Bundesanstalt für Materialforschung und -prüfung (BAM)

in cooperation with the WG 'Special Materials' of the Committee of Chemists of GDMB Gesellschaft der Metallurgen und Bergleute e.V.

Certification Report

Certified Reference Material

BAM-S010

High Purity Graphite Powder

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Summary

This report describes the preparation and certification of the reference material BAM-S010, a high purity graphite powder with certified impurity contents for 27 elements. The certification work was carried out by Bundesanstalt für Materialforschung und -prüfung (BAM) in co-operation with the working group "Special Materials" of Committee of Chemists of GDMB.

The following mass fractions and uncertainties have been certified:

Parameter	Mass fraction in mg/kg	
	Certified value [1]	Uncertainty [2]
Al	0.047	0.011
Ba	0.0023	0.0009
Be	0.000016	0.000008
Ca	0.061	0.029
Co	0.0008	0.0004
Cr	0.011	0.005
Cu	0.0049	0.0020
Fe	0.21	0.06
K	0.021	0.009
Li	0.00017	0.00010
Mg	0.019	0.005
Mn	0.0029	0.0008
Na	0.037	0.011
Ni	0.087	0.024
P	0.027	0.011
Pb	0.0029	0.0014
S	5.7	0.5
Sr	0.0008	0.0004
Ti	0.011	0.008
W	0.0036	0.0011
Y	0.00019	0.00010
Zn	0.014	0.008
Zr	0.0016	0.0005

[1] Unweighted mean value of the means of accepted sets of data, 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 approx. 95 %, as defined in the Guide to the expression of uncertainty in measurement, (GUM, ISO/IEC Guide 98-3:2008).

The following mass fractions and uncertainties are given as values for information:

Parameter	Mass fraction in mg/kg	
	Informative value [1]	Uncertainty [2]
Ag	< 0.001	
As	0.0034	0.0008
B[3]	0.0040	0.0019
Cd	0.00016	0.00009
Mo	0.0040	0.0019
Sb	< 0.03	
Si[3]	0.27	0.09
Sn	< 0.02	
Te	< 0.002	
V	0.005	0.003

[1] Unweighted mean value of the means of accepted sets of data, each set being obtained by a different laboratory and/or a different method of measurement. Values were not certified, but given for information, when the number of accepted datasets was considered to be too low (< 5) or when the uncertainty from the inter-laboratory certification was considerably larger than the expected range or homogeneity data are lacking.

[2] Estimated expanded uncertainty *U* with a coverage factor of *k* = 2, corresponding to a level of confidence of approx. 95 %, as defined in the Guide to the expression of uncertainty in measurement, (GUM, ISO/IEC Guide 98-3:2008).

[3] Method specific value obtained using ETV-ICP-OES with an evaporation temperature of 2300 °C, according to DIN 51457: 2017-05, Testing of ceramic raw and basic materials - Direct determination of mass fractions of trace impurities in powders, granules and lumps of graphite by optical emission spectroscopy by inductively coupled plasma (ICP-OES) and by electrothermal vaporization (ETV) under the action of a halogenated reaction gas (modifiers). Using ETV-ICP-OES for determination, lower mass fractions could be obtained than with other methods. In case of BAM-S010 the differences could not be observed because there was no dataset for boron and only one data set for Si obtained with other methods than ETV-ICP-OES. Therefore, the mass fractions of B and Si are only given for information.

Further informative values (laboratory mean values without statistical evaluation)

	Mass fraction in mg/kg							
Line no.	*Au*	*Bi*	*Cs*	*Eu*	*Hg*	*La*	*Rb*	*Re*
1	*< 0.00003*	*0.0013*	*<0.0004*	*< 0.00017*	*0.0049*	*0.0031*	*<0.006*	*0.00051*
2		*0.0023*	*<0.002*	*<0.0003*	*0.0071*			

Line no.	Sc	Se	Sm	Ta	Tb	Th	U	
1	*0.00004*	*<0.0042*	*< 0.00005*	*0.00054*	*< 0.0008*	*< 0.0011*	*< 0.0008*	
2	*< 0.00018*			*<0.0009*				
3	*0.00021*							

BAM-S010 is intended for the calibration of analytical instruments and to validate or verify analytical methods suitable for the determination of impurities in graphite materials. In particular, this CRM can be used as multielement standard for calibration of solid sampling methods (e.g. ETV-ICP-OES, ETV-ICP-MS). It is not suitable for the production of pressed pellets without additives.

Contents

List of abbreviations
(if not explained elsewhere)

ETV-ICP-OES	Inductively coupled plasma optical emission spectrometry with electrothermal vaporisation
INAA	Instrumental neutron activation analysis
k_0-INAA	k_0- Instrumental neutron activation analysis
SS-ET AAS	Solid sampling electrothermal atomic absorption spectrometry
ANOVA	Analysis of variance
ILC	Inter-laboratory comparison

1 Introduction

1.1 Scope

Owing to its unique combination of chemical and physical properties, graphite and in particular synthetic graphite is an important material with a very broad spectrum of industrial and scientific application. Due to its inertness to irradiation and chemical attack, graphite is used as moderator, reflector and structural material in several types of nuclear reactors. The highly refractory character combined with the high electrical and thermal conductivity makes high-purity graphite indispensable as a material for crucibles and furnaces in various analytical instruments, for electrodes, insulators, conductors, and high resistance engineering parts in space and aeronautic technology. Due to its lubricating properties graphite is widely used as a friction modifier in breaking aggregates and carbon brushes of electric motors. It is also the outgoing material for a number of modern high-tech materials such as carbon fibres, carbon-resin bonded composites, hard metals, lightweight alloys, and high-performance carbon ceramics.
The growing application of graphite materials in various fields of technology and science enhances the demand for strictly controlled properties which are often directly correlated to the contents of trace element impurities. Therefore, the availability of powerful, rapid and reliable analytical methods for the determination of trace impurities is essential for process and quality control. Certified reference materials, indispensable for the development and validation of appropriate trace analytical methods for the characterization of special purity graphite materials are still lacking. BAM-S010 was developed for the calibration of analytical instruments and to validate or verify analytical methods intended to be used for the determination of typical impurities in graphite materials. Together with CRM BAM-S009 Medium Purity Graphite there are now two graphite CRMs with different impurity levels available.

1.2 Certification procedure

Certification of reference material BAM-S010 was carried out on the basis of ISO 17034 [1] and the relevant ISO-Guides [2, 3].
The high purity graphite powder material was a commercial product (KRB2000) taken from the customary production line of the producer SGL CARBON, Bonn (Germany). It was filled into 350 polypropylene bottles each containing ca. 40 g of the material. For certification analysis one bottle was distributed to each participant of the certification inter-laboratory comparison. All participating laboratories were asked to carry out six independent determinations using an analytical method of their own choice. A technical discussion on analytical methods and on the results of the certification inter-laboratory comparison took place during the biannual sessions of the working group "Special Materials" of GDMB.
The statistical evaluation of all analytical results was performed using the software program SoftCRM 1.2.2. [4]. After removal of technical and statistical outliers the certified values were calculated as means of the laboratory means reported from the participating laboratories of the inter-laboratory comparison. The certified uncertainties were calculated taking into account the contributions from inter-laboratory comparison, from inhomogeneity of the material and contributions reflecting the average precision of accepted laboratory means.

2 Participating laboratories

2.1 Allocation and preparation of the material

- The material was produced by SGL CARBON, Bonn (Germany).
- The material was filled into cleaned sample bottles by BAM under clean air conditions.

2.2 Homogeneity testing

The analytical investigations for the homogeneity testing were carried out by Schunk Kohlenstofftechnik GmbH, Germany. For details of the investigations see Section 3.
All statistical evaluations for homogeneity testing were carried out by BAM.

2.3 Characterisation study

10 laboratories from four different countries participated in the inter-laboratory comparison for certification. These laboratories were either involved in daily analysis of graphitic and carbonaceous materials or had well known ability to analyse refractory materials by adequate analytical methods. Most of them already participated successfully in certification inter-laboratory comparisons of other special materials CRMs. Therefore, no preceding qualification round robin was carried out.
The participating laboratories are listed in alphabetical order:

Bundesanstalt für Materialforschung und –prüfung (BAM), Berlin (Germany)
3M Technical Ceramics ZNL der 3M Deutschland GmbH, Kempten (Germany)
ESK-SiC GmbH, Frechen (Germany)
HORIBA Jobin Yvon S.A.S., Longjumeau Cedex (France)
Institut "Jožef Stefan", Ljubljana (Slovenia)
Leibniz-Institut für Kristallzüchtung, Berlin (Germany)
MERSEN US, Bay City - MI (USA)
Schunk Kohlenstofftechnik GmbH, Heuchelheim (Germany)
SGL CARBON GmbH, Bonn (Germany)
Spectro Analytical Instruments, Kleve (Germany)

3 Homogeneity investigation of the material

For homogeneity testing 10 bottles were representatively taken from a total of 350 bottles by a combination of random access and systematic selection (see Table 1). From each of the N = 10 bottles four subsamples were taken for analysis.

Table 1: Selected bottles analysed for homogeneity of BAM-S010

11	40	76	99	139	172	207	231	270	300

From each of the bottles five measurements were carried out weighing 30 mg ± 5 mg directly into a graphite boat. The analyses were performed using an ETV 4000c combined with an ICP-spectrometer Arcos EOP (Spectro, Kleve, Germany). Dichlorodifluoromethane (R12) was used as reaction gas.
For the parameters Ca, Cr, Cu, Fe, Mg, Mn, Mo, Na, Ni, S, Sn, Sr, Ti, V, and Zn the analytical method used for homogeneity testing was sensitive enough to give concrete measurement results. For all other elements contents below the limit of detection of the method were found. For these elements an inhomogeneity contribution to the combined uncertainty was estimated considering similar elements.
All measurement results (raw data) are listed in Annex 1 (Tables A.1 – A.30).
The estimates of analyte-specific inhomogeneity contributions u_{bb} to be included into the total uncertainty budget were calculated according to ISO Guide 35 [3] using Eq. (1) and Eq. (2):

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

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

with:

$MS_{between}$	mean of squared deviations between bottles (from one-way ANOVA)
MS_{within}	mean of squared deviations within bottles (from one-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}. Eq. (1) does not apply if MS_{within} is larger than $MS_{between}$.
The ANOVA results together with the calculated values of s_{bb}, u^*_{bb}, and u_{bb} are listed in Table 2.

Table 2: Results from one-way ANOVA and relative uncertainty contributions due to possible inhomogeneity

Element	$MS_{between}$[a] ($\mu g^2\ kg^{-2}$)	MS_{within}[b] ($\mu g^2\ kg^{-2}$)	F_{obs}[c]	F_{crit}[d]	$s_{bb,r}$ (%)	$u^*_{bb,r}$ (%)	$u_{bb,r}$ (%)
Ca	2376.4	922.5	2.5759	2.1696	17.05	6.69	17.05
Cr	2130.2	1511.0	1.4098	2.1240	11.13	8.22	11.13
Cu	19087	12987	1.4697	2.2107	34.93	25.90	34.93
Fe	19998	29089	0.7151	2.1608	$MS_{between} < MS_{within}$	37.29	37.29
Mg	156.54	113.51	1.3791	2.1306	2.93	2.27	2.93
Mn	33.097	24.086	1.3741	2.1240	1.34	1.04	1.34
Mo	3095.6	153.32	20.1895	2.1240	24.26	2.62	2.1240
Na	936.4	1584.3	0.5911	2.1240	$MS_{between} < MS_{within}$	8.42	8.42
Ni	305.5	160.1	1.9084	2.2549	5.39	2.85	5.39
S	35874	32257	1.1121	2.1240	26.90	37.98	37.98
Sn	2036.2	77.40	26.3064	2.1240	19.79	1.86	19.79
Sr	0.7048	1.0319	0.6830	2.1240	$MS_{between} < MS_{within}$	0.22	0.22
Ti	26.091	12.047	2.1657	2.1240	1.68	0.74	1.68
V	1083	31.82	34.0235	2.2490	14.50	1.20	14.50
Zn	247.30	69.64	3.5512	2.3463	5.96	1.91	5.96

[a] Mean square between bottles from one-way ANOVA
[b] Mean square within bottles from one-way ANOVA
[c] Observed F-value: $MS_{between}/MS_{within}$
[d] Critical F-value $F_{0.05}$

The results of this study indicate that for some of the elements the material is not perectly homogeneous. The reason for this is the extremely low trace contents in the material and the resulting large scatter in the homogeneity measurements. Nevertheless, the material is suitable as a reference material.

4 Stability of the material

Graphite is known to be stable. At room temperature and atmospheric pressure, graphite is the thermodynamically stable allotrope of carbon. Graphite is one of the most chemically inert materials. Therefore, no specific stability test was performed and no contribution of long-term stability to the total uncertainty has been considered.

5 Inter-laboratory comparison for characterisation

5.1 Analytical methods used

Ten laboratories participated in the certification inter-laboratory comparison. The laboratories were asked to analyse six subsamples. They were free to choose any suitable method for analysis. For

several elements part of the laboratories used more than one analytical method and therefore reported more than one dataset.

All participating laboratories were instructed to use only calibrants prepared from pure metals or stoichiometric compounds or well checked commercial calibration solutions.
Since the element contents are very low and possible contamination from chemicals to be used for decomposition is a high risk for wrong results only methods without sample preparation (INAA, k_0-INAA) as well as typical solid sampling methods (ETV-ICP-OES, SS-ET AAS,) were used to characterise the material. The combination of results from methods based on completely different principles virtually should rule out undetected method bias.

Table 3 summarises the analytical procedures used by the participating laboratories. The laboratory code is a random number and does not correspond to the order of laboratories in Section 2.3.

Table 3: Summary of analytical procedures used by the participating laboratories

Lab code	Element	Sub-sample mass	Sample pre-treatment	Calibrants	Analytical method
3	Al, As, B, Ca, Co, Cr, Cu, Fe, K, Li, Mg, Mn, Mo, Na, Ni, Pb, S, Sb, Si, Ti, V, W, Zn, Zr	app. 25 mg	Solid sampling technique	1 g/l Merck Certipur	ETV-ICP-OES
6	Ag, Al, As, B, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Li, Mg, Mn, Mo, Na, Ni, Pb, S, Sb, Sc, Si, Sr, Te, Ti, V, W, Y, Zn, Zr	105 - 175 mg	Solid sampling technique	1 g/l Merck Certipur, SGL1 (1.4 ng/mg B)	ETV-ICP-OES
9	Ag, Au, Ba, Co, Cr, Cs, Eu, Hg, K, Mo, Na, Re, Sb, Sc, Sm, Ta, Tb, Th, U, W, Zn	0.24-0.27 g	Not applicable	Al-Au(0.1%) Alloy	k_0-INAA
	Hg	100 mg	Solid sampling technique	1 g/l commercial Hg standard solution	SS-ET AAS (DMA-80)
10	Ag, Al, As, B, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Li, Mg, Mn, Mo, Na, Ni, P, Pb, S, Si, Ta, Ti, V, W, Zn, Zr	50 - 140 mg	Solid sampling technique	1 g/l Merck Certipur, SGL1 (1.4 ng/mg B)	ETV-ICP-OES
11	Al, As, Ba, Ca, Co, Cr, Cu, Fe, K, Mg, Mn, Mo, Na, Ni, P, Si, Sr, Ti, V, W, Zn, Zr	80 - 120 mg	Solid sampling technique	1 g/l Merck Certipur or Alfa Aesar Specpure (Mo, W), checked against solutions prepared from pure metals or metal salts (Alfa Johnson Matthey)	ETV-ICP-OES
13(1)	Al, Ba, Be, Bi, Ca, Cd, Co, Cr, Cu, Fe, K, Li, Mg, Mn, Mo, Na, Ni, P, Pb, S, Sb, Si, Sn, Sr, Te, Ti, V, W, Y, Zn, Zr	58 – 158 mg	Solid sampling technique	1 g/l Merck Certipur	ETV-ICP-OES
13(2)	Al, As, B, Ba, Be, Bi, Ca, Cd, Co, Cr, Cu, Fe, K, Li, Mg, Mn, Mo, Na, Ni, P, Pb, S, Sb, Si, Sn, Sr, Ti, V, W, Y, Zn, Zr	85-180 mg	Solid sampling technique	Stock standard solutions prepared from pure metals or metal salts (Alfa Johnson Matthey), 1 g/l Merck Certipur	ETV-ICP-OES

Lab code	Element	Sub-sample mass	Sample pre-treatment	Calibrants	Analytical method
13(3)	Al, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Li, Mg, Mn, Mo, Na, Ni, P, Pb, S, Sb, Si, Sn, Sr, Te, Ti, V, W, Y, Zn, Zr	30-130 mg	Solid sampling technique	1 g/l Merck Certipur	ETV-ICP-OES
16	Ag, Al, B, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, Li, Mg, Mn, Mo, Na, Ni, P, Pb, S, Si, Ti, V, W, Zn, Zr	29 - 77 mg	Solid sampling technique	1 g/l Merck Certipur	ETV-ICP-OES
22	Al, B, Ba, Ca, Co, Cr, Cu, Fe, K, Li, Mg, Mn, Na, Ni, Pb, Si, Sr, Ti, V, Y, Zn, Zr	60 - 145 mg	Solid sampling technique	1 g/l single element standard solutions (Bernd Kraft GmbH)	ETV-ICP-OES
24	Al, B, Ba, Ca, Co, Cr, Cu, Fe, K, Li, Mg, Mn, Na, Ni, Pb, Si, Sr, Ti, V, Y, Zn, Zr	48 - 147 mg	Solid sampling technique	1 g/l Merck Certipur single element standard solutions	ETV-ICP-OES
26	Al, B, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Li, Mg, Mn, Mo, Na, Ni, P, Pb, S, Sb, Si, Sr, Ti, V, Y, Zn, Zr	19 - 45 mg	Solid sampling technique	Stock standard solutions prepared from pure metals or metal salts (Alfa Johnson Matthey), 1 g/l Merck Certipur	ETV-ICP-OES
27	Ag, As, Ba, Co, Cr, Cs, Eu, Fe, La, Na, Rb, Sb, Sc, Se, Zn	100 mg	Not applicable	Spex multi-element standard solutions	INAA
28	Al, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Pb, Si, Zn	12 - 60 mg	Solid sampling technique	Stock standard solutions prepared from pure metals or metal salts (Alfa Johnson Matthey), 1 g/l Merck Certipur	SS-ET AAS
31	Al, Ba, Ca, Cr, Cu, Fe, Mg, Mn, Ni, S, Ti, V, Zn, Zr	104 - 112 mg	Solid sampling technique	Stock standard solutions prepared from pure metals or metal salts; commercial standard solutions	ETV-ICP-OES

5.2 Statistical evaluation of ILC results

Individual results of the participants, grouped per element are compiled in Tables B.1 to B.32 of Annex B. These tables show the single results of each laboratory, the corresponding laboratories' mean values together with the intra-laboratory standard deviation (s_i). All results which were excluded from the evaluation for technical or statistical reasons are marked grey.
The graphical presentation of the results as bar graphs include the mean and standard deviation of each lab's results (accepted data sets), the certified or informative value (mean of lab means) and the corresponding expanded uncertainty (Figures B.1 – B.32).

Statistical tests and data evaluation were performed using the software program SoftCRM 1.2.2. [6].

The following tests at significance levels of 0.05 and 0.01 were carried out:

- Grubbs test for identification of outlying means
- Dixon and Nalimov test for verification of possible outlier indications
- Cochran test for identification of outlying variances
- Kolmogorov-Smirnov-Lillifors test for normality of laboratories' means distribution
- Skewness and kurtosis test for normality of laboratories' means distribution
- Snedecor F-test for equality of two standard deviations
- Bartlett test for homogeneity of variances

The results of these tests are summarised in Tables C.1 to C.31 in Annex C (with indication of the respective level of significance, α = 0.05 or α = 0.01).

An individual data set was excluded from further data processing only in case that it had been identified as Grubbs test or paired Grubbs test outlier at a significance level of α = 0.01.

Most datasets showed some outlying variances. The set of variances is often not homogeneous which is due to the fact that different methods are used each having a different repeatability and reproducibility. Furthermore, the majority of the Cochran test outliers fulfilled the criterion that the standard error of the mean ($s_i/\sqrt{n_i}$) of the dataset did not exceed the standard deviation of the distribution of all laboratories' means. Therefore, only a few flagged outlying variances were removed.

All data were technically discussed at several meetings of the Working Group "Special Materials" of the Committee of Chemists of the GDMB where some of the participating laboratories were present. After a discussion with the laboratories concerned and further examination of the methods applied, some contributions were withdrawn or excluded from the evaluation. No values were excluded from the evaluation for statistical reasons alone, data were only excluded if a sound technical reason was given. At the sessions, it was also decided to take a parameter as certified or informative value.

All accepted laboratories' mean values of the characterisation study together with the mean of the laboratory means and the standard deviation of the laboratory means are listed in Table 4.

