

Certification Report

Certified Reference Material

BAM-M320

Al-Alloy 5028 AlMgSc

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Coordinator: Dr. Sebastian Recknagel
Bundesanstalt für Materialforschung und -prüfung (BAM)
Division 1.6 „Inorganic Reference Materials“
Richard-Willstätter-Str. 11
D-12489 Berlin
Phone: +49 30 8104 1111
Fax: +49 30 8104 71111
E-mail: sebastian.recknagel@bam.de

Summary

This report describes preparation, analysis and certification of the aluminium alloy reference material BAM-M320. 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 validation and quality control of wet chemical analysis methods. The following mass fractions and uncertainties have been certified:

Element	Mass fraction ¹⁾ in %	Uncertainty ²⁾ in %
Si	0.197	0.007
Fe	0.206	0.006
Cu	0.147	0.005
Mn	0.699	0.008
Mg	3.98	0.08
Cr	0.1044	0.0023
Zn	0.252	0.006
Ti	0.102	0.004
Ga	0.0208	0.0008
Sc	0.282	0.007
Zr	0.102	0.005
	in mg/kg	in mg/kg
Ni	20.9	1.3
Be	22.4	0.7
Ca	11.7	2.2
Cd	15.2	1.9
Co	20.9	1.2
Li	9.1	0.4
Na	6.4	1.3
Pb	44.8	2.4
Sn	45.6	2.9
V	75.9	2.5

- 1 Unweighted mean value of the means of accepted sets of data (consisting of at least 6 single results), each set being obtained by a different laboratory and/or a different method of measurement.
- 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.

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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 – 22)
I(R)	ICP-OES, revised value (Tables 2 – 22)
IMS	ICP-MS (Tables 2 – 22)
A	FAAS (Tables 2 – 22)
EA	ETAAS (Tables 2 – 22)
P	spectrophotometry (Tables 2 – 22)
-s	dissolution in acid (Tables 2 – 22)
-a	dissolution in base (Tables 2 – 22)

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-M320 is based on the aluminium alloy AA5028 AlMgSc, which is mainly used in the aircraft industry.

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 Aluminum Duffel BVBA, Duffel, Belgium
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
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).

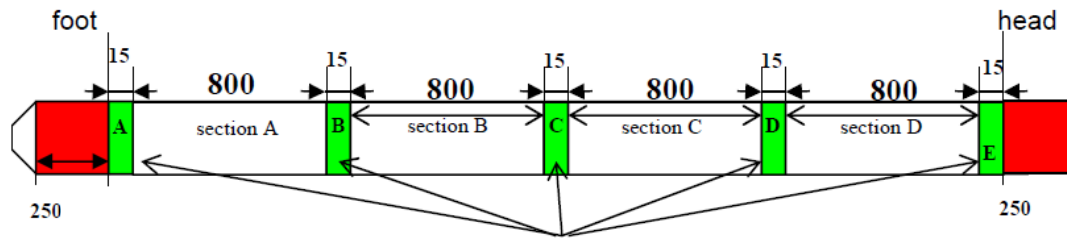


Fig.1: Preparation of the rods cast (all figures in mm)

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):

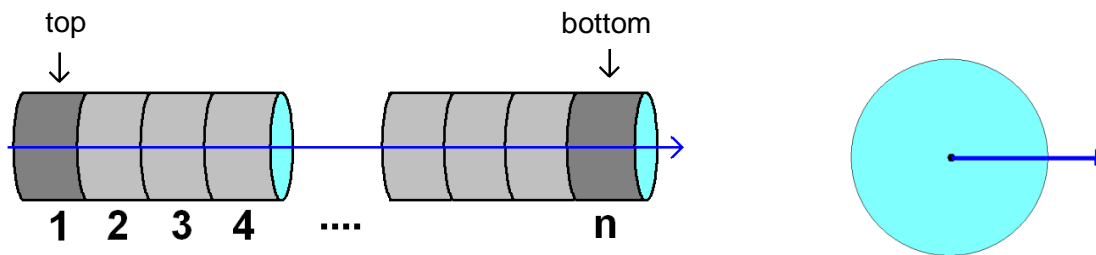


Fig. 2: Axial and radial composition gradient

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. For the elements Zn and Sc XRF was used to investigate homogeneity. This was done in BAM. 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, mean circle: 16 sparks, inner circle: 8 sparks; centre: 1 spark). For Sc data from the accompanying spark emission round robin test was used because there was no Sc-channel implemented in the BAM-spectrometer. Calculation was done in the same way as for the other elements while the number of sparks were different (outer circle: 4 sparks, inner circle: 4 sparks; centre: 1 spark).

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 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), r_mean (mean 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 uncertainty calculation.

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, Sc, Zr	0.5 g	Dissolution with NaOH	ICP-OES, commercial mono-element solutions, matrix matching with pure Al
	Ni, Be, Ca, Cd, Co, Li, Na, Pb, Sn, V	0.5 g	Dissolution with HCl/HNO ₃	ICP-OES, commercial mono-element solutions, matrix matching with pure Al
2	Si, Fe, Cu, Mn, Mg, Cr, Zn, Ga	0.5 g	Dissolution with NaOH	ICP-OES, calibration with pure metals or pure chemicals, matrix matching with pure Al (5N5)
	Ti, Sc, Zr, Ni, Ca, Na	0.5 g	Dissolution with HNO ₃ /HF	ICP-OES, calibration with pure metals or chemicals, matrix matching with pure Al (5N5)
	Be, Cd, V	0.5 g	Dissolution with HNO ₃ /HF	ICP-OES, commercial mono-element solution (Merck certipur), matrix matching with pure Al (5N5)
	Co, Li, Pb, Sn	0.5 g	Dissolution with HNO ₃ /HF	ICP-MS, 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, matrix matching with pure Al
	Fe, Cu, Mn, Mg, Cr, Zn, Ti, Ga, Sc, Zr	0.2 g	Dissolution with HNO ₃ /HCl	ICP-OES, commercial mono-element solutions, matrix matching with pure Al
	Ni, Be, Ca, Cd, Co, Li, Na, Pb, Sn, V	1 g	Dissolution with HNO ₃ /HCl	ICP-OES, commercial mono-element solutions, matrix matching with pure Al
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, Cr, Ti, Sc, Zr; Ni, Be, Sn	0.5 g	Dissolution with NaOH	ICP-OES, calibration with pure metals or pure chemicals, matrix matching with pure Al
	Cu, Mn, Mg, Zn, Ga, Ca, Cd, Co, Li, Na, Pb, V	0.5 g	Dissolution with HCl	ICP-OES, calibration with pure metals or pure chemicals, matrix matching with pure Al
7	Si, Fe, Cu, Mn, Mg, Cr, Zn, Ti, Ga, Sc, Zr, Ni, Cd, Co, Pb, Sn, V	0.5 g	Dissolution with HNO ₃ /HCl/HF	ICP-OES, calibration with matrix matched standards, commercial multi-element standard solutions
	Ga, Ni, Be, Cd, Li, Pb, Sn, V	0.5 g	Dissolution with HNO ₃ /HCl/HF	ICP-MS, with matrix matched standards, commercial mono-element standard solutions (Perkin Elmer)

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

Lab-No.	Element	Sample mass	Sample pretreatment	Analytical method
8	Si, Fe, Cu, Mn, Mg, Cr, Zn, Ti, Ga, Zr, Ni, Co, Pb, Sn, V	0.3 g	Dissolution with NaOH/HNO ₃	ICP-OES with matrix matched standards, commercial mono-element solutions (Merck)
	Fe, Cu, Mn, Mg, Cr, Zn, Ti, Ga, Zr, Ni, Cd, Co, Pb, Sn, V	0.3 g	Dissolution with HCl/H ₂ O ₂	ICP-OES with matrix matched standards, commercial mono-element solutions (Merck)
9	Si, Mn	0.25 g	Dissolution with NaOH	Spectrophotometry, calibration with commercial mono-element solutions (Merck)
	Fe	0.5 g	Dissolution with HCl/H ₂ O ₂	Spectrophotometry, calibration with commercial mono-element solutions (Merck)
	Zr	0.5 g	Dissolution with NaOH,	Spectrophotometry, calibration with commercial mono-element solutions (Merck)
	Fe, Cu, Mn, Mg, Zn	1 g	Dissolution with HCl/H ₂ O ₂	FAAS, calibration with matrix matched standards, calibration with commercial mono-element solutions (Merck)
	Na	0.25 g	Dissolution with HCl/HNO ₃ /HF	ETAAS, calibration with commercial mono-element solution (Merck)
	Cu, Mg, Cr, Zn, Ti, Ga, Sc, Zr, Ni, Be, Cd, Co, Li, Pb, Sn, Zr	1 g	Dissolution with HCl/HNO ₃ /HF	ICP- OES, calibration with matrix matched standards, commercial mono-element solutions
	Mg	0.25 g	Dissolution with NaOH	ICP-OES, calibration with matrix matched standards, commercial mono-element solution
10	Si, Fe, Cu, Mn, Mg, Cr, Zn, Ti, Ga, Sc, Zr, Ni, Be, Cd, Co, Sn, V	0.25 g	Dissolution with NaOH/HNO ₃	ICP-OES, calibration with commercial mono-element solutions
11	Si, Fe, Cu, Mn, Mg, Cr, Ti, Ga, Sc	0.5 g	Dissolution with NaOH	ICP-OES, calibration with commercial mono-element solutions
	Ni, Be, Ca, Co, Li, Na, V	1 g	Dissolution with HCl/HNO ₃	ICP-OES, calibration with commercial mono-element solutions
	Fe, Cu, Cr, Zn, Ti, Sc, Zr	1 g	Dissolution with HCl/HNO ₃	ICP-OES, calibration with commercial mono-element solutions
	Ga, Ni, Be, Cd, Co, Pb, Sn	1 g	Dissolution with HCl/HNO ₃	ICP-MS, calibration with commercial mono-element solutions
12	Si, Fe, Cu, Mn, Mg, Cr, Zn, Ga, Sc, Ti, Zr, Ni, Ca, Cd, Co, Li, Pb, Sn, V	0.5 g	Dissolution with NaOH/HNO ₃	ICP-OES, calibration with matrix matched standards, commercial mono-element solutions (Merck certipur)
15	Si, Fe, Cu, Mn, Mg, Cr, Zn, Ti, Zr, Ni, Ca, Cd, Li, Pb, Sn, V	0.5 g	Dissolution with NaOH	ICP-OES, calibration with matrix matched standards, commercial mono-element solutions (Merck certipur)

5.2 Analytical results and statistical evaluation

The analytical results of the inter-laboratory certification comparison are listed in Tables 2 to 22. 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/l-a	5/l-a	1/l-a	2/l-a	9/P	15/l-a	10/l-a	6/l-a	12/l-a	7/l-s	4/P	11/l-a		
M_i [%]	0.1894	0.189	0.190	0.193	0.195	0.194	0.197	0.200	0.201	0.204	0.206	0.209		n 12
	0.1884	0.191	0.189	0.192	0.195	0.197	0.195	0.202	0.203	0.202	0.201	0.211		
	0.1902	0.190	0.190	0.192	0.195	0.196	0.196	0.198	0.201	0.203	0.202	0.210		
	0.1899	0.189	0.191	0.192	0.195	0.194	0.200	0.194	0.200	0.202	0.200	0.211		
	0.1875	0.190	0.190	0.188	0.194	0.197	0.196	0.197	0.201	0.204	0.203	0.204		
	0.1879	0.192	0.190	0.191	0.191	0.195	0.197	0.196	0.195	0.200	0.204	0.207	0.210	
					0.194									
M [%]	0.189	0.190	0.190	0.191	0.195	0.196	0.197	0.198	0.201	0.203	0.203	0.209		0.197
s [%]	0.0011	0.0010	0.0004	0.0018	0.0006	0.0015	0.0018	0.0029	0.0010	0.0009	0.0028	0.0027	s_M [%]	0.0063
s_{rel}	0.00587	0.00551	0.00235	0.00929	0.00312	0.00765	0.00890	0.01459	0.00475	0.00453	0.01372	0.01275	\bar{s}_i [%]	0.0017
														0.03217

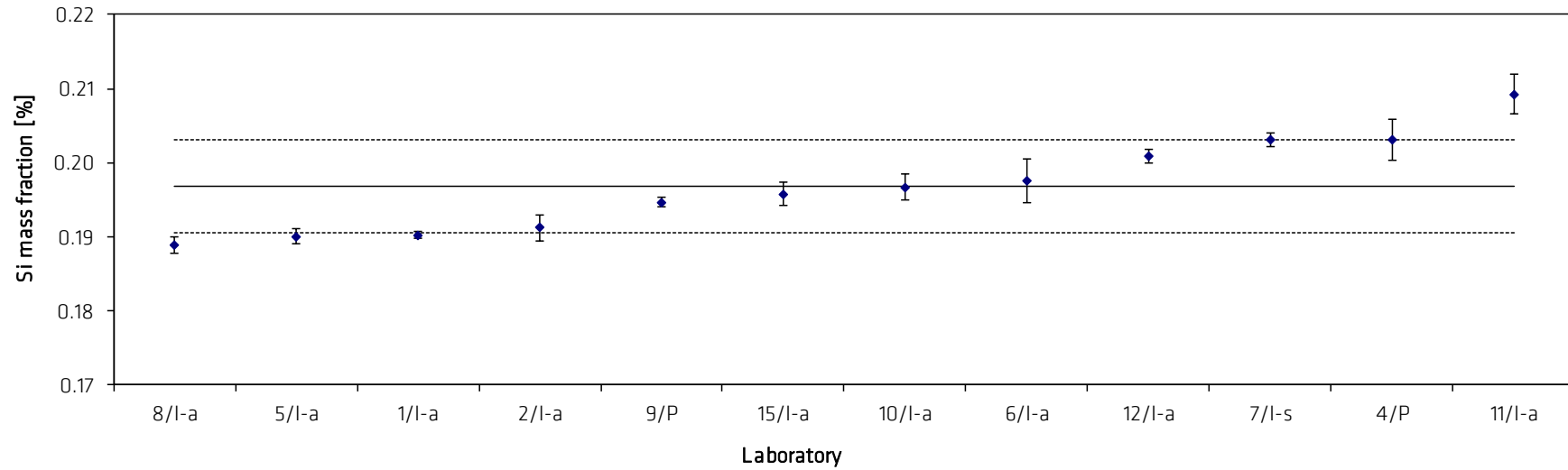


Table 3: Results for Fe

Lab./Meth.	6/l-a(R)	8/l-a	7/IMS-s	4/l-s	1/l-a	5/l-a	8/l-s	12/l-a	9/P	7/l-s	2/l-a	9/A-s	15/l-a	11/l-s	10/l-a	11/l-a		
M_i [%]	0.2033	0.2044	0.2050	0.203	0.204	0.2043	0.2039	0.205	0.206	0.2070	0.2072	0.2058	0.2065	0.209	0.2120	0.2134		n 16
	0.2018	0.2032	0.2080	0.205	0.205	0.2061	0.2060	0.208	0.208	0.2056	0.2056	0.2098	0.2075	0.210	0.2130	0.2130		
	0.2010	0.2028	0.2030	0.204	0.205	0.2038	0.2059	0.204	0.207	0.2070	0.2067	0.2076	0.2069	0.209	0.2080	0.2125		
	0.2077	0.2041	0.2060	0.204	0.204	0.2032	0.2053	0.207	0.203	0.2078	0.2091	0.2086	0.2061	0.209	0.2100	0.2128		
	0.2023	0.2024	0.2010	0.204	0.204	0.2043	0.2061	0.206	0.206	0.2062	0.2075	0.2080	0.2087	0.210	0.2090	0.2044		
	0.2000	0.2021	0.1990	0.204	0.204	0.2045	0.2071	0.206	0.204	0.2058	0.2080	0.2081	0.2090	0.210	0.2120	0.2122		
M [%]	0.2027	0.2032	0.2037	0.2040	0.2043	0.2044	0.2057	0.2059	0.2060	0.2066	0.2074	0.2074	0.2075	0.2093	0.2107	0.2114		0.2062
s [%]	0.0027	0.0009	0.0033	0.0006	0.0005	0.0010	0.0011	0.0014	0.0015	0.0008	0.0012	0.0020	0.0012	0.0004	0.0020	0.0034	s_M [%]	0.0026
s_{rel}	0.01340	0.00454	0.01633	0.00310	0.00253	0.00470	0.00517	0.00663	0.00722	0.00408	0.00567	0.00971	0.00570	0.00207	0.00933	0.01630	\bar{s}_i [%]	0.0018
																		0.01256

