Certification Report

Certified Reference Material

BAM-M308a

AlZnMgCu1,5

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Summary

This report describes preparation, analysis and certification of the aluminium alloy reference material BAM-M308a.
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	0.072	0.003
Fe	0.164	0.005
Cu	1.36	0.03
Mn	0.0343	0.0005
Mg	2.28	0.05
Cr	0.192	0.004
Zn	5.61	0.08
	in mg/kg	in mg/kg
Ni	147	3
Ti	257	7
Ag	6.5	0.6
Be	1.8	0.1
Na	15.8	2.2
Pb	43.6	2.7
Zr	87.3	2.6

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 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 ten laboratories which participated in the certification inter-laboratory comparison. In addition to the elements mentioned above. The mass fraction of Ca is given for information.

Content

Page

List of abbreviations 5

1. Introduction........ 6

2. Companies/laboratories involved 6

3. Candidate material 7

4. Homogeneity testing........ 7

5. Characterisation study........ 9

   5.1 Analytical methods 9
   5.2 Analytical results and statistical evaluation........ 10

6. Instructions for users and stability........ 32

7. References 33

8. Information on and purchase of the CRM........ 33

Annex 1: Calculation of uncertainty contribution of potential inhomogeneity (length), SOES 34

Annex 2: Calculation of uncertainty contribution of potential inhomogeneity (area) 49

List of abbreviations
(if not explained elsewhere)

CRM	certified 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
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 – 16)
I(R)	ICP-OES, revised value (Tables 2 – 16)
IMS	ICP-MS (Tables 2 – 16)
IMS(R)	ICP-MS, revised value (Tables 2 – 16)
A	FAAS (Tables 2 – 16)
P	spectrophotometry (Tables 2 – 16)
XRF	X-ray fluorescence spectrometry (Tables 2 – 16)
XRF(R)	X-ray fluorescence spectrometry, revised value (Tables 2 – 16)
-s	dissolution in acid (Tables 2 – 16)
-a	dissolution in base (Tables 2 – 16)

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-M308a is based on the aluminium alloy AlZnMgCu1,5. It replaces the out of stock CRM BAM-308.

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 the relevant ISO-Guides [1-3], the „Guidelines for the development and 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

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:

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
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 casted 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).

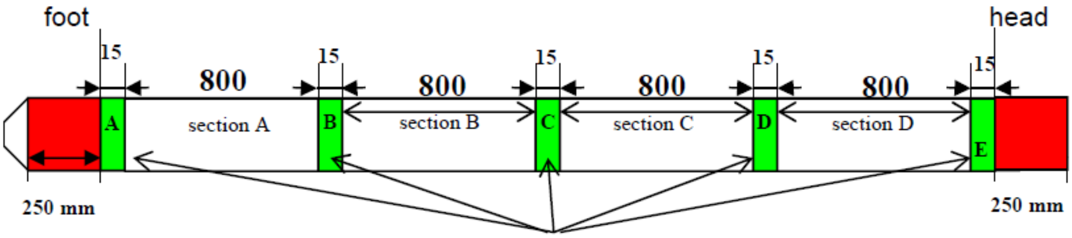


Fig.1: Preparation of the rods casted

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 volatize 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):

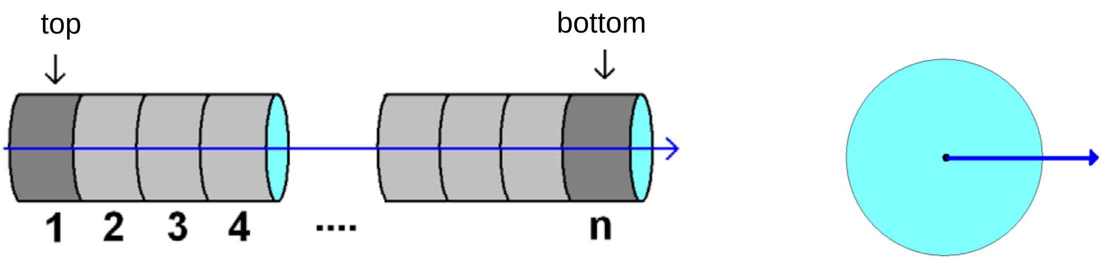


Fig. 2: Axial and radial composition gradient

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 [4] using Eq. (1) and Eq. (2):

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

$$u^{*}_{\text{bb}} = \sqrt{\frac{MS_{\text{within}}}{n}} \sqrt[4]{\frac{2}{N(n-1)}} \qquad (2)$$

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 $u_{\text{bb}}(1)$. 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: 1 spark, Pb: outer circle: 8 sparks, inner circle: 8 sparks; centre: 1 spark).

The analyte-specific within-disc uncertainty component $u_{\text{bb}}(2)$ 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 is different for the centre, the inner and the outer circle. For technical reasons, at r_0 (centre) only one measurement is possible. An ANOVA requires a minimum of 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 where 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 component for the radius of the disc is obtained. From these values, a combined inhomogeneity component is calculated. This component is compared with the within standard deviation calculated from the ANOVA-data. The higher component is used for the uncertainty calculation.

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

5. Characterisation study

5.1 Analytical methods

Ten laboratories participated in the certification inter-laboratory comparison. 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, Ni, Ti, Pb, Ag, Be, Ca, Na, Zr	0.5 g	Dissolution with NaOH	ICP-OES, commercial mono-element solutions
2	Si, Fe, Cu, Mn, Mg, Zn	0.5 g	Dissolution with NaOH	ICP-OES, calibration with pure metals or pure chemicals, matrix matching with pure Al (5N5)
	Zr, Pb	0.5 g	Dissolution with HNO_3/HF	ICP-MS, calibration with pure chemicals, matrix matching with pure Al (5N5)
	Ag	0.5 g	Dissolution with HNO_3/HF	ICP-MS, commercial mono-element solution (Merck certipur), matrix matching with pure Al (5N5)
	Cr, Ni, Ti, Ag, Be, Ca, Na	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 solutions
	Fe, Cu, Mn, Mg, Cr, Zn, Ni, Ti, Pb, Be, Ca, Na, Zr	0.5 g	Dissolution with HCl	ICP-OES, commercial mono-element solutions
	Ag	0.5 g	Dissolution with HNO_3	ICP-OES, commercial mono-element solutions
5	Si, Fe, Cu, Mn, Mg, Cr, Ni, Zn, Ti, Pb, Sn, Ga, Be, Cd, Co, V, Zr	0.5 g	Dissolution with NaOH	ICP-OES, commercial mono-element solutions (NIST)
6	Si, Fe, Cu, Mn, Cr, Ni, Ti, Pb, Be, Zr	0.5 g	Dissolution with NaOH	ICP-OES, calibration with pure metals or pure chemicals
	Mg, Zn, Ag, Ca, Na, Zr	0.5 g	Dissolution with HNO_3	ICP-OES, calibration with pure metals or pure chemicals
7	Si, Fe, Cu, Mn, Mg, Cr, Ni, Ti, Zr	1 g	Dissolution with HNO_3/HCl/HF	ICP-MS, calibration with matrix matched standards, commercial multi-element standard solutions
	Ag, Be, Ca, Pb	1 g	Dissolution with HNO_3/HF	ICP-MS, with matrix matched standards, commercial mono-element standard solutions (Perkin Elmer)
	Zn	0.2 g	Dissolution with HCl/H_2O_2	FAAS with matrix matched standards, commercial multi-element standard solution

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

7	Na	1 g	Dissolution with HNO_3/HF	FAAS, with matrix matched standards, commercial mono-element standard solution (Merck)
8	Si, Fe, Cu, Mn, Mg, Cr, Zn, Ni, Ti, Ag, Be, Zr	0.5 g	Dissolution with NaOH	ICP-OES with matrix matched standards, commercial mono-element solutions (Merck)
	Si, Fe, Cu, Mn, Mg, Cr, Zn, Ni, Ti, Ag, Be, Na, Pb, Zr	0.5 g	Dissolution with HCl/H_2O_2	ICP-OES with matrix matched standards, commercial mono-element solutions (Merck)
	Si, Fe, Cu, Mn, Cr, Zn, Ni, Ti, Ag, Pb, Zr			XRF, calibration with BAM-CRMs
9	Si	0.25 g	Dissolution with NaOH	Spectrophotometry
	Fe	0.5 g	Dissolution with HCl/H_2O_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, Mn, Cr, Ni, Ti, Be, Zr	0.5 g	Dissolution with HCl/HNO_3	ICP-OES, calibration with matrix matched standards, commercial mono-element solutions (Merck)
	Si, Fe, Cu, Mn, Mg, Zn, Ni. Ti, Be, Zr	0.5 g	Dissolution with NaOH	ICP-OES, calibration with matrix matched standards, commercial mono-element solutions (Merck)
	Pb, Ni	0.25 g	Dissolution with NaOH	ETAAS, calibration with commercial solution (Merck)
	Be, Pb, Ni	0.25 g	Dissolution with HCl	ETAAS, calibration with commercial solution (Merck)
10	Si, Fe, Cu, Mn, Mg, Cr, Zn, Ni, Ti, Ag, Be, Pb, Zr	0.25 g	Dissolution with NaOH	ICP-OES, calibration with commercial mono-element solutions
11	Si, Fe, Cu, Mn, Mg, Zn, Ni. Ti,	0.5 g	Dissolution with NaOH	ICP-OES, commercial mono-element solutions
	Si, Fe, Cu, Mn, Mg, Cr, Zn, Ni, Ti, Ag, Be, N	1 g	Dissolution with HCl/HNO_3	ICP-OES, commercial mono-element solutions
	Pb, Zr	1 g	Dissolution with HCl/HNO_3	ICP-MS, commercial mono-element solutions
	Fe, Mg, Ti, Na	1 g	Dissolution with HCl	FAAS, commercial mono-element solutions

5.2 Analytical results and statistical evaluation

The analytical results of the certification inter-laboratory comparison are listed in Tables 2 to 16. 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 ($\overline{s_i}$) 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.	6/I-a	8/XRF	9/P	7/I-s	9/I-a	2/I-a	11/I-a	4/P	5/I-a	10/I-a	1/I-a	8/I-a		
M_i [%]	0.067	0.069	0.0701	0.073	0.071	0.073	0.071	0.074	0.073	0.075	0.075	0.083		*n*
	0.066	0.068	0.0695	0.070	0.071	0.071	0.073	0.072	0.075	0.073	0.076	0.082		12
	0.067	0.069	0.0695	0.069	0.070	0.071	0.072	0.072	0.072	0.075	0.076	0.074		
	0.066	0.069	0.0690	0.071	0.071	0.070	0.071	0.073	0.073	0.075	0.075	0.073		
	0.068	0.069	0.0681	0.070	0.071	0.072	0.072	0.074	0.074	0.073	0.075	0.072		
	0.066	0.069	0.0691	0.068	0.072	0.070	0.073	0.074	0.073	0.074	0.074	0.073		
			0.0695											
			0.0688											
***M* [%]**	**0.067**	**0.069**	**0.069**	**0.070**	**0.071**	**0.071**	**0.072**	**0.073**	**0.073**	**0.074**	**0.075**	**0.076**		**0.072**
s [%]	0.0009	0.0005	0.0006	0.0017	0.0007	0.0010	0.0008	0.0010	0.0009	0.0008	0.0008	0.0050	s_M [%]	0.0028
													$\bar{s}_i$ [%]	0.0019
s_{rel}	0.01288	0.00695	0.00838	0.02410	0.01023	0.01335	0.01154	0.01344	0.01245	0.01043	0.01001	0.06540		0.03890

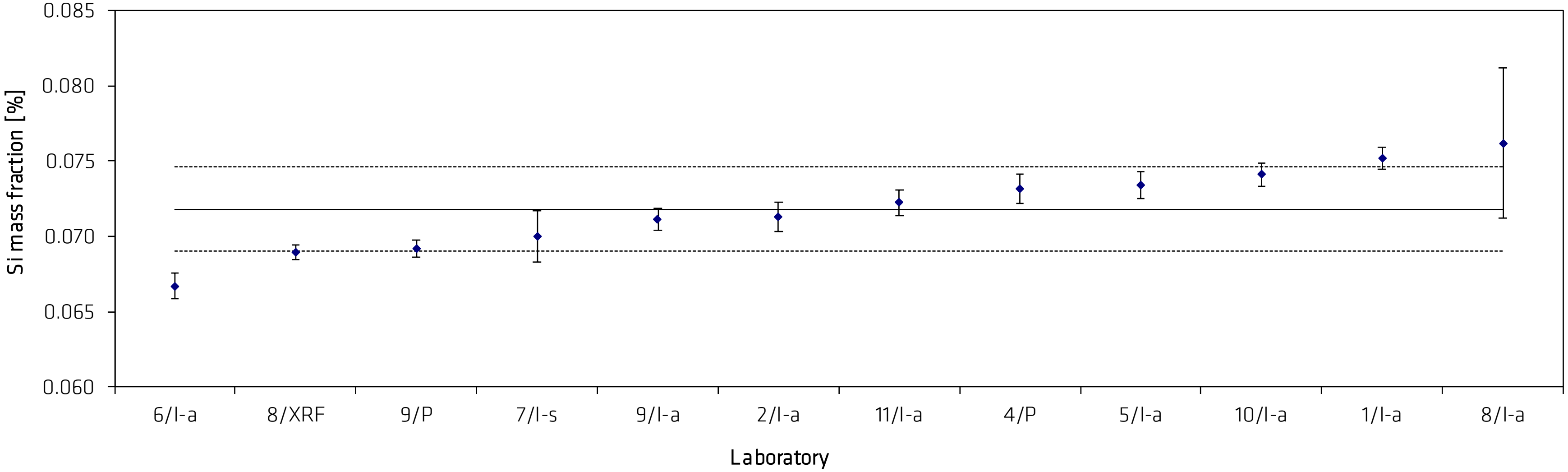


Table 3: Results for Fe

Lab./Meth.	9/I-s	6/I-a(R)	8/I-a	7/I-s	9/I-a	8/I-s	9/P	2/I-a	4/I-s	11/I-a	5/I-a	1/I-a	11/A-s	8/XRF	11/I-s	10/I-a		
M_i [%]	0.1662	0.1604	0.1624	0.1630	0.1635	0.1630	0.1636	0.1657	0.164	0.1646	0.1644	0.1660	0.168	0.1682	0.1694	0.1670		*n*
	0.1578	0.1597	0.1655	0.1630	0.1635	0.1633	0.1642	0.1635	0.164	0.1643	0.1654	0.1660	0.169	0.1681	0.1666	0.1670		16
	0.1581	0.1588	0.1612	0.1626	0.1625	0.1625	0.1621	0.1633	0.165	0.1644	0.1639	0.1640	0.166	0.1679	0.1667	0.1710		
	0.1596	0.1614	0.1618	0.1622	0.1653	0.1636	0.1666	0.1638	0.164	0.1636	0.1648	0.1650	0.169	0.1679	0.1702	0.1740		
	0.1584	0.1617	0.1593	0.1619	0.1632	0.1634	0.1638	0.1635	0.165	0.1641	0.1642	0.1640	0.168	0.1679	0.1682	0.1690		
	0.1550	0.1626	0.1602	0.1626	0.1608	0.1644	0.1642	0.1639	0.163	0.1647	0.1651	0.1640	0.166	0.1677	0.1685	0.1700		
							0.1619											
							0.1651											
***M* [%]**	**0.1592**	**0.1608**	**0.1617**	**0.1626**	**0.1631**	**0.1634**	**0.1639**	**0.1640**	**0.1642**	**0.1643**	**0.1646**	**0.1648**	**0.1676**	**0.1679**	**0.1683**	**0.1697**		**0.1644**
s [%]	0.0038	0.0014	0.0022	0.0004	0.0015	0.0006	0.0015	0.0009	0.0008	0.0004	0.0006	0.0010	0.0012	0.0002	0.0014	0.0027	s_M [%]	0.0028
																	$\bar{s}_i$ [%]	0.0007
s_{rel}	0.02361	0.00870	0.01332	0.00269	0.00901	0.00392	0.00930	0.00537	0.00459	0.00242	0.00357	0.00596	0.00719	0.00105	0.00854	0.01567		0.01718