Table 4: Means of accepted data sets

Certified values (Mass fraction in mg/kg)

Line no.	Al	Ba	Be	Ca	Co	Cr	Cu	Fe	K	Li	Mg	Mn
1	0.0328	0.00120	0.0000106	0.0282	0.000333	0.0058	0.00151	0.070	0.0084	0.000041	0.0086	0.00154
2	0.0368	0.00128	0.0000119	0.0369	0.000375	0.0063	0.00276	0.168	0.0100	0.000057	0.0123	0.00207
3	0.0368	< 0.002	0.0000133	0.0408	0.000520	0.0064	0.00333	0.173	0.0111	0.000082	0.0150	0.00262
4	0.0410	0.00203	0.0000148	0.0439	0.000590	0.0070	0.00345	0.192	0.0155	0.000130	0.0152	0.00268
5	0.0414	0.00217	0.0000286	0.0511	0.000646	0.0077	0.00392	0.197	0.0183	0.000150	0.0161	0.00278
6	0.0420	0.00232	---	0.0542	0.000696	0.0084	0.00428	0.200	0.0194	0.000175	0.0182	0.00283
7	0.0432	0.00242	---	0.0665	0.000772	0.0085	0.00440	0.202	< 0.02	0.000363	0.0205	< 0.003
8	0.0499	0.00266		0.0684	< 0.0015	0.0092	0.00750	0.212	< 0.24	0.000384	0.0209	0.00332
9	0.0517	0.00280		0.0688	0.001756	0.0095	0.00758	0.223	0.0292	---	0.0218	0.00338
10	0.0534	0.00337		0.0794	0.001882	0.0114	<0.01	0.265	0.0323	<0.005	0.0220	0.00395
11	0.0583	---		0.0845	< 0.002	0.0144	0.01028	0.292			0.0225	0.00422
12	0.0606	< 0.27		0.1042	< 0.004	0.0177		0.295			0.0234	---
13	0.0606			---	< 0.005	0.0252		---			0.0246	
14					---	---		---				
M :	0.0468	0.00225	0.0000158	0.0606	0.000841	0.0106	0.00490	0.207	0.0209	0.000173	0.0185	0.00294
s_M :	0.0095	0.00070	0.0000073	0.0222	0.000578	0.0056	0.00269	0.061	0.0120	0.000132	0.0048	0.00081

Line no.	Na	Ni	P	Pb	S	Sr	Ti	V	W	Y	Zn	Zr
1	0.0168	0.0668	0.0125	0.00116	---	0.000450	0.0044	0.00200	0.00177	0.000075	0.0059	0.00057
2	0.0198	0.0690	0.0168	0.00129	4.73	0.000597	< 0.005	0.00294	0.00261	0.000103	0.0073	<0.001
3	0.0258	0.0763	0.0201	0.00145	5.37	0.000619	0.0051	0.00333	0.00263	0.000145	0.0076	0.00113
4	0.0261	0.0773	0.0342	0.00176	5.38	0.000689	0.0059	0.00407	0.00307	0.000197	0.0081	0.00149
5	0.0279	0.0778	0.0357	< 0.0025	5.77	0.000819	0.0074	0.00489	0.00433	0.000289	0.0090	0.00151
6	0.0295	0.0789	0.0396	0.00266	5.93	0.001483	0.0090	0.00490	0.00447	0.000342	0.0097	0.00187
7	0.0367	0.0802	---	0.00359	6.14	< 0.002	0.0095	< 0.005	0.00477	<0.001	0.0105	0.00198
8	0.0368	0.0815		0.00505	6.15	---	0.0098	0.00504	0.00487		0.0109	0.00202
9	0.0372	0.0855		0.00586	6.28		0.0233	0.00673	<0.1		0.0112	0.00226
10	0.0596	0.0902		---			0.0257	0.00959			0.0160	< 0.004
11	0.0599	0.1055		<0.1				---			0.0216	
12	0.0679	0.1141									0.0280	
13	---	0.1274									0.0346	
14											---	
15											< 0.08	
M :	0.0370	0.0870	0.0265	0.00285	5.72	0.000776	0.0111	0.00483	0.00356	0.000192	0.0139	0.00160
s_M :	0.0167	0.0180	0.0114	0.00181	0.53	0.000367	0.0079	0.00226	0.00119	0.000106	0.0089	0.00055

Informative values (Mass fraction in mg/kg)

Line no.	*Ag*	*As*	*B*	*Cd*	*Mo*	*Sb*	*Si*	*Sn*	*Te*
1	*0.000058*	*0.00245*	*0.00117*	*0.000071*	*0.00248*	*0.00114*	*0.155*	*0.00162*	*0.00028*
2	*0.000159*	*0.00350*	*0.00171*	*0.000072*	*0.00286*	*0.00195*	*0.167*	*0.00423*	*0.00095*
3	*0.000296*	*0.00377*	*0.00227*	*0.000103*	*0.00370*	*0.00242*	*0.180*	*0.01785*	*0.00193*
4	*<0.0013*	*0.00386*	*0.00388*	*0.000122*	*0.00386*	*0.00606*	*0.202*		
5	*<0.012*	---	*0.00612*	*< 0.00013*	*0.00422*	*0.00650*	*0.216*		
6		*<0.1*	*0.00614*	*0.000270*	*0.00491*	*0.00650*	*0.249*		
7			*0.00635*	*0.000300*	*0.00509*	*0.02032*	*0.250*		
8			*<0.02*	---	*0.00518*	*<0.1*	*0.306*		
9					*< 0.006*		*0.341*		
10					*<0.1*		*0.440*		
11							*0.443*		
12									
M :	*0.000171*	*0.00339*	*0.00395*	*0.000156*	*0.00404*	*0.00641*	*0.268*	*0.00790*	*0.00105*
s_M :	*0.000119*	*0.00065*	*0.00227*	*0.000102*	*0.00101*	*0.00655*	*0.101*	*0.00872*	*0.00083*

The omission of outlying values is indicated by "---".

6 Certified and informative values

6.1 Certified values and uncertainties

Certified values for all analytes are based on the unweighted mean of the means of the accepted datasets as shown in Table 4. Full uncertainty budgets following the 'Guide to the Expression of Uncertainty in Measurement' [7] were established.

The uncertainty of the certified values contains contributions of the certification inter-laboratory comparison (u_{ilc}), the potential between-unit inhomogeneity (u_{bb}). The different contributions to the uncertainty are then combined using the following equation:

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

- u_{ilc} was calculated using Equation (4), where s_M is the standard deviation of the laboratories' means (see Table 4) and N is the number of accepted data sets

$$u_{\mathrm{ilc}} = \sqrt{\frac{s_{\mathrm{M}}^2}{N}} \qquad (4)$$

- u_{bb} was estimated as described in Section 3.

The expanded uncertainty of the certified value U_{CRM} was calculated according to GUM [4] with a coverage factor of k = 2, representing a level of confidence of approximately 95%, as

$$U_{\mathrm{CRM}} = 2 \cdot u_{\mathrm{c}} \qquad (5)$$

The certified mass fractions and the results from the calculation of the combined uncertainty and their associated expanded uncertainties are summarised in Table 5.
Finally, a summary of the certified mass fractions with their respective expanded uncertainties after rounding according to DIN 1333 [8] is given on Page 2 of this report.

Table 5: Certified values and their uncertainties (before rounding)

Element	Certified value (mg/kg)	U_{CRM} (mg/kg)	s_M (mg/kg)	n	u_{ilc} (mg/kg)	u_{bb} (mg/kg)	u_c (mg/kg)
Al	0.0468	0.0107	0.0095	13	0.0026	0.0047*	0.0054
Ba	0.00225	0.00082	0.00070	9	0.00023	0.00039*	0.00041
Be	0.0000158	0.000008	0.0000073	5	0.000003	0.000002*	0.000004
Ca	0.0606	0.034	0.0222	12	0.0064	0.0127	0.0142
Co	0.000841	0.00042	0.000578	9	0.00019	0.00008*	0.00021
Cr	0.0106	0.0042	0.0056	13	0.00154	0.0014	0.0021
Cu	0.00490	0.00221	0.00269	10	0.00085	0.00071	0.00111
Fe	0.207	0.0591	0.061	12	0.0175	0.024	0.0296
K	0.0209	0.0083	0.0120	9	0.0040	0.0011*	0.0041
Li	0.000173	0.000095	0.000132	8	0.000047	0.000009*	0.000047
Mg	0.0185	0.005	0.0048	13	0.00133	0.0021	0.0025
Mn	0.00294	0.00079	0.00081	10	0.00026	0.00030	0.00040
Na	0.0370	0.0102	0.0167	12	0.00483	0.00161	0.00509
Ni	0.0870	0.0292	0.0180	13	0.0050	0.0109	0.0120
P	0.0265	0.0107	0.0114	6	0.0047	0.0027	0.0054
Pb	0.00285	0.00140	0.00181	8	0.00064	0.00029*	0.00070
S	5.72	0.42	0.53	8	0.19	0.093	0.21
Sr	0.000776	0.00031	0.000367	6	0.00015	0.00005	0.00016
Ti	0.0111	0.0079	0.0079	9	0.0026	0.0030	0.0040
V	0.00483	0.0031	0.00226	9	0.00075	0.00103	0.0013
W	0.00356	0.0011	0.00119	8	0.00042	0.00036*	0.00055
Y	0.000192	0.00010	0.000106	6	0.00004	0.00002*	0.00005
Zn	0.0139	0.0073	0.0089	13	0.0025	0.0027	0.0037
Zr	0.00160	0.0005	0.00055	8	0.00020	0.00016*	0.00025

*estimated from the results for other elements

According to DIN 51457 [6] for the elements B and Si method-dependent differences could be observed. Using ETV-ICP-OES for determination lower mass fractions could be obtained than with other methods. In case of BAM-S010 the differences could not be observed because there was no dataset for boron and only one data set for Si obtained with other methods than ETV-ICP-OES. Therefore, the mass fractions of B and Si are only given for information.

6.2 Informative values and uncertainties

The data obtained for the elements Ag, As, Cd, Sb, Sn and Te did not allow a certification.

For these elements, only 3-8 valid data sets are available, which scatter more than for the elements listed above (Table 5).

The informative values and the results from the calculation of the combined uncertainty and their associated expanded uncertainties are summarised in Table 6.

Finally, a summary of the informative mass fractions with their respective expanded uncertainties after rounding according to DIN 1333 [7] is given on Page 3 of this report.

Table 6: Informative values and their uncertainties

Element	Informative value (mg/kg)	U_{CRM} (mg/kg)	s_M (mg/kg)	n	u_{ilc} (mg/kg)	u_{bb} (mg/kg)	u_c (mg/kg)
Ag	0.000171	0.00014	0.000119	3	0.00007	0.00002*	0.00007
As	0.00339	0.00073	0.00065	4	0.00032	0.00017*	0.00037
B	0.0040	0.0019	0.00227	7	0.00086	0.00039*	0.00094
Cd	0.000156	0.000089	0.000102	6	0.000042	0.000016*	0.000044
Sb	0.00641	0.0051	0.00655	7	0.0025	0.00064*	0.0026
Mo	0.00404	0.00185	0.00101	8	0.00036	0.00085	0.00092
Si	0.268	0.084	0.101	10	0.032	0.027*	0.042
Sn	0.00790	0.0104	0.00872	3	0.00503	0.0013*	0.0052
Te	0.00105	0.00098	0.00083	3	0.00048	0.00011*	0.00049

*estimated from the results for other elements

Some additional data are available for Au, Bi, Cs, Eu, Hg, La, Rb, Re, Sc, Se, Sm, Ta, Tb, Th and U. Data are based on 1 – 3 series of results without statistical evaluation, some of them were obtained by one method only. A major part of them is given as "less than" value. The complete tabular summary is given on Page 3 of this report.

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 traceable commercial calibration solutions.

8 Instructions for use

8.1 Area of application

The main area of application is to check the trueness of results when one or more of the certified parameters in graphite powder are determined by a laboratory. Based on own results and on certified values the uncertainty of own measurements can be calculated.
As any reference material, it can also be used for routine performance checks (control charts) or validation studies.

8.2 Recommendations for correct sampling and sample preparation

To ensure a representative sub-sampling for the analysis the bottle containing the CRM should be shaken in different directions for about 20 seconds before taking the sub-sample. Each sub-sample has to be taken separately. The analytical sample for analysis should be taken as it is. According to the sub-sample masses used for the homogeneity testing the minimum sample intake for analysis is 35 mg. The opening duration of the bottle should be as short as possible. The lid of the bottle should be locked tightly immediately after usage.

8.3 Recommendations for correct storage

The sample should be stored at room temperature in a dust-free and dry environment avoiding contamination and moisture.

8.4 Safety guidelines

The usual laboratory safety precautions must be applied. Graphite powder is not known to be toxic. No hazardous effect is to be expected if the material is used under conditions usually adopted in analytical laboratories when handling finely dispersed powder materials. 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.

9 References

[1] DIN EN ISO 17034, General requirements for the competence of reference material producers, 2017

[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] G. Bonas, M. Zervou, T. Papaeoannou, M. Lees, "SoftCRM": a new software for the certification of reference materials, Accred. Qual. Assur. 8 (2003) 101-107.

[5] ISO/IEC Guide 98-3:2008, Uncertainty in measurement – Part 3: Guide to the expression of uncertainty in measurement (GUM:1995).

[6] DIN 51457:2017: Prüfung keramischer Roh- und Werkstoffe - Direkte Bestimmung der Massenanteile von Spurenverunreinigungen in pulver-, kornförmigem und stückigem Graphit mittels optischer Emissionsspektrometrie mit induktiv gekoppeltem Plasma (ICP-OES) und elektrothermischer Verdampfung (ETV) unter Einwirkung eines halogenierenden Reaktionsgases (Modifiers)

[7] DIN 1333:1992, Zahlenangaben (Presentation of numerical data)

10 Regulatory information

- ASTM C560-15 Standard Test Methods for Chemical Analysis of Graphite.
- DIN 51457: 2017-05, Testing of ceramic raw and basic materials - Direct determination of mass fractions of trace impurities in powders, granules and lumps of graphite by optical emission spectroscopy by inductively coupled plasma (ICP-OES) and by electrothermal vaporization (ETV) under the action of a halogenated reaction gas (modifiers)

11 Informative references

- H.O. Pierson, Handbook of carbon, graphite, diamond and fullerenes. Properties, processing and applications, Noyes Publications 1993, Park Ridge New Jersey, USA.
- H. Hashitani, Wet oxidation decomposition of graphite for determination of impurity elements, Bunseki Kagaku 35 (1986) 911-915.
- Y. Koshino, A. Narukawa, Determination of trace metal impurities in graphite powders by acid pressure decomposition and inductively coupled plasma atomic emission spectrometry, Analyst 118 (1993) 827-830.
- Z. Hladky, M. Fišera, Determination of trace impurities in high-purity graphite by electrothermal atomic absorption spectrometry and inductively coupled plasma atomic emission spectrometry, J. Anal. Ato. Spectrom. 9 (1994) 1285-1287.

- U. Schäffer, V. Krivan, Slurry sampling electrothermal atomic absorption spectrometry for the analysis of graphite powders, Spectrochim. Acta Part B 51 (1996) 1211-1222.
- W. Bögershausen, R. Cicciarelli, B. Gercken, E. König, V. Krivan, R. Müller-Käfer, J. Pavel, H. Seltner, J. Schelcher, Pure graphite as a reference material for the determination of trace elements – an interlaboratory collaborative study, Fresenius J. Anal. Chem. 357 (1997) 266-273.
- C. Pickhardt, J.S. Becker, Trace analysis of high-purity graphite by LA-ICP-MS, Fresenius J. Anal. Chem. 370 (1997) 534-540.
- U. Schäffer, V. Krivan, Multielement analysis of graphite and silicon carbide by inductively coupled plasma atomic emission spectrometry using solid sampling and electrothermal vaporization, Anal. Chem. 71 (1999) 849-854.
- M. Watanabe, A. Narukawa, Determination of impurity elements in high purity graphite by inductively coupled plasma atomic emission spectrometry after microwave decomposition, Analyst 125 (2000) 1189-1191.
- U. Schäffer, V. Krivan, Analysis of high purity graphite and silicon carbide by direct solid sampling electrothermal atomic absorption spectrometry, Fresenius J. Anal. Chem. 371 (2001) 859-866.
- P.A. Mello, M.F. Pedrotti, S.M. Cruz, E.I. Muller, V.L. Dressler, E.M.M. Flores, Determination of rare earth elements in graphite by solid sampling electrothermal vaporization-inductively coupled plasma mass spectrometry, J. Anal. At. Spectrom. 30 (2015) 2048-2055.
- S.M. Cruz, L. Schmidt, F.M. Dalla Nora, M.F. Pedrotti, C.A. Bizzi, J.S. Barin, E.M.M. Flores, Microwave-induced combustion method for the determination of trace and ultratrace element impurities in graphite samples by ICP-OES and ICP-MS, Microchem. J. 123 (2015) 28-32.

12 Annexes

Annex A: Data from homogeneity study

Annex B: Results obtained in the characterisation study

Annex C: Outcome of statistical tests on results from the ILC

Annex A: Data from homogeneity study

Table A.1: Homogeneity study, results for Ca

Bottle No.	Ca mass fraction (µg/kg)							RSD (%)
	#1	#2	#3	#4	#5	Mean	SD_i	
11	38.6	---	49.3	53.7	23.6	41.3	13.4	32.4
40	50.3	43.2	40.9	29.4	---	41.0	8.7	21.2
76	68.4	69.8	81.9	115.5	---	83.9	21.9	26.1
90	90.0	120.1	---	103.6	88.1	100.5	14.8	14.7
139	76.1	38.1	101.7	68.1	92.5	75.3	24.6	32.7
172	112.5	107.0	118.7	---	76.8	103.8	18.6	17.9
207	149.3	113.5	126.7	123.9	35.7	109.8	43.4	39.6
231	84.3	158.1	94.2	24.4	35.0	79.2	53.5	67.5
270	144.6	88.1	75.4	64.8	---	93.2	35.5	38.1
300	49.6	76.9	97.2	105.0	105.3	86.8	23.8	27.4

Table A.2: Homogeneity study, results for Cr

Bottle No.	Cr mass fraction (µg/kg)							RSD (%)
	#1	#2	#3	#4	#5	Mean	SD_i	
11	102.2	93.7	189.8	54.2	61.1	100.2	54.1	54.0
40	80.6	71.6	78.7	68.6	68.2	73.5	5.8	7.8
76	85.1	73.0	73.9	114.3	78.2	84.9	17.1	20.2
90	72.9	65.0	75.4	74.4	80.8	73.7	5.7	7.7
139	83.2	62.5	64.9	109.3	65.2	77.0	19.9	25.8
172	58.9	185.1	279.7	67.7	66.8	131.6	97.9	74.4
207	66.3	90.3	73.3	75.7	128.8	86.9	25.0	28.8
231	83.1	100.0	77.9	107.6	78.9	89.5	13.5	15.1
270	80.1	66.4	61.5	64.7	2.7	55.1	30.1	54.7
300	80.0	53.0	77.7	69.1	76.3	71.2	11.0	15.4

Table A.3: Homogeneity study, results for Cu

Bottle No.	Cu mass fraction (µg/kg)						RSD (%)
	#1	#2	#3	#4	Mean	SD_i	
11	351.5	375.0	313.0	243.4	320.7	57.5	17.9
40	457.9	330.9	333.8	257.1	344.9	83.3	24.1
76	290.7	412.3	359.2	249.7	328.0	72.1	22.0
90	328.8	273.5	268.6	163.8	258.7	68.9	26.6
139	397.9	391.7	238.1	256.9	321.2	85.4	26.6
172	303.8	442.3	693.5	284.8	431.1	188.5	43.7
207	672.3	227.5	397.4	563.0	465.1	194.6	41.8
231	510.9	300.2	456.2	565.3	458.2	114.3	25.0
270	475.8	332.2	279.5	336.6	356.0	84.0	23.6
300	299.9	401.3	463.2	520.0	421.1	94.2	22.4

Table A.4: Homogeneity study, results for Fe

Bottle No.	Fe mass fraction (µg/kg)							RSD (%)
	#1	#2	#3	#4	#5	Mean	SD_i	
11	257.8	495.6	---	607.5	210.7	392.9	189.8	48.3
40	213.5	357.3	254.8	687.8	204.6	343.6	201.7	58.7
76	328.9	284.9	436.9	505.0	593.2	429.8	126.1	29.3
90	334.2	194.5	261.8	190.8	248.8	246.0	58.6	23.8
139	339.5	177.9	226.3	---	339.8	270.9	81.8	30.2
172	195.3	994.1	---	115.4	178.9	370.9	416.9	112.4
207	329.8	459.6	379.6	274.2	345.7	357.8	68.5	19.1
231	238.3	444.7	231.1	571.8	229.2	343.0	157.4	45.9
270	---	240.1	234.8	206.7	---	227.2	18.0	7.9
300	324.3	194.6	266.9	241.7	295.8	264.7	49.9	18.9

Table A.5: Homogeneity study, results for Mg

Bottle No.	Mg mass fraction (µg/kg)							RSD (%)
	#1	#2	#3	#4	#5	Mean	SD_i	
11	17.9	25.5	22.7	29.8	16.1	22.4	5.6	24.9
40	19.3	18.5	22.4	15.0	14.3	17.9	3.3	18.5
76	28.6	26.4	25.1	27.9	28.7	27.3	1.6	5.7
90	25.6	30.9	41.2	24.9	28.6	30.2	6.6	21.8
139	23.9	15.5	26.6	30.1	17.4	22.7	6.2	27.1
172	33.4	24.7	42.4	7.0	25.9	26.7	13.1	49.0
207	42.5		56.7	30.9	27.6	39.4	13.2	33.4
231	16.6	11.4	57.1	7.7	10.2	20.6	20.7	100.3
270	34.6	37.0	29.5	23.4	1.9	25.3	14.1	55.8
300	17.3	20.2	30.4	26.5	35.5	26.0	7.4	28.5

Table A.6: Homogeneity study, results for Mn

Bottle No.	Mn mass fraction (µg/kg)							RSD (%)
	#1	#2	#3	#4	#5	Mean	SD_i	
11	11.2	19.3	8.7	9.7	9.9	11.8	4.3	36.6
40	21.1	13.3	9.0	38.8	8.5	18.1	12.6	69.6
76	14.4	9.5	8.6	10.0	10.3	10.6	2.2	21.3
90	15.3	13.1	14.0	13.6	13.2	13.8	0.9	6.4
139	14.1	13.1	13.1	12.8	12.8	13.2	0.5	4.1
172	9.2	9.4	18.9	13.7	12.7	12.8	4.0	31.0
207	12.3	17.5	11.7	25.3	12.3	15.8	5.8	36.6
231	10.6	9.3	14.4	13.3	9.0	11.3	2.4	21.3
270	13.5	13.8	15.5	13.3	14.4	14.1	0.9	6.3
300	8.4	8.9	8.9	9.6	11.2	9.4	1.1	11.6

Table A.7: Homogeneity study, results for Mo

Bottle No.	Mo mass fraction (µg/kg)							RSD (%)
	#1	#2	#3	#4	#5	Mean	SD_i	
11	160.1	164.8	172.4	163.7	135.5	159.3	14.0	8.8
40	142.3	137.2	152.9	138.6	165.1	147.2	11.7	8.0
76	137.5	139.5	138.7	141.3	149.8	141.4	4.9	3.5
90	127.6	116.7	122.1	113.8	66.8	109.4	24.4	22.3
139	108.5	116.6	119.2	102.4	91.7	107.7	11.1	10.3
172	109.9	109.1	111.0	99.0	100.4	105.9	5.7	5.4
207	118.8	104.7	99.2	106.3	84.3	102.7	12.5	12.2
231	91.5	94.1	91.7	92.9	78.8	89.8	6.2	6.9
270	99.9	98.6	92.3	87.7	78.0	91.3	8.9	9.8
300	91.8	97.5	100.6	111.7	78.6	96.0	12.1	12.6

