

Certification Report

Certified Reference Material

BAM-M318

AlSi1,2Mg0,4

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Summary

This report describes preparation, analysis and certification of the aluminium alloy reference material BAM-M318.

The certified reference material (CRM) is available in the form of discs (65 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. It is also suitable for wet chemical analysis.

The following mass fractions and uncertainties have been certified:

Element	Mass fraction ¹⁾ in %	Uncertainty ²⁾ in %
Si	1.211	0.017
Fe	0.246	0.008
Cu	0.0908	0.0025
Mn	0.0985	0.0017
Mg	0.356	0.009
Cr	0.0208	0.0004
Zn	0.0486	0.0011
Ti	0.0238	0.0010
Ga	0.0189	0.0005
V	0.0104	0.0003
	in mg/kg	in mg/kg
Ni	50.0	1.9
Be	4.7	0.3
Ca	9.1	1.6
Cd	9.6	1.2
Hg	7.6	1.1
Li	6.0	0.7
Pb	56	3
Sn	20.6	1.1
Zr	32.9	1.4

- 1 Unweighted mean value of the means of accepted sets of data, each set being obtained by at least 5 laboratories and/or with different methods of measurement. The values are traceable to the SI (Système International d'Unités) by the use of pure substances of known stoichiometry or certified standard solutions for calibration.
- 2 Estimated expanded uncertainty U with a coverage factor of $k = 2$, corresponding to a level of confidence of about 95%, as defined in the ISO/IEC Guide 98-3:2008 [Uncertainty of measurement -- Part 3: Guide to the Expression of Uncertainty in Measurement (GUM:1995)].

This report contains detailed information on the preparation of the CRM as well as on homogeneity investigations and on the analytical methods used for certification analysis.

The certified values are based on the results of twelve laboratories which participated in the certification inter-laboratory comparison. In addition to the elements mentioned above the mass fractions of B and Na are given for information.

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List of abbreviations

(if not explained elsewhere)

CRM	certified reference material
FAAS	flame atomic absorption spectrometry
ETAAS	Electrothermal 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
s	standard deviation of an individual data set
s_M	standard deviation of laboratory means
s_{rel}	relative standard deviation
\bar{s}_i	square root of mean of variances of data sets under repeatability conditions
M_i	single result
I	ICP-OES (Tables 2 – 21)
I(R)	ICP-OES, revised value (Tables 2 – 21)
IMS	ICP-MS (Tables 2 – 21)
A	FAAS (Tables 2 – 21)
AF	Atomic fluorescence (Tables 2 – 21)
CVAAS	Cold vapor atomic absorption spectrometry (Tables 2 – 21)
EA	ETAAS (Tables 2 – 21)
P	spectrophotometry (Tables 2 – 21)
XRF	X-ray fluorescence spectrometry (Tables 2 – 21)
XRF(R)	X-ray fluorescence spectrometry, revised value (Tables 2 – 21)
-s	dissolution in acid (Tables 2 – 21)
-a	dissolution in base (Tables 2 – 21)

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 BAM-M318 is based on the aluminium alloy AlSi1,2Mg0,4.

The CRM was produced in close cooperation with the working group „Aluminium“ of the Committee of Chemists of the Society of Metallurgists und Miners (GDMB). Since all the laboratories participating in this certification project are highly experienced with aluminium analysis and had already participated in earlier inter-laboratory comparisons, there was no preceding round robin for qualification necessary.

Certification was carried out on the basis of ISO 17034 [1] and the relevant ISO-Guides [2, 3].

2. Companies/laboratories involved

Manufacturing of the material:

- Constellium, Centre de Recherches de Voreppe, Voreppe, France

Test for homogeneity:

- Bundesanstalt für Materialforschung und -prüfung (BAM), Berlin, Germany
- Constellium, Centre de Recherches de Voreppe, Voreppe, France

Participants in the certification inter-laboratory comparison:

ALERIS Rolled Products Germany GmbH, Koblenz, Germany
AMAG Austria Metall AG, Ranshofen, Austria
Bundesanstalt für Materialforschung und -prüfung (BAM), Berlin, Germany
Constellium, Centre de Recherches de Voreppe, Voreppe, France
Hydro Aluminium Rolled Products GmbH, R&D, Bonn, Germany
Hydro Aluminium Rolled Products GmbH, Hamburg, Germany
Institute of Non-Ferrous Metals, Gliwice, Poland
Leichtmetall Aluminium Giesserei Hannover GmbH, Hannover, Germany
Otto Fuchs KG, Meinerzhagen, Germany
Revierlabor, Essen, Germany
Suisse Technology Partners AG, Neuhausen, Switzerland
TRIMET Aluminium SE, Essen, Germany

Statistical evaluation of the data:

- Bundesanstalt für Materialforschung und -prüfung (BAM), Berlin, Germany

3. Candidate material

The candidate material was produced by Constellium, Centre de Recherches de Voreppe, Voreppe, France. About 500 kg of an aluminium melt were doped with the desired elements. The melt was cast into six rods (A – F) with a length of 3775 mm each. 250 mm on both ends of each rod were discarded. The rods were cut into segments of 800 mm length (A1, A2, A3, A4, B1, B2, ..., F3, F4). Between the segments 15-mm discs (AA, AB, AC, AD, AE, BA, BB, ..., FD, FE) were taken for homogeneity testing (see Fig. 1).

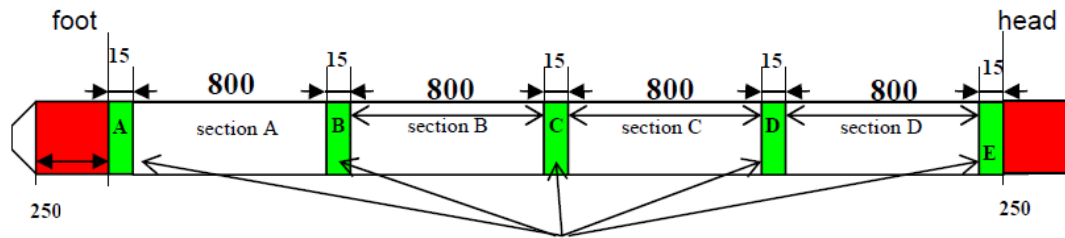


Fig.1: Preparation of the rods cast (all figures in mm)

In total, approx. 500 discs with a diameter of ca. 65 mm and 30 mm height were obtained.

4. Homogeneity testing

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 volatilize or because of possible segregation 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 Figure 2):

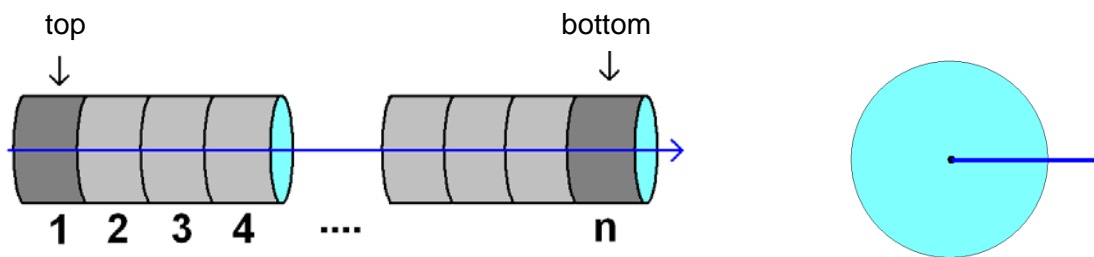


Fig. 2: Axial and radial composition gradient

Therefore, it is necessary to investigate the raw material for both axial and radial inhomogeneities. Radial homogeneity testing of the candidate material using spark emission spectrometry was performed at Constellium, Centre de Recherches de Voreppe on the discs taken from the rods as shown in Fig. 1. In total 30 discs were investigated, this corresponds to 6 % of the whole batch.

The estimate of analyte-specific inhomogeneity contribution u_{bb} to be included into the total uncertainty budget was calculated according to ISO Guide 35 [3] using Eq. (1) and Eq. (2):

$$s_{bb} = \sqrt{\frac{MS_{among} - MS_{within}}{n}} \quad (1)$$

$$u_{bb}^* = \sqrt{\frac{MS_{within}}{n}} \sqrt[4]{\frac{2}{N(n-1)}} \quad (2)$$

where:

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

s_{bb} signifies the between-discs standard deviation whereas u_{bb}^* denotes the maximum heterogeneity that can potentially be hidden by an insufficient repeatability of the applied measurement method (which has to be considered as the minimum uncertainty contribution). In any case the larger of the two values was used as $u_{bb}(1)$. Eq. (1) does not apply if MS_{within} is larger than MS_{among} .

In addition to the tests performed over the length of the rods three discs were tested for homogeneity over the area (possible segregation from the outer part to the centre). To perform this test SOES analysis was carried out in circles (outer circle: 16 sparks, inner circle: 8 sparks; centre: 5 sparks, Sn: outer circle: 8 sparks, inner circle: 8 sparks; centre: 4 sparks).

The analyte-specific within-disc uncertainty component $u_{bb}(2)$ was calculated in the same way as for the total batch. To calculate the necessary data an unbalanced ANOVA was carried out taking into account that the number of single measurements was different for the centre, the inner and the outer circle.

Annex 1 and 2 show the results of the homogeneity calculations.

5. Characterisation study

5.1 Analytical methods

Twelve laboratories participated in the certification inter-laboratory comparison. All laboratories were highly experienced in the analysis of aluminium and aluminium alloys and participated successfully in former certification inter-laboratory comparisons. For some elements part of the laboratories used more than one analytical method reporting more than one data set.

The laboratories were asked to analyse six subsamples. They were free to choose any suitable analytical method. Table 1 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 1: Analytical procedures used by the participating laboratories

Lab-No.	Element	Sample mass	Sample pretreatment	Analytical method
1	Si, Fe, Cu, Mn, Mg, Cr, Zn, Ti, Ga, V, Ni, Pb, Zr	0.5 g	Dissolution with NaOH	ICP-OES, commercial mono-element solutions, matrix matching with pure Al
	B, Be, Cd, Hg, Li, Sn	0.5 g	Dissolution with HNO ₃ /HF	ICP-OES, commercial mono-element solutions
2	Si, Fe, Cu, Mn, Mg, Zn, Ti, Ga	0.5 g	Dissolution with NaOH	ICP-OES, calibration with pure metals or pure chemicals, matrix matching with pure Al (5N5)
	B, Cd, Hg, Pb, Sn	0.5 g	Dissolution with HNO ₃ /HF	ICP-MS, calibration with pure metals or chemicals, matrix matching with pure Al (5N5)
	Be	0.5 g	Dissolution with HNO ₃ /HF	ICP-OES, commercial mono-element solution (Merck certipur), matrix matching with pure Al (5N5)
	Cr, Ni, Ca, Li, Na, V, Zr	0.5 g	Dissolution with HNO ₃ /HF	ICP-OES, calibration with pure metals or pure chemicals, matrix matching with pure Al (5N5)
4	Si	0.5 g	Dissolution with NaOH	Spectrophotometry, commercial mono-element solution
	Fe, Cu, Mn, Mg, Cr, Zn, Ti, Ga, V	0.5 g	Dissolution with HCl	ICP-OES, commercial mono-element solutions
	Pb, Ni, Be, Cd, Hg, Li, Sn, Zr	1 g	Dissolution with HCl	ICP-OES, commercial mono-element solutions
5	Si, Fe, Cu, Mn, Mg, Cr, Ni, Zn, Ti, Pb, Sn, Ga, Be, Cd, Li, V, Zr	0.5 g	Dissolution with NaOH	ICP-OES, commercial mono-element solutions (NIST)
6	Si, Fe, Cu, Mn, Mg, Cr, Zn, Ti, Ga, V, Ni, B, Be, Cd, Li, Pb, Sn, Zr	0.5 g	Dissolution with NaOH	ICP-OES, calibration with pure metals or pure chemicals
	B, Cs, Hg, Na	0.5 g	Dissolution with HNO ₃ /HF	ICP-OES, calibration with pure metals or pure chemicals
7	Si, Fe, Cu, Mn, Mg, Cr, Zn, Ni, Ti, Ga, Pb, Sn, V, Zr	0.5 g	Dissolution with HNO ₃ /HF	ICP-OES, calibration with matrix matched standards, commercial multi-element standard solutions
	Mn, Cr, Ti, Ni, Be, Cd, Li, Pb, Sn	1 g	Dissolution with HNO ₃ /HF	ICP-MS, with matrix matched standards, commercial mono-element standard solutions (Perkin Elmer)
	Si	1 g	Dissolution with NaOH/HClO ₄	Gravimetry
8	Si, Fe, Cu, Mn, Mg, Cr, Zn, Ni, Ti, Ga, Sn, V, Zr	0.3 g	Dissolution with NaOH/HNO ₃	ICP-OES with matrix matched standards, commercial mono-element solutions (Merck)
	Si, Fe, Cu, Mn, Mg, Cr, Zn, Ni, Ti, Ga, Be, Ca, Cd, Li, Na, Pb, Sn, V, Zr	0.3 g	Dissolution with HCl/H ₂ O ₂	ICP-OES with matrix matched standards, commercial mono-element solutions (Merck)
	Si, Fe, Cu, Mn, Mg, Cr, Zn, Ti, Pb, V			XRF, calibration with BAM-CRMs

Table 1 (cont.): Analytical procedures used by the participating laboratories

Lab-No.	Element	Sample mass	Sample pretreatment	Analytical method
9	Si, Mn	0.25 g	Dissolution with NaOH	Spectrophotometry
	Fe	0.5 g	Dissolution with HCl/H ₂ O ₂	Spectrophotometry, calibration with pure metals or pure chemicals
	Zr	0.5 g	Dissolution with NaOH,	Spectrophotometry, calibration with pure metals or pure chemicals
	Fe, Cu, Zn, Ni	2 g	Dissolution with HCl/H ₂ O ₂	FAAS, calibration with matrix matched standards, calibration with pure metals or pure chemicals
	Mn, Mg, Cr	2 g	Dissolution with HCl/H ₂ O ₂	FAAS, calibration with matrix matched standards, calibration with commercial mono-element solutions (Merck)
	Li	2 g	Dissolution with HCl/H ₂ O ₂	FAAS (measurement in emission mode), calibration with matrix matched standards, calibration with commercial mono-element solution (Merck)
	Pb	2 g	Dissolution with HCl/H ₂ O ₂	ETAAS, calibration with matrix matched standards, calibration with commercial mono-element solution (Merck)
	Hg	0.4 g	Dissolution with HCl/HNO ₃ /HF	CVAAS, calibration with commercial mono-element solution (Merck)
	Hg	0.3 g	Dissolution with HCl/HNO ₃ /HF	Atomic fluorescence, calibration with commercial mono-element solution (Merck)
10	Si, Fe, Cu, Mn, Mg, Cr, Zn, Ni, Ti, Ga, Be, Cd, Pb, Sn, V, Zr	0.25 g	Dissolution with NaOH/HNO ₃	ICP-OES, calibration with commercial mono-element solutions
11	Si, Fe, Cu, Mn, Mg, Cr, Zn, Ti, V, Be	0.5 g	Dissolution with NaOH	ICP-OES, calibration with matrix matched standards, commercial mono-element solutions
	Si, Fe, Cu, Mn, Mg, Cr, Zn, Ni, Ti, Ga, Be, Cd, Li, Na, Pb, Sn, V, Zr	1 g	Dissolution with HCl/HNO ₃	ICP-OES, calibration with matrix matched standards, commercial mono-element solutions
	Pb	1 g	Dissolution with HCl/HNO ₃	ICP-MS, calibration with matrix matched standard, commercial mono-element solution
14	Si, Fe, Cu, Mn, Mg, Cr, Zn, Ti, Ni, Be, Pb, Zr	0.5 g	Dissolution with NaOH	ICP-OES, calibration with matrix matched standards, commercial mono-element solutions
15	Si, Fe, Cu, Mn, Mg, Cr, Zn, Ti, Ga, V, Ni, B, Be, Ca, Cd, Li, Pb, Sn	0.5 g	Dissolution with NaOH	ICP-OES, calibration with matrix matched standards, commercial mono-element solutions

5.2 Analytical results and statistical evaluation

The analytical results of the inter-laboratory certification comparison are listed in Tables 2 to 21. These tables show the single results (M_i) of each laboratory, the respective laboratories' mean values (M), absolute and relative intra-laboratory standard deviation (s and s_{rel} , respectively), the standard deviation of laboratory means (s_M), and in addition the square root of mean of variances of data sets under repeatability conditions (\bar{s}_i) where n is the number of accepted data sets. 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. Outliers which have been excluded are highlighted in yellow.