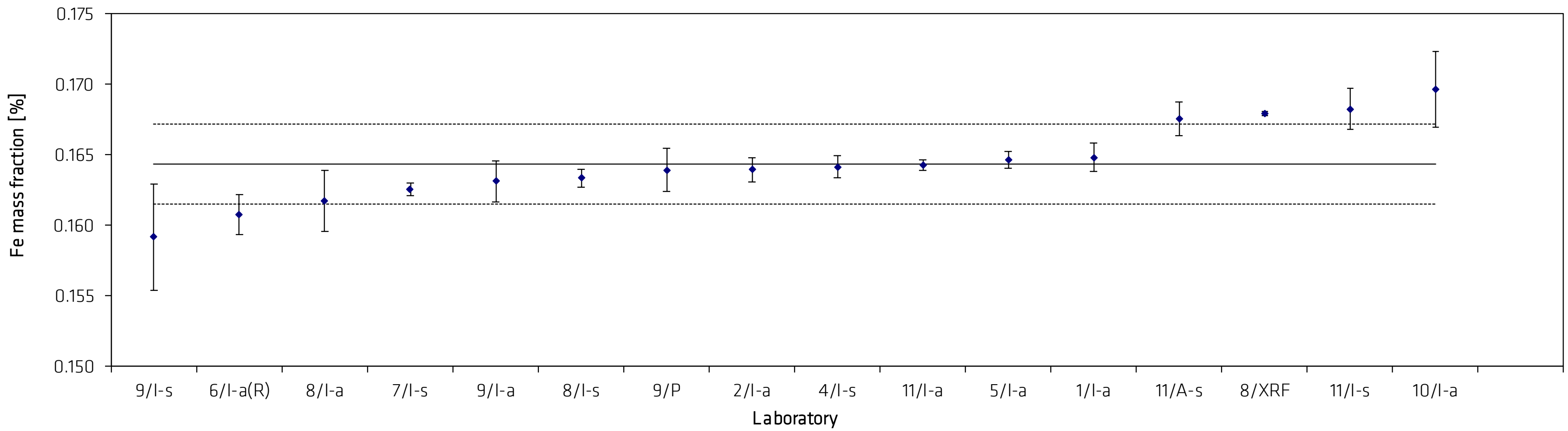


Table 4: Results for Cu

Lab./Meth.	6/I-a(R)	5/I-a	9/I-s	9/I-a	8/I-a	8/I-s	11/I-a	11/I-s	10/I-a	2/I-a	7/I-s	8/XRF	1/I-a	4/I-s		
M_i [%]	1.333	1.340	1.408	1.345	1.354	1.352	1.360	1.373	1.351	1.372	1.372	1.367	1.370	1.391		n
	1.329	1.349	1.338	1.336	1.389	1.354	1.357	1.352	1.352	1.359	1.366	1.367	1.380	1.407		13
	1.316	1.339	1.347	1.343	1.344	1.341	1.362	1.356	1.375	1.360	1.371	1.366	1.370	1.395		
	1.310	1.342	1.357	1.365	1.348	1.362	1.353	1.349	1.381	1.366	1.360	1.368	1.380	1.366		
	1.325	1.342	1.344	1.354	1.340	1.360	1.355	1.354	1.348	1.366	1.376	1.367	1.370	1.376		
	1.316	1.350	1.308	1.360	1.344	1.360	1.356	1.372	1.351	1.367	1.355	1.367	1.360	1.369		
M [%]	**1.322**	**1.344**	**1.350**	**1.351**	**1.353**	**1.355**	**1.357**	**1.359**	**1.360**	**1.365**	**1.367**	**1.367**	**1.372**	**1.384**		**1.360**
s [%]	0.0087	0.0045	0.0328	0.0110	0.0182	0.0078	0.0033	0.0105	0.0144	0.0048	0.0079	0.0006	0.0075	0.0162	s_M [%]	0.0107
															$\bar{s}_i$ [%]	0.0140
s_{rel}	0.00658	0.00338	0.02426	0.00818	0.01344	0.00576	0.00244	0.00774	0.01058	0.00352	0.00581	0.00043	0.00549	0.01170		0.00784

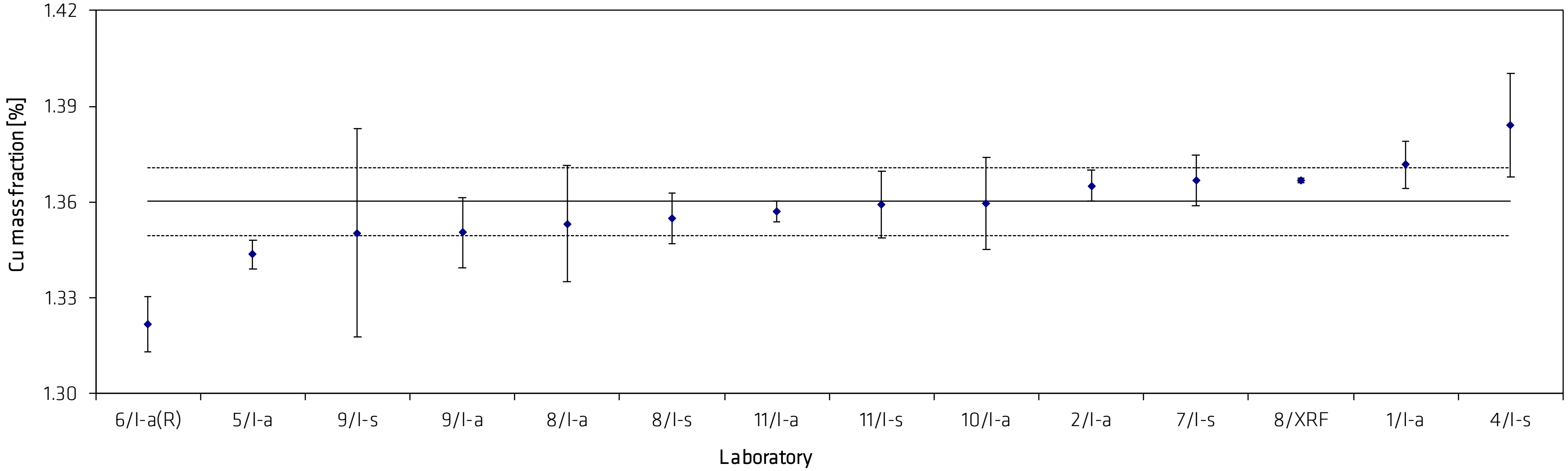


Table 5: Results for Mn

Lab./Meth.	9/I-s	8/XRF	8/I-a(R)	6/I-a(R)	11/I-a	10/I-a	7/I-s	1/I-a	8/I-s	5/I-a	4/I-s	2/I-a	11/I-s	9/I-a		
M_i [%]	0.0321	0.0336	0.0343	0.0344	0.0343	0.0344	0.0346	0.0346	0.0345	0.0346	0.0347	0.0354	0.0351	0.0348		*n*
	0.0337	0.0336	0.0340	0.0341	0.0343	0.0337	0.0348	0.0347	0.0346	0.0347	0.0346	0.0348	0.0347	0.0352		14
	0.0339	0.0334	0.0339	0.0341	0.0342	0.0346	0.0346	0.0344	0.0344	0.0344	0.0348	0.0351	0.0346	0.0351		
	0.0338	0.0334	0.0339	0.0337	0.0341	0.0347	0.0347	0.0345	0.0345	0.0346	0.0345	0.0345	0.0345	0.0343		
	0.0334	0.0334	0.0335	0.0340	0.0342	0.0336	0.0344	0.0342	0.0345	0.0345	0.0346	0.0345	0.0351	0.0350		
	0.0327	0.0334	0.0337	0.0339	0.0342	0.0344	0.0334	0.0344	0.0348	0.0347	0.0345	0.0346	0.0351	0.0350		
M [%]	**0.0333**	**0.0334**	**0.0339**	**0.0340**	**0.0342**	**0.0342**	**0.0344**	**0.0345**	**0.0346**	**0.0346**	**0.0346**	**0.0348**	**0.0349**	**0.0349**		**0.0343**
s [%]	0.0007	0.0001	0.0003	0.0002	0.0001	0.0005	0.0005	0.0002	0.0001	0.0001	0.0001	0.0004	0.0003	0.0003	s_M [%]	0.0005
															$\bar{s}_i$ [%]	0.0004
s_{rel}	0.02157	0.00321	0.00801	0.00687	0.00220	0.01366	0.01498	0.00508	0.00325	0.00308	0.00338	0.01010	0.00807	0.00899		0.01461

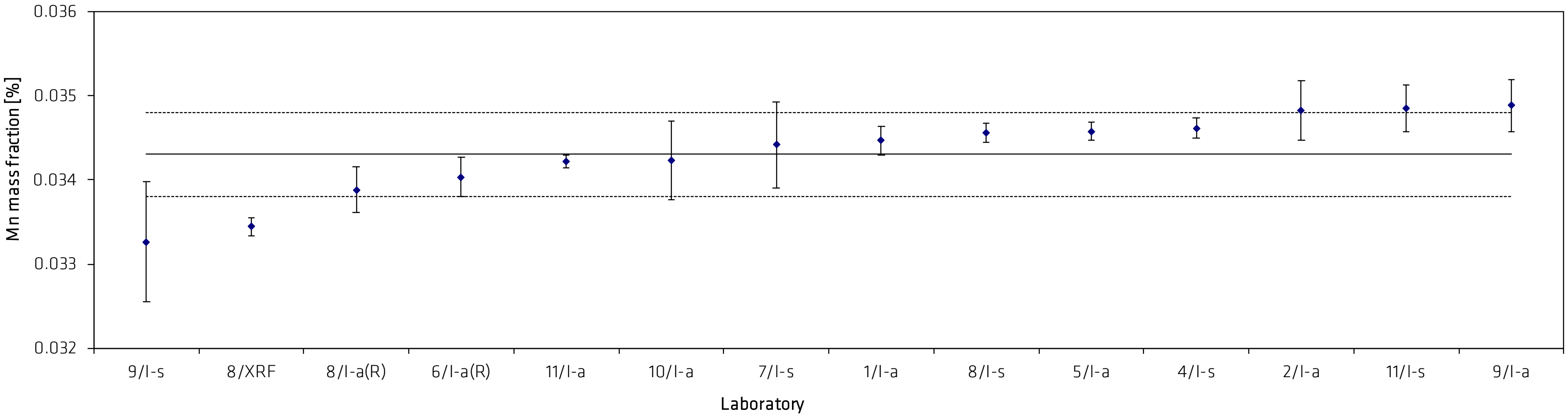


Table 6: Results for Mg

Lab./Meth.	9/I-a	10/I-a	11/I-s	8/I-a	6/I-s	8/I-s	11/I-a	2/I-a	1/I-a	11/A-s	5/I-a	4/I-s	7/I-s		
M_i [%]	2.203	2.25	2.285	2.280	2.222	2.269	2.272	2.297	2.290	2.331	2.296	2.338	2.329		*n*
	2.184	2.24	2.252	2.300	2.280	2.266	2.281	2.275	2.290	2.278	2.303	2.349	2.328		13
	2.222	2.29	2.259	2.259	2.316	2.262	2.270	2.281	2.280	2.290	2.291	2.337	2.350		
	2.223	2.30	2.255	2.260	2.268	2.279	2.276	2.285	2.290	2.308	2.298	2.322	2.359		
	2.221	2.24	2.273	2.269	2.298	2.278	2.282	2.286	2.290	2.275	2.294	2.318	2.331		
	2.224	2.25	2.285	2.254	2.253	2.288	2.268	2.286	2.280	2.285	2.301	2.301	2.362		
M [%]	**2.213**	**2.262**	**2.268**	**2.270**	**2.273**	**2.274**	**2.275**	**2.285**	**2.287**	**2.294**	**2.297**	**2.328**	**2.343**		**2.282**
s [%]	0.0161	0.0264	0.0149	0.0172	0.0332	0.0097	0.0057	0.0073	0.0052	0.0213	0.0045	0.0172	0.0157	s_M [%]	0.0316
														$\bar{s}_i$ [%]	0.0184
s_{rel}	0.00728	0.01167	0.00655	0.00757	0.01463	0.00426	0.00252	0.00318	0.00226	0.00927	0.00194	0.00741	0.00670		0.01383

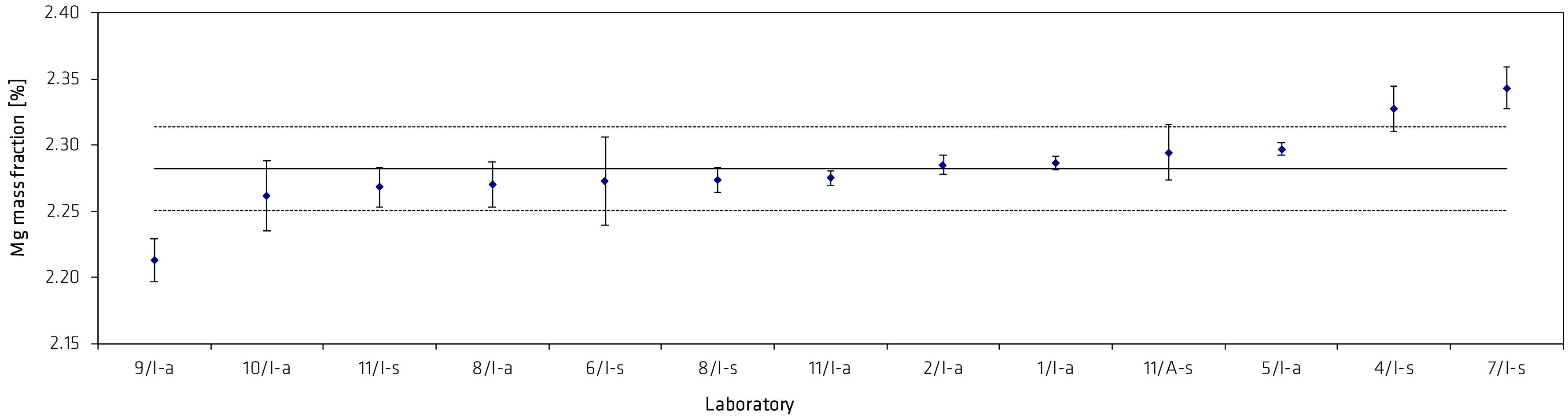


Table 7: Results for Cr

Lab./Meth.	10/I-a	6/I-a(R)	8/XRF(R)	9/I-s	8/I-a	1/I-a	8/I-s	5/I-a	2/I-s	7/I-s	11/I-s	4/I-s		
M_i [%]	0.187	0.1917	0.1899	0.1989	0.1932	0.194	0.1929	0.1926	0.1926	0.1931	0.1953	0.196		*n*
	0.186	0.1888	0.1900	0.1883	0.1923	0.193	0.1929	0.1937	0.1927	0.1953	0.1930	0.195		12
	0.190	0.1883	0.1898	0.1888	0.1909	0.192	0.1924	0.1921	0.1925	0.1945	0.1926	0.196		
	0.191	0.1870	0.1902	0.1883	0.1913	0.192	0.1932	0.1934	0.1932	0.1934	0.1975	0.195		
	0.186	0.1880	0.1898	0.1839	0.1892	0.191	0.1927	0.1929	0.1938	0.1948	0.1950	0.194		
	0.187	0.1876	0.1901	0.1942	0.1904	0.192	0.1941	0.1936	0.1934	0.1951	0.1961	0.196		
M [%]	**0.1878**	**0.1886**	**0.1899**	**0.1904**	**0.1912**	**0.1923**	**0.1930**	**0.1930**	**0.1930**	**0.1944**	**0.1949**	**0.1953**		**0.1920**
s [%]	0.0021	0.0017	0.0002	0.0053	0.0014	0.0010	0.0006	0.0006	0.0005	0.0009	0.0019	0.0008	s_M [%]	0.0024
													$\bar{s}_i$ [%]	0.0019
s_{rel}	0.01138	0.00876	0.00087	0.02782	0.00738	0.00537	0.00297	0.00317	0.00257	0.00469	0.00953	0.00418		0.01268