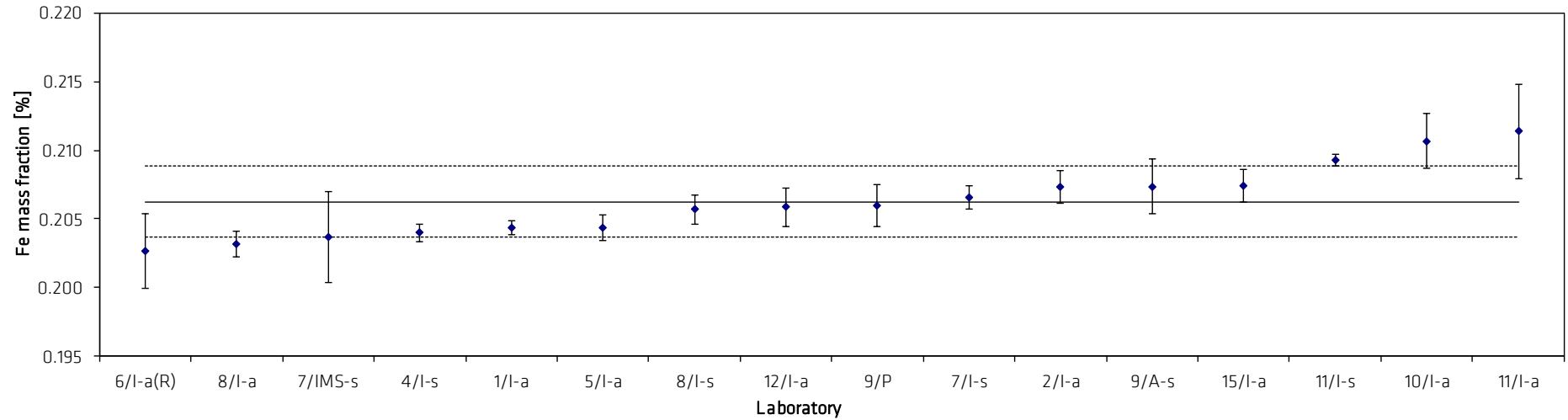


Table 4: Results for Cu

Lab./Meth.	9/l-s	4/l-s	9/A-s	6/l-s	10/l-a	12/l-a	2/l-a	1/l-a	5/l-a	11/l-s	8/l-s	8/l-a	11/l-a	7/l-s		
M_i [%]	0.141	0.143	0.143	0.145	0.145	0.145	0.146	0.146	0.146	0.147	0.146	0.149	0.151	0.153		n 14
	0.142	0.145	0.144	0.144	0.145	0.147	0.145	0.147	0.146	0.148	0.148	0.149	0.150	0.152		
	0.143	0.144	0.144	0.144	0.148	0.144	0.145	0.147	0.147	0.147	0.148	0.148	0.150	0.153		
	0.143	0.144	0.145	0.144	0.144	0.147	0.147	0.147	0.146	0.147	0.147	0.151	0.152	0.154		
	0.140	0.144	0.145	0.145	0.145	0.145	0.147	0.146	0.147	0.147	0.148	0.149	0.148	0.152		
	0.142	0.144	0.145	0.146	0.143	0.146	0.147	0.147	0.148	0.148	0.149	0.148	0.151	0.153		
			0.144													
M [%]	0.1418	0.1440	0.1444	0.1446	0.1450	0.1458	0.1462	0.1467	0.1468	0.1473	0.1476	0.1490	0.1504	0.1527		0.1466
s [%]	0.0010	0.0006	0.0006	0.0009	0.0017	0.0011	0.0010	0.0005	0.0006	0.0003	0.0009	0.0012	0.0014	0.0008	s_M [%]	0.0028
s_{rel}	0.00717	0.00439	0.00398	0.00642	0.01154	0.00744	0.00704	0.00352	0.00382	0.00174	0.00636	0.00800	0.00921	0.00492	\overline{s}_i [%]	0.0010
																0.01902

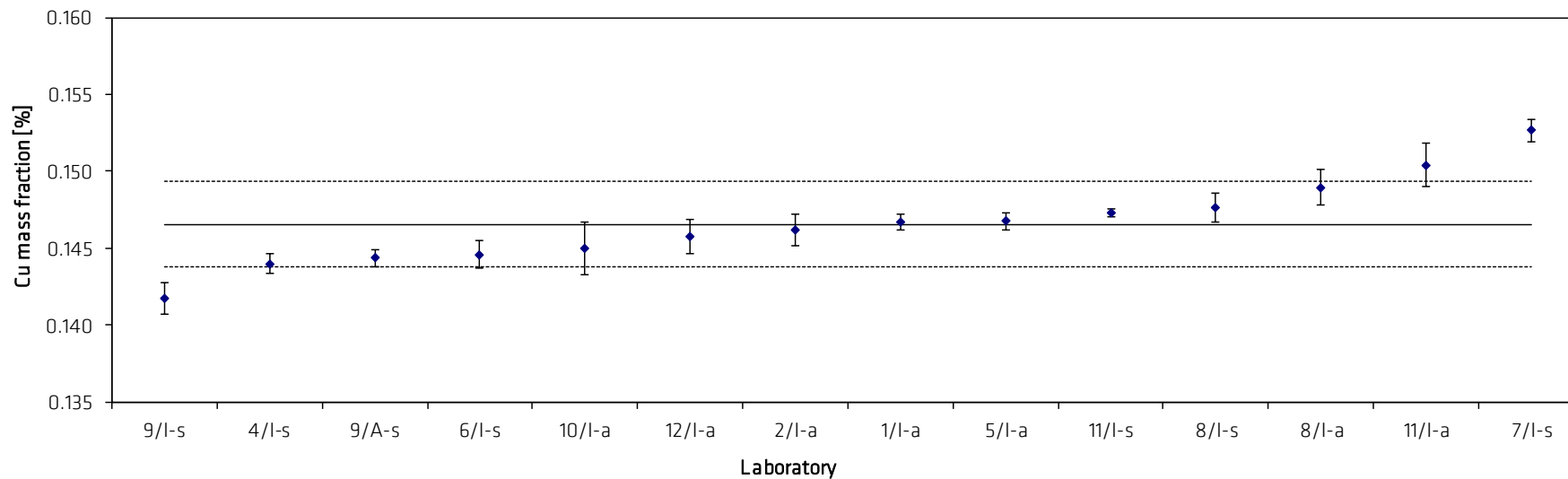


Table 5: Results for Mn

Lab./Meth.	5/l-a	10/l-a	8/l-a	11/l-a	6/l-a	1/l-a	7/l-s	9/P	8/l-s	12/l-a	2/l-a	4/l-s	15/l-a	9/A-s		
M_i [%]	0.6840	0.686	0.6937	0.6982	0.6992	0.70	0.7031	0.7018	0.6963	0.7048	0.7029	0.706	0.7053	0.708		n 14
	0.6810	0.685	0.6878	0.6978	0.6989	0.70	0.7026	0.7015	0.7033	0.7008	0.7007	0.708	0.7105	0.712		
	0.6793	0.684	0.6891	0.6978	0.6988	0.70	0.6992	0.7033	0.7000	0.7001	0.7014	0.707	0.7076	0.715		
	0.6785	0.679	0.6908	0.6904	0.6954	0.70	0.7016	0.7008	0.7022	0.7024	0.7071	0.706	0.7066	0.714		
	0.6808	0.695	0.6861	0.6946	0.6982	0.70	0.6984	0.7010	0.7017	0.7052	0.7099	0.708	0.7108	0.715		
	0.6785	0.685	0.6858	0.6958	0.7030	0.70	0.7023	0.6994	0.7078	0.7023	0.7077	0.710	0.7173	0.704		
									0.7006						0.713	
M [%]	0.680	0.686	0.689	0.696	0.699	0.700	0.701	0.701	0.702	0.703	0.705	0.708	0.710	0.712		0.699
s [%]	0.002	0.005	0.003	0.003	0.002	0.000	0.002	0.001	0.004	0.002	0.004	0.002	0.004	0.004	s_M [%]	0.009
s_{rel}	0.00306	0.00759	0.00437	0.00428	0.00346	0.00000	0.00276	0.00158	0.00540	0.00293	0.00538	0.00214	0.00608	0.00579	\bar{s}_i [%]	0.003
																0.01285

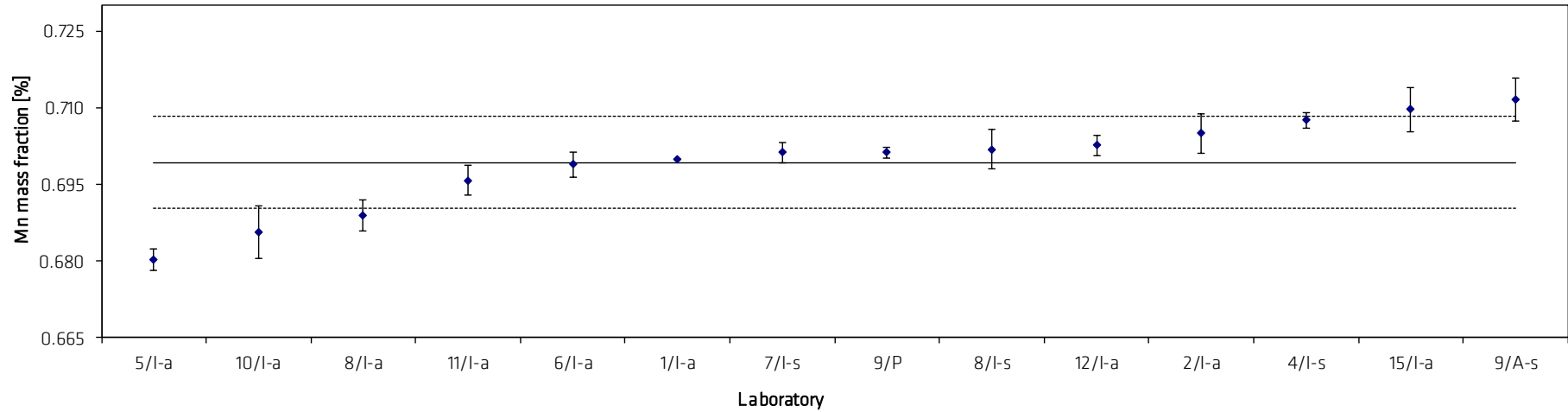


Table 6: Results for Mg

Lab./Meth.	8/l-a	9/l-s	4/l-s	10/l-a	5/l-a	15/l-a	12/l-a	2/l-a	8/l-s	11/l-a	7/l-s	9/l-a	1/l-a	9/A-s	6/l-s		
M_i [%]	3.908	3.922	3.92	3.97	3.971	3.968	3.989	3.996	3.951	4.022	3.998	3.993	4.02	4.107	4.065		n 15
	3.895	3.958	3.92	3.97	3.959	3.966	3.960	3.973	3.969	4.015	3.962	3.986	4.01	4.076	4.076		
	3.896	3.940	3.99	3.95	3.965	3.978	3.968	3.985	3.973	4.008	3.989	4.003	4.03	4.112	4.050		
	3.910	3.966	3.87	3.97	3.953	3.964	3.980	3.979	3.978	3.968	4.007	4.018	4.02	4.052	4.064		
	3.889	3.893	3.94	3.96	3.960	3.972	3.978	3.957	3.986	3.957	4.000	4.008	4.03	3.976	4.076		
	3.887	3.950	4.00	3.94	3.972	3.972	3.966	3.961	4.016	3.968	3.997	3.993	4.02	4.070	4.086		
												3.997		3.976			
M [%]	3.90	3.94	3.94	3.96	3.96	3.97	3.97	3.98	3.98	3.99	3.99	4.00	4.02	4.05	4.07		3.98
s [%]	0.0094	0.0269	0.0486	0.0126	0.0074	0.0051	0.0106	0.0148	0.0215	0.0285	0.0159	0.0113	0.0075	0.0564	0.0126	s_M [%]	0.044
s_{rel}	0.00242	0.00684	0.01233	0.00319	0.00187	0.00127	0.00268	0.00373	0.00539	0.00713	0.00397	0.00284	0.00187	0.01391	0.00311	\bar{s}_i [%]	0.024
																	0.01097

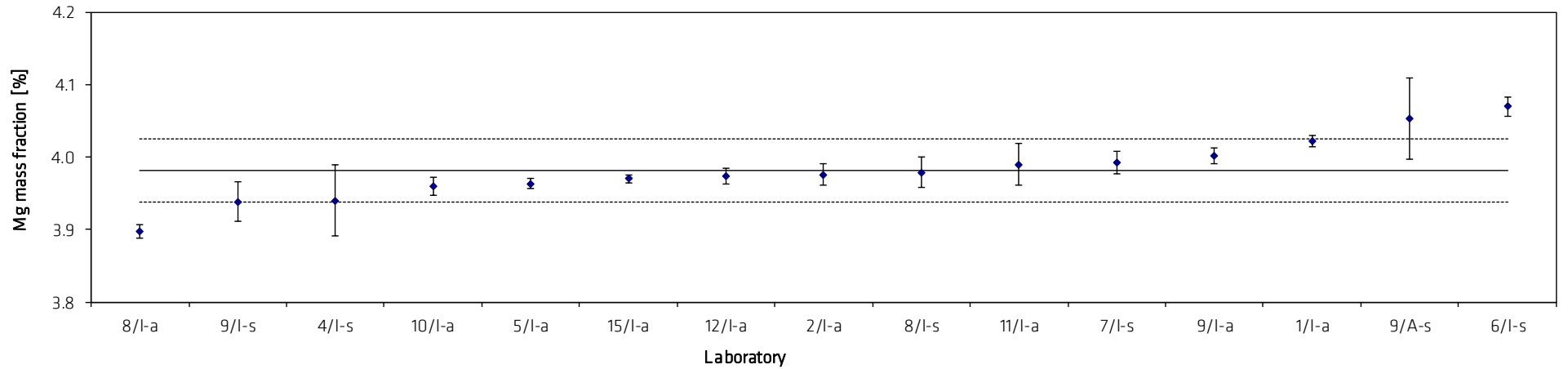


Table 7: Results for Cr

Lab./Meth.	5/l-a	7/l-s	9/l-s	11/l-s	8/l-a	6/l-a	12/l-a	7/IMS-s	8/l-s	4/l-s	15/l-a	10/l-a	1/l-a	11/l-a	2/l-a		
M_i [%]	0.1007	0.101	0.1021	0.1027	0.1037	0.1033	0.1049	0.106	0.1040	0.104	0.1049	0.107	0.107	0.1080	0.1069		n 15
	0.1000	0.102	0.1028	0.1034	0.1029	0.1048	0.1055	0.107	0.1046	0.105	0.1059	0.106	0.108	0.1075	0.1067		
	0.1003	0.101	0.1023	0.1024	0.1031	0.1041	0.1030	0.104	0.1047	0.105	0.1055	0.106	0.108	0.1082	0.1067		
	0.0998	0.102	0.1027	0.1023	0.1035	0.1034	0.1044	0.106	0.1045	0.104	0.1055	0.105	0.108	0.1075	0.1087		
	0.1004	0.102	0.1012	0.1022	0.1025	0.1039	0.1050	0.103	0.1047	0.105	0.1061	0.107	0.107	0.1055	0.1089		
	0.1003	0.101	0.1028	0.1027	0.1030	0.1033	0.1041	0.101	0.1054	0.105	0.1067	0.106	0.107	0.1088	0.1087		
M [%]	0.1002	0.1013	0.1023	0.1026	0.1031	0.1038	0.1045	0.1045	0.1047	0.1047	0.1058	0.1062	0.1075	0.1076	0.1078		0.1044
s [%]	0.0003	0.0006	0.0006	0.0004	0.0004	0.0006	0.0009	0.0023	0.0005	0.0005	0.0006	0.0008	0.0002	0.0011	0.0011	s_M [%]	0.0023
s_{rel}	0.00313	0.00596	0.00604	0.00424	0.00418	0.00547	0.00837	0.02161	0.00431	0.00493	0.00582	0.00709	0.00207	0.01051	0.01000	\bar{s}_i [%]	0.0009
																	0.02184

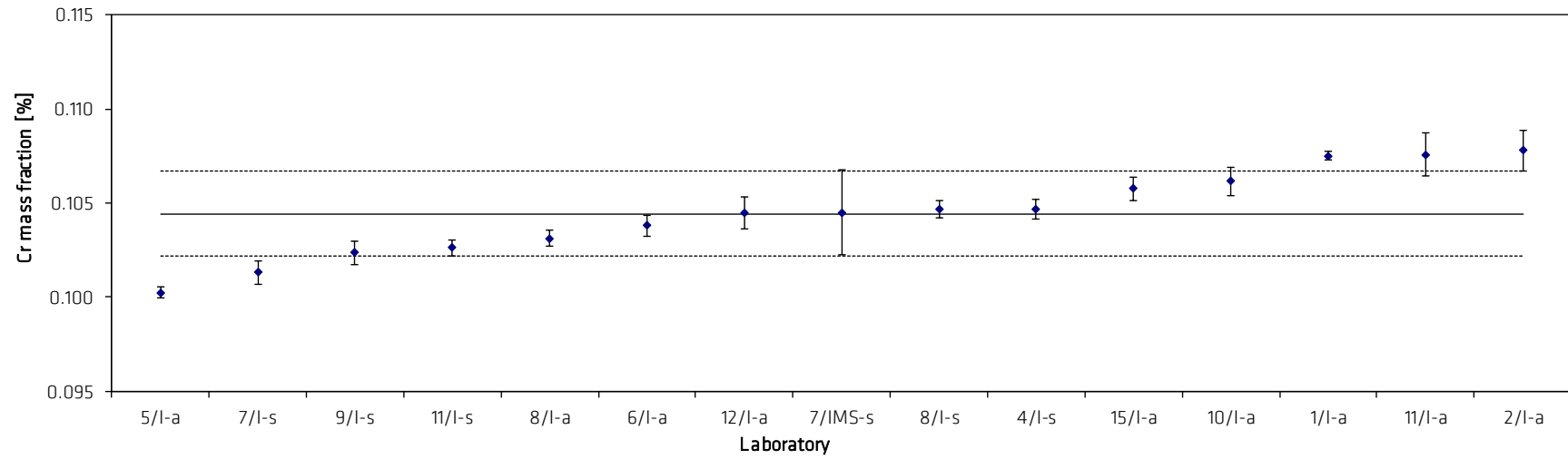


Table 8: Results for Zn

Lab./Meth.	8/l-a	7/l-s(R)	5/l-a	11/l-s	4/l-s	12/l-a	2/l-a	9/l-s	6/l-s	10/l-a	9/A-s	15/l-a	1/l-a	8/l-s		
M_i [%]	0.244	0.2416	0.251	0.2482	0.251	0.2536	0.2516	0.2537	0.255	0.260	0.255	0.2537	0.260	0.258		n 14
	0.243	0.2412	0.248	0.2491	0.251	0.2540	0.2506	0.2547	0.257	0.257	0.257	0.2581	0.259	0.262		
	0.242	0.2412	0.245	0.2478	0.252	0.2493	0.2510	0.2537	0.253	0.256	0.255	0.2559	0.260	0.260		
	0.243	0.2431	0.244	0.2479	0.251	0.2502	0.2546	0.2556	0.254	0.257	0.258	0.2553	0.259	0.261		
	0.239	0.2438	0.246	0.2479	0.250	0.2528	0.2548	0.2529	0.254	0.255	0.255	0.2584	0.260	0.261		
	0.241	0.2422	0.245	0.2489	0.250	0.2509	0.2545	0.2560	0.254	0.253	0.257	0.2625	0.259	0.260		
M [%]	0.242	0.242	0.247	0.248	0.251	0.252	0.253	0.254	0.255	0.256	0.257	0.257	0.260	0.260		0.252
s [%]	0.002	0.001	0.003	0.001	0.001	0.002	0.002	0.001	0.002	0.002	0.002	0.003	0.001	0.001	s_M [%] \bar{s}_i [%]	0.006
s_{rel}	0.00681	0.00442	0.01134	0.00226	0.00300	0.00768	0.00790	0.00473	0.00595	0.00912	0.00736	0.01202	0.00211	0.00439		0.002
																0.02319

