.005	0.005	0.006	0.004	0.010	0.010	0.020	0.008	0.000		0.027

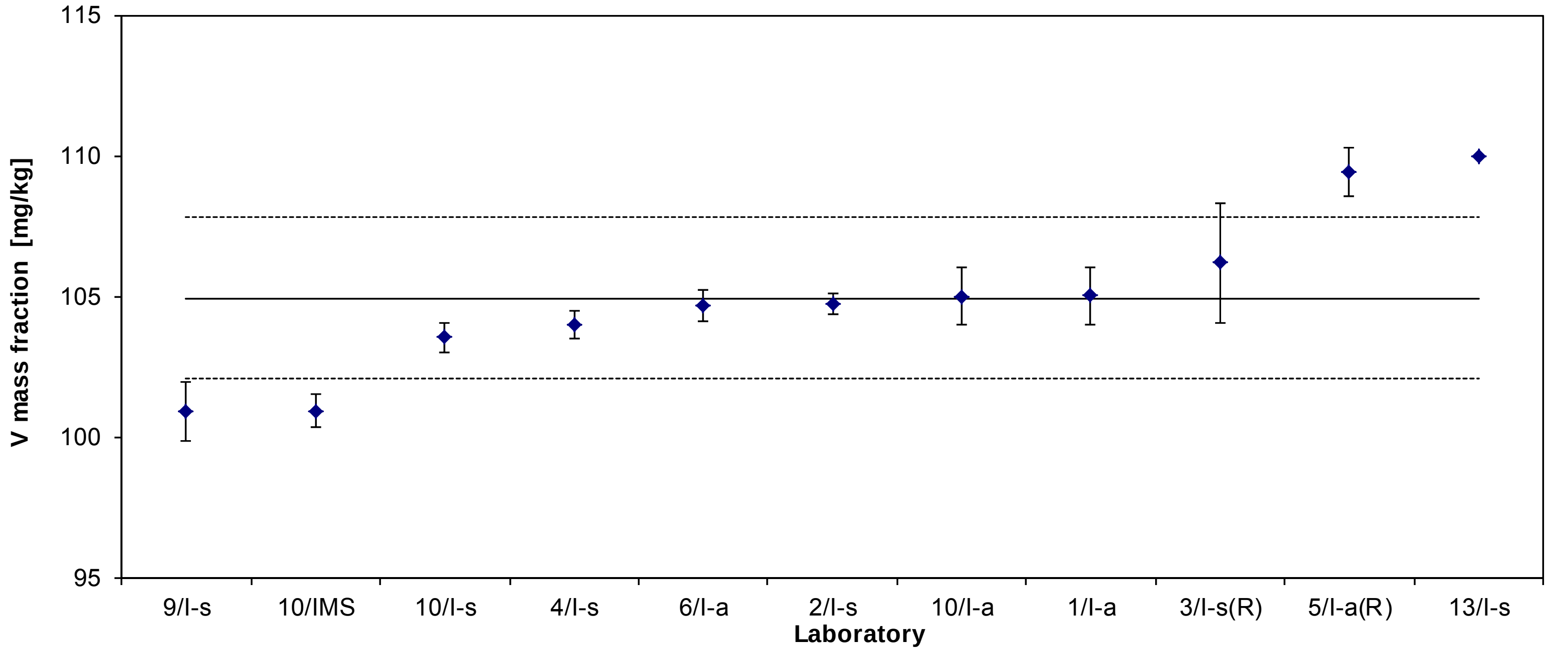

Table 41: Results for V

Lab./Meth.	6/I-a	11/I-s	10/I-s	6/I-s	4/IMS-s	13/I-s	1/I-a		N
M_i [mg/kg]	28.0	31.0	33.8	36.7	41.5	43.0	47		
	27.0	29.0	34.2	34.0	39.5	44.0	52		7
	33.0	31.0	33.9	36.0	39.8	43.0	48		
		30.0	34.1		40.2	44.0	46		
		30.0	33.7		40.4	43.0	45		
		34.0	34.1		40.6	43.0	46		
M [mg/kg]	**29.33**	**30.83**	**33.97**	**35.57**	**40.34**	**43.33**	**47.33**		**37.24**
s_M [mg/kg]	3.215	1.722	0.197	1.401	0.703	0.516	2.503		6.654
$\bar{s}_i$ [mg/kg]									1.786
s_{rel}	0.110	0.056	0.006	0.039	0.017	0.012	0.053		0.179