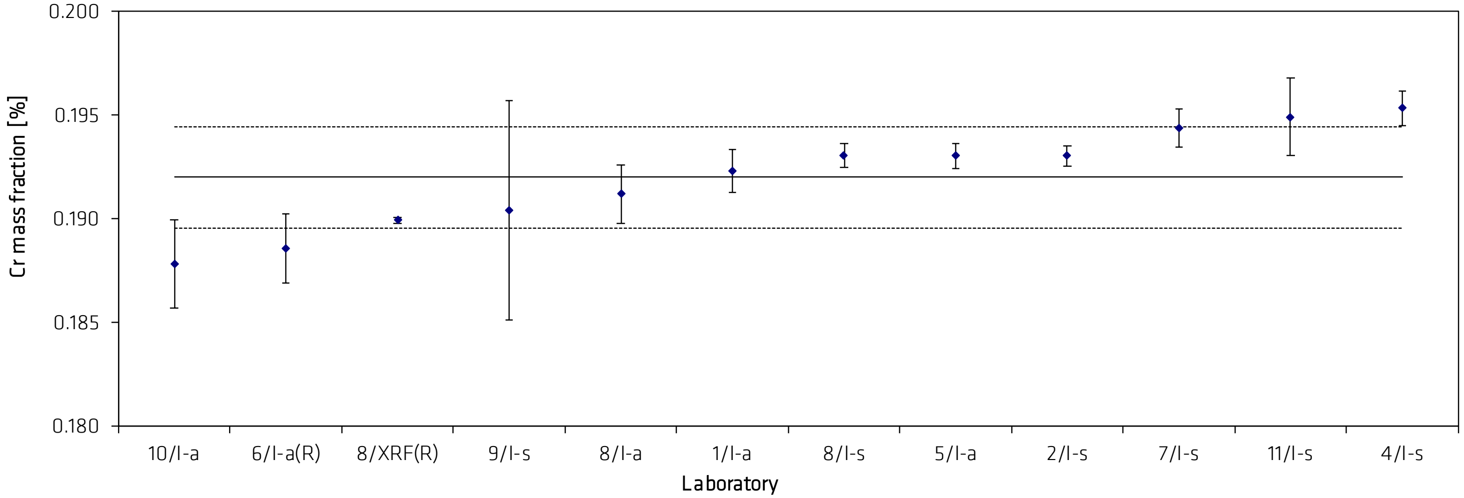


Table 8: Results for Zn

Lab./Meth.	8/I-s	8/I-a	11/I-s	7/A-s	8/XRF	5/I-a	2/I-a	11/I-a	1/I-a	6/I-s	10/I-a	4/I-s	9/I-a		
M_i [%]	5.544	5.568	5.616	5.546	5.575	5.5703	5.639	5.6472	5.64	5.5866	5.620	5.703	5.723		*n*
	5.584	5.606	5.487	5.580	5.574	5.5932	5.596	5.6389	5.65	5.7318	5.580	5.736	5.730		13
	5.489	5.494	5.528	5.554	5.572	5.5758	5.582	5.6247	5.62	5.7881	5.720	5.710	5.784		
	5.481	5.542	5.536	5.527	5.573	5.5861	5.616	5.6118	5.67	5.5521	5.720	5.592	5.656		
	5.510	5.494	5.598	5.605	5.574	5.5680	5.613	5.6686	5.63	5.5632	5.620	5.663	5.797		
	5.567	5.508	5.597	5.560	5.574	5.5833	5.626	5.6373	5.62	5.6553	5.630	5.640	5.565		
M [%]	**5.529**	**5.535**	**5.560**	**5.562**	**5.574**	**5.579**	**5.612**	**5.638**	**5.638**	**5.646**	**5.648**	**5.674**	**5.709**		**5.608**
s [%]	0.0423	0.0453	0.0506	0.0273	0.0010	0.0098	0.0203	0.0194	0.0194	0.0968	0.0581	0.0529	0.0867	s_M [%]	0.0558
														$\bar{s}_i$ [%]	0.0512
s_{rel}	0.00766	0.00819	0.00910	0.00491	0.00017	0.00175	0.00361	0.00344	0.00344	0.01714	0.01029	0.00932	0.01518		0.00995

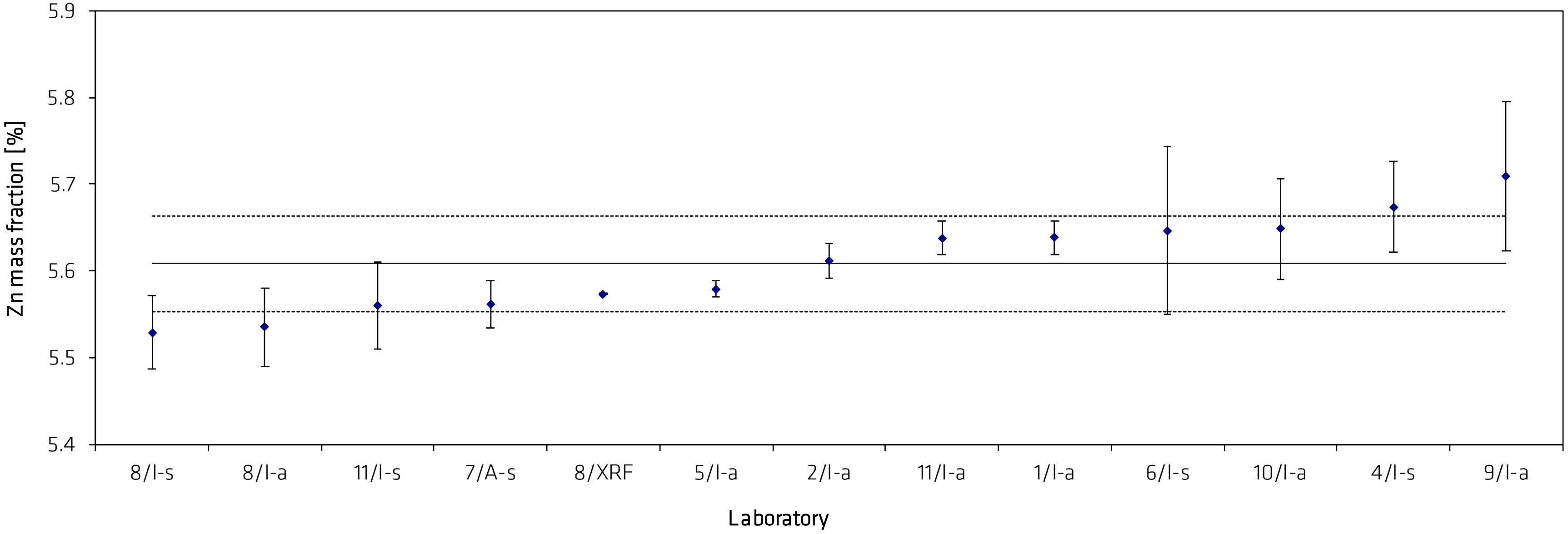


Table 9: Results for Ni

Lab./Meth.	6/I-a(R)	9/I-s	9/EA-a	8/I-a	2/I-s	4/I-s	9/EA-s	8/XRF	8/I-s	7/I-s(R)	10/I-a	1/I-a	11/I-a	5/I-a	11/I-s		
M_i [mg/kg]	142.2	135	142.0	144.6	146.9	145.9	148	146.5	146.7	149.7	147	150	149.2	150.9	153.9		*n*
	142.1	149	146.0	145.3	146.6	145.8	146	147.0	147.2	145.7	147	151	149.3	152.6	151.3		15
	139.6	143	143.0	145.9	146.8	145.5	147	147.0	146.4	146.2	150	148	150.6	150.9	151.2		
	143.8	149	142.0	146.4	144.2	146.8	146	146.5	146.5	146.2	152	151	149.7	151.3	150.4		
	142.3	142	145.0	144.7	143.8	145.2	146	146.2	147.9	148.3	148	148	149.5	151.3	153.2		
	140.5	141	142.0	144.6	144.2	146.8	147	147.4	148.3	148.4	149	148	148.4	151.8	153.0		
M [mg/kg]	**141.8**	**143.2**	**143.3**	**145.2**	**145.4**	**146.0**	**146.7**	**146.8**	**147.1**	**147.4**	**148.8**	**149.3**	**149.5**	**151.5**	**152.2**		**146.9**
s [mg/kg]	1.5	5.3	1.8	0.8	1.5	0.7	0.8	0.4	0.8	1.6	1.9	1.5	0.7	0.7	1.4	s_M [mg/kg]	2.975
																$\bar{s}_i$ [mg/kg]	1.8200
s_{rel}	0.01047	0.03707	0.01222	0.00535	0.01021	0.00456	0.00557	0.00301	0.00555	0.01088	0.01304	0.01008	0.00480	0.00433	0.00909		0.02025

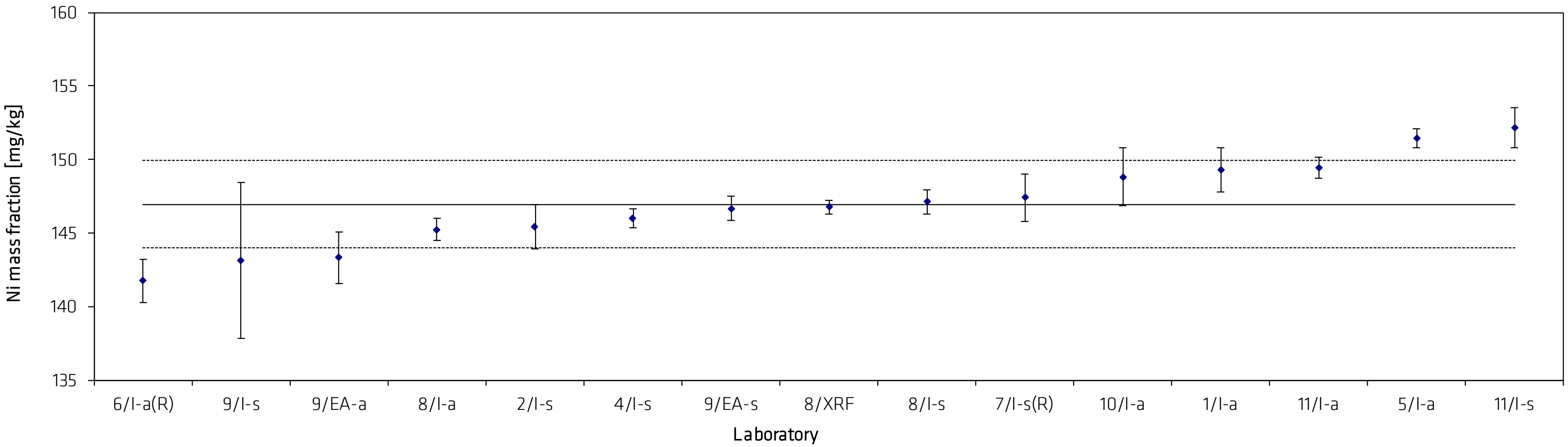


Table 10: Results for Ti

Lab./Meth.	9/I-a(R)	8/I-a	8/I-s	11/I-a	11/A-s	9/I-s	6/I-a	7/I-s	5/I-a	2/I-s	9/P	4/I-s	1/I-a	11/I-s	8/XRF	10/I-a		
M_i [mg/kg]	246.4	250.9	255.3	254.7	258.5	268	257.0	261.1	258.1	256.7	257.8	261.8	261	263.1	262.0	262		n
	244.5	251.9	255.5	255.6	263.2	252	256.0	262.9	258.9	258.2	259.6	261.7	261	260.7	261.6	278		15
	244.1	255.8	254.4	255.8	261.2	256	258.3	270.9	257.1	257.3	258.3	255.9	259	259.5	259.3	267		
	251.8	251.6	255.6	255.2	249.4	254	256.6	265.4	258.8	259.4	259.6	255.5	260	257.9	261.8	263		
	259.1	249.2	253.9	255.5	254.3	256	256.9	241.3	257.1	259.7	258.6	260.8	259	263.4	260.2	269		
		250.6	256.9	255.3	250.7	255	256.6	241.2	258.2	259.4	260.1	259.5	261	263.3	263.3	282		
											260.0							
											258.7							
M [mg/kg]	**249.2**	**251.7**	**255.3**	**255.4**	**256.2**	**256.8**	**256.9**	**257.1**	**258.0**	**258.4**	**259.1**	**259.2**	**260.2**	**261.3**	**261.4**	**270.2**		**257.1**
s [mg/kg]	6.3	2.2	1.0	0.4	5.6	5.7	0.8	12.7	0.8	1.2	0.8	2.8	1.0	2.3	1.4	8.1	s_M [mg/kg]	3.34
																	$\bar{s}_i$ [mg/kg]	2.73
s_{rel}	0.0254	0.0089	0.0041	0.0015	0.0220	0.0221	0.0030	0.0495	0.0031	0.0048	0.0032	0.0109	0.0038	0.0089	0.0054	0.0301		0.0130

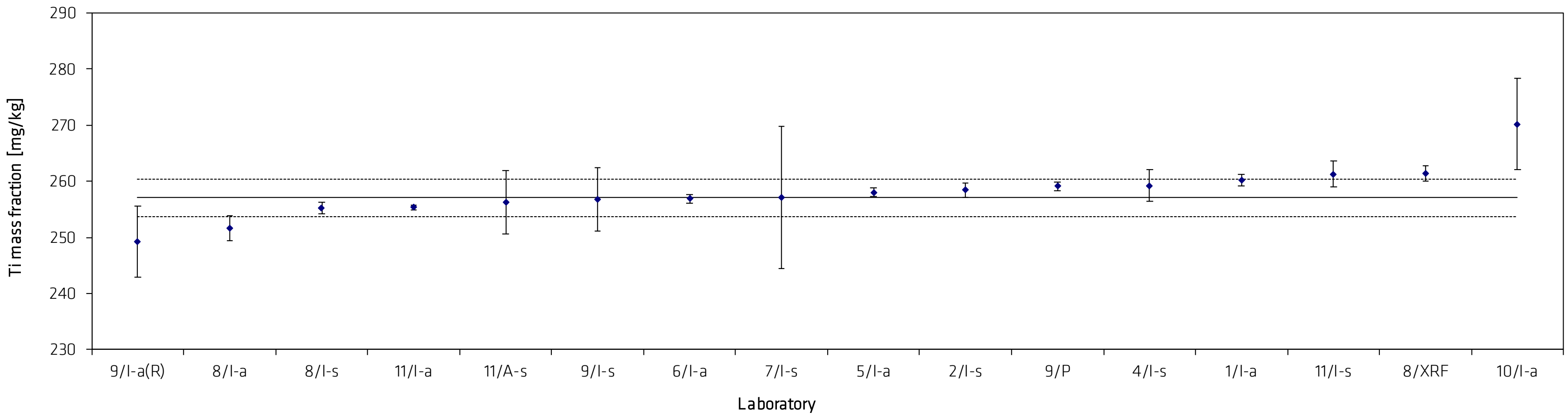


Table 11: Results for Ag

Lab./Meth.	8/XRF	8/I-s	5/I-a	2/IMS-s	11/I-s	9/I-s	4/I-s	1/I-a		
M_i [mg/kg]	5.1	5.8	6.3	6.6	6.8	7.0	7.0	7		*n*
	5.4	5.6	6.5	6.7	6.7	7.1	7.1	7		8
	5.1	5.0	6.4	6.7	6.7	7.0	7.1	7		
	5.4	5.1	6.4	6.5	6.6	7.0	7.2	7		
	5.1	5.7	6.4	6.5	6.8	7.0	7.2	8		
	5.4	5.5	6.3	6.6	6.8	7.1	7.2	7		
						7.1				
M [mg/kg]	**5.25**	**5.43**	**6.38**	**6.61**	**6.72**	**7.04**	**7.13**	**7.17**		**6.47**
s [mg/kg]	0.164	0.335	0.073	0.110	0.073	0.058	0.082	0.408	s_M [mg/kg]	0.748
									$\bar{s}_i$ [mg/kg]	0.157
s_{rel}	0.031	0.062	0.011	0.017	0.011	0.008	0.011	0.057		0.116