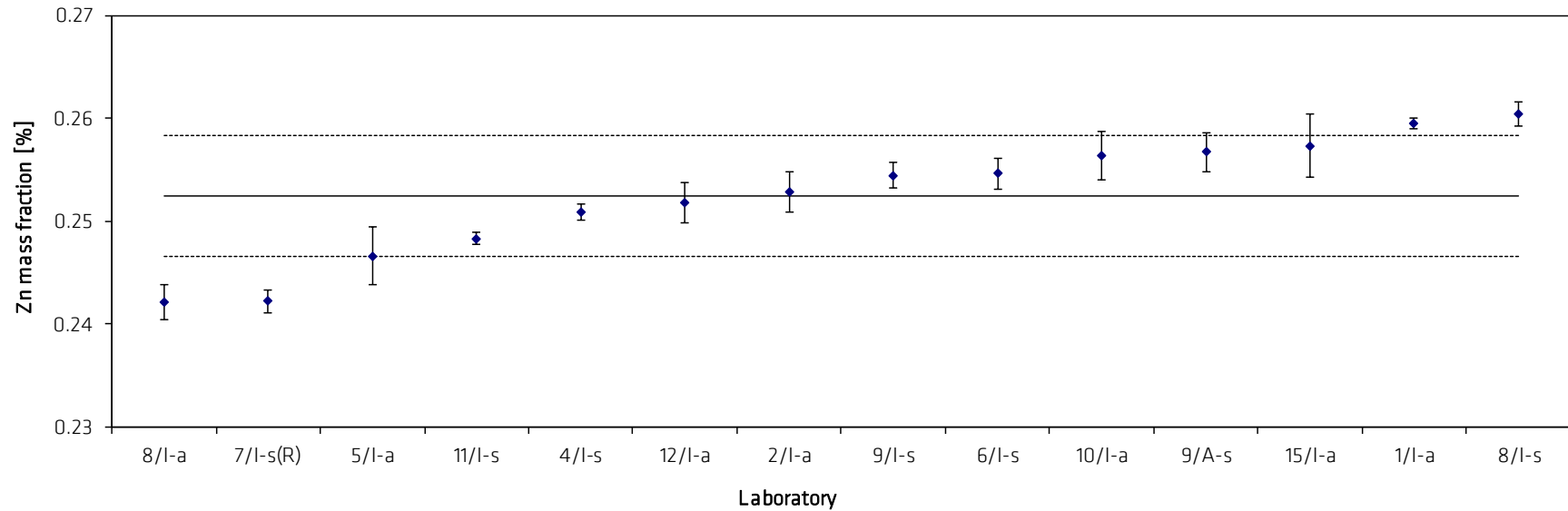


Table 9: Results for Ti

Lab./Meth.	5/l-a	9/l-s	10/l-a	6/l-a	12/l-a	11/l-s	15/l-a	9/P	1/l-a	2/l-s	8/l-a	8/l-s	11/l-a	7/l-s	4/l-s		
M_i [%]	0.0989	0.0995	0.101	0.1000	0.102	0.1010	0.1012	0.102	0.1022	0.1027	0.1034	0.1029	0.1042	0.1045	0.106		n 14
	0.0992	0.0998	0.100	0.1009	0.101	0.1016	0.1014	0.102	0.1021	0.1026	0.1028	0.1034	0.1037	0.1050	0.109		
	0.0984	0.0996	0.100	0.1006	0.099	0.1008	0.1013	0.101	0.1021	0.1025	0.1033	0.1033	0.1057	0.1039	0.109		
	0.0983	0.1004	0.099	0.1001	0.100	0.1004	0.1013	0.102	0.1023	0.1027	0.1034	0.1036	0.1048	0.1042	0.108		
	0.0984	0.0990	0.101	0.1007	0.101	0.1007	0.1015	0.102	0.1022	0.1022	0.1028	0.1037	0.1014	0.1047	0.107		
	0.0985	0.1008	0.100	0.1001	0.101	0.1010	0.1015	0.101	0.1021	0.1024	0.1023	0.1032	0.1055	0.1045	0.107		
M [%]	0.0986	0.0998	0.1000	0.1004	0.1007	0.1009	0.1014	0.1015	0.1022	0.1025	0.1030	0.1034	0.1042	0.1045	0.1077		0.1016
s [%]	0.00036	0.00065	0.00095	0.00037	0.00089	0.00040	0.00012	0.00035	0.00008	0.00018	0.00044	0.00029	0.00157	0.00038	0.00121	s_M [%]	0.00173
s_{rel}	0.00362	0.00647	0.00949	0.00372	0.00883	0.00398	0.00119	0.00349	0.00080	0.00172	0.00430	0.00279	0.01510	0.00367	0.01125	\bar{s}_i [%]	0.00063
																	0.01699

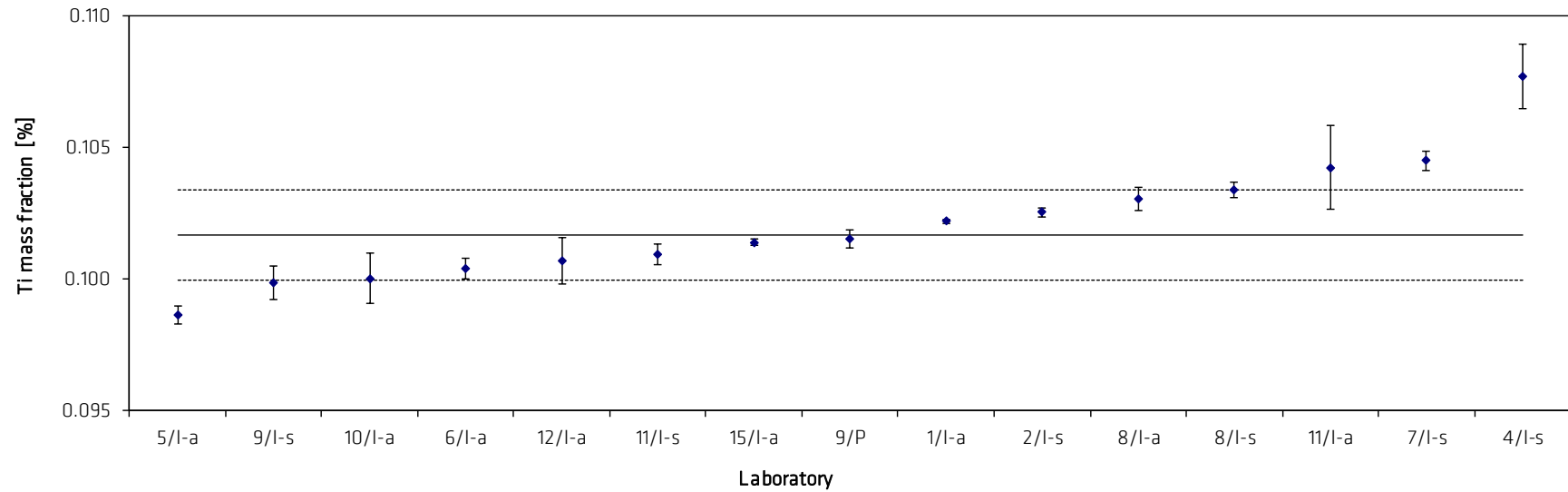


Table 10: Results for Ga

Lab./Meth.	5/l-a	7/l-s(R)	4/l-s	2/l-a	8/l-s	7/IMS-s	12/l-a	9/l-s	8/l-a	10/l-a	1/l-a	6/l-s	11/l-a	11/l-s		
M_i [%]	0.0192	0.0198	0.0202	0.0205	0.0204	0.020	0.0209	0.0206	0.0210	0.0214	0.0215	0.0218	0.0219	0.0221		n 14
	0.0192	0.0197	0.0201	0.0204	0.0204	0.021	0.0204	0.0208	0.0208	0.0216	0.0215	0.0213	0.0222	0.0222		
	0.0186	0.0197	0.0203	0.0204	0.0204	0.021	0.0206	0.0210	0.0206	0.0214	0.0215	0.0215	0.0225	0.0223		
	0.0191	0.0198	0.0200	0.0203	0.0205	0.021	0.0205	0.0207	0.0210	0.0213	0.0215	0.0215	0.0222	0.0225		
	0.0190	0.0199	0.0202	0.0202	0.0203	0.021	0.0205	0.0206	0.0209	0.0214	0.0215	0.0215	0.0206	0.0225		
	0.0190	0.0198	0.0204	0.0203	0.0206	0.020	0.0208	0.0209	0.0210	0.0215	0.0215	0.0215	0.0226	0.0227		
M [%]	0.0190	0.0198	0.0202	0.0203	0.0204	0.0205	0.0206	0.0208	0.0209	0.0214	0.0215	0.0215	0.0220	0.0224		0.0208
s [%]	0.00024	0.00008	0.00014	0.00011	0.00010	0.00026	0.00019	0.00015	0.00016	0.00010	0.00000	0.00015	0.00071	0.00022	s_M [%]	0.00089
s_{rel}	0.01237	0.00381	0.00700	0.00539	0.00505	0.01249	0.00941	0.00730	0.00767	0.00482	0.00000	0.00677	0.03227	0.00993	\bar{s}_i [%]	0.00024
																0.04287

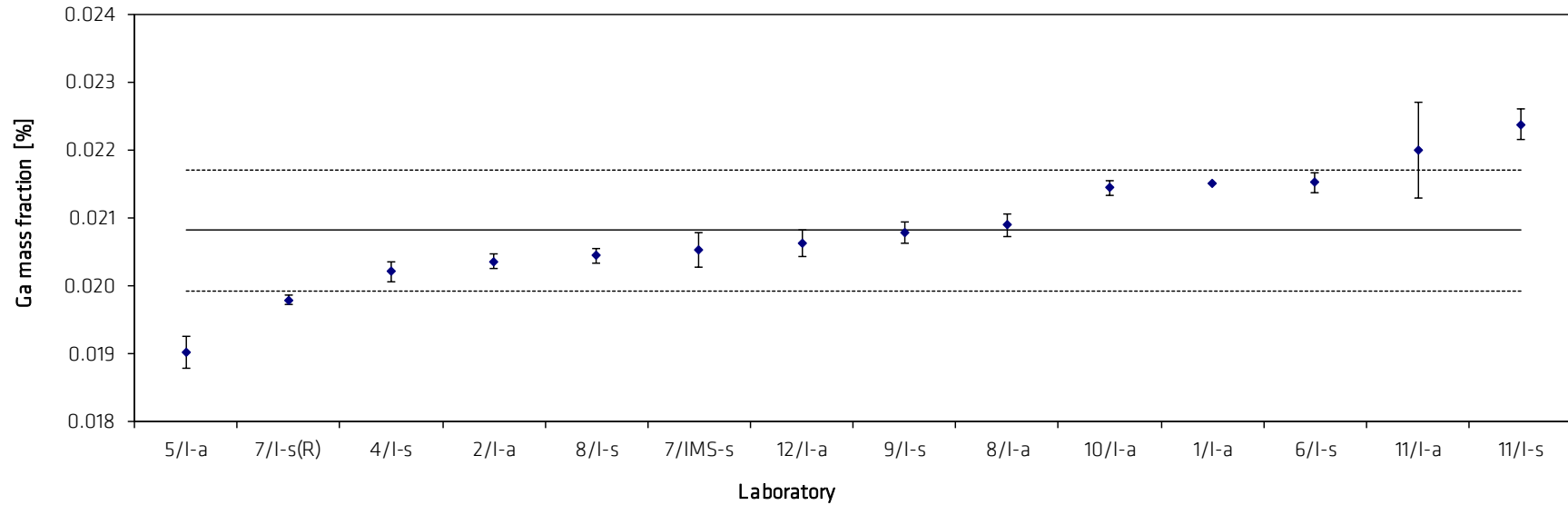


Table 11: Results for Sc

Lab./Meth.	5/l-a	8/l-a	11/l-s	1/l-a	10/l-a	2/l-s	9/l-s	6/l-a	12/l-a	11/l-a	4/l-s	7/l-s(R)		
M_i [%]	0.275	0.269	0.2794	0.280	0.282	0.2826	0.2840	0.2839	0.287	0.289	0.290	0.302		n
	0.274	0.279	0.2805	0.280	0.283	0.2835	0.2781	0.2869	0.289	0.285	0.289	0.302		11
	0.276	0.285	0.2790	0.280	0.283	0.2824	0.2802	0.2853	0.282	0.289	0.289	0.301		
	0.275	0.277	0.2781	0.280	0.276	0.2814	0.2842	0.2839	0.286	0.289	0.292	0.302		
	0.275	0.270	0.2780	0.280	0.280	0.2805	0.2833	0.2855	0.284	0.279	0.289	0.301		
	0.276	0.273	0.2790	0.280	0.287	0.2808	0.2866	0.2828	0.289	0.289	0.286	0.302		
M [%]	0.275	0.275	0.279	0.280	0.282	0.282	0.283	0.285	0.286	0.286	0.289	0.302		0.282
s [%]	0.0007	0.0061	0.0009	0.0000	0.0037	0.0012	0.0031	0.0015	0.0027	0.0042	0.0019	0.0006	s_M [%]	0.0045
s_{rel}	0.00244	0.02199	0.00329	0.00000	0.01297	0.00414	0.01086	0.00511	0.00944	0.01454	0.00671	0.00186	\bar{s}_i [%]	0.0029
														0.01581

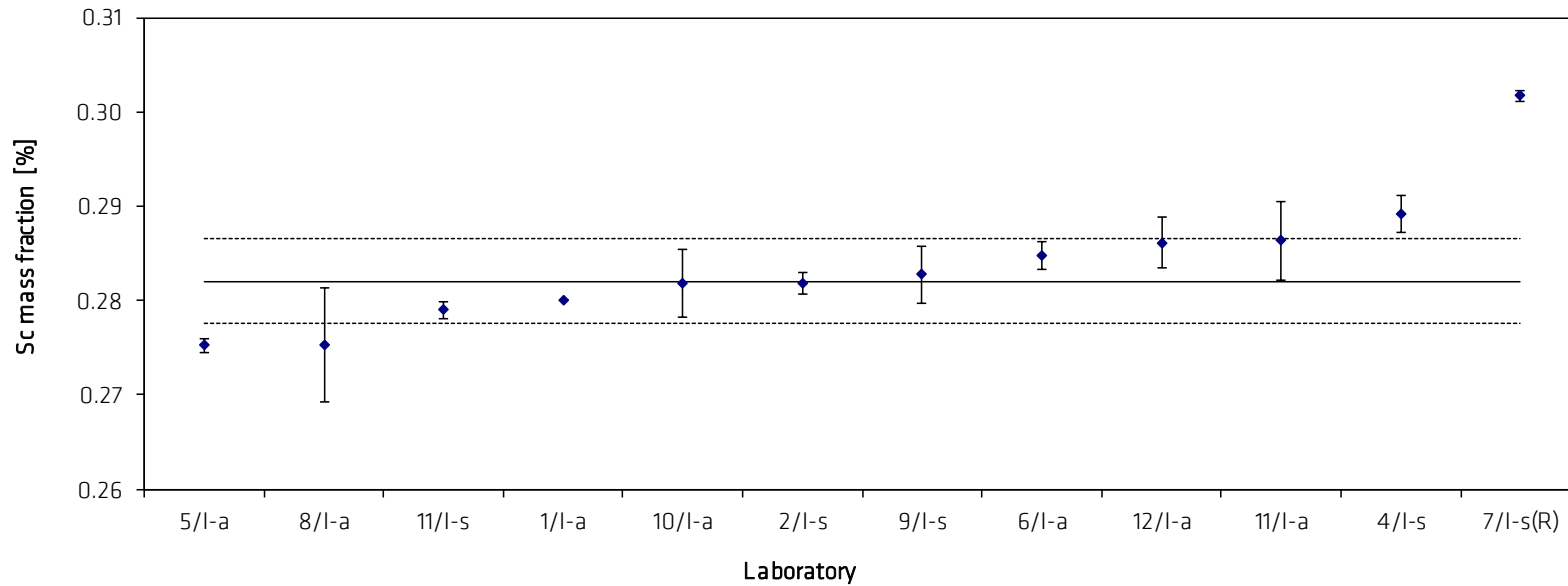


Table 12: Results for Zr

Lab./Meth.	5/l-a	12/l-a	10/l-a	8/l-a	6/l-a	1/l-a	8/l-s	9/P	7/l-s	15/l-a	11/l-s	2/l-s	4/l-s	9/l-s		
M_i [%]	0.0991	0.0988	0.1000	0.1010	0.1004	0.1024	0.1031	0.1025	0.1033	0.1032	0.1023	0.1047	0.104	0.104		n 14
	0.0987	0.0994	0.0994	0.1007	0.1016	0.1021	0.1029	0.1013	0.1028	0.1038	0.1046	0.1047	0.104	0.104		
	0.0989	0.0992	0.1000	0.1005	0.1013	0.1022	0.1033	0.1047	0.1035	0.1036	0.1041	0.1044	0.105	0.105		
	0.0987	0.0999	0.0985	0.1013	0.1004	0.1023	0.1029	0.1016	0.1050	0.1041	0.1041	0.1042	0.105	0.105		
	0.0984	0.0988	0.0998	0.1002	0.1012	0.1022	0.1039	0.1034	0.1034	0.1037	0.1045	0.1038	0.104	0.104		
	0.0991	0.0998	0.1010	0.1006	0.1001	0.1021	0.0975	0.1050	0.1026	0.1041	0.1045	0.1040	0.105	0.105		
								0.1022	0.1025							
M [%]	0.0988	0.0993	0.0998	0.1007	0.1009	0.1022	0.1023	0.1029	0.1034	0.1038	0.1040	0.1043	0.1045	0.1045		0.1022
s [%]	0.0003	0.0005	0.0008	0.0004	0.0006	0.0001	0.0024	0.0014	0.0008	0.0003	0.0009	0.0004	0.0005	0.0005	s_M [%] \bar{s}_i [%]	0.0020
s_{rel}	0.00257	0.00478	0.00823	0.00384	0.00615	0.00114	0.02312	0.01337	0.00817	0.00327	0.00835	0.00357	0.00524	0.00524		0.0009

