Bundesanstalt für Materialforschung und -prüfung (BAM)

ECRM 578-2

Ferro-Molybdenum

Certification report

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S. Recknagel
Bundesanstalt für Materialforschung und -prüfung (BAM)
Division 1.6 „Inorganic Reference Materials"
Richard-Willstätter-Str. 11
D-12489 Berlin

Abstract

This report describes the preparation and certification of reference material ECRM 578-2, Ferro-Molybdenum (FeMo). The certified mass fractions are listed below.

Element	Mass fraction [1)] in %	Uncertainty *U* in %
C	0.0200	0.0014
P	0.0221	0.0007
S	0.0311	0.0010
Mo	72.19	0.18
Ni	0.0299	0.0007
Cu	0.3497	0.0029
Co	0.0069	0.0004
Sb	0.0018	0.0003
Sn	0.00305	0.00012

[1)] Unweighted mean value of the means of accepted sets of data (consisting of 4 single results), each set being obtained by a different laboratory and/or a different method of measurement.

The mass fractions of the following elements are given for information:

Element	Mass fraction [1)] in %
Si	0.185
Mn	0.0075
Cr	0.012
Fe	27.3
Pb	0.00039
Bi	0.00013

[1)] Unweighted mean value of the means of accepted sets of data (consisting of 4 single results), each set being obtained by a different laboratory and/or a different method of measurement.

ECRM 578-2 is available as a powder with a particle size in the range of 100 - 250 µm and is supplied in 60 mL glass bottles containing 100 g. The minimum amount of sample to be used for the determination is 0.2 g.

Contents

List of abbreviations

(if not explained elsewhere)

ETAAS	Electrothermal atomic absorption spectrometry
FAAS	Flame atomic absorption spectrometry
ICP-OES	Inductively coupled plasma - optical emission spectrometry
ICP-MS	Inductively coupled plasma – mass spectrometry
XRF	X-Ray fluorescence

1 Introduction

Ferro-molybdenum (FeMo), a molybdenum-iron alloy with 60 to 75 % molybdenum (mass fraction) is used in the steel industry as a master alloy. In steels, molybdenum is used as an alloying component to improve temperability, high-temperature strength and corrosion behaviour. The exact composition of the ferro-molybdenum used in steel production is important for the correct dosage of molybdenum and the input of accompanying elements, as well as due to the high price of molybdenum. The ECRM 578-2 is used to control the accompanying analysis and to calibrate the analytical methods used. ECRM 578-2 replaces the sold-out ECRM 578-1. The CRM was produced in accordance with the relevant ISO documents [1, 2].

2 Candidate material

About 240 kg of FeMo-powder (< 250 µm) was obtained from AG der Dillinger Hüttenwerke, Dillingen-Saar (Germany). This material was sieved over a 100 µm sieve in BAM. A total amount of 77 kg in a particle size range of 100 – 250 µm was taken as candidate material for ECRM 578-2.
The material is stored at ambient temperature in five containers. From these containers three samples each were taken from the top, the middle, and the bottom to test the homogeneity of the total batch.
ECRM 578-2 will be bottled in 60 mL glass bottles containing 100 g of material.

3 Homogeneity study

A total of 15 subsamples of the candidate material were selected from the total batch to give complete coverage of it. From each unit two independent test portions were analysed using different analytical methods (see Table 1). All measurements were performed in BAM.

Table 1. Analytical methods used for homogeneity testing

Element	Analytical method
C, S	Combustion/Infrared detection
Si, Mo, Fe	X-ray fluorescence spectrometry
Mn, P, Cr, Ni, Cu, Co, Sn, Sb, Pb	ICP-OES

All measurement results from the homogeneity study are given in Annex 2. The estimate of inhomogeneity contribution u_{bb} was calculated according to ISO Guide 35 [2] on the basis of the results of 1-way Analysis of Variance (ANOVA). The maximum of the following equations was used:

$$s_{bb} = \max\left(\sqrt{\frac{MS_{among} - MS_{within}}{n}}, 0\right) \quad (1)$$

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

where:

MS_{among}	mean of squared deviations between bottles (from 1-way ANOVA)
MS_{within}	mean of squared deviations within bottles (from 1-way ANOVA)
n	number of replicate sub-samples per bottle
N	number of bottles selected for homogeneity study

s_{bb} signifies the between-bottle 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}.

4 Stability

From former batches (ECRM 578-1) it is well known that the material is stable.
Therefore, the CRM will remain stable, provided that the bottle remains sealed and is stored in a cool and dry atmosphere. When the bottle has been opened the lid should be secured immediately after use. If the contents should become discoloured (e.g. oxidised) due to atmospheric contamination they should be discarded.

5 Certification study

5.1 Design of the study

The certification study was organised as an interlaboratory comparison. 26 laboratories invited to participate were selected on the basis of their expertise demonstrated in former certification interlaboratory comparisons in the framework of ECRM-certifications.
Most of the laboratories hold an accreditation according to ISO/IEC 17025.
Each participant received one unit of bottled candidate material and was asked to analyse four independent sub-samples for their element contents.
All participants were asked to give information on details of the applied analytical procedures. Table 2 shows the analytical parameters reported by the participating laboratories.

Table 2: Analytical procedures used by the participating laboratories

Lab-No.	Element	Sample mass	Sample pretreatment	Analytical method
1*	all elements	50 mg	Dissolution with aqua regia/HF	ICP-MS, calibration with commercial calibration solutions
2	Mn, Cr, Mo, Ni, Cu, Co, Fe	0.1 g	Microwave assisted digestion	ICP-OES, calibration with commercial calibration solutions (Merck)
3*	C	0.1 g		Combustion/Infrared detection, calibration with $CaCO_3$ (DIN EN ISO 15350)
	S	0.1 g		Combustion/Infrared detection, calibration with sulphur (DIN EN ISO 15350)
	Mn, P, Cr, Ni, Cu, Co, Sn, Sb, Pb	0.5 g	Dissolution in acid	ICP-OES, calibration with different pure chemicals (DIN 11885)
	Mo, Fe	0.5 g		XRF, calibration with different pure chemicals (DIN 51418-2)
4*	C	0.25 g		Combustion/Infrared detection, calibration with $NaHCO_3$ (a) or $BaCO_3$ (b)
	S	0.25 g		Combustion/Infrared detection, calibration with K_2SO_4 (a) or $BaSO_4$ (b)
	Si	4 g	Dissolution with HCl/HNO_3	Gravimetry (HCl-method)
	Mo	0.3 g	Decomposition with Na_2O_2 and Na_2CO_2	Gravimetry
	Mn	0.25 g	Dissolution with H_2SO_4/H_3PO_4	Spectrophotometry, calibration with commercial calibration solutions (Merck)
	Ni	0.5 g	Dissolution with HCl/HNO_3	Spectrophotometry, calibration with commercial calibration solutions (Merck)
	Sb	0.5 g	Dissolution with HCl/HNO_3	ETAAS, calibration with commercial calibration solutions (Merck)
	Mn, P, Cr, Mo, Ni, Cu, Co, Pb, Fe	0.5 g	Dissolution with HCl/HNO_3	ICP-OES, calibration with commercial calibration solutions (Merck)

*accredited acc. to ISO 17025

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

Lab-No.	Element	Sample mass	Sample pretreatment	Analytical method
5	Si, Mn, P, Cr, Mo, Ni, Cu, Co, Fe	0.25 g	Dissolution with HCl/HNO_3 on a hot plate	ICP-OES, calibration with commercial calibration solutions (Accu Standard)
6*	Si, Mn, P, Cr, Mo, Ni, Cu, Co, Fe	0.5 g	Dissolution with HNO_3 /HCl	ICP-OES, calibration with commercial calibration solutions
7	C	1 g		Combustion/Infrared detection, calibration with $CaCO_3$
	S	1 g		Combustion/Infrared detection, calibration with K_2SO_4
	Si, Mn, Cr, Ni, Cu, Sn, Pb, Fe	0.5 g	Microwave assisted digestion	ICP-OES, calibration with commercial calibration solutions (Spectrascan)
8*	C	0.25 g		Combustion/Infrared detection, calibration with WC (DIN EN ISO 15350)
	S	0.15 g		Combustion/Infrared detection, calibration with K_2SO_4 (DIN EN ISO 15350)
	Si, P, Mo, Fe	0.05 g	Dissolution in acid	ICP-OES, calibration with calibration solutions prepared from pure substances
	Mn, Cr, Ni, Cu, Co, Sn, Sb	0.05 g	Dissolution in acid	ICP-MS, calibration with calibration solutions prepared from pure substances
9*	Si, P, Mo, Cu	1 g	Dissolution in acid	ICP-OES, calibration with commercial calibration solutions
10*	Mn, P, Cr, Ni, Cu, Co, Sn	1 g	Dissolution with HCl/HNO_3	ICP-OES, calibration with calibration solutions prepared from pure substances
	Si	0.5 g	Dissolution with HCl/HNO_3	ICP-OES, calibration with calibration solutions prepared from pure substances
	Mo, Fe	0.1 g	Dissolution with H_2SO_4/H_3PO_4	ICP-OES, calibration with calibration solutions prepared from pure substances
11	C	0.3 g		Combustion/Infrared detection, calibration with $CaCO_3$
	S	0.3 g		Combustion/Infrared detection, calibration with $CaSO_4$
	Si, Mn, P, Cr, Mo, Ni, Cu, Co, Sn, Sb, Pb, Fe	1 g	Dissolution with aqua regia	ICP-OES, calibration with calibration solutions prepared from pure substances
12*	C			Combustion/Infrared detection, calibration with CO_2
	S			Combustion/Infrared detection, calibration with SO_3
	Si, Mn, P, Cr, Mo, Ni, Cu, Co, Fe			ICP-OES, calibration with commercial calibration solutions (NIST traceable)
13	Si	0.2 g	Dissolution with HNO_3/H_2SO_4	Gravimetry (HCl-method)
	Mo	0.2 g		XRF, calibration with different pure chemicals
14*	Sn, Sb, Pb	0.5 g	Dissolution in acid	ICP-MS, calibration with commercial calibration solutions (Supelco, Sigma Aldrich, Roth)
15*	C	0.1 g		Combustion/Infrared detection, calibration with $CaCO_3$ (DIN EN ISO 15350)
	S	0.1 g		Combustion/Infrared detection, calibration with $BaSO_4$ (DIN EN ISO 15350)
	Mo	0.5 g	Dissolution with HNO_3/H_2SO_4	Gravimetry (ISO 4173)
	P, Cr, Ni, Cu, Co, Sb, Pb	0.5 g	Dissolution with HNO_3, HCl, H_2SO_4 and HF	ICP-OES, calibration with commercial calibration solutions (DIN 11885)

*accredited acc. to ISO 17025

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

Lab-No.	Element	Sample mass	Sample pretreatment	Analytical method
16	C	0.25 g	Flux: W/Sn+ Fe	Combustion/Infrared detection, calibration with CO_2
	S	0.25 g	Flux: W/Sn+ Fe	Combustion/Ultraviolet detection, calibration with K_2SO_4
	Mo, Ni, Cu, Fe	0.05 g	Microwave assisted digestion with HNO_3, HCl and HF	ICP-OES, calibration with calibration solutions prepared from pure substances
	Si, Mn, Cr	0.05 g	Microwave assisted digestion with HNO_3, HCl and HF	ICP-OES, calibration with commercial calibration solutions (NIST traceable)
17*	Si, Mn, P, S, Mo, Ni, Cu, Sn, Fe	0.1 g	Microwave assisted acid digestion	ICP-OES, calibration with commercial calibration solutions (Merck)
18*	C	1 g		Combustion/Infrared detection, calibration with $CaCO_3$
	S	1 g		Combustion/Infrared detection, calibration with $BaSO_4$
19	C	0.25 g		Combustion/Infrared detection, calibration with $NaHCO_3$ (ASTM 1019)
	S	0.25 g		Combustion/Infrared detection, calibration with K_2SO_4 (ASTM 1019)
20	Mo	6.5 g		XRF, calibration with different pure chemicals
	Si, P, S, Ni, Cu	0.25 g	Dissolution with HNO_3/H_2O_2	ICP-OES, calibration with commercial calibration solutions (LabKings, Merck)
	Mn, Cr, Co, Sn, Sb, Pb	0.25 g	Dissolution with HNO_3/H_2O_2	ICP-MS, calibration with commercial calibration solutions (LabKings, Merck)
22	C	1 g		Combustion/Infrared detection, calibration with Sucrose (ISO 9556)
	S	1 g		Combustion/Infrared detection, calibration with K_2SO_4 (ISO 4935)
	Si, Cr, Co	0.5 g	Dissolution in acid	ICP-OES, calibration with commercial calibration solutions (Spectrascan)
	P, Sn, Sb, Pb, As	0.1 g	Dissolution in acid	ICP-MS, calibration with commercial calibration solutions (Spectrascan)
23*	C	0.3 g		Combustion/Infrared detection, calibration with $CaCO_3$
	S	0.3 g		Combustion/Infrared detection, calibration with $CsSO_4$
	Si, Mn, Cr, Mo, Ni, Fe	0.25 g		XRF, calibration with different pure chemicals
	P, Co, Sn, Sb, Pb	0.1 g	Dissolution in acid	ICP-MS, calibration with calibration solutions prepared from pure substances
	Cu, Mn	0.1 g	Dissolution in acid	ICP-OES, calibration with calibration solutions prepared from pure substances
24*	C	0.2 g		Combustion/Infrared detection, calibration with $CaCO_3$ (ASTM E1019)
	S	0.2 g		Combustion/Infrared detection, calibration with $BaSO_4$ (ASTM E1019)
	Si	1 g	Dissolution with HNO_3/H_2SO_4	Gravimetry (IS 12614 (P-3))
	P	0.5 g	Dissolution with HCl/HF	Titration (IS 12614 (P-5))
	Mn, Cr, Ni, Co, Sn, Sb, Pb	0.5 g	Dissolution with HCl/HF	ICP-OES, calibration with commercial calibration solutions (Merck)
	Cu, Fe	0.5 g	Dissolution with HCl/HF	FAAS, calibration with commercial calibration solutions (Merck)