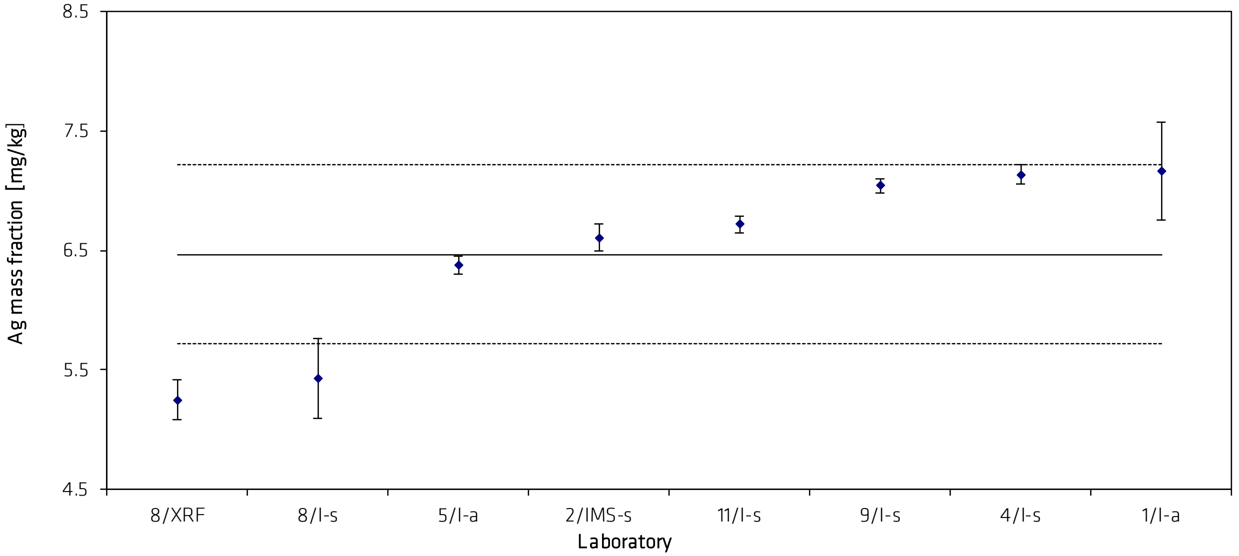


Table 12: Results for Be

Lab./Meth.	4/I-s	10/I-a	6/I-a	8/I-a	9/I-s	2/I-s	11/I-s	9/EA-s	8/I-s	5/I-a	7/IMS-s	9/I-a(R)	1/I-a(R)		
M_i [mg/kg]	1.7	1.7	1.73	1.75	1.88	1.84	1.85	1.83	1.85	1.86	1.95	2.01	1.9		*n*
	1.7	1.7	1.73	1.76	1.78	1.83	1.82	1.84	1.85	1.88	1.90	1.86	1.9		13
	1.7	1.8	1.74	1.74	1.77	1.83	1.82	1.89	1.84	1.86	1.92	1.81	1.9		
	1.7	1.7	1.73	1.74	1.79	1.75	1.81	1.81	1.85	1.86	1.79	1.85	1.9		
	1.7	1.7	1.73	1.75	1.77	1.74	1.85	1.82	1.84	1.85	1.79	1.84	1.9		
	1.7	1.7	1.72	1.74	1.73	1.74	1.84	1.86	1.86	1.87	1.84		1.9		
***M* [mg/kg]**	**1.70**	**1.72**	**1.73**	**1.75**	**1.79**	**1.79**	**1.83**	**1.84**	**1.85**	**1.86**	**1.87**	**1.87**	**1.90**		**1.81**
s [mg/kg]	0.000	0.041	0.008	0.008	0.050	0.048	0.017	0.029	0.008	0.010	0.068	0.078	0.000	s_M [mg/kg]	0.067
														$\bar{s}_i$ [mg/kg]	0.038
s_{rel}	0.000	0.024	0.004	0.005	0.028	0.027	0.009	0.016	0.004	0.006	0.037	0.042	0.000		0.037

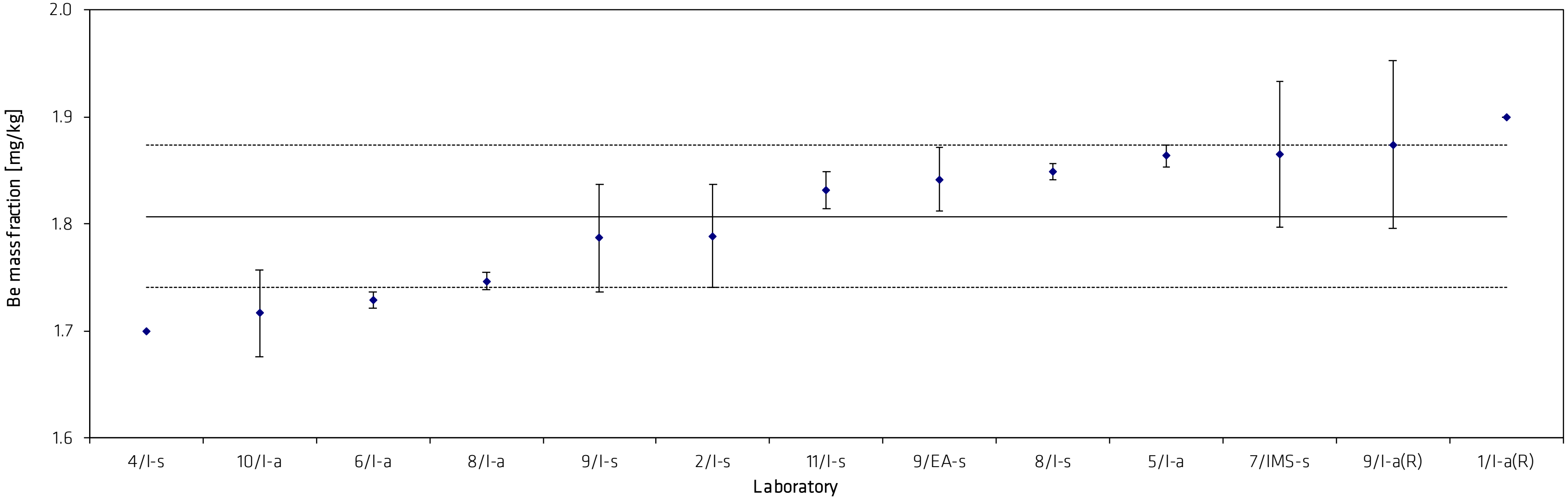


Table 13: Results for Ca

Lab./Meth.	2/I-s	1/I-a	6/I-s	4/I-s		
M_i [mg/kg]	9.63	12.0	11.1	12.2		n
	9.58	11.0	11.8	11.8		4
	9.53	10.0	10.7	11.2		
	9.18	11.0	12.9	11.4		
	9.17	11.0	11.2	11.6		
	9.25	11.0	10.1	11.4		
M [mg/kg]	**9.39**	**11.00**	**11.29**	**11.60**		**10.82**
s [mg/kg]	0.211	0.632	0.978	0.358	s_M [mg/kg]	0.986
					$\bar{s}_i$ [mg/kg]	0.618
s_{rel}	0.022	0.057	0.087	0.031		0.091

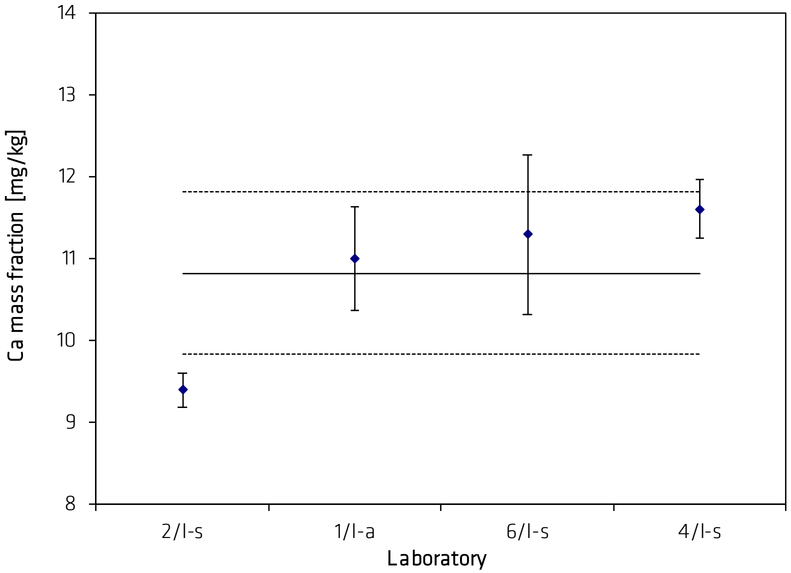


Table 14: Results for Na

Lab./Meth.	9/I-s	7/A-s	4/I-s	1/I-a	11/A-s	11/I-s	6/I-s(R)	2/I-s		
M_i [mg/kg]	13.6	14.0	16.1	16	16.4	17.1	16.6	18.42		*n*
	13.2	13.8	15.8	16	16.4	16.2	16.6	18.39		8
	13.0	14.8	15.6	16	17.3	15.9	16.3	18.07		
	13.2	13.9	15.3	16	16.0	16.4	16.8	17.21		
	13.9	14.2	15.3	16	15.9	16.4	16.3	17.15		
		14.0	15.8	16	15.8		17.1	17.15		
M [mg/kg]	**13.38**	**14.12**	**15.65**	**16.00**	**16.29**	**16.38**	**16.60**	**17.73**		**15.77**
s [mg/kg]	0.361	0.360	0.315	0.000	0.533	0.442	0.290	0.627	s_M [mg/kg]	1.398
									$\bar{s}_i$ [mg/kg]	0.405
s_{rel}	0.027	0.026	0.020	0.000	0.033	0.027	0.017	0.035		0.089

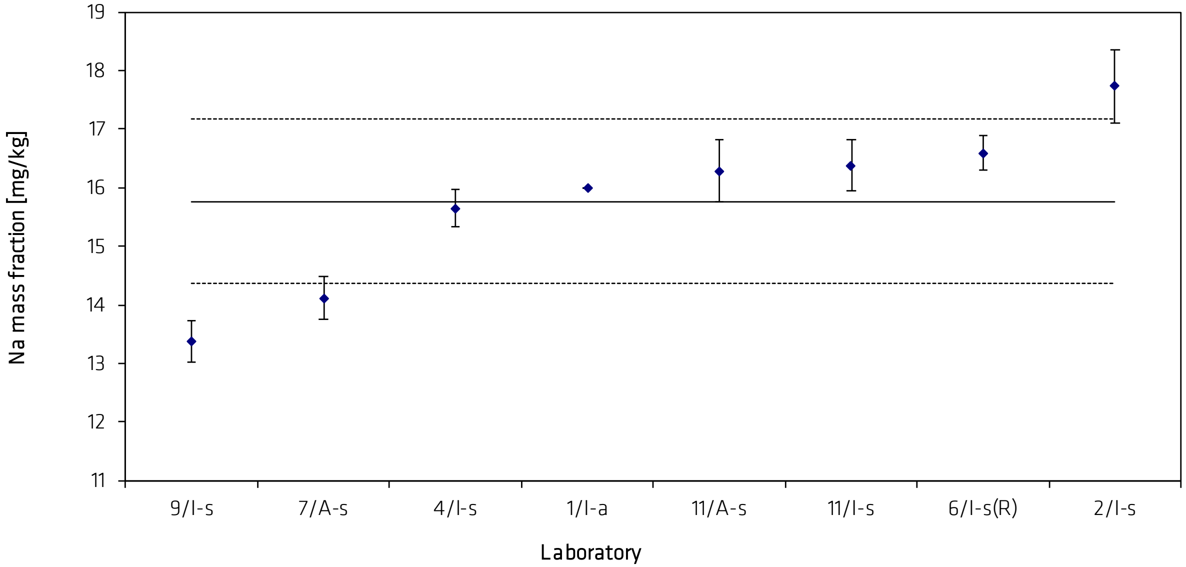


Table 15: Results for Pb

Lab./Meth.	1/I-a	7/IMS-s	10/I-a	2/IMS-s	6/I-a(R)	8/I-s	4/I-s	8/XRF	9/EA-s	5/I-a	11/IMS-s(R)		
M_i [mg/kg]	41	42.2	41.6	43.1	45.1	41.9	43.4	44.4	46.0	43.8	46.0		*n*
	42	41.9	42.8	42.2	45.3	45.2	43.8	44.3	44.0	42.6	45.0		11
	41	43.1	43.1	42.7	43.6	46.2	44.2	44.3	44.0	45.0	45.5		
	42	42.2	42.8	42.9	42.4	47.1	45.1	44.8	44.0	46.9	45.4		
	41	42.0	41.3	42.9	43.6	42.8	44.9	44.7	45.0	46.6	45.0		
	41	41.5	41.5	43.0	41.7	42.7	44.6	44.6	45.0	48.0	46.5		
M [mg/kg]	**41.3**	**42.1**	**42.2**	**42.8**	**43.6**	**44.3**	**44.3**	**44.5**	**44.7**	**45.5**	**45.5**		**43.7**
s [mg/kg]	0.516	0.533	0.799	0.305	1.431	2.154	0.656	0.214	0.816	2.046	0.575	s_M [mg/kg]	1.416
												$\bar{s}_i$ [mg/kg]	1.165
s_{rel}	0.0125	0.0127	0.0189	0.0071	0.0328	0.0486	0.0148	0.0048	0.0183	0.0450	0.0126		0.032