Table A.8: Homogeneity study, results for Na

Bottle No.	Na mass fraction (µg/kg)							RSD (%)
	#1	#2	#3	#4	#5	Mean	SD_i	
11	230.0	246.8	170.7	181.1	159.0	197.5	38.6	19.5
40	256.2	167.2	242.8	253.1	100.3	203.9	68.5	33.6
76	120.7	181.6	159.4	203.2	176.2	168.2	30.8	18.3
90	155.0	276.3	237.4	249.1	178.7	219.3	50.6	23.1
139	201.9	146.1	219.4	211.5	150.1	185.8	35.0	18.8
172	188.4	223.5	169.8	168.2	195.2	189.0	22.5	11.9
207	213.2	122.7	203.5	207.7	230.6	195.5	42.0	21.5
231	195.7	174.3	235.3	146.1	176.4	185.6	33.0	17.8
270	179.3	204.2	197.1	165.5	187.4	186.7	15.2	8.1
300	151.5	213.8	250.1	183.9	208.1	201.5	36.6	18.2

Table A.9: Homogeneity study, results for Ni

Bottle No.	Ni mass fraction (µg/kg)							RSD (%)
	#1	#2	#3	#4	#5	Mean	SD_i	
11	32.5	13.9	---	59.4	52.4	39.6	20.5	52.0
76	49.9	39.3	49.0	69.1	53.2	52.1	10.8	20.8
90	45.5	47.2	45.7	32.0	37.5	41.6	6.6	15.8
139	37.3	38.7	42.7	77.8	59.1	51.1	17.3	33.8
172	42.9	---	---	---	40.4	41.7	1.8	4.2
207	42.0	21.0	27.6	34.3	16.1	28.2	10.3	36.6
231	31.4	55.5	33.0	38.4	32.1	38.1	10.1	26.6
270	64.0	39.6	36.7	33.6	---	43.5	13.9	32.0
300	54.7	66.3	50.2	36.9	53.0	52.2	10.5	20.2

Table A.10: Homogeneity study, results for S

Bottle No.	S mass fraction (µg/kg)							RSD (%)
	#1	#2	#3	#4	#5	Mean	SD_i	
11	2534	2235	2181	1989	2270	2242	196	8.7
40	2490	2351	2384	2367	2350	2388	58	2.4
76	2365	2394	2143	2275	2548	2345	150	6.4
90	2457	2280	2273	2104	2078	2238	154	6.9
139	2212	2301	2413	2346	2357	2326	75	3.2
172	2424	2474	2465	2447	2214	2405	108	4.5
207	2491	2453	2303	2100	2695	2408	222	9.2
231	2273	2324	2202	1994	2485	2256	180	8.0
270	2527	2396	1906	2265	2708	2360	302	12.8
300	2220	2617	2491	2404	2776	2502	211	8.4

Table A.11: Homogeneity study, results for Sn

Bottle No.	Sn mass fraction (µg/kg)							RSD (%)
	#1	#2	#3	#4	#5	Mean	SD_i	
11	208.9	147.9	156.1	155.5	159.5	165.6	24.6	14.8
40	141.7	135.9	143.3	139.0	151.3	142.2	5.8	4.1
76	135.6	131.1	127.5	135.9	137.3	133.5	4.1	3.1
90	115.1	107.6	115.0	107.7	105.5	110.2	4.5	4.1
139	114.2	111.6	116.2	107.1	108.1	111.4	3.9	3.5
172	103.4	107.5	107.9	113.0	112.6	108.9	4.0	3.7
207	110.4	112.0	103.8	112.1	109.7	109.6	3.4	3.1
231	108.9	105.6	108.2	105.5	104.4	106.5	1.9	1.8
270	112.4	116.3	100.9	102.6	101.7	106.8	7.1	6.6
300	108.3	107.8	107.3	109.5	105.1	107.6	1.6	1.5

Table A.12: Homogeneity study, results for Sr

Bottle No.	Sr mass fraction (µg/kg)							RSD (%)
	#1	#2	#3	#4	#5	Mean	SD_i	
11	3.80	5.39	3.49	2.94	0.85	3.29	1.64	49.9
40	3.92	3.91	3.83	3.84	3.88	3.88	0.04	1.0
76	4.10	3.40	3.66	3.84	5.10	4.02	0.66	16.3
90	3.71	3.80	5.12	2.51	0.25	3.08	1.83	59.6
139	3.11	3.24	3.68	3.01	2.66	3.14	0.37	11.8
172	3.66	3.63	3.31	2.88	3.74	3.44	0.36	10.3
207	4.43	3.61	3.49	4.96	2.99	3.90	0.79	20.2
231	3.67	3.55	3.72	3.77	2.53	3.45	0.52	15.1
270	3.21	3.14	3.71	3.00	1.89	2.99	0.67	22.4
300	4.08	3.46	3.36	6.07	1.97	3.79	1.49	39.4

Table A.13: Homogeneity study, results for Ti

Bottle No.	Ti mass fraction (µg/kg)							RSD (%)
	#1	#2	#3	#4	#5	Mean	SD_i	
11	12.40	10.70	11.20	6.32	0.27	8.18	4.98	60.9
40	4.54	1.92	3.30	3.08	3.86	3.34	0.97	29.2
76	3.17	3.51	3.87	4.34	3.73	3.72	0.43	11.7
90	14.80	11.70	10.40	6.97	0.89	8.95	5.31	59.3
139	7.79	7.89	8.75	5.50	2.48	6.48	2.54	39.2
172	11.50	14.10	11.90	5.89	10.70	10.82	3.03	28.0
207	13.90	4.12	2.99	9.65	---	7.67	5.07	66.2
231	7.50	8.27	9.17	5.60	3.89	6.89	2.13	30.9
270	7.53	6.45	5.79	5.64	---	6.35	0.86	13.5
300	9.31	14.10	7.79	9.55	1.62	8.47	4.50	53.1

Table A.14: Homogeneity study, results for V

Bottle No.	V mass fraction (µg/kg)							RSD (%)
	#1	#2	#3	#4	#5	Mean	SD_i	
76	92.6	95.3	92.2	95.7	104.6	96.08	5.01	5.2
90	81.2	83.7	83.3	82.3	54.3	76.96	12.70	16.5
139	71.9	65.3	71.2	65.5	60.3	66.84	4.78	7.2
172	71.7	71.2	68.9	62.2	65.5	67.90	4.02	5.9
207	73.3	65.6	58.1	65.8	58.5	64.26	6.27	9.7
231	53.6	53.8	53.1	58.4	56.2	55.02	2.24	4.1
270	61.7	55.7	57.5	53.0	46.6	54.90	5.62	10.2
300	62.4	64.8	64.0	63.0	54.6	61.76	4.11	6.7

Table A.15: Homogeneity study, results for Zn

Bottle No.	Zn mass fraction (µg/kg)							RSD (%)
	#1	#2	#3	#4	#5	Mean	SD_i	
40	33.8	34.2	---	32.3	34.3	33.7	0.93	2.8
76	18.1	22.1	23.9	30.7	29.6	24.9	5.26	21.2
90	31.2	31.0	34.2	---	20.9	29.3	5.80	19.8
139	30.7	26.5	29.5	22.7	45.8	31.0	8.81	28.4
172	47.3	56.3	62.7	16.8	45.1	45.6	17.60	38.6
231	21.3	22.8	24.8	24.9	19.4	22.6	2.35	10.4
270	35.3	29.7	21.2	19.4	---	26.4	7.44	28.2
300	24.2	26.2	31.7	33.4	32.3	29.6	4.09	13.8

Annex B: Results obtained in the characterisation study

Table B.1: Individual results for Ag (values in mg/kg)
Data not used for certification is indicated by a grey background.

Lab No.	Lab code	Method	Sub-sample #1	Sub-sample #2	Sub-sample #3	Sub-sample #4	Sub-sample #5	Sub-sample #6	**Mean**	s_i
L1	6	ETV-ICP-OES	0.00006	0.00005	0.00005	0.00008	0.00006	0.00005	**0.00006**	0.00001
L2	13	ETV-ICP-OES	0.00006	0.00006	0.00005	0.00008	0.00006	0.00005	**0.00006**	0.00001
L3	16	ETV-ICP-OES	0.00015	0.00017	0.00015	0.00018	0.00015	0.00014	**0.00016**	0.00002
L4	10	ETV-ICP-OES	0.00019	0.00021	0.00034	0.00040	0.00023	0.00041	**0.00030**	0.00010
L5	27	INAA	<0.0012	<0.0012	<0.0013	<0.0012	0.00120	<0.0012	**<0.0013**	
L6	9	k_0-INAA	<0.010	<0.011	<0.012	<0.012	<0.011	< 0.009	**<0.012**	

[1] Data given as below limit of quantification

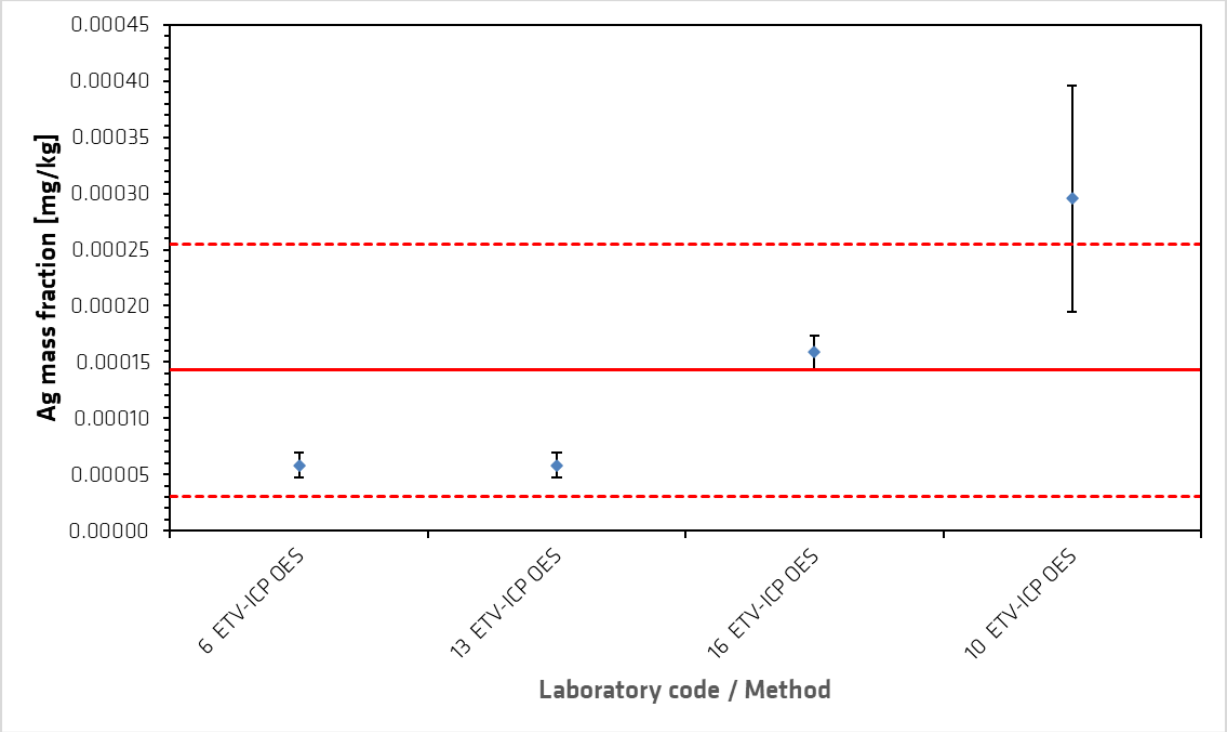

Figure B.1: Accepted laboratory means for Ag. Uncertainty bars represent the standard deviation of each lab's results. The solid red line represents the informative value (the mean of laboratory means), while the broken red lines give the standard deviation of the laboratories' mans.

Table B.2: Individual results for Al (values in mg/kg)

Lab No.	Lab code	Method	Sub-sample #1	Sub-sample #2	Sub-sample #3	Sub-sample #4	Sub-sample #5	Sub-sample #6	**Mean**	s_i
L1	3	ETV-ICP-OES	0.02337	0.03548	0.02000	0.05624	0.03061	0.03106	**0.03279**	0.01278
L2	24	ETV-ICP-OES	0.03661	0.03864	0.02722	0.03996	0.03743	0.04070	**0.03676**	0.00491
L3	28	SS-ET AAS	0.02948	0.04009	0.03838	0.04057	0.03359	0.03873	**0.03681**	0.00436
L4	13(3)	ETV-ICP-OES	0.03931	0.04710	0.04781	0.03698	0.03385	0.04066	**0.04095**	0.00555
L5	13(1)	ETV-ICP-OES	0.05105	0.03347	0.03719	0.04689	0.03557	0.04413	**0.04138**	0.00700
L6	11	ETV-ICP-OES	0.03760	0.04410	0.03590	0.04540	0.03640	0.05260	**0.04200**	0.00658
L7	6	ETV-ICP-OES	0.04413	0.04100	0.05988	0.02861	0.05193	0.03372	**0.04321**	0.01151
L8	10	ETV-ICP-OES	0.06069	0.03799	0.05633	0.03637	0.05419	0.05379	**0.04989**	0.01016
L9	31	ETV-ICP-OES	0.05458	0.05482	0.03807	0.05935			**0.05170**	0.00935
L10	13(2)	ETV-ICP-OES	0.05284	0.05836	0.05397	0.06006	0.04308	0.05219	**0.05342**	0.00596
L11	26	ETV-ICP-OES	0.06506	0.06856	0.05769	0.06512	0.05009	0.04301	**0.05826**	0.00999
L12	16	ETV-ICP-OES	0.05030	0.05634	0.05442	0.06907	0.05957	0.07376	**0.06058**	0.00904
L13	22	ETV-ICP-OES	0.06530	0.06570	0.04480	0.07050	0.05480	0.06270	**0.06063**	0.00931

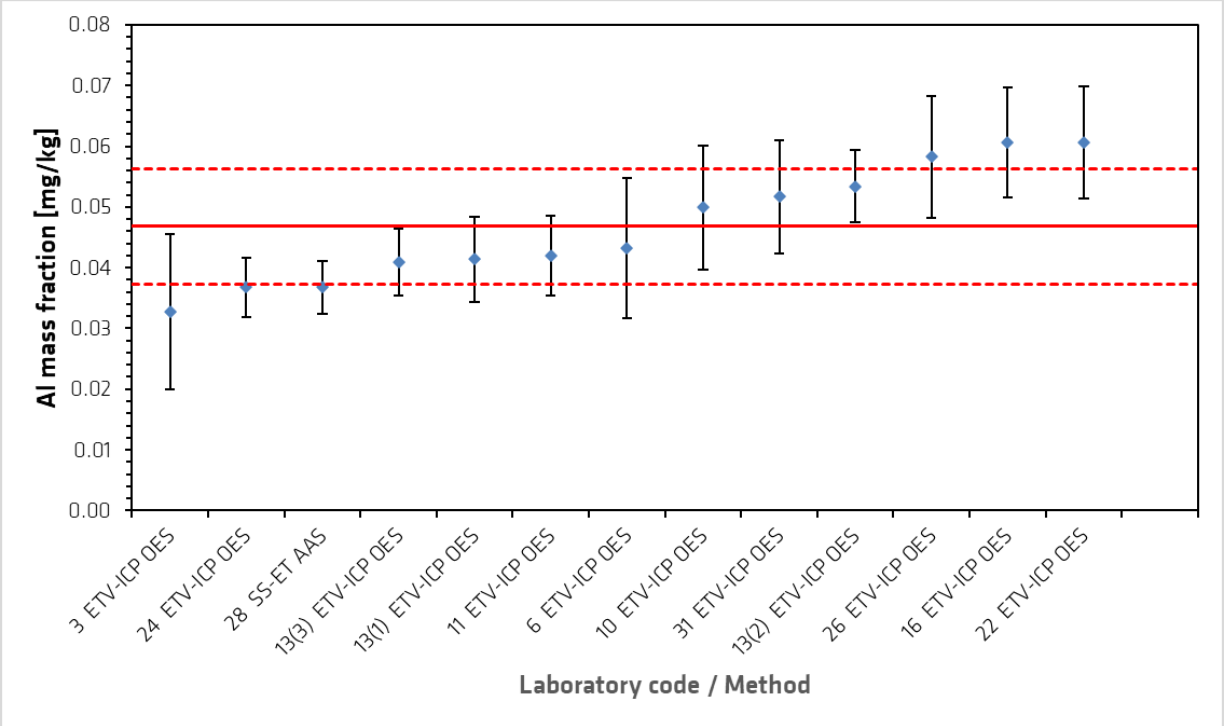

Figure B.2: Accepted laboratory means for Al. Uncertainty bars represent the standard deviation of each lab's results. The solid red line represents the certified value (the mean of laboratory means), while the broken red lines give the standard deviation of the laboratories' mans.

Table B.3: Individual results for As (values in mg/kg)
Data not used for certification is indicated by a grey background.

Lab No.	Lab code	Method	Sub-sample #1	Sub-sample #2	Sub-sample #3	Sub-sample #4	Sub-sample #5	Sub-sample #6	**Mean**	s_i
L1	13(2)	ETV-ICP-OES	0.00258	0.00215	0.00264	0.00268	0.00226	0.00238	**0.00245**	0.00022
L2	27	INAA	0.00350	0.00370	0.00290	0.00300	0.00400	0.00390	**0.00350**	0.00046
L3	11	ETV-ICP-OES	0.00440	0.00390	0.00340	0.00380	0.00330	0.00380	**0.00377**	0.00039
L4	6	ETV-ICP-OES	0.00399	0.00386	0.00413	0.00391	0.00352	0.00373	**0.00386**	0.00021
L5 [3)]	10	ETV-ICP-OES	0.00451	0.00141	0.00444	0.00170	0.01157	0.00139	**0.00417**	0.00391
L6 [1)]	3	ETV-ICP-OES	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	**<0.1**	

[1)] Data given as below limit of quantification
[3)] Outlier, dataset rejected

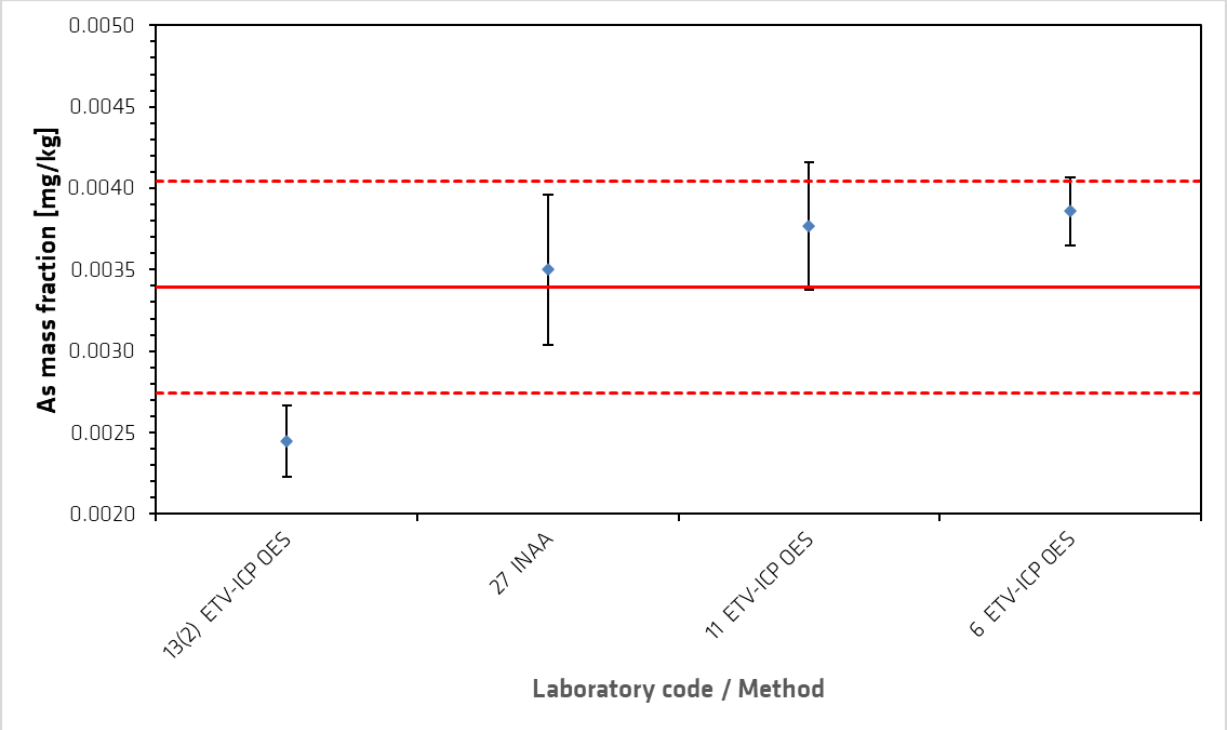

Figure B.3: Accepted laboratory means for As. Uncertainty bars represent the standard deviation of each lab's results. The solid red line represents the informative value (the mean of laboratory means), while the broken red lines give the standard deviation of the laboratories' means.