*accredited acc. to ISO 17025

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

Lab-No.	Element	Sample mass	Sample pretreatment	Analytical method
25	C	0.2 g	Flux: 1.5 g W+ 0.8 g Fe	Combustion/Infrared detection, calibration with $BaCO_3$
	S	0.2 g	Flux: 1.5 g W+ 0.8 g Fe	Combustion/Ultraviolet detection, calibration with K_2SO_4
26	C	0.5 g	Flux: W/Sn+ Fe	Combustion/Infrared detection, calibration with $CaCO_3$
27	C	0.15 g		Combustion/Infrared detection, calibration with $BaCO_3$
	S	0.15 g		Combustion/Ultraviolet detection, calibration with $BaSO_4$
	Si, Sn, Sb	0.1 g	Microwave assisted HNO_3, HCl, and HF	ICP-OES, calibration with commercial calibration solutions (Merck)
	Sb	0.1 g	Microwave assisted HNO_3, HCl, and HF	ICP-MS, calibration with commercial calibration solutions (Merck)

*accredited acc. to ISO 17025

5.2 Participants

Afarak Elektrowerk Weisweiler GmbH, Eschweiler (Germany)
AG der Dillinger Hüttenwerke, Dillingen-Saar (Germany)
Alleima Tube AB, Sandviken (Sweden)
ALS Scandinavia AB, Luleå (Sweden)
ArcelorMittal Maizières Research SAS, Maizières-lès-Metz (France)
Bruker AXS GmbH, Karlsruhe (Germany)
Bundesanstalt für Materialforschung und -prüfung (BAM), Berlin (Germany)
Chemad GmbH, Duisburg (Germany)
CMC POLAND Sp. z o.o., Zawiercie (Poland)
CSIR-National Metallurgical Laboratory, Jamshedpur (India)
Dunaferr Labor Nonprofit Ltd., Dunaújváros (Hungary)
Elementar Analysensysteme GmbH, Langenselbold (Germany)
Eltra GmbH, Haan (Germany)
Höganäs Sweden AB, Höganäs (Sweden)
Horn & Co. Analytics GmbH, Wenden-Hünsborn (Germany)
IFW Dresden e.V., Dresden (Germany)
Inspectorate Griffith India Pvt. Lyd., Bhubaneswar Laboratory, Bhubaneswar (India)
Łukasiewicz Research Network – Upper Silesian Institute of Technology, Gliwice (Poland)
Narema, Närpiö (Finland)
Österreichisches Gießerei-Institut (ÖGI), Leoben (Austria)
Saarstahl AG, Völklingen (Germany)
Salzgitter Flachstahl GmbH, Salzgitter (Germany)
Tata Steel Limited, Jamshedpur (India)
ThyssenKrupp Steel Europe AG, Duisburg (Germany)
Treibacher Industrie AG, Althofen (Austria)
voestalpine Böhler Edelstahl GmbH & Co KG, Kapfenberg (Austria)

5.3 Analytical results and statistical evaluation

Following the rules of the EURONORM CRM producers' group in a first step, the data obtained were checked for technical errors, miscalculations and typing errors [3, 4]. After that, the mean of all datasets (*M*) was calculated together with the standard deviation of

the laboratories' means (*s*). All results being outside the interval "*M* ± 3 * *s*" (obvious erroneous value, OEV) were removed before testing the whole data for outliers (Grubbs (95%), Cochran (99%)). After removal of outliers the recalculated standard deviation was compared with the data from [5] to evaluate the results of the interlaboratory comparison. OEVs, outliers and results which increase the standard deviation significantly were referred to the laboratories for reanalysis. A second statistical evaluation of the data after inclusion of reanalysed results was performed to detect additional outliers. These data are presented in Annex 1. The bars in the graphic presentations indicate the standard deviation of individual results.

5.4 Calculation of uncertainty

The uncertainty of the certified values considering the uncertainty contributions from the homogeneity-test and the certification interlaboratory comparison was calculated following Equation (3)

$$u_c = \sqrt{u_{char}^2 + u_{bb}^2} \qquad (3)$$

with

$$u_{char} = \frac{s_M}{\sqrt{n}} \qquad (4)$$

n: number of data sets used for calculating the certified mass fractions

In older EURONORM-CRMs as a measure of the uncertainty of the certified values the half-width confidence interval C (95 %) of the certified mass content wass calculated according to Equation (5) and reported in the certificate

$$C(95\ \%) = \frac{t \times S_M}{\sqrt{n}} \qquad (5)$$

with
n: number of accepted mean values;
t: the Student's value at the chosen probability with n-1 degrees of freedom.

Table 3 shows the results of both calculations.

Table 3: results of uncertainty calculation

		uncertainty contribution from							
	M	n	s_M	u_{char}	u_{bb}*	$u_{combined}$	U	C(95%)	u_{bb} (rel)
	%		%	%	%	%	%		
C	0.0200	17	0.0024	0.0006	0.0003	0.0007	0.00133	0.00142	1.5834
Si	0.1845	18	0.0159	0.0038	0.0045	0.0058	0.01168	0.00830	2.4250
Mn	0.0075	17	0.0012	0.0003	0.0005	0.0006	0.00119	0.00060	7.0786
P	0.0221	15	0.0012	0.0003	0.0001	0.0003	0.00063	0.00065	0.4163
S	0.0311	19	0.0018	0.0004	0.0002	0.0005	0.00094	0.00090	0.7898
Cr	0.0117	18	0.0021	0.0005	0.0003	0.0006	0.00114	0.00107	2.2479
Mo	72.19	18	0.2320	0.0547	0.0659	0.0856	0.17122	0.11490	0.0912
Ni	0.0299	17	0.0014	0.0003	0.0000	0.0003	0.00068	0.00071	0.1478
Cu	0.3497	19	0.0056	0.0013	0.0006	0.0014	0.00285	0.00269	0.1778
Co	0.0069	15	0.0006	0.0002	0.0001	0.0002	0.00036	0.00034	1.3783
Sn	0.0031	14	0.0002	0.0000	0.0000	0.0001	0.00012	0.00011	1.1761
Sb	0.0018	14	0.0003	0.0001	0.0001	0.0002	0.00030	0.00018	7.5588
Pb	0.00039	8	0.00003	0.00001	0.00005	0.00005	0.00009	0.00013	11.8102
Fe	27.33	14	0.3790	0.1013	0.1153	0.1535	0.30693	0.23390	0.4218

*calculated from u_{bb}(rel): $u_{bb} = \frac{M \cdot u_{bb}(rel)}{100}$

It can be seen that for S, Mo, Cu, Co, Sb, and Sn C(95%) is lower than *U* ($U = 2 \cdot u_c$). For the elements C, P, and Ni C(95%) is slightly higher than *U*. Certified mass fractions and their respective uncertainties are rounded according to DIN 1333 [6].

The mass fractions of the elements Si, Mn, Cr, Pb, Bi (no homogeneity data available), and Fe are only given for information reporting only the mean value of the laboratories' means.

6 Information on the proper use of ECRM 578-2

6.1 Shelf life

As mentioned in Chapter 4 ECRM 578-2 is stable. The certificate is valid until there is a revocation from the producer of the material.

6.2 Recommendations for transport, storage and use

Transportation of the bottled sample does not require special precautions. The stability of the material allows the dispatch of the material at ambient temperature. When the bottle has been opened the lid should be secured immediately after use. The bottle should be stored in a cool and dry atmosphere.

6.3 Safety instructions

No hazardous effect is to be expected when the material is used under conditions usually adopted for the analysis of ferro-alloy powders. However, it is strongly recommended to handle and dispose the reference material in accordance with the guidelines for hazardous materials legally in force at the site of end use and disposal.

7 Metrological Traceability

The assigned values for ECRM 578-2 are achieved by inter-laboratory characterization, each laboratory using the method of their choice, details of which are given in Chapter 5. These methods are either stoichiometric analytical techniques or methods which are calibrated against pure metals or stoichiometric compounds. Most methods used were either international or national standard methods or methods which are technically equivalent.

8 References

[1] ISO 17034, General requirements for the competence of reference material producers, 2016

[2] ISO Guide 35, Reference materials - Guidance for characterization and assessment of homogeneity and stability, 2017

[3] ECRM-PG doc 2012-02 (October 2013) Statistical treatment of results of analysis for the certification of European certified reference materials (EURONORM-CRMs)

[4] CEN TR 10317:2020 European certified reference materials (EURONORM-CRMs) for the determination of the chemical composition of iron and steel products

[5] Review of the standard deviation [s(m)] of European certified reference material (EURONORM-CRM) produced between 1980 and 2021

[6] DIN 1333:1992-02 Zahlenangaben

9 Information on and purchase of the CRM

Certified reference material ECRM 578-2 is supplied by

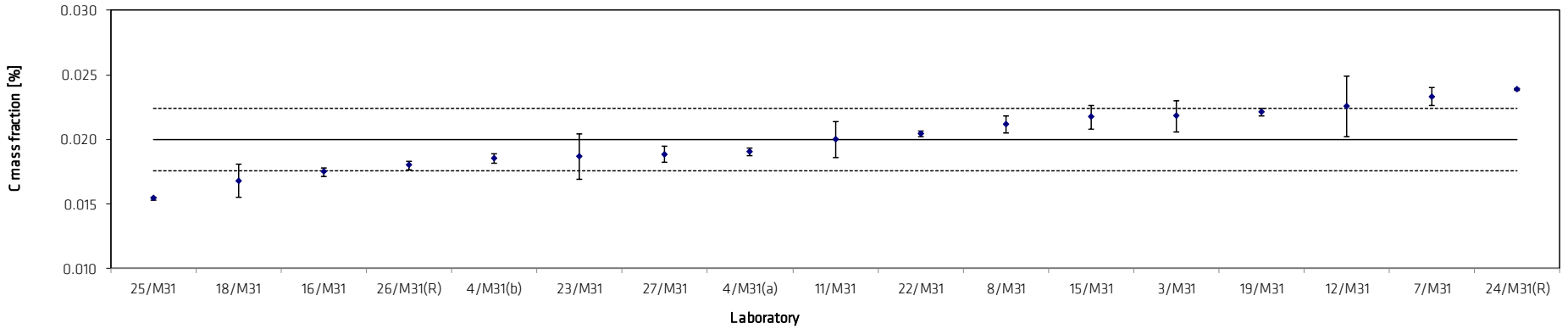
Bundesanstalt für Materialforschung und -prüfung (BAM)
Fachbereich 1.6: Anorganische Referenzmaterialien
Richard-Willstätter-Str. 11, D-12489 Berlin, Germany
Phone +49 (0)30 - 8104 2061
Fax: +49 (0)30 - 8104 72061
Email: sales.crm@bam.de
https://www.webshop.bam.de

Each unit 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.