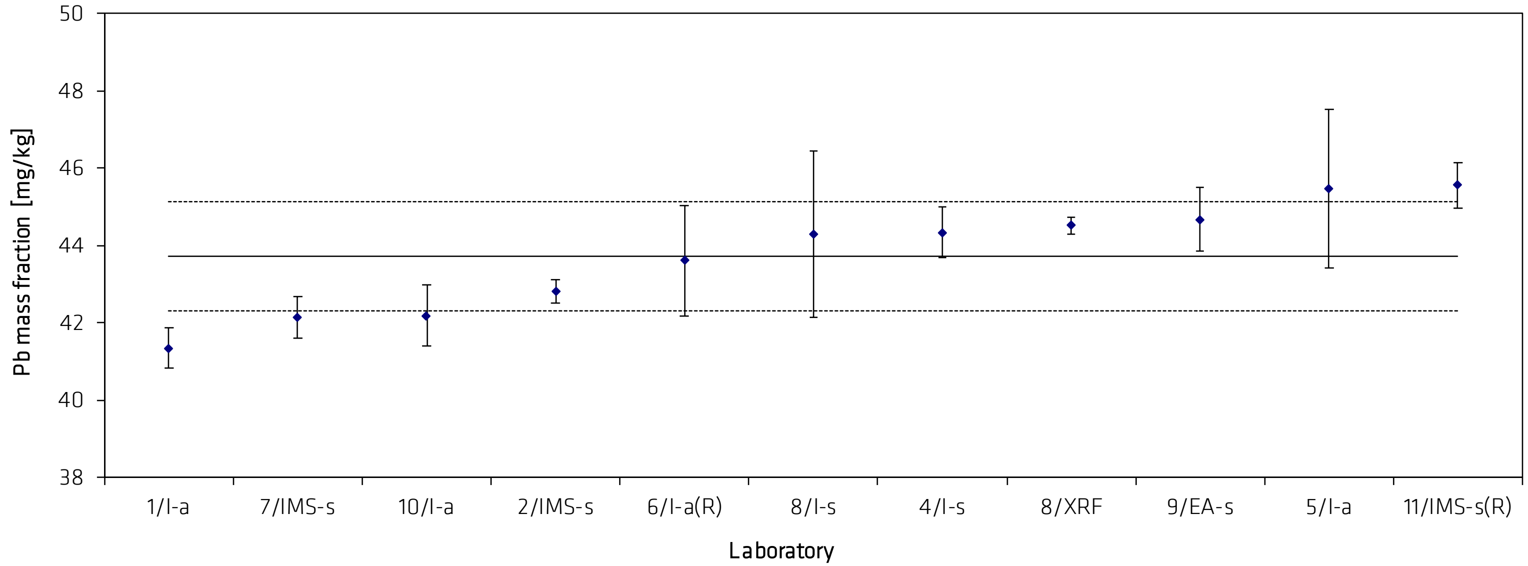
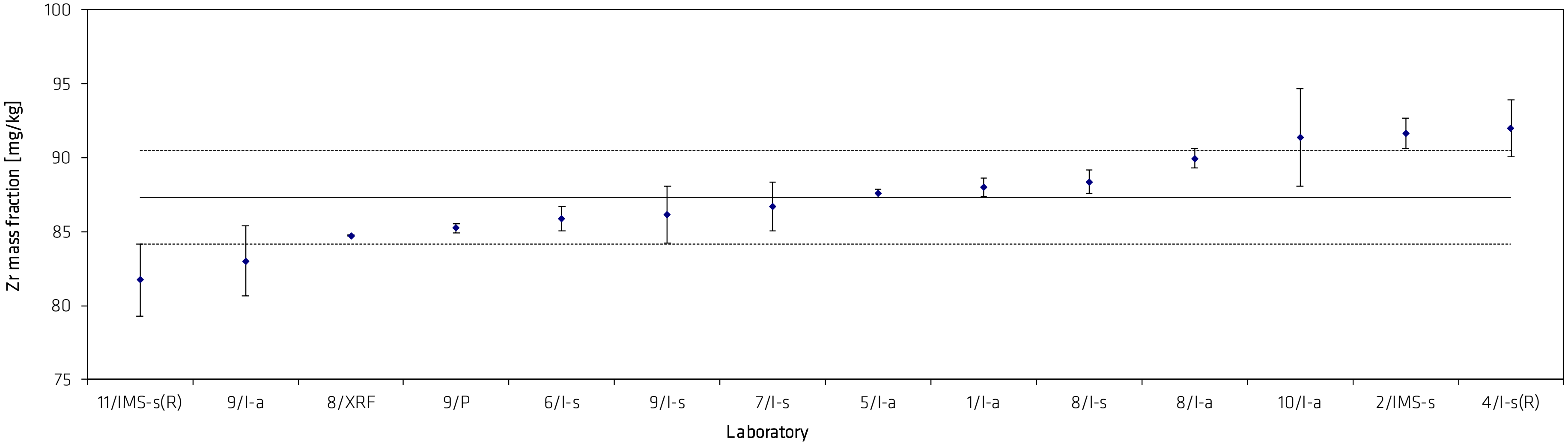


Table 16: Results for Zr

Lab./Meth.	11/IMS-s(R)	9/I-a	8/XRF	9/P	6/I-s	9/I-s	7/I-s	5/I-a	1/I-a	8/I-s	8/I-a	10/I-a	2/IMS-s	4/I-s(R)		
M_i [mg/kg]	82.3	85.1	84.8	85.1	85.1	84	85.3	87.9	89	87.9	89.9	95.4	90.5	94.0		*n*
	85.7	82.4	84.7	85.4	86.3	87	85.3	87.8	88	88.0	90.6	88.0	92.2	94.0		14
	81.0	82.4	84.7	85.7	87.3	86	88.7	87.5	88	87.6	90.2	90.3	90.1	93.0		
	80.9	85.5	84.7	84.8	86.0	89	87.4	87.7	88	88.1	90.6	90.4	92.6	90.0		
	78.2	79.7	84.7	85.4	85.4	87	88.4	87.1	87	89.4	88.9	88.6	92.4	90.0		
	82.3		84.8	85.1	85.1	84	85.3	87.6	88	89.4	89.5	95.5	92.1	91.0		
M [mg/kg]	**81.7**	**83.0**	**84.7**	**85.2**	**85.9**	**86.2**	**86.7**	**87.6**	**88.0**	**88.4**	**89.9**	**91.4**	**91.6**	**92.0**		**87.3**
s [mg/kg]	2.4549	2.3594	0.0516	0.3160	0.8299	1.9408	1.6343	0.2721	0.6325	0.8104	0.6616	3.2989	1.0550	1.8974	s_M [mg/kg]	3.1639
															$\bar{s}_i$ [mg/kg]	1.603
s_{rel}	0.03004	0.02842	0.00061	0.00371	0.00966	0.02252	0.01885	0.00311	0.00719	0.00917	0.00736	0.03611	0.01151	0.02062		0.03624



The statistical evaluation of the data was performed using the software program SoftCRM 1.2.2. [6]. The following results were obtained:

Tab. 17: Outcome of statistical tests on the results obtained for Si and Fe

	Si	Fe
Number of data sets	12	15
Scheffe's test (data compatible?)	yes	yes
Snedecor-F-Test and Bartlett-Test	Pooling not allowed	Pooling not allowed
Dixon (α = 0.05)	---	---
Dixon (α = 0.01)	---	---
Nalimov (α = 0.05)	---	Lab. 10
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	Distribution: normal
Kolmogorov-Smirnov-Lilliefors Test (α = 0.01)	Distribution: normal	Distribution: normal
Skewness & Kurtosis Test (α = 0.05)	Distribution: normal	Distribution: normal
Skewness & Kurtosis Test (α = 0.01)	Distribution: normal	Distribution: normal

The straggler (Lab. 10; Fe) was not removed.

Tab. 18: Outcome of statistical tests on the results obtained for Cu

	1^{st} run	2^{nd} run
Number of data sets	14	13
Scheffe's test (data compatible?)	yes	yes
Snedecor-F-Test and Bartlett-Test	Pooling not allowed	Pooling not allowed
Dixon (α = 0.05)	Lab. 6	---
Dixon (α = 0.01)	---	---
Nalimov (α = 0.05)	Lab. 6	Lab. 4
Nalimov (α = 0.01)	Lab. 6	---
Grubbs (α = 0.05)	Lab. 6	---
Grubbs (α = 0.01)	---	---
Grubbs Pair (α = 0.05)	---	---
Grubbs Pair (α = 0.01)	---	---
Cochran	---	---
Kolmogorov-Smirnov-Lilliefors Test (α = 0.05)	Distribution: normal	Distribution: normal
Kolmogorov-Smirnov-Lilliefors Test (α = 0.01)	Distribution: normal	Distribution: normal
Skewness & Kurtosis Test (α = 0.05)	Distribution: not normal	Distribution: normal
Skewness & Kurtosis Test (α = 0.01)	Distribution: normal	Distribution: normal

The outlier (Lab. 6, 1^{st} run) was removed, the straggler (Lab. 4, 2^{nd} run) was not removed.

Tab. 19: Outcome of statistical tests on the results obtained for Mn and Fe

	Mn	Mg
Number of data sets	14	13
Scheffe's test (data compatible?)	yes	yes
Snedecor-F-Test and Bartlett-Test	Pooling not allowed	Pooling not allowed
Dixon (α = 0.05)	---	Lab. 7
Dixon (α = 0.01)	---	---
Nalimov (α = 0.05)	Lab. 9/I-s	Labs. 7 and 9/I
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	Distribution: normal
Kolmogorov-Smirnov-Lilliefors Test (α = 0.01)	Distribution: normal	Distribution: normal
Skewness & Kurtosis Test (α = 0.05)	Distribution: normal	Distribution: normal
Skewness & Kurtosis Test (α = 0.01)	Distribution: normal	Distribution: normal

The stragglers (Lab. 9; Mg and Labs. 7 and 9, Mg) were not removed.

Tab. 20: Outcome of statistical tests on the results obtained for Cr and Zn

	Cr	Zn
Number of data sets	12	13
Scheffe's test (data compatible?)	yes	yes
Snedecor-F-Test and Bartlett-Test	Pooling not allowed	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	Distribution: normal
Kolmogorov-Smirnov-Lilliefors Test (α = 0.01)	Distribution: normal	Distribution: normal
Skewness & Kurtosis Test (α = 0.05)	Distribution: normal	Distribution: normal
Skewness & Kurtosis Test (α = 0.01)	Distribution: normal	Distribution: normal

Tab. 21: Outcome of statistical tests on the results obtained for Ni and Ag

	Ni	Ag
Number of data sets	15	8
Scheffe's test (data compatible?)	yes	yes
Snedecor-F-Test and Bartlett-Test	Pooling not allowed	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	Distribution: normal
Kolmogorov-Smirnov-Lilliefors Test (α = 0.01)	Distribution: normal	Distribution: normal
Skewness & Kurtosis Test (α = 0.05)	Distribution: normal	Distribution: normal
Skewness & Kurtosis Test (α = 0.01)	Distribution: normal	Distribution: normal

Tab. 22: Outcome of statistical tests on the results obtained for Ti

	1^{st} run	2^{nd} run
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 (α = 0.05)	Lab. 10	---
Dixon (α = 0.01)	---	---
Nalimov (α = 0.05)	Lab. 10	Lab. 0/I-a
Nalimov (α = 0.01)	Lab. 10	---
Grubbs (α = 0.05)	Lab. 10	---
Grubbs (α = 0.01)	---	---
Grubbs Pair (α = 0.05)	---	---
Grubbs Pair (α = 0.01)	---	---
Cochran	---	---
Kolmogorov-Smirnov-Lilliefors Test (α = 0.05)	Distribution: normal	Distribution: normal
Kolmogorov-Smirnov-Lilliefors Test (α = 0.01)	Distribution: normal	Distribution: normal
Skewness & Kurtosis Test (α = 0.05)	Distribution: not normal	Distribution: normal
Skewness & Kurtosis Test (α = 0.01)	Distribution: normal	Distribution: normal

The outlier (Lab. 10, 1^{st} run) was removed, the straggler (Lab. 9, 2^{nd} run) was not removed.

Tab. 23: Outcome of statistical tests on the results obtained for Be and Ca

	Be	Ca
Number of data sets	13	4
Scheffe's test (data compatible?)	yes	yes
Snedecor-F-Test and Bartlett-Test	Pooling not allowed	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	Distribution: normal
Kolmogorov-Smirnov-Lilliefors Test (α = 0.01)	Distribution: normal	Distribution: normal
Skewness & Kurtosis Test (α = 0.05)	Distribution: normal	Distribution: insufficient data
Skewness & Kurtosis Test (α = 0.01)	Distribution: normal	Distribution: insufficient data

Tab. 24: Outcome of statistical tests on the results obtained for Na and Pb

	Na	Pb
Number of data sets	8	10
Scheffe's test (data compatible?)	yes	yes
Snedecor-F-Test and Bartlett-Test	Pooling not allowed	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	Distribution: normal
Kolmogorov-Smirnov-Lilliefors Test (α = 0.01)	Distribution: normal	Distribution: normal
Skewness & Kurtosis Test (α = 0.05)	Distribution: normal	Distribution: normal
Skewness & Kurtosis Test (α = 0.01)	Distribution: normal	Distribution: normal

Tab. 25: Outcome of statistical tests on the results obtained for Zr

	1st run
Number of data sets	14
Scheffe's test (data compatible?)	yes
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
Skewness & Kurtosis Test (α = 0.05)	Distribution: normal
Skewness & Kurtosis 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 26.
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.

$$u_{\mathrm{combined}} = \sqrt{u_{ilc}^2 + u_{bb}^2(1) + u_{bb}^2(2)} \qquad (3)$$

with

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

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

Table 26: Uncertainty calculation

	uncertainty contribution from									u_{bb} (rel)	
	M	n	s_M	u_{ilc}	u_{bb} (1) Length	u_{bb} (2) Area	u(comb)	U		Length	Area
	%		%	%	%	%	%	%			
Si	0.0720	12	0.0028	0.0008	0.0005	0.0008	0.0013	0.00253		0.7364	1.1308
Fe	0.1644	15	0.0028	0.0007	0.0008	0.0019	0.0022	0.00431		0.5099	1.1261
Cu	1.3602	13	0.0107	0.0030	0.0065	0.0120	0.0140	0.02801		0.4798	0.8844
Mn	0.0343	14	0.0005	0.0001	0.0002	0.0000	0.0002	0.00046		0.5320	0.1262
Mg	2.2820	13	0.0316	0.0088	0.0097	0.0153	0.0201	0.04026		0.4234	0.6718
Cr	0.1920	12	0.0024	0.0007	0.0011	0.0009	0.0016	0.00320		0.5939	0.4594
Zn	5.6080	13	0.0558	0.0155	0.0322	0.0107	0.0373	0.07459		0.5740	0.1913
	mg/kg		mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg			
Ni	146.90	15	2.9750	0.7681	0.9942	0.7807	1.4792	2.9583		0.6768	0.5314
Ti	257.10	15	3.3000	0.8521	0.3596	3.2250	3.3549	6.7099		0.1399	1.2544
Ag	6.47	8	0.7480	0.2645	0.0396	0.1172	0.2920	0.5839		0.6120	1.8110
Be	1.81	13	0.0670	0.0186	0.0066	0.0337	0.0391	0.0782		0.3621	1.8644
Ca	10.82	4	0.9860	0.4930	0.1167	0.1630	0.5322	1.5966		1.0784	1.5063
Na	15.77	8	1.3980	0.4943	0.9425	0.1720	1.0780	2.1560		5.9763	1.0905
Pb	43.60	10	1.4556	0.4603	0.4848	1.1170	1.3017	2.6035		1.1118	2.5619
Zr	87.30	14	3.1640	0.8456	0.4971	0.7930	1.2614	2.5227		0.5694	0.9084

The expanded uncertainties U are calculated by multiplication of $u_{combined}$ with a coverage factor of k = 2 (Ca: k = 3) using Equation 4.

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

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

In addition to the wet chemical characterization some of the laboratories analysed the material with spark emission to check if there is agreement between SOES and wet chemistry. Tab. 27 shows the mean values of wet chemical and spark emission results as well as their standard deviations. The agreement between wet chemistry and SOES is good for all elements except of Ag.