Table 2: Results for Si

Lab./Meth.	8/XRF	8/l-a	4/P	7/l-s	14/l-a	1/l-a	7/G	5/l-a	15/l-a	2/l-a	11/l-a	10/l-a	6/l-a		
M_i [%]	1.198	1.206	1.207	1.207	1.210	1.210	1.197	1.210	1.219	1.217	1.232	1.220	1.235		n 13
	1.195	1.204	1.196	1.207	1.210	1.200	1.202	1.213	1.226	1.213	1.226	1.230	1.231		
	1.199	1.197	1.212	1.203	1.200	1.210	1.204	1.211	1.210	1.217	1.220	1.220	1.234		
	1.201	1.209	1.207	1.207	1.210	1.210	1.206	1.214	1.214	1.220	1.198	1.220	1.233		
	1.200	1.201	1.196	1.202	1.200	1.200	1.209	1.211	1.210	1.216	1.221	1.220	1.221		
	1.198	1.182	1.209	1.203	1.200	1.200	1.213	1.212	1.211	1.214	1.219	1.220	1.232		
M [%]	1.199	1.200	1.205	1.205	1.205	1.205	1.205	1.212	1.215	1.216	1.219	1.222	1.231		1.211
s [%]	0.0021	0.0097	0.0068	0.0024	0.0055	0.0055	0.0056	0.0014	0.0066	0.0023	0.0114	0.0041	0.0051	s_M [%]	0.0095
s_{rel}	0.0017	0.0081	0.0057	0.0020	0.0045	0.0045	0.0046	0.0012	0.0054	0.0019	0.0094	0.0033	0.0042	\bar{s}_i [%]	0.0060
															0.0079

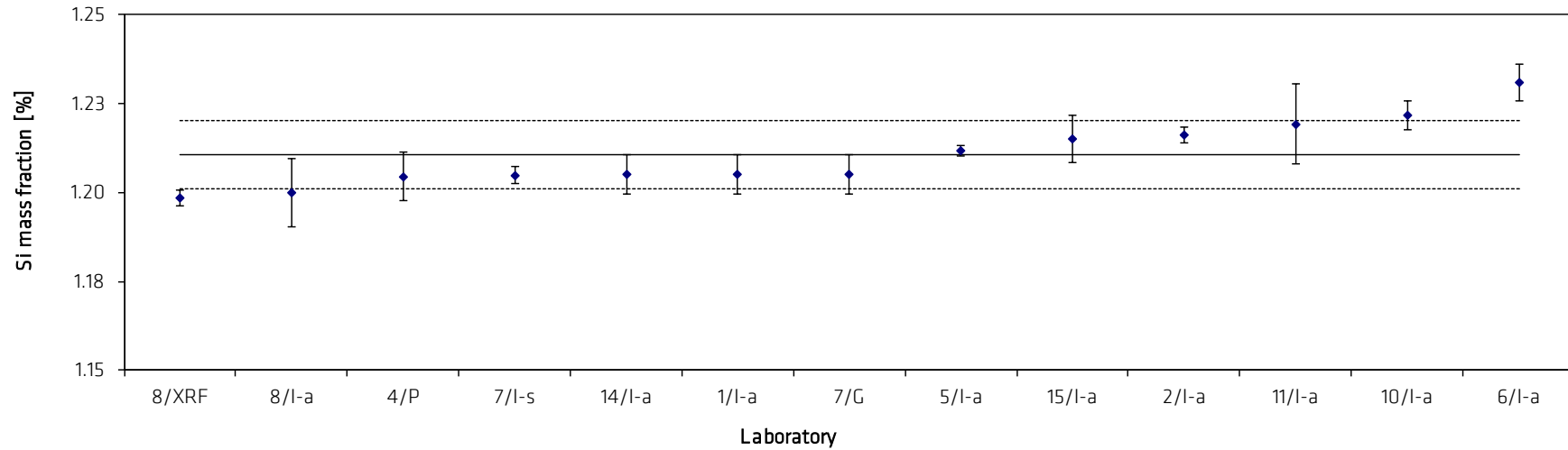


Table 3: Results for Fe

Lab./Meth.	9/l-s	6/l-a	5/l-a	8/l-s	4/l-s	9/A-s	1/l-a	8/l-a	14/l-a	2/l-a	15/l-a	8/XRF	11/l-a	11/l-s	7/l-s	10/l-a	9/P		
M_i [%]	0.2377	0.2380	0.2415	0.2437	0.245	0.2452	0.245	0.2446	0.250	0.2464	0.248	0.249	0.2512	0.251	0.2491	0.251	0.2570		n 17
	0.2327	0.2390	0.2422	0.2404	0.244	0.2453	0.243	0.2443	0.240	0.2473	0.247	0.249	0.2507	0.251	0.2499	0.253	0.2532		
	0.2346	0.2416	0.2415	0.2412	0.244	0.2453	0.243	0.2450	0.240	0.2464	0.247	0.249	0.2498	0.250	0.2506	0.251	0.2549		
	0.2393	0.2377	0.2423	0.2424	0.243	0.2361	0.244	0.2464	0.250	0.2474	0.247	0.249	0.2486	0.249	0.2511	0.251	0.2534		
	0.2420	0.2403	0.2417	0.2420	0.244	0.2507	0.244	0.2456	0.240	0.2485	0.249	0.249	0.2479	0.248	0.2492	0.250	0.2541		
	0.2407	0.2418	0.2415	0.243	0.2414	0.246	0.2408	0.250	0.2471	0.248	0.248	0.2466	0.247	0.2505	0.250	0.2543	0.2554		
M [%]	0.2373	0.2396	0.2418	0.2419	0.2438	0.2440	0.2442	0.2445	0.2450	0.2472	0.2477	0.2488	0.2491	0.2491	0.2501	0.2510	0.2546		0.2459
s [%]	0.0037	0.0016	0.0003	0.0011	0.0008	0.0049	0.0012	0.0019	0.0055	0.0008	0.0008	0.0004	0.0018	0.0018	0.0008	0.0011	0.0013	s_M [%]	0.0044
s_{rel}	0.0155	0.0065	0.0014	0.0047	0.0031	0.0200	0.0048	0.0080	0.0224	0.0032	0.0033	0.0016	0.0070	0.0070	0.0032	0.0044	0.0052	\bar{s}_i [%]	0.0023
																			0.0181

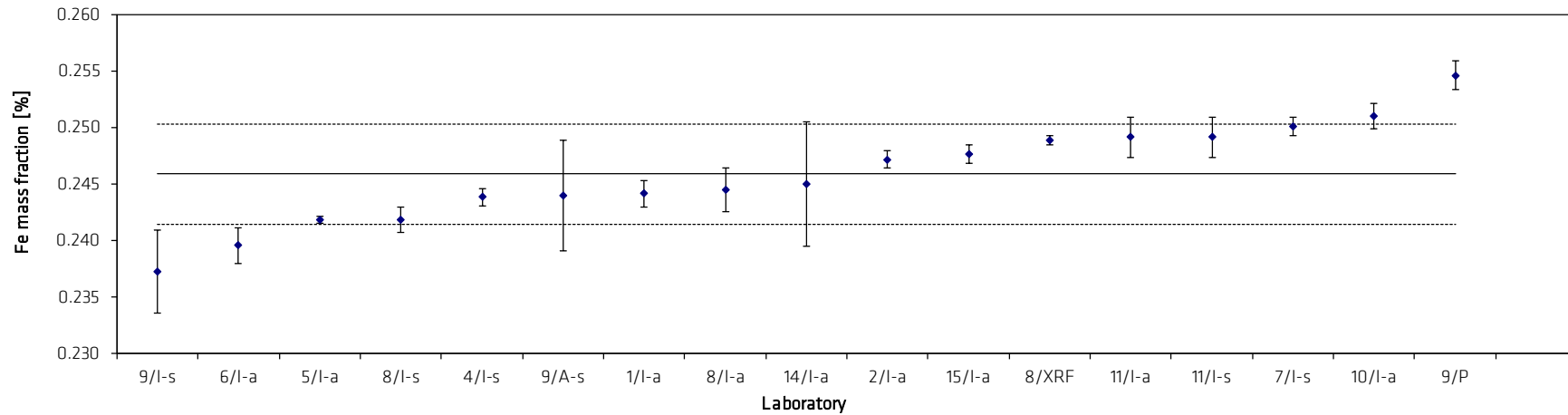


Table 4: Results for Cu

Lab./Meth.	9/A-s	8/l-s	9/l-s	6/l-a	4/l-s	11/l-s	5/l-a	15/l-a	14/l-a	2/l-a	1/l-a	11/l-a	10/l-a	7/l-s	8/XRF	8/l-a		
M_i [%]	0.0899	0.0892	0.0886	0.0890	0.089	0.0895	0.0905	0.091	0.091	0.0909	0.0910	0.0934	0.0924	0.0910	0.094	0.0910		n 16
	0.0901	0.0881	0.0898	0.0894	0.090	0.0908	0.0906	0.089	0.091	0.0907	0.0910	0.0921	0.0916	0.0917	0.094	0.0914		
	0.0876	0.0879	0.0876	0.0899	0.090	0.0900	0.0901	0.091	0.091	0.0909	0.0910	0.0925	0.0910	0.0922	0.094	0.0974		
	0.0893	0.0884	0.0882	0.0888	0.091	0.0898	0.0903	0.090	0.090	0.0912	0.0910	0.0907	0.0933	0.0923	0.094	0.0915		
	0.0831	0.0888	0.0884	0.0905	0.090	0.0894	0.0899	0.090	0.090	0.0916	0.0920	0.0914	0.0929	0.0921	0.094	0.0980		
	0.0889	0.0882	0.0888	0.0901	0.089	0.0906	0.0900	0.091	0.090	0.0915	0.0910	0.0905	0.0906	0.0936	0.094	0.0966		
M [%]	0.0882	0.0884	0.0886	0.0896	0.0898	0.0900	0.0902	0.0903	0.0905	0.0911	0.0912	0.0918	0.0920	0.0922	0.0940	0.0943		0.0908
s [%]	0.0026	0.0005	0.0007	0.0007	0.0008	0.0006	0.0003	0.0008	0.0005	0.0004	0.0004	0.0011	0.0011	0.0009	0.0000	0.0033	s_M [%]	0.0018
s_{rel}	0.0298	0.0054	0.0084	0.0077	0.0084	0.0062	0.0031	0.0090	0.0061	0.0039	0.0045	0.0120	0.0117	0.0093	0.0000	0.0352	\bar{s}_i [%]	0.0012
																		0.0198

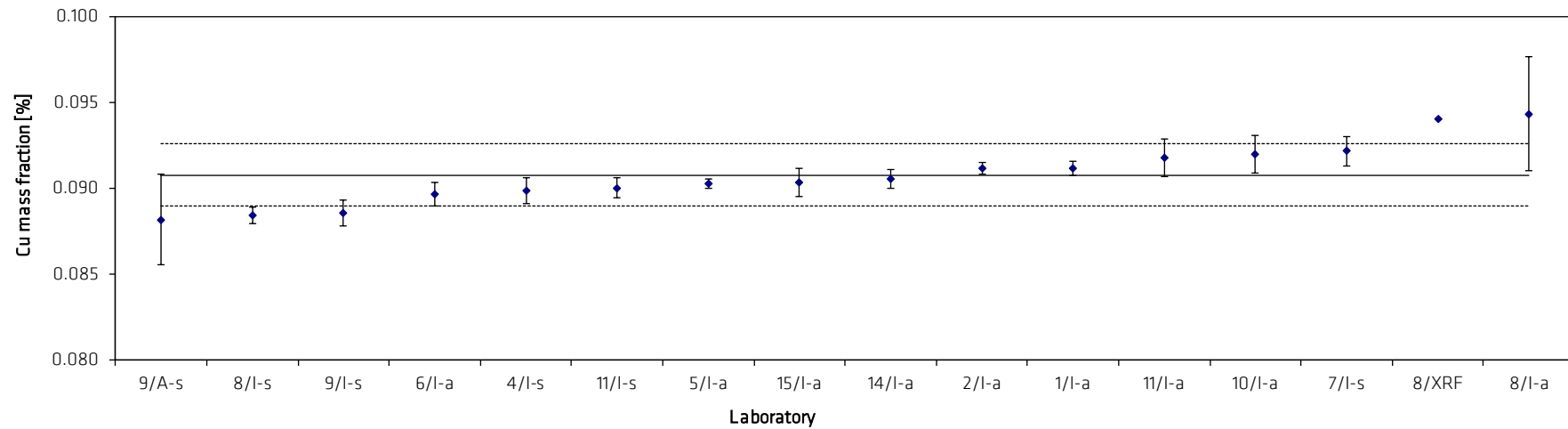


Table 5: Results for Mn

Lab./Meth.	6/l-a	8/l-a	14/l-a	15/l-a	11/l-a	8/XRF	1/l-a	8/l-s	9/A-s	9/l-s	2/l-a	5/l-a	4/l-s	9/P	11/l-s	7/IMS-s	10/l-a	7/l-s		
M_i [%]	0.0969	0.0980	0.097	0.098	0.0978	0.098	0.098	0.0991	0.098	0.0982	0.0985	0.0985	0.100	0.0983	0.0988	0.0980	0.102	0.1006		n 18
	0.0960	0.0914	0.097	0.098	0.0980	0.098	0.098	0.0976	0.096	0.0986	0.0983	0.0986	0.099	0.0985	0.0990	0.1008	0.102	0.1014		
	0.0965	0.0974	0.097	0.097	0.0978	0.098	0.098	0.0978	0.098	0.0977	0.0984	0.0984	0.098	0.0995	0.0987	0.1006	0.102	0.1034		
	0.0967	0.0983	0.098	0.097	0.0974	0.098	0.098	0.0982	0.100	0.0980	0.0984	0.0987	0.098	0.0998	0.0987	0.1000	0.102	0.1005		
	0.0967	0.0980	0.098	0.097	0.0973	0.098	0.098	0.0982	0.099	0.0985	0.0984	0.0984	0.100	0.0984	0.0994	0.0999	0.101	0.1037		
	0.0963	0.0966	0.098	0.098	0.0985	0.098	0.098	0.0980			0.0982	0.0984	0.097	0.0980	0.0992	0.0995	0.101	0.1015		
M [%]	0.0965	0.0966	0.0975	0.0975	0.0978	0.0980	0.0980	0.0981	0.0982	0.0982	0.0984	0.0985	0.0987	0.0988	0.0989	0.0998	0.1017	0.1019		0.0985
s [%]	0.0004	0.0026	0.0005	0.0005	0.0004	0.0000	0.0000	0.0005	0.0015	0.0004	0.0001	0.0001	0.0012	0.0007	0.0003	0.0010	0.0005	0.0014	s_M [%]	0.00142
s_{rel}	0.0036	0.0270	0.0056	0.0056	0.0045	0.0000	0.0000	0.0052	0.0151	0.0036	0.0008	0.0013	0.0123	0.0069	0.0028	0.0100	0.0051	0.0136	\bar{s}_i [%]	0.00093

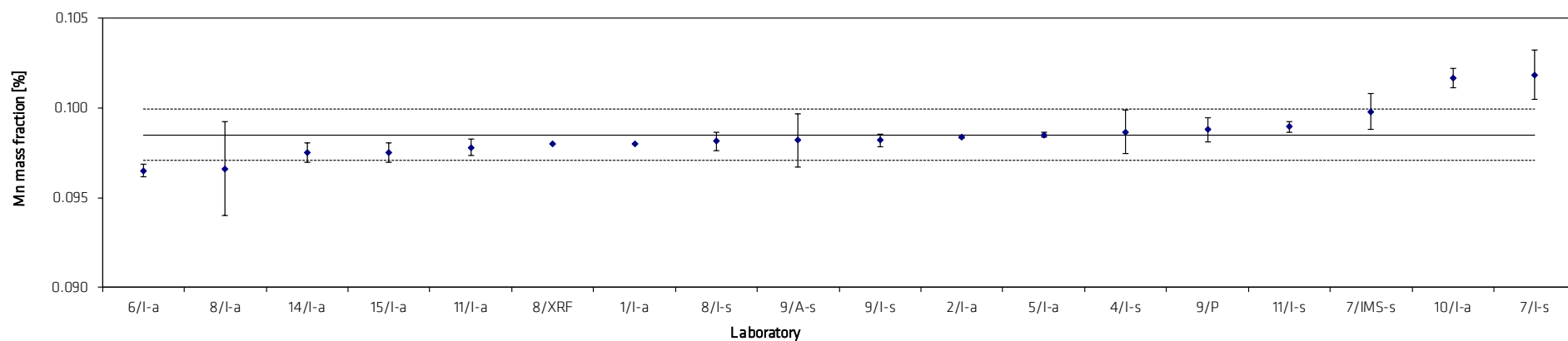


Table 6: Results for Mg

Lab./Meth.	8/l-s	5/l-a	14/l-a	4/l-s	8/l-a	7/l-s	9/l-s	1/l-a	15/l-a	2/l-a	11/l-a	11/l-s	9/A-s	8/XRF	6/l-a	10/l-a		
M_i [%]	0.330	0.342	0.34	0.346	0.351	0.353	0.351	0.356	0.353	0.356	0.358	0.362	0.362	0.366	0.379	0.40		n 16
	0.324	0.341	0.34	0.349	0.350	0.356	0.361	0.353	0.359	0.355	0.357	0.366	0.363	0.366	0.379	0.39		
	0.326	0.341	0.34	0.346	0.354	0.354	0.353	0.354	0.354	0.356	0.358	0.363	0.366	0.365	0.378	0.39		
	0.328	0.341	0.34	0.349	0.354	0.353	0.351	0.356	0.354	0.356	0.356	0.360	0.366	0.366	0.376	0.40		
	0.325	0.341	0.34	0.344	0.353	0.354	0.356	0.355	0.354	0.356	0.357	0.354	0.365	0.366	0.382	0.40		
	0.325	0.342	0.35	0.347	0.348	0.353	0.353	0.354	0.356	0.355	0.354	0.355	0.365	0.365	0.380	0.39		
M [%]	0.326	0.341	0.342	0.347	0.352	0.354	0.354	0.355	0.355	0.356	0.357	0.360	0.364	0.366	0.379	0.393		0.356
s [%]	0.0024	0.0005	0.0041	0.0019	0.0022	0.0014	0.0038	0.0012	0.0022	0.0007	0.0014	0.0050	0.0016	0.0005	0.0020	0.0039	s_M [%]	0.0153
s_{rel}	0.0072	0.0014	0.0119	0.0056	0.0063	0.0039	0.0107	0.0034	0.0062	0.0020	0.0039	0.0138	0.0045	0.0014	0.0052	0.0099	\bar{s}_i [%]	0.0025
																		0.0430