Annex 1: Certification study

Table A1: Measurement results for carbon

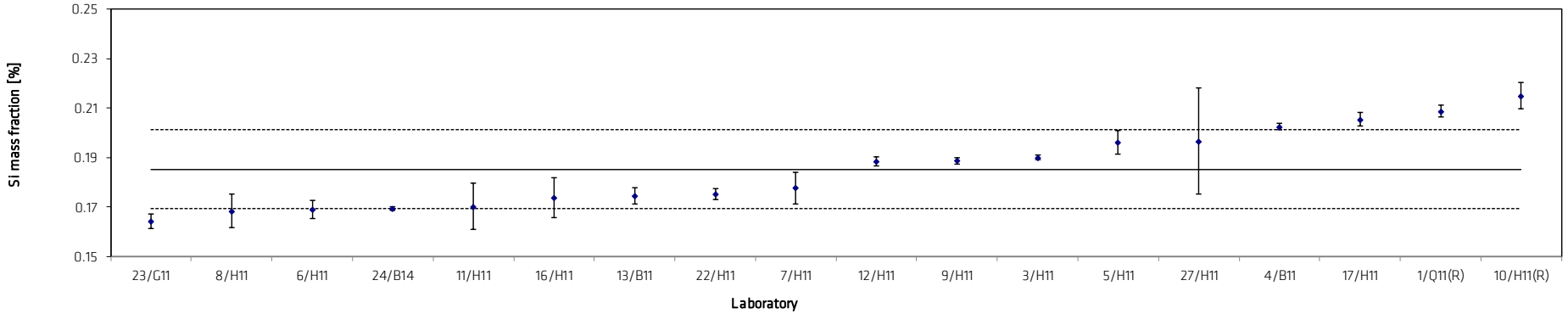
Lab./Meth.	25/M31	18/M31	16/M31	26/M31(R)	4/M31(b)	23/M31	27/M31	4/M31(a)	11/M31	22/M31	8/M31	15/M31	3/M31	19/M31	12/M31	7/M31	24/M31(R)		
M_i [%]	0.0155	0.0168	0.0172	0.0175	0.0186	0.0179	0.0194	0.0190	0.0185	0.02069	0.02089	0.02130	0.0213	0.02240	0.0216	0.0238	0.0239		n
	0.0153	0.0151	0.0179	0.0182	0.0190	0.0169	0.0193	0.0189	0.0193	0.02035	0.02103	0.02110	0.0233	0.02170	0.0251	0.0240	0.0240		17
	0.0153	0.0182	0.0174	0.0180	0.0184	0.0188	0.0187	0.0189	0.0216	0.02058	0.02207	0.02310	0.0222	0.02230	0.0197	0.0228	0.0238		
	0.0156	0.0172	0.0174	0.0183	0.0181	0.0210	0.0180	0.0194	0.0206	0.02017	0.02060	0.02140	0.0204	0.02210	0.0237	0.0226	0.0240		
M [%]	**0.0154**	**0.0168**	**0.0175**	**0.0180**	**0.0185**	**0.0187**	**0.0189**	**0.0190**	**0.0200**	**0.0204**	**0.0211**	**0.0217**	**0.0218**	**0.0221**	**0.0225**	**0.0233**	**0.0239**		**0.0200**
s [%]	0.00013	0.00127	0.00032	0.00032	0.00038	0.00176	0.00065	0.00027	0.00137	0.00023	0.00064	0.00093	0.00124	0.00031	0.00235	0.00070	0.00008	s_M [%]	0.00241
																		$\bar{s}_i$ [%]	0.00098
s_{rel}	0.00816	0.07587	0.01808	0.01793	0.02038	0.09414	0.03424	0.01397	0.06868	0.01140	0.03029	0.04258	0.05694	0.01399	0.10424	0.03014	0.00323		0.12037
Calibrant	BaCO3	CaCO3	CO2	CaCO3	BaCO3	CaCO3	BaCO3	Na2CO3	CaCO3	Succrose	WC	CaCO3	CaCO3	NaHCO3	CO2	CaCO3	CaCO3		



M31: Combustion/Infrared-detection
(R): reanalysed values

Table A2: Measurement results for silicon

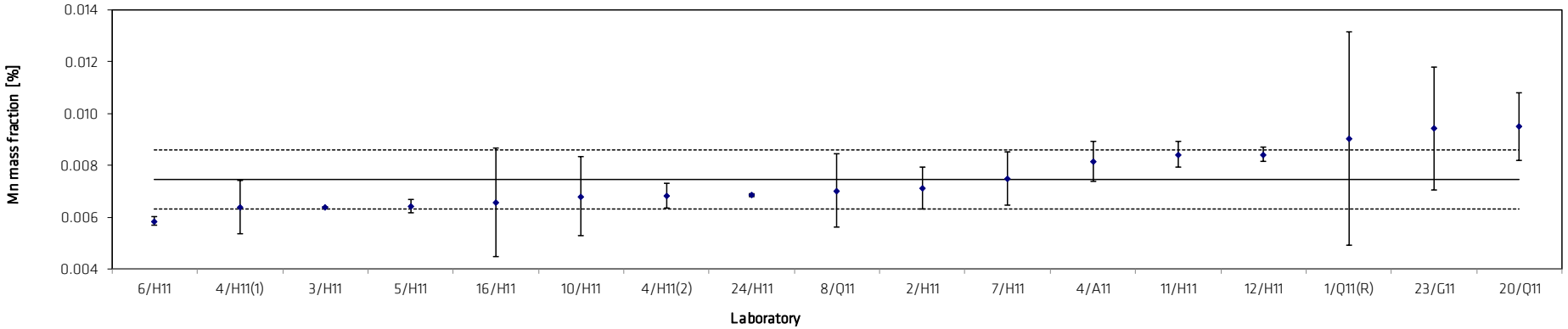
Lab./Meth.	23/G11	8/H11	6/H11	24/B14	11/H11	16/H11	13/B11	22/H11	7/H11	12/H11	9/H11	3/H11	5/H11	27/H11	4/B11	17/H11	1/Q11(R)	10/H11(R)		
M_i [%]	0.1640	0.1656	0.1688	0.1703	0.1671	0.1800	0.1780	0.1734	0.1738	0.1892	0.1890	0.1900	0.2030	0.1722	0.2020	0.2021	0.2064	0.2153		*n*
	0.1627	0.1787	0.1734	0.1693	0.1592	0.1805	0.1750	0.1778	0.1707	0.1901	0.1900	0.1911	0.1960	0.2244	0.2041	0.2049	0.2087	0.2090		18
	0.1619	0.1650	0.1696	0.1685	0.1734	0.1703	0.1750	0.1731	0.1840	0.1859	0.1870	0.1889	0.1920	0.1938	0.2012	0.2055	0.2077	0.2216		
	0.1684	0.1643	0.1643	0.1689	0.1810	0.1640	0.1700	0.1765	0.1821	0.1877	0.1890	0.1900	0.1940	0.1962	0.2028	0.2090	0.2119	0.2140		
M [%]	**0.1643**	**0.1684**	**0.1690**	**0.1693**	**0.1702**	**0.1737**	**0.1745**	**0.1752**	**0.1776**	**0.1882**	**0.1888**	**0.1900**	**0.1963**	**0.1967**	**0.2025**	**0.2054**	**0.2087**	**0.2150**		**0.1852**
s [%]	0.00290	0.00689	0.00373	0.00077	0.00926	0.00800	0.00332	0.00231	0.00643	0.00184	0.00126	0.00090	0.00479	0.02142	0.00124	0.00283	0.00235	0.00518	s_M [%]	0.01592
																			$\bar{s}_i$ [%]	0.00670
s_{rel}	0.01765	0.04090	0.02210	0.00456	0.05444	0.04606	0.01901	0.01316	0.03621	0.00975	0.00667	0.00473	0.02439	0.10891	0.00611	0.01380	0.01125	0.02412		0.08598



G11: X-ray fluorescence spectrometry
H11: ICP-OES
B14: Gravimetry, dehydration with nitrosulfuric acid
B11: Gravimetry, dehydration with hydrochloric acid
Q11: ICP-MS
(R): reanalysed values

Table A3: Measurement results for manganese

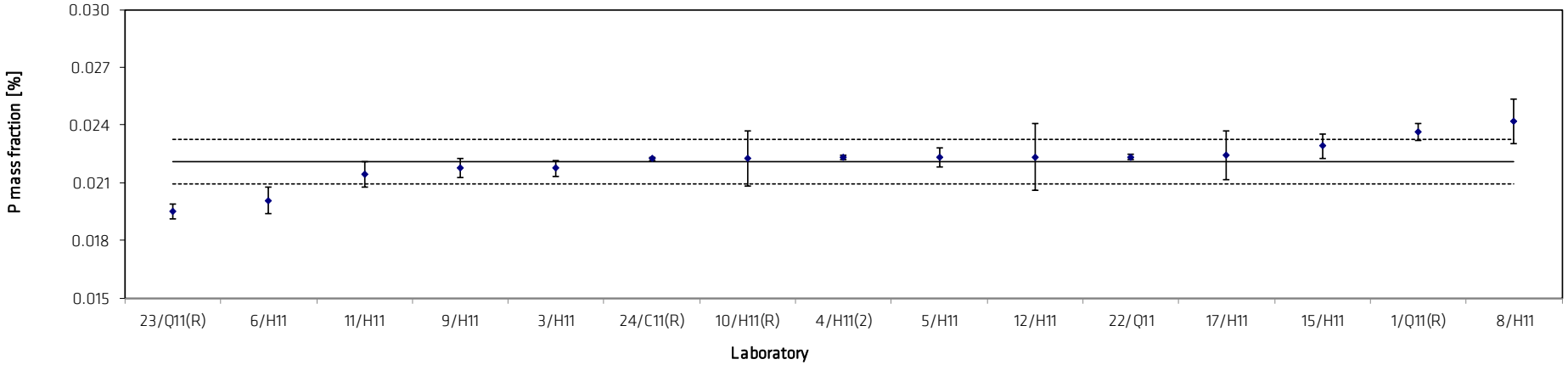
Lab./Meth.	6/H11	4/H11(1)	3/H11	5/H11	16/H11	10/H11	4/H11(2)	24/H11	8/Q11	2/H11	7/H11	4/A11	11/H11	12/H11	1/Q11(R)	23/G11	20/Q11		
M_i [%]	0.0057	0.0058	0.0064	0.0062	0.0066	0.0090	0.0063	0.0068	0.0075	0.0078	0.0082	0.0077	0.0081	0.0087	0.0077	0.0121	0.0080		*n*
	0.0058	0.0057	0.0064	0.0068	0.0038	0.0057	0.0071	0.0069	0.0050	0.0061	0.0060	0.0086	0.0079	0.0082	0.0066	0.0088	0.0110		17
	0.0058	0.0079	0.0064	0.0064	0.0088	0.0067	0.0073	0.0068	0.0083	0.0069	0.0076	0.0073	0.0090	0.0082	0.0152	0.0103	0.0090		
	0.0061	0.0061	0.0064	0.0063	0.0071	0.0058	0.0066	0.0069	0.0073	0.0077	0.0082	0.0090	0.0086	0.0086	0.0066	0.0065	0.0100		
***M* [%]**	**0.00585**	**0.00638**	**0.00640**	**0.00643**	**0.00657**	**0.00680**	**0.00683**	**0.00685**	**0.00703**	**0.00712**	**0.00749**	**0.00815**	**0.00842**	**0.00843**	**0.00903**	**0.00943**	**0.00950**		**0.00745**
s [%]	0.00017	0.00104	0.00000	0.00026	0.00210	0.00153	0.00049	0.00006	0.00142	0.00082	0.00103	0.00079	0.00049	0.00026	0.00411	0.00237	0.00129	s_M [%]	0.00115
																		$\bar{s}_i$ [%]	0.00148
s_{rel}	0.02961	0.16231	0.00000	0.04093	0.31947	0.22483	0.07132	0.00803	0.20212	0.11492	0.13698	0.09635	0.05796	0.03096	0.45568	0.25158	0.13589		0.15389