Tab. 27: Comparison wet chemistry (incl. XRF) vs. SOES

Element	Wet chemical analysis			Spark emission		
	Mass fraction in %	**Std.-dev.** in %	***n***	**Mass fraction** in %	**Std.-dev.** in %	***n***
Si	0.072	0.0028	12	0.073	0.0030	10
Fe	0.1644	0.0028	15	0.1672	0.0059	10
Cu	1.360	0.011	13	1.372	0.023	9
Mn	0.0343	0.0005	14	0.0344	0.0013	10
Mg	2.282	0.032	13	2.331	0.061	10
Cr	0.1920	0.0024	12	0.1908	0.0045	10
Zn	5.61	0.06	13	5.70	0.15	10
	in mg/kg	in mg/kg		in mg/kg	in mg/kg	
Ni	146.9	3.0	15	149.7	6.3	9
Ti	257.1	3.3	15	259.1	9.8	9
Ag	6.47	0.75	8	8.8	1.7	8
Be	1.81	0.07	13	1.87	0.12	9
Ca	10.8	1.0	4	11.7	0.9	3
Na	15.8	1.4	8	18.2	2.3	9
Pb	43.6	1.5	10	43.1	4.0	9
Zr	87.3	3.2	14	87.6	3.9	9

6. Instructions for users and stability

The certified reference material BAM-M308a 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).

7. References

[1] ISO Guide 31, Reference materials - Contents of certificates, labels and accompanying documentation, 2015

[2] ISO Guide 34, General requirements for the competence of reference material producers, 2009

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

[4] Guidelines for the development and production of BAM Reference Materials, 2016

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

[6] 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 BAM-M308a 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-M308a 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.

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

Silicon:

Sample	*Number*	*Sum*	*Mean*	*Variance*		
A1	5	0.3582	0.07164	1.53E-07		
A2	5	0.3553	0.07106	1.18E-07		
A3	5	0.3515	0.0703	9E-08		
A4	5	0.3565	0.0713	9.5E-08		
A5	5	0.3544	0.07088	1.12E-07		
B1	5	0.3585	0.0717	3.5E-08		
B2	5	0.3544	0.07088	3.7E-08		
B3	5	0.3539	0.07078	3.7E-08		
B4	5	0.3528	0.07056	1.33E-07		
B5	5	0.3546	0.07092	1.72E-07		
C1	5	0.3571	0.07142	3.2E-08		
C2	5	0.3563	0.07126	1.43E-07		
C3	5	0.3558	0.07116	7.3E-08		
C4	5	0.3521	0.07042	2.67E-07		
C5	5	0.3539	0.07078	6.2E-08		
D1	5	0.3569	0.07138	5.2E-08		
D2	5	0.3554	0.07108	7.7E-08		
D3	5	0.3525	0.0705	8E-08		
D4	5	0.3517	0.07034	9.3E-08		
D5	5	0.3529	0.07058	8.2E-08		
E1	5	0.3604	0.07208	6.7E-08		
E2	5	0.3535	0.0707	1.45E-07		
E3	5	0.3547	0.07094	2.53E-07		
E4	5	0.3515	0.0703	2.45E-07		
E5	5	0.3514	0.07028	2.7E-08		
F1	5	0.3603	0.07206	2.3E-08		
F2	5	0.3542	0.07084	1.33E-07		
F3	5	0.3552	0.07104	6.3E-08		
F4	5	0.3558	0.07116	2.53E-07		
F5	5	0.3533	0.07066	4.3E-08		
			0.07096667			

ANOVA

Source of variation	*sums of squares (SS)*	*degrees of freedom (df)*	*Mean squares (MS)*	*F-value*	*P-value*	*critical F-value*
Between groups	3.4773E-05	29	1.1991E-06	11.2589715	1.8322E-22	1.56207098
Within groups	0.00001278	120	1.065E-07			
Total	4.7553E-05	149				
within-sd	0.000326					
effective n	4.00					
s_bb	0.000523					
s_bb_min	5.86E-05					
u_bb	0.000523	0.522633				
u_bb(rel.)	0.73644838					

Iron:

Sample	*Number*	*Sum*	*Mean*	*Variance*		
A1	5	0.656	0.1312	9.385E-06		
A2	5	0.6686	0.13372	7.92E-07		
A3	5	0.6575	0.1315	2.185E-06		
A4	5	0.6766	0.13532	1.047E-06		
A5	5	0.6736	0.13472	4.867E-06		
B1	5	0.6694	0.13388	1.322E-06		
B2	5	0.6696	0.13392	2.547E-06		
B3	5	0.6701	0.13402	7.062E-06		
B4	5	0.6691	0.13382	2.417E-06		
B5	5	0.6665	0.1333	1.2235E-05		
C1	5	0.6646	0.13292	2.927E-06		
C2	5	0.6701	0.13402	5.912E-06		
C3	5	0.6629	0.13258	3.107E-06		
C4	5	0.6659	0.13318	5.632E-06		
C5	5	0.6693	0.13386	5.503E-06		
D1	5	0.6643	0.13286	2.288E-06		
D2	5	0.6737	0.13474	1.128E-06		
D3	5	0.6703	0.13406	3.928E-06		
D4	5	0.6634	0.13268	7.077E-06		
D5	5	0.664	0.1328	3.3E-06		
E1	5	0.6692	0.13384	4.283E-06		
E2	5	0.6632	0.13264	4.83E-07		
E3	5	0.6719	0.13438	6.102E-06		
E4	5	0.6649	0.13298	1.457E-06		
E5	5	0.6546	0.13092	5.007E-06		
F1	5	0.6625	0.1325	4.55E-06		
F2	5	0.6666	0.13332	3.567E-06		
F3	5	0.6708	0.13416	1.3183E-05		
F4	5	0.6788	0.13576	7.743E-06		
F5	5	0.6749	0.13498	6.282E-06		
			0.133486			
ANOVA						
Source of variation	*sums of squares (SS)*	*degrees of freedom (df)*	*Mean squares (MS)*	*F-value*	*P-value*	*critical F-value*
Between groups	0.000186489	29	6.43064E-06	1.40490862	0.10455229	1.56207098
Within groups	0.000549272	120	4.57727E-06			
Total	0.000735761	149				
within-sd	0.00213945					
effective n	4.00					
s_bb	0.00068069					
s_bb_min	0.00038436					
u_bb	0.00068069	0.68069353				
u_bb(rel.)	0.509936268					

Copper:

Sample	Number	Sum	Mean	Variance		
A1	5	6.8647	1.37294	2.9843E-05		
A2	5	6.9161	1.38322	1.3017E-05		
A3	5	6.8821	1.37642	3.0417E-05		
A4	5	6.9809	1.39618	3.9017E-05		
A5	5	6.9176	1.38352	6.4317E-05		
B1	5	6.8941	1.37882	1.9972E-05		
B2	5	6.9031	1.38062	1.9392E-05		
B3	5	6.8988	1.37976	3.3248E-05		
B4	5	6.8894	1.37788	1.3832E-05		
B5	5	6.9214	1.38428	5.3167E-05		
C1	5	6.8573	1.37146	2.8588E-05		
C2	5	6.9471	1.38942	2.5862E-05		
C3	5	6.9485	1.3897	5.253E-05		
C4	5	6.8785	1.3757	0.00010106		
C5	5	6.9008	1.38016	3.8243E-05		
D1	5	6.8567	1.37134	1.623E-06		
D2	5	6.9287	1.38574	1.7923E-05		
D3	5	6.8917	1.37834	1.2328E-05		
D4	5	6.8661	1.37322	5.0852E-05		
D5	5	6.8614	1.37228	3.0937E-05		
E1	5	6.9175	1.3835	2.916E-05		
E2	5	6.8756	1.37512	4.0557E-05		
E3	5	6.9249	1.38498	6.8207E-05		
E4	5	6.8811	1.37622	8.5377E-05		
E5	5	6.8476	1.36952	9.77E-07		
F1	5	6.9096	1.38192	3.5387E-05		
F2	5	6.9	1.38	6.4255E-05		
F3	5	6.935	1.387	3.006E-05		
F4	5	6.9556	1.39112	0.00011968		
F5	5	6.878	1.3756	0.00001126		
			1.380199333			
ANOVA						
Source of variation	*sums of squares (SS)*	*degrees of freedom (df)*	*Mean squares (MS)*	*F-value*	*P-value*	*critical F-value*
Between groups	0.00620945	29	0.000214119	5.53240853	9.6398E-12	1.56207098
Within groups	0.00464432	120	3.87027E-05			
Total	0.01085377	149				
within-sd	0.00622115					
effective n	4.00					
s_bb	0.00662224					
s_bb_min	0.00111764					
u_bb	0.00662224	6.6222409				
u_bb(rel.)	0.479803222					

Manganese:

Sample	*Number*	*Sum*	*Mean*	*Variance*		
A1	5	0.1793	0.03586	8E-09		
A2	5	0.1792	0.03584	3E-09		
A3	5	0.1791	0.03582	2.2E-08		
A4	5	0.1803	0.03606	8E-09		
A5	5	0.1785	0.0357	1E-08		
B1	5	0.1798	0.03596	3E-09		
B2	5	0.1792	0.03584	3E-09		
B3	5	0.1785	0.0357	0		
B4	5	0.1783	0.03566	3E-09		
B5	5	0.1793	0.03586	8E-09		
C1	5	0.1796	0.03592	7E-09		
C2	5	0.1795	0.0359	1E-08		
C3	5	0.1808	0.03616	3E-09		
C4	5	0.178	0.0356	2E-08		
C5	5	0.1791	0.03582	7E-09		
D1	5	0.1786	0.03572	1.7E-08		
D2	5	0.1795	0.0359	1E-08		
D3	5	0.1787	0.03574	8E-09		
D4	5	0.1777	0.03554	3E-09		
D5	5	0.1776	0.03552	1.2E-08		
E1	5	0.1806	0.03612	1.7E-08		
E2	5	0.178	0.0356	5E-09		
E3	5	0.1794	0.03588	1.7E-08		
E4	5	0.179	0.0358	1.5E-08		
E5	5	0.1774	0.03548	2E-09		
F1	5	0.1806	0.03612	1.7E-08		
F2	5	0.179	0.0358	5E-09		
F3	5	0.179	0.0358	1.5E-08		
F4	5	0.1799	0.03598	1.7E-08		
F5	5	0.1784	0.03568	2.2E-08		
			0.03581267			
ANOVA						
Source of variation	*sums of squares (SS)*	*degrees of freedom (df)*	*Mean squares (MS)*	*F-value*	*P-value*	*critical F-value*
Between groups	4.4979E-06	29	1.551E-07	15.6667828	1.4233E-28	1.56207098
Within groups	1.188E-06	120	9.9E-09			
Total	5.6859E-06	149				
within-sd	9.95E-05					
effective n	4.00					
s_bb	0.000191					
s_bb_min	1.79E-05					
u_bb	0.000191	0.190526				
u_bb(rel.)	0.53200825					

Magnesium:

Sample	Number	Sum	Mean	Variance		
A1	5	11.3248	2.26496	0.00012659		
A2	5	11.3954	2.27908	7.5727E-05		
A3	5	11.3668	2.27336	7.5713E-05		
A4	5	11.5002	2.30004	0.00013639		
A5	5	11.3902	2.27804	0.00019725		
B1	5	11.3574	2.27148	6.2597E-05		
B2	5	11.3438	2.26876	0.00010747		
B3	5	11.3581	2.27162	6.6052E-05		
B4	5	11.3733	2.27466	1.1848E-05		
B5	5	11.3902	2.27804	0.00016203		
C1	5	11.2954	2.25908	8.0477E-05		
C2	5	11.4269	2.28538	0.00012872		
C3	5	11.4328	2.28656	0.00011616		
C4	5	11.3369	2.26738	0.0001586		
C5	5	11.3675	2.2735	0.00014879		
D1	5	11.2971	2.25942	8.237E-06		
D2	5	11.4175	2.2835	0.00011511		
D3	5	11.357	2.2714	1.7245E-05		
D4	5	11.3248	2.26496	0.00016562		
D5	5	11.3103	2.26206	7.2268E-05		
E1	5	11.3875	2.2775	8.6915E-05		
E2	5	11.3037	2.26074	0.00013159		
E3	5	11.4086	2.28172	0.00014174		
E4	5	11.3708	2.27416	0.00020802		
E5	5	11.2977	2.25954	6.933E-06		
F1	5	11.37	2.274	0.00019753		
F2	5	11.3618	2.27236	0.00022285		
F3	5	11.4168	2.28336	9.7513E-05		
F4	5	11.4438	2.28876	0.00017456		
F5	5	11.3225	2.2645	5.316E-05		
			2.273664			
ANOVA						
Source of variation	*sums of squares (SS)*	*degrees of freedom (df)*	*Mean squares (MS)*	*F-value*	*P-value*	*critical F-value*
Between groups	0.01399097	29	0.00048245	4.31563837	6.9851E-09	1.56207098
Within groups	0.01341486	120	0.00011179			
Total	0.02740583	149				
within-sd	0.010573					
effective n	4.00					
s_bb	0.009626					
s_bb_min	0.001899					
u_bb	0.009626	9.626224				
u_bb(rel.)	0.42337936					

Chromium:

Sample	*Anzahl*	*Summe*	*Mittelwert*	*Varianz*
A1	5	0.9396	0.18792	1.762E-06
A2	5	0.9491	0.18982	1.067E-06
A3	5	0.9421	0.18842	1.317E-06
A4	5	0.962	0.1924	2.385E-06
A5	5	0.9481	0.18962	3.527E-06
B1	5	0.9445	0.1889	1.285E-06
B2	5	0.9413	0.18826	1.433E-06
B3	5	0.942	0.1884	1.34E-06
B4	5	0.9453	0.18906	3.18E-07
B5	5	0.9484	0.18968	2.707E-06
C1	5	0.9392	0.18784	7.23E-07
C2	5	0.9495	0.1899	1.51E-06
C3	5	0.9523	0.19046	1.373E-06
C4	5	0.9394	0.18788	6.7E-08
C5	5	0.9418	0.18836	9.58E-07
D1	5	0.9375	0.1875	4.25E-07
D2	5	0.9459	0.18918	1.092E-06
D3	5	0.9429	0.18858	3.62E-07
D4	5	0.9403	0.18806	1.883E-06
D5	5	0.9376	0.18752	1.237E-06
E1	5	0.9463	0.18926	1.783E-06
E2	5	0.9387	0.18774	6.78E-07
E3	5	0.9474	0.18948	1.167E-06
E4	5	0.945	0.189	2.33E-06
E5	5	0.9398	0.18796	4.73E-07
F1	5	0.9416	0.18832	3.647E-06
F2	5	0.9434	0.18868	1.527E-06
F3	5	0.9532	0.19064	4.073E-06
F4	5	0.9524	0.19048	2.217E-06
F5	5	0.9363	0.18726	1.073E-06
			0.188886	

ANOVA

Source of variation	*sums of squares (SS)*	*degrees of freedom (df)*	*Mean squares (MS)*	*F-value*	*P-value*	*critical F-value*
Between groups	0.00019018	29	6.5581E-06	4.30142088	7.5669E-09	1.56207098
Within groups	0.00018296	120	1.5246E-06			
Total	0.00037314	149				

within-sd	0.001235	
effective n	4.00	
s_bb	0.001122	
s_bb_min	0.000222	
u_bb	0.001122	1.121768
u_bb(rel.)	0.59388641	

Nickel:

Sample	Anzahl	Summe	Mittelwert	Varianz		
A1	5	0.0623	0.01246	1.3E-08		
A2	5	0.0625	0.0125	1E-08		
A3	5	0.0631	0.01262	2E-09		
A4	5	0.0633	0.01266	8E-09		
A5	5	0.0628	0.01256	2.3E-08		
B1	5	0.0628	0.01256	8E-09		
B2	5	0.0627	0.01254	3E-09		
B3	5	0.0625	0.0125	1.5E-08		
B4	5	0.0631	0.01262	2E-09		
B5	5	0.0622	0.01244	1.3E-08		
C1	5	0.0621	0.01242	2E-09		
C2	5	0.063	0.0126	1E-08		
C3	5	0.0631	0.01262	2.2E-08		
C4	5	0.0623	0.01246	3E-09		
C5	5	0.0623	0.01246	1.3E-08		
D1	5	0.0625	0.0125	5E-09		
D2	5	0.0627	0.01254	3E-09		
D3	5	0.0627	0.01254	3E-09		
D4	5	0.0623	0.01246	3E-09		
D5	5	0.0616	0.01232	1.2E-08		
E1	5	0.0622	0.01244	8E-09		
E2	5	0.0617	0.01234	3E-09		
E3	5	0.0627	0.01254	1.3E-08		
E4	5	0.0629	0.01258	7E-09		
E5	5	0.0618	0.01236	3E-09		
F1	5	0.0625	0.0125	5E-09		
F2	5	0.0628	0.01256	8E-09		
F3	5	0.0629	0.01258	7E-09		
F4	5	0.063	0.0126	2E-08		
F5	5	0.0622	0.01244	1.3E-08		
			0.01251067			
ANOVA						
Source of variation	*sums of squares (SS)*	*degrees of freedom (df)*	*Mean squares (MS)*	*F-value*	*P-value*	*critical F-value*
Between groups	1.0829E-06	29	3.7343E-08	4.30875332	7.261E-09	1.56207098
Within groups	0.00000104	120	8.6667E-09			
Total	2.1229E-06	149				
within-sd	9.31E-05					
effective n	4.00					
s_bb	8.47E-05					
s_bb_min	1.67E-05					
u_bb	8.47E-05	0.08467				
u_bb(rel.)	0.67678043					

Zinc:

Sample	Number	Sum	Mean	Variance
A1	5	28.1812	5.63624	0.00047581
A2	5	28.1009	5.62018	0.00073898
A3	5	28.5019	5.70038	0.00077322
A4	5	28.4207	5.68414	0.0009062
A5	5	28.3026	5.66052	0.00021765
B1	5	28.3586	5.67172	0.00041884
B2	5	28.2161	5.64322	0.00102941
B3	5	28.0179	5.60358	0.00013324
B4	5	28.4098	5.68196	0.00011307
B5	5	28.0508	5.61016	0.00075217
C1	5	28.0315	5.6063	0.00022306
C2	5	28.2464	5.64928	0.00053187
C3	5	28.3739	5.67478	0.00044465
C4	5	28.2454	5.64908	0.00059259
C5	5	28.1589	5.63178	0.00066269
D1	5	28.1833	5.63666	0.00030385
D2	5	28.312	5.6624	0.0003348
D3	5	28.273	5.6546	0.00027724
D4	5	28.1381	5.62762	0.00094975
D5	5	27.9381	5.58762	0.00051091
E1	5	28.1173	5.62346	0.00025768
E2	5	27.8586	5.57172	0.00077382
E3	5	28.2141	5.64282	0.00043467
E4	5	28.4948	5.69896	0.00075788
E5	5	28.1187	5.62374	0.00024128
F1	5	28.1557	5.63114	0.00081535
F2	5	28.3091	5.66182	0.00098003
F3	5	28.2447	5.64894	0.00031592
F4	5	28.2109	5.64218	0.00019843
F5	5	28.0872	5.61744	0.00047935
			5.64181467	

ANOVA

Source of variation	sums of squares (SS)	degrees of freedom (df)	Mean squares (MS)	F-value	P-value	critical F-value
Between groups	0.13677207	29	0.00471628	9.04400774	8.8274E-19	1.56207098
Within groups	0.06257772	120	0.00052148			
Total	0.19934979	149				

within-sd	0.022836	
effective n	4.00	
s_bb	0.032384	
s_bb_min	0.004103	
u_bb	0.032384	32.38363
u_bb(rel.)	0.5739931	

Titanium:

Sample	*Number*	*Sum*	*Mean*	*Variance*		
A1	5	0.143	0.0286	2.5E-08		
A2	5	0.1434	0.02868	4.7E-08		
A3	5	0.1432	0.02864	2.3E-08		
A4	5	0.1436	0.02872	1.2E-08		
A5	5	0.1433	0.02866	1.03E-07		
B1	5	0.1433	0.02866	1.8E-08		
B2	5	0.1433	0.02866	3.3E-08		
B3	5	0.1432	0.02864	8E-09		
B4	5	0.1429	0.02858	6.7E-08		
B5	5	0.1427	0.02854	1.3E-08		
C1	5	0.1436	0.02872	3.2E-08		
C2	5	0.1435	0.0287	1.35E-07		
C3	5	0.1429	0.02858	5.2E-08		
C4	5	0.1435	0.0287	5.5E-08		
C5	5	0.1427	0.02854	1.18E-07		
D1	5	0.1432	0.02864	3E-09		
D2	5	0.1426	0.02852	1.27E-07		
D3	5	0.1434	0.02868	1.52E-07		
D4	5	0.1427	0.02854	8.3E-08		
D5	5	0.1426	0.02852	5.7E-08		
E1	5	0.1437	0.02874	2.8E-08		
E2	5	0.1429	0.02858	4.7E-08		
E3	5	0.1427	0.02854	2.3E-08		
E4	5	0.1437	0.02874	2.3E-08		
E5	5	0.1424	0.02848	9.7E-08		
F1	5	0.1436	0.02872	2.2E-08		
F2	5	0.1432	0.02864	1.8E-08		
F3	5	0.1445	0.0289	5E-09		
F4	5	0.1442	0.02884	1.3E-08		
F5	5	0.1421	0.02842	5.2E-08		
			0.02863733			

ANOVA

Source of variation	*sums of squares (SS)*	*degrees of freedom (df)*	*Mean squares (MS)*	*F-value*	*P-value*	*critical F-value*
Between groups	1.5669E-06	29	5.4032E-08	1.08716668	0.36446954	1.56207098
Within groups	5.964E-06	120	4.97E-08			
Total	7.5309E-06	149				

within-sd	0.000223	
effective n	4.00	
s_bb	3.29E-05	
s_bb_min	4.01E-05	
u_bb	4.01E-05	0.040051
u_bb(rel.)	0.13985494	

Silver:

Sample	*Number*	*Sum*	*Mean*	*Variance*
A1	5	59.13	11.826	0.00358
A2	5	59.04	11.808	0.00352
A3	5	59.97	11.994	0.00233
A4	5	59.53	11.906	0.00188
A5	5	59.42	11.884	0.00583
B1	5	59.12	11.824	0.00683
B2	5	59.19	11.838	0.00277
B3	5	58.74	11.748	0.00352
B4	5	59.63	11.926	0.00248
B5	5	59.24	11.848	0.00447
C1	5	58.66	11.732	0.00167
C2	5	59.43	11.886	0.00708
C3	5	59.41	11.882	0.00817
C4	5	59.34	11.868	0.00107
C5	5	58.82	11.764	0.00313
D1	5	59.09	11.818	0.00602
D2	5	58.69	11.738	0.00217
D3	5	59.08	11.816	0.00468
D4	5	58.99	11.798	0.00337
D5	5	58.49	11.698	0.00177
E1	5	58.75	11.75	0.00135
E2	5	58.56	11.712	0.01037
E3	5	59.2	11.84	0.00235
E4	5	59.68	11.936	0.01243
E5	5	58.93	11.786	0.00333
F1	5	58.96	11.792	0.00567
F2	5	59.16	11.832	0.00387
F3	5	59.23	11.846	0.00093
F4	5	59.48	11.896	0.00613
F5	5	58.77	11.754	0.00173
			11.8248667	

ANOVA

Source of variation	*sums of squares (SS)*	*degrees of freedom (df)*	*Mean squares (MS)*	*F-value*	*P-value*	*critical F-value*
Between groups	0.72794733	29	0.02510163	6.04858607	6.92E-13	1.56207098
Within groups	0.498	120	0.00415			
Total	1.22594733	149				

within-sd	0.06442	
effective n	4.00	
s_bb	0.072373	
s_bb_min	0.011573	
u_bb	0.072373	72.37339
u_bb(rel.)	0.61204405	

Beryllium:

Sample	Number	Sum	Mean	Variance		
A1	5	9.53	1.906	0.00023		
A2	5	9.53	1.906	0.00013		
A3	5	9.59	1.918	7E-05		
A4	5	9.58	1.916	0.00013		
A5	5	9.52	1.904	8E-05		
B1	5	9.56	1.912	0.00012		
B2	5	9.58	1.916	0.00013		
B3	5	9.49	1.898	0.00002		
B4	5	9.55	1.91	0.00015		
B5	5	9.54	1.908	0.00022		
C1	5	9.53	1.906	8E-05		
C2	5	9.57	1.914	8E-05		
C3	5	9.63	1.926	8E-05		
C4	5	9.52	1.904	3E-05		
C5	5	9.52	1.904	0.00018		
D1	5	9.5	1.9	0.00025		
D2	5	9.57	1.914	8E-05		
D3	5	9.58	1.916	0.00018		
D4	5	9.52	1.904	3E-05		
D5	5	9.46	1.892	0.00012		
E1	5	9.58	1.916	0.00033		
E2	5	9.48	1.896	0.00013		
E3	5	9.56	1.912	0.00012		
E4	5	9.56	1.912	7E-05		
E5	5	9.5	1.9	5E-05		
F1	5	9.61	1.922	7E-05		
F2	5	9.54	1.908	7E-05		
F3	5	9.56	1.912	0.00012		
F4	5	9.59	1.918	7E-05		
F5	5	9.51	1.902	0.00027		
			1.90906667			
ANOVA						
Source of variation	sums of squares (SS)	degrees of freedom (df)	Mean squares (MS)	F-value	P-value	critical F-value
Between groups	0.00910933	29	0.00031411	2.55378002	0.0002042	1.56207098
Within groups	0.01476	120	0.000123			
Total	0.02386933	149				
within-sd	0.011091					
effective n	4.00					
s_bb	0.006912					
s_bb_min	0.001992					
u_bb	0.006912	6.912216				
u_bb(rel.)	0.36207308					

Calcium:

Sample	*Number*	*Sum*	*Mean*	*Variance*		
A1	5	61.11	12.222	0.01167		
A2	5	60.55	12.11	0.01115		
A3	5	61.81	12.362	0.03492		
A4	5	61.72	12.344	0.01813		
A5	5	60.45	12.09	0.0305		
B1	5	61.07	12.214	0.00963		
B2	5	60.8	12.16	0.00255		
B3	5	59.85	11.97	0.01265		
B4	5	61.21	12.242	0.00677		
B5	5	60.33	12.066	0.01108		
C1	5	60.89	12.178	0.01052		
C2	5	61.72	12.344	0.01313		
C3	5	61.33	12.266	0.02563		
C4	5	60.85	12.17	0.01485		
C5	5	59.65	11.93	0.0082		
D1	5	61.18	12.236	0.01123		
D2	5	60.79	12.158	0.01067		
D3	5	60.64	12.128	0.00962		
D4	5	60.11	12.022	0.01527		
D5	5	59.29	11.858	0.00557		
E1	5	60.63	12.126	0.01303		
E2	5	60.24	12.048	0.01587		
E3	5	60.42	12.084	0.01013		
E4	5	61.44	12.288	0.03697		
E5	5	59.94	11.988	0.00432		
F1	5	61.38	12.276	0.00648		
F2	5	60.95	12.19	0.01075		
F3	5	60.56	12.112	0.00302		
F4	5	60.85	12.17	0.02995		
F5	5	59.71	11.942	0.00587		
			12.1431333			
ANOVA						
Source of variation	*sums of squares (SS)*	*degrees of freedom (df)*	*Mean squares (MS)*	*F-value*	*P-value*	*critical F-value*
Between groups	2.38570733	29	0.08226577	6.01753859	8.0865E-13	1.56207098
Within groups	1.64052	120	0.013671			
Total	4.02622733	149				
within-sd	0.116923					
effective n	4.00					
s_bb	0.130953					
s_bb_min	0.021005					
u_bb	0.130953	130.953				
u_bb(rel.)	1.07841208					

Sodium:

Sample	Number	Sum	Mean	Variance		
A1	5	70.45	14.09	0.0225		
A2	5	66.73	13.346	0.00748		
A3	5	64.81	12.962	0.00687		
A4	5	63.41	12.682	0.01887		
A5	5	60.74	12.148	0.02642		
B1	5	70.1	14.02	0.0221		
B2	5	66.6	13.32	0.0046		
B3	5	64.55	12.91	0.02985		
B4	5	62.66	12.532	0.01087		
B5	5	60.48	12.096	0.00253		
C1	5	70	14	0.0125		
C2	5	67.43	13.486	0.03463		
C3	5	65.78	13.156	0.03333		
C4	5	62.28	12.456	0.00883		
C5	5	60.09	12.018	0.00247		
D1	5	69.86	13.972	0.00827		
D2	5	66.52	13.304	0.00288		
D3	5	64.57	12.914	0.00803		
D4	5	62.07	12.414	0.02183		
D5	5	59.93	11.986	0.00343		
E1	5	70.17	14.034	0.00763		
E2	5	66.19	13.238	0.01937		
E3	5	65.06	13.012	0.01122		
E4	5	62.66	12.532	0.02427		
E5	5	59.86	11.972	0.00592		
F1	5	70.39	14.078	0.03212		
F2	5	67.03	13.406	0.01323		
F3	5	65.61	13.122	0.00947		
F4	5	63.31	12.662	0.02472		
F5	5	60.09	12.018	0.01152		
			12.9962			
ANOVA						
Source of variation	sums of squares (SS)	degrees of freedom (df)	Mean squares (MS)	F-value	P-value	critical F-value
Between groups	70.410494	29	2.42794807	162.672954	6.7495E-83	1.56207098
Within groups	1.79104	120	0.01492533			
Total	72.201534	149				
within-sd	0.122169					
effective n	4.00					
s_bb	0.776695					
s_bb_min	0.021948					
u_bb	0.776695	776.6954				
u_bb(rel.)	5.97632663					

Lead:

Sample	*Number*	*Sum*	*Mean*	*Variance*
A1	5	0.0196	0.00392	2E-09
A2	5	0.0197	0.00394	3E-09
A3	5	0.0203	0.00406	3E-09
A4	5	0.0199	0.00398	2E-09
A5	5	0.0198	0.00396	3E-09
B1	5	0.0198	0.00396	3E-09
B2	5	0.02	0.004	0
B3	5	0.0194	0.00388	2E-09
B4	5	0.0199	0.00398	2E-09
B5	5	0.0196	0.00392	2E-09
C1	5	0.0196	0.00392	2E-09
C2	5	0.0198	0.00396	3E-09
C3	5	0.0197	0.00394	3E-09
C4	5	0.0198	0.00396	3E-09
C5	5	0.0194	0.00388	2E-09
D1	5	0.0198	0.00396	3E-09
D2	5	0.0194	0.00388	2E-09
D3	5	0.0197	0.00394	3E-09
D4	5	0.0196	0.00392	2E-09
D5	5	0.0192	0.00384	3E-09
E1	5	0.0196	0.00392	2E-09
E2	5	0.0193	0.00386	3E-09
E3	5	0.0197	0.00394	3E-09
E4	5	0.0198	0.00396	3E-09
E5	5	0.0195	0.0039	0
F1	5	0.0195	0.0039	0
F2	5	0.0198	0.00396	3E-09
F3	5	0.0195	0.0039	0
F4	5	0.0198	0.00396	3E-09
F5	5	0.0197	0.00394	3E-09
			0.00393467	