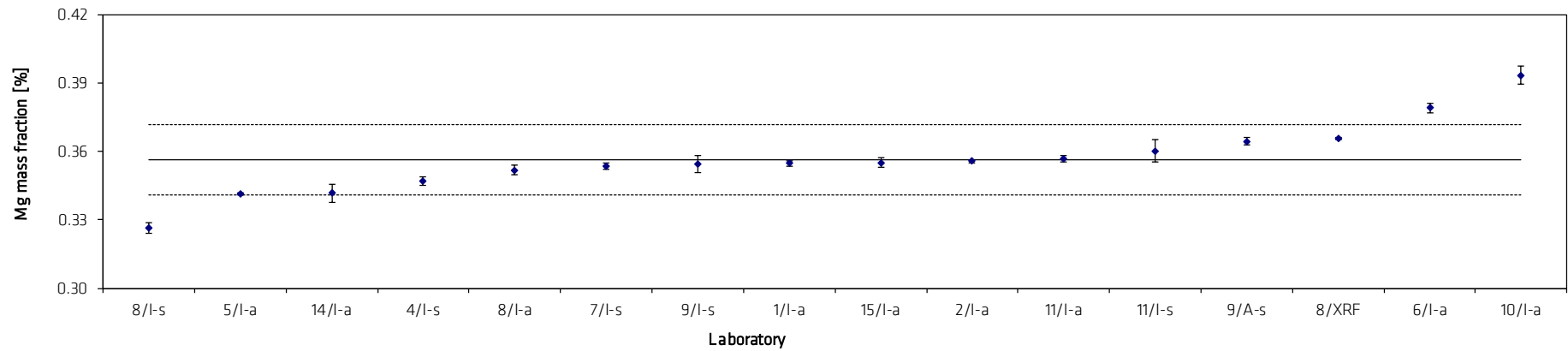


Table 7: Results for Cr

Lab./Meth.	7/I-s	7/IMS-s	9/A-s	8/I-a	6/I-a	8/I-s	4/I-s	1/I-a	5/I-a	15/I-a	11/I-s	14/I-a	8/XRF(R)	11/I-a	2/I-s	10/I-a		
M_i [%]	0.0197	0.0199	0.0204	0.0205	0.0205	0.021	0.020	0.0209	0.0210	0.0209	0.0209	0.021	0.0212	0.0212	0.0212	0.0213		n 16
	0.0201	0.0201	0.0207	0.0204	0.0205	0.021	0.021	0.0210	0.0209	0.0210	0.0211	0.021	0.0211	0.0214	0.0212	0.0213		
	0.0205	0.0206	0.0206	0.0205	0.0206	0.020	0.021	0.0209	0.0209	0.0210	0.0211	0.021	0.0212	0.0211	0.0215	0.0212		
	0.0207	0.0209	0.0205	0.0207	0.0206	0.021	0.021	0.0207	0.0209	0.0210	0.0209	0.021	0.0213	0.0211	0.0213	0.0213		
	0.0205	0.0202	0.0203	0.0205	0.0205	0.021	0.021	0.0208	0.0209	0.0209	0.0209	0.021	0.0213	0.0213	0.0212	0.0217		
	0.0205	0.0208	0.0200	0.0202	0.0204	0.020	0.021	0.0209	0.0209	0.0208	0.0209	0.021	0.0212	0.0213	0.0212	0.0209		
M [%]	0.0203	0.0204	0.0204	0.0205	0.0205	0.0206	0.0208	0.0209	0.0209	0.0209	0.0210	0.0212	0.0212	0.0212	0.0213	0.0213		0.0208
s [%]	0.0004	0.0004	0.0002	0.0002	0.0001	0.0002	0.0004	0.0001	0.0000	0.0001	0.0001	0.0004	0.0001	0.0001	0.0001	0.0003	s_M [%]	0.0003
s_{rel}	0.018	0.020	0.012	0.008	0.003	0.009	0.020	0.005	0.002	0.004	0.004	0.019	0.003	0.005	0.005	0.012	\bar{s}_i [%]	0.0002
																		0.016

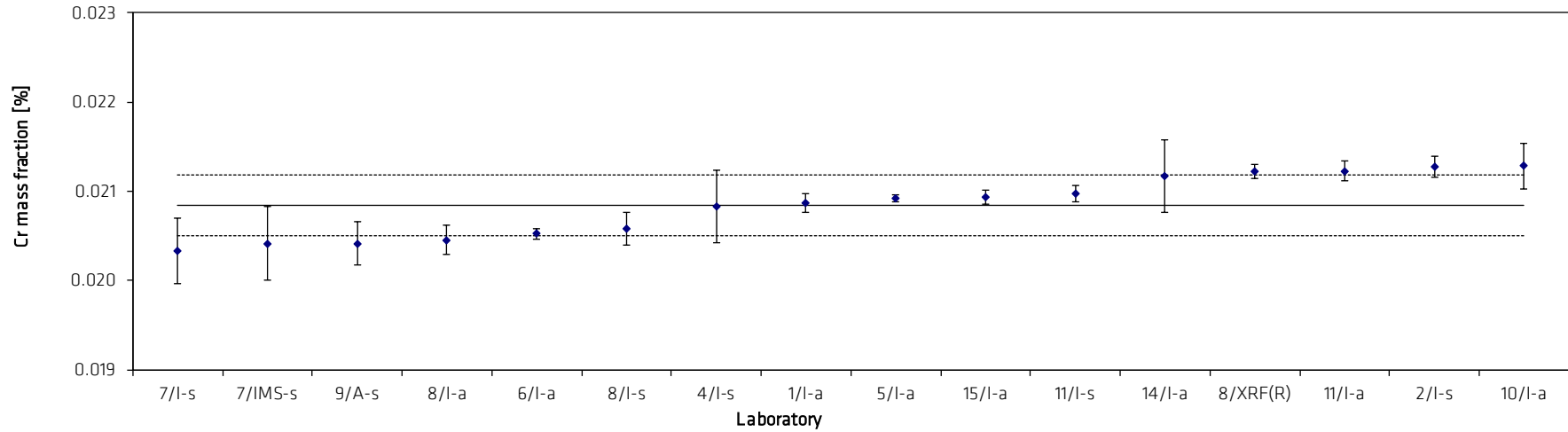


Table 8: Results for Zn

Lab./Meth.	4/l-s	10/l-a	6/l-a	8/l-a	11/l-s	1/l-a	2/l-a	8/XRF(R)	15/l-a	11/l-a	5/l-a	9/A-s	7/l-s	8/l-s	14/l-a		
M_i [%]	0.0480	0.0480	0.0475	0.0478	0.0476	0.048	0.0481	0.0483	0.050	0.0493	0.0486	0.049	0.049	0.052	0.050		n
	0.0470	0.0477	0.0477	0.0472	0.0480	0.048	0.0478	0.0483	0.047	0.0487	0.0491	0.050	0.051	0.049	0.050		15
	0.0470	0.0476	0.0477	0.0477	0.0478	0.048	0.0478	0.0484	0.048	0.0495	0.0490	0.050	0.051	0.050	0.050		
	0.0470	0.0475	0.0477	0.0480	0.0475	0.048	0.0482	0.0479	0.049	0.0475	0.0487	0.049	0.050	0.050	0.051		
	0.0470	0.0475	0.0478	0.0481	0.0474	0.048	0.0482	0.0483	0.048	0.0487	0.0487	0.050	0.050	0.051	0.051		
	0.0480	0.0476	0.0476	0.0473	0.0481	0.048	0.0482	0.0483	0.048	0.0480	0.0489	0.049	0.049	0.051	0.051		
M [%]	0.0473	0.0477	0.0477	0.0477	0.0477	0.0480	0.0481	0.0482	0.0483	0.0486	0.0488	0.0495	0.0501	0.0504	0.0505		0.0486
s [%]	0.0005	0.0002	0.0001	0.0004	0.0003	0.0000	0.0002	0.0002	0.0009	0.0008	0.0002	0.0005	0.0008	0.0011	0.0005	s_M [%]	0.0011
s_{rel}	0.011	0.004	0.002	0.007	0.006	0.000	0.004	0.003	0.018	0.015	0.004	0.011	0.016	0.021	0.011	\bar{s}_i [%]	0.0005
																	0.022

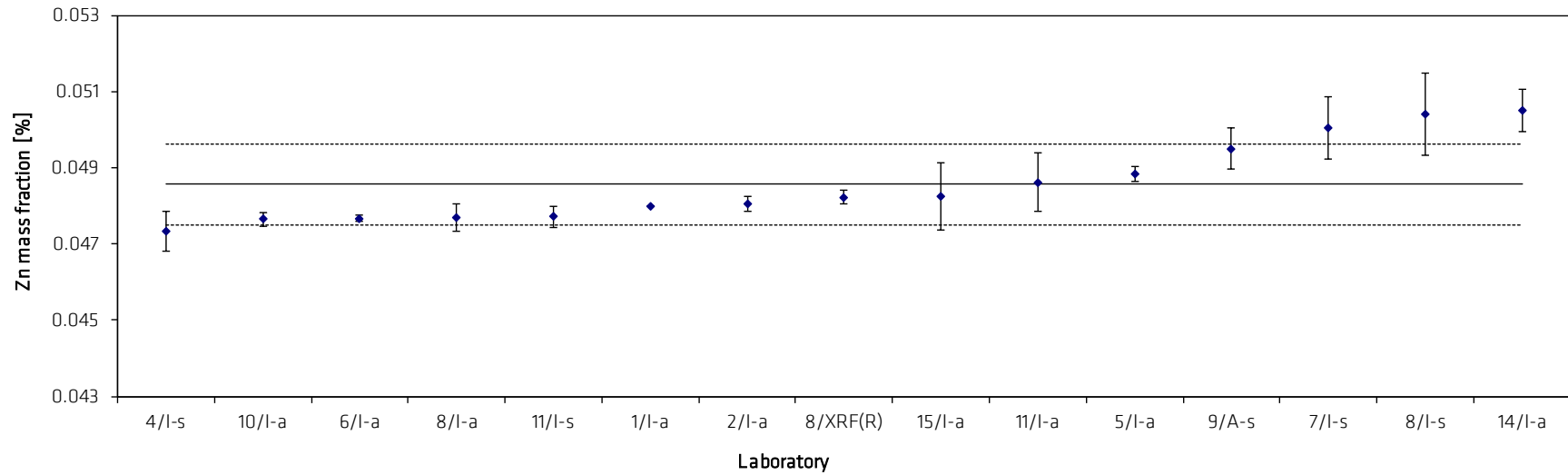


Table 9: Results for Ti

Lab./Meth.	4/l-s	8/l-a	7/l-s	9/l-s	15/l-a	8/l-s	5/l-a	9/P	7/IMS-s	11/l-a	11/l-s	6/l-a	1/l-a	2/l-a	10/l-a(R)	14/l-a	8/XRF		
M_i [%]	0.0231	0.0231	0.0227	0.0228	0.0233	0.0236	0.0236	0.0238	0.0231	0.0238	0.0240	0.0239	0.0249	0.0238	0.0251	0.025	0.025		n 17
	0.0226	0.0231	0.0232	0.0237	0.0232	0.0238	0.0236	0.0237	0.0242	0.0240	0.0241	0.0239	0.0241	0.0241	0.0246	0.024	0.025		
	0.0236	0.0230	0.0232	0.0231	0.0234	0.0232	0.0235	0.0236	0.0241	0.0238	0.0241	0.0238	0.0238	0.0240	0.0239	0.025	0.025		
	0.0228	0.0232	0.0233	0.0230	0.0234	0.0234	0.0236	0.0238	0.0238	0.0238	0.0239	0.0241	0.0236	0.0243	0.0237	0.024	0.025		
	0.0231	0.0233	0.0230	0.0231	0.0232	0.0235	0.0235	0.0235	0.0237	0.0238	0.0239	0.0243	0.0237	0.0243	0.0235	0.025	0.025		
	0.0224	0.0228	0.0235	0.0234	0.0233	0.0233	0.0235	0.0236	0.0238	0.0240	0.0239	0.0241	0.0241	0.0243	0.0249	0.025	0.025		
M [%]	0.0229	0.0231	0.0232	0.0232	0.0233	0.0235	0.0236	0.0237	0.0238	0.0239	0.0240	0.0240	0.0240	0.0241	0.0243	0.0247	0.0250		0.0238
s [%]	0.00043	0.00019	0.00027	0.00032	0.00009	0.00024	0.00005	0.00011	0.00039	0.00009	0.00011	0.00016	0.00047	0.00021	0.00067	0.00052	0.00000	s_M [%]	0.00057
s_{rel}	0.0186	0.0083	0.0118	0.0140	0.0038	0.0101	0.0023	0.0046	0.0163	0.0039	0.0045	0.0068	0.0196	0.0088	0.0276	0.0209	0.0000	\bar{s}_i [%]	0.00031
																			0.0239

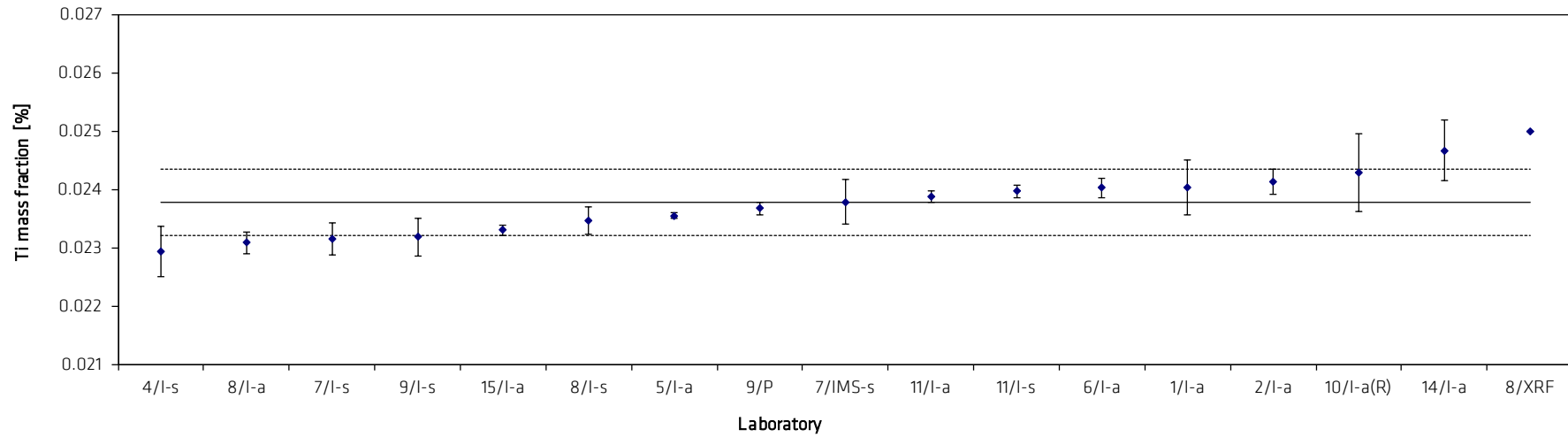


Table 10: Results for Ga

Lab./Meth.	7/IMS-s	5/l-a	9/l-s	8/l-a	10/l-a(R)	15/l-a	2/l-a	8/l-s	6/l-a	1/l-a	11/l-s	4/l-s			
M_i [%]	0.0177	0.0182	0.0187	0.0185	0.0188	0.0189	0.0187	0.0190	0.0188	0.0192	0.0191	0.0195		n 11	
	0.0178	0.0182	0.0190	0.0187	0.0186	0.0188	0.0189	0.0189	0.0190	0.0191	0.0194	0.0193			
	0.0177	0.0184	0.0184	0.0188	0.0190	0.0187	0.0188	0.0188	0.0191	0.0190	0.0194	0.0192			
	0.0181	0.0183	0.0185	0.0188	0.0184	0.0188	0.0189	0.0189	0.0189	0.0192	0.0192	0.0192			
	0.0176	0.0185	0.0186	0.0187	0.0187	0.0190	0.0189	0.0189	0.0189	0.0187	0.0191	0.0192	0.0195		
	0.0180	0.0185	0.0187	0.0183	0.0186	0.0187	0.0189	0.0189	0.0187	0.0188	0.0191	0.0192	0.0192		
M [%]	0.0178	0.0184	0.0186	0.0187	0.0187	0.0188	0.0189	0.0189	0.0189	0.0191	0.0192	0.0193		0.0189	
s [%]	0.00019	0.00014	0.00021	0.00021	0.00020	0.00013	0.00008	0.00009	0.00013	0.00008	0.00011	0.00015	s_M [%]	0.00028	
s_{rel}	0.0109	0.0075	0.0115	0.0110	0.0109	0.0067	0.0042	0.0046	0.0068	0.0039	0.0055	0.0076	\bar{s}_i [%]	0.00015	
														0.0151	

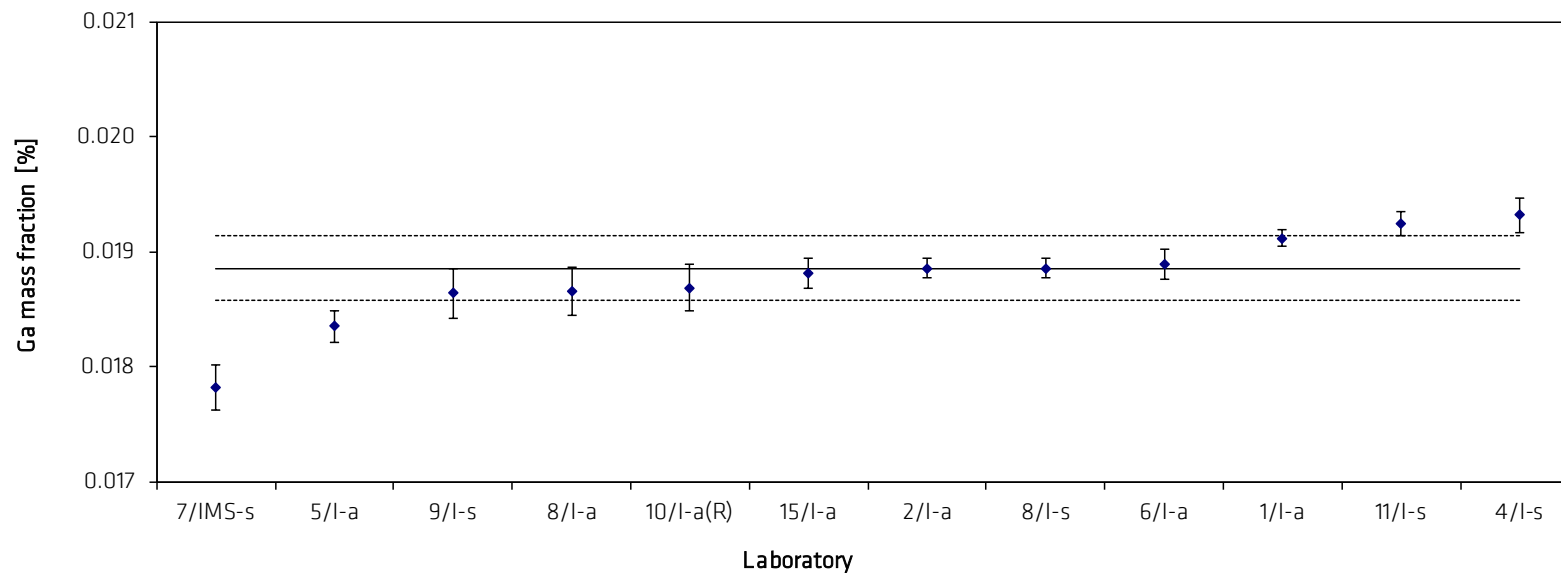


Table 11: Results for V

Lab./Meth.	7/l-s	7/IMS-s	15/l-a	9/P	9/l-s	10/l-a	5/l-a	6/l-a	2/l-s	1/l-a	8/l-a	8/l-s	11/l-a	11/l-s	8/XRF	4/l-s			
M_i [%]	0.0098	0.0096	0.0102	0.0100	0.0100	0.0102	0.0104	0.0103	0.0104	0.0105	0.0105	0.0106	0.0105	0.0106	0.011	0.0108		n 16	
	0.0099	0.0100	0.0100	0.0102	0.0104	0.0104	0.0104	0.0104	0.0104	0.0105	0.0104	0.0105	0.0104	0.0106	0.010	0.0108			
	0.0095	0.0102	0.0101	0.0100	0.0101	0.0103	0.0103	0.0104	0.0105	0.0104	0.0105	0.0105	0.0105	0.0106	0.011	0.0108			
	0.0101	0.0103	0.0101	0.0101	0.0101	0.0103	0.0103	0.0104	0.0103	0.0104	0.0106	0.0105	0.0105	0.0105	0.011	0.0108			
	0.0100	0.0100	0.0100	0.0102	0.0102	0.0103	0.0103	0.0103	0.0103	0.0103	0.0104	0.0106	0.0104	0.0105	0.0106	0.011	0.0108		
	0.0101	0.0105	0.0102	0.0103	0.0103	0.0103	0.0103	0.0103	0.0103	0.0104	0.0104	0.0104	0.0104	0.0106	0.0106	0.010	0.0107		
M [%]	0.0099	0.0101	0.0101	0.0101	0.0102	0.0103	0.0103	0.0104	0.0104	0.0104	0.0105	0.0105	0.0105	0.0106	0.0107	0.0108		0.0104	
s [%]	0.0002	0.0003	0.0001	0.0001	0.0001	0.0001	0.0001	0.0000	0.0001	0.0001	0.0001	0.0001	0.0000	0.0000	0.0005	0.0000		s_M [%] 0.00023	
s_{rel}	0.023	0.031	0.009	0.014	0.013	0.006	0.005	0.003	0.007	0.005	0.008	0.007	0.004	0.004	0.048	0.004		\bar{s}_i [%] 0.00018	
																		0.022	