A11: Spectrophotometry, periodate oxidation

Table A4: Measurement results for phosphor

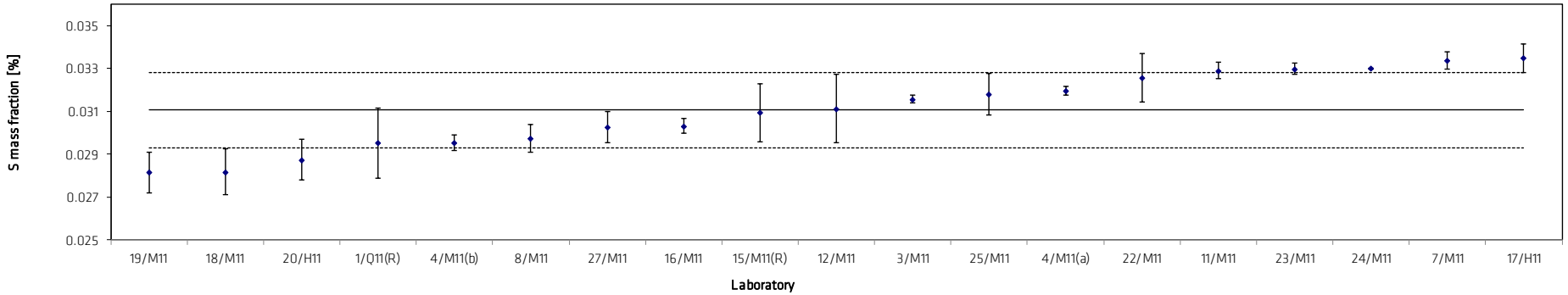
Lab./Meth.	23/Q11(R)	6/H11	11/H11	9/H11	3/H11	24/C11(R)	10/H11(R)	4/H11(2)	5/H11	12/H11	22/Q11	17/H11	15/H11	1/Q11(R)	8/H11		
M_i [%]	0.01910	0.0209	0.02210	0.0220	0.02181	0.02225	0.02202	0.02245	0.02201	0.02471	0.02212	0.02191	0.02320	0.02309	0.02316		*n*
	0.01990	0.0196	0.02194	0.0220	0.02130	0.02210	0.02055	0.02227	0.02304	0.02251	0.02243	0.02209	0.02300	0.02382	0.02564		15
	0.01970	0.0204	0.02085	0.0210	0.02160	0.02232	0.02237	0.02228	0.02211	0.02126	0.02236	0.02139	0.02200	0.02348	0.02456		
	0.01930	0.0194	0.02091	0.0220	0.02230	0.02227	0.02404	0.02222	0.02212	0.02087	0.02246	0.02428	0.02340	0.02409	0.02338		
M [%]	**0.01950**	**0.02008**	**0.02145**	**0.02175**	**0.02175**	**0.02224**	**0.02225**	**0.02231**	**0.02232**	**0.02234**	**0.02234**	**0.02242**	**0.02290**	**0.02362**	**0.02419**		**0.02210**
s [%]	0.00037	0.00070	0.00066	0.00050	0.00042	0.00009	0.00143	0.00010	0.00048	0.00173	0.00015	0.00128	0.00062	0.00043	0.00115	s_M [%]	0.00117
																$\bar{s}_i$ [%]	0.00082
s_{rel}	0.01873	0.03484	0.03086	0.02299	0.01934	0.00426	0.06442	0.00452	0.02162	0.07747	0.00690	0.05695	0.02715	0.01831	0.04748		0.05296



C11: Titration

Table A5: Measurement results for sulphur

Lab./Meth.	19/M11	18/M11	20/H11	1/Q11(R)	4/M11(b)	8/M11	27/M11	16/M11	15/M11(R)	12/M11	3/M11	25/M11	4/M11(a)	22/M11	11/M11	23/M11	24/M11	7/M11	17/H11		
M_i [%]	0.02690	0.02765	0.0300	0.03140	0.02957	0.02994	0.0295	0.03059	0.03130	0.0303	0.03140	0.0310	0.03216	0.03387	0.0324	0.03330	0.0330	0.03390	0.03374		*n*
	0.02790	0.02896	0.0290	0.03020	0.02983	0.02899	0.0312	0.03012	0.03210	0.0320	0.03150	0.0310	0.03212	0.03195	0.0332	0.03286	0.0330	0.03350	0.03374		19
	0.02890	0.02694	0.0280	0.02760	0.02902	0.02958	0.0305	0.02995	0.03140	0.0294	0.03160	0.0324	0.03183	0.03307	0.0328	0.03307	0.0330	0.03310	0.03249		
	0.02890	0.02916	0.0280	0.02891	0.02975	0.03053	0.0299	0.03063	0.02900	0.0329	0.03180	0.0329	0.03178	0.03135	0.0332	0.03269	0.0330	0.03300	0.03399		
***M* [%]**	**0.02815**	**0.02818**	**0.02875**	**0.02953**	**0.02954**	**0.02976**	**0.03028**	**0.03032**	**0.03095**	**0.03114**	**0.03158**	**0.03181**	**0.03197**	**0.03256**	**0.03290**	**0.03298**	**0.03301**	**0.03338**	**0.03349**		**0.03107**
s [%]	0.00096	0.00106	0.00096	0.00164	0.00037	0.00065	0.00074	0.00034	0.00135	0.00161	0.00017	0.00096	0.00020	0.00113	0.00038	0.00026	0.00001	0.00041	0.00068	s_M [%]	0.00176
																				$\bar{s}_i$ [%]	0.00087
s_{rel}	0.03401	0.03771	0.03330	0.05549	0.01238	0.02170	0.02448	0.01120	0.04355	0.05169	0.00541	0.03009	0.00610	0.03462	0.01164	0.00800	0.00043	0.01232	0.02022		0.05661
Calibrant	K2SO4	BaSO4			BaSO4	K2SO4	BaSO4	K2SO4	K2SO4	SO2	sulfur	K2SO4	K2SO4	K2SO4	CaSO4	Cs2SO4	BaSO4	K2SO4			



M11: Combustion/Infrared-detection

Table A6: Measurement results for chromium

Lab./Meth.	3/H11	23/G11	12/H11	4/H11(2)	4/H11(1)	22/H11	6/H11	20/Q11	8/Q11	2/H11	1/Q11	16/H11	5/H11	11/H11(R)	10/H11	7/H11	15/H11	24/H11		
M_i [%]	0.0088	0.0095	0.0095	0.00947	0.00960	0.01089	0.0104	0.0110	0.01128	0.01080	0.0122	0.0113	0.0116	0.01411	0.0148	0.0175	0.0166	0.01502		*n*
	0.0084	0.0088	0.0089	0.00938	0.01011	0.01082	0.0108	0.0110	0.01246	0.01291	0.0114	0.0130	0.0129	0.01501	0.0151	0.0130	0.0144	0.01498		18
	0.0083	0.0096	0.0092	0.00929	0.00976	0.01017	0.0104	0.0110	0.01135	0.01196	0.0118	0.0110	0.0118	0.01294	0.0150	0.0122	0.0139	0.01501		
	0.0097	0.0082	0.0094	0.00924	0.00987	0.01051	0.0109	0.0110	0.01154	0.01121	0.0119	0.0124	0.0120	0.01404	0.0135	0.0165	0.0145	0.01501		
***M* [%]**	**0.00880**	**0.00901**	**0.00924**	**0.00934**	**0.00984**	**0.01060**	**0.01063**	**0.01100**	**0.01166**	**0.01172**	**0.01183**	**0.01189**	**0.01210**	**0.01403**	**0.01459**	**0.01480**	**0.01484**	**0.01501**		**0.01172**
s [%]	0.00064	0.00063	0.00028	0.00010	0.00021	0.00033	0.00026	0.00000	0.00055	0.00093	0.00033	0.00092	0.00058	0.00085	0.00075	0.00260	0.00120	0.00002	s_M [%]	0.00214
																			$\bar{s}_i$ [%]	0.00085
s_{rel}	0.07247	0.07024	0.02981	0.01046	0.02178	0.03108	0.02475	0.00000	0.04685	0.07915	0.02827	0.07715	0.04760	0.06043	0.05128	0.17575	0.08094	0.00115		0.18272

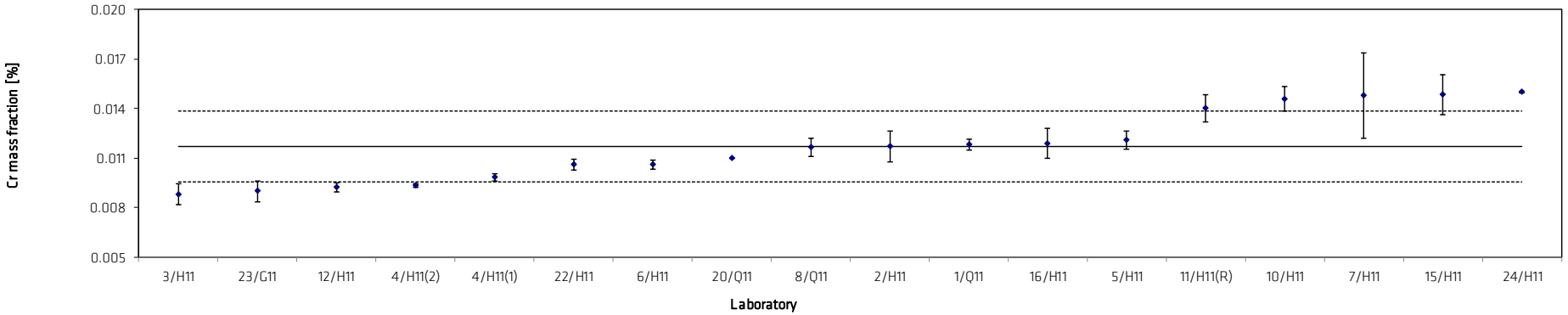
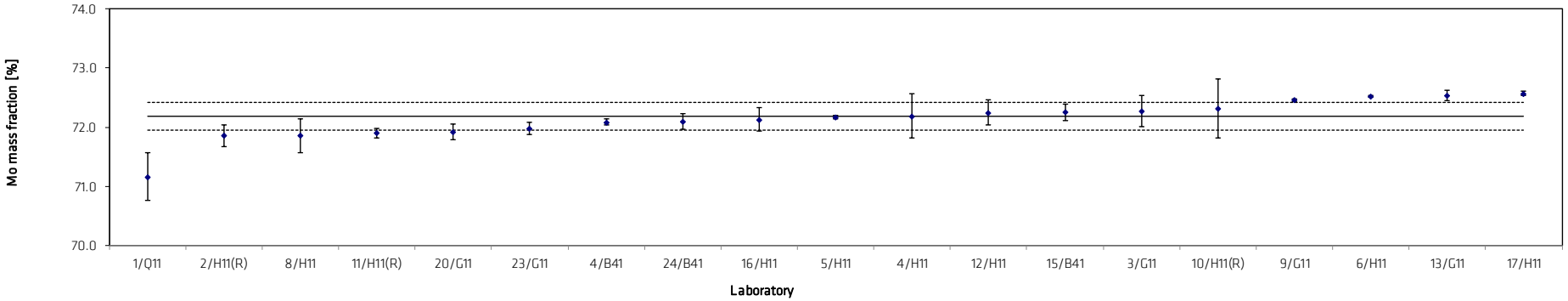


Table A7: Measurement results for molybdenum

Lab./Meth.	1/Q11	2/H11(R)	8/H11	11/H11(R)	20/G11	23/G11	4/B41	24/B41	16/H11	5/H11	4/H11	12/H11	15/B41	3/G11	10/H11(R)	9/G11	6/H11	13/G11	17/H11		
M_i [%]	*70.64*	71.77	71.57	71.83	71.94	71.96	72.12	72.10	72.33	72.14	71.78	72.39	72.21	72.08	72.68	72.48	72.51	72.44	72.55		*n*
	71.18	72.11	71.67	71.86	72.09	72.09	72.12	72.21	71.89	72.20	72.01	72.47	72.43	72.20	72.79	72.46	72.52	72.58	72.63		18
	71.23	71.71	72.17	72.01	71.87	71.85	72.10	71.92	72.24	72.15	72.34	72.07	72.27	72.13	72.02	72.42	72.51	72.63	72.56		
	71.61	71.84	72.02	71.91	71.77	72.04	72.01	72.18	72.08	72.18	72.63	72.06	72.10	72.66	71.78	72.47	72.54	72.49	72.54		
M [%]	**71.16**	**71.86**	**71.86**	**71.90**	**71.92**	**71.99**	**72.09**	**72.10**	**72.13**	**72.17**	**72.19**	**72.25**	**72.25**	**72.27**	**72.32**	**72.46**	**72.52**	**72.53**	**72.57**		**72.19**
s [%]	0.39873	0.17677	0.28420	0.07847	0.13451	0.10318	0.05258	0.13196	0.19289	0.02754	0.37265	0.21383	0.13769	0.26371	0.49227	0.02630	0.01442	0.08751	0.04038	s_M [%]	0.23199
																				$\bar{s}_i$ [%]	0.20147
s_{rel}	0.00560	0.00246	0.00395	0.00109	0.00187	0.00143	0.00073	0.00183	0.00267	0.00038	0.00516	0.00296	0.00191	0.00365	0.00681	0.00036	0.00020	0.00121	0.00056		0.00321