Table B.4: Individual results for B (values in mg/kg)

Lab No.	Lab code	Method	Sub-sample #1	Sub-sample #2	Sub-sample #3	Sub-sample #4	Sub-sample #5	Sub-sample #6	**Mean**	s_i
L1	24	ETV-ICP-OES	0.00154	0.00130	0.00100	0.00119	0.00081	0.00121	**0.00117**	0.00025
L2	10	ETV-ICP-OES	0.00185	0.00240	0.00170	0.00149	0.00138	0.00145	**0.00171**	0.00038
L3	6	ETV-ICP-OES	0.00245	0.00171	0.00251	0.00247	0.00239	0.00208	**0.00227**	0.00031
L4	22	ETV-ICP-OES	0.00380	0.00210	0.00550	0.00400	0.00340	0.00450	**0.00388**	0.00113
L5	16	ETV-ICP-OES	0.00636	0.00540	0.00541	0.00765	0.00697	0.00493	**0.00612**	0.00106
L6	13(2)	ETV-ICP-OES	0.00687	0.00831	0.00586	0.00610	0.00414	0.00556	**0.00614**	0.00139
L7	26	ETV-ICP-OES	0.00740	0.00762	0.00624	0.00615	0.00465	0.00604	**0.00635**	0.00107
L8 [1)]	3	ETV-ICP-OES	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	**<0.02**	

[1)] Data given as below limit of quantification

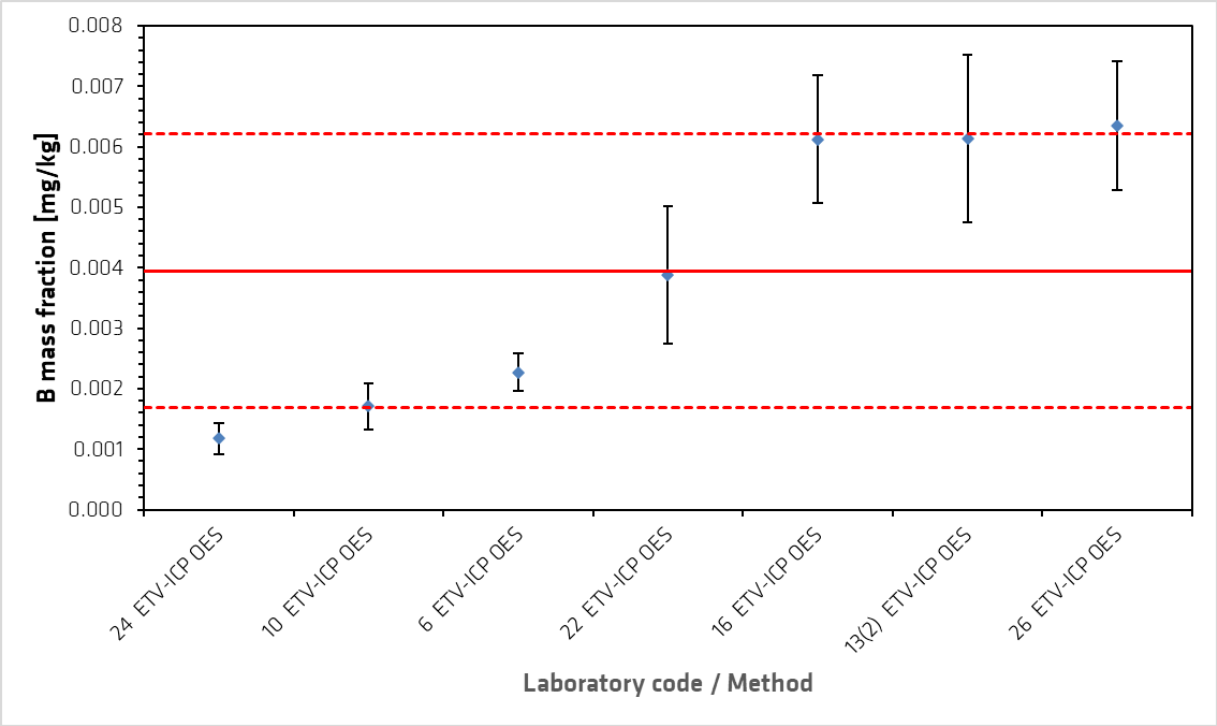

Figure B.4: Accepted laboratory means for B. Uncertainty bars represent the standard deviation of each lab's results. The solid red line represents the informative value (the mean of laboratory means), while the broken red lines give the standard deviation of the laboratories' means.

Table B.5: Individual results for Ba (values in mg/kg)
Data not used for certification is indicated by a grey background.

Lab No.	Lab code	Method	Sub-sample #1	Sub-sample #2	Sub-sample #3	Sub-sample #4	Sub-sample #5	Sub-sample #6	**Mean**	s_i
L1	13(3)	ETV-ICP-OES	0.00104	0.00100	0.00100	0.00184	0.00122	0.00110	**0.00120**	0.00033
L2	6	ETV-ICP-OES	0.00158	0.00114	0.00140	0.00126	0.00110	0.00117	**0.00128**	0.00018
L3 [1)]	11	ETV-ICP-OES	< 0.002	< 0.002	< 0.002	< 0.002	< 0.002	< 0.002	**< 0.002**	
L4	13(1)	ETV-ICP-OES	0.00228	0.00235	0.00131	0.00230	0.00189	0.00204	**0.00203**	0.00039
L5	16	ETV-ICP-OES	0.00191	0.00240	0.00193	0.00226	0.00232	0.00220	**0.00217**	0.00020
L6	22	ETV-ICP-OES	0.00300	0.00270	0.00280	0.00180	0.00150	0.00210	**0.00232**	0.00060
L7	26	ETV-ICP-OES	0.00301	0.00178	0.00261	0.00243	0.00176	0.00295	**0.00242**	0.00055
L8	10	ETV-ICP-OES	0.00390	0.00251	0.00268	0.00232	0.00182	0.00276	**0.00266**	0.00069
L9	13(2)	ETV-ICP-OES	0.00312	0.00229	0.00353	0.00292	0.00321	0.00173	**0.00280**	0.00067
L10	24	ETV-ICP-OES	0.00349	0.00400	0.00274	0.00265	0.00355	0.00379	**0.00337**	0.00055
L11 [3)]	31	ETV-ICP-OES	0.00644	0.00762	0.00781	0.00835			**0.00756**	0.00081
L12 [2)]	27	INAA	0.14200	0.14400	<0.036	0.10200	0.09400	0.07700	**0.11180**	0.02989
L13 [1)]	9	K_0-INAA	<0.18	< 0.21	< 0.24	< 0.27	<0.22	< 0.20	**< 0.27**	

[1)] Data given as below limit of quantification
[2)] Data not accepted on technical grounds
[3)] Outlier, dataset rejected

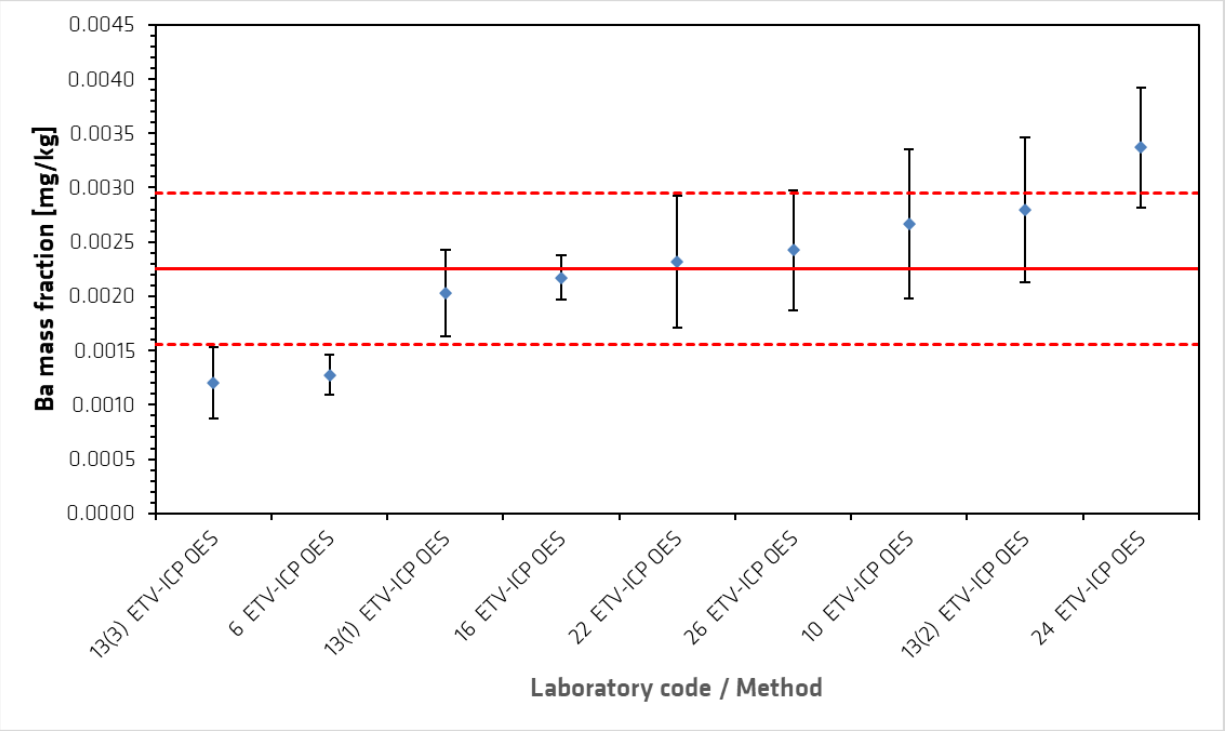

Figure B.5: Accepted laboratory means for Ba. Uncertainty bars represent the standard deviation of each lab's results. The solid red line represents the certified value (the mean of laboratory means), while the broken red lines give the standard deviation of the laboratories' means.

Table B.6: Individual results for Be (values in mg/kg)

Lab No.	Lab code	Method	Sub-sample #1	Sub-sample #2	Sub-sample #3	Sub-sample #4	Sub-sample #5	Sub-sample #6	**Mean**	s_i
L1	13(2)	ETV-ICP-OES	0.00002	0.00001	0.00001	0.00001	0.00001	0.00001	**0.000011**	0.000003
L2	13(1)	ETV-ICP-OES	0.00001	0.00001	0.00001	0.00001	0.00001	0.00001	**0.000012**	0.000002
L3	13(3)	ETV-ICP-OES	0.00001	0.00002	0.00001	0.00002	0.00001	0.00001	**0.000013**	0.000004
L4	6	ETV-ICP-OES	0.00002	0.00001	0.00002	0.00001	0.00001	0.00001	**0.000015**	0.000001
L5	26	ETV-ICP-OES	0.00004	0.00003	0.00002	0.00003	0.00003	0.00002	**0.000029**	0.000007
L6 [4)]	10	ETV-ICP-OES	0.00008	0.00006	0.00007	0.00006	0.00005	0.00009	**0.000067**	0.000014
L7 [4)]	16	ETV-ICP-OES	0.00012	0.00012	0.00008	0.00008	0.00013	0.00012	**0.000109**	0.000023

[4)] Paired Grubbs test outlier (α = 0.01)

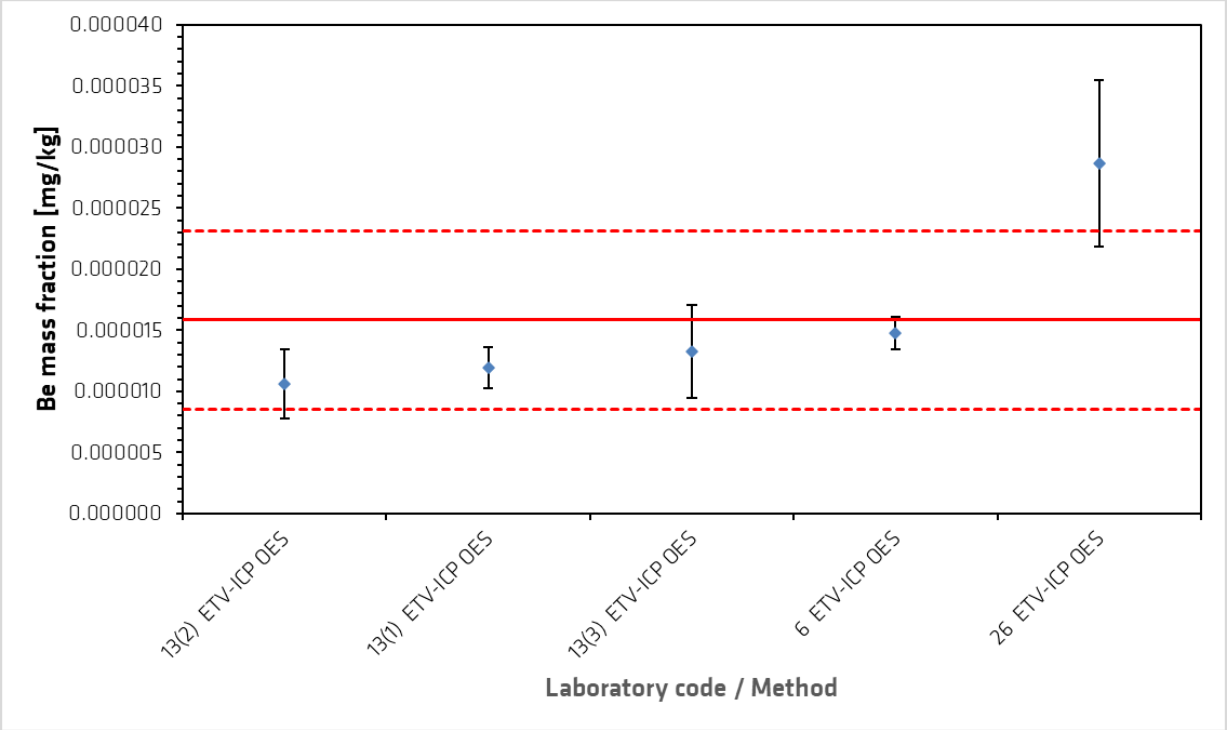

Figure B.6: Accepted laboratory means for Be. Uncertainty bars represent the standard deviation of each lab's results. The solid red line represents the certified value (the mean of laboratory means), while the broken red lines give the standard deviation of the laboratories' means.

Table B.7: Individual results for Ca (values in mg/kg)

Lab No.	Lab code	Method	Sub-sample #1	Sub-sample #2	Sub-sample #3	Sub-sample #4	Sub-sample #5	Sub-sample #6	**Mean**	s_i
L1	11	ETV-ICP-OES	0.0270	0.0330	0.0220	0.0380	0.0230	0.0260	**0.0282**	0.0062
L2	28	SS-ET AAS	0.0272	0.0387	0.0469	0.0280	0.0307	0.0498	**0.0369**	0.0098
L3	6	ETV-ICP-OES	0.0699	0.0350	0.0348	0.0320	0.0418	0.0311	**0.0408**	0.0148
L4	26	ETV-ICP-OES	0.0615	0.0539	0.0241	0.0341	0.0505	0.0394	**0.0439**	0.0139
L5	13(3)	ETV-ICP-OES	0.0517	0.0470	0.0639	0.0467	0.0493	0.0482	**0.0511**	0.0065
L6	10	ETV-ICP-OES	0.0488	0.0415	0.0657	0.0457	0.0665	0.0571	**0.0542**	0.0105
L7	16	ETV-ICP-OES	0.0792	0.0623	0.0572	0.0393	0.0747	0.0861	**0.0665**	0.0171
L8	3	ETV-ICP-OES	0.0601	0.0893	0.0200	0.1031	0.0718	0.0660	**0.0684**	0.0285
L9	31	ETV-ICP-OES	0.0736	0.0615	0.0630	0.0769			**0.0688**	0.0076
L10	13(1)	ETV-ICP-OES	0.0543	0.1053	0.0734	0.0700	0.0934	0.0801	**0.0794**	0.0180
L11	24	ETV-ICP-OES	0.1002	0.0938	0.0709	0.0669	0.0782	0.0968	**0.0845**	0.0143
L12	13(2)	ETV-ICP-OES	0.1046	0.1210	0.1030	0.1012	0.0916	0.1041	**0.1042**	0.0095
L13 [3)]	22	ETV-ICP-OES	0.1384	0.1751	0.2926	0.1971	0.2882	0.2481	**0.2233**	0.0630

[3)] Outlier, dataset rejected

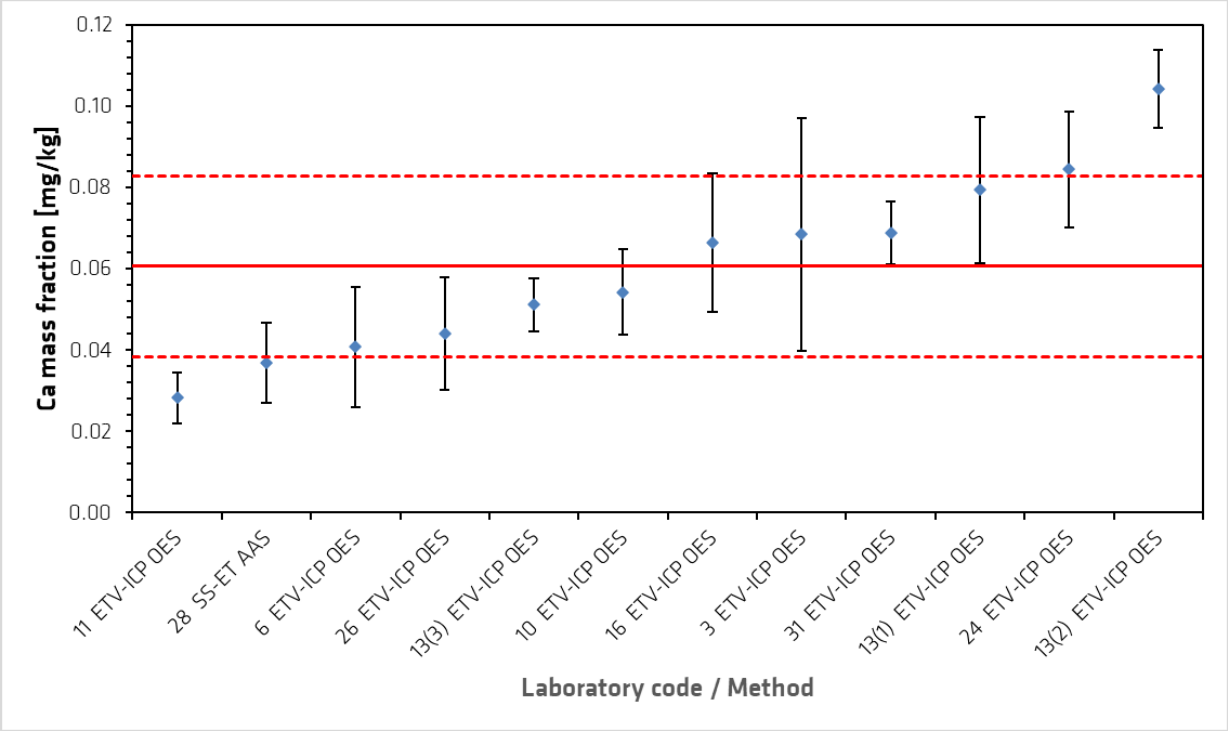

Figure B.7: Accepted laboratory means for Ca. Uncertainty bars represent the standard deviation of each lab's results. The solid red line represents the certified value (the mean of laboratory means), while the broken red lines give the standard deviation of the laboratories' means.

Table B.8: Individual results for Cd (values in mg/kg)
Data not used for certification is indicated by a grey background.

Lab No.	Lab code	Method	Sub-sample #1	Sub-sample #2	Sub-sample #3	Sub-sample #4	Sub-sample #5	Sub-sample #6	**Mean**	s_i
L1	13(3)	ETV-ICP-OES	0.00005	0.00006	0.00006	0.00012	0.00007	0.00006	**0.000071**	0.000027
L2	13(2)	ETV-ICP-OES	0.00006	0.00008	0.00008	0.00006	0.00008	0.00007	**0.000072**	0.000011
L3	6	ETV-ICP-OES	0.00011	0.00011	0.00013	0.00008	0.00006	0.00014	**0.000103**	0.000030
L4	13(1)	ETV-ICP-OES	0.00015	0.00012	0.00009	0.00016	0.00012	0.00011	**0.000122**	0.000026
L5 [1)]	28	SS-ET AAS	< 0.00013	< 0.00013	< 0.00013	< 0.00013	< 0.00013	< 0.00013	**< 0.00013**	
L6	10	ETV-ICP-OES	0.00047	0.00040	0.00018	0.00012	0.00031	0.00014	**0.000270**	0.000144
L7	26	ETV-ICP-OES	0.00036	0.00015	0.00041	0.00028	0.00044	0.00016	**0.000300**	0.000126
L8 [3)]	16	ETV-ICP-OES	0.00161	0.00086	0.00102	0.00168	0.00104	0.00126	**0.001247**	0.000335

[1)] Data given as below limit of quantification
[3)] Outlier, dataset rejected

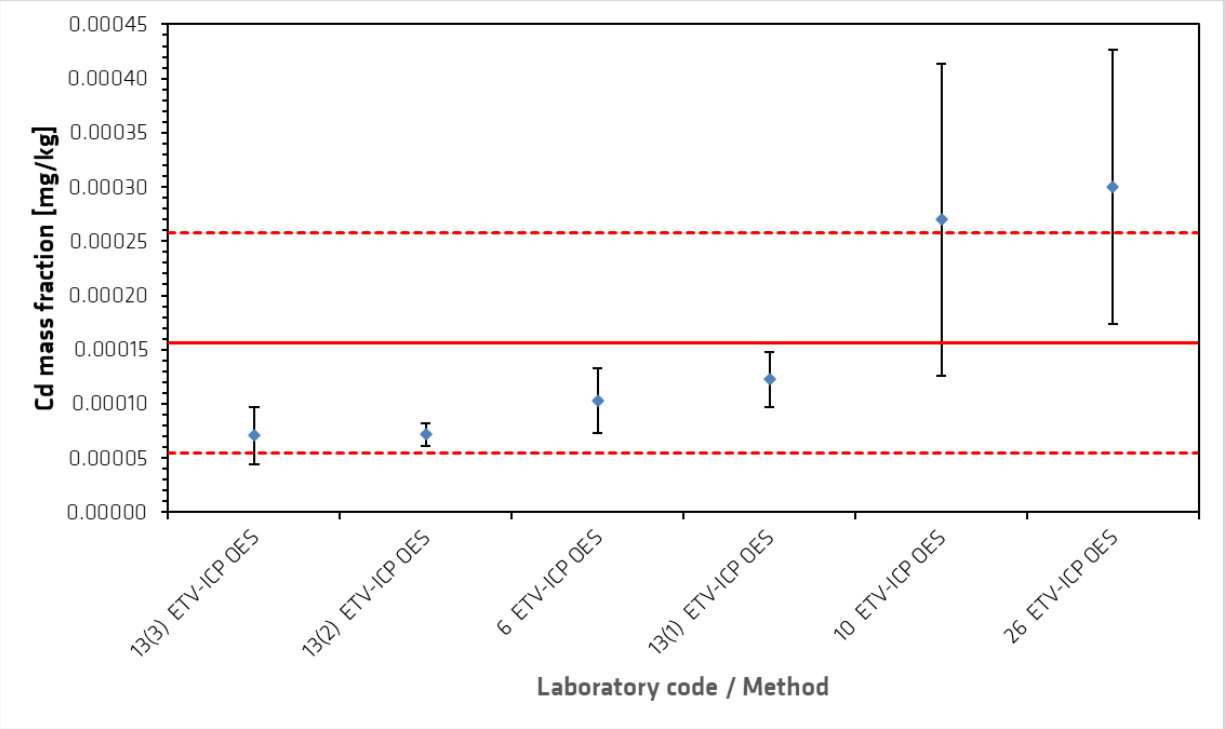

Figure B.8: Accepted laboratory means for Cd. Uncertainty bars represent the standard deviation of each lab's results. The solid red line represents the informative value (the mean of laboratory means), while the broken red lines give the standard deviation of the laboratories' means.

Table B.9: Individual results for Co (values in mg/kg)
Data not used for certification is indicated by a grey background.