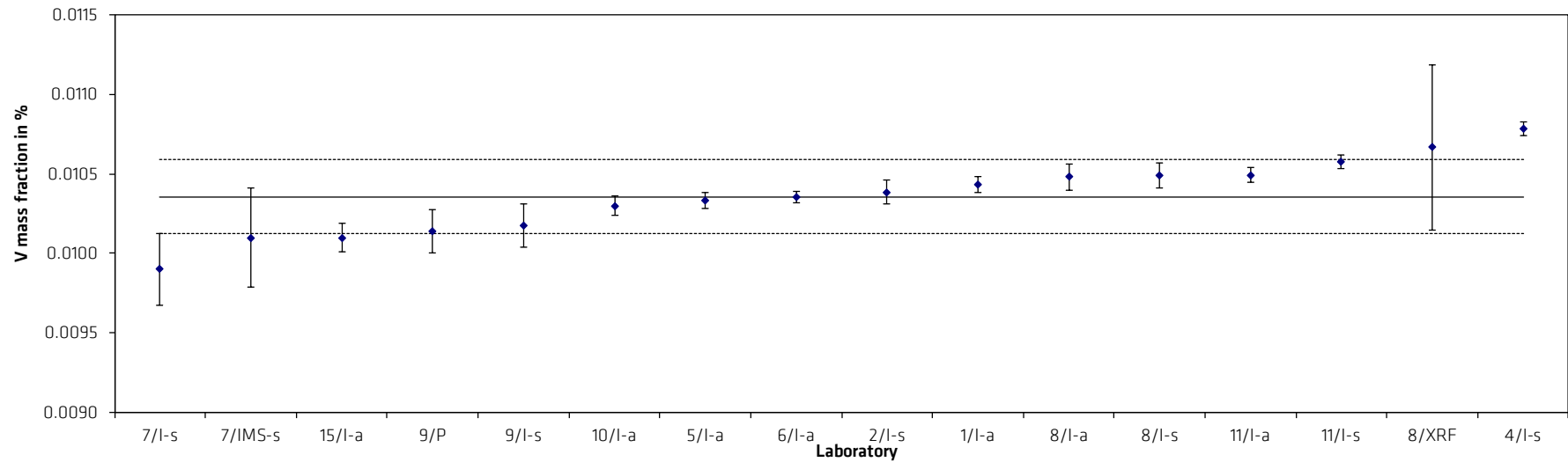


Table 12: Results for Ni

Lab./Meth.	8/l-s(R)	9/A-s	7/l-s	9/l-s	11/l-s	8/l-a	7/IMS-s	2/l-s	6/l-a	10/l-a	15/l-a	5/l-a	1/l-a	4/l-s	14/l-a		
M_i [mg/kg]	46.3	48	47.6	49.0	48.5	49.9	49.6	49.5	49.8	49.4	50.8	51.8	52	53	56		n 15
	45.1	49	49.2	49.5	49.4	48.9	48.2	49.5	50.3	50.2	51.0	51.8	52	53	52		
	45.2	50	51.8	48.3	49.7	49.1	50.6	49.8	50.6	50.3	50.7	51.6	51	53	54		
	46.3	48	48.1	48.5	48.7	49.8	51.3	50.5	49.9	50.1	50.0	51.5	52	53	53		
	45.3	47	47.6	49.4	49.0	49.8	49.0	50.2	49.8	50.4	50.8	51.4	52	53	53		
	45.2	47	46.2	49.1	48.6	48.8	50.0	50.3	50.0	50.2	51.0	51.4	56	52	53		
M [mg/kg]	45.6	48.2	48.4	48.9	49.0	49.4	49.8	50.0	50.1	50.1	50.7	51.6	52.5	52.8	53.5		50.0
s [mg/kg]	0.57	1.17	1.93	0.48	0.47	0.50	1.11	0.43	0.32	0.36	0.37	0.19	1.76	0.41	1.38	s_M [mg/kg]	2.028
s_{rel}	0.0125	0.0243	0.0398	0.0098	0.0095	0.0102	0.0223	0.0087	0.0064	0.0071	0.0073	0.0037	0.0335	0.0077	0.0258	\bar{s}_i [mg/kg]	0.9345
																	0.0405

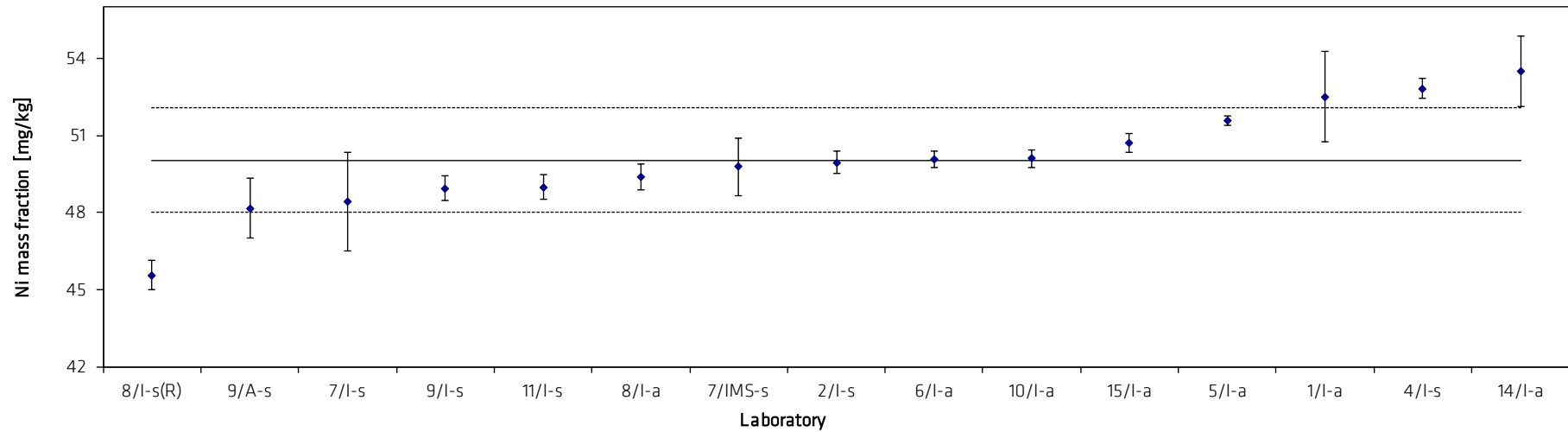


Table 13: Results for Be

Lab./Meth.	4/l-s	5/l-a	9/l-s	1/l-s	8/l-s	11/l-s	11/l-a	6/l-a	15/l-a	2/l-s	14/l-a	10/l-a	7/IMS-s		
M_i [mg/kg]	4.0	4.1	4.29	4.6	4.70	4.65	4.84	4.83	4.9	4.92	5.0	5.2	5.22		n 13
	4.0	4.1	4.48	4.5	4.65	4.74	4.84	4.88	4.9	4.90	5.0	5.2	5.25		
	4.0	4.1	4.30	4.6	4.67	4.72	4.81	4.78	4.9	5.03	5.0	5.2	5.36		
	4.0	4.1	4.33	4.5	4.67	4.69	4.79	4.80	5.0	4.91	5.0	5.2	5.34		
	4.0	4.1	4.50	4.7	4.61	4.67	4.80	4.81	4.9	4.93	5.0	5.1	5.27		
	4.0	4.1	4.53	4.5	4.62	4.66	4.75	4.86	4.9	4.99	5.0	5.2	5.28		
M [mg/kg]	4.00	4.06	4.40	4.57	4.65	4.69	4.80	4.83	4.92	4.94	5.00	5.18	5.29		4.72
s [mg/kg]	0.000	0.000	0.108	0.082	0.034	0.036	0.034	0.035	0.041	0.053	0.000	0.041	0.054	s_M [mg/kg]	0.388
s_{rel}	0.0000	0.0000	0.0246	0.0179	0.0073	0.0077	0.0072	0.0073	0.0083	0.0107	0.0000	0.0079	0.0101	\bar{s}_i [mg/kg]	0.050
															0.0822

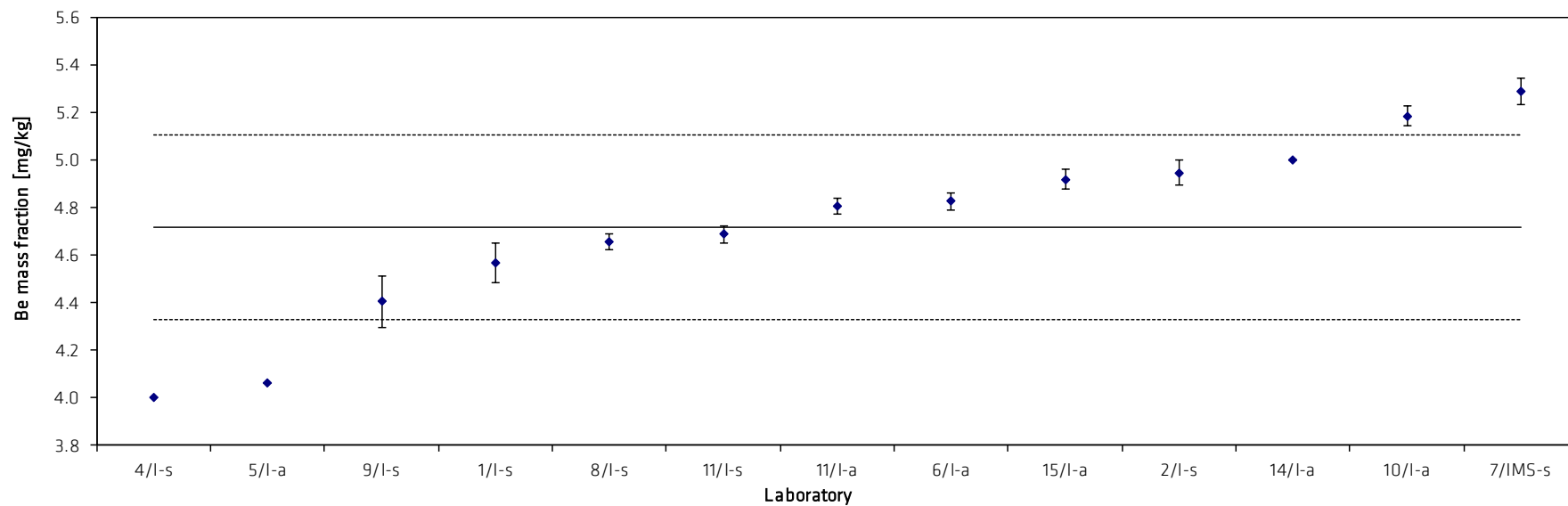


Table 14: Results for Ca

Lab./Meth.	9/l-s	2/l-s	15/l-a	6/l-s	8/l-s		
M_i [mg/kg]	7.29	8.3	9	10.0	9.7		n 5
	6.51	8.3	9	8.2	8.8		
	7.38	8.4	9	10.1	16.9		
	5.30	8.3	8	10.3	10.1		
	7.60	8.5	9	10.3	9.3		
		8.4	9	11.3	12.8		
M [mg/kg]	6.82	8.37	8.83	10.04	11.25		9.06
s [mg/kg]	0.945	0.079	0.408	1.014	3.079	s_M [mg/kg]	1.682
s_{rel}	0.139	0.009	0.046	0.101	0.274	\bar{s}_i [mg/kg]	1.522
							0.186

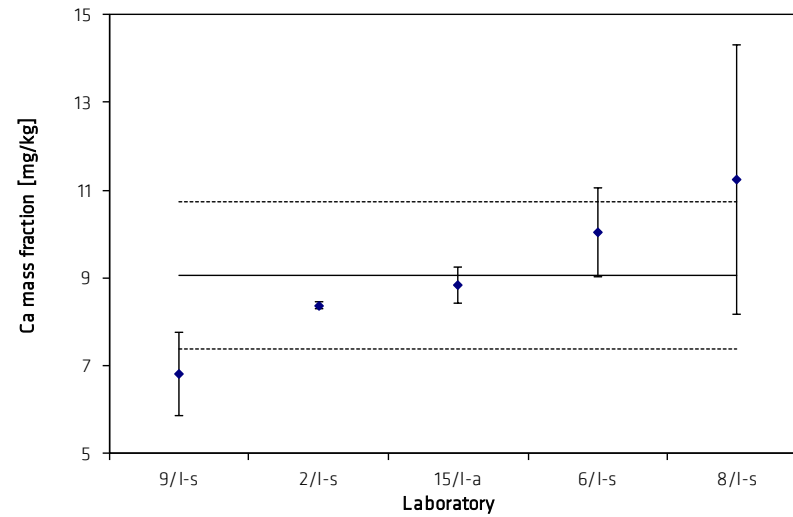


Table 15: Results for Cd

Lab./Meth.	8/l-s	11/l-s	9/l-s	10/l-a(R)	2/IMS-s	5/l-a	15/l-a	6/l-a	7/IMS-s	1/l-s	4/l-s		
M_i [mg/kg]	9.21	9.29	10.83	9.65	9.34	9.60	9.7	9.88	9.79	10	10		n
	9.29	9.42	9.27	9.75	9.56	9.61	9.7	9.79	9.72	10	10		10
	9.29	9.39	9.04	9.89	9.55	9.60	9.6	9.76	9.92	10	10		
	9.35	9.34	9.06	9.03	9.62	9.74	9.7	9.46	9.81	10	11		
	9.31	9.31	9.26	9.27	9.64	9.64	9.7	9.74	9.91	10	11		
	9.28	9.28	9.27	9.55	9.58	9.59	9.7	9.60	9.78	10	11		
M [mg/kg]	9.3	9.3	9.5	9.5	9.5	9.6	9.7	9.7	9.8	10.0	10.5		9.60
s [mg/kg]	0.05	0.05	0.68	0.32	0.11	0.06	0.04	0.15	0.08	0.00	0.55	s_M [mg/kg]	0.22
s_{rel}	0.0049	0.0059	0.0722	0.0335	0.0111	0.0059	0.0042	0.0152	0.0080	0.0000	0.0522	\bar{s}_i [mg/kg]	0.25
													0.0226

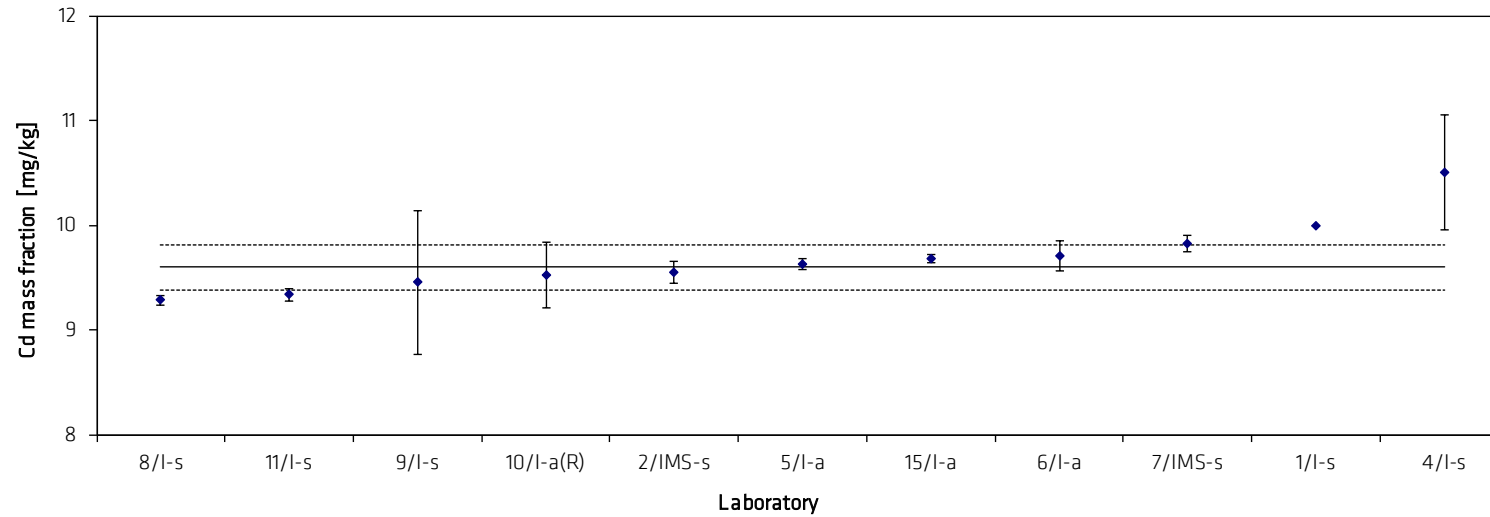


Table 16: Results for Hg

Lab./Meth.	2/IMS-s	4/l-s	9/CVAAS	1/l-s	9/AF-s	6/l-s		
M_i [mg/kg]	6.18	7	7.67	9	7.8	8.4		n 6
	6.15	7	7.70	7	7.7	8.0		
	6.41	7	7.57	8	8.1	8.9		
	6.69	7	7.67	8	8.0	9.4		
	6.76	7	7.39	8	7.2	8.8		
	6.69	7	7.57	7	8.3	9.5		
M [mg/kg]	6.48	7.00	7.60	7.83	7.85	8.83		7.60
s [mg/kg]	0.27	0.00	0.11	0.75	0.38	0.58	s_M [mg/kg]	0.804
							\bar{s}_i [mg/kg]	0.434
s_{rel}	0.042	0.000	0.015	0.096	0.048	0.065		0.106