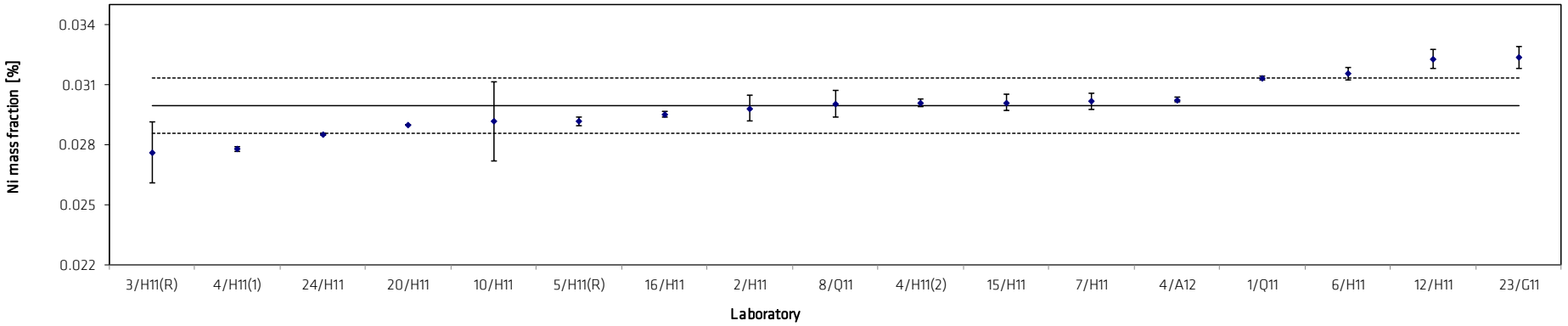


*Dataset 1/Q11 removed for technical reasons

B41: Gravimetric with 8-hydroxyquinoline

Table A8: Measurement results for nickel

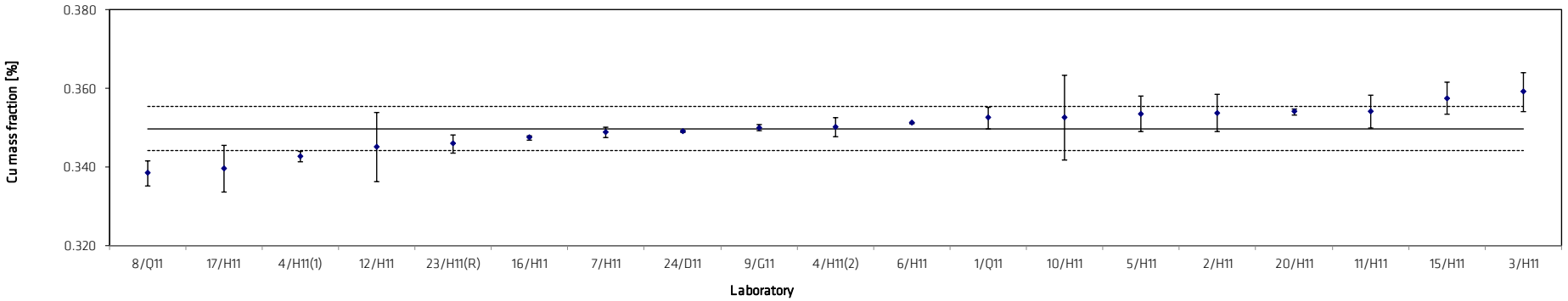
Lab./Meth.	3/H11(R)	4/H11(1)	24/H11	20/H11	10/H11	5/H11(R)	16/H11	2/H11	8/Q11	4/H11(2)	15/H11	7/H11	4/A12	1/Q11	6/H11	12/H11	23/G11		
M_i [%]	0.0274	0.0279	0.0285	0.0290	0.0274	0.0289	0.0297	0.0306	0.0296	0.0303	0.0295	0.0299	0.0303	0.0315	0.0318	0.03197	0.0323		*n*
	0.0298	0.0276	0.0285	0.0290	0.0307	0.0293	0.0293	0.0291	0.0296	0.0301	0.0303	0.0299	0.0304	0.0313	0.0318	0.03187	0.0319		17
	0.0263	0.0279	0.0285	0.0290	0.0311	0.0291	0.0295	0.0301	0.0300	0.0300	0.0303	0.0307	0.0302	0.0314	0.0314	0.03249	0.0322		
	0.0270	0.0278	0.0286	0.0290	0.0276	0.0294	0.0295	0.0295	0.0310	0.0299	0.0303	0.0302	0.0301	0.0313	0.0312	0.03287	0.0331		
M [%]	**0.02763**	**0.02779**	**0.02852**	**0.02900**	**0.02917**	**0.02918**	**0.02952**	**0.02982**	**0.03005**	**0.03009**	**0.03010**	**0.03017**	**0.03025**	**0.03134**	**0.03155**	**0.03230**	**0.03237**		**0.02993**
s [%]	0.00152	0.00012	0.00003	0.00000	0.00197	0.00022	0.00015	0.00065	0.00066	0.00018	0.00040	0.00040	0.00013	0.00010	0.00030	0.00047	0.00054	s_M [%]	0.00138
																		$\bar{s}_i$ [%]	0.00069
s_{rel}	0.05501	0.00420	0.00097	0.00000	0.06755	0.00760	0.00507	0.02171	0.02196	0.00591	0.01329	0.01339	0.00427	0.00331	0.00951	0.01446	0.01654		0.04609



A12: Spectrophotometry, dimethylglyoxime, extraction

Table A9: Measurement results for copper

Lab./Meth.	8/Q11	17/H11	4/H11(1)	12/H11	23/H11(R)	16/H11	7/H11	24/D11	9/G11	4/H11(2)	6/H11	1/Q11	10/H11	5/H11	2/H11	20/H11	11/H11	15/H11	3/H11		
M_i [%]	0.3381	0.3380	0.34309	0.35310	0.3476	0.34788	0.3474	0.3492	0.350	0.35366	0.35120	0.3503	0.3602	0.3590	0.3597	0.3540	0.35660	0.3530	0.3576		*n*
	0.3367	0.3377	0.34218	0.35213	0.3479	0.34700	0.3481	0.3490	0.350	0.35010	0.35110	0.3562	0.3408	0.3540	0.3488	0.3550	0.34930	0.3570	0.3665		19
	0.3359	0.3482	0.34127	0.33724	0.3447	0.34692	0.3503	0.3488	0.351	0.34832	0.35140	0.3505	0.3630	0.3530	0.3550	0.3540	0.35210	0.3630	0.3562		
	0.3430	0.3343	0.34420	0.33748	0.3433	0.34797	0.3494	0.3492	0.349	0.34852	0.35100	0.3531	0.3462	0.3480	0.3516	0.3530	0.35830	0.3570	0.3563		
M [%]	**0.3384**	**0.3396**	**0.3427**	**0.3450**	**0.3459**	**0.3474**	**0.3488**	**0.3491**	**0.3500**	**0.3501**	**0.3512**	**0.3525**	**0.3526**	**0.3535**	**0.3538**	**0.3540**	**0.3541**	**0.3575**	**0.3592**		**0.3497**
s [%]	0.00319	0.00601	0.00125	0.00882	0.00224	0.00056	0.00131	0.00019	0.00082	0.00247	0.00017	0.00276	0.01074	0.00451	0.00469	0.00082	0.00412	0.00412	0.00494	s_M [%]	0.00558
																				$\bar{s}_i$ [%]	0.00438
s_{rel}	0.00943	0.01769	0.00366	0.02556	0.00648	0.00161	0.00377	0.00055	0.00233	0.00706	0.00049	0.00784	0.03047	0.01276	0.01327	0.00231	0.01164	0.01153	0.01376		0.01595



D11: flame atomic absorption spectrometry

Table A10: Measurement results for cobalt

Lab./Meth.	23/Q11(R)	6/H11	10/H11	12/H11	22/H11	24/H11	8/Q11	4/H11(2)	5/H11	20/Q11	4/H11(1)	1/Q11	11/H11	2/H11	15/H11		
M_i [%]	0.00612	0.00630	0.00668	0.00654	0.00676	0.00661	0.00665	0.00693	0.00651	0.0070	0.00698	0.00708	0.00744	0.00737	0.0081		*n*
	0.00645	0.00630	0.00638	0.00657	0.00658	0.00662	0.00665	0.00676	0.00733	0.0070	0.00710	0.00721	0.00711	0.00818	0.0080		15
	0.00556	0.00620	0.00642	0.00664	0.00657	0.00663	0.00671	0.00672	0.00662	0.0070	0.00711	0.00716	0.00802	0.00804	0.0082		
	0.00564	0.00640	0.00642	0.00653	0.00642	0.00661	0.00668	0.00669	0.00667	0.0070	0.00693	0.00729	0.00813	0.00820	0.0081		
M [%]	**0.00594**	**0.00630**	**0.006475**	**0.00657**	**0.00658**	**0.00662**	**0.00667**	**0.00677**	**0.00678**	**0.00700**	**0.00703**	**0.00719**	**0.00768**	**0.00795**	**0.00810**		**0.00691**
s [%]	0.00042	0.00008	0.00014	0.00005	0.00014	0.00001	0.00003	0.00011	0.00037	0.00000	0.00009	0.00009	0.00048	0.00039	0.00008	s_M [%]	0.00060
																$\overline{s}_i$ [%]	0.00023
s_{rel}	0.07052	0.01296	0.02131	0.00756	0.02114	0.00145	0.00430	0.01587	0.05471	0.00000	0.01267	0.01249	0.06296	0.04931	0.01008		0.08709

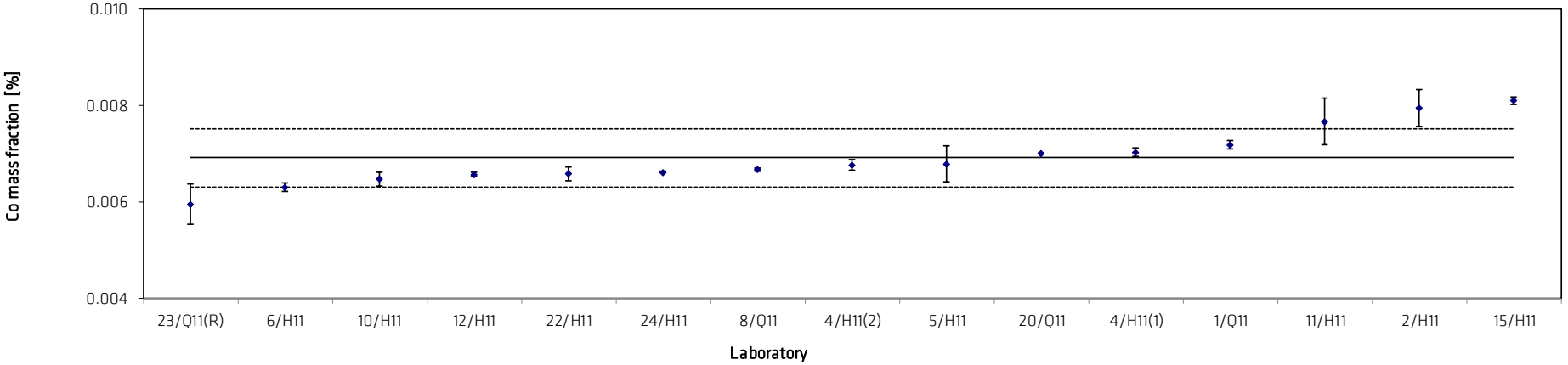


Table A11: Measurement results for tin

Lab./Meth.	5/H11	4/H11	1/Q11	10/H11	23/Q11(R)	14/Q11	11/H11(R)	24/H11	8/Q11	27/H11	22/Q11	6/H11	7/H11	3/H11	20/Q11		
M_i [%]	0.00260	0.00295	0.00295	0.00307	0.00308	0.00299	0.00321	0.00301	0.00296	0.00314	0.00312	0.00320	0.00326	0.0033			*n*
	0.00250	0.00291	0.00296	0.00282	0.00316	0.00295	0.00299	0.00302	0.00301	0.00307	0.00330	0.00340	0.00332	0.0033			14
	0.00280	0.00296	0.00296	0.00308	0.00274	0.00319	0.00266	0.00301	0.00303	0.00325	0.00320	0.00330	0.00328	0.0033			
	0.00310	0.00292	0.00294	0.00285	0.00285	0.00275	0.00304	0.00302	0.00311	0.00312	0.00316	0.00310	0.00339	0.0034			
M [%]	**0.00275**	**0.00294**	**0.00295**	**0.00296**	**0.00296**	**0.00297**	**0.00298**	**0.00302**	**0.00303**	**0.00315**	**0.00320**	**0.00325**	**0.00331**	**0.00333**	**< 0,01**		**0.00305**
s [%]	0.00026	0.00002	0.00001	0.00014	0.00020	0.00018	0.00023	0.00001	0.00006	0.00008	0.00008	0.00013	0.00006	0.00005	#DIV/0!	s_M [%]	0.00017
																$\bar{s}_i$ [%]	0.00013
s_{rel}	0.09621	0.00771	0.00324	0.04709	0.06616	0.06073	0.07736	0.00191	0.02061	0.02415	0.02418	0.03972	0.01792	0.01504	#DIV/0!		0.05428