Lab No.	Lab code	Method	Sub-sample #1	Sub-sample #2	Sub-sample #3	Sub-sample #4	Sub-sample #5	Sub-sample #6	**Mean**	s_i
L1 [1)]	22	ETV-ICP-OES	0.00030	0.00040	0.00040	0.00030	0.00020	0.00040	**0.00033**	0.00008
L2	13(3)	ETV-ICP-OES	0.00052	0.00037	0.00031	0.00041	0.00029	0.00035	**0.00037**	0.00008
L3	10	ETV-ICP-OES	0.00068	0.00045	0.00043	0.00056	0.00034	0.00067	**0.00052**	0.00014
L4	6	ETV-ICP-OES	0.00062	0.00063	0.00062	0.00055	0.00047	0.00065	**0.00059**	0.00007
L5	13(1)	ETV-ICP-OES	0.00055	0.00063	0.00087	0.00086	0.00053	0.00045	**0.00065**	0.00018
L6	13(2)	ETV-ICP-OES	0.00075	0.00068	0.00076	0.00075	0.00079	0.00045	**0.00070**	0.00013
L7	24	ETV-ICP-OES	0.00027	0.00045	0.00056	0.00121	0.00117	0.00098	**0.00077**	0.00040
L8 [1)]	28	SS-ET AAS	< 0.0015	< 0.0015	< 0.0015	< 0.0015	< 0.0015	< 0.0015	**< 0.0015**	
L9	26	ETV-ICP-OES	0.00253	0.00102	0.00175	0.00193	0.00201	0.00129	**0.00176**	0.00054
L10	16	ETV-ICP-OES	0.00191	0.00130	0.00260	0.00175	0.00150	0.00222	**0.00188**	0.00048
L11 [1)]	11	ETV-ICP-OES	< 0.002	< 0.002	< 0.002	< 0.002	< 0.002	< 0.002	**< 0.002**	
L12 [1)]	9	k_0-INAA	<0.003	< 0.003	< 0.003	< 0.004	< 0.002	< 0.001	**< 0.004**	
L13 [1)]	3	ETV-ICP-OES	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	**<0.005**	
L14 [3)]	27	INAA	0.00480	0.00400	0.00400	0.00560	0.01460	0.00340	**0.00607**	0.00425

[1)] Data given as below limit of quantification
[3)] Outlier, dataset rejected

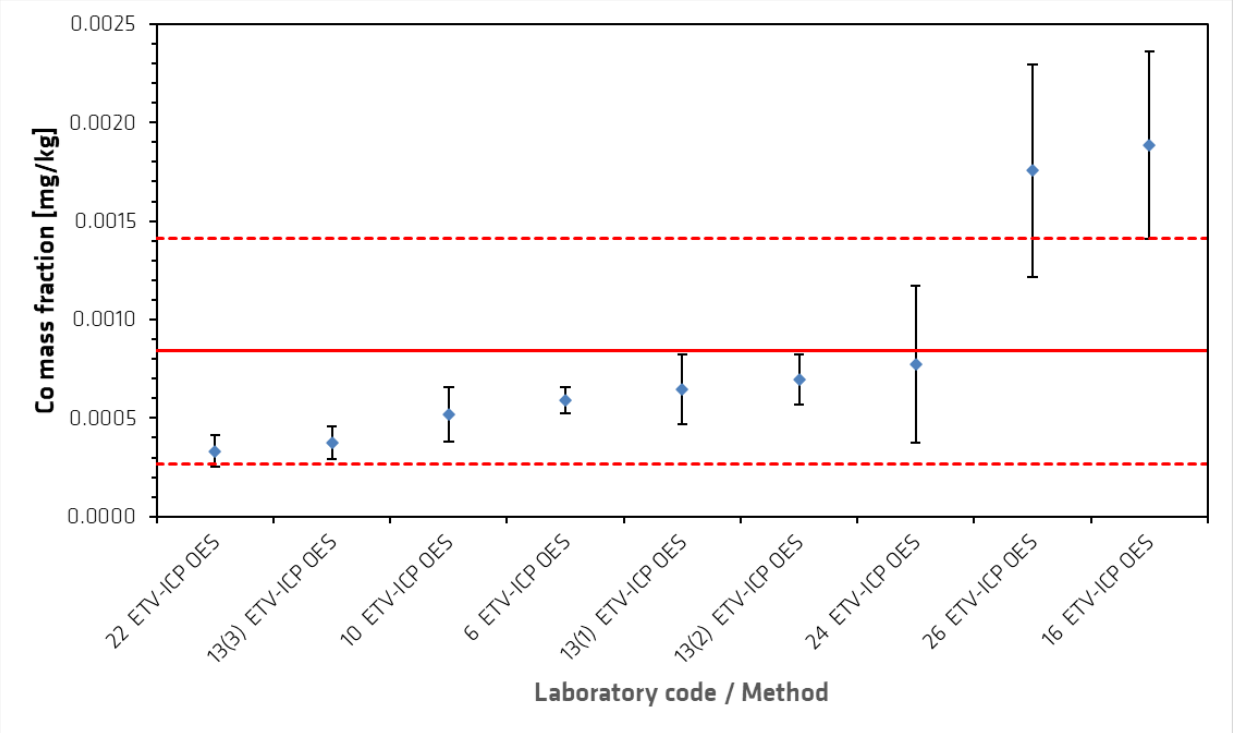

Figure B.9: Accepted laboratory means for Co. Uncertainty bars represent the standard deviation of each lab's results. The solid red line represents the certified value (the mean of laboratory means), while the broken red lines give the standard deviation of the laboratories' means.

Table B.10: Individual results for Cr (values in mg/kg)
Data not used for certification is indicated by a grey background.

Lab No.	Lab code	Method	Sub-sample #1	Sub-sample #2	Sub-sample #3	Sub-sample #4	Sub-sample #5	Sub-sample #6	**Mean**	s_i
L1	26	ETV-ICP-OES	0.00685	0.00333	0.00490	0.01299	0.00373	0.00291	**0.00578**	0.00380
L2	11	ETV-ICP-OES	0.00690	0.00490	0.00700	0.00380	0.00990	0.00530	**0.00630**	0.00215
L3	13(3)	ETV-ICP-OES	0.00618	0.00496	0.00852	0.00629	0.00520	0.00709	**0.00637**	0.00131
L4	16	ETV-ICP-OES	0.00752	0.00645	0.00716	0.00839	0.00858	0.00403	**0.00702**	0.00167
L5	13(2)	ETV-ICP-OES	0.00956	0.00891	0.00764	0.00726	0.00657	0.00628	**0.00770**	0.00130
L6	6	ETV-ICP-OES	0.00700	0.01048	0.00649	0.00831	0.00945	0.00848	**0.00837**	0.00149
L7	13(1)	ETV-ICP-OES	0.01222	0.00519	0.00441	0.00917	0.01009	0.01012	**0.00854**	0.00307
L8	10	ETV-ICP-OES	0.01186	0.00983	0.00567	0.01504	0.00766	0.00501	**0.00918**	0.00385
L9	24	ETV-ICP-OES	0.00697	0.01120	0.01033	0.00904	0.00945	0.00983	**0.00947**	0.00144
L10	28	SS-ET AAS	0.00428	0.00589	0.02112	0.00362	0.02310	0.01023	**0.01137**	0.00865
L11	22	ETV-ICP-OES	0.01200	0.01120	0.01160	0.01280	0.02180	0.01680	**0.01437**	0.00417
L12	27	INAA	0.02850	0.02050	0.01800	0.01060		0.01090	**0.01770**	0.00744
L13	9	k_0-INAA	0.02100	0.03900	0.02100	0.02600	0.01900		**0.02520**	0.00814
L14 [2)]	3	ETV-ICP-OES	<0.005	0.05437	0.00500	<0.005	0.02124	<0.005	**0.02687**	
L15 [3)]	31	ETV-ICP-OES	0.02852	0.03100	0.03153	0.03565			**0.03167**	0.00296

[2)] Data not accepted on technical grounds
[3)] Outlier, dataset rejected

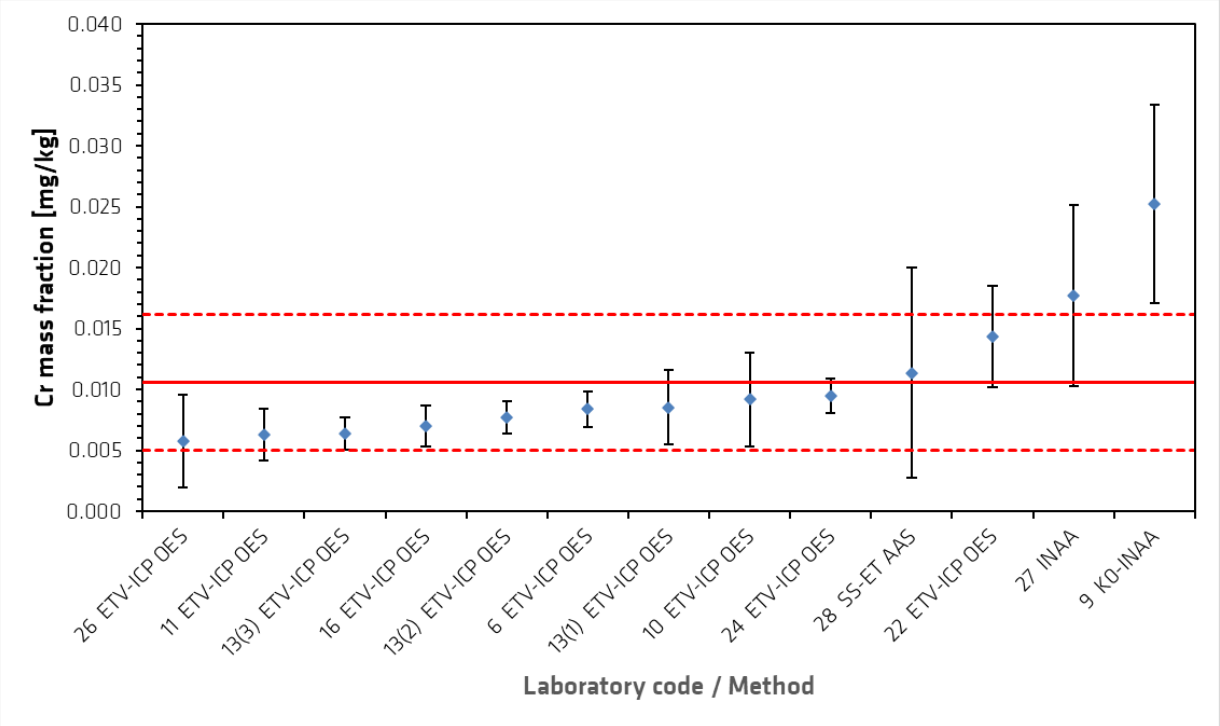

Figure B.10: Accepted laboratory means for Cr. Uncertainty bars represent the standard deviation of each lab's results. The solid red line represents the certified value (the mean of laboratory means), while the broken red lines give the standard deviation of the laboratories' means.

Table B.11: Individual results for Cu (values in mg/kg)
Data not used for certification is indicated by a grey background.

Lab No.	Lab code	Method	Sub-sample #1	Sub-sample #2	Sub-sample #3	Sub-sample #4	Sub-sample #5	Sub-sample #6	**Mean**	s_i
L1	28	SS-ET AAS	0.00143	0.00190	0.00086	0.00122	0.00175	0.00188	**0.00151**	0.00042
L2	10	ETV-ICP-OES	0.00293	0.00391	0.00322	0.00287	0.00195	0.00165	**0.00276**	0.00083
L3	24	ETV-ICP-OES	0.00369	0.00268	0.00408	0.00514	0.00231	0.00208	**0.00333**	0.00118
L4	6	ETV-ICP-OES	0.00328	0.00386	0.00361	0.00291	0.00296	0.00408	**0.00345**	0.00048
L5	13(3)	ETV-ICP-OES	0.00346	0.00393	0.00416	0.00500	0.00329	0.00367	**0.00392**	0.00062
L6	13(1)	ETV-ICP-OES	0.00396	0.00407	0.00529	0.00404	0.00379	0.00451	**0.00428**	0.00055
L7	22	ETV-ICP-OES	0.00410	0.00530	0.00570	0.00390	0.00520	0.00220	**0.00440**	0.00129
L8	26	ETV-ICP-OES	0.00838	0.00536	0.01203	0.00510	0.00946	0.00464	**0.00750**	0.00296
L9	13(2)	ETV-ICP-OES	0.00856	0.00815	0.00833	0.00502	0.00673	0.00870	**0.00758**	0.00144
L10 [1)]	11	ETV-ICP-OES	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	**<0.01**	
L11	16	ETV-ICP-OES	0.01282	0.00737	0.01321	0.00792	0.01170	0.00869	**0.01028**	0.00259
L12 [2)]	31	ETV-ICP-OES	0.03084	<0.009	0.02815	0.02873			**0.02924**	0.00141
L13 [2)]	3	ETV-ICP-OES	0.00759	0.00286	<0.005	<0.005	0.14057	<0.005	**0.05034**	0.07818

[1)] Data given as below limit of quantification
[2)] Data not accepted on technical grounds

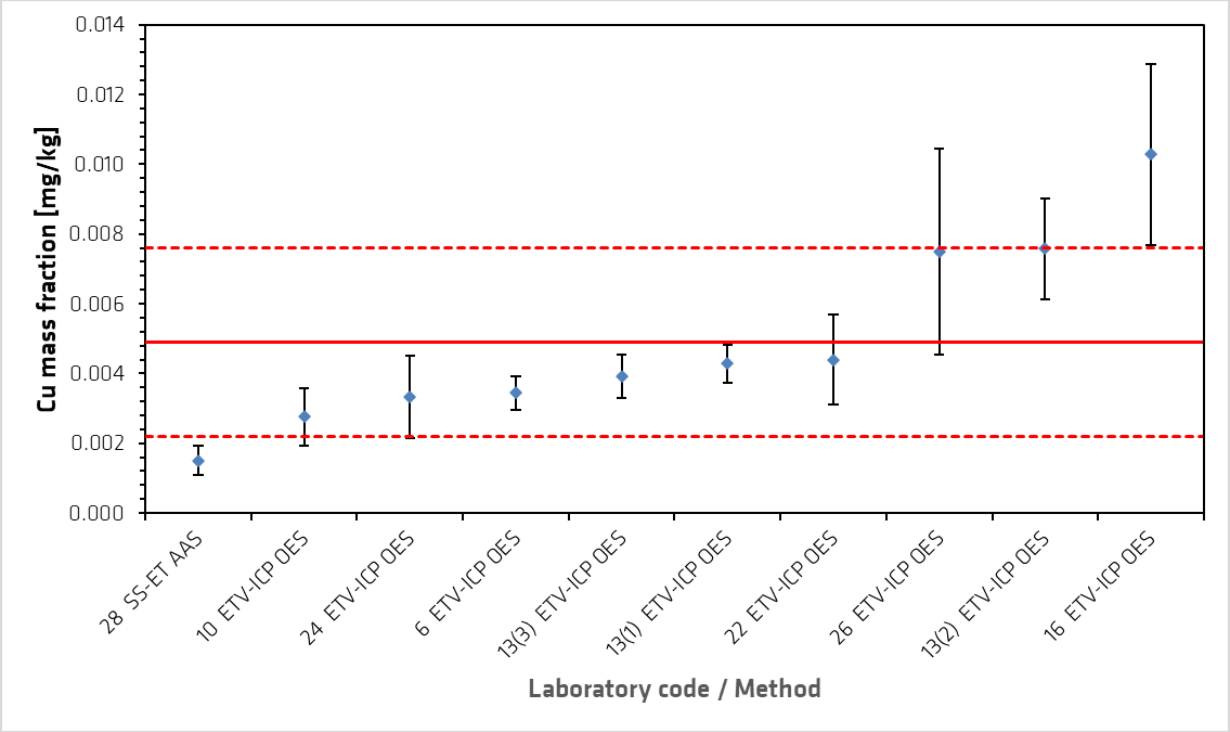

Figure B.11: Accepted laboratory means for Cu. Uncertainty bars represent the standard deviation of each lab's results. The solid red line represents the certified value (the mean of laboratory means), while the broken red lines give the standard deviation of the laboratories' means.

Table B.12: Individual results for Fe (values in mg/kg)
Data not used for certification is indicated by a grey background.

Lab No.	Lab code	Method	Sub-sample #1	Sub-sample #2	Sub-sample #3	Sub-sample #4	Sub-sample #5	Sub-sample #6	**Mean**	s_i
L1	28	SS-ET AAS	0.0592	0.0437	0.0783	0.0640	0.1018	0.0722	**0.0699**	0.0196
L2	24	ETV-ICP-OES	0.2055	0.1050	0.1359	0.1990	0.1818	0.1837	**0.1685**	0.0395
L3	11	ETV-ICP-OES	0.1280	0.2030	0.1990	0.1420	0.1050	0.2620	**0.1732**	0.0585
L4	10	ETV-ICP-OES	0.1921	0.1526	0.1458	0.2067	0.2334	0.2201	**0.1918**	0.0358
L5	6	ETV-ICP-OES	0.2003	0.2499	0.1171	0.1838	0.2045	0.2278	**0.1972**	0.0455
L6	16	ETV-ICP-OES	0.1898	0.2321	0.1757	0.1634	0.2165	0.2206	**0.1997**	0.0274
L7	13(3)	ETV-ICP-OES	0.1489	0.2129	0.2387	0.2151	0.1629	0.2340	**0.2021**	0.0374
L8	13(1)	ETV-ICP-OES	0.1904	0.2570	0.2062	0.2450	0.2071	0.1678	**0.2122**	0.0334
L9	31	ETV-ICP-OES	0.2414	0.2048	0.2071	0.2383			**0.2229**	0.0196
L10	22	ETV-ICP-OES	0.2104	0.3039	0.2643	0.2589	0.2932	0.2604	**0.2652**	0.0327
L11	26	ETV-ICP-OES	0.3008	0.2043	0.3759	0.2206	0.3188	0.3304	**0.2918**	0.0665
L12	13(2)	ETV-ICP-OES	0.3531	0.3317	0.3360	0.2515	0.2650	0.2305	**0.2946**	0.0517
L13 [3)]	3	ETV-ICP-OES	0.2810	0.2503	0.1100	0.0855	0.5701	0.8517	**0.3581**	0.2973
L14 [3)]	27	INAA	0.6600	0.4700	0.4300	0.5000		0.9700	**0.6060**	0.2214

[3)] Outlier, dataset rejected

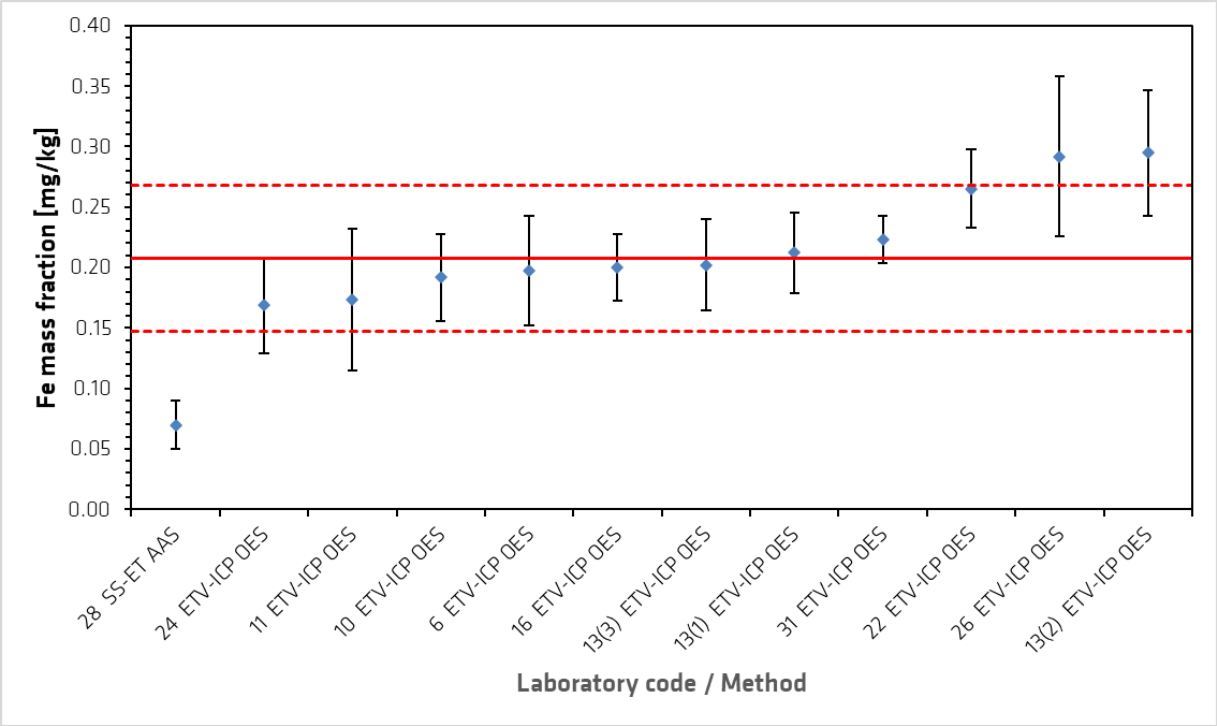

Figure B.12: Accepted laboratory means for Fe. Uncertainty bars represent the standard deviation of each lab's results. The solid red line represents the certified value (the mean of laboratory means), while the broken red lines give the standard deviation of the laboratories' means.

Table B.13: Individual results for K (values in mg/kg)
Data not used for certification is indicated by a grey background.