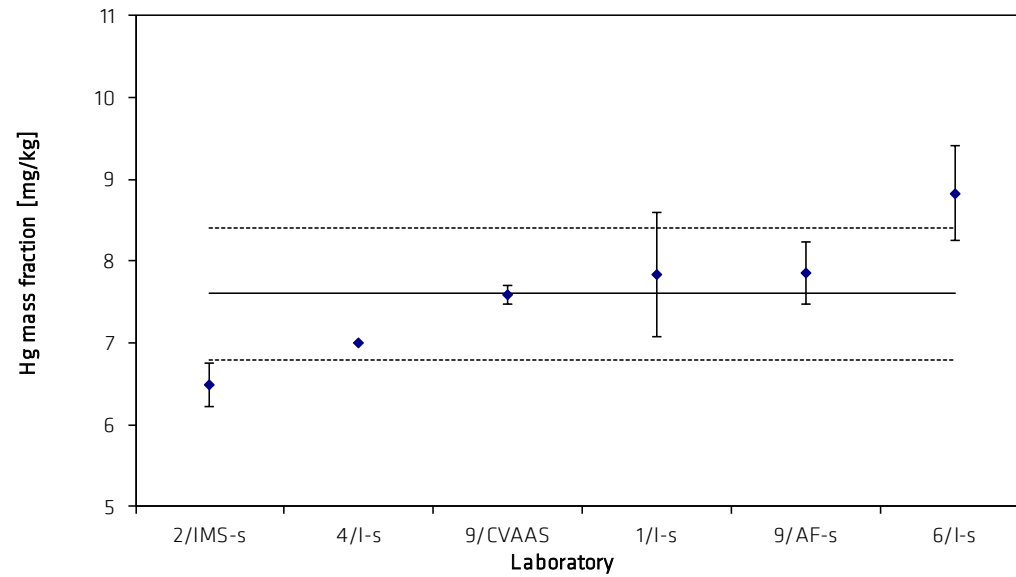


Table 17: Results for Li

Lab./Meth.	4/l-s	15/l-a	8/l-s(R)	9/AE-s	6/l-a	7/IMS-s	1/l-s(R)	9/l-s	2/l-s	5/l-a	11/l-s		
M_i [mg/kg]	5	5.6	5.9	5.9	5.9	5.8	6.0	5.9	6.0	6.1	6		n
	5	5.5	6.1	6.0	6.1	6.0	6.0	6.1	6.1	6.0	6		10
	5	5.6	5.4	5.9	6.0	6.0	6.0	6.0	6.2	6.2	6		
	5	5.6	5.8	6.0	6.0	6.0	6.0	6.0	6.0	6.0	6		
	5	5.5	5.6	5.9	6.0	6.0	6.0	6.0	6.0	6.2	6		
	5	5.5	5.8	6.0	6.0	6.1			6.0	5.9	6		
M [mg/kg]	5.00	5.55	5.77	5.95	5.98	5.98	6.00	6.00	6.06	6.07	6.16		5.95
s [mg/kg]	0.000	0.055	0.242	0.056	0.049	0.096	0.000	0.092	0.107	0.121	0.038	s_M [mg/kg]	0.174
s_{rel}	0.0000	0.0099	0.0420	0.0094	0.0082	0.0161	0.0000	0.0153	0.0176	0.0200	0.0061	\bar{s}_i [mg/kg]	0.111
													0.0292

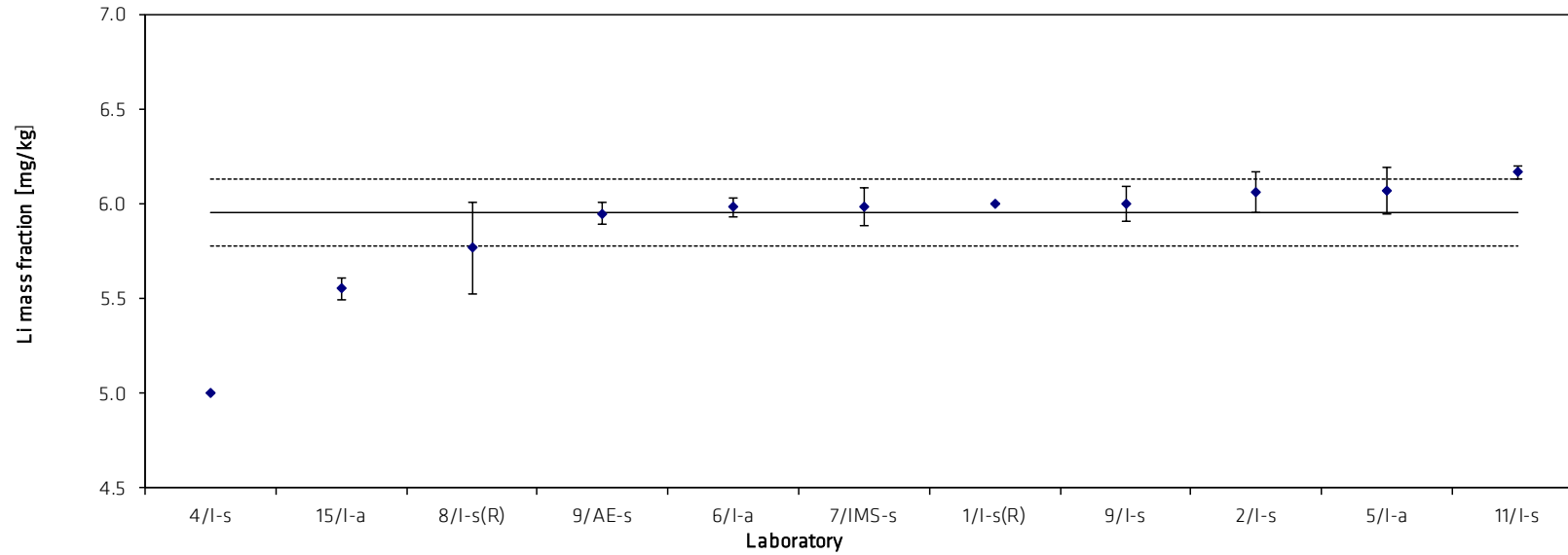


Table 18: Results for Na

Lab./Meth.	2/l-s	11/l-s	6/l-s	8/l-s		
M_i [mg/kg]	3.0	3.4	4.0	4		n
	3.0	3.4	4.2	4		4
	3.1	3.3	4.2	4		
	3.1	3.1	4.2	4		
	3.1	3.1	4.2	5		
	3.0	3.3	4.1	4		
M [mg/kg]	3.06	3.29	4.16	4.33		3.71
s [mg/kg]	0.089	0.136	0.099	0.169	s_M [mg/kg]	0.629
s_{rel}	0.029	0.041	0.024	0.039	\bar{s}_i [mg/kg]	0.127
						0.170

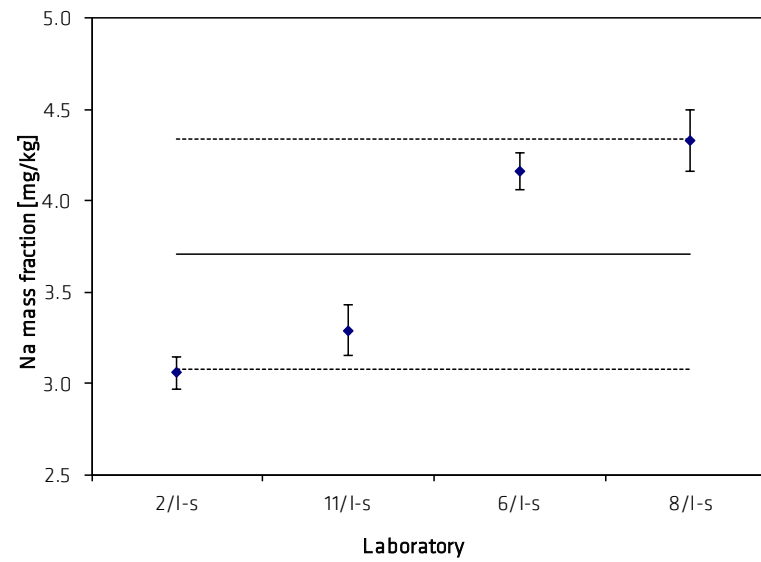


Table 19: Results for Pb

Lab./Meth.	9/EA	6/l-a	5/l-a	11/IMS-s	10/l-a	7/l-s	15/l-a	4/l-s	2/IMS-s	9/l-s	1/l-a	7/IMS-s	8/XRF	14/l-a	8/l-s		
M_i [mg/kg]	50	53.5	53.8	52.8	52.8	59.1	54	56	54.7	57.1	57	58.4	60.6	62	61.6		n 15
	52	52.2	52.7	53.4	54.9	55.8	55	54	54.5	57.6	57	58.9	60.9	59	57.3		
	52	52.9	52.9	54.2	54.4	50.3	52	54	54.5	53.8	57	62.0	59.9	60	60.2		
	[46.6]	53.5	53.9	53.7	55.7	53.0	54	55	55.0	54.9	57	57.8	60.5	60	67.0		
	52	52.3	53.4	[49.5]	53.4	50.7	55	55	55.5	56.8	57	57.2	60.5	64	61.9		
	51	53.3	53.1	53.8	52.4	54.8	54	54	55.1	55.3	57	57.9	59.9	58	66.1		
M [mg/kg]	51.4	52.9	53.3	53.6	53.9	54.0	54.0	54.7	54.9	55.9	57.0	58.7	60.4	60.5	62.3		55.8
s [mg/kg]	0.77	0.57	0.49	0.51	1.28	3.33	1.10	0.82	0.38	1.48	0.00	1.72	0.40	2.17	3.65	s_M [mg/kg]	3.23
s_{rel}	0.015	0.011	0.009	0.010	0.024	0.062	0.020	0.015	0.007	0.026	0.000	0.029	0.007	0.036	0.059	\bar{s}_i [mg/kg]	1.62
																	0.058

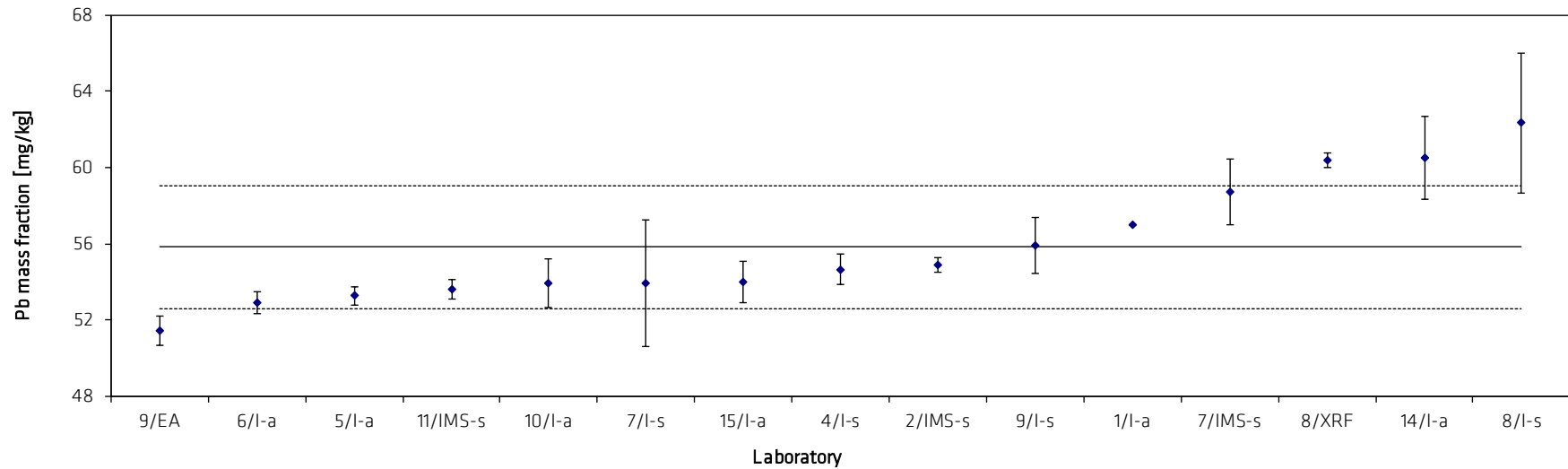


Table 20: Results for Sn

Lab./Meth.	8/l-s	10/l-a	9/l-s	11/l-s	5/l-a	8/l-a	15/l-a	2/IMS-s	7/IMS-s	7/l-s	6/l-a	1/l-s	4/l-s		
M_i [mg/kg]	17.3	19.0	18.6	20.6	20.8	23.0	21	20.6	21.4	19.6	20.1	21	22		n 13
	18.3	18.9	18.7	20.0	20.9	22.1	21	20.9	21.8	23.6	20.3	22	22		
	18.5	18.8	19.3	20.3	21.5	17.8	21	21.4	21.4	19.9	22.8	22	21		
	19.8	18.4	19.3	21.3	20.4	21.2	21	20.7	21.2	20.7	23.2	21	21		
	18.3	18.7	19.1	20.6	20.2	20.7	21	21.3	21.0	24.9	20.7	23	22		
	17.1	19.5		20.6	20.1	19.7	21	21.3	21.4	20.0	21.5	21	26		
M [mg/kg]	18.21	18.88	19.00	20.57	20.65	20.75	21.00	21.03	21.37	21.45	21.45	21.67	22.33		20.64
s [mg/kg]	0.97	0.37	0.35	0.44	0.55	1.84	0.00	0.36	0.27	2.22	1.32	0.82	1.86	s_M [mg/kg]	1.216
s_{rel}	0.054	0.019	0.018	0.022	0.027	0.089	0.000	0.017	0.012	0.104	0.062	0.038	0.083	\bar{s}_i [mg/kg]	1.113
															0.059

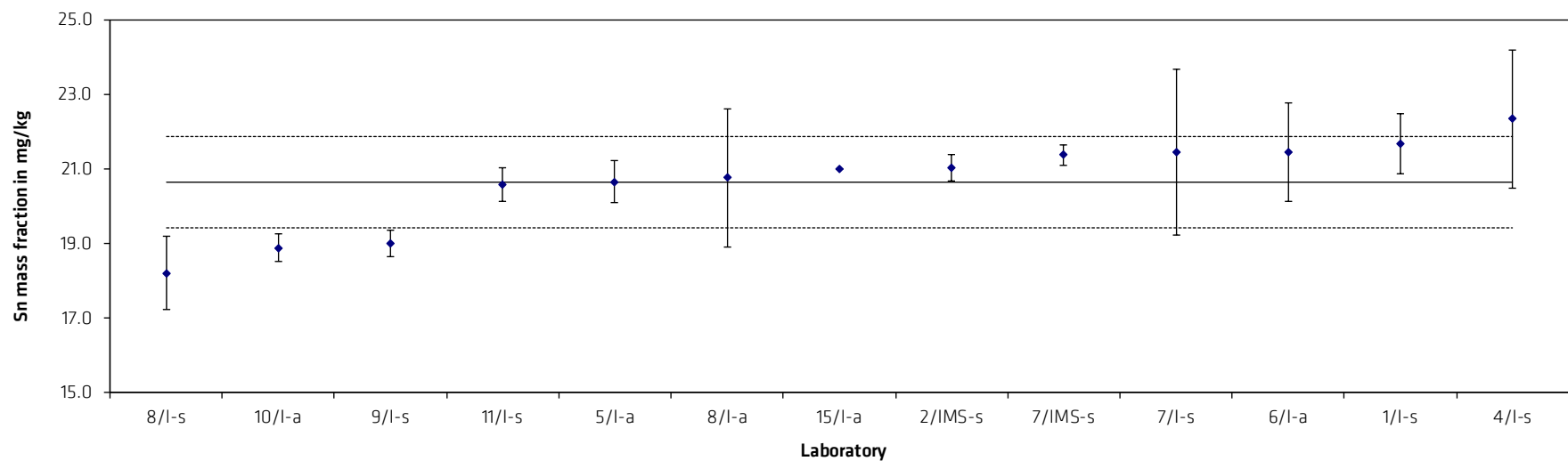
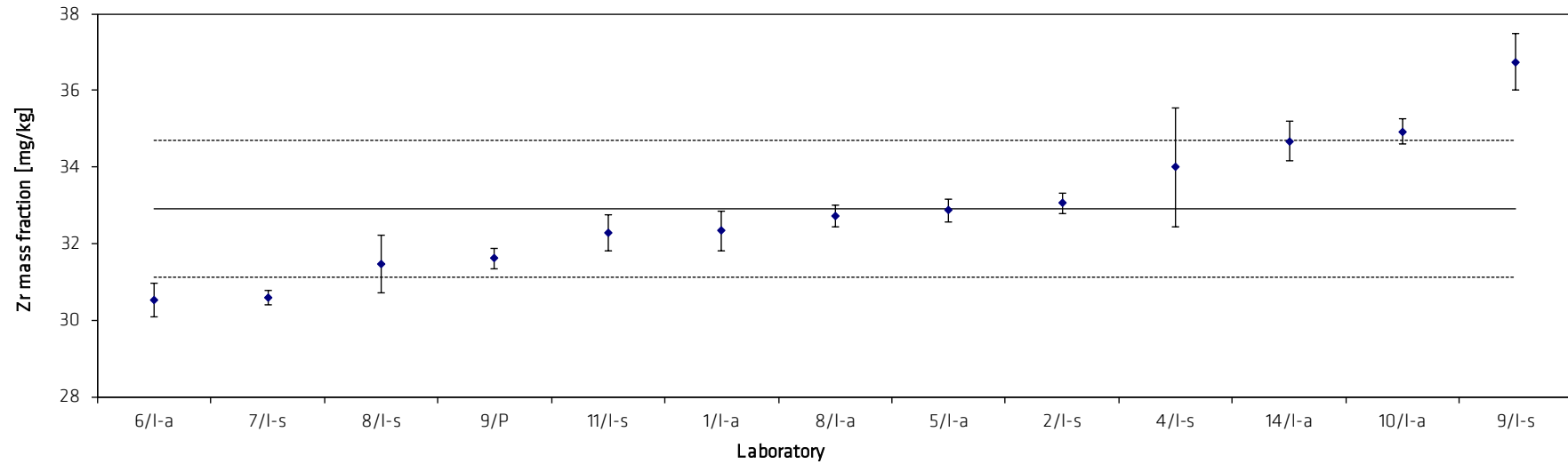


Table 21: Results for Zr

Lab./Meth.	6/l-a	7/l-s	8/l-s	9/P	11/l-s	1/l-a	8/l-a	5/l-a	2/l-s	4/l-s	14/l-a	10/l-a	9/l-s		
M_i [mg/kg]	30.2	30.4	32.1	31.7	31.4	33	32.8	33.4	33	34	34	34.7	35.7		n
	31.1	30.8	30.9	31.2	32.7	32	32.5	33.0	33	35	34	35.1	37.1		13
	30.7	30.4	31.5	31.5	32.7	32	32.7	32.7	33	34	35	34.7	36.6		
	30.1	30.8	32.6	31.3	32.3	32	32.6	32.8	33	35	35	35.3	36.6		
	31.0	30.4	30.9	31.9	32.3	33	33.2	32.6	33	35	35	35.2	37.6		
	30.2	30.7	30.8	31.9	32.3	32	32.4	32.6	33	31	35	34.5			
				31.74											
M [mg/kg]	30.5	30.6	31.5	31.6	32.3	32.3	32.7	32.9	33.1	34.0	34.7	34.9	36.7		32.9
s [mg/kg]	0.45	0.19	0.76	0.27	0.46	0.52	0.27	0.31	0.26	1.55	0.52	0.33	0.73	s_M [mg/kg]	1.80
														\bar{s}_i [mg/kg]	0.614
s_{rel}	0.0146	0.0062	0.0241	0.0085	0.0143	0.0160	0.0084	0.0094	0.0079	0.0456	0.0149	0.0093	0.0198		0.0546



Four laboratories determined boron and found 0.4 mg/kg, 1.1 mg/kg, < 1 mg/kg and < 2 mg/kg. Homogeneity data is not available.