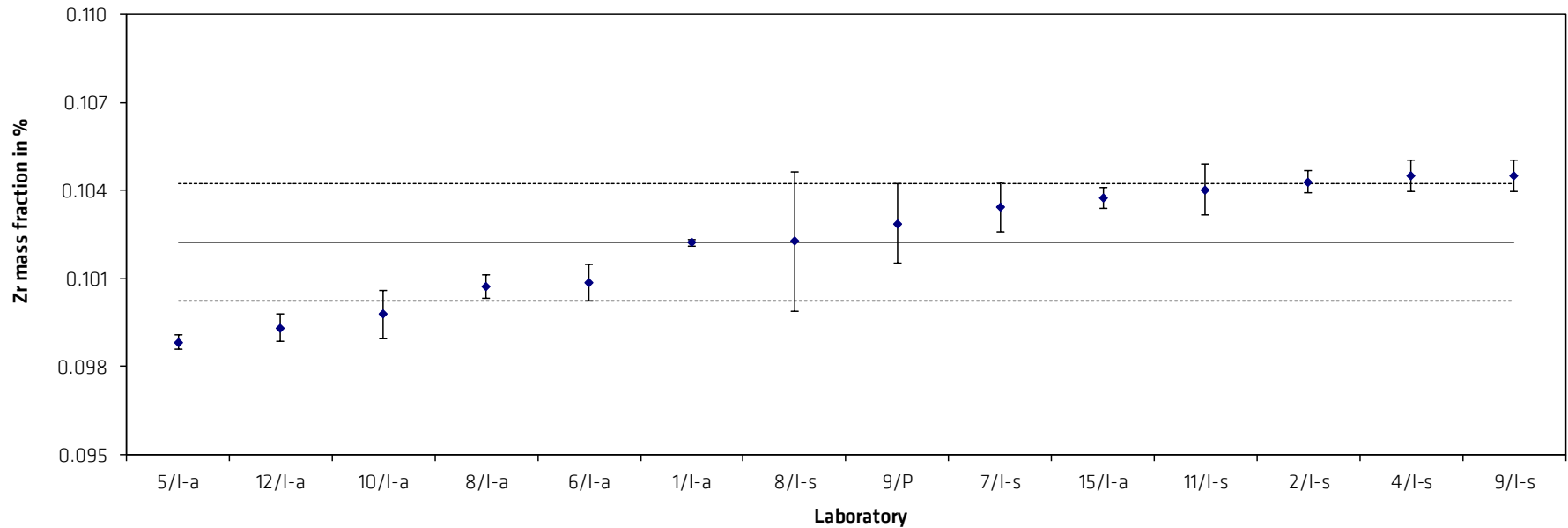


Table 13: Results for Ni

Lab./Meth.	1/l-s	8/l-a	12/l-a	9/l-s	4/l-s	11/l-s	2/l-s	8/l-s	5/l-a	6/l-a	7/l-s(R)	11/IMS-s	10/l-a	7/IMS-s	15/l-a		
M_i [mg/kg]	17	19.0	19	19.5	19.9	19.8	19.6	20.0	20.5	20.2	20.5	22.6	23.3	23.3	23.5		n 15
	19	19.0	20	19.6	19.8	19.9	19.4	21.0	20.7	20.7	25.2	22.6	22.9	23.3	22.6		
	18	19.0	19	20.0	19.6	19.9	19.4	20.0	20.6	21.0	20.8	22.6	23.1	23.0	25.4		
	17	19.0	20	19.7	19.8	20.0	20.8	20.0	20.4	22.3	23.1	22.8	22.4	23.0	24.0		
	17	18.0	19	19.7	19.9	20.2	20.7	20.0	20.4	23.3	24.7	22.9	22.8	22.8	22.5		
	17	19.0	19	19.9	19.9	19.9	20.1	20.3	20.0	20.4	24.3	21.4	23.0	23.2	23.2	26.6	
M [mg/kg]	17.5	18.8	19.3	19.7	19.8	20.0	20.0	20.2	20.5	22.0	22.6	22.8	23.0	23.1	24.1		20.9
s [mg/kg]	0.8	0.4	0.6	0.2	0.1	0.1	0.6	0.4	0.1	1.6	2.0	0.2	0.3	0.2	1.6	s_M [mg/kg]	1.888
s_{rel}	0.04781	0.02168	0.03274	0.00924	0.00590	0.00745	0.03227	0.02024	0.00658	0.07280	0.08936	0.00829	0.01425	0.00866	0.06721	\bar{s}_i [mg/kg]	0.8713
																	0.09036

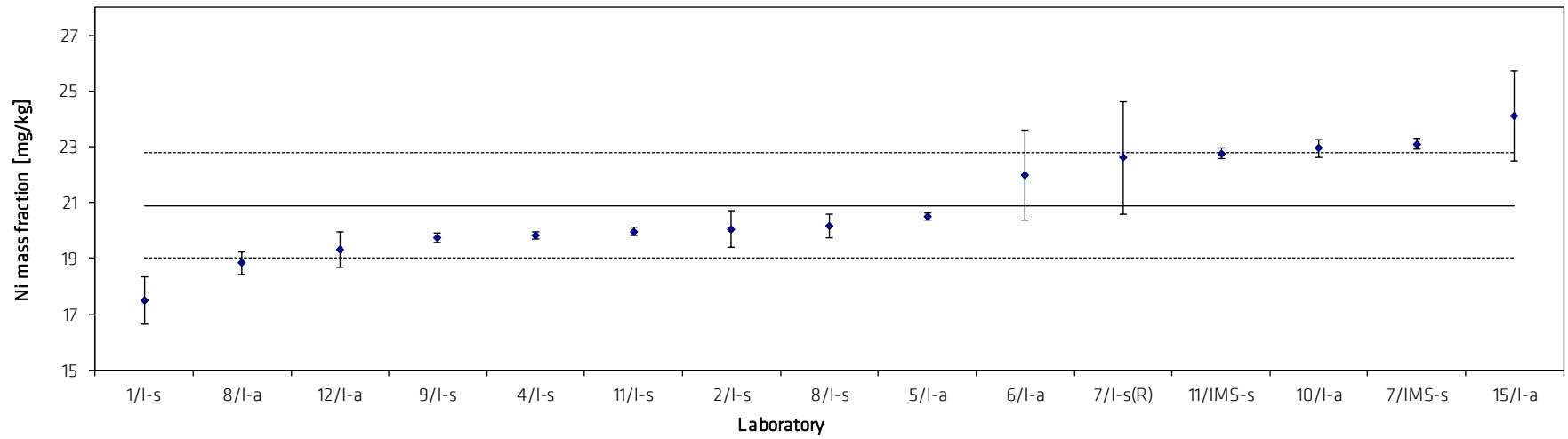


Table 14: Results for Be

Lab./Meth.	8/l-a	2/l-s	4/l-s	8/l-s	10/l-a	5/l-a	7/IMS-s	9/l-s	12/l-a	6/l-a	11/l-s	1/l-s	11/IMS-s		
M_i [mg/kg]	22.0	21.63	22.1	22.0	22.50	22.2	22.0	21.90	22.76	22.64	22.85	23.0	23.09		n
	22.0	21.75	22.2	22.0	22.20	22.3	22.9	21.89	22.70	22.89	22.87	23.0	23.45		13
	22.0	21.63	22.2	22.0	22.00	22.2	22.4	23.14	22.27	22.87	22.89	23.0	23.41		
	22.0	22.26	22.1	22.0	22.10	22.2	22.5	22.46	22.54	22.61	23.00	23.0	23.49		
	22.0	22.58	22.0	23.0	22.20	22.2	22.3	22.93	22.36	22.64	23.09	23.0	23.25		
	22.0	22.32	22.0	22.0	22.10	22.2	21.4	21.73	22.76	22.53	23.05	23.0	23.42		
M [mg/kg]	22.0	22.0	22.1	22.2	22.2	22.2	22.3	22.3	22.6	22.7	23.0	23.0	23.4		22.4
s [mg/kg]	0.000	0.408	0.089	0.408	0.172	0.043	0.509	0.595	0.212	0.148	0.102	0.000	0.152	s_M [mg/kg]	0.43
s_{rel}	0.000	0.019	0.004	0.018	0.008	0.002	0.023	0.027	0.009	0.007	0.004	0.000	0.007	\bar{s}_i [mg/kg]	0.29
															0.01915

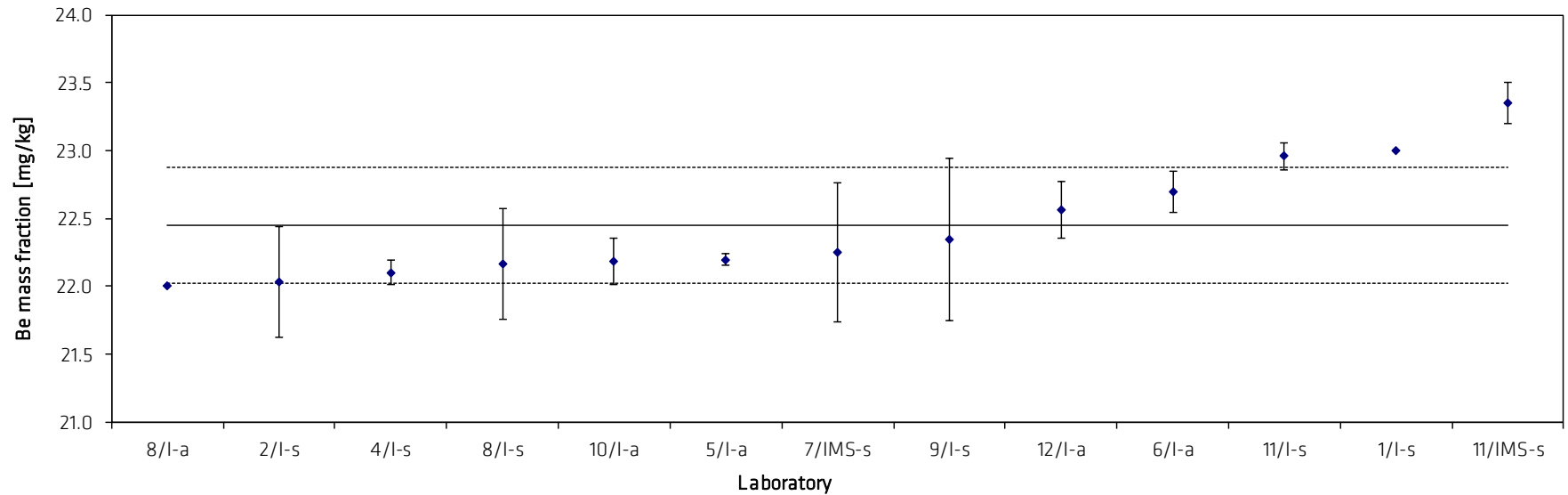


Table 15: Results for Ca

Lab./Meth.	1/l-s	4/l-s	2/l-s	11/l-s	6/l-s	15/l-a	12/l-a		
M_i [mg/kg]	7.0	11.6	11.3	11.5	13.2	13.2	15.0		n 7
	7.0	11.5	11.3	11.7	13.1	12.8	15.0		
	7.0	11.0	10.9	11.4	14.6	14.4	15.0		
	6.0	10.9	10.9	11.4	12.7	13.9	16.0		
	7.0	10.3	11.2	12.2	13.5	13.3	15.0		
	6.0	10.4	10.9	11.6	10.7	14.3	14.0		
M [mg/kg]	6.7	11.0	11.1	11.6	13.0	13.7	15.0		11.7
s [mg/kg]	0.52	0.54	0.20	0.28	1.30	0.65	0.63	s_M [mg/kg]	2.66
s_{rel}	0.077	0.049	0.018	0.025	0.100	0.047	0.042	\bar{s}_i [mg/kg]	0.67
									0.228

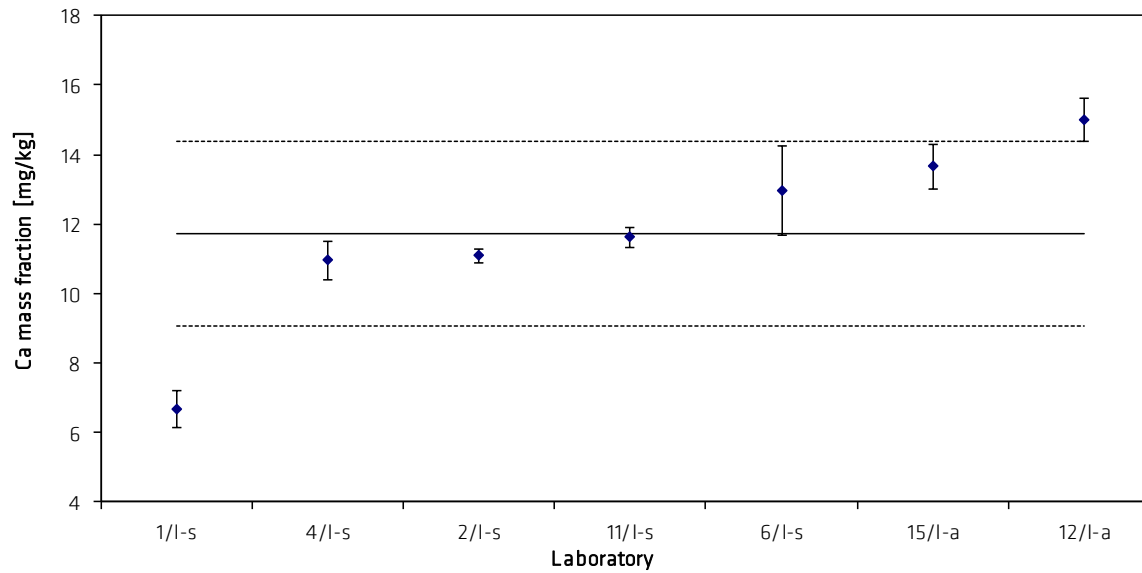


Table 16: Results for Cd

Lab./Meth.	8/l-s	2/l-s	15/l-a	9/l-s	7/IMS-s	12/l-a	6/l-s(R)	11/IMS-s	1/l-s	4/l-s	5/l-a		
M_i [mg/kg]	13.0	14.3	14.3	15.1	15.1	15.6	15.7	15.7	16.0	17.3	18.5		n
	13.0	14.4	14.9	15.2	15.5	15.8	15.9	15.7	16.0	17.2	18.4		10
	13.0	14.2	14.7	15.2	15.5	15.4	15.5	15.8	16.0	17.0	18.4		
	13.0	14.2	14.5	15.1	15.3	15.9	15.6	15.9	15.0	17.1	18.3		
	13.0	14.1	14.4	15.1	15.4	15.4	15.9	15.9	16.0	17.1	18.3		
	14.0	14.1	14.9	15.3	14.8	15.4	15.7	15.9	16.0	17.0	18.5		
M [mg/kg]	13.2	14.2	14.6	15.2	15.3	15.6	15.7	15.8	15.8	17.1	18.4		15.2
s [mg/kg]	0.4	0.1	0.3	0.1	0.3	0.2	0.1	0.1	0.4	0.1	0.1	s_M [mg/kg]	1.07
s_{rel}	0.0310	0.0063	0.0175	0.0061	0.0179	0.0143	0.0089	0.0054	0.0258	0.0068	0.0039	\bar{s}_i [mg/kg]	0.23
													0.0700

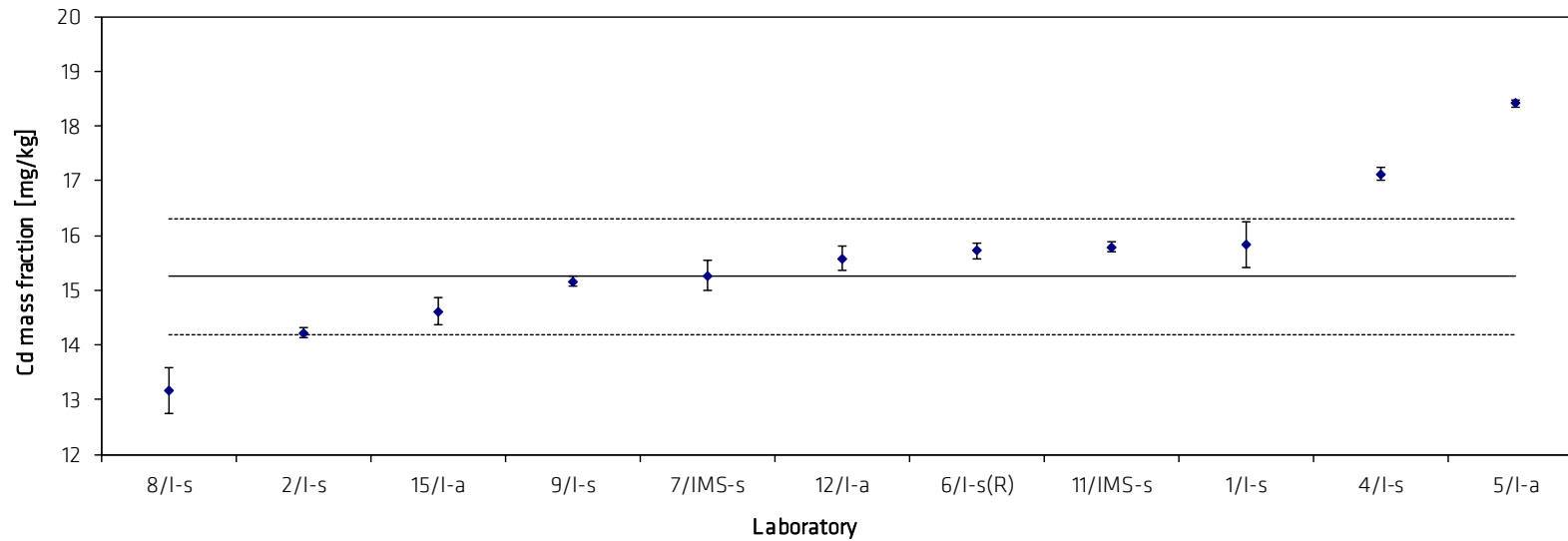


Table 17: Results for Co

Lab./Meth.	7/IMS-s	8/l-a	7/l-s	10/l-a	2/IMS-s	12/l-a	8/l-s	6/l-s	9/l-s	11/l-s	4/l-s	5/l-a	1/l-s	11/IMS-s		
M_i [mg/kg]	18.9	20.0	19.8	20.8	20.86	21.0	21.0	20.6	20.9	21.2	21.3	21.9	22.0	22.0		n
	19.4	19.0	20.0	20.7	20.95	21.0	21.0	21.7	20.8	21.2	21.4	21.9	22.0	22.0		14
	19.4	19.0	20.4	19.9	20.71	20.7	21.0	20.7	21.9	21.2	21.4	21.6	22.0	22.1		
	19.2	19.0	20.2	19.8	20.98	20.8	21.0	21.0	21.7	21.3	21.2	21.5	22.0	22.3		
	18.9	19.0	20.0	20.4	20.62	20.9	21.0	22.1	21.7	21.4	21.3	21.6	22.0	22.3		
	19.1	19.0	19.9	19.9	20.95	20.8	21.0	20.3	20.5	21.3	21.3	21.5	23.0	22.3		
M [mg/kg]	19.2	19.2	20.0	20.3	20.8	20.9	21.0	21.1	21.3	21.3	21.3	21.6	22.2	22.2		20.9
s [mg/kg]	0.23	0.41	0.20	0.44	0.15	0.12	0.00	0.70	0.55	0.09	0.08	0.18	0.41	0.15	s_M [mg/kg]	0.94
s_{rel}	0.012	0.021	0.010	0.022	0.007	0.006	0.000	0.033	0.026	0.004	0.004	0.008	0.018	0.007	\bar{s}_i [mg/kg]	0.33
																0.045