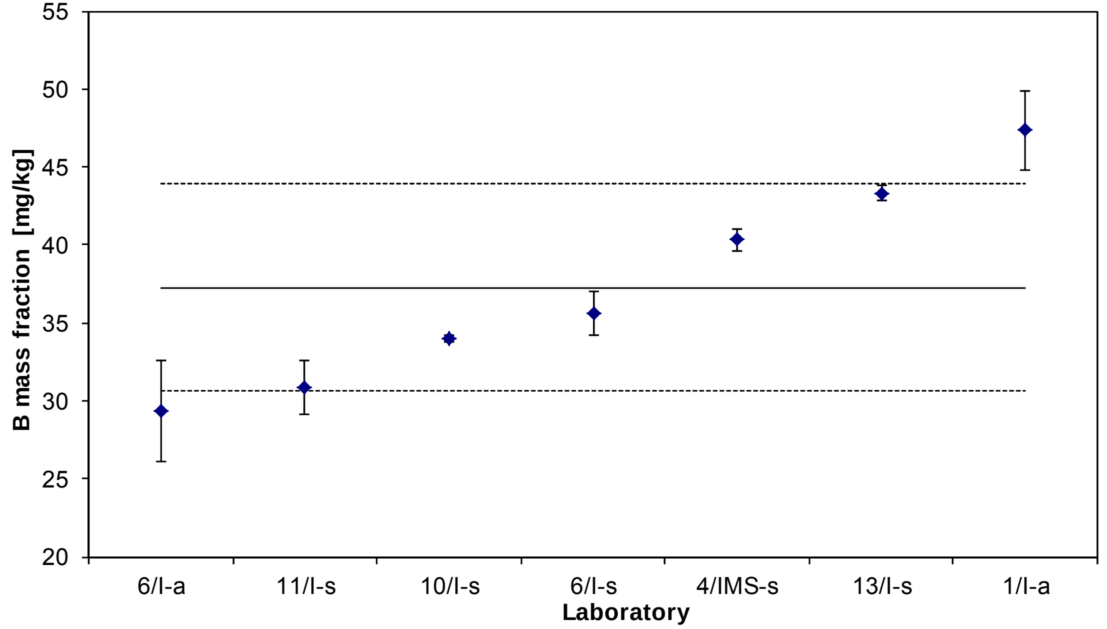

Table 42: Results for B

Lab./Meth.	6/l-s	10/l-s	4/l-s	11/l-s		N
M_i [mg/kg]	2.4	5.8	5.8	8.1		
	5.1	5.9	5.9	7.7		4
	5.2	5.8	5.8	9.0		
	5.3	5.8	6.1	5.9		
	5.4	5.7	6.2	6.1		
		5.9	6.1	9.5		
***M* [mg/kg]**	**4.68**	**5.83**	**5.97**	**7.72**		**6.049**
s_M [mg/kg]	1.279	0.057	0.157	1.476		1.253
$\bar{s}_i$ [mg/kg]						0.9801
s_{rel}	0.273	0.010	0.026	0.191		0.207