The statistical evaluation of the data was performed using the software program SoftCRM 1.2.2. [4]. The following results were obtained:

Tab. 22: Outcome of statistical tests on the results obtained for Si and Fe

	Si	Fe
Number of data sets	13	17
Scheffe's test (data compatible?)	yes	yes
Snedecor-F-Test and Bartlett-Test	Pooling not allowed	Pooling not allowed
Dixon ($\alpha = 0.05$)	---	---
Dixon ($\alpha = 0.01$)	---	---
Nalimov ($\alpha = 0.05$)	Lab. 6	Labs. 9/I, 9/P
Nalimov ($\alpha = 0.01$)	---	---
Grubbs ($\alpha = 0.05$)	---	---
Grubbs ($\alpha = 0.01$)	---	---
Grubbs Pair ($\alpha = 0.05$)	---	---
Grubbs Pair ($\alpha = 0.01$)	---	---
Cochran	---	---
Kolmogorov-Smirnov-Lilliefors Test ($\alpha = 0.05$)	Distribution: not normal	Distribution: normal
Kolmogorov-Smirnov-Lilliefors Test ($\alpha = 0.01$)	Distribution: normal	Distribution: normal
Skewness & Kurtosis Test ($\alpha = 0.05$)	Distribution: normal	Distribution: normal
Skewness & Kurtosis Test ($\alpha = 0.01$)	Distribution: normal	Distribution: normal

The stragglers (Lab. 6; Si and Labs. 9/I, 9/P; Fe) were not removed.

Tab. 23: Outcome of statistical tests on the results obtained for Cu and Mn

	Cu	Mn
Number of data sets	16	18
Scheffe's test (data compatible?)	yes	yes
Snedecor-F-Test and Bartlett-Test	Pooling not allowed	Pooling not allowed
Dixon ($\alpha = 0.05$)	---	---
Dixon ($\alpha = 0.01$)	---	---
Nalimov ($\alpha = 0.05$)	Lab. 8/I-a	Labs. 7/I, 10/I
Nalimov ($\alpha = 0.01$)	---	Lab. 7/I
Grubbs ($\alpha = 0.05$)	---	---
Grubbs ($\alpha = 0.01$)	---	---
Grubbs Pair ($\alpha = 0.05$)	---	---
Grubbs Pair ($\alpha = 0.01$)	---	---
Cochran	---	---
Kolmogorov-Smirnov-Lilliefors Test ($\alpha = 0.05$)	Distribution: normal	Distribution: not normal
Kolmogorov-Smirnov-Lilliefors Test ($\alpha = 0.01$)	Distribution: normal	Distribution: normal
Skewness & Kurtosis Test ($\alpha = 0.05$)	Distribution: normal	Distribution: not normal
Skewness & Kurtosis Test ($\alpha = 0.01$)	Distribution: normal	Distribution: normal

The stragglers (Lab. 8/I-a; Cu and Labs. 7/I, 10/I; Mn) were not removed.

Tab. 24: Outcome of statistical tests on the results obtained for Mg and Cr

	Mg	Cr
Number of data sets	16	16
Scheffe's test (data compatible?)	yes	yes
Snedecor-F-Test and Bartlett-Test	Pooling not allowed	Pooling not allowed
Dixon ($\alpha = 0.05$)	---	---
Dixon ($\alpha = 0.01$)	---	---
Nalimov ($\alpha = 0.05$)	Labs. 10 and 8/I-a	---
Nalimov ($\alpha = 0.01$)	Lab. 10	---
Grubbs ($\alpha = 0.05$)	---	---
Grubbs ($\alpha = 0.01$)	---	---
Grubbs Pair ($\alpha = 0.05$)	---	---
Grubbs Pair ($\alpha = 0.01$)	---	---
Cochran	---	---
Kolmogorov-Smirnov-Lilliefors Test ($\alpha = 0.05$)	Distribution: normal	Distribution: normal
Kolmogorov-Smirnov-Lilliefors Test ($\alpha = 0.01$)	Distribution: normal	Distribution: normal
Skewness & Kurtosis Test ($\alpha = 0.05$)	Distribution: normal	Distribution: normal
Skewness & Kurtosis Test ($\alpha = 0.01$)	Distribution: normal	Distribution: normal

The stragglers (Labs. 10 and 8/I-a, Mg) were not removed.

Tab. 25: Outcome of statistical tests on the results obtained for Zn and Ti

	Zn	Ti
Number of data sets	15	17
Scheffe's test (data compatible?)	yes	yes
Snedecor-F-Test and Bartlett-Test	Pooling not allowed	Pooling not allowed
Dixon ($\alpha = 0.05$)	---	---
Dixon ($\alpha = 0.01$)	---	---
Nalimov ($\alpha = 0.05$)	---	Lab. 8/X
Nalimov ($\alpha = 0.01$)	---	---
Grubbs ($\alpha = 0.05$)	---	---
Grubbs ($\alpha = 0.01$)	---	---
Grubbs Pair ($\alpha = 0.05$)	---	---
Grubbs Pair ($\alpha = 0.01$)	---	---
Cochran	---	---
Kolmogorov-Smirnov-Lilliefors Test ($\alpha = 0.05$)	Distribution: normal	Distribution: normal
Kolmogorov-Smirnov-Lilliefors Test ($\alpha = 0.01$)	Distribution: normal	Distribution: normal
Skewness & Kurtosis Test ($\alpha = 0.05$)	Distribution: normal	Distribution: normal
Skewness & Kurtosis Test ($\alpha = 0.01$)	Distribution: normal	Distribution: normal

The straggler (Lab. 8/X; Ti) was not removed.

Tab. 26: Outcome of statistical tests on the results obtained for Ga

	1 st run	2 nd run
Number of data sets	12	11
Scheffe's test (data compatible?)	yes	yes
Snedecor-F-Test and Bartlett-Test	Pooling not allowed	Pooling not allowed
Dixon ($\alpha = 0.05$)	Lab. 7/IMS	---
Dixon ($\alpha = 0.01$)	---	---
Nalimov ($\alpha = 0.05$)	Lab. 7/IMS	---
Nalimov ($\alpha = 0.01$)	Lab. 7/IMS	---
Grubbs ($\alpha = 0.05$)	Lab. 7/IMS	---
Grubbs ($\alpha = 0.01$)	---	---
Grubbs Pair ($\alpha = 0.05$)	---	---
Grubbs Pair ($\alpha = 0.01$)	---	---
Cochran	---	---
Kolmogorov-Smirnov-Lilliefors Test ($\alpha = 0.05$)	Distribution: normal	Distribution: normal
Kolmogorov-Smirnov-Lilliefors Test ($\alpha = 0.01$)	Distribution: normal	Distribution: normal
Skewness & Kurtosis Test ($\alpha = 0.05$)	Distribution: normal	Distribution: normal
Skewness & Kurtosis Test ($\alpha = 0.01$)	Distribution: normal	Distribution: normal

The outlier (Lab. 7/IMS) was removed.

Tab. 27: Outcome of statistical tests on the results obtained for V and Ni

	V	Ni
Number of data sets	15	15
Scheffe's test (data compatible?)	yes	yes
Snedecor-F-Test and Bartlett-Test	Pooling not allowed	Pooling not allowed
Dixon ($\alpha = 0.05$)	---	---
Dixon ($\alpha = 0.01$)	---	---
Nalimov ($\alpha = 0.05$)	Lab. 7/I-s	Labs. 8/I-s, 14
Nalimov ($\alpha = 0.01$)	---	---
Grubbs ($\alpha = 0.05$)	---	---
Grubbs ($\alpha = 0.01$)	---	---
Grubbs Pair ($\alpha = 0.05$)	---	---
Grubbs Pair ($\alpha = 0.01$)	---	---
Cochran	---	---
Kolmogorov-Smirnov-Lilliefors Test ($\alpha = 0.05$)	Distribution: normal	Distribution: normal
Kolmogorov-Smirnov-Lilliefors Test ($\alpha = 0.01$)	Distribution: normal	Distribution: normal
Skewness & Kurtosis Test ($\alpha = 0.05$)	Distribution: normal	Distribution: normal
Skewness & Kurtosis Test ($\alpha = 0.01$)	Distribution: normal	Distribution: normal

The stragglers (Lab. 7/I-s, V; Labs. 14 and 8/I-s, Ni) were not removed.

Tab. 28: Outcome of statistical tests on the results obtained for Be and Ca

	Be	Ca
Number of data sets	13	5
Scheffe's test (data compatible?)	yes	yes
Snedecor-F-Test and Bartlett-Test	Pooling not allowed	Pooling not allowed
Dixon ($\alpha = 0.05$)	---	---
Dixon ($\alpha = 0.01$)	---	---
Nalimov ($\alpha = 0.05$)	---	---
Nalimov ($\alpha = 0.01$)	---	---
Grubbs ($\alpha = 0.05$)	---	---
Grubbs ($\alpha = 0.01$)	---	---
Grubbs Pair ($\alpha = 0.05$)	---	---
Grubbs Pair ($\alpha = 0.01$)	---	---
Cochran	---	---
Kolmogorov-Smirnov-Lilliefors Test ($\alpha = 0.05$)	Distribution: normal	Distribution: normal
Kolmogorov-Smirnov-Lilliefors Test ($\alpha = 0.01$)	Distribution: normal	Distribution: normal
Skewness & Kurtosis Test ($\alpha = 0.05$)	Distribution: normal	Distribution: insufficient data
Skewness & Kurtosis Test ($\alpha = 0.01$)	Distribution: normal	Distribution: insufficient data

Tab. 29: Outcome of statistical tests on the results obtained for Hg and Na

	Hg	Na
Number of data sets	6	4
Scheffe's test (data compatible?)	yes	yes
Snedecor-F-Test and Bartlett-Test	Pooling not allowed	Pooling not allowed
Dixon ($\alpha = 0.05$)	---	---
Dixon ($\alpha = 0.01$)	---	---
Nalimov ($\alpha = 0.05$)	---	---
Nalimov ($\alpha = 0.01$)	---	---
Grubbs ($\alpha = 0.05$)	---	---
Grubbs ($\alpha = 0.01$)	---	---
Grubbs Pair ($\alpha = 0.05$)	---	---
Grubbs Pair ($\alpha = 0.01$)	---	---
Cochran	---	---
Kolmogorov-Smirnov-Lilliefors Test ($\alpha = 0.05$)	Distribution: normal	Distribution: normal
Kolmogorov-Smirnov-Lilliefors Test ($\alpha = 0.01$)	Distribution: normal	Distribution: normal
Skewness & Kurtosis Test ($\alpha = 0.05$)	Distribution: insufficient data	Distribution: insufficient data
Skewness & Kurtosis Test ($\alpha = 0.01$)	Distribution: insufficient data	Distribution: insufficient data

Tab. 30: Outcome of statistical tests on the results obtained for Cd

	1 st run	2 nd run
Number of data sets	11	10
Scheffe's test (data compatible?)	yes	yes
Snedecor-F-Test and Bartlett-Test	Pooling not allowed	Pooling not allowed
Dixon ($\alpha = 0.05$)	---	---
Dixon ($\alpha = 0.01$)	---	---
Nalimov ($\alpha = 0.05$)	Lab. 4	Lab. 1
Nalimov ($\alpha = 0.01$)	Lab. 4	---
Grubbs ($\alpha = 0.05$)	Lab. 4	---
Grubbs ($\alpha = 0.01$)	---	---
Grubbs Pair ($\alpha = 0.05$)	---	---
Grubbs Pair ($\alpha = 0.01$)	---	---
Cochran	---	---
Kolmogorov-Smirnov-Lilliefors Test ($\alpha = 0.05$)	Distribution: normal	Distribution: normal
Kolmogorov-Smirnov-Lilliefors Test ($\alpha = 0.01$)	Distribution: normal	Distribution: normal
Skewness & Kurtosis Test ($\alpha = 0.05$)	Distribution: not normal	Distribution: normal
Skewness & Kurtosis Test ($\alpha = 0.01$)	Distribution: normal	Distribution: normal

The outlier (Lab. 4) was removed, the straggler (Lab. 1) was not removed.

Tab. 31: Outcome of statistical tests on the results obtained for Li

	1 st run	2 nd run
Number of data sets	11	9
Scheffe's test (data compatible?)	yes	yes
Snedecor-F-Test and Bartlett-Test	Pooling not allowed	Pooling not allowed
Dixon ($\alpha = 0.05$)	Lab. 4	---
Dixon ($\alpha = 0.01$)	Lab. 4	---
Nalimov ($\alpha = 0.05$)	Lab. 4	Lab. 15/I-a
Nalimov ($\alpha = 0.01$)	Lab. 4	Lab. 15/I-a
Grubbs ($\alpha = 0.05$)	Lab. 4	Lab. 15/I-a
Grubbs ($\alpha = 0.01$)	Lab. 4	---
Grubbs Pair ($\alpha = 0.05$)	---	---
Grubbs Pair ($\alpha = 0.01$)	---	---
Cochran	---	---
Kolmogorov-Smirnov-Lilliefors Test ($\alpha = 0.05$)	Distribution: not normal	Distribution: normal
Kolmogorov-Smirnov-Lilliefors Test ($\alpha = 0.01$)	Distribution: not normal	Distribution: normal
Skewness & Kurtosis Test ($\alpha = 0.05$)	Distribution: not normal	Distribution: normal
Skewness & Kurtosis Test ($\alpha = 0.01$)	Distribution: not normal	Distribution: normal

The outlier (Lab. 4) was removed, the straggler (Lab. 15/I-a) was not removed.

Tab. 32: Outcome of statistical tests on the results obtained for Pb and Zr

	Pb	Zr
Number of data sets	15	14
Scheffe's test (data compatible?)	yes	yes
Snedecor-F-Test and Bartlett-Test	Pooling not allowed	Pooling not allowed
Dixon ($\alpha = 0.05$)	---	---
Dixon ($\alpha = 0.01$)	---	---
Nalimov ($\alpha = 0.05$)	Lab. 8/l-s	Labs. 9/l
Nalimov ($\alpha = 0.01$)	---	---
Grubbs ($\alpha = 0.05$)	---	---
Grubbs ($\alpha = 0.01$)	---	---
Grubbs Pair ($\alpha = 0.05$)	---	---
Grubbs Pair ($\alpha = 0.01$)	---	---
Cochran	---	---
Kolmogorov-Smirnov-Lilliefors Test ($\alpha = 0.05$)	Distribution: normal	Distribution: normal
Kolmogorov-Smirnov-Lilliefors Test ($\alpha = 0.01$)	Distribution: normal	Distribution: normal
Skewness & Kurtosis Test ($\alpha = 0.05$)	Distribution: normal	Distribution: normal
Skewness & Kurtosis Test ($\alpha = 0.01$)	Distribution: normal	Distribution: normal

The stragglers (Lab. 8/l-s, Pb; Lab. 9/l, Zr) were not removed.

Tab. 33: Outcome of statistical tests on the results obtained for Sn

	Sn
Number of data sets	13
Scheffe's test (data compatible?)	yes
Snedecor-F-Test and Bartlett-Test	Pooling not allowed
Dixon ($\alpha = 0.05$)	---
Dixon ($\alpha = 0.01$)	---
Nalimov ($\alpha = 0.05$)	Lab. 8/l-s
Nalimov ($\alpha = 0.01$)	---
Grubbs ($\alpha = 0.05$)	---
Grubbs ($\alpha = 0.01$)	---
Grubbs Pair ($\alpha = 0.05$)	---
Grubbs Pair ($\alpha = 0.01$)	---
Cochran	---
Kolmogorov-Smirnov-Lilliefors Test ($\alpha = 0.05$)	Distribution: normal
Kolmogorov-Smirnov-Lilliefors Test ($\alpha = 0.01$)	Distribution: normal
Skewness & Kurtosis Test ($\alpha = 0.05$)	Distribution: normal
Skewness & Kurtosis Test ($\alpha = 0.01$)	Distribution: normal

The straggler (Lab. 8/l-s) was not removed.