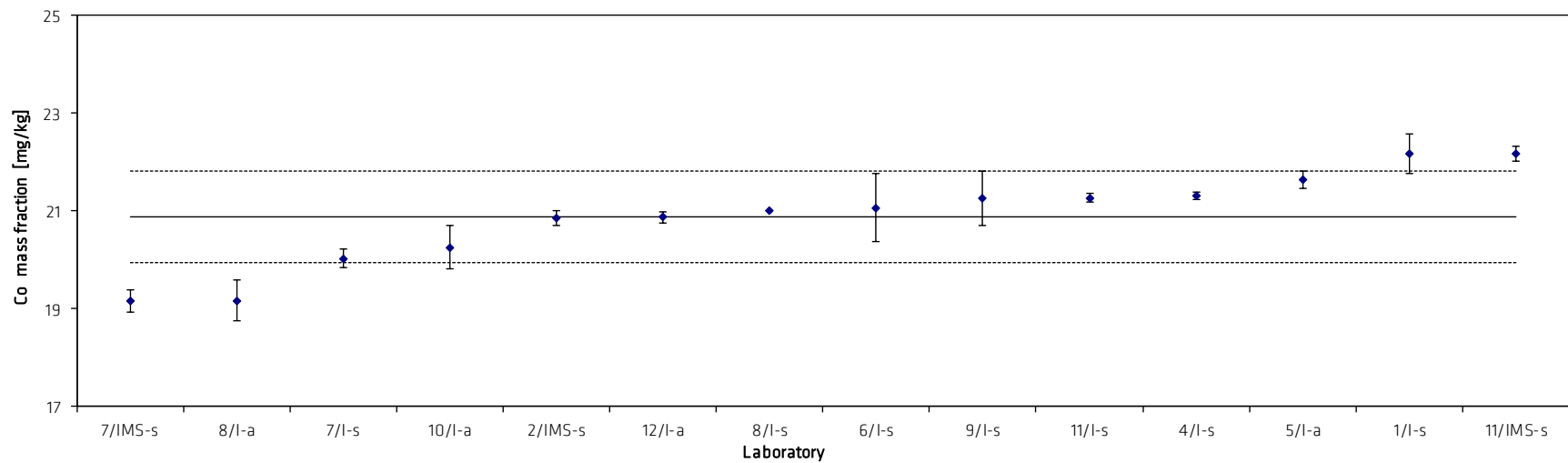


Table 18: Results for Li

Lab./Meth.	8/l-a	7/IMS-s	12/l-a	15/l-a	8/l-s	2/l-s	6/l-s	1/l-s	9/l-s	4/l-s	11/l-s		
M_i [mg/kg]	8	8.5	8.8	9.0	9.0	9.2	9.3	9.0	9.2	9.2	9.6		n
	8	8.8	8.9	9.1	9.0	9.0	9.1	9.0	9.3	9.2	9.7		10
	8	8.8	8.8	8.7	9.0	9.1	9.1	9.0	9.2	9.2	9.7		
	8	8.9	8.8	8.7	9.0	9.0	9.1	9.0	9.2	9.2	9.6		
	8	8.7	8.8	8.9	9.0	9.1	9.1	10.0	9.1	9.2	9.7		
	8	8.4	8.7	8.9	9.0	9.0	9.1	9.0	9.2	9.1	9.8		
M [mg/kg]	8.0	8.7	8.8	8.9	9.0	9.1	9.1	9.2	9.2	9.2	9.7		9.1
s [mg/kg]	0.00	0.19	0.06	0.16	0.00	0.10	0.10	0.41	0.07	0.04	0.05	s_M [mg/kg]	0.27
s_{rel}	0.000	0.022	0.007	0.018	0.000	0.011	0.010	0.045	0.007	0.004	0.005	\bar{s}_i [mg/kg]	0.16
													0.030

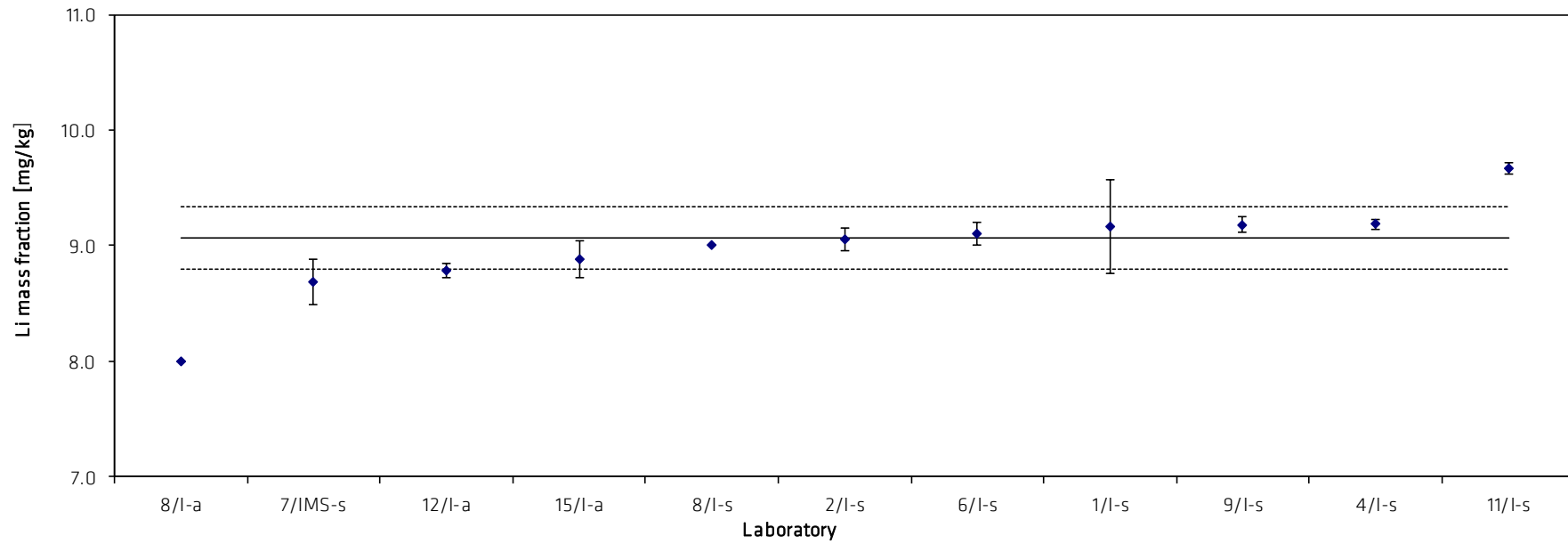


Table 19: Results for Na

Lab./Meth.	9/EA-s	2/l-s	1/l-s	11/l-s	6/l-s	4/l-s		
M_i [mg/kg]	6.0	5.4	6.0	6.5	5.4	7.4		n
	5.8	6.0	6.0	6.6	6.0	7.3		6
	6.1	5.9	6.0	6.5	6.9	7.7		
	5.9	6.0	6.0	6.6	7.9	6.6		
	5.7	6.0	6.0	6.9	7.1	7.1		
	5.9	6.0	6.0	6.5	8.0	6.7		
M [mg/kg]	5.9	5.9	6.0	6.6	6.9	7.1		6.4
s [mg/kg]	0.12	0.26	0.00	0.14	1.01	0.42	s_M [mg/kg]	0.54
							\bar{s}_i [mg/kg]	0.46
s_{rel}	0.021	0.044	0.000	0.020	0.147	0.059		0.085

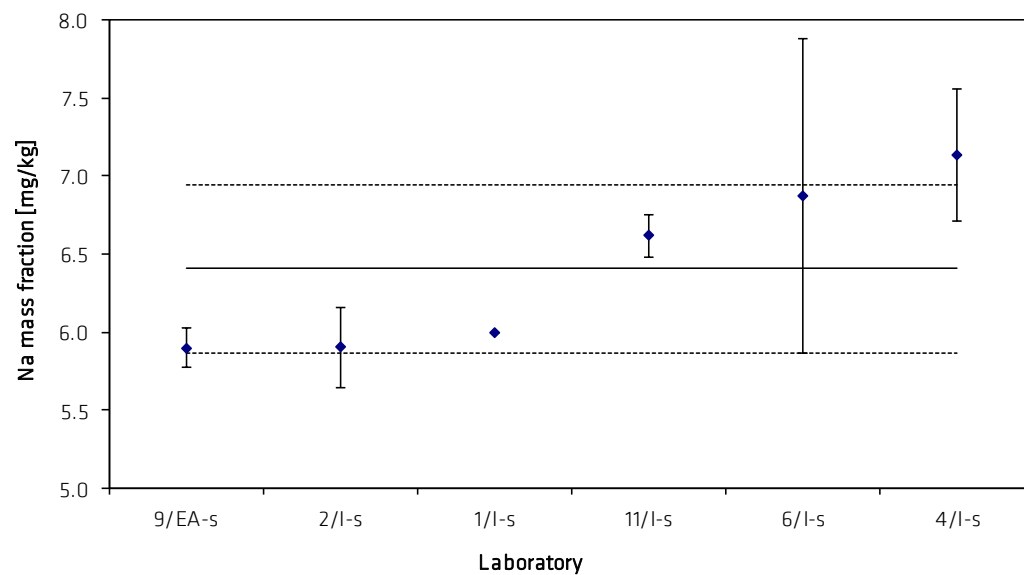


Table 20: Results for Pb

Lab./Meth.	1/l-s	6/l-s	12/l-a	4/l-s	11/IMS-s	2/IMS-s	8/l-a	9/l-s	5/l-a	7/IMS-s	7/l-s	8/l-s		
M_i [mg/kg]	41.0	42.7	45.0	42.7	43.0	43.1	43.0	43.7	45.9	47.9	51.2	51.0		n
	41.0	41.2	42.0	42.8	43.1	43.9	50.0	43.8	46.6	48.9	50.4	52.0		12
	41.0	41.3	42.0	42.5	42.9	43.3	43.0	44.6	46.4	47.7	50.0	51.0		
	41.0	41.4	43.0	42.7	42.9	43.6	42.0	46.0	44.3	47.5	50.2	50.0		
	41.0	40.6	41.0	42.8	43.0	43.0	39.0	43.7	44.5	48.0	50.3	50.0		
	42.0	41.8	42.0	43.2	42.6	43.4	45.0	45.2	46.2	49.0	50.0	55.0		
M [mg/kg]	41.2	41.5	42.5	42.8	42.9	43.4	43.7	44.5	45.6	48.2	50.4	51.5		44.8
s [mg/kg]	0.41	0.71	1.38	0.23	0.17	0.35	3.67	0.95	1.00	0.63	0.46	1.87	s_M [mg/kg]	3.41
s_{rel}	0.0099	0.0170	0.0324	0.0054	0.0040	0.0080	0.0840	0.0213	0.0218	0.0131	0.0091	0.0363	\bar{s}_i [mg/kg]	1.36
														0.076

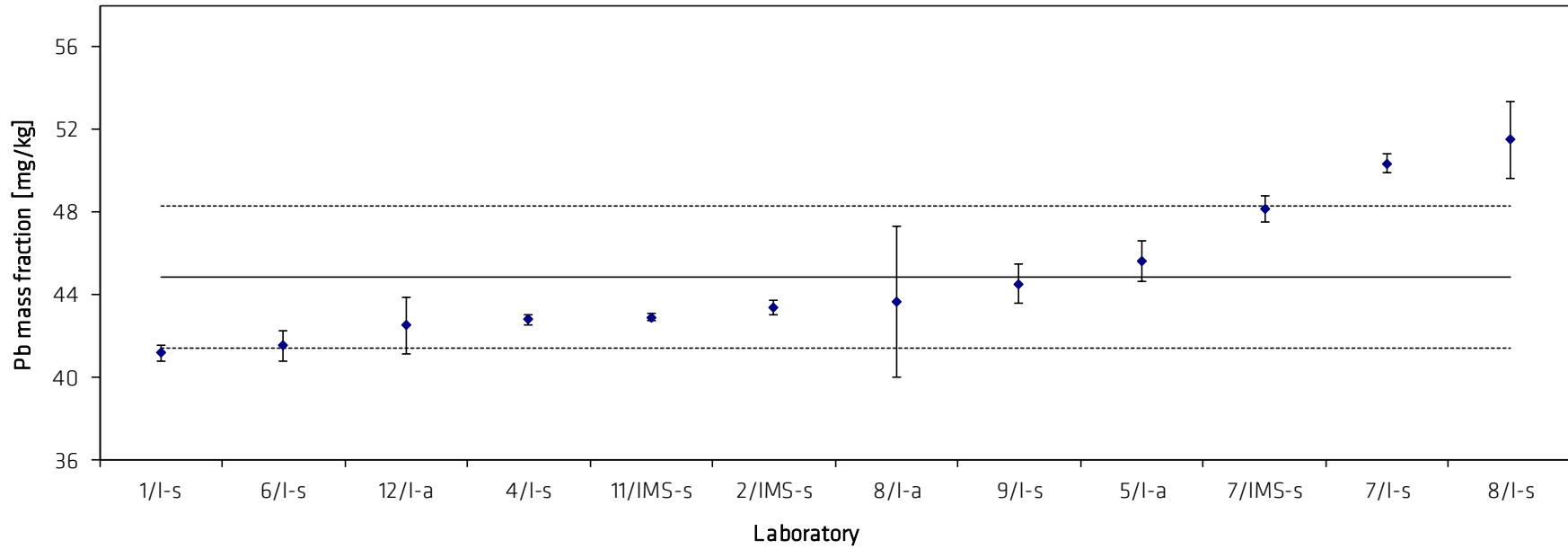


Table 21: Results for Sn

Lab./Meth.	7/l-s	10/l-a	4/l-s	2/IMS-s	1/l-s	11/IMS-s	7/IMS-s	9/l-s	15/l-a	8/l-a	6/l-a	8/l-s	12/l-a	5/l-a		
M_i [mg/kg]	37.2	41.7	41.6	42.1	44.0	43.0	42.8	44.4	44.2	48.0	50.2	50.0	52.2	53.5		n 14
	37.2	41.4	41.8	41.7	43.0	43.2	43.9	43.3	45.8	48.0	49.4	52.0	52.3	54.8		
	40.2	40.7	41.4	42.1	43.0	43.2	43.6	45.1	48.0	47.0	50.6	51.0	51.1	53.7		
	38.2	41.0	41.9	41.6	43.0	43.4	43.3	44.4	46.3	51.0	50.2	51.0	52.1	54.2		
	40.1	39.0	41.8	42.3	43.0	43.8	43.6	44.4	45.4	48.0	49.8	50.0	52.1	53.3		
	38.4	40.9	41.7	42.1	43.0	43.4	43.4	44.9	47.7	49.0	49.6	51.0	52.2	54.0		
M [mg/kg]	38.5	40.8	41.7	42.0	43.2	43.3	43.4	44.4	46.2	48.5	50.0	50.8	52.0	53.9		45.6
s [mg/kg]	1.32	0.95	0.18	0.29	0.41	0.27	0.37	0.62	1.43	1.38	0.45	0.75	0.45	0.55	s_M [mg/kg]	4.67
s_{rel}	0.034	0.023	0.004	0.007	0.009	0.006	0.009	0.014	0.031	0.028	0.009	0.015	0.009	0.010	\bar{s}_i [mg/kg]	0.79
																0.102