Lab No.	Lab code	Method	Sub-sample #1	Sub-sample #2	Sub-sample #3	Sub-sample #4	Sub-sample #5	Sub-sample #6	**Mean**	s_i
L1	6	ETV-ICP-OES	0.00904	0.00647	0.00715	0.00882	0.00697	0.01181	**0.00838**	0.00198
L2	28	SS-ET AAS	0.01066	0.01281	0.00697	0.00822	0.00815	0.01308	**0.00998**	0.00259
L3	13(3)	ETV-ICP-OES	0.00913	0.01221	0.01295	0.01134	0.01297	0.00811	**0.01112**	0.00205
L4	13(2)	ETV-ICP-OES	0.01143	0.01866	0.00990	0.02088	0.01222	0.01985	**0.01549**	0.00483
L5	13(1)	ETV-ICP-OES	0.02062	0.02676	0.02310	0.00666	0.01762	0.01486	**0.01827**	0.00704
L6	24	ETV-ICP-OES	0.02227	0.01325	0.01623	0.01649	0.03283	0.01547	**0.01942**	0.00722
L7 [1)]	11	ETV-ICP-OES	< 0.02	< 0.02	< 0.02	< 0.02	< 0.02	< 0.02	**< 0.02**	
L8 [1)]	9	k_0-INAA	<0.19	< 0.20	< 0 23	<0.18	< 0.24	< 0.22	**< 0.24**	
L9 [2)]	3	ETV-ICP-OES	<0.02	<0.02	0.05000	0.03119	0.01000	0.02000	**0.02780**	0.01715
L10	10	ETV-ICP-OES	0.02501	0.03014	0.04941	0.04532	0.00247	0.02266	**0.02917**	0.01700
L11	26	ETV-ICP-OES	0.04679	0.02514	0.02995	0.03045	0.02924		**0.03231**	0.00836
L12	22	ETV-ICP-OES	0.02490	0.09380	0.04220	0.00850	0.08560	0.00930	**0.04405**	0.03753

[1)] Data given as below limit of quantification
[2)] Data not accepted on technical grounds

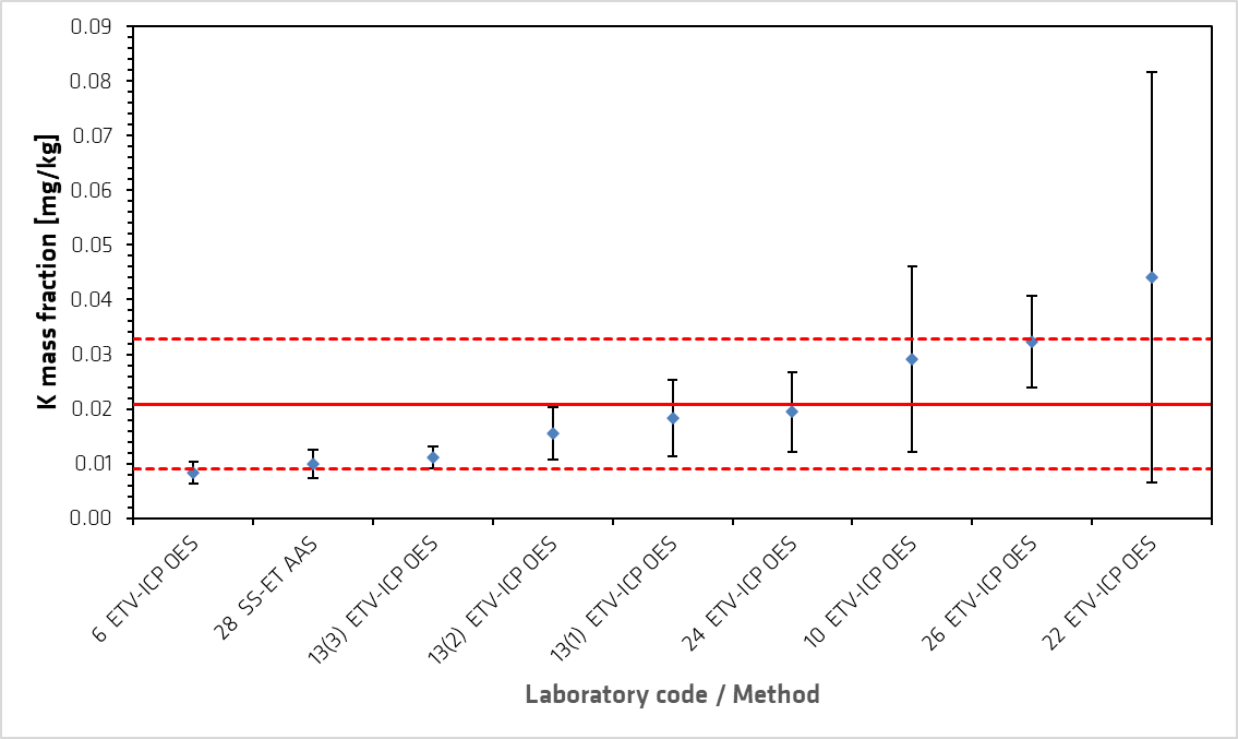

Figure B.13: Accepted laboratory means for K. Uncertainty bars represent the standard deviation of each lab's results. The solid red line represents the certified value (the mean of laboratory means), while the broken red lines give the standard deviation of the laboratories' means.

Table B.14: Individual results for Li (values in mg/kg)
Data not used for certification is indicated by a grey background.

Lab No.	Lab code	Method	Sub-sample #1	Sub-sample #2	Sub-sample #3	Sub-sample #4	Sub-sample #5	Sub-sample #6	**Mean**	s_i
L1	16	ETV-ICP-OES	0.00004	0.00007	0.00005	0.00003	0.00003	0.00003	**0.000041**	0.000017
L2	6	ETV-ICP-OES	0.00005	0.00005	0.00005	0.00006	0.00007	0.00006	**0.000057**	0.000009
L3	24	ETV-ICP-OES	0.00011	0.00005	0.00007	0.00015	0.00005	0.00006	**0.000082**	0.000040
L4	13(2)	ETV-ICP-OES	0.00013	0.00015	0.00011	0.00015	0.00011	0.00013	**0.000130**	0.000018
L5	22	ETV-ICP-OES	0.00020	0.00010	0.00010	0.00030	0.00010	0.00010	**0.000150**	0.000084
L6	10	ETV-ICP-OES	0.00015	0.00015	0.00002	0.00017	0.00044	0.00012	**0.000175**	0.000140
L7	13(3)	ETV-ICP-OES	0.00047	0.00034	0.00049	0.00025	0.00033	0.00030	**0.000363**	0.000095
L8	26	ETV-ICP-OES	0.00052	0.00050	0.00031	0.00037	0.00043	0.00018	**0.000384**	0.000128
L9 [3)]	13(1)	ETV-ICP-OES	0.00097	0.00108	0.00104	0.00052	0.00074	0.00091	**0.000878**	0.000212
L10 [1)]	3	ETV-ICP-OES	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	**<0.005**	

[1)] Data given as below limit of quantification
[3)] Outlier, dataset rejected

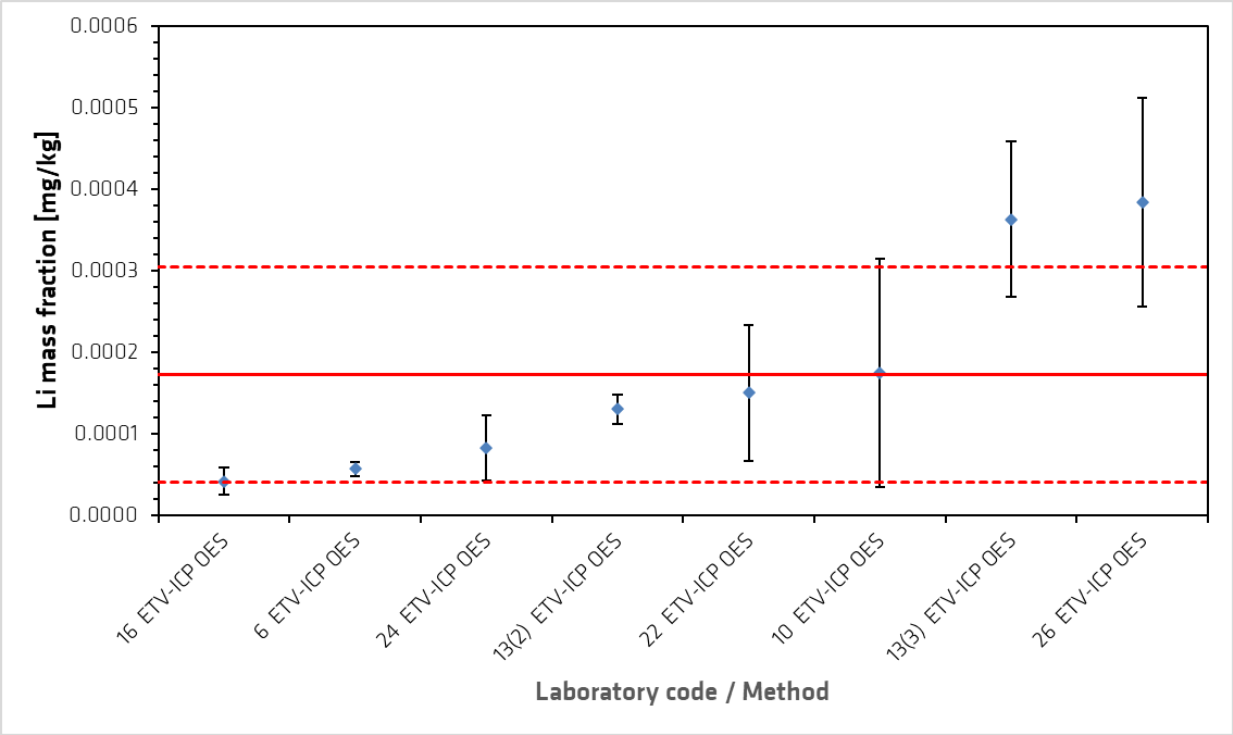

Figure B.14: Accepted laboratory means for Li. Uncertainty bars represent the standard deviation of each lab's results. The solid red line represents the certified value (the mean of laboratory means), while the broken red lines give the standard deviation of the laboratories' means.

Table B.15: Individual results for Mg (values in mg/kg)

Lab No.	Lab code	Method	Sub-sample #1	Sub-sample #2	Sub-sample #3	Sub-sample #4	Sub-sample #5	Sub-sample #6	**Mean**	s_i
L1	6	ETV-ICP-OES	0.00753	0.00987	0.00717	0.01014	0.00883	0.00824	**0.00863**	0.00121
L2	28	SS-ET AAS	0.01020	0.01646	0.00949	0.01225	0.01230	0.01306	**0.01229**	0.00246
L3	24	ETV-ICP-OES	0.01479	0.01789	0.01413	0.01360	0.01524	0.01409	**0.01496**	0.00155
L4	13(3)	ETV-ICP-OES	0.01343	0.01513	0.01803	0.01408	0.01426	0.01612	**0.01517**	0.00168
L5	10	ETV-ICP-OES	0.02027	0.01038	0.01539	0.01666	0.01679	0.01682	**0.01605**	0.00322
L6	11	ETV-ICP-OES	0.01842	0.01692	0.01361	0.02319	0.01838	0.01875	**0.01821**	0.00310
L7	13(1)	ETV-ICP-OES	0.02127	0.02404	0.01522	0.02441	0.02375	0.01448	**0.02053**	0.00454
L8	16	ETV-ICP-OES	0.02163	0.01844	0.01465	0.02396	0.01781	0.02877	**0.02088**	0.00503
L9	26	ETV-ICP-OES	0.01598	0.02285	0.02772	0.02144	0.02557	0.01749	**0.02184**	0.00454
L10	3	ETV-ICP-OES	0.02984	0.02687	0.00500	0.02492	0.01505	0.03036	**0.02201**	0.01001
L11	13(2)	ETV-ICP-OES	0.02296	0.02267	0.02331	0.02940	0.01704	0.01943	**0.02247**	0.00419
L12	22	ETV-ICP-OES	0.02270	0.01570	0.02290	0.02910	0.02670	0.02330	**0.02340**	0.00455
L13	31	ETV-ICP-OES	0.02274	0.02499	0.02296	0.02751			**0.02455**	0.00222

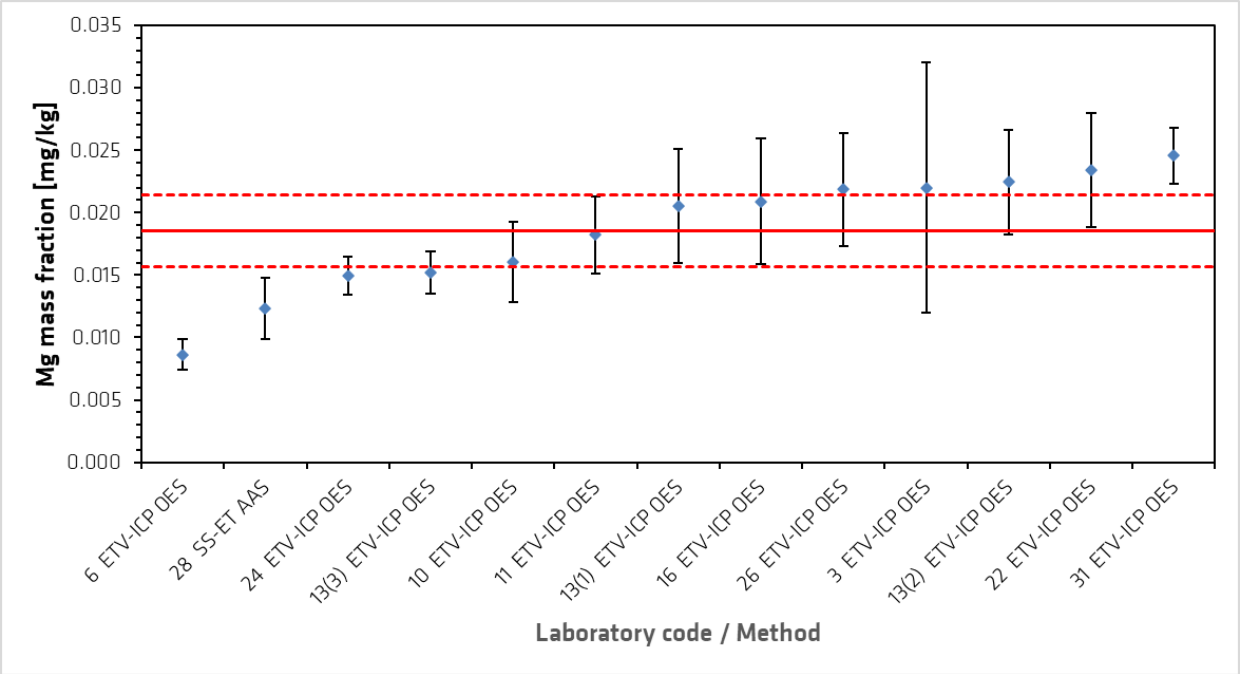

Figure B.15: Accepted laboratory means for Mg. Uncertainty bars represent the standard deviation of each lab's results. The solid red line represents the certified value (the mean of laboratory means), while the broken red lines give the standard deviation of the laboratories' means.

Table B.16: Individual results for Mn (values in mg/kg)
Data not used for certification is indicated by a grey background.

Lab No.	Lab code	Method	Sub-sample #1	Sub-sample #2	Sub-sample #3	Sub-sample #4	Sub-sample #5	Sub-sample #6	**Mean**	s_i
L1	6	ETV-ICP-OES	0.00230	0.00129	0.00145	0.00114	0.00121	0.00182	**0.00154**	0.00045
L2	24	ETV-ICP-OES	0.00178	0.00159	0.00175	0.00281	0.00209	0.00240	**0.00207**	0.00046
L3	13(2)	ETV-ICP-OES	0.00292	0.00255	0.00231	0.00260	0.00255	0.00282	**0.00262**	0.00021
L4	10	ETV-ICP-OES	0.00290	0.00232	0.00273	0.00259	0.00374	0.00178	**0.00268**	0.00065
L5	13(3)	ETV-ICP-OES	0.00226	0.00352	0.00266	0.00320	0.00301	0.00204	**0.00278**	0.00057
L6	16	ETV-ICP-OES	0.00301	0.00243	0.00242	0.00372	0.00272	0.00267	**0.00283**	0.00049
L7 [1)]	11	ETV-ICP-OES	< 0.003	< 0.003	< 0.003	< 0.003	< 0.003	< 0.003	**< 0.003**	
L8	28	SS-ET AAS	0.00307	0.00210	0.00451	0.00537	0.00329	0.00158	**0.00332**	0.00143
L9	22	ETV-ICP-OES	0.00290	0.00420	0.00360	0.00410	0.00250	0.00300	**0.00338**	0.00069
L10	26	ETV-ICP-OES	0.00422	0.00260	0.00435	0.00469	0.00491	0.00293	**0.00395**	0.00095
L11	13(1)	ETV-ICP-OES	0.00466	0.00322	0.00296	0.00467	0.00455	0.00529	**0.00422**	0.00092
L12 [2)]	3	ETV-ICP-OES	<0.005	0.01176	<0.005	<0.005	0.01089	0.00534	**0.00933**	0.00349
L13 [3)]	31	ETV-ICP-OES	0.01374	0.01861	0.01313	0.01380			**0.01482**	0.00255

[1)] Data given as below limit of quantification
[2)] Data not accepted on technical grounds
[3)] Outlier, dataset rejected

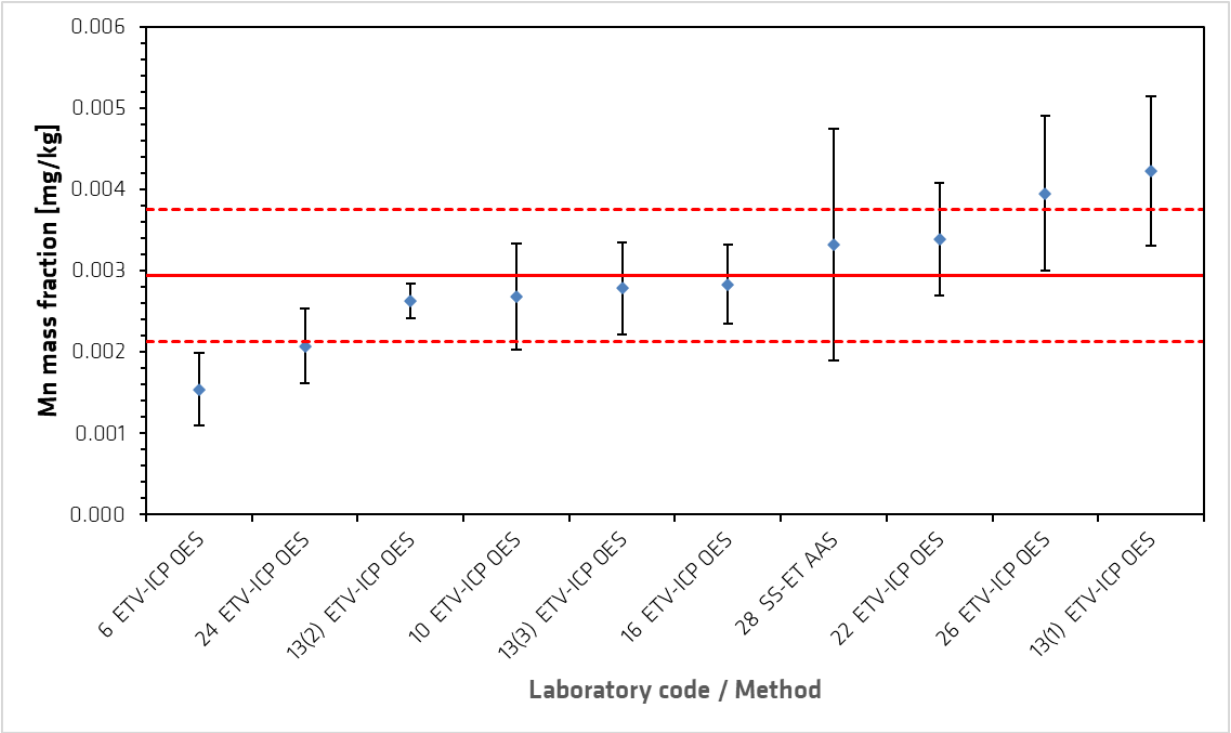

Figure B.16: Accepted laboratory means for Mn. Uncertainty bars represent the standard deviation of each lab's results. The solid red line represents the certified value (the mean of laboratory means), while the broken red lines give the standard deviation of the laboratories' means.

Table B.17: Individual results for Mo (values in mg/kg)
Data not used for certification is indicated by a grey background.

Lab No.	Lab code	Method	Sub-sample #1	Sub-sample #2	Sub-sample #3	Sub-sample #4	Sub-sample #5	Sub-sample #6	**Mean**	s_i
L1	6	ETV-ICP-OES	0.00253	0.00238	0.00291	0.00218	0.00181	0.00305	**0.00248**	0.00046
L2 [2)]	13(2)	ETV-ICP-OES	0.00279	0.00293	0.00263	0.00326	0.00212	0.00346	**0.00286**	0.00048
L3	10	ETV-ICP-OES	0.00488	0.00535	0.00214	0.00259	0.00412	0.00312	**0.00370**	0.00129
L4	13(1)	ETV-ICP-OES	0.00403	0.00371	0.00368	0.00328	0.00350	0.00497	**0.00386**	0.00060
L5	16	ETV-ICP-OES	0.00233	0.00431	0.00394	0.00552	0.00335	0.00587	**0.00422**	0.00133
L6	26	ETV-ICP-OES	0.00204	0.00659	0.00489	0.00506	0.00796	0.00293	**0.00491**	0.00221
L7	13(3)	ETV-ICP-OES	0.00593	0.00499	0.00437	0.00536	0.00466	0.00523	**0.00509**	0.00055
L8	11	ETV-ICP-OES	0.00460	0.00450	0.00520	0.00510	0.00610	0.00560	**0.00518**	0.00060
L9	9	k_0-INAA	< 0.005	< 0.006	< 0.004	< 0.003	< 0.002	< 0.005	**< 0.006**	
L10	3	ETV-ICP-OES	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	**<0.1**	

[1)] Data given as below limit of quantification

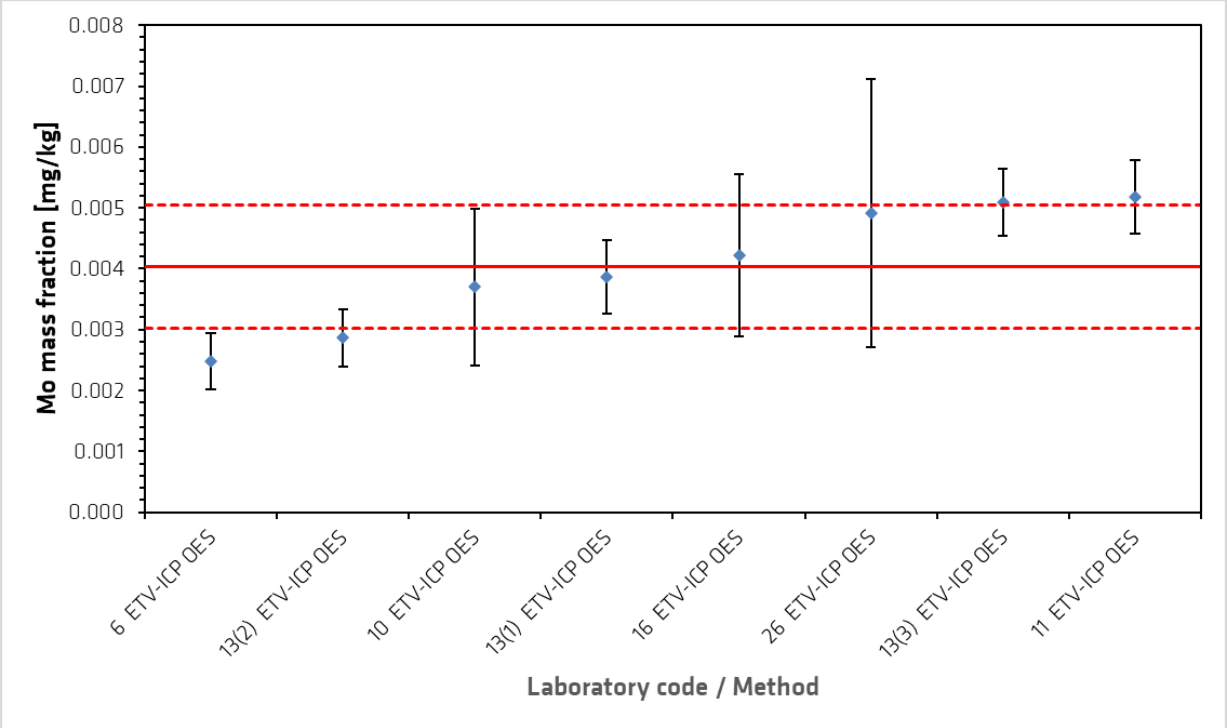

Figure B.17: Accepted laboratory means for Mo. Uncertainty bars represent the standard deviation of each lab's results. The solid red line represents the certified value (the mean of laboratory means), while the broken red lines give the standard deviation of the laboratories' means.