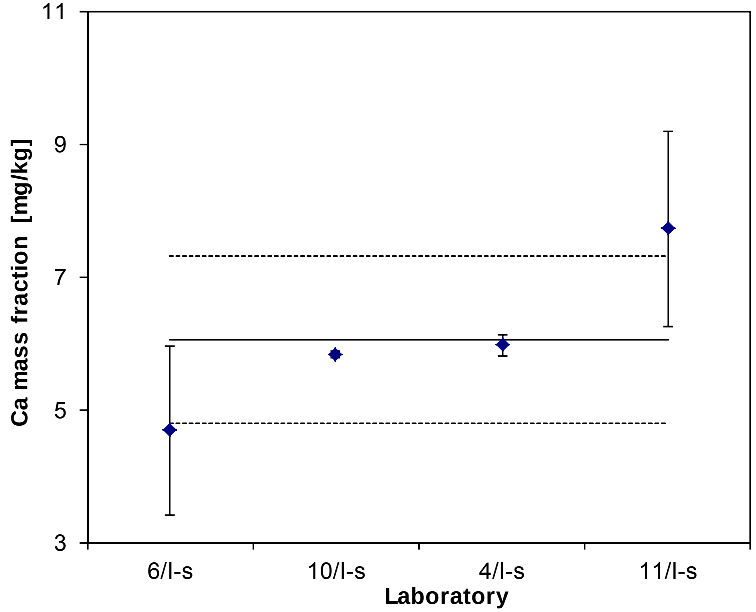

Table 43: Results for Ca

Lab./Meth.	4/l-s	13/l-s	2/l	6/l-s	11/l-s		N
M_i [mg/kg]	18.3	17	29.1	30.4	35		
	15.7	20	28.2	33.7	33		5
	15.9	22	29.4	33.4	34		
	19.6	17	30.3	31.0	33		
	20.6	16	30.3	33.0	33		
	20.3	22	35.4	30.0	34		
***M* [mg/kg]**	**18.40**	**19.00**	**30.46**	**31.92**	**33.67**		**26.69**
s_M [mg/kg]	2.165	2.683	2.550	1.635	0.816		7.383
$\bar{s}_i$ [mg/kg]							2.0847
s_{rel}	0.118	0.141	0.084	0.051	0.024		0.277

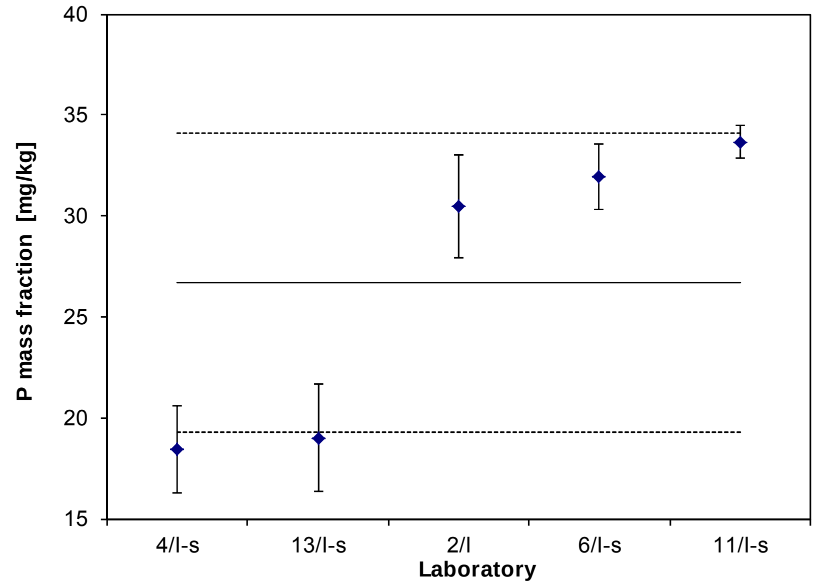

Table 44: Results for P

6. Instructions for use and stability

The certified reference material ERM®-EB317 is intended for the calibration and quality control of spark emission and X-ray fluorescence spectrometer used for the analysis of similar materials.
Before analysis the surface of the material should be cleaned by turning or milling.
If chips prepared from the compact material are used for wet chemical analysis, a minimum sample intake of 0.5 g has to be used.
The material will remain stable provided that it is not subjected to excessive heat (e.g. during preparation of the working surface).

7. Literature

[1] ISO Guide 31, Contents of certificates of reference materials, 1981

[2] ISO Guide 34, General requirements for the competence of reference material producers, 2000

[3] ISO Guide 35, Reference materials - General and statistical principles for certification. Third edition, 2006

[4] Guidelines for the production of BAM Reference Materials, 2006

[5] Technical Guidelines for the Production and Acceptance of a European Reference Material (www.erm-crm.org)

[6] ASTM Designation E 826-90, Standard Practice for Testing Homogeneity of Materials for the Development of Reference Materials

[7] Bonas G, Zervou M, Papaeoannou T, Lees M: Accred Qual Assur (2003) 8:101-107

8. Information on and purchase of the CRM

Certified reference material ERM®-EB317 is supplied by

BAM Bundesanstalt für Materialforschung und -prüfung
Fachbereich 1.6: Anorganische Referenzmaterialien
Richard-Willstätter-Straße 11, 12489 Berlin
Phone +49 (0)30 - 8104 2061 or 1119
Fax: +49 (0)30 - 8104 1117
E-Mail: sales.crm@bam.de

Each disc of ERM®-EB317 will be distributed together with a detailed certificate containing the certified values and their uncertainties, the mean values and standard deviations of all accepted data sets and information on the analytical methods used and the names of the participating laboratories.
Information on certified reference materials can be obtained from BAM, http://www.bam.de/en/fachthemen/referenzmaterialien/index.htm
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