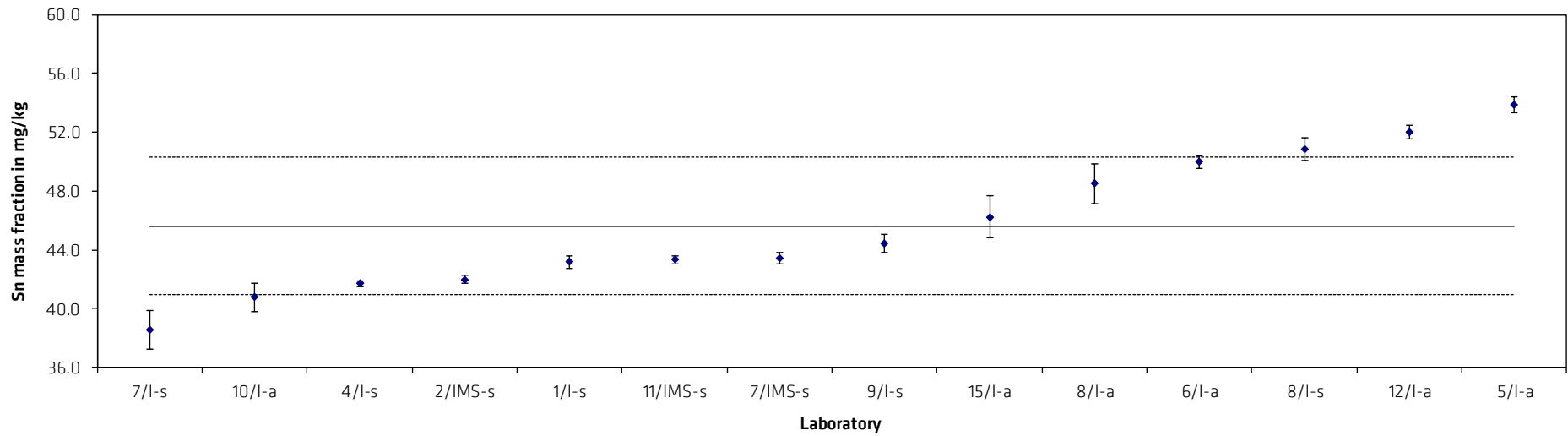
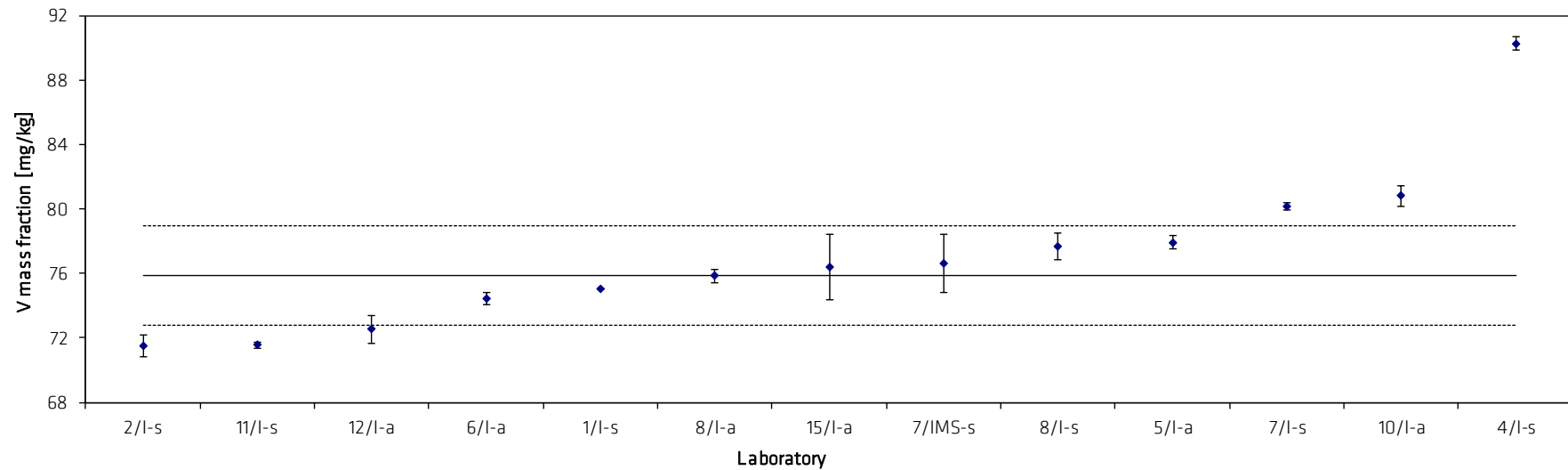


Table 22: Results for V

Lab./Meth.	2/l-s	11/l-s	12/l-a	6/l-a	1/l-s	8/l-a	15/l-a	7/IMS-s	8/l-s	5/l-a	7/l-s	10/l-a	4/l-s		
M_i [mg/kg]	72.1	71.4	72.8	75.0	75	76.0	76.2	77.6	78.0	78.1	80.5	81.5	90.5		n
	72.1	71.6	73.9	74.3	75	75.0	75.6	79.1	78.0	78.3	79.8	81.6	89.6		11
	72.1	71.4	71.2	73.9	75	76.0	75.9	76.5	78.0	78.0	79.9	80.5	90.6		
	70.9	71.5	72.5	74.4	75	76.0	80.4	77.3	78.0	77.2	80.2	80.8	90.0		
	70.9	71.5	72.1	74.2	75	76.0	75.6	75.0	78.0	77.8	80.3	80.0	90.8		
	70.9	71.9	72.7	74.6	75	76.0	74.6	74.2	76.0	78.1	80.2	80.4	90.1		
M [mg/kg]	71.5	71.5	72.5	74.4	75.0	75.8	76.4	76.6	77.7	77.9	80.2	80.8	90.3		75.9
s [mg/kg]	0.67	0.17	0.87	0.37	0.00	0.41	2.04	1.79	0.82	0.39	0.24	0.64	0.45	s_M [mg/kg]	3.06
s_{rel}	0.00940	0.00235	0.01197	0.00494	0.00000	0.00538	0.02671	0.02340	0.01051	0.00502	0.00302	0.00787	0.00494	\bar{s}_i [mg/kg]	0.89
															0.04030



The statistical evaluation of the data was performed using the software program BAM-Tool [4]. The Cochran-test was only performed once. The following results were obtained:

Tab. 23: Outcome of statistical tests on the results obtained for Si and Fe

	Si	Fe
Number of data sets	12	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 ($\alpha = 0.05$)	Lab. 11	Lab. 11-a
Nalimov ($\alpha = 0.01$)	---	---
Grubbs ($\alpha = 0.05$)	---	---
Grubbs ($\alpha = 0.01$)	---	---
Grubbs Pair ($\alpha = 0.05$)	---	---
Grubbs Pair ($\alpha = 0.01$)	---	---
Cochran ($\alpha = 0.01$)	---	Lab. 11-a
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 outliers were not removed.

Tab. 24: Outcome of statistical tests on the results obtained for Cu and Mn

	Cu	Mn
Number of data sets	14	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. 7	Lab. 5
Nalimov ($\alpha = 0.01$)	---	---
Grubbs ($\alpha = 0.05$)	---	---
Grubbs ($\alpha = 0.01$)	---	---
Grubbs Pair ($\alpha = 0.05$)	---	---
Grubbs Pair ($\alpha = 0.01$)	---	---
Cochran ($\alpha = 0.01$)	---	---
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 were not removed.

Tab. 25: Outcome of statistical tests on the results obtained for Mg and Cr

	Mg	Cr
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 ($\alpha = 0.05$)	Labs. 8/I-a, 6	---
Nalimov ($\alpha = 0.01$)	---	---
Grubbs ($\alpha = 0.05$)	---	---
Grubbs ($\alpha = 0.01$)	---	---
Grubbs Pair ($\alpha = 0.05$)	---	---
Grubbs Pair ($\alpha = 0.01$)	---	---
Cochran ($\alpha = 0.01$)	Labs. 9/A	Lab. 7/IMS
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 outliers were not removed.

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

	Zn	Ga
Number of data sets	14	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. 5
Nalimov ($\alpha = 0.01$)	---	---
Grubbs ($\alpha = 0.05$)	---	---
Grubbs ($\alpha = 0.01$)	---	---
Grubbs Pair ($\alpha = 0.05$)	---	---
Grubbs Pair ($\alpha = 0.01$)	---	---
Cochran ($\alpha = 0.01$)	---	Lab. 11-a
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 outliers were not removed.

Tab. 27: 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 ($\alpha = 0.05$)	---	---
Dixon ($\alpha = 0.01$)	---	---
Nalimov ($\alpha = 0.05$)	Lab. 4	---
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 ($\alpha = 0.01$)	Lab. 11-a	Lab. 11-a
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. 4, 1st run) was removed.

Tab. 28: Outcome of statistical tests on the results obtained for Sc

	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	---
Dixon ($\alpha = 0.01$)	Lab. 7	---
Nalimov ($\alpha = 0.05$)	Lab. 7	Lab. 5
Nalimov ($\alpha = 0.01$)	Lab. 7	---
Grubbs ($\alpha = 0.05$)	Lab. 7	---
Grubbs ($\alpha = 0.01$)	Lab. 7	---
Grubbs Pair ($\alpha = 0.05$)	---	---
Grubbs Pair ($\alpha = 0.01$)	---	---
Cochran ($\alpha = 0.01$)	---	---
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. 7, 1st run) was removed.

Tab. 29: Outcome of statistical tests on the results obtained for Zr and Ni

	Zr	Ni
Number of data sets	14	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 ($\alpha = 0.05$)	---	---
Nalimov ($\alpha = 0.01$)	---	---
Grubbs ($\alpha = 0.05$)	---	---
Grubbs ($\alpha = 0.01$)	---	---
Grubbs Pair ($\alpha = 0.05$)	---	---
Grubbs Pair ($\alpha = 0.01$)	---	---
Cochran ($\alpha = 0.01$)	Lab. 8-s	Lab. 7/l
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 outliers were not removed.

Tab. 30: Outcome of statistical tests on the results obtained for Be and Ca

	Be	Ca
Number of data sets	13	7
Scheffe's test (data compatible?)	yes	yes
Snedecor-F-Test and Bartlett-Test	Pooling not allowed	Pooling not allowed
Dixon ($\alpha = 0.05$)	---	Lab. 1
Dixon ($\alpha = 0.01$)	---	---
Nalimov ($\alpha = 0.05$)	Lab. 11/IMS	Lab. 1
Nalimov ($\alpha = 0.01$)	---	---
Grubbs ($\alpha = 0.05$)	---	---
Grubbs ($\alpha = 0.01$)	---	---
Grubbs Pair ($\alpha = 0.05$)	---	---
Grubbs Pair ($\alpha = 0.01$)	---	---
Cochran ($\alpha = 0.01$)	Lab. 9	Lab. 6
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	insufficient data
Skewness & Kurtosis Test ($\alpha = 0.01$)	Distribution: normal	insufficient data

The outliers were not removed.

Tab. 31: Outcome of statistical tests on the results obtained for Co and Na

	Co	Na
Number of data sets	14	6
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 ($\alpha = 0.01$)	Lab. 6	Lab. 6
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	Insufficient data
Skewness & Kurtosis Test ($\alpha = 0.01$)	Distribution: normal	Insufficient data

The outliers were not removed.

Tab. 32: Outcome of statistical tests on the results obtained for Li

	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 ($\alpha = 0.05$)	Lab. 8/I-a	Lab. 11/I-s
Dixon ($\alpha = 0.01$)	---	---
Nalimov ($\alpha = 0.05$)	---	Lab. 11/I-s
Nalimov ($\alpha = 0.01$)	---	---
Grubbs ($\alpha = 0.05$)	Lab. 8/I-a	Lab. 11/I-s
Grubbs ($\alpha = 0.01$)	---	---
Grubbs Pair ($\alpha = 0.05$)	---	---
Grubbs Pair ($\alpha = 0.01$)	---	---
Cochran ($\alpha = 0.01$)	Lab. 1	Lab. 1
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. 8/I-a) was removed.

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 ($\alpha = 0.05$)	Lab. 5	---
Dixon ($\alpha = 0.01$)	---	---
Nalimov ($\alpha = 0.05$)	Lab. 5	Lab. 8/I-s
Nalimov ($\alpha = 0.01$)	---	---
Grubbs ($\alpha = 0.05$)	---	---
Grubbs ($\alpha = 0.01$)	---	---
Grubbs Pair ($\alpha = 0.05$)	---	---
Grubbs Pair ($\alpha = 0.01$)	---	---
Cochran ($\alpha = 0.01$)	Lab. 1	---
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. 5) was removed, the straggler (Lab. 8) was not removed.

Tab. 31: Outcome of statistical tests on the results obtained for Pb and Sn

	Pb	Sn
Number of data sets	12	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 ($\alpha = 0.05$)	---	---
Nalimov ($\alpha = 0.01$)	---	---
Grubbs ($\alpha = 0.05$)	---	---
Grubbs ($\alpha = 0.01$)	---	---
Grubbs Pair ($\alpha = 0.05$)	---	---
Grubbs Pair ($\alpha = 0.01$)	---	---
Cochran ($\alpha = 0.01$)	Lab. 8/I-a	Lab. 8/I-s
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 outliers were not removed.

Tab. 32: Outcome of statistical tests on the results obtained for V

	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. 4	---
Dixon ($\alpha = 0.01$)	---	---
Nalimov ($\alpha = 0.05$)	Lab. 4	---
Nalimov ($\alpha = 0.01$)	Lab. 4	---
Grubbs ($\alpha = 0.05$)	Lab. 4	---
Grubbs ($\alpha = 0.01$)	Lab. 4	---
Grubbs Pair ($\alpha = 0.05$)	---	---
Grubbs Pair ($\alpha = 0.01$)	---	---
Cochran ($\alpha = 0.01$)	Lab. 8/l-s	Lab. 8/l-s
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, 1st run) was removed.

The certified mass fractions of all elements were calculated as mean of the accepted data sets. These values are given in Table 33.

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 33: 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	0.1968	12	0.0063	0.0018	0.0011	0.0026	0.0034	0.00670	0.5339	1.3263		
Fe	0.2062	16	0.0026	0.0007	0.0012	0.0021	0.0025	0.00507	0.5895	1.0314		
Cu	0.1466	14	0.0028	0.0007	0.0010	0.0018	0.0022	0.00440	0.7022	1.2240		
Mn	0.6993	14	0.0090	0.0024	0.0005	0.0028	0.0037	0.00739	0.0706	0.3951		
Mg	3.9820	15	0.0437	0.0113	0.0080	0.0359	0.0385	0.07692	0.2005	0.9013		
Cr	0.1044	15	0.0023	0.0006	0.0005	0.0008	0.0011	0.00227	0.4851	0.7889		
Zn	0.2524	14	0.0059	0.0016	0.0007	0.0024	0.0030	0.00592	<i>0.2788</i>	0.9532		
Ti	0.1016	14	0.0017	0.0005	0.0012	0.0014	0.0019	0.00383	1.1518	1.4225		
Sc	0.2820	11	0.0045	0.0013	0.0024	0.0018	0.0033	0.00668	<i>0.8654</i>	0.6515		
Ga	0.0208	14	0.0009	0.0002	0.0002	0.0002	0.0004	0.00077	1.1017	0.9475		
Zr	0.1022	14	0.0020	0.0005	0.0023	0.0004	0.0024	0.00478	2.2565	0.3479		
	mg/kg		mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg				
Ni	20.9	15	1.89	0.4875	0.2200	0.3524	0.6405	1.2810	1.0524	1.6863		
Be	22.4	13	0.43	0.1193	0.2581	0.1930	0.3437	0.6873	1.1522	0.8618		
Ca	11.70	7	2.66	1.0054	0.2538	0.2717	1.0719	2.1439	2.1693	2.3226		
Cd	15.20	10	1.07	0.3384	0.8084	0.2188	0.9033	1.8065	5.3184	1.4394		
Co	20.9	14	0.94	0.2512	0.0983	0.5251	0.5904	1.1807	0.4709	2.5162		
Li	9.09	9	0.28	0.0933	0.1543	0.0235	0.1819	0.3638	1.6980	0.2585		
Pb	44.8	12	3.41	0.9850	0.1398	0.6231	1.1739	2.3477	0.3122	1.3910		
Sn	45.6	14	4.67	1.2478	0.5525	0.4706	1.4435	2.8871	1.2117	1.0319		
V	75.9	11	3.06	0.9218	0.6515	0.4173	1.2035	2.4069	0.8588	0.5500		
Na	6.40	6	0.54	0.2213	0.5610	0.0276	0.6037	1.2074	8.7655	0.4306		

italic: measurement performed with XRF

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. 34 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. 34: Comparison wet chemistry 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	0.197	0.007	12	0.197	0.009	16
Fe	0.2062	0.0026	16	0.215	0.006	16
Cu	0.1466	0.0028	14	0.145	0.010	16
Mn	0.699	0.009	14	0.685	0.015	16
Mg	3.98	0.05	15	4.01	0.06	16
Cr	0.1044	0.0023	15	0.107	0.005	15
Zn	0.252	0.006	14	0.262	0.009	15
Ti	0.1016	0.0017	14	0.099	0.005	16
Ga	0.0208	0.0009	14	0.0232	0.0024	14
Sc	0.282	0.005	11	0.275	0.035	8
Zr	0.1022	0.0020	14	0.097	0.006	16
	in mg/kg	in mg/kg		in mg/kg	in mg/kg	
Ni	20.9	1.9	15	29.2	14.7	12
Be	22.4	0.5	13	21.6	1.4	15
Ca	11.7	2.7	7	11.8	1.0	16
Cd	15.2	1.1	10	20.11	2.4	15
Co	20.9	1.0	14	22.4	3.7	16
Li	9.07	0.27	10	9.5	1.1	16
Na	6.4	0.6	6	6.3	1.2	15
Pb	44.8	3.5	12	50.3	6.9	15
Sn	45.6	4.7	14	47.9	5.6	13
V	75.9	3.1	12	78.3	5.2	13

6. Instructions for users and stability

The certified reference material BAM-M320 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 validation and quality control of wet chemical analysis methods.