The certified mass fractions of all elements were calculated as mean of the accepted data sets. These values are given in Table 34.

The resp. combined uncertainties were calculated from the spread resulting from the certification inter-laboratory comparison (u_{ilc}) and the uncertainty contributions from possible inhomogeneity over the length ($u_{bb}(1)$) and over area ($u_{bb}(2)$) of the material using Equation 3.

$$U_{\text{combined}} = \sqrt{u_{ilc}^2 + u_{bb}^2(1) + u_{bb}^2(2)} \quad (3)$$

with

$$u_{ilc} = \sqrt{\frac{s_M^2}{n}} : \text{uncertainty contribution resulting from inter-laboratory comparison}$$

n : number of data sets used for calculating the certified mass fraction of each element

Table 34: Uncertainty calculation ($u_{bb}(\text{rel})$) was calculated with the data from the homogeneity test (see Annex 1 and 2) and used for the calculation of $u_{bb}(2)$ and $u_{bb}(2)$ by multiplication with M)

	uncertainty contribution from				$u_{bb}(1)$ Length	$u_{bb}(2)$ Area	$u(\text{comb})$	U	$u_{bb}(\text{rel})$			
	M	n	s_M	u_{ilc}					Length	Area	Length	Area
	%		%	%					%	%		
Si	1.2110	13	0.0095	0.0026	0.0059	0.0055	0.0084	0.01689	0.4851	0.4511		
Fe	0.2459	17	0.0044	0.0011	0.0024	0.0027	0.0037	0.00748	0.9682	1.0890		
Cu	0.0908	16	0.0018	0.0004	0.0007	0.0009	0.0012	0.00244	0.7221	1.0230		
Mn	0.0985	18	0.0014	0.0003	0.0007	0.0003	0.0008	0.00168	0.7343	0.2710		
Mg	0.3560	16	0.0153	0.0038	0.0017	0.0013	0.0044	0.00874	0.4787	0.3517		
Cr	0.0208	16	0.0003	0.0001	0.0001	0.0001	0.0002	0.00035	0.5939	0.4427		
Zn	0.0486	15	0.0011	0.0003	0.0004	0.0003	0.0005	0.00103	0.7250	0.5366		
Ti	0.0238	17	0.0006	0.0001	0.0001	0.0004	0.0005	0.00095	0.3413	1.8770		
V	0.0104	16	0.0002	0.0001	0.0001	0.0001	0.0001	0.00026	0.7783	0.8243		
Ga	0.0189	11	0.0003	0.0001	0.0001	0.0001	0.0002	0.00042	0.6207	0.7886		
	mg/kg		mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg				
Ni	50.0	15	2.03	0.5241	0.6487	0.4180	0.9329	1.8657	1.2974	0.8360		
Be	4.72	13	0.39	0.1082	0.0302	0.0970	0.1484	0.2968	0.6401	2.0550		
Cd	9.60	10	0.22	0.0687	0.3925	0.3987	0.5637	1.1273	4.0882	4.1531		
Li	5.95	10	0.17	0.0550	0.3159	0.0235	0.3215	0.6430	5.3086	0.3957		
Pb	55.8	15	3.23	0.8340	0.2154	1.2255	1.4979	2.9959	0.3861	2.1963		
Sn	20.64	13	1.22	0.3373	0.2918	0.2589	0.5157	1.0313	1.4137	1.2542		
Zr	32.9	13	1.80	0.4980	0.3761	0.1902	0.6524	1.3049	1.1432	0.5781		
Ca	9.06	5	1.69	0.7558	0.2323	0.1131	0.7987	1.5975	2.5640	1.2486		
Hg	7.60	6	0.80	0.3282	0.2599	0.3334	0.5352	1.0704	3.4198	4.3867		
Na	3.71	4	0.63	0.3150	0.5220	0.0883	0.6160	1.2320	14.0688	2.3796		

The expanded uncertainties U are calculated by multiplication of u_{combined} with a coverage factor of $k = 2$ using Equation 4.

$$U = k \cdot u_{\text{combined}} \quad (4)$$

The calculated mass fractions and their resp. expanded uncertainties are given on Page 3 of this report. Rounding was done according to DIN 1333 [5].

In addition to the wet chemical characterisation an accompanying inter-laboratory comparison with spark emission was performed to check if there is agreement between SOES and wet chemistry. Tab. 35 shows the mean values of wet chemical and spark emission results as well as their standard deviations. The data of wet chemistry and SOES are consistent for all elements considering their uncertainties. The data from the spark emission inter-laboratory comparison was not used for the calculation of the certified values.

Tab. 35: Comparison wet chemistry (incl. XRF) vs. SOES

Element	Wet chemical analysis			Spark emission		
	Mass fraction in %	Std.-dev. in %	<i>n</i>	Mass fraction in %	Std.-dev. in %	<i>n</i>
Si	1.211	0.010	13	1.199	0.037	16
Fe	0.246	0.005	17	0.254	0.008	16
Cu	0.0908	0.0018	16	0.0909	0.0031	16
Mn	0.0985	0.0015	18	0.0984	0.0027	15
Mg	0.356	0.016	16	0.358	0.015	16
Cr	0.0208	0.0004	16	0.0208	0.0012	15
Zn	0.0486	0.0011	15	0.0467	0.0021	16
Ti	0.0238	0.0006	16	0.0236	0.0016	15
V	0.0104	0.0003	16	0.0106	0.0008	16
Ga	0.0189	0.0003	11	0.0191	0.0007	14
	in mg/kg	in mg/kg		in mg/kg	in mg/kg	
Ni	50.0	2.1	15	49.0	3.7	16
Be	4.72	0.39	13	4.90	0.36	14
Cd	9.60	0.22	10	9.1	1.9	14
Li	5.95	0.18	10	5.79	1.10	15
Pb	55.8	3.3	15	51.8	5.6	15
Sn	20.6	1.3	13	24.5	6.7	16
Zr	32.9	1.8	13	32.6	2.6	16
Ca	9.06	1.69	5	7.89	0.73	14
Hg	7.60	0.81	6	7.37	2.5	10
Na	3.71	0.63	4	2.80	0.45	14

6. Instructions for users and stability

The certified reference material BAM-M318 is intended for the calibration and quality control of spark emission and X-ray fluorescence spectrometers used for the analysis of similar materials. It is also suitable for wet chemical analysis.

The surface of the material should be cleaned by turning or milling before analysis.

If chips prepared from the compact material are used for wet chemical analysis, a minimum sample intake of 0.2 g has to be used.

The material will remain stable provided that it is not subjected to excessive heat (eg, during preparation of the working surface).

An area 8mm in diameter in the centre of the discs should be avoided for spark optical emission spectrometry.

7. Metrological Traceability

To ensure traceable of the certified mass fractions to the SI (Système International d'Unités) calibration was done using standard solutions prepared from pure metals or stoichiometric compounds or well checked commercial calibration solutions.

8. Information on and purchase of the CRM

Certified reference material BAM-M318 is supplied by

Bundesanstalt für Materialforschung und -prüfung (BAM)

Division 1.6 „Inorganic Reference Materials“

Richard-Willstätter-Str. 11, D-12489 Berlin, Germany

Phone +49 (0)30 - 8104 2061

Fax: +49 (0)30 - 8104 72061

E-Mail: sales.crm@bam.de

Each disc of BAM-M318 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:

<https://www.bam.de>.

Tel. +49 30 8104 1111.

9. References

- [1] ISO 17034, General requirements for the competence of reference material producers, 2016
- [2] ISO Guide 31, Reference materials - Contents of certificates, labels and accompanying documentation, 2015
- [3] ISO Guide 35, Reference materials - Guidance for characterization and assessment of homogeneity and stability, 2017
- [4] Bonas G, Zervou M, Papaeoannou T, Lees M: Accred Qual Assur (2003) 8:101-107
- [5] DIN 1333:1992-02 Zahlenangaben

Annex 1: Calculation of uncertainty contribution of potential inhomogeneity (length), SOES

Silicon:

<i>Sample</i>	<i>Number</i>	<i>Sum</i>	<i>Mean</i>	<i>Variance</i>		
A1	5	5.9425	1.1885	5.9966E-06		
A2	5	5.9064	1.1813	2.7665E-05		
A3	5	5.9058	1.1812	1.6073E-05		
A4	5	5.9131	1.1826	2.3714E-06		
A5	5	5.9206	1.1841	2.3404E-05		
B1	5	5.9187	1.1837	9.1637E-06		
B2	5	5.9094	1.1819	8.4629E-06		
B3	5	5.9061	1.1812	8.9719E-06		
B4	5	5.9616	1.1923	9.0903E-06		
B5	5	5.8954	1.1791	3.0223E-06		
C1	5	5.9073	1.1815	3.0321E-06		
C2	5	5.9146	1.1829	3.2914E-06		
C3	5	5.9928	1.1986	5.3076E-06		
C4	5	5.9322	1.1864	8.1576E-06		
C5	5	5.9135	1.1827	3.5155E-06		
D1	5	5.9733	1.1947	2.4536E-05		
D2	5	5.9125	1.1825	9.5767E-06		
D3	5	5.9213	1.1843	2.7881E-05		
D4	5	5.9066	1.1813	1.1683E-05		
D5	5	5.9096	1.1819	6.7844E-06		
E1	5	5.9462	1.1892	3.5009E-06		
E2	5	5.9373	1.1875	1.4668E-05		
E3	5	5.9328	1.1866	7.7876E-06		
E4	5	5.9211	1.1842	2.9218E-05		
E5	5	5.9190	1.1838	3.618E-05		
F1	5	5.9116	1.1823	4.399E-05		
F2	5	5.9144	1.1829	3.4333E-05		
F3	5	5.9142	1.1828	9.3399E-06		
F4	5	5.9132	1.1826	3.3358E-05		
F5	5	6.0135	1.2027	2.0853E-05		
			1.1852			
ANOVA						
<i>Source of variation</i>	<i>sums of squares (SS)</i>	<i>degrees of freedom (df)</i>	<i>Mean squares (MS)</i>	<i>F-value</i>	<i>P-value</i>	<i>critical F-value</i>
Between groups	0.004270704	29	0.000147266	9.7912584	4.4419E-20	1.56207098
Within groups	0.001804863	120	1.50405E-05			
Total	0.006075568	149				
within-sd	0.00387821					
effective n	4.00					
s_bb	0.00574946					
s_bb_min	0.00069673					
u_bb	0.00574946	5.7494597				
u_bb(rel.)	0.485086414					

Chromium:

Sample	Number	Sum	Mean	Variance		
A1	5	0.09897792	0.019795584	7.2372E-09		
A2	5	0.09877456	0.019754912	5.4796E-08		
A3	5	0.0990796	0.01981592	1.5508E-08		
A4	5	0.09938464	0.019876928	8.2711E-09		
A5	5	0.09846952	0.019693904	7.2372E-09		
B1	5	0.10131656	0.020263312	1.344E-08		
B2	5	0.09928296	0.019856592	2.8949E-08		
B3	5	0.09826616	0.019653232	2.3779E-08		
B4	5	0.09735104	0.019470208	3.1016E-09		
B5	5	0.09735104	0.019470208	1.344E-08		
C1	5	0.09968968	0.019937936	7.2372E-09		
C2	5	0.09877456	0.019754912	8.2711E-09		
C3	5	0.09816448	0.019632896	7.2372E-09		
C4	5	0.09938464	0.019876928	8.2711E-09		
C5	5	0.09938464	0.019876928	8.2711E-09		
D1	5	0.10019808	0.020039616	4.8592E-08		
D2	5	0.09724936	0.019449872	3.1016E-09		
D3	5	0.09938464	0.019876928	8.2711E-09		
D4	5	0.09836784	0.019673568	1.861E-08		
D5	5	0.09846952	0.019693904	2.7915E-08		
E1	5	0.10101152	0.020202304	1.2407E-08		
E2	5	0.0975544	0.01951088	5.1694E-09		
E3	5	0.09846952	0.019693904	7.2372E-09		
E4	5	0.09714768	0.019429536	1.7576E-08		
E5	5	0.09918128	0.019836256	4.8592E-08		
F1	5	0.10141824	0.020283648	2.3779E-08		
F2	5	0.099588	0.0199176	4.6525E-08		
F3	5	0.09796112	0.019592224	7.2372E-09		
F4	5	0.09867288	0.019734576	2.2745E-08		
F5	5	0.09826616	0.019653232	2.3779E-08		
			0.019777282			
ANOVA						
Source of variation	sums of squares (SS)	degrees of freedom (df)	Mean squares (MS)	F-value	P-value	critical F-value
Between groups	7.23449E-06	29	2.49465E-07	13.9473789	2.3662E-26	1.56207098
Within groups	2.14634E-06	120	1.78862E-08			
Total	9.38083E-06	149				
within-sd	0.0001337					
effective r	4.00					
s_bb	0.0002406					
s_bb_min	2.403E-05					
u_bb	0.0002406	0.2406132				
u_bb(rel.)	1.216614323					

Zirconium:

Sample	Number	Sum	Mean	Variance		
A1	5	0.01599448	0.003198896	2.135E-09		
A2	5	0.0160978	0.00321956	0		
A3	5	0.01620112	0.003240224	2.135E-09		
A4	5	0.0160978	0.00321956	0		
A5	5	0.0160978	0.00321956	0		
B1	5	0.0160978	0.00321956	0		
B2	5	0.01620112	0.003240224	2.135E-09		
B3	5	0.0160978	0.00321956	0		
B4	5	0.0160978	0.00321956	0		
B5	5	0.01599448	0.003198896	8.0063E-10		
C1	5	0.01599448	0.003198896	2.135E-09		
C2	5	0.01620112	0.003240224	8.0063E-10		
C3	5	0.0160978	0.00321956	0		
C4	5	0.0166144	0.00332288	0		
C5	5	0.0160978	0.00321956	0		
D1	5	0.0160978	0.00321956	0		
D2	5	0.01599448	0.003198896	2.135E-09		
D3	5	0.0166144	0.00332288	0		
D4	5	0.0160978	0.00321956	0		
D5	5	0.0160978	0.00321956	0		
E1	5	0.0160978	0.00321956	0		
E2	5	0.01599448	0.003198896	2.135E-09		
E3	5	0.0160978	0.00321956	0		
E4	5	0.0160978	0.00321956	0		
E5	5	0.01651108	0.003302216	2.135E-09		
F1	5	0.01651108	0.003302216	2.135E-09		
F2	5	0.0160978	0.00321956	0		
F3	5	0.01599448	0.003198896	2.135E-09		
F4	5	0.0160978	0.00321956	0		
F5	5	0.0160978	0.00321956	0		
			0.0032			
ANOVA						
Source of variation	sums of squares (SS)	degrees of freedom (df)	Mean squares (MS)	F-value	P-value	critical F-value
Between groups	1.78273E-07	29	6.14734E-09	8.85941645	1.8884E-18	1.56207098
Within groups	8.32652E-08	120	6.93876E-10			
Total	2.61538E-07	149				
within-sd	2.634E-05					
effective n	4.00					
s_bb	3.692E-05					
s_bb_min	4.732E-06					
u_bb	3.692E-05	0.0369238				
u_bb(rel.)	1.143189508					

Annex 2: Calculation of uncertainty contribution of potential inhomogeneity (area)

Silicon:

Si	outer region	1.170	1.183	1.185	1.177	1.179	1.174	1.181	1.172
	inner region	1.164	1.178	1.169	1.158	1.160	1.182	1.167	1.173
	centre	1.175	1.169	1.168	1.183	1.154			
	<i>Sample</i>	<i>Number</i>	<i>Sum</i>	<i>Mean</i>	<i>Variance</i>				
	outer region	8	9.421	1.177625	2.85536E-05				
	inner region	8	9.351	1.168875	7.09821E-05				
	centre	5	5.849	1.1698	0.0001137				
				1.1721					
	ANOVA								
	<i>Source of variation</i>	<i>sums of squares (SS)</i>	<i>degrees of freedom (df)</i>	<i>Mean squares (MS)</i>	<i>F-value</i>	<i>P-value</i>	<i>critical F-value</i>		
	Between groups	0.000351593	2	0.000175796	2.747892592	0.0909	3.5545571		
	Within groups	0.00115155	18	6.3975E-05					
	Total	0.001503143	20						
	within-sd	0.0079984							
	effective n	4.00							
	s_bb	0.0052873							
	s_bb_min	0.0023089							
	u_bb	0.0052873							
	u_bb(rel.)	0.451094837							

Iron:

Fe	outer region	0.26	0.262	0.263	0.26	0.258	0.258	0.259	0.261
	inner region	0.259	0.259	0.256	0.255	0.257	0.257	0.257	0.258
	centre	0.254	0.256	0.256	0.259	0.253			
	<i>Sample</i>	<i>Number</i>	<i>Sum</i>	<i>Mean</i>	<i>Variance</i>				
	outer region	8	2.081	0.260125	3.26786E-06				
	inner region	8	2.058	0.25725	1.92857E-06				
	centre	5	1.278	0.2556	5.3E-06				
				0.257658333					
	ANOVA								
	<i>Source of variation</i>	<i>sums of squares (SS)</i>	<i>degrees of freedom (df)</i>	<i>Mean squares (MS)</i>	<i>F-value</i>	<i>P-value</i>	<i>critical F-value</i>		
	Between groups	6.93774E-05	2	3.46887E-05	10.84492277	0.00081	3.5545571		
	Within groups	5.7575E-05	18	3.19861E-06					
	Total	0.000126952	20						
	within-sd	0.0017885							
	effective n	4.00							
	s_bb	0.0028058							
	s_bb_min	0.0005163							
	u_bb	0.0028058							
	u_bb(rel.)	1.088961909							

Copper:

Cu	outer region	0.09	0.09	0.092	0.091	0.092	0.091	0.092	0.091
	inner region	0.091	0.092	0.091	0.09	0.09	0.092	0.091	0.091
	centre	0.09	0.089	0.089	0.091	0.089			
	<i>Sample</i>	<i>Number</i>	<i>Sum</i>	<i>Mean</i>	<i>Variance</i>				
	outer region	8	0.729	0.091125	6.96429E-07				
	inner region	8	0.728	0.091	5.71429E-07				
	centre	5	0.448	0.0896	8E-07				
				0.090575					
	ANOVA								
	<i>Source of variation</i>	<i>sums of squares (SS)</i>	<i>degrees of freedom (df)</i>	<i>Mean squares (MS)</i>	<i>F-value</i>	<i>P-value</i>	<i>critical F-value</i>		
	Between groups	8.21071E-06	2	4.10536E-06	6.119787045	0.00938	3.5545571		
	Within groups	0.000012075	18	6.70833E-07					
	Total	2.02857E-05	20						
	within-sd	0.000819							
	effective n	4.00							
	s_bb	0.0009266							
	s_bb_min	0.0002364							
	u_bb	0.0009266							
	u_bb(rel.)	1.023045448							