ANOVA

Source of variation	*sums of squares (SS)*	*degrees of freedom (df)*	*Mean squares (MS)*	*F-value*	*P-value*	*critical F-value*
Between groups	2.8773E-07	29	9.9218E-09	4.37728195	4.9414E-09	1.56207098
Within groups	2.72E-07	120	2.2667E-09			
Total	5.5973E-07	149				

within-sd	4.76E-05	
effective n	4.00	
s_bb	4.37E-05	
s_bb_min	8.55E-06	
u_bb	4.37E-05	0.043747
u_bb(rel.)	1.11183297	

Zirconium:

Sample	*Number*	*Sum*	*Mean*	*Variance*		
A1	5	0.0437	0.00874	3E-09		
A2	5	0.044	0.0088	0		
A3	5	0.0442	0.00884	3E-09		
A4	5	0.0445	0.0089	0		
A5	5	0.0439	0.00878	2E-09		
B1	5	0.044	0.0088	0		
B2	5	0.044	0.0088	0		
B3	5	0.0439	0.00878	2E-09		
B4	5	0.0439	0.00878	2E-09		
B5	5	0.044	0.0088	0		
C1	5	0.0441	0.00882	2E-09		
C2	5	0.0443	0.00886	3E-09		
C3	5	0.0447	0.00894	3E-09		
C4	5	0.0437	0.00874	3E-09		
C5	5	0.044	0.0088	0		
D1	5	0.0438	0.00876	3E-09		
D2	5	0.0442	0.00884	3E-09		
D3	5	0.044	0.0088	5E-09		
D4	5	0.0438	0.00876	3E-09		
D5	5	0.0437	0.00874	3E-09		
E1	5	0.044	0.0088	0		
E2	5	0.0436	0.00872	2E-09		
E3	5	0.0441	0.00882	2E-09		
E4	5	0.0441	0.00882	2E-09		
E5	5	0.0438	0.00876	3E-09		
F1	5	0.0442	0.00884	3E-09		
F2	5	0.044	0.0088	0		
F3	5	0.0441	0.00882	2E-09		
F4	5	0.0443	0.00886	3E-09		
F5	5	0.0437	0.00874	3E-09		
			0.008802			

ANOVA

Source of variation	*sums of squares (SS)*	*degrees of freedom (df)*	*Mean squares (MS)*	*F-value*	*P-value*	*critical F-value*
Between groups	3.494E-07	29	1.2048E-08	6.02413793	7.8229E-13	1.56207098
Within groups	2.4E-07	120	2E-09			
Total	5.894E-07	149				

within-sd	4.47E-05	
effective n	4.00	
s_bb	5.01E-05	
s_bb_min	8.03E-06	
u_bb	5.01E-05	0.050121
u_bb(rel.)	0.56942223	

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

Silicon:

r_0	0.059609905	0.060390095														
r_in	0.061	0.061	0.061	0.061	0.061	0.061	0.061	0.061								
r_out	0.061	0.063	0.062	0.062	0.061	0.063	0.062	0.062	0.062	0.061	0.061	0.062	0.062	0.062	0.061	0.061
Source of variation	*sums of squares (SS)*	*degrees of freedom (df)*	*Mean squares (MS)*	*F-value*	*P-value*	*critical F-value*										
Between groups	7.15385E-06	2	3.57692E-06	11.26304945	0.000388933	3.422132208										
Within groups	7.30435E-06	23	3.1758E-07													
Total	1.44582E-05	25														
within-sd	0.000563543															
effective n	6.77															
s_bb	0.000693898															
s_bb_min	0.00011762															
u_bb	0.000693898			0.061363636												
u_bb(rel.)	1.130796011															

Iron:

r_0	0.154743756	0.165256244														
r_in	0.153	0.159	0.155	0.151	0.154	0.155	0.158	0.155								
r_out	0.155	0.16	0.158	0.157	0.154	0.157	0.158	0.159	0.157	0.156	0.158	0.155	0.158	0.161	0.161	0.158
Source of variation	*sums of squares (SS)*	*degrees of freedom (df)*	*Mean squares (MS)*	*F-value*	*P-value*	*critical F-value*										
Between groups	5.625E-05	2	2.8125E-05	3.968407064	0.033067461	3.422132208										
Within groups	0.000163006	23	7.08723E-06													
Total	0.000219256	25														
within-sd	0.002662185															
effective n	6.77															
s_bb	0.00176291															
s_bb_min	0.000555641															
u_bb	0.00176291			0.156545455												
u_bb(rel.)	1.126133207															

Copper:

r_0	1.519760748	1.560239252													
r_in	1.56	1.55	1.57	1.56	1.57	1.57	1.58	1.57							
r_out	1.59	1.55	1.57	1.58	1.58	1.55	1.57	1.58	1.57	1.6	1.58	1.6	1.58	1.59	1.59

Source of variation	*sums of squares (SS)*	*degrees of freedom (df)*	*Mean squares (MS)*	*F-value*	*P-value*	*critical F-value*
Between groups	0.002975167	2	0.001487583	7.145459518	0.004063234	3.443356779
Within groups	0.004580088	22	0.000208186			
Total	0.007555255	24				

within-sd	0.014428646			
effective n	6.64			
s_bb	0.013880935			
s_bb_min	0.003074634			
u_bb	0.013880935			1.569545455
u_bb(rel.)	0.884392018			

Manganese:

r_0	0.03072172	0.03127828														
r_in	0.0311	0.0309	0.0308	0.0311	0.031	0.0311	0.0308	0.031								
r_out	0.0311	0.0312	0.0313	0.0307	0.0313	0.0311	0.0313	0.0308	0.0309	0.0311	0.031	0.031	0.0309	0.0308	0.0312	0.031

reuungsursac	*dratsummen (*	*heitsgrade (*	*e Quadratsum*	*rüfgröße (F)*	*P-Wert*	*tischer F-Wert*
Unterschiede	2.60096E-08	2	1.30048E-08	0.369612425	0.695032277	3.422132208
Innerhalb de	8.09255E-07	23	3.5185E-08			
Gesamt	8.35264E-07	25				

within-sd	0.000187577			
effective n	6.77			
s_bb	0			
s_bb_min	3.91503E-05			
u_bb	3.91503E-05			0.031027273
u_bb(rel.)	0.126180128			

Magnesium:

r_0	2.385077761	2.434922239													
r_in	2.44	2.44	2.44	2.44	2.45	2.46	2.45	2.42							
r_out	2.47	2.43	2.46	2.46	2.45	2.42	2.45	2.45	2.45	2.47	2.47	2.47	2.45	2.47	2.47

Source of variation	*sums of squares (SS)*	*degrees of freedom (df)*	*Mean squares (MS)*	*F-value*	*P-value*	*critical F-value*
Between groups	0.00409	2	0.002045	8.103041693	0.002307766	3.443356779
Within groups	0.005552236	22	0.000252374			
Total	0.009642236	24				

within-sd	0.015886295			
effective n	6.64			
s_bb	0.016430878			
s_bb_min	0.003385247			
u_bb	0.016430878			2.445909091
u_bb(rel.)	0.671769761			

Chromium:

r_0	0.198550005	0.201449995													
r_in	0.197	0.198	0.197	0.197	0.197	0.197	0.196	0.198							
r_out	0.199	0.199	0.199	0.197	0.198	0.199	0.198	0.197	0.198	0.197	0.196	0.195	0.197	0.197	0.197

Source of variation	*sums of squares (SS)*	*degrees of freedom (df)*	*Mean squares (MS)*	*F-value*	*P-value*	*critical F-value*
Between groups	1.33917E-05	2	6.69583E-06	5.493852708	0.011608505	3.443356779
Within groups	2.68133E-05	22	1.21879E-06			
Total	4.0205E-05	24				

within-sd	0.001103987			
effective n	6.64			
s_bb	0.000908216			
s_bb_min	0.000235251			
u_bb	0.000908216			0.197681818
u_bb(rel.)	0.459433292			

Zinc:

r_in	6.01	6.02	6.03	6.01	6.01	6.07	6.03	5.97							
r_out	6.02	5.96	6.03	6.01	6	5.95	6.02	6.02	6.01	6.05	6.03	6.04	6	6.06	6.08
Source of variation	*sums of squares (SS)*	*degrees of freedom (df)*	*Mean squares (MS)*	*F-value*	*P-value*	*critical F-value*									
Between groups	0.004363167	2	0.002181583	1.673128773	0.21066734	3.443356779									
Within groups	0.028685678	22	0.001303894												
Total	0.033048845	24													
within-sd	0.036109479														
effective n	6.64														
s_bb	0.011497046														
s_bb_min	0.007694652														
u_bb	0.011497046			6.010454545											
u_bb(rel.)	0.19128413														

Nickel:

r_0	0.012474405	0.013525595														
r_in	0.0124	0.0124	0.0128	0.0129	0.013	0.0128	0.0128	0.0124								
r_out	0.0123	0.0121	0.0122	0.013	0.0127	0.013	0.0128	0.013	0.013	0.0128	0.013	0.0128	0.013	0.013	0.0129	0.013
reuungsursac	*dratsummen (*	*heitsgrade (*	*e Quadratsum*	*rüfgröße (F)*	*P-Wert*	*tischer F-Wert*										
Unterschiede	1.64904E-07	2	8.24519E-08	0.784038958	0.468383443	3.422132208										
Innerhalb de	2.41875E-06	23	1.05163E-07													
Gesamt	2.58365E-06	25														
within-sd	0.000324289															
effective n	6.77															
s_bb	0															
s_bb_min	6.76842E-05															
u_bb	6.76842E-05			0.012736364												
u_bb(rel.)	0.531425027															

Titanium:

r_0	0.02885792	0.03114208													
r_in	0.029	0.028	0.028	0.028	0.029	0.029	0.029	0.029							
r_out	0.029	0.03	0.029	0.029	0.029	0.031	0.029	0.029	0.028	0.03	0.029	0.029	0.028	0.028	0.028

Source of variation	*sums of squares (SS)*	*degrees of freedom (df)*	*Mean squares (MS)*	*F-value*	*P-value*	*critical F-value*
Between groups	3.085E-06	2	1.5425E-06	2.342979362	0.119546972	3.443356779
Within groups	1.44837E-05	22	6.5835E-07			
Total	1.75687E-05	24				

within-sd	0.000811388			status:	homogeneous
effective n	6.64				
s_bb	0.000364904				
s_bb_min	0.0001729				
u_bb	0.000364904			0.029090909	
u_bb(rel.)	1.254358541				

Silver:

r_0	8.935918269	10.50408173													
r_in	10.48	9.22	10.07	9.96	10.1	10.1	10.22	9.62							
r_out	10.53	10.19	10.21	10.06	10.21	10.53	10.5	10.01	10.07	10.11	9.89	10.15	10.25	10.32	10.54

Source of variation	*sums of squares (SS)*	*degrees of freedom (df)*	*Mean squares (MS)*	*F-value*	*P-value*	*critical F-value*
Between groups	0.7039365	2	0.35196825	2.689303905	0.090182967	3.443356779
Within groups	2.879295823	22	0.130877083			
Total	3.583232323	24				

within-sd	0.361769378			
effective n	6.64			
s_bb	0.182474279			
s_bb_min	0.077090275			
u_bb	0.182474279			10.07590909
u_bb(rel.)	1.810995687			

Beryllium:

r_0	2.266931196	2.293068804													
r_in	2.34	2.31	2.33	2.32	2.32	2.31	2.31	2.28							
r_out	2.4	2.38	2.39	2.38	2.39	2.37	2.39	2.37	2.37	2.34	2.34	2.35	2.35	2.37	2.38

reuungsursac	*dratsummen (*	*heitsgrade (*	*e Quadratsum*	*Prüfgröße (F)*	*P-Wert*	*tischer F-Wert*
Unterschiede	0.026026667	2	0.013013333	38.09665428	7.14083E-08	3.443356779
Innerhalb de	0.007514921	22	0.000341587			
Gesamt	0.033541587	24				
within-sd	0.018482081					
effective n	6.64					
s_bb	0.043685186			u_bb(rel.)	1.864353239	
s_bb_min	0.003938389					
u_bb	0.043685186			2.343181818		

Calcium:

r_0	15.4301353	15.6898647													
r_in	16.03	15.8	15.93	15.95	15.91	15.92	16.12	15.96							
r_out	15.96	15.97	15.96	15.91	16.39	15.91	16.24	16.2	16.14	16.21	16.28	16.48	16.5	16.17	16.51

reuungsursac	*dratsummen (*	*heitsgrade (*	*e Quadratsum*	*Prüfgröße (F)*	*P-Wert*	*tischer F-Wert*
Unterschiede	0.840580667	2	0.420290333	12.46054815	0.000240769	3.443356779
Innerhalb de	0.742053016	22	0.033729683			
Gesamt	1.582633683	24				
within-sd	0.183656425					
effective n	6.64					
s_bb	0.241281921			u_bb(rel.)	1.506343048	
s_bb_min	0.039135773					
u_bb	0.241281921			16.01772727		

Sodium:

r_0	6.888209888	7.111790112														
r_in	6.99	7.24	7.06	6.87	7.02	7.21	7.18	7.2								
r_out	7.03	6.83	7.07	7.17	7.26	7.2	7.17	7.26	7.26	7.28	7.65	7.35	7.3	7.26	7.2	7.16
Source of variation	*sums of squares (SS)*	*degrees of freedom (df)*	*Mean squares (MS)*	*F-value*	*P-value*	*critical F-value*										
Between groups	0.133080288	2	0.066540144	2.620698677	0.094336341	3.422132208										
Within groups	0.583975308	23	0.025390231													
Total	0.717055597	25														
within-sd	0.159343123															
effective n	6.77															
s_bb	0.077967714			u_bb(rel.)	1.090457544											
s_bb_min	0.03325747															
u_bb	0.077967714			7.15												

Lead:

r_0	0.004738995	0.005061005						
r_in	0.0051	0.0051	0.0047	0.0053	0.0049	0.0051	0.0057	0.005
r_out	0.0054	0.0055	0.0054	0.0054	0.0053	0.0049	0.0052	0.0053
Source of variation	*sums of squares (SS)*	*degrees of freedom (df)*	*Mean squares (MS)*	*F-value*	*P-value*	*critical F-value*		
Between groups	3.07361E-07	2	1.53681E-07	2.559649703	0.110557457	3.682320344		
Within groups	9.00595E-07	15	6.00397E-08					
Total	1.20796E-06	17						
within-sd	0.00024503							
effective n	5.33							
s_bb	0.000132505			u_bb(rel.)	2.561864686			
s_bb_min	6.41142E-05							
u_bb	0.000132505			0.005172222				

Zirconium:

r_0	92.85034655	94.14965345													
r_in	92.7	89.9	90.8	91.4	91	89.8	90.6	91.7							
r_out	91.1	91.6	91.8	91	91.6	92.6	90.8	91.1	91.4	89.8	90.1	89.2	91.1	91.9	92.1
reuungsursac	*dratsummen (*	*heitsgrade (*	*e Quadratsum*	*rüfgröße (F)*	*P-Wert*	*itischer F-Wert*									
Unterschiede	10.80751667	2	5.403758333	6.401804779	0.006438465	3.443356779									
Innerhalb de	18.57018254	22	0.844099206												
Gesamt	29.37769921	24													
within-sd	0.918748718														
effective n	6.64														
s_bb	0.828671015			u_bb(rel.)	0.908358861										
s_bb_min	0.195778292														
u_bb	0.828671015			91.22727273											