Table B.18: Individual results for Na (values in mg/kg)
Data not used for certification is indicated by a grey background.

Lab No.	Lab code	Method	Sub-sample #1	Sub-sample #2	Sub-sample #3	Sub-sample #4	Sub-sample #5	Sub-sample #6	**Mean**	s_i
L1	6	ETV-ICP-OES	0.01529	0.01593	0.01731	0.02039	0.01833	0.01366	**0.01682**	0.00238
L2	24	ETV-ICP-OES	0.02073	0.02225	0.01364	0.02007	0.02015	0.02212	**0.01983**	0.00317
L3	13(3)	ETV-ICP-OES	0.02094	0.03195	0.02601	0.02841	0.02843	0.01909	**0.02580**	0.00491
L4	28	SS-ET AAS	0.04161	0.02177	0.02549	0.02296	0.02115	0.02340	**0.02606**	0.00776
L5	13(2)	ETV-ICP-OES	0.03412	0.02627	0.02923	0.02592	0.02555	0.02605	**0.02786**	0.00334
L6	10	ETV-ICP-OES	0.03208	0.02958	0.03060	0.03571	0.02688	0.02207	**0.02949**	0.00466
L7	13(1)	ETV-ICP-OES	0.02959	0.02476	0.04607	0.04417	0.03495	0.04054	**0.03668**	0.00842
L8	22	ETV-ICP-OES	0.03970	0.04420	0.02400	0.03900	0.03700	0.03660	**0.03675**	0.00681
L9	9	k_0-INAA	0.03000	0.03900		0.03700	0.03500	0.04500	**0.03720**	0.00550
L10	3	ETV-ICP-OES	0.02288	0.08000	0.12732	0.03328	0.03419	0.06000	**0.05961**	0.03921
L11	26	ETV-ICP-OES	0.05861	0.08789	0.04304	0.07224	0.05092	0.04695	**0.05994**	0.01714
L12	16	ETV-ICP-OES	0.08211	0.06424	0.08835	0.06572	0.05708	0.04995	**0.06791**	0.01468
L13 [3)]	27	INAA	0.11000	0.10400	0.06400	0.09000	0.11200	0.12500	**0.10083**	0.02134

[3)] Outlier, dataset rejected

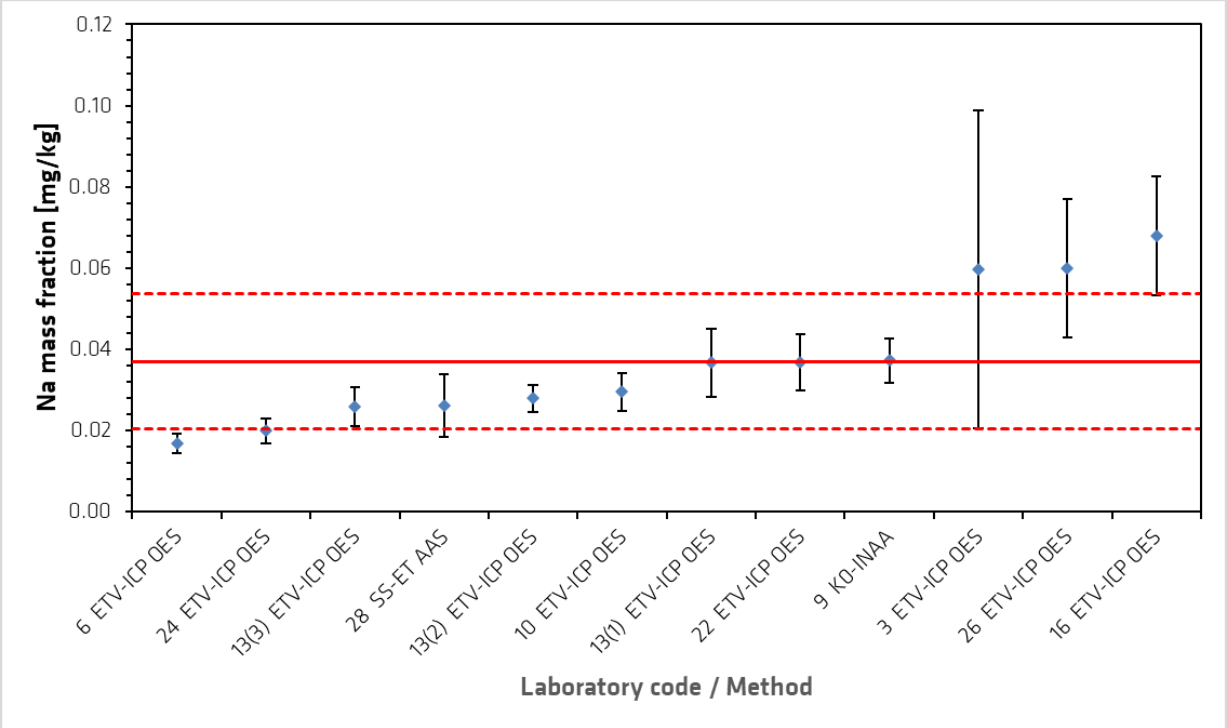

Figure B.18: Accepted laboratory means for Na. Uncertainty bars represent the standard deviation of each lab's results. The solid red line represents the certified value (the mean of laboratory means), while the broken red lines give the standard deviation of the laboratories' means.

Table B.19: Individual results for Ni (values in mg/kg)

Lab No.	Lab code	Method	Sub-sample #1	Sub-sample #2	Sub-sample #3	Sub-sample #4	Sub-sample #5	Sub-sample #6	**Mean**	s_i
L1	10	ETV-ICP-OES	0.07634	0.06508	0.06190	0.06137	0.06235	0.07357	**0.06677**	0.00653
L2	13(1)	ETV-ICP-OES	0.06357	0.08027	0.07516	0.06102	0.06589	0.06786	**0.06896**	0.00733
L3	24	ETV-ICP-OES	0.08775	0.07560	0.07449	0.07261	0.07468	0.07243	**0.07626**	0.00576
L4	6	ETV-ICP-OES	0.08151	0.08082	0.07812	0.06945	0.08566	0.06823	**0.07730**	0.00699
L5	16	ETV-ICP-OES	0.06969	0.09220	0.07441	0.06866	0.08238	0.07923	**0.07776**	0.00884
L6	13(3)	ETV-ICP-OES	0.07996	0.08471	0.07216	0.07605	0.08318	0.07719	**0.07888**	0.00468
L7	13(2)	ETV-ICP-OES	0.08342	0.07446	0.09084	0.07577	0.07788	0.07891	**0.08021**	0.00605
L8	11	ETV-ICP-OES	0.07600	0.07300	0.07700	0.07300	0.08500	0.10500	**0.08150**	0.01232
L9	22	ETV-ICP-OES	0.07510	0.08530	0.07990	0.08410	0.09120	0.09730	**0.08548**	0.00792
L10	26	ETV-ICP-OES	0.08606	0.08251	0.09019	0.10100	0.10865	0.07273	**0.09019**	0.01295
L11	31	ETV-ICP-OES	0.09987	0.10768	0.11190	0.10264			**0.10552**	0.00534
L12	28	SS-ET AAS	0.11302	0.11590	0.11205	0.11776	0.11001	0.11592	**0.11411**	0.00290
L13	3	ETV-ICP-OES	0.09505	0.16406	0.10000	0.10157	0.14739	0.15646	**0.12742**	0.03179

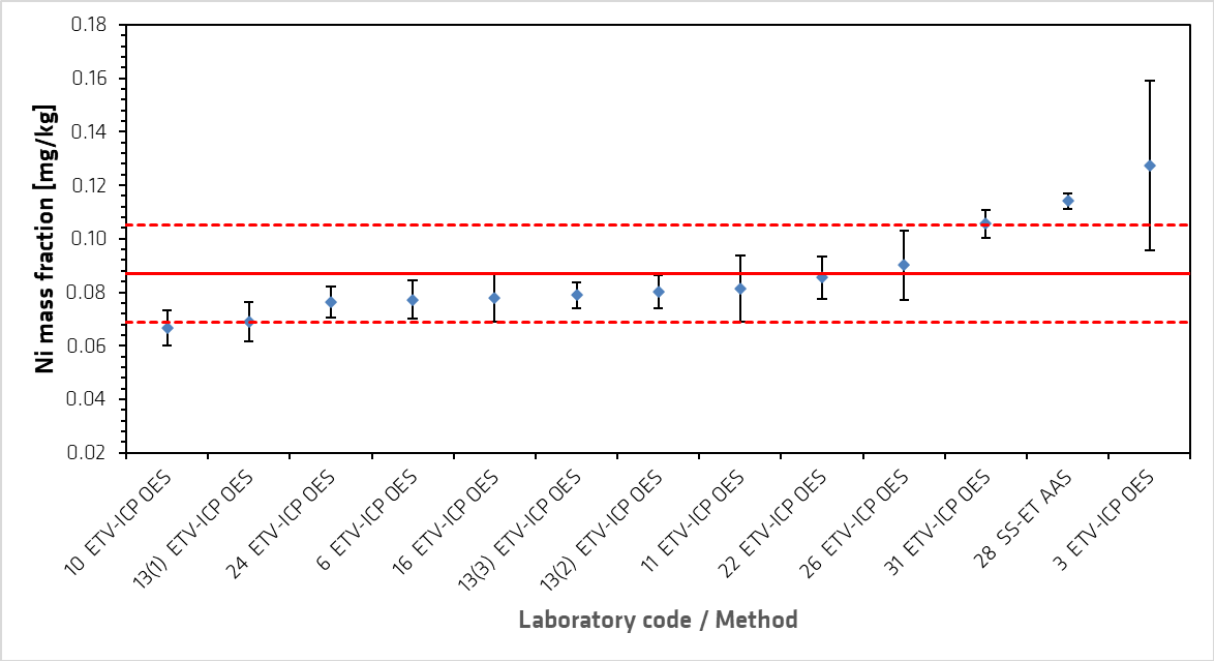

Figure B.19: Accepted laboratory means for Ni. Uncertainty bars represent the standard deviation of each lab's results. The solid red line represents the certified value (the mean of laboratory means), while the broken red lines give the standard deviation of the laboratories' means.

Table B.20: Individual results for P (values in mg/kg)
Data not used for certification is indicated by a grey background.

Lab No.	Lab code	Method	Sub-sample #1	Sub-sample #2	Sub-sample #3	Sub-sample #4	Sub-sample #5	Sub-sample #6	**Mean**	s_i
L1	11	ETV-ICP-OES	0.01080	0.01140	0.01370	0.01320	0.01150	0.01410	**0.01245**	0.00138
L2	10	ETV-ICP-OES	0.01120	0.01774	0.02647	0.01358	0.01565	0.01620	**0.01681**	0.00525
L3	13(3)	ETV-ICP-OES	0.01816	0.02236	0.02298	0.02170	0.01828	0.01730	**0.02013**	0.00249
L4	16	ETV-ICP-OES	0.03037	0.02834	0.03503	0.03357	0.02443	0.05344	**0.03420**	0.01016
L5	13(1)	ETV-ICP-OES	0.03101	0.03836	0.04428	0.03009	0.03322	0.03738	**0.03572**	0.00535
L6	13(2)	ETV-ICP-OES	0.03924	0.02598	0.04541	0.04445	0.03652	0.04629	**0.03965**	0.00771
L7 [3)]	26	ETV-ICP-OES	0.08608	0.07905	0.08726	0.07514	0.06698	0.07114	**0.07761**	0.00810

[3)] Outlier, dataset rejected

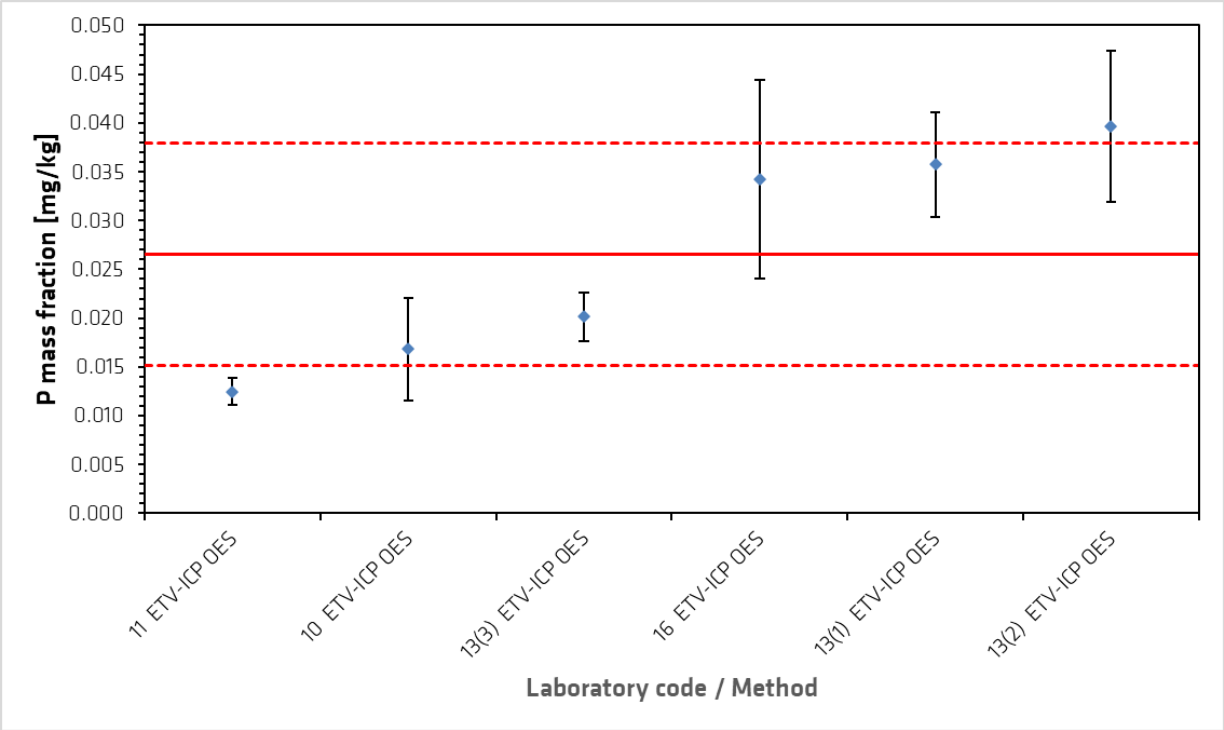

Figure B.20: Accepted laboratory means for P. Uncertainty bars represent the standard deviation of each lab's results. The solid red line represents the certified value (the mean of laboratory means), while the broken red lines give the standard deviation of the laboratories' means.

Table B.21: Individual results for Pb (values in mg/kg)
Data not used for certification is indicated by a grey background.

Lab No.	Lab code	Method	Sub-sample #1	Sub-sample #2	Sub-sample #3	Sub-sample #4	Sub-sample #5	Sub-sample #6	Mean	s_i
L1	6	ETV-ICP-OES	0.00166	0.00086	0.00121	0.00097	0.00121	0.00103	**0.00116**	0.00028
L2	13(3)	ETV-ICP-OES	0.00119	0.00120	0.00159	0.00161	0.00145	0.00070	**0.00129**	0.00034
L3	13(1)	ETV-ICP-OES	0.00165	0.00126	0.00153	0.00133	0.00064	0.00232	**0.00145**	0.00055
L4	24	ETV-ICP-OES	0.00084	0.00092	0.00190	0.00276	0.00222	0.00190	**0.00176**	0.00075
L5 [1)]	28	SS-ET AAS	< 0.0025	< 0.0025	< 0.0025	< 0.0025	< 0.0025	< 0.0025	**< 0.0025**	
L6	10	ETV-ICP-OES	0.00142	0.00435	0.00227	0.00297	0.00287	0.00208	**0.00266**	0.00100
L7	13(2)	ETV-ICP-OES	0.00289	0.00374	0.00425	0.00224	0.00376	0.00465	**0.00359**	0.00088
L8	22	ETV-ICP-OES	0.00630	0.00580	0.00800	0.00460	0.00380	0.00180	**0.00505**	0.00215
L9	26	ETV-ICP-OES	0.00527	0.00322	0.00773	0.00596	0.00482	0.00818	**0.00586**	0.00186
L10 [3)]	16	ETV-ICP-OES	0.00928	0.02270	0.00645	0.00723	0.01612	0.00535	**0.01119**	0.00682
L11 [1)]	3	ETV-ICP-OES	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	**<0.1**	<0.1

[1)] Data given as below limit of quantification
[3)] Outlier, dataset rejected

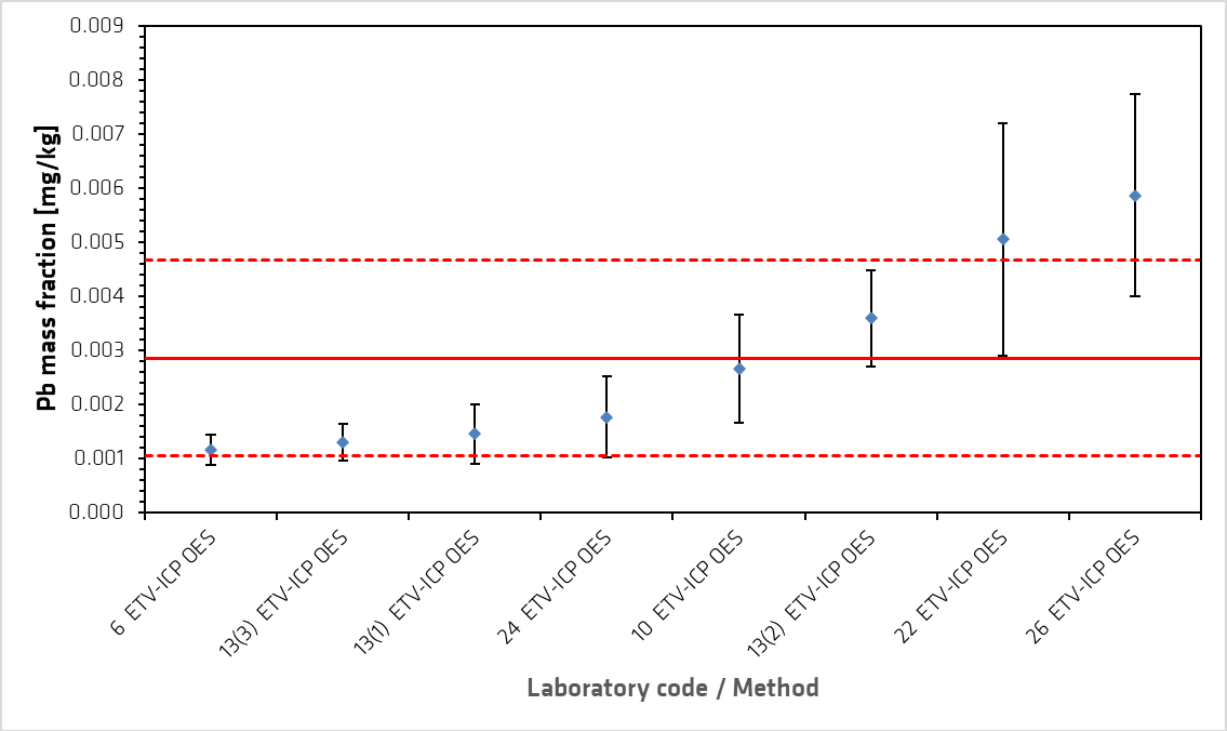

Figure B.21: Accepted laboratory means for Pb. Uncertainty bars represent the standard deviation of each lab's results. The solid red line represents the certified value (the mean of laboratory means), while the broken red lines give the standard deviation of the laboratories' means.