Manganese:

Mn	outer region	0.077	0.078	0.078	0.077	0.077	0.077	0.077	0.077
	inner region	0.078	0.078	0.077	0.077	0.077	0.077	0.077	0.077
	centre	0.077	0.077	0.077	0.077	0.076			
	<i>Sample</i>	<i>Number</i>	<i>Sum</i>	<i>Mean</i>	<i>Variance</i>				
	outer region	8	0.618	0.07725	2.14286E-07				
	inner region	8	0.618	0.07725	2.14286E-07				
	centre	5	0.384	0.0768	0.0000002				
				0.0771					
	ANOVA								
	<i>Source of variation</i>	<i>sums of squares (SS)</i>	<i>degrees of freedom (df)</i>	<i>Mean squares (MS)</i>	<i>F-value</i>	<i>P-value</i>	<i>critical F-value</i>		
	Between groups	7.71429E-07	2	3.85714E-07	1.827067669	0.18949	3.5545571		
	Within groups	3.8E-06	18	2.11111E-07					
	Total	4.57143E-06	20						
	within-sd	0.0004595							
	effective n	4.00							
	s_bb	0.0002089							
	s_bb_min	0.0001326							
	u_bb	0.0002089							
	u_bb(rel.)	0.27098278							

Magnesium:

Mg	outer region	0.365	0.365	0.363	0.367	0.365	0.365	0.365	0.368
	inner region	0.366	0.368	0.367	0.366	0.368	0.368	0.368	0.368
	centre	0.369	0.366	0.367	0.367	0.365			
	<i>Sample</i>	<i>Number</i>	<i>Sum</i>	<i>Mean</i>	<i>Variance</i>				
	outer region	8	2.923	0.365375	2.26786E-06				
	inner region	8	2.939	0.367375	8.39286E-07				
	centre	5	1.834	0.3668	0.0000022				
				0.366516667					
	ANOVA								
	<i>Source of variation</i>	<i>sums of squares (SS)</i>	<i>degrees of freedom (df)</i>	<i>Mean squares (MS)</i>	<i>F-value</i>	<i>P-value</i>	<i>critical F-value</i>		
	Between groups	1.66881E-05	2	8.34405E-06	4.916296469	0.01979	3.5545571		
	Within groups	3.055E-05	18	1.69722E-06					
	Total	4.72381E-05	20						
	within-sd	0.0013028							
	effective n	4.00							
	s_bb	0.0012891							
	s_bb_min	0.0003761							
	u_bb	0.0012891							
	u_bb(rel.)	0.351708943							

Chromium:

Cr	outer region	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02
	inner region	0.02	0.02	0.02	0.02	0.019	0.02	0.02	0.02
	centre	0.02	0.02	0.02	0.02	0.019			
	<i>Sample</i>	<i>Number</i>	<i>Sum</i>	<i>Mean</i>	<i>Variance</i>				
	outer region	8	0.16	0.02	0				
	inner region	8	0.159	0.019875	0.000000125				
	centre	5	0.099	0.0198	0.0000002				
				0.019891667					
	ANOVA								
	<i>Source of variation</i>	<i>sums of squares (SS)</i>	<i>degrees of freedom (df)</i>	<i>Mean squares (MS)</i>	<i>F-value</i>	<i>P-value</i>	<i>critical F-value</i>		
	Between groups	1.34524E-07	2	6.72619E-08	0.722814499	0.49895	3.5545571		
	Within groups	0.000001675	18	9.30556E-08					
	Total	1.80952E-06	20						
	within-sd	0.0003051							
	effective n	4.00							
	s_bb	0							
	s_bb_min	8.806E-05							
	u_bb	8.806E-05							
	u_bb(rel.)	0.442699832							

Zinc:

Zn	outer region	0.049	0.048	0.048	0.049	0.048	0.049	0.047	0.049
	inner region	0.048	0.047	0.048	0.048	0.05	0.049	0.047	0.049
	centre	0.049	0.049	0.049	0.049	0.047			
	<i>Sample</i>	<i>Number</i>	<i>Sum</i>	<i>Mean</i>	<i>Variance</i>				
	outer region	8	0.387	0.048375	5.53571E-07				
	inner region	8	0.386	0.04825	1.07143E-06				
	centre	5	0.243	0.0486	8E-07				
				0.048408333					
	ANOVA								
	<i>Source of variation</i>	<i>sums of squares (SS)</i>	<i>degrees of freedom (df)</i>	<i>Mean squares (MS)</i>	<i>F-value</i>	<i>P-value</i>	<i>critical F-value</i>		
	Between groups	3.77381E-07	2	1.8869E-07	0.23303112	0.79448	3.5545571		
	Within groups	0.000014575	18	8.09722E-07					
	Total	1.49524E-05	20						
	within-sd	0.0008998							
	effective n	4.00							
	s_bb	0							
	s_bb_min	0.0002598							
	u_bb	0.0002598							
	u_bb(rel.)	0.536608164							

Titanium:

Ti	outer region	0.023	0.023	0.022	0.022	0.022	0.023	0.022	0.022
	inner region	0.022	0.021	0.022	0.022	0.022	0.022	0.021	0.022
	centre	0.022	0.022	0.022	0.022	0.021			
	<i>Sample</i>	<i>Number</i>	<i>Sum</i>	<i>Mean</i>	<i>Variance</i>				
	outer region	8	0.179	0.022375	2.67857E-07				
	inner region	8	0.174	0.02175	2.14286E-07				
	centre	5	0.109	0.0218	2E-07				
				0.021975					
	ANOVA								
	<i>Source of variation</i>	<i>sums of squares (SS)</i>	<i>degrees of freedom (df)</i>	<i>Mean squares (MS)</i>	<i>F-value</i>	<i>P-value</i>	<i>critical F-value</i>		
	Between groups	0.000001825	2	9.125E-07	3.934131737	0.03824	3.5545571		
	Within groups	4.175E-06	18	2.31944E-07					
	Total	6E-06	20						
	within-sd	0.0004816							
	effective n	4.00							
	s_bb	0.0004125							
	s_bb_min	0.000139							
	u_bb	0.0004125							
	u_bb(rel.)	1.877037341							

Gallium:

Ga	outer region	142.1	140.4	142.3	142.5	141.2	141.3	141.6	143.4
	inner region	140.6	141.6	141.2	142	143.1	143.4	141.1	142.9
	centre	140.2	139.3	140.4	140.9	140.1			
	<i>Sample</i>	<i>Number</i>	<i>Sum</i>	<i>Mean</i>	<i>Variance</i>				
	outer region	8	1134.8	141.85	0.854285714				
	inner region	8	1135.9	141.9875	1.078392857				
	centre	5	700.9	140.18	0.337				
				141.3391667					
	ANOVA								
	<i>Source of variation</i>	<i>sums of squares (SS)</i>	<i>degrees of freedom (df)</i>	<i>Mean squares (MS)</i>	<i>F-value</i>	<i>P-value</i>	<i>critical F-value</i>		
	Between groups	11.59277381	2	5.796386905	7.013290153	0.0056	3.5545571		
	Within groups	14.87675	18	0.826486111					
	Total	26.46952381	20						
	within-sd	0.9091128							
	effective n	4.00							
	s_bb	1.1146637							
	s_bb_min	0.2624383							
	u_bb	1.1146637							
	u_bb(rel.)	0.788644607							

Nickel:

Ni	outer region	0.0046	0.0046	0.0046	0.0046	0.0044	0.0045	0.0044	0.0046
	inner region	0.0044	0.0043	0.0044	0.0044	0.0046	0.0046	0.0042	0.0046
	centre	0.0045	0.0045	0.0046	0.0047	0.0043			
	<i>Sample</i>	<i>Number</i>	<i>Sum</i>	<i>Mean</i>	<i>Variance</i>				
	outer region	8	0.0363	0.0045375	8.39286E-09				
	inner region	8	0.0355	0.0044375	2.26786E-08				
	centre	5	0.0226	0.00452	0.000000022				
				0.004498333					
	ANOVA								
	<i>Source of variation</i>	<i>sums of squares (SS)</i>	<i>degrees of freedom (df)</i>	<i>Mean squares (MS)</i>	<i>F-value</i>	<i>P-value</i>	<i>critical F-value</i>		
	Between groups	4.40238E-08	2	2.20119E-08	1.296937105	0.29772	3.5545571		
	Within groups	3.055E-07	18	1.69722E-08					
	Total	3.49524E-07	20						
	within-sd	0.0001303							
	effective n	4.00							
	s_bb	3.55E-05							
	s_bb_min	3.761E-05							
	u_bb	3.761E-05							
	u_bb(rel.)	0.836040079							

Beryllium:

Be	outer region	5.04	5.01	5.02	5	4.97	4.98	4.99	4.97
	inner region	4.9	4.91	4.9	4.87	4.88	4.88	4.89	4.89
	centre	4.86	4.84	4.86	4.88	4.79			
	<i>Sample</i>	<i>Number</i>	<i>Sum</i>	<i>Mean</i>	<i>Variance</i>				
	outer region	8	39.98	4.9975	0.000621429				
	inner region	8	39.12	4.89	0.000171429				
	centre	5	24.23	4.846	0.00118				
				4.911166667					
	ANOVA								
	<i>Source of variation</i>	<i>sums of squares (SS)</i>	<i>degrees of freedom (df)</i>	<i>Mean squares (MS)</i>	<i>F-value</i>	<i>P-value</i>	<i>critical F-value</i>		
	Between groups	0.082625238	2	0.041312619	72.40770622	2.5E-09	3.5545571		
	Within groups	0.01027	18	0.000570556					
	Total	0.092895238	20						
	within-sd	0.0238863							
	effective n	4.00							
	s_bb	0.1009233							
	s_bb_min	0.0068954							
	u_bb	0.1009233							
	u_bb(rel.)	2.054976417							

Cadmium:

Cd	outer region	7.66	8.35	5.87	8.41	7.41	9.67	6.99	6.27
	inner region	6.17	6.69	9.61	8.06	7.32	8.31	7.77	7.81
	centre	8.07	7.48	5.87	7.3	7.39			
	<i>Sample</i>	<i>Number</i>	<i>Sum</i>	<i>Mean</i>	<i>Variance</i>				
	outer region	8	60.63	7.57875	1.524783929				
	inner region	8	61.74	7.7175	1.095678571				
	centre	5	36.11	7.222	0.66197				
				7.506083333					
	ANOVA								
	<i>Source of variation</i>	<i>sums of squares (SS)</i>	<i>degrees of freedom (df)</i>	<i>Mean squares (MS)</i>	<i>F-value</i>	<i>P-value</i>	<i>critical F-value</i>		
	Between groups	0.768749167	2	0.384374583	0.329603343	0.72346	3.5545571		
	Within groups	20.9911175	18	1.166173194					
	Total	21.75986667	20						
	within-sd	1.079895							
	effective n	4.00							
	s_bb	0							
	s_bb_min	0.3117388							
	u_bb	0.3117388							
	u_bb(rel.)	4.153149098							

Lithium:

Li	outer region	6.8	6.65	6.47	6.82	6.82	6.83	6.81	6.84
	inner region	6.79	6.75	6.69	6.81	6.76	6.74	6.82	6.67
	centre	6.67	6.63	6.73	6.75	6.78			
	<i>Sample</i>	<i>Number</i>	<i>Sum</i>	<i>Mean</i>	<i>Variance</i>				
	outer region	8	54.04	6.755	0.016942857				
	inner region	8	54.03	6.75375	0.002883929				
	centre	5	33.56	6.712	0.00372				
				6.74025					
	ANOVA								
	<i>Source of variation</i>	<i>sums of squares (SS)</i>	<i>degrees of freedom (df)</i>	<i>Mean squares (MS)</i>	<i>F-value</i>	<i>P-value</i>	<i>critical F-value</i>		
	Between groups	0.006846786	2	0.003423393	0.401002629	0.67548	3.5545571		
	Within groups	0.1536675	18	0.008537083					
	Total	0.160514286	20						
	within-sd	0.0923963							
	effective n	4.00							
	s_bb	0							
	s_bb_min	0.0266725							
	u_bb	0.0266725							
	u_bb(rel.)	0.39572012							

Lead:

Pb	outer region	58	59.6	62.6	58.9	55.5	55.1	58	56.3
	inner region	62.6	57.7	58.6	52.5	53.1	52.3	56.1	54.6
	centre	59.3	59.5	60.3	61	54.2			
	<i>Sample</i>	<i>Number</i>	<i>Sum</i>	<i>Mean</i>	<i>Variance</i>				
	outer region	8	464	58	6.011428571				
	inner region	8	447.5	55.9375	12.78553571				
	centre	5	294.3	58.86	7.243				
				57.59916667					
	ANOVA								
	<i>Source of variation</i>	<i>sums of squares (SS)</i>	<i>degrees of freedom (df)</i>	<i>Mean squares (MS)</i>	<i>F-value</i>	<i>P-value</i>	<i>critical F-value</i>		
	Between groups	30.64163095	2	15.32081548	1.717679167	0.20762	3.5545571		
	Within groups	160.55075	18	8.919486111					
	Total	191.192381	20						
	within-sd	2.9865509							
	effective n	4.00							
	s_bb	1.2650424							
	s_bb_min	0.862143							
	u_bb	1.2650424							
	u_bb(rel.)	2.196285988							

Zirconium:

Zr	outer region	34.93	34.52	34.52	33.53	33.69	34.51	34.39	33.58
	inner region	34.23	34.48	34.49	33.63	32.74	33.26	33.37	34.6
	centre	34.86	34.88	34.9	34.72	32.88			
	<i>Sample</i>	<i>Number</i>	<i>Sum</i>	<i>Mean</i>	<i>Variance</i>				
	outer region	8	273.67	34.20875	0.280383929				
	inner region	8	270.8	33.85	0.481771429				
	centre	5	172.24	34.448	0.77332				
				34.16891667					
	ANOVA								
	<i>Source of variation</i>	<i>sums of squares (SS)</i>	<i>degrees of freedom (df)</i>	<i>Mean squares (MS)</i>	<i>F-value</i>	<i>P-value</i>	<i>critical F-value</i>		
	Between groups	1.182413452	2	0.591206726	1.262607625	0.30681	3.5545571		
	Within groups	8.4283675	18	0.468242639					
	Total	9.610780952	20						
	within-sd	0.6842826							
	effective n	4.00							
	s_bb	0.1753312							
	s_bb_min	0.1975354							
	u_bb	0.1975354							
	u_bb(rel.)	0.578114213							

Calcium:

Ca	outer region	13.26	13.36	13.4	13.39	13.51	13.52	13.42	13.4
	inner region	13.66	13.7	13.51	13.62	13.33	13.73	13.66	13.75
	centre	13.7	13.55	13.67	13.7	13.63			
	<i>Sample</i>	<i>Number</i>	<i>Sum</i>	<i>Mean</i>	<i>Variance</i>				
	outer region	8	107.26	13.4075	0.006821429				
	inner region	8	108.96	13.62	0.019257143				
	centre	5	68.25	13.65	0.00395				
				13.55916667					
	ANOVA								
	<i>Source of variation</i>	<i>sums of squares (SS)</i>	<i>degrees of freedom (df)</i>	<i>Mean squares (MS)</i>	<i>F-value</i>	<i>P-value</i>	<i>critical F-value</i>		
	Between groups	0.251345238	2	0.125672619	11.40462386	0.00063	3.5545571		
	Within groups	0.19835	18	0.011019444					
	Total	0.449695238	20						
	within-sd	0.1049735							
	effective n	4.00							
	s_bb	0.1693024							
	s_bb_min	0.0303033							
	u_bb	0.1693024							
	u_bb(rel.)	1.248619311							

Mercury:

Hg	outer region	6	4.7	7	7.275	6	5.5	7	7.897
	inner region	5	5.7	6	5.8	7	7.122	6	7.8
	centre	6	5.4	6	7.0433				
	<i>Sample</i>	<i>Number</i>	<i>Sum</i>	<i>Mean</i>	<i>Variance</i>				
	outer region	8	51.372	6.4215	1.106133714				
	inner region	8	50.422	6.30275	0.842231929				
	centre	4	24.4433	6.110825	0.466448723				
				6.278358333					
	ANOVA								
	<i>Source of variation</i>	<i>sums of squares (SS)</i>	<i>degrees of freedom (df)</i>	<i>Mean squares (MS)</i>	<i>F-value</i>	<i>P-value</i>	<i>critical F-value</i>		
	Between groups	0.258491658	2	0.129245829	0.146109381	0.86514	3.5915306		
	Within groups	15.03790567	17	0.884582686					
	Total	15.29639733	19						
	within-sd	0.9405226							
	effective n	4.00							
	s_bb	0							
	s_bb_min	0.275413							
	u_bb	0.275413							
	u_bb(rel.)	4.386704611							

Sodium:

Na	outer region	3.17	3.13	3.07	3.13	3.14	3.17	3.04	3.19
	inner region	3.15	3.14	3.13	3.21	3.13	3.24	3.14	3.31
	centre	3.27	3.22	3.33	3.32	3.17			
	<i>Sample</i>	<i>Number</i>	<i>Sum</i>	<i>Mean</i>	<i>Variance</i>				
	outer region	8	25.04	3.13	0.002657143				
	inner region	8	25.45	3.18125	0.004355357				
	centre	5	16.31	3.262	0.00457				
				3.191083333					
	ANOVA								
	<i>Source of variation</i>	<i>sums of squares (SS)</i>	<i>degrees of freedom (df)</i>	<i>Mean squares (MS)</i>	<i>F-value</i>	<i>P-value</i>	<i>critical F-value</i>		
	Between groups	0.053613452	2	0.026806726	7.162520079	0.00515	3.5545571		
	Within groups	0.0673675	18	0.003742639					
	Total	0.120980952	20						
	within-sd	0.0611771							
	effective n	4.00							
	s_bb	0.0759343							
	s_bb_min	0.0176603							
	u_bb	0.0759343							
	u_bb(rel.)	2.379578275							