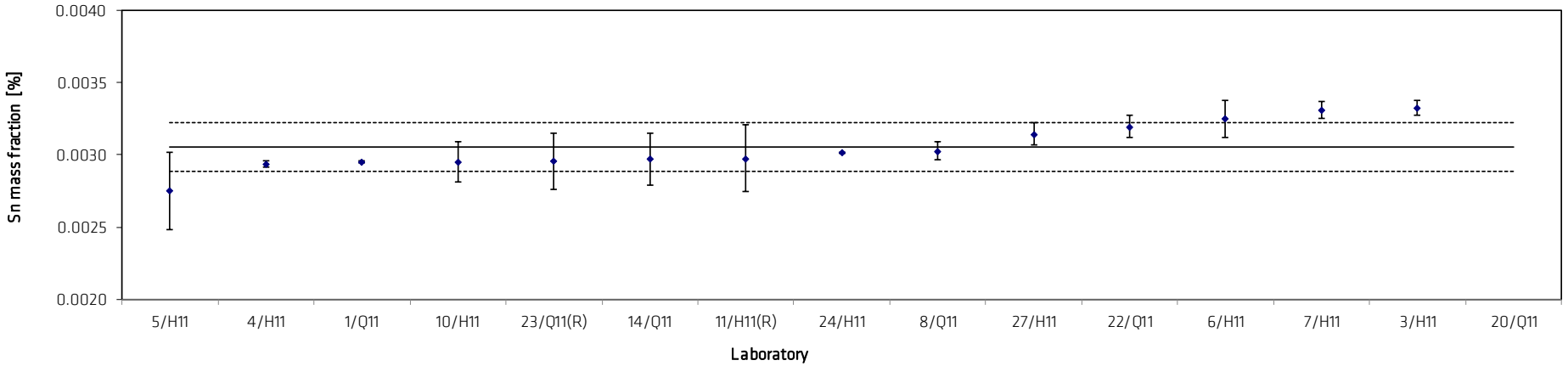
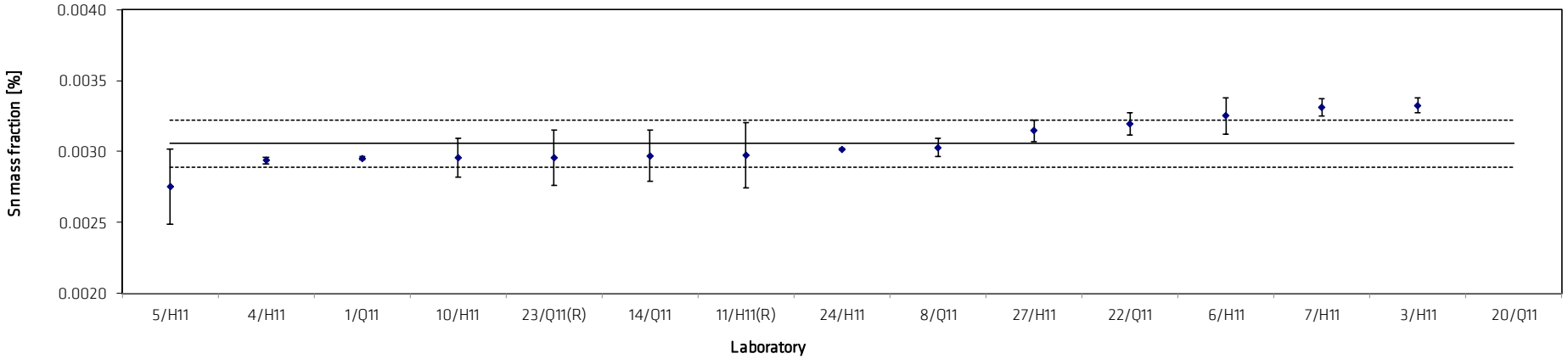


Table A12: Measurement results for antimony

Lab./Meth.	5/H11	4/H11	1/Q11	10/H11	23/Q11(R)	14/Q11	11/H11(R)	24/H11	8/Q11	27/H11	22/Q11	6/H11	7/H11	3/H11	20/Q11		
M_i [%]	0.00260	0.00295	0.00295	0.00307	0.00308	0.00299	0.00321	0.00301	0.00296	0.00314	0.00312	0.00320	0.00326	0.0033			*n*
	0.00250	0.00291	0.00296	0.00282	0.00316	0.00295	0.00299	0.00302	0.00301	0.00307	0.00330	0.00340	0.00332	0.0033			14
	0.00280	0.00296	0.00296	0.00308	0.00274	0.00319	0.00266	0.00301	0.00303	0.00325	0.00320	0.00330	0.00328	0.0033			
	0.00310	0.00292	0.00294	0.00285	0.00285	0.00275	0.00304	0.00302	0.00311	0.00312	0.00316	0.00310	0.00339	0.0034			
***M* [%]**	**0.00275**	**0.00294**	**0.00295**	**0.00296**	**0.00296**	**0.00297**	**0.00298**	**0.00302**	**0.00303**	**0.00315**	**0.00320**	**0.00325**	**0.00331**	**0.00333**	**< 0,01**		**0.00305**
s [%]	0.00026	0.00002	0.00001	0.00014	0.00020	0.00018	0.00023	0.00001	0.00006	0.00008	0.00008	0.00013	0.00006	0.00005	#DIV/0!	s_M [%]	0.00017
																$\bar{s}_i$ [%]	0.00013
s_{rel}	0.09621	0.00771	0.00324	0.04709	0.06616	0.06073	0.07736	0.00191	0.02061	0.02415	0.02418	0.03972	0.01792	0.01504	#DIV/0!		0.05428



E11: Electrothermal atomic absorption spectrometry

Table A13: Measurement results for lead

Lab./Meth.	4/E11	22/Q11	20/Q11	1/Q11	23/Q11(R)	24/H11	8/Q11	14/Q11	7/H11	4/H11	6/H11	10/H11	3/H11		
M_i [%]	0.00032	0.00035	0.00038	0.00039	0.00040	0.00041	0.00040	0.00040	0.00070	0.00076	0.00090				*n*
	0.00033	0.00038	0.00039	0.00039	0.00043	0.00040	0.00042	0.00039	0.00066	0.00070	0.00090				11
	0.00034	0.00036	0.00039	0.00040	0.00036	0.00040	0.00039	0.00049	0.00065	0.00081	0.00070				
	0.00033	0.00036	0.00039	0.00039	0.00038	0.00040	0.00046	0.00040	0.00067	0.00087	0.00080				
***M* [%]**	**0.00033**	**0.00036**	**0.00039**	**0.00039**	**0.00039**	**0.00040**	**0.00042**	**0.00042**	**0.00067**	**0.00079**	**0.00083**	**< 0.005**	**< 0.001**		**0.00039**
s [%]	0.00001	0.00001	0.00000	0.00000	0.00003	0.00000	0.00003	0.00005	0.00002	0.00007	0.00010			s_M [%]	0.00003
														$\bar{s}_i$ [%]	0.00004
s_{rel}	0.02910	0.03667	0.01290	0.01212	0.07608	0.01242	0.07415	0.11168	0.03214	0.09252	0.11605				0.07538

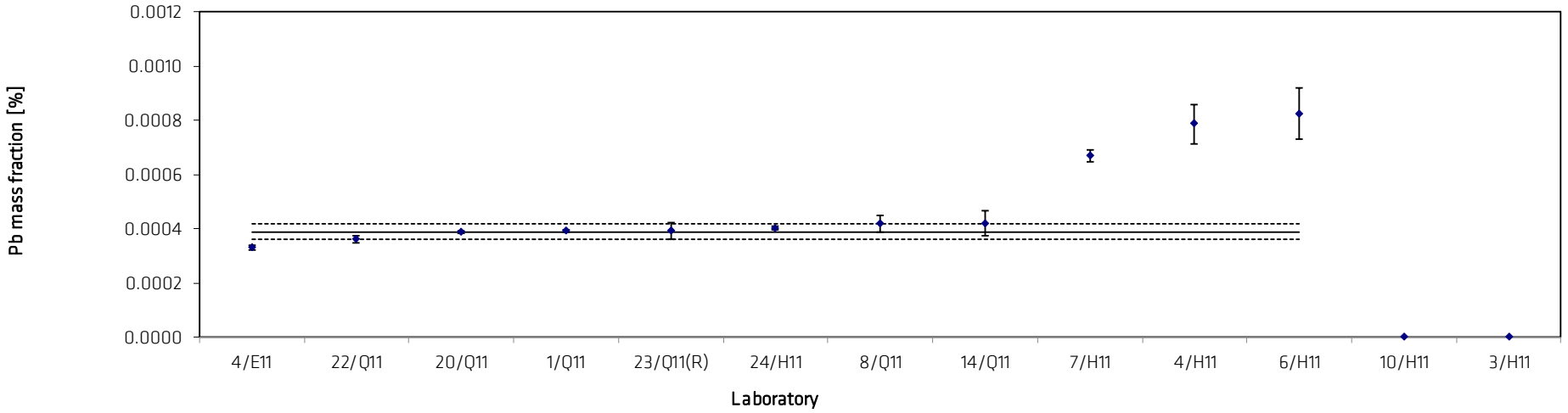


Table A14: Measurement results for bismuth

Lab./Meth.	23/Q11	22/Q11	1/Q11	14/Q11	8/Q11	4/H11	6/H11	12/H11	15/H11	11/H11	20/Q11	3/H11		
M_i [%]	0.00004	0.00012	0.00012	0.00014	0.00014	0.00939	0.00960	0.01299	0.01550					*n*
	0.00000	0.00012	0.00013	0.00014	0.00014	0.00933	0.01060	0.01084	0.01470					9
	0.00003	0.00012	0.00012	0.00016	0.00014	0.00928	0.01030	0.01018	0.01520					
	0.00000	0.00012	0.00013	0.00009	0.00014	0.00924	0.00980	0.01513	0.01520					
***M* [%]**	**0.00002**	**0.00012**	**0.00013**	**0.00013**	**0.00014**	**0.00931**	**0.01008**	**0.01229**	**0.01515**	**< 0.003**	**< 0.01**	**< 0.1**		**0.00013**
s [%]	0.00002	0.00000	0.00001	0.00003	0.00000	0.00007	0.00046	0.00224	0.00033	#DIV/0!	#DIV/0!	#DIV/0!	s_M [%]	0.00001
													$\bar{s}_i$ [%]	0.00002
s_{rel}	1.17803	0.02076	0.04619	0.22536	0.00000	0.00708	0.04539	0.18268	0.02189	#DIV/0!	#DIV/0!	#DIV/0!		0.07364