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

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

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. Metrological Traceability

To ensure traceability of the certified mass fractions to the SI (Système International d'Unités) calibration was performed 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-M320 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-M320 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] DIN EN 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] Lisec, J.: BAM Ecerto Softwaretool (BAM 2019)
- [5] DIN 1333:1992-02 Zahlenangaben

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

Silicon:

Sample	Number	Sum	Mean	Variance		
A1	5	0.9560	0.1912	3.2E-06		
A2	5	0.9500	0.1900	0.000001		
A3	5	0.9520	0.1904	0.0000028		
A4	5	0.9440	0.1888	0.0000012		
A5	5	0.9430	0.1886	8E-07		
B1	5	0.9520	0.1904	0.0000013		
B2	5	0.9510	0.1902	7E-07		
B3	5	0.9430	0.1886	8E-07		
B4	5	0.9450	0.1890	0.0000015		
B5	5	0.9490	0.1898	7E-07		
C1	5	0.9550	0.1910	0.000002		
C2	5	0.9530	0.1906	3E-07		
C3	5	0.9420	0.1884	8E-07		
C4	5	0.9470	0.1894	8E-07		
C5	5	0.9500	0.1900	0.0000025		
D1	5	0.9440	0.1888	7E-07		
D2	5	0.9550	0.1910	0.000001		
D3	5	0.9550	0.1910	5E-07		
D4	5	0.9390	0.1878	0.0000002		
D5	5	0.9470	0.1894	0.0000013		
E1	5	0.9570	0.1914	0.0000018		
E2	5	0.9490	0.1898	0.0000017		
E3	5	0.9430	0.1886	3E-07		
E4	5	0.9490	0.1898	0.0000017		
E5	5	0.9400	0.1880	0.0000015		
F1	5	0.9560	0.1912	7E-07		
F2	5	0.9540	0.1908	7E-07		
F3	5	0.9500	0.1900	0.000002		
F4	5	0.9470	0.1894	3E-07		
F5	5	0.9450	0.1890	5E-07		
			0.1897			
ANOVA						
Source of variation	sums of squares (SS)	degrees of freedom (df)	Mean squares (MS)	F-value	P-value	critical F-value
Between groups	0.00015317	29	5.2818E-06	4.48881508	2.6502E-09	1.56207098
Within groups	0.0001412	120	1.1767E-06			
Total	0.00029437	149				
within-sd	0.001085					
effective n	4.00					
s_bb	0.001013					
s_bb_min	0.000195					
u_bb	0.001013					
u_bb(rel.)	0.533902					

Iron:

<i>Sample</i>	<i>Number</i>	<i>Sum</i>	<i>Mean</i>	<i>Variance</i>		
A1	5	1.0170	0.2034	3.3E-06		
A2	5	1.0130	0.2026	1.8E-06		
A3	5	1.0150	0.2030	4.5E-06		
A4	5	1.0080	0.2016	0.0000023		
A5	5	1.0010	0.2002	7E-07		
B1	5	1.0160	0.2032	7E-07		
B2	5	1.0170	0.2034	2.3E-06		
B3	5	1.0040	0.2008	0.0000022		
B4	5	1.0070	0.2014	0.0000023		
B5	5	1.0100	0.2020	0.000001		
C1	5	1.0200	0.2040	2.5E-06		
C2	5	1.0160	0.2032	1.2E-06		
C3	5	1.0010	0.2002	0.0000022		
C4	5	1.0090	0.2018	0.0000002		
C5	5	1.0120	0.2024	4.3E-06		
D1	5	1.0100	0.2020	0.000001		
D2	5	1.0130	0.2026	1.3E-06		
D3	5	1.0190	0.2038	7E-07		
D4	5	1.0040	0.2008	2.7E-06		
D5	5	1.0100	0.2020	0.000002		
E1	5	1.0240	0.2048	2.2E-06		
E2	5	1.0080	0.2016	0.0000013		
E3	5	1.0040	0.2008	0.0000022		
E4	5	1.0120	0.2024	1.3E-06		
E5	5	1.0010	0.2002	0.0000017		
F1	5	1.0170	0.2034	3E-07		
F2	5	1.0200	0.2040	2E-06		
F3	5	1.0150	0.2030	1.5E-06		
F4	5	1.0110	0.2022	7E-07		
F5	5	1.0090	0.2018	7E-07		
			0.2023			
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	0.00021627	29	7.4577E-06	4.21339048	1.2437E-08	1.56207098
Within groups	0.0002124	120	1.77E-06			
Total	0.00042867	149				
within-sd	0.00133					
effective n	4.00					
s_bb	0.001192					
s_bb_min	0.000239					
u_bb	0.001192					
u_bb(rel.)	0.58948279					

Copper:

Sample	Number	Sum	Mean	Variance		
A1	5	0.7270	0.1454	0.0000013		
A2	5	0.7250	0.1450	0.0000015		
A3	5	0.7130	0.1426	0.0000018		
A4	5	0.7190	0.1438	0.0000012		
A5	5	0.7220	0.1444	3E-07		
B1	5	0.7170	0.1434	0.0000018		
B2	5	0.7180	0.1436	8E-07		
B3	5	0.7220	0.1444	3E-07		
B4	5	0.7130	0.1426	0.0000018		
B5	5	0.7140	0.1428	0.0000012		
C1	5	0.7180	0.1436	0.0000023		
C2	5	0.7100	0.1420	0.0000015		
C3	5	0.7230	0.1446	3E-07		
C4	5	0.7210	0.1442	0.0000002		
C5	5	0.7110	0.1422	3.2E-06		
D1	5	0.7200	0.1440	0		
D2	5	0.7180	0.1436	8E-07		
D3	5	0.7100	0.1420	0.0000015		
D4	5	0.7230	0.1446	8E-07		
D5	5	0.7130	0.1426	0.0000018		
E1	5	0.7210	0.1442	0.0000022		
E2	5	0.7150	0.1430	0.0000002		
E3	5	0.7250	0.1450	5E-07		
E4	5	0.7160	0.1432	0.0000012		
E5	5	0.7190	0.1438	0.0000012		
F1	5	0.7280	0.1456	3E-07		
F2	5	0.7170	0.1434	0.0000018		
F3	5	0.7100	0.1420	0.0000015		
F4	5	0.7190	0.1438	0.0000012		
F5	5	0.7120	0.1424	8E-07		
			0.1436			
ANOVA						
Source of variation	sums of squares (SS)	degrees of freedom (df)	Mean squares (MS)	F-value	P-value	critical F-value
Between groups	0.00015379	29	5.3032E-06	4.28831676	8.1466E-09	1.56207098
Within groups	0.0001484	120	1.2367E-06			
Total	0.00030219	149				
within-sd	0.001112					
effective n	4.00					
s_bb	0.001008					
s_bb min	0.0002					
u_bb	0.001008					
u_bb(rel.)	0.70218068					

Magnesium:

Sample	Number	Sum	Mean	Variance		
A1	5	20.2700	4.0540	0.00053		
A2	5	20.2300	4.0460	0.00083		
A3	5	20.2800	4.0560	0.00028		
A4	5	20.1900	4.0380	0.00047		
A5	5	20.1700	4.0340	0.00018		
B1	5	20.3200	4.0640	0.00013		
B2	5	20.2400	4.0480	0.00037		
B3	5	20.1700	4.0340	0.00048		
B4	5	20.1900	4.0380	2E-05		
B5	5	20.1400	4.0280	0.00017		
C1	5	20.2900	4.0580	0.00052		
C2	5	20.2700	4.0540	0.00043		
C3	5	20.2200	4.0440	0.00013		
C4	5	20.1800	4.0360	0.00023		
C5	5	20.1600	4.0320	0.00027		
D1	5	20.1100	4.0220	0.00022		
D2	5	20.2300	4.0460	8E-05		
D3	5	20.2600	4.0520	0.00017		
D4	5	20.1800	4.0360	0.00053		
D5	5	20.1800	4.0360	0.00073		
E1	5	20.2200	4.0440	0.00033		
E2	5	20.1600	4.0320	0.00047		
E3	5	20.1800	4.0360	0.00013		
E4	5	20.1500	4.0300	0.0001		
E5	5	20.1000	4.0200	0.00055		
F1	5	20.2200	4.0440	8E-05		
F2	5	20.2800	4.0560	0.00023		
F3	5	20.2300	4.0460	0.00038		
F4	5	20.2200	4.0440	0.00068		
F5	5	20.1600	4.0320	0.00017		
			4.0413			
ANOVA						
Source of variation	sums of squares (SS)	degrees of freedom (df)	Mean squares (MS)	F-value	P-value	critical F-value
Between groups	0.01717333	29	0.00059218	1.79631115	0.01508117	1.56207098
Within groups	0.03956	120	0.00032967			
Total	0.05673333	149				
within-sd	0.018157					
effective n	4.00					
s_bb	0.008101					
s_bb_min	0.003262					
u_bb	0.008101					
u_bb(rel.)	0.20045839					

Chromium:

<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	0.0042707	29	0.00014727	9.7912584	4.4419E-20	1.56207098
Within groups	0.00180486	120	1.5041E-05			
Total	0.00607557	149				
within-sd	0.003878					
effective n	4.00					
s_bb	0.005749					
s_bb_min	0.000697					
u_bb	0.005749					
u_bb (rel.)	0.48508641					

Nickel:

<i>Sample</i>	<i>Number</i>	<i>Sum</i>	<i>Mean</i>	<i>Variance</i>		
A1	5	0.0340	0.0068	0.0000002		
A2	5	0.0330	0.0066	0.0000003		
A3	5	0.0350	0.0070	9.404E-37		
A4	5	0.0350	0.0070	9.404E-37		
A5	5	0.0350	0.0070	9.404E-37		
B1	5	0.0350	0.0070	9.404E-37		
B2	5	0.0340	0.0068	0.0000002		
B3	5	0.0350	0.0070	9.404E-37		
B4	5	0.0350	0.0070	9.404E-37		
B5	5	0.0350	0.0070	9.404E-37		
C1	5	0.0350	0.0070	9.404E-37		
C2	5	0.0350	0.0070	9.404E-37		
C3	5	0.0350	0.0070	9.404E-37		
C4	5	0.0350	0.0070	9.404E-37		
C5	5	0.0340	0.0068	0.0000002		
D1	5	0.0350	0.0070	9.404E-37		
D2	5	0.0350	0.0070	9.404E-37		
D3	5	0.0350	0.0070	9.404E-37		
D4	5	0.0350	0.0070	9.404E-37		
D5	5	0.0350	0.0070	9.404E-37		
E1	5	0.0350	0.0070	9.404E-37		
E2	5	0.0340	0.0068	0.0000002		
E3	5	0.0350	0.0070	9.404E-37		
E4	5	0.0350	0.0070	9.404E-37		
E5	5	0.0350	0.0070	9.404E-37		
F1	5	0.0350	0.0070	9.404E-37		
F2	5	0.0330	0.0066	0.0000003		
F3	5	0.0350	0.0070	9.404E-37		
F4	5	0.0350	0.0070	9.404E-37		
F5	5	0.0350	0.0070	9.404E-37		
			0.0069			
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	1.9733E-06	29	6.8046E-08	1.45812808	0.0820305	1.56207098
Within groups	0.0000056	120	4.6667E-08			
Total	7.5733E-06	149				
within-sd	0.000216					
effective n	4.00					
s_bb	7.31E-05					
s_bb_min	3.88E-05					
u_bb	7.31E-05					
u_bb(rel.)	1.05242314					

Beryllium:

<i>Sample</i>	<i>Number</i>	<i>Sum</i>	<i>Mean</i>	<i>Variance</i>		
A1	5	98.0000	19.6000	0.3		
A2	5	100.0000	20.0000	0		
A3	5	96.0000	19.2000	0.2		
A4	5	98.0000	19.6000	0.3		
A5	5	97.0000	19.4000	0.3		
B1	5	96.0000	19.2000	0.2		
B2	5	98.0000	19.6000	0.3		
B3	5	96.0000	19.2000	0.2		
B4	5	97.0000	19.4000	0.3		
B5	5	98.0000	19.6000	0.3		
C1	5	99.0000	19.8000	0.2		
C2	5	96.0000	19.2000	0.2		
C3	5	96.0000	19.2000	0.2		
C4	5	98.0000	19.6000	0.3		
C5	5	100.0000	20.0000	0		
D1	5	100.0000	20.0000	0		
D2	5	98.0000	19.6000	0.3		
D3	5	96.0000	19.2000	0.2		
D4	5	99.0000	19.8000	0.2		
D5	5	99.0000	19.8000	0.2		
E1	5	100.0000	20.0000	0		
E2	5	96.0000	19.2000	0.2		
E3	5	99.0000	19.8000	0.2		
E4	5	99.0000	19.8000	0.2		
E5	5	98.0000	19.6000	0.3		
F1	5	100.0000	20.0000	0		
F2	5	98.0000	19.6000	0.3		
F3	5	96.0000	19.2000	0.2		
F4	5	98.0000	19.6000	0.3		
F5	5	97.0000	19.4000	0.3		
			19.5733			
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	11.8933333	29	0.41011494	1.98442714	0.00541796	1.56207098
Within groups	24.8	120	0.20666667			
Total	36.6933333	149				
within-sd	0.454606					
effective n	4.00					
s_bb	0.225526					
s_bb_min	0.081671					
u_bb	0.225526					
u_bb (rel.)	1.15221154					

Cobalt:

Sample	Number	Sum	Mean	Variance		
A1	5	111.0000	22.2000	0.2		
A2	5	110.0000	22.0000	0		
A3	5	110.0000	22.0000	0		
A4	5	110.0000	22.0000	0		
A5	5	110.0000	22.0000	0		
B1	5	111.0000	22.2000	0.2		
B2	5	110.0000	22.0000	0		
B3	5	109.0000	21.8000	0.2		
B4	5	110.0000	22.0000	0		
B5	5	110.0000	22.0000	0		
C1	5	112.0000	22.4000	0.3		
C2	5	112.0000	22.4000	0.3		
C3	5	109.0000	21.8000	0.2		
C4	5	109.0000	21.8000	0.2		
C5	5	110.0000	22.0000	0		
D1	5	110.0000	22.0000	0		
D2	5	110.0000	22.0000	0		
D3	5	111.0000	22.2000	0.2		
D4	5	110.0000	22.0000	0		
D5	5	110.0000	22.0000	0		
E1	5	111.0000	22.2000	0.2		
E2	5	110.0000	22.0000	0		
E3	5	110.0000	22.0000	0		
E4	5	110.0000	22.0000	0		
E5	5	110.0000	22.0000	0		
F1	5	110.0000	22.0000	0		
F2	5	112.0000	22.4000	0.3		
F3	5	110.0000	22.0000	0		
F4	5	110.0000	22.0000	0		
F5	5	110.0000	22.0000	0		
			22.0467			
ANOVA						
Source of variation	sums of squares (SS)	degrees of freedom (df)	Mean squares (MS)	F-value	P-value	critical F-value
Between groups	3.47333333	29	0.11977011	1.56221889	0.04996387	1.56207098
Within groups	9.2	120	0.07666667			
Total	12.6733333	149				
within-sd	0.276887					
effective n	4.00					
s_bb	0.103807					
s_bb_min	0.049743					
u_bb	0.103807					
u_bb(rel.)	0.47085054					

Lithium:

Sample	Number	Sum	Mean	Variance		
A1	5	40.0000	8.0000	0.005		
A2	5	39.3000	7.8600	0.013		
A3	5	39.5000	7.9000	0.005		
A4	5	38.9000	7.7800	0.007		
A5	5	38.3000	7.6600	0.003		
B1	5	40.3000	8.0600	0.003		
B2	5	39.8000	7.9600	0.008		
B3	5	38.8000	7.7600	0.013		
B4	5	38.8000	7.7600	0.013		
B5	5	38.6000	7.7200	0.012		
C1	5	40.1000	8.0200	0.007		
C2	5	40.0000	8.0000	0.005		
C3	5	38.5000	7.7000	0.005		
C4	5	38.6000	7.7200	0.002		
C5	5	38.8000	7.7600	0.003		
D1	5	39.8000	7.9600	0.003		
D2	5	39.7000	7.9400	0.008		
D3	5	40.1000	8.0200	0.007		
D4	5	38.9000	7.7800	0.007		
D5	5	38.8000	7.7600	0.003		
E1	5	40.3000	8.0600	0.003		
E2	5	39.4000	7.8800	0.002		
E3	5	39.0000	7.8000	0.005		
E4	5	39.0000	7.8000	0.005		
E5	5	38.4000	7.6800	0.002		
F1	5	39.4000	7.8800	0.002		
F2	5	40.1000	8.0200	0.012		
F3	5	39.7000	7.9400	0.008		
F4	5	38.8000	7.7600	0.003		
F5	5	38.8000	7.7600	0.008		
			7.8567			
ANOVA						
Source of variation	sums of squares (SS)	degrees of freedom (df)	Mean squares (MS)	F-value	P-value	critical F-value
Between groups	2.24033333	29	0.07725287	12.7339901	1.1486E-24	1.56207098
Within groups	0.728	120	0.00606667			
Total	2.96833333	149				
within-sd	0.077889					
effective n	4.00					
s_bb	0.133404					
s_bb_min	0.013993					
u_bb	0.133404					
u_bb(rel.)	1.6979684					

Lead:

<i>Sample</i>	<i>Number</i>	<i>Sum</i>	<i>Mean</i>	<i>Variance</i>		
A1	5	266.7000	53.3400	1.128		
A2	5	262.3000	52.4600	0.723		
A3	5	266.6000	53.3200	1.127		
A4	5	264.9000	52.9800	0.192		
A5	5	262.4000	52.4800	0.362		
B1	5	265.7000	53.1400	0.128		
B2	5	264.9000	52.9800	0.957		
B3	5	262.4000	52.4800	0.362		
B4	5	263.9000	52.7800	1.352		
B5	5	264.1000	52.8200	0.957		
C1	5	266.6000	53.3200	0.362		
C2	5	267.5000	53.5000	0.515		
C3	5	264.0000	52.8000	0.57		
C4	5	264.0000	52.8000	0.57		
C5	5	264.9000	52.9800	0.192		
D1	5	264.0000	52.8000	0.57		
D2	5	264.1000	52.8200	0.957		
D3	5	268.4000	53.6800	0.992		
D4	5	262.4000	52.4800	0.362		
D5	5	262.4000	52.4800	0.362		
E1	5	267.5000	53.5000	0.515		
E2	5	263.1000	52.6200	1.272		
E3	5	266.6000	53.3200	1.127		
E4	5	264.9000	52.9800	0.192		
E5	5	261.6000	52.3200	0.162		
F1	5	264.9000	52.9800	0.192		
F2	5	268.3000	53.6600	0.648		
F3	5	265.8000	53.1600	0.498		
F4	5	265.8000	53.1600	0.498		
F5	5	265.7000	53.1400	0.128		
			52.9760			
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	20.5456	29	0.70846897	1.18262124	0.26102695	1.56207098
Within groups	71.888	120	0.59906667			
Total	92.4336	149				
within-sd	0.773994					
effective n	4.00					
s_bb	0.16538					
s_bb_min	0.13905					
u_bb	0.16538					
u_bb (rel.)	0.31217927					

Vanadium:

<i>Sample</i>	<i>Number</i>	<i>Sum</i>	<i>Mean</i>	<i>Variance</i>		
A1	5	563.0000	112.6000	2.3		
A2	5	562.0000	112.4000	2.3		
A3	5	570.0000	114.0000	1.5		
A4	5	565.0000	113.0000	0.5		
A5	5	558.0000	111.6000	0.8		
B1	5	571.0000	114.2000	0.7		
B2	5	568.0000	113.6000	0.8		
B3	5	558.0000	111.6000	2.3		
B4	5	564.0000	112.8000	2.7		
B5	5	564.0000	112.8000	2.7		
C1	5	570.0000	114.0000	0.5		
C2	5	571.0000	114.2000	0.7		
C3	5	557.0000	111.4000	1.8		
C4	5	564.0000	112.8000	0.2		
C5	5	564.0000	112.8000	0.7		
D1	5	566.0000	113.2000	0.7		
D2	5	564.0000	112.8000	0.2		
D3	5	577.0000	115.4000	2.3		
D4	5	565.0000	113.0000	0.5		
D5	5	565.0000	113.0000	0		
E1	5	568.0000	113.6000	0.3		
E2	5	562.0000	112.4000	1.8		
E3	5	560.0000	112.0000	1.5		
E4	5	567.0000	113.4000	0.8		
E5	5	560.0000	112.0000	1.5		
F1	5	559.0000	111.8000	2.7		
F2	5	576.0000	115.2000	2.7		
F3	5	571.0000	114.2000	0.7		
F4	5	567.0000	113.4000	0.3		
F5	5	568.0000	113.6000	0.8		
			113.0933			
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	145.493333	29	5.01701149	4.03512989	3.4281E-08	1.56207098
Within groups	149.2	120	1.24333333			
Total	294.693333	149				
within-sd	1.115049					
effective n	4.00					
s_bb	0.971298					
s_bb_min	0.200321					
u_bb	0.971298					
u_bb(rel.)	0.85884626					

Copper:

r_0	0.136306832	0.146093168															
r_in	0.142	0.1407	0.1407	0.1389	0.1448	0.1431	0.1404	0.142									
r_middle	0.142	0.1463	0.1448	0.144	0.144	0.1441	0.1438	0.1451	0.1443	0.1443	0.1435	0.1443	0.1435	0.1425	0.1434	0.1446	
r_out	0.141	0.1407	0.1406	0.1404	0.1402	0.1438	0.1418	0.1422	0.14	0.1408	0.1407	0.1389	0.14	0.1404	0.1392	0.1408	
Source of variation	sums of squares (SS)	degrees of freedom (df)	Mean squares (MS)	F-value	P-value	critical F-value											
Between groups	9.38396E-05	3	3.12799E-05	11.20931148	2.07581E-05	2.851741336											
Within groups	0.00010604	38	2.79052E-06														
Total	0.00019988	41															
within-sd	0.001670486																
effective n	9.40																
s_bb	0.001741208																
s_bb min	0.000261014																
u_bb	0.001741208			0.142252941													
u_bb (rel.)	1.224022309																

Manganese:

r_0	0.641358356	0.673241644															
r_in	0.6604	0.6522	0.6512	0.6526	0.6625	0.652	0.6511	0.655									
r_middle	0.6598	0.6613	0.6581	0.6572	0.6635	0.6645	0.6603	0.659	0.6632	0.6661	0.6609	0.6597	0.6579	0.6654	0.659	0.6578	
r_out	0.6583	0.6645	0.6521	0.6495	0.6518	0.6616	0.6579	0.6565	0.6542	0.654	0.6549	0.658	0.6579	0.6645	0.6501	0.6516	
Source of variation	sums of squares (SS)	degrees of freedom (df)	Mean squares (MS)	F-value	P-value	critical F-value											
Between groups	0.000276946	3	9.23155E-05	3.195401878	0.034208664	2.851741336											
Within groups	0.001097824	38	2.88901E-05														
Total	0.00137477	41															
within-sd	0.005374951																
effective n	9.40																
s_bb	0.002598011																
s_bb min	0.000839838																
u_bb	0.002598011			0.657602941													
u_bb (rel.)	0.395072888																

Magnesium:

r_0	3.90624708	4.15575292															
r_in	4.0412	4.007	4.0236	4.0336	4.0413	4.0499	3.9522	4.0415									
r_middle	4.0323	4.0905	4.0405	4.0455	4.0802	4.0449	4.0659	4.0796	4.0779	4.096	4.1133	4.0878	4.1091	4.0866	4.0745	4.126	
r_out	3.9764	3.9785	3.964	4.0024	3.9896	3.9402	4.0338	3.9886	4.0136	4.02	4.0824	4.0297	4.0517	4.06	4.0049	3.977	
Source of variation	sums of squares (SS)	degrees of freedom (df)	Mean squares (MS)	F-value	P-value	critical F-value											
Between groups	0.042839584	3	0.014279861	7.666518693	0.000398198	2.851741336											
Within groups	0.070779808	38	0.001862627														
Total	0,113619392	41															
within-sd	0.043158157																
effective n	9.40																
s_bb	0.036351461																
s_bb min	0.006743478																
u_bb	0.036351461			4.033114706													
u_bb (rel.)	0.901324738																

Chromium:

r_0	0.102492921	0.105507079															
r_in	0.1047	0.1036	0.1036	0.1034	0.1035	0.1038	0.1033	0.1033									
r_middle	0.1051	0.1041	0.1038	0.1028	0.1036	0.1036	0.1029	0.1025	0.1032	0.1034	0.103	0.1029	0.1028	0.1029	0.1026	0.1021	
r_out	0.1052	0.1055	0.1043	0.1046	0.1049	0.1052	0.1046	0.1047	0.1045	0.1054	0.1043	0.104	0.1044	0.1049	0.1048	0.105	
Source of variation	sums of squares (SS)	degrees of freedom (df)	Mean squares (MS)	F-value	P-value	critical F-value											
Between groups	2.02724E-05	3	6.75748E-06	15.53789494	9.41698E-07	2.851741336											
Within groups	1.65263E-05	38	4.34903E-07														
Total	3.67988E-05	41															
within-sd	0.000659472																
effective n	9.40																
s_bb	0.000820269																
s_bb min	0.000103043																
u_bb	0.000820269			0.103979412													
u_bb (rel.)	0.788876697																

Zinc:

r_0	0.266900261	0.279499739															
r_in	0.2722	0.2728	0.2694	0.2689	0.2709	0.2711	0.2725	0.274									
r_middle	0.277	0.2742	0.2755	0.2763	0.2747	0.2744	0.274	0.2755	0.2757	0.2779	0.2772	0.2752	0.2775	0.2745	0.276	0.2753	
r_out	0.2683	0.2743	0.2696	0.2705	0.2729	0.2725	0.2726	0.2727	0.268	0.2701	0.2704	0.2712	0.2706	0.2697	0.2685	0.2724	
Source of variation	sums of squares (SS)	degrees of freedom (df)	Mean squares (MS)	F-value	P-value	critical F-value											
Between groups	0.00020453	3	6.81768E-05	14.846331	1.49504E-06	2.851741336											
Within groups	0.000174502	38	4.59216E-06														
Total	0.000379032	41															
within-sd	0.002142933																
effective n	9.40																
s_bb	0.00260127																
s_bb_min	0.000334834																
u_bb	0.00260127			0.272908824													
u_bb(rel.)	0.95316443																

Titanium:

r_0	0.081903733	0.119896267															
r_in	0.1054	0.0999	0.1023	0.1033	0.1034	0.1052	0.1295	0.1033									
r_middle	0.1023	0.1039	0.1044	0.1003	0.1047	0.1019	0.1042	0.1027	0.1016	0.1025	0.1025	0.1026	0.1012	0.1052	0.098	0.1014	
r_out	0.1056	0.1054	0.1051	0.1064	0.1063	0.1066	0.1059	0.1071	0.1053	0.1073	0.1065	0.1066	0.1049	0.1071	0.1069	0.1068	
Source of variation	sums of squares (SS)	degrees of freedom (df)	Mean squares (MS)	F-value	P-value	critical F-value											
Between groups	0.000173416	3	5.78053E-05	1.564944296	0.213745532	2.851741336											
Within groups	0.00140363	38	3.69376E-05														
Total	0.001577046	41															
within-sd	0.006077634																
effective n	9.40																
s_bb	0.001490208																
s_bb_min	0.000949632																
u_bb	0.001490208			0.104758824													
u_bb(rel.)	1.422512695																

Gallium:

r_0	193.3522158	201.2477842															
r_in	198.341	198.741	200.041	197.841	200.141	202.041	196.541	198.341									
r_middle	199.4	200	200.5	200.1	200.2	200.4	200.7	200.5	200.3	201	202.4	201.5	202.2	200.7	200.6	201.5	
r_out	197.3	196.2	197.5	196.9	198.3	197.2	197.5	196.9	196.9	198.4	198	197.4	196.1	197.8	196	197	
Source of variation	sums of squares (SS)	degrees of freedom (df)	Mean squares (MS)	F-value	P-value	critical F-value											
Between groups	105.5269603	3	35.17565343	19.49464686	8.1224E-08	2.851741336											
Within groups	68.56625	38	1.804375														
Total	174.0932103	41															
within-sd	1.343270263																
effective n	9.40																
s_bb	1.884498706																
s_bb min	0.20988646																
u_bb	1.884498706			198.8861176													
u_bb (rel.)	0.947526518																

Zirconium:

r_0	926.5124508	1061.687549															
r_in	987.1	985.5	986.6	982.7	978.5	998.1	956	1068.1									
r_middle	1001.5	993.7	990.9	971.3	990.3	980.2	973.3	969.6	986.9	986.8	984.6	982.9	985.4	973.4	984.1	969.4	
r_out	1000.1	1001.4	988	987.9	986.3	997.8	995	993.3	985.8	1006.3	992.5	994.6	997.8	992.2	992.9	1002.6	
Source of variation	sums of squares (SS)	degrees of freedom (df)	Mean squares (MS)	F-value	P-value	critical F-value											
Between groups	1278.436012	3	426.1453373	0.876601967	0.461764049	2.851741336											
Within groups	18473.06237	38	486.1332202														
Total	19751.49838	41															
within-sd	22.04842897																
effective n	9.40																
s_bb	0																
s_bb min	3.445074921																
u_bb	3.445074921			990.3470588													
u_bb (rel.)	0.347865417																

Nickel:

r_0	0.001757878	0.002442122																		
r_in	0.0022	0.0022	0.0019	0.002	0.002	0.0022	0.0021	0.0021												
r_middle	0.0022	0.0022	0.0022	0.0023	0.0023	0.0023	0.0022	0.0021	0.0023	0.0025	0.0021	0.0023	0.0022	0.002	0.0021	0.0021	0.0021	0.0021	0.0021	0.0021
r_out	0.0021	0.0022	0.0021	0.0022	0.002	0.0022	0.0022	0.0021	0.0019	0.0022	0.0021	0.0021	0.002	0.0022	0.0021	0.0021	0.0021	0.0021	0.0021	0.0022
Source of variation	sums of squares (SS)	degrees of freedom (df)	Mean squares (MS)	F-value	P-value	critical F-value														
Between groups	8.97321E-08	3	2.99107E-08	1.703496204	0.182604561	2.851741336														
Within groups	6.6722E-07	38	1.75584E-08																	
Total	7.56952E-07	41																		
within-sd	0.000132508																			
effective n	9.40																			
s_bb	3.62563E-05																			
s_bb_min	2.07045E-05																			
u_bb	3.62563E-05			0.00215																
u_bb(rel.)	1.686338082																			

Beryllium:

r_0	17.99417283	19.80582717																		
r_in	19.073	18.733	18.883	18.673	18.983	18.783	18.313	19.073												
r_middle	19.2	19.11	18.89	19.22	18.68	19.24	19.21	18.91	18.89	19.04	19.18	19.05	19.1	19.06	18.83	19.05				
r_out	18.68	19.18	18.7	18.79	18.6	18.89	18.65	18.11	18.43	18.91	19.11	18.46	18.65	18.77	18.43	18.63				
Source of variation	sums of squares (SS)	degrees of freedom (df)	Mean squares (MS)	F-value	P-value	critical F-value														
Between groups	1.024546321	3	0.34151544	3.667174233	0.020483615	2.851741336														
Within groups	3.538851964	38	0.093127683																	
Total	4.563398286	41																		
within-sd	0.305168287																			
effective n	9.40																			
s_bb	0.162582768																			
s_bb_min	0.047682654																			
u_bb	0.162582768			18.866																
u_bb(rel.)	0.861776574																			

Calcium:

r_0	10.21652562	12.42347438															
r_in	12.69	11.82	11.51	11.29	11.61	11.46	12.34	11.54									
r_middle	11.83	11.8	11.56	11.89	12.17	11.69	12.06	12.84	11.7	11.82	12.1	12.07	11.8	12.95	11.89	11.84	
r_out	12	11.3	11.48	11.29	11.48	11.56	11.35	11.3	11.52	11.43	11.52	11.61	11.23	11.45	11.34	11.63	
Source of variation	sums of squares (SS)	degrees of freedom (df)	Mean squares (MS)	F-value	P-value	critical F-value											
Between groups	2.625702976	3	0.875234325	4.871529995	0.005793019	2.851741336											
Within groups	6.827198929	38	0.17966313														
Total	9.452901905	41															
within-sd	0.423866877																
effective n	9.40																
s_bb	0.272069724																
s_bb_min	0.066229351																
u_bb	0.272069724			11.71382353													
u_bb (rel.)	2.322638063																

Cadmium:

r_0	17.90050138	30.13949862															
r_in	24.37	24.78	26.21	23.11	20.75	24.59	21.68	23.27									
r_middle	24.99	22.87	21.71	21.73	21.37	22.3	24.06	23.51	25.93	24.31	21.99	24.11	21.03	24.26	21.33	21.54	
r_out	24.05	26.25	24.37	25.68	25.19	21.66	22.61	24.39	18.89	22.98	23.61	23.94	22.57	22.01	22.55	23.34	
Source of variation	sums of squares (SS)	degrees of freedom (df)	Mean squares (MS)	F-value	P-value	critical F-value											
Between groups	3.899918155	3	1.299972718	0.277188362	0.841490486	2.851741336											
Within groups	178.2144204	38	4.689853169														
Total	182.1143386	41															
within-sd	2.165606882																
effective n	9.40																
s_bb	0																
s_bb_min	0.338376851																
u_bb	0.338376851			23.50882353													
u_bb (rel.)	1.439361056																

Cobalt:

r_0	14.82494589	21.75505411															
r_in	19.16	21.17	17.83	18.25	19.89	19.5	19.09	19.15									
r_middle	19.01	19.96	21.87	20.89	19.66	20.55	19.95	20.76	20.34	19.2	21.09	20.43	19.93	19.83	21.07	20.6	
r_out	21.49	19.54	20	19.39	20.18	20.57	19.13	20.18	20.18	20.12	20.45	21.89	18.45	20.4	19.15	18.06	
Source of variation	sums of squares (SS)	degrees of freedom (df)	Mean squares (MS)	F-value	P-value	critical F-value											
Between groups	11.37204048	3	3.790680159	2.653024748	0.062401013	2.851741336											
Within groups	54.29495	38	1.428814474														
Total	65.66699048	41															
within-sd	1.195330278																
effective n	9.40																
s_bb	0.501345383																
s_bb min	0.186770784																
u_bb	0.501345383			19.925													
u_bb (rel.)	2.516162523																

Lithium:

r_0	11.33219294	12.50780706															
r_in	11.85	11.92	11.92	11.9	11.68	11.82	12.4	11.84									
r_middle	11.74	11.86	11.74	11.89	11.89	11.98	12.02	11.95	11.91	11.98	11.97	11.96	11.96	12.16	11.89	11.92	
r_out	11.84	11.73	11.75	11.65	11.91	11.46	11.9	11.97	12.02	11.94	11.86	11.91	11.73	11.96	11.8	11.96	
Source of variation	sums of squares (SS)	degrees of freedom (df)	Mean squares (MS)	F-value	P-value	critical F-value											
Between groups	0.07349375	3	0.024497917	0.633822524	0.597795063	2.851741336											
Within groups	1.468740536	38	0.038651067														
Total	1.542234286	41															
within-sd	0.196598745																
effective n	9.40																
s_bb	0																
s_bb min	0.030718624																
u_bb	0.030718624			11.88235294													
u_bb (rel.)	0.258523076																

Tin:

r_0	90.20790239	99.99209761															
r_in	98.3	100.3	95.2	96.2	97	97.3	97.2	97.6									
r_middle	95.6	98.6	100.6	100.4	98	98.5	98	99.7	99.8	96.7	99.9	99.1	97.9	98.4	100.6	100.2	
r_out	100.1	97.2	97.4	96.9	97.4	98.9	95.5	96.8	98.4	97.7	97.2	99.1	95.6	97.2	96.5	94.9	
Source of variation	sums of squares (SS)	degrees of freedom (df)	Mean squares (MS)	F-value	P-value	critical F-value											
Between groups	38.42529762	3	12.80843254	3.96791629	0.014848871	2.851741336											
Within groups	122.6639881	38	3.227999687														
Total	161.0892857	41															
within-sd	1.796663487																
effective n	9.40																
s_bb	1.009722391																
s_bb_min	0.280729313																
u_bb	1.009722391			97.84705882													
u_bb(rel.)	1.03193944																

Vanadium:

r_0	128.8526383	158.9473617															
r_in	147	141.8	143	142.8	144.9	137.2	157	144.2									
r_middle	146.6	144.5	144.6	147	140.8	146.3	148.5	140.1	143.3	141	144.9	147.2	144.8	148.7	140.9	140.7	
r_out	148.4	152.4	148.8	146.7	145.7	146.4	144.9	139	142.3	145	149.6	140.2	144	146.2	142.4	144.6	
Source of variation	sums of squares (SS)	degrees of freedom (df)	Mean squares (MS)	F-value	P-value	critical F-value											
Between groups	10.62080357	3	3.540267857	0.135881271	0.938013885	2.851741336											
Within groups	990.0568155	38	26.05412672														
Total	1000.677619	41															
within-sd	5.104324316																
effective n	9.40																
s_bb	0																
s_bb_min	0.797552502																
u_bb	0.797552502			144.9970588													
u_bb(rel.)	0.550047365																

Scandium:

r_0	0.295197657	0.304802343				
r_in	0.296	0.294	0.29	0.296		
r_out	0.3	0.296	0.291	0.288		
<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	6.015E-05	2	3.0075E-05	1.359322034	0.317119736	4.737414128
Within groups	0.000154875	7	2.2125E-05			
Total	0.000215025	9				
within-sd	0.004703722					
effective n	3.20					
s_bb	0.00157619					
s_bb min	0.001922426					
u_bb	0.001922426			0.2951		
u_bb(rel.)	0.651449012					