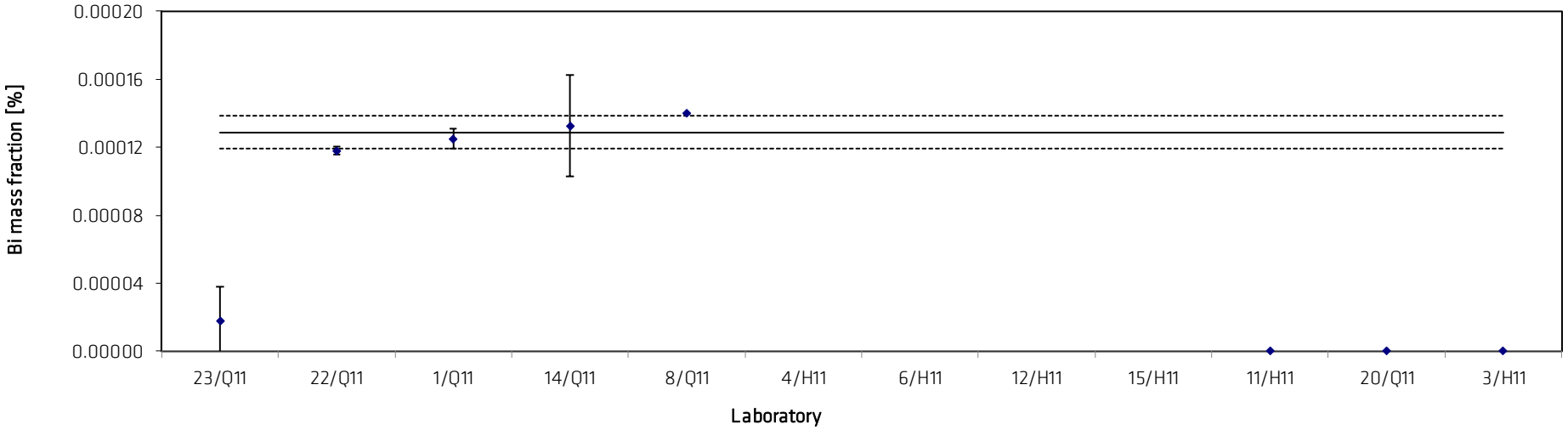
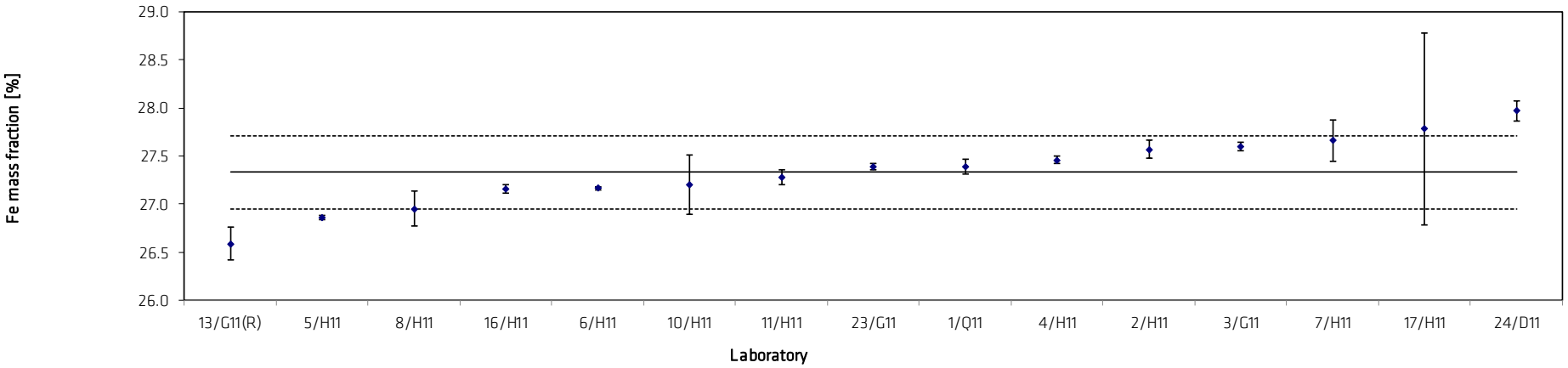


Table A15: Measurement results for iron

Lab./Meth.	13/G11(R)	5/H11	8/H11	16/H11	6/H11	10/H11	11/H11	23/G11	1/Q11	4/H11	2/H11	3/G11	7/H11	17/H11	24/D11		
M_i [%]	26.42	26.88	26.71	27.19	27.19	27.56	27.25	27.38	*27.48*	27.44	27.69	27.65	27.73	28.85	28.00		*n*
	26.82	26.83	26.99	27.09	27.15	27.10	27.39	27.43	*27.33*	27.42	27.47	27.56	27.93	27.33	28.10		14
	26.52	26.89	26.95	27.17	27.17	26.83	27.24	27.39	*27.42*	27.46	27.60	27.57	27.56	28.32	27.86		
	26.60	26.86	27.15	27.18	27.16	27.32	27.24	27.35	*27.33*	27.51	27.52	27.62	27.43	26.62	27.93		
M [%]	**26.59**	**26.86**	**26.95**	**27.16**	**27.17**	**27.20**	**27.28**	**27.39**	27.39	**27.46**	**27.57**	**27.60**	**27.66**	**27.78**	**27.97**		**27.33**
s [%]	0.169	0.025	0.181	0.044	0.017	0.312	0.075	0.033	0.075	0.039	0.096	0.045	0.215	0.996	0.103	s_M [%]	0.379
																$\bar{s}_i$ [%]	0.13754
s_{rel}	0.00637	0.00092	0.00670	0.00163	0.00062	0.01145	0.00274	0.00120	0.00272	0.00141	0.00347	0.00163	0.00779	0.03586	0.00369		0.01386



*Dataset 1/Q11 removed for technical reasons

Table A15: results for additional elements

	As	As	B	Ba	Be	Ca	Ce	Ga	Ge	Hf	Ir	Mg	Pt
	0.00921	0.01019	0.0005	0.00009	<0.000001	0.00303	0.0003	0.00072	0.00013	<0.000001	<0.000001	0.001420	0.00002
	0.00911	0.01042	0.0006	0.00009	<0.000001	0.00234	0.0003	0.00071	0.00015	<0.000001	<0.000001	0.002940	0.00002
	0.00937	0.01029	0.0005	0.00010	<0.000001	0.00341	0.0003	0.00074	0.00014	<0.000001	<0.000001	0.002660	0.00002
	0.00907	0.01050	0.0005	0.00011	<0.000001	0.00284	0.0003	0.00073	0.00013	<0.000001	<0.000001	0.002240	0.00002
Mean	**0.00919**	**0.01035**	**0.00051**	**0.00010**		**0.00291**	**0.00027**	**0.00073**	**0.00014**			**0.00232**	**0.00002**
Lab	1/Q11	22/Q11	1/Q11	1/Q11	1/Q11	1/Q11	1/Q11	1/Q11	1/Q11	1/Q11	1/Q11	1/Q11	1/Q11

	Rb	Re	Ta	Th	Ti	U	V	W	Zn	Zr
	0.00001	0.00365	0.00001	<0.000001	0.00324	<0.000001	0.00168	0.01390	0.000940	0.000050
	0.00001	0.00372	0.00001	<0.000001	0.00272	<0.000001	0.00165	0.01410	0.000970	0.000050
	0.00001	0.00369	0.00001	<0.000001	0.00261	<0.000001	0.00172	0.01480	0.000820	0.000050
	0.00001	0.00368	0.00001	<0.000001	0.00264	<0.000001	0.00169	0.01430	0.000890	0.000050
Mean	**0.00001**	**0.00369**	**0.00001**		**0.00280**		**0.00169**	**0.01428**	**0.00091**	**0.00005**
Lab	1/Q11	1/Q11	1/Q11	1/Q11	1/Q11	1/Q11	1/Q11	1/Q11	1/Q11	1/Q11

Annex 2: Homogeneity study (mass fraction in %)

Carbon:

	1	2	3
Bottle 1	0.01993	0.01917	0.01880
Bottle 2	0.01766	0.01961	0.01945
Bottle 3	0.01952	0.01935	0.01873
Bottle 4	0.01893	0.01865	0.01895
Bottle 5	0.01822	0.01875	0.02001
Bottle 6	0.02009	0.01992	0.01932
Bottle 7	0.01993	0.01921	0.01928
Bottle 8	0.02008	0.01980	0.02000
Bottle 9	0.01884	0.01878	0.01816
Bottle 10	0.02017	0.01863	0.01833
Bottle 11	0.01879	0.02147	0.01779
Bottle 12	0.01974	0.01904	0.01944
Bottle 13	0.02162	0.01965	0.01966
Bottle 14	0.02388	0.01910	0.01970
Bottle 15	0.01902	0.01752	0.01823

Source of variation	*sums of squares (SS)*	*degrees of freedom (df)*	*Mean squares (MS)*	*F-value*	*P-value*	*critical F-value*
Between groups	1.90562E-05	14	1.36116E-06	1.2474569	0.2949043	2.0374204
Within groups	3.27344E-05	30	1.09115E-06			
Total	5.17906E-05	44				
within-sd	0.001044579			status:	homogeneous	
effective n	3.00					
s_{bb}	0.000300006					
u^*_{bb}	0.000306449					
u_{bb}	0.000306449	0.306448868				
u_{bb}(rel.)	1.678810194					

Silicon:

Bottle 1	0.1919	0.1925
Bottle 2	0.1921	0.1920
Bottle 3	0.2023	0.1978
Bottle 4	0.1830	0.1847
Bottle 5	0.1983	0.1806
Bottle 6	0.2014	0.2062
Bottle 7	0.1857	0.1870
Bottle 8	0.1880	0.1823
Bottle 9	0.1951	0.1992
Bottle 10	0.1774	0.1799
Bottle 11	0.1895	0.2019
Bottle 12	0.1956	0.1875
Bottle 13	0.1995	0.1948
Bottle 14	0.2082	0.1903
Bottle 15	0.2071	0.1848

Source of variation	*sums of squares (SS)*	*degrees of freedom (df)*	*Mean squares (MS)*	*F-value*	*P-value*	*critical F-value*
Between groups	0.001329288	14	9.49492E-05	1.928696102	0.109633637	2.424364357
Within groups	0.000738446	15	4.92297E-05			
Total	0.002067734	29				
within-sd	0.007016389			status:	homogeneous	
effective n	2.00					
s_{bb}	0.004781184					
u^*_{bb}	0.002998012					
u_{bb}	0.004781184	4.781184226				
u_{bb}(rel.)	2.424990605					

Manganese:

Bottle 1	0.0053	0.0068
Bottle 2	0.0080	0.0051
Bottle 3	0.0063	0.0062
Bottle 4	0.0087	0.0058
Bottle 5	0.0077	0.0052
Bottle 6	0.0056	0.0059
Bottle 7	0.0059	0.0059
Bottle 8	0.0089	0.0055
Bottle 9	0.0085	0.0072
Bottle 10	0.0054	0.0074
Bottle 11	0.0085	0.0073
Bottle 12	0.0050	0.0058
Bottle 13	0.0080	0.0084
Bottle 14	0.0073	0.0089
Bottle 15	0.0085	0.0061

Source of variation	*sums of squares (SS)*	*degrees of freedom (df)*	*Mean squares (MS)*	*F-value*	*P-value*	*critical F-value*
Between groups	2.32878E-05	14	1.66342E-06	0.9299009	0.5516215	2.4243644
Within groups	2.68321E-05	15	1.78881E-06			
Total	5.012E-05	29				
within-sd	0.001337464			status:	homogeneous	
effective n	2.00					
s_{bb}	0					
u^*_{bb}	0.000571481					
u_{bb}	0.000571481	0.571480994				
u_{bb}(rel.)	7.078588499					

Phosphor:

Bottle 1	0.0222	0.0224
Bottle 2	0.0225	0.0222
Bottle 3	0.0221	0.0225
Bottle 4	0.0222	0.0224
Bottle 5	0.0221	0.0220
Bottle 6	0.0225	0.0225
Bottle 7	0.0223	0.0223
Bottle 8	0.0224	0.0222
Bottle 9	0.0222	0.0224
Bottle 10	0.0222	0.0222
Bottle 11	0.0227	0.0226
Bottle 12	0.0224	0.0223
Bottle 13	0.0223	0.0220
Bottle 14	0.0221	0.0223
Bottle 15	0.0223	0.0224

Source of variation	*sums of squares (SS)*	*degrees of freedom (df)*	*Mean squares (MS)*	*F-value*	*P-value*	*critical F-value*
Between groups	4.87458E-07	14	3.48184E-08	1.9636044	0.103584	2.4243644
Within groups	2.65979E-07	15	1.77319E-08			
Total	7.53437E-07	29				
within-sd	0.000133161			status:	homogeneous	
effective n	2.00					
s_{bb}	9.24298E-05					
u^*_{bb}	5.6898E-05					
u_{bb}	9.24298E-05	0.092429809				
u_{bb}(rel.)	0.416292735					

Sulphur:

Bottle 1	0.03051	0.03140	0.03186
Bottle 2	0.03017	0.03131	0.03132
Bottle 3	0.03146	0.03201	0.03280
Bottle 4	0.03135	0.03134	0.03188
Bottle 5	0.03122	0.03203	0.03195
Bottle 6	0.03083	0.03254	0.03175
Bottle 7	0.03174	0.03195	0.03195
Bottle 8	0.03231	0.03223	0.03227
Bottle 9	0.03171	0.03252	0.03179
Bottle 10	0.03205	0.03185	0.03165
Bottle 11	0.03215	0.03214	0.03236
Bottle 12	0.03118	0.03232	0.03120
Bottle 13	0.03185	0.03106	0.03165
Bottle 14	0.03197	0.03216	0.03269
Bottle 15	0.03222	0.03251	0.03132

Source of variation	*sums of squares (SS)*	*degrees of freedom (df)*	*Mean squares (MS)*	*F-value*	*P-value*	*critical F-value*
Between groups	6.17323E-06	14	4.40945E-07	1.75099889	0.09686233	2.03742044
Within groups	7.55474E-06	30	2.51825E-07			
Total	1.3728E-05	44				
within-sd	0.000501821			status:	homogeneous	
effective n	3.00					
s_{bb}	0.000251078					
u^*_{bb}	0.00014722					
u_{bb}	0.000251078	0.251077744				
u_{bb}(rel.)	0.784453138					

Chromium:

Bottle 1	0.0093	0.0096
Bottle 2	0.0096	0.0092
Bottle 3	0.0093	0.0067
Bottle 4	0.0094	0.0091
Bottle 5	0.0093	0.0090
Bottle 6	0.0095	0.0095
Bottle 7	0.0094	0.0093
Bottle 8	0.0094	0.0092
Bottle 9	0.0092	0.0093
Bottle 10	0.0092	0.0093
Bottle 11	0.0095	0.0096
Bottle 12	0.0094	0.0094
Bottle 13	0.0094	0.0094
Bottle 14	0.0093	0.0094
Bottle 15	0.0095	0.0093

Source of variation	*sums of squares (SS)*	*degrees of freedom (df)*	*Mean squares (MS)*	*F-value*	*P-value*	*critical F-value*
Between groups	3.6865E-06	14	2.63319E-07	1.10719474	0.42206279	2.42436436
Within groups	3.5674E-06	15	2.37826E-07			
Total	7.2539E-06	29				
within-sd	0.00048767			status:	homogeneous	
effective n	2.00					
s_{bb}	0.0001129					
u^*_{bb}	0.00020838					
u_{bb}	0.00020838	0.208376706				
u_{bb}(rel.)	2.22907329					

Molybdenum

Bottle 1	72.70	72.67
Bottle 2	72.45	72.82
Bottle 3	73.07	72.80
Bottle 4	72.55	72.78
Bottle 5	72.70	72.67
Bottle 6	72.33	72.63
Bottle 7	72.63	72.63
Bottle 8	72.41	72.55
Bottle 9	72.48	72.63
Bottle 10	72.57	72.50
Bottle 11	72.70	72.58
Bottle 12	72.81	72.70
Bottle 13	72.79	72.29
Bottle 14	72.47	72.46
Bottle 15	72.84	72.65

Source of variation	*sums of squares (SS)*	*degrees of freedom (df)*	*Mean squares (MS)*	*F-value*	*P-value*	*critical F-value*
Between grou	0.444147299	14	0.031724807	1.321453441	0.298954571	2.424364357
Within groups	0.360112654	15	0.02400751			
Total	0.804259953	29				
within-sd	0.154943571			status:	homogeneous	
effective n	2.00					
s_{bb}	0.06211802					
u^*_{bb}	0.066205375					
u_{bb}	0.066205375	66.20537474				
u_{bb}(rel.)	0.09124702					

Nickel:

Bottle 1	0.0303	0.0304
Bottle 2	0.0304	0.0303
Bottle 3	0.0303	0.0305
Bottle 4	0.0304	0.0306
Bottle 5	0.0305	0.0304
Bottle 6	0.0304	0.0305
Bottle 7	0.0305	0.0305
Bottle 8	0.0304	0.0304
Bottle 9	0.0305	0.0304
Bottle 10	0.0308	0.0306
Bottle 11	0.0305	0.0305
Bottle 12	0.0306	0.0303
Bottle 13	0.0305	0.0303
Bottle 14	0.0304	0.0304
Bottle 15	0.0305	0.0305

Source of variation	*sums of squares (SS)*	*degrees of freedom (df)*	*Mean squares (MS)*	*F-value*	*P-value*	*critical F-value*
Between groups	2.06684E-07	14	1.47632E-08	1.3304667	0.2945633	2.4243644
Within groups	1.66444E-07	15	1.10962E-08			
Total	3.73128E-07	29				
within-sd	0.000105339			status:	homogeneous	
effective n	2.00					
s_{bb}	4.2819E-05					
u^*_{bb}	4.50099E-05					
u_{bb}	4.50099E-05	0.045009851				
u_{bb}(rel.)	0.147918973					

Copper:

Bottle 1	0.3541	0.3573
Bottle 2	0.3582	0.3571
Bottle 3	0.3579	0.3608
Bottle 4	0.3560	0.3569
Bottle 5	0.3561	0.3548
Bottle 6	0.3565	0.3563
Bottle 7	0.3581	0.3571
Bottle 8	0.3569	0.3579
Bottle 9	0.3593	0.3556
Bottle 10	0.3578	0.3556
Bottle 11	0.3564	0.3562
Bottle 12	0.3574	0.3561
Bottle 13	0.3591	0.3569
Bottle 14	0.3588	0.3596
Bottle 15	0.3573	0.3570

Source of variation	*sums of squares (SS)*	*degrees of freedom (df)*	*Mean squares (MS)*	*F-value*	*P-value*	*critical F-value*
Between groups	3.49038E-05	14	2.49313E-06	1.4780518	0.2307985	2.4243644
Within groups	2.53015E-05	15	1.68676E-06			
Total	6.02052E-05	29				
within-sd	0.001298755			status:	homogeneous	
effective n	2.00					
s_{bb}	0.000634965					
u^*_{bb}	0.000554941					
u_{bb}	0.000634965	0.634964888				
u_{bb}(rel.)	0.176764312					

Cobalt:

Bottle 1	0.0068	0.0068
Bottle 2	0.0069	0.0068
Bottle 3	0.0067	0.0067
Bottle 4	0.0067	0.0066
Bottle 5	0.0067	0.0067
Bottle 6	0.0068	0.0069
Bottle 7	0.0068	0.0068
Bottle 8	0.0067	0.0067
Bottle 9	0.0067	0.0067
Bottle 10	0.0067	0.0067
Bottle 11	0.0073	0.0069
Bottle 12	0.0071	0.0068
Bottle 13	0.0068	0.0068
Bottle 14	0.0067	0.0067
Bottle 15	0.0067	0.0067

Source of variation	*sums of squares (SS)*	*degrees of freedom (df)*	*Mean squares (MS)*	*F-value*	*P-value*	*critical F-value*
Between groups	3.84576E-07	14	2.74697E-08	2.74555954	0.0308863	2.42436436
Within groups	1.50077E-07	15	1.00051E-08			
Total	5.34653E-07	29				
within-sd	0.000100026			status:	inhomogeneous	
effective n	2.00					
s_{bb}	9.34467E-05					
u^*_{bb}	4.27397E-05					
u_{bb}	9.34467E-05	0.093446729				
u_{bb}(rel.)	1.38766419					

Tin:

Bottle 1	0.0028	0.0029
Bottle 2	0.0029	0.0029
Bottle 3	0.0030	0.0030
Bottle 4	0.0030	0.0028
Bottle 5	0.0031	0.0031
Bottle 6	0.0030	0.0030
Bottle 7	0.0030	0.0030
Bottle 8	0.0029	0.0029
Bottle 9	0.0030	0.0030
Bottle 10	0.0028	0.0028
Bottle 11	0.0030	0.0031
Bottle 12	0.0029	0.0030
Bottle 13	0.0029	0.0030
Bottle 14	0.0029	0.0030
Bottle 15	0.0028	0.0031

Source of variation	*sums of squares (SS)*	*degrees of freedom (df)*	*Mean squares (MS)*	*F-value*	*P-value*	*critical F-value*
Between groups	5.94553E-09	4	1.48638E-09	0.1748919	0.94922074	2.42436436
Within groups	2.12472E-07	25	8.49887E-09			
Total	2.18417E-07	29				
within-sd	9.21893E-05			status:	homogeneous	
effective n	2.00					
s_{bb}	0					
u^*_{bb}	3.46687E-05					
u_{bb}	3.46687E-05	0.03466874				
u_{bb}(rel.)	1.171547949					

Antimony:

Bottle 1	0.0017	0.0014
Bottle 2	0.0021	0.0018
Bottle 3	0.0020	0.0017
Bottle 4	0.0019	0.0015
Bottle 5	0.0019	0.0017
Bottle 6	0.0017	0.0016
Bottle 7	0.0020	0.0020
Bottle 8	0.0019	0.0020
Bottle 9	0.0018	0.0019
Bottle 10	0.0019	0.0020
Bottle 11	0.0020	0.0021
Bottle 12	0.0017	0.0017
Bottle 13	0.0020	0.0024
Bottle 14	0.0017	0.0016

Source of variation	*squares (SS)*	*degrees of freedom (df)*	*Mean squares (MS)*	*F-value*	*P-value*	*critical F-value*
Between groups	8.874E-07	13	6.82608E-08	2.401233	0.058232	2.507263
Within groups	3.98E-07	14	2.84274E-08			
Total	1.285E-06	27				
within-sd	0.0001686			status:	homogeneous	
effective n	2.00					
s_{bb}	0.0001411					
u^*_{bb}	7.33E-05					
u_{bb}	0.0001411	0.141126556				
u_{bb}(rel.)	7.5588161					

Lead:

Bottle 1	0.0008	0.0010
Bottle 2	0.0007	0.0006
Bottle 3	0.0007	0.0003
Bottle 4	0.0004	0.0010
Bottle 5	0.0007	0.0011
Bottle 6	0.0011	0.0004
Bottle 7	0.0005	0.0006
Bottle 8	0.0006	0.0010
Bottle 9	0.0010	0.0005
Bottle 10	0.0009	0.0007
Bottle 11	0.0012	0.0002
Bottle 12	0.0007	0.0006
Bottle 13	0.0005	0.0008
Bottle 14	0.0012	0.0011
Bottle 15	0.0011	0.0007

Source of variation	*sums of squares (SS)*	*degrees of freedom (df)*	*Mean squares (MS)*	*F-value*	*P-value*	*critical F-value*
Between groups	6.92459E-07	14	4.94613E-08	0.49582729	0.90114195	2.42436436
Within groups	1.49633E-06	15	9.97552E-08			
Total	2.18879E-06	29				
within-sd	0.00031584			status:	homogeneous	
effective n	2.00					
s_{bb}	0					
u^*_{bb}	0.000134955					
u_{bb}	0.000134955	0.134954505				
u_{bb}(rel.)	11.81019477					

Iron:

Bottle 1	28.10	28.04
Bottle 2	28.11	28.07
Bottle 3	28.01	27.99
Bottle 4	28.08	28.11
Bottle 5	28.03	27.94
Bottle 6	28.07	28.04
Bottle 7	28.16	27.84
Bottle 8	28.16	28.14
Bottle 9	27.84	27.91
Bottle 10	27.79	27.82
Bottle 11	27.73	27.82
Bottle 12	28.02	28.23
Bottle 13	27.98	27.97
Bottle 14	27.96	27.87
Bottle 15	27.79	27.64

Source of variation	*sums of squares (SS)*	*degrees of freedom (df)*	*Mean squares (MS)*	*F-value*	*P-value*	*critical F-value*
Between groups	0.485908775	14	0.03470777	5.0636648	0.0017462	2.4243644
Within groups	0.10281418	15	0.006854279			
Total	0.588722955	29				
within-sd	0.082790571			status:	inhomogeneous	
effective n	2.00					
s_{bb}	0.118011633					
u^*_{bb}	0.035375335					
u_{bb}	0.118011633	118.0116327				
u_{bb}(rel.)	0.423301652					