Table B.22: Individual results for S (values in mg/kg)

Lab No.	Lab code	Method	Sub-sample #1	Sub-sample #2	Sub-sample #3	Sub-sample #4	Sub-sample #5	Sub-sample #6	**Mean**	s_i
L1[3)]	10	ETV-ICP-OES	4.285	3.510	3.410	4.347	3.624	2.756	**3.655**	0.594
L2	6	ETV-ICP-OES	4.991	5.171	4.670	4.384	4.633	4.523	**4.729**	0.296
L3	16	ETV-ICP-OES	5.381	5.381	5.310	5.305	5.440	5.396	**5.369**	0.052
L4	31	ETV-ICP-OES	4.802	5.187	5.830	5.716			**5.384**	0.479
L5	13(2)	ETV-ICP-OES	6.462	5.409	5.829	5.117	5.160	6.615	**5.766**	0.652
L6	26	ETV-ICP-OES	6.006	5.917	6.052	5.865	5.968	5.760	**5.928**	0.105
L7	3	ETV-ICP-OES	6.406	7.871	5.200	5.165	5.636	6.586	**6.144**	1.035
L8	13(1)	ETV-ICP-OES	6.350	6.928	5.205	6.204	6.551	5.665	**6.151**	0.622
L9	13(3)	ETV-ICP-OES	6.581	6.975	5.998	7.642	5.014	5.472	**6.280**	0.976

3) Outlier, dataset rejected

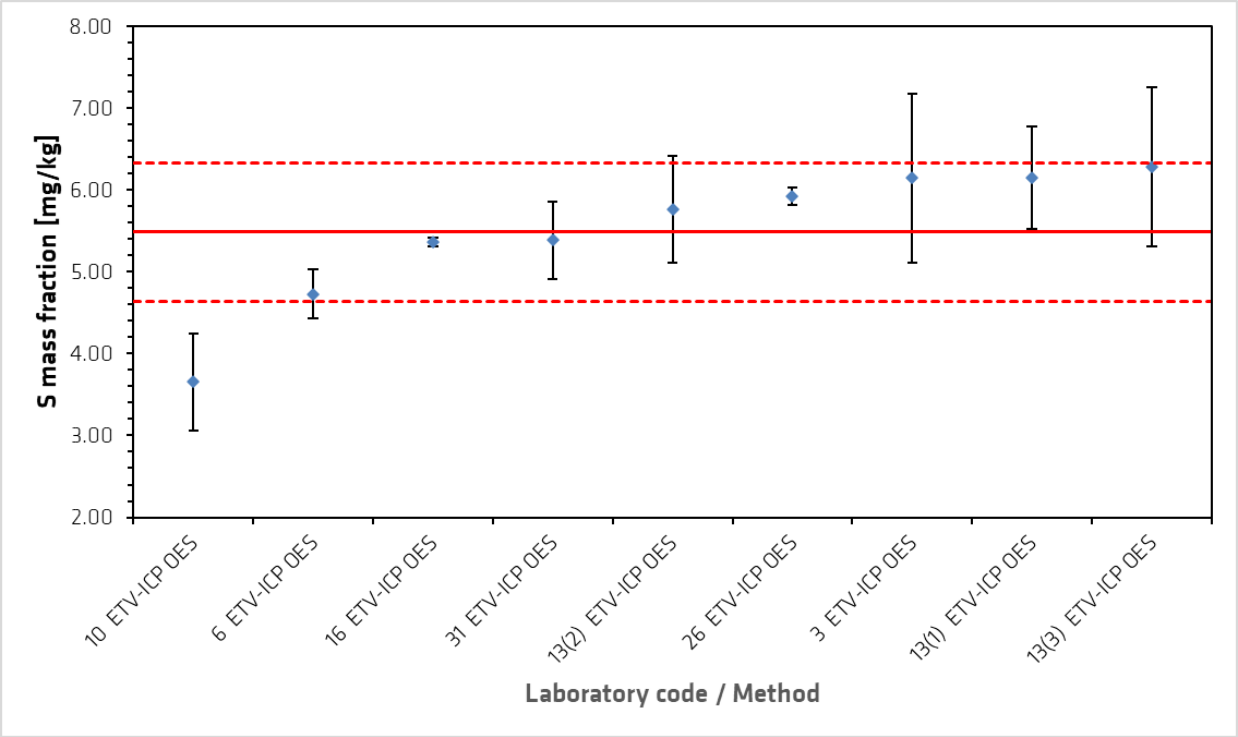

Figure B.22: Accepted laboratory means for S. Uncertainty bars represent the standard deviation of each lab's results. The solid red line represents the certified value (the mean of laboratory means), while the broken red lines give the standard deviation of the laboratories' means.

Table B.23: Individual results for Sb (values in mg/kg)
Data not used for certification is indicated by a grey background.

Lab No.	Lab code	Method	Sub-sample #1	Sub-sample #2	Sub-sample #3	Sub-sample #4	Sub-sample #5	Sub-sample #6	**Mean**	s_i
L1	9	k_0-INAA	0.00081	0.00082	0 00107	0 00100	0.00179		**0.00114**	0.00056
L2	27	INAA	0.00090	0.00590	0.00080	0.00170	0.00130	0.00110	**0.00195**	0.00196
L3	6	ETV-ICP-OES	0.00319	0.00162	0.00316	0.00236	0.00231	0.00186	**0.00242**	0.00065
L4	13(3)	ETV-ICP-OES	0.00693	0.00700	0.00757	0.00514	0.00395	0.00577	**0.00606**	0.00136
L5	13(1)	ETV-ICP-OES	0.00481	0.00894	0.00693	0.00463	0.00858	0.00508	**0.00650**	0.00194
L6	26	ETV-ICP-OES	0.00481	0.00894	0.00693	0.00463	0.00858	0.00508	**0.00650**	0.00194
L7	13(2)	ETV-ICP-OES	0.02210	0.01999	0.01547	0.02173	0.01843	0.02418	**0.02032**	0.00307
L8 [1)]	3	ETV-ICP-OES	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	**<0.1**	

[1)] Data given as below limit of quantification

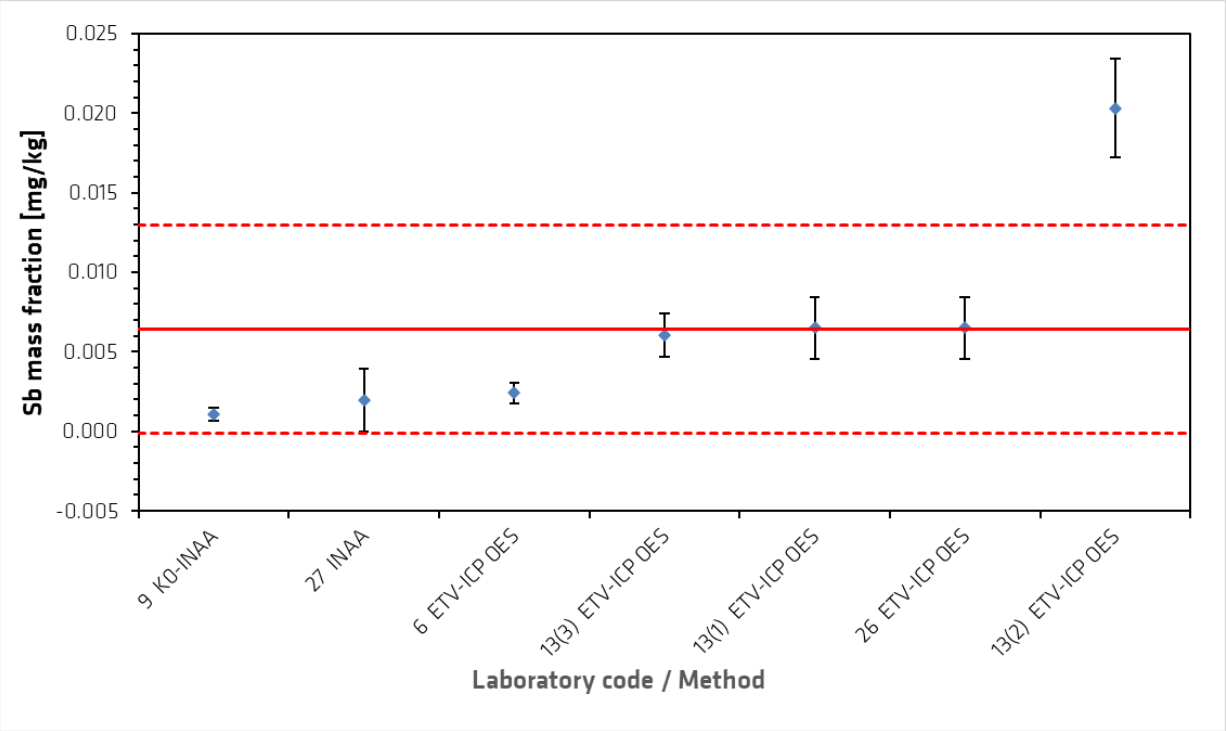

Figure B.23: Accepted laboratory means for Sb. Uncertainty bars represent the standard deviation of each lab's results. The solid red line represents the informative value (the mean of laboratory means), while the broken red lines give the standard deviation of the laboratories' means.

Table B.24: Individual results for Si (values in mg/kg)
Data not used for certification is indicated by a grey background.

Lab No.	Lab code	Method	Sub-sample #1	Sub-sample #2	Sub-sample #3	Sub-sample #4	Sub-sample #5	Sub-sample #6	**Mean**	s_i
L1	28	SS-ET AAS	0.1897	0.1666	0.0963	0.1727	0.1428	0.1598	**0.1547**	0.0325
L2	26	ETV-ICP-OES	0.1527	0.1205	0.1673	0.2456	0.1488	0.1699	**0.1675**	0.0422
L3	11	ETV-ICP-OES	0.1360	0.1490	0.2530	0.2320	0.1770	0.1300	**0.1795**	0.0518
L4	10	ETV-ICP-OES	0.1994	0.2252	0.2471	0.1629	0.2037	0.1708	**0.2015**	0.0319
L5	16	ETV-ICP-OES	0.2804	0.2073	0.2073	0.1849	0.2488	0.1644	**0.2155**	0.0424
L6	13(3)	ETV-ICP-OES	0.2047	0.2478	0.2829	0.2532	0.2304	0.2728	**0.2486**	0.0284
L7	6	ETV-ICP-OES	0.2160	0.2204	0.2712	0.2673	0.2790	0.2438	**0.2496**	0.0270
L8	24	ETV-ICP-OES	0.2787	0.3941	0.2879	0.2994	0.2900	0.2878	**0.3063**	0.0435
L9	13(1)	ETV-ICP-OES	0.4155	0.2480	0.3316	0.3302	0.3559	0.3673	**0.3414**	0.0554
L10	13(2)	ETV-ICP-OES	0.3964	0.4321	0.3931	0.3871	0.5785	0.4534	**0.4401**	0.0726
L11	22	ETV-ICP-OES	0.5206	0.4990	0.4192	0.4335	0.3652	0.4190	**0.4428**	0.0573
L12 [2)]	3	ETV-ICP-OES	<0.5	0.5000	<0.5	<0.5	<0.5	1.1011	**0.8005**	0.4250

[2)] Data not accepted on technical grounds

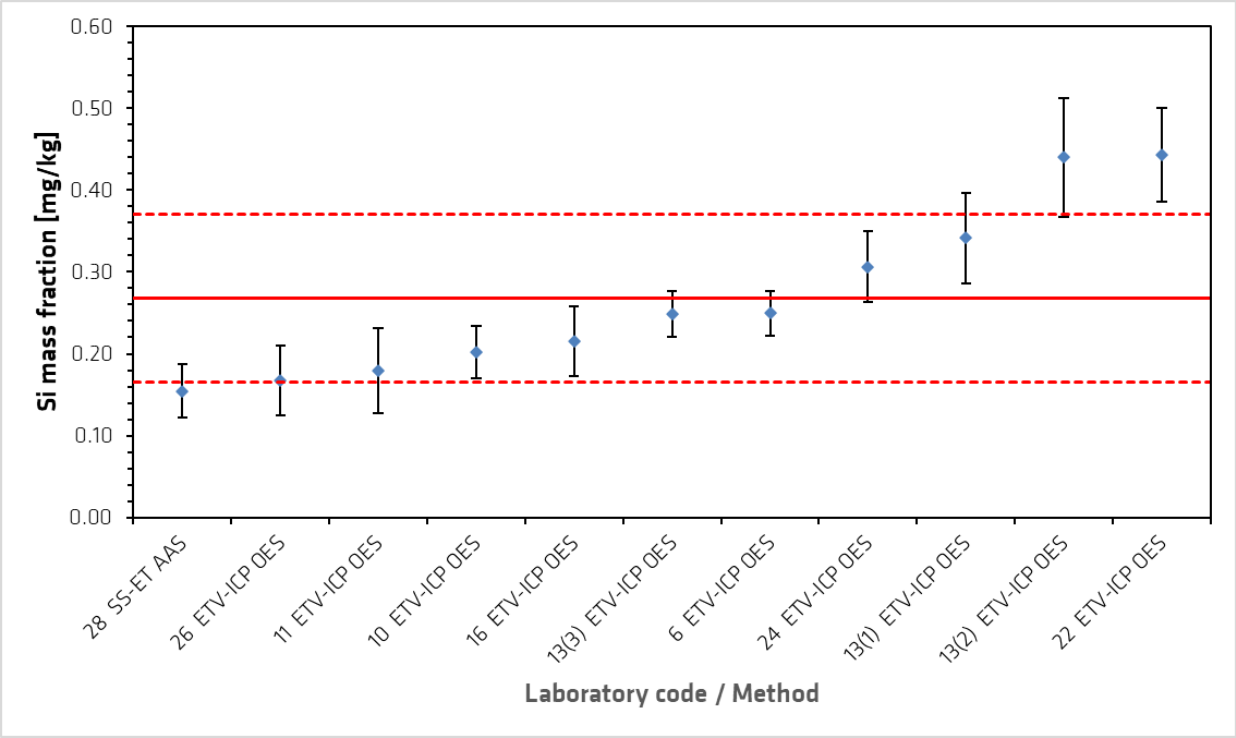

Figure B.24: Accepted laboratory means for Si. Uncertainty bars represent the standard deviation of each lab's results. The solid red line represents the certified value (the mean of laboratory means), while the broken red lines give the standard deviation of the laboratories' means.

Table B.25: Individual results for Sr (values in mg/kg)

Lab No.	Lab code	Method	Sub-sample #1	Sub-sample #2	Sub-sample #3	Sub-sample #4	Sub-sample #5	Sub-sample #6	**Mean**	s_i
L1	13(3)	ETV-ICP-OES	0.00072	0.00041	0.00042	0.00035	0.00040	0.00040	**0.00045**	0.00013
L2	6	ETV-ICP-OES	0.00050	0.00079	0.00041	0.00046	0.00051	0.00090	**0.00060**	0.00020
L3	24	ETV-ICP-OES	0.00052	0.00062	0.00071	0.00043	0.00041	0.00101	**0.00062**	0.00022
L4	13(1)	ETV-ICP-OES	0.00055	0.00091	0.00074	0.00064	0.00079	0.00050	**0.00069**	0.00015
L5	13(2)	ETV-ICP-OES	0.00088	0.00077	0.00071	0.00065	0.00081	0.00111	**0.00082**	0.00016
L6	26	ETV-ICP-OES	0.00150	0.00129	0.00163	0.00174	0.00141	0.00133	**0.00148**	0.00017
L7[1)]	11	ETV-ICP-OES	< 0.002	< 0.002	< 0.002	< 0.002	< 0.002	< 0.002	**< 0.002**	
L8[3)]	22	ETV-ICP-OES	0.00210	0.00250	0.00180	0.00660	0.00210	0.00270	**0.00297**	0.00181

[1)] Data given as below limit of quantification
[3)] Outlier, dataset rejected

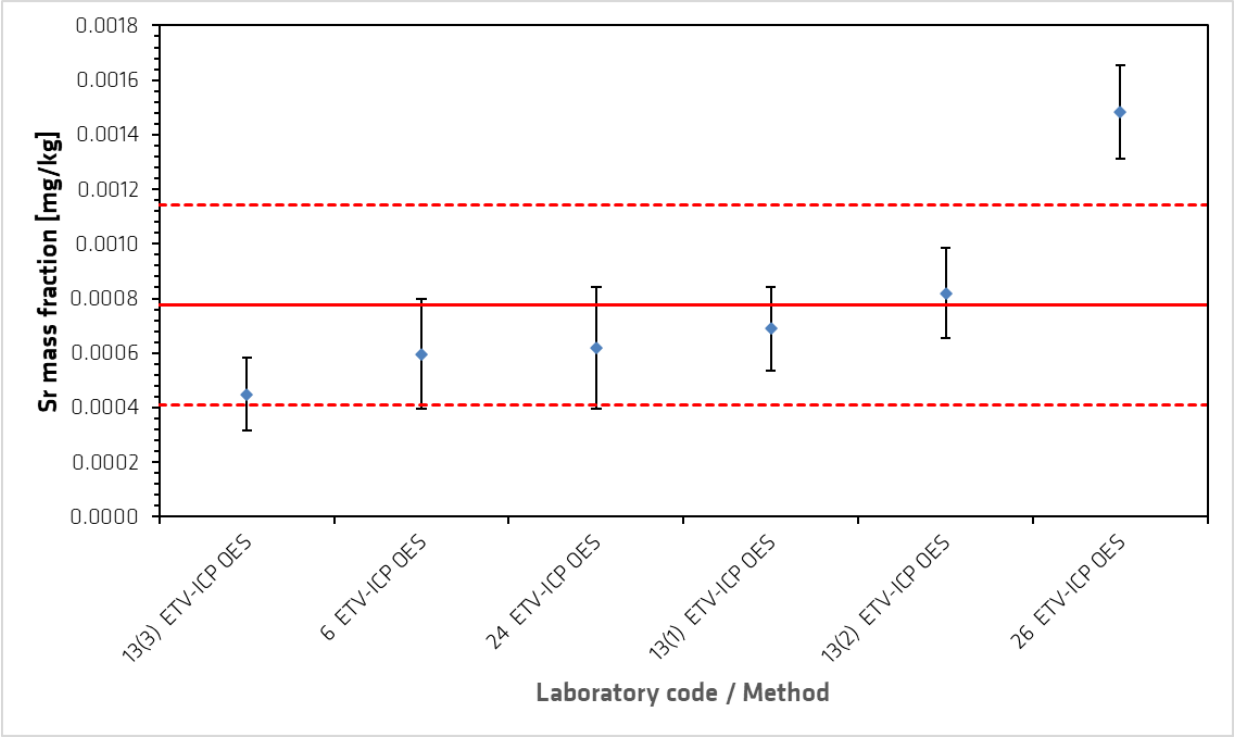

Figure B.25: Accepted laboratory means for Sr. Uncertainty bars represent the standard deviation of each lab's results. The solid red line represents the certified value (the mean of laboratory means), while the broken red lines give the standard deviation of the laboratories' means.

Table B.26: Individual results for Ti (values in mg/kg)
Data not used for certification is indicated by a grey background.

Lab No.	Lab code	Method	Sub-sample #1	Sub-sample #2	Sub-sample #3	Sub-sample #4	Sub-sample #5	Sub-sample #6	**Mean**	s_i
L1 [2)]	10	ETV-ICP-OES	0.00602	0.00219	0.00429	0.00378	0.00573	0.00462	**0.00444**	0.00139
L2 [2)]	11	ETV-ICP-OES	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	**< 0.005**	
L3	6	ETV-ICP-OES	0.00509	0.00576	0.00610	0.00542	0.00384	0.00418	**0.00507**	0.00089
L4	22	ETV-ICP-OES	0.00580	0.00700	0.00540	0.00500	0.00650	0.00550	**0.00587**	0.00075
L5	13(3)	ETV-ICP-OES	0.00776	0.00406	0.00885	0.00520	0.01261	0.00572	**0.00736**	0.00310
L6	13(2)	ETV-ICP-OES	0.00999	0.01002	0.01402	0.00715	0.00474	0.00808	**0.00900**	0.00315
L7	13(1)	ETV-ICP-OES	0.00666	0.00828	0.01609	0.00769	0.01126	0.00690	**0.00948**	0.00364
L8	16	ETV-ICP-OES	0.01178	0.01247	0.00536	0.01129	0.00864	0.00909	**0.00977**	0.00264
L9 [2)]	3	ETV-ICP-OES	<0.005	0.02548	<0.005	0.00755	0.01002	<0.005	**0.01435**	0.00972
L10 [2)]	31	ETV-ICP-OES	<0.009	0.01539	0.01835	0.01616			**0.01663**	0.00154
L11	24	ETV-ICP-OES	0.04494	0.04954	0.01931	0.01234	0.00902	0.00447	**0.02327**	0.01924
L12	26	ETV-ICP-OES	0.02107	0.03315	0.03082	0.02955	0.02027	0.01956	**0.02574**	0.00608

[1)] Data given as below limit of quantification
[2)] Data not accepted on technical grounds

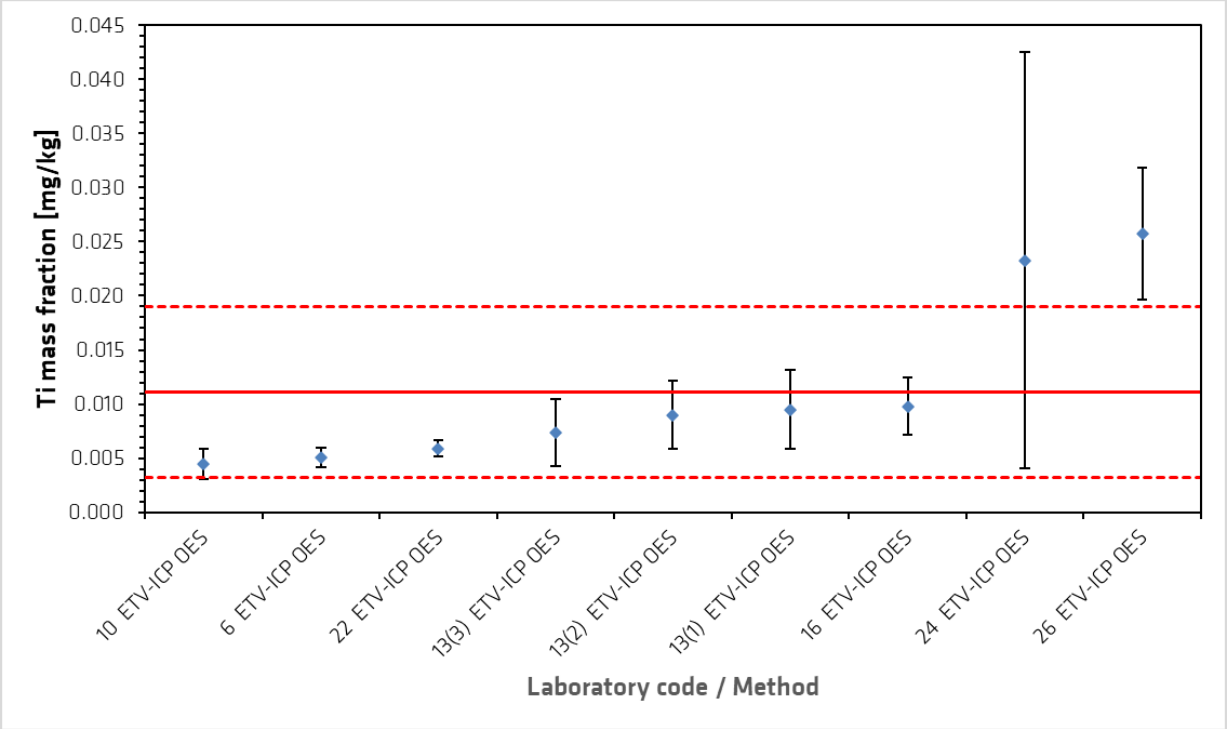

Figure B.26: Accepted laboratory means for Ti. Uncertainty bars represent the standard deviation of each lab's results. The solid red line represents the certified value (the mean of laboratory means), while the broken red lines give the standard deviation of the laboratories' means.

Table B.27: Individual results for V (values in mg/kg)
Data not used for certification is indicated by a grey background.

Lab No.	Lab code	Method	Sub-sample #1	Sub-sample #2	Sub-sample #3	Sub-sample #4	Sub-sample #5	Sub-sample #6	**Mean**	s_i
L1	16	ETV-ICP-OES	0.00120	0.00150	0.00134	0.00343	0.00233	0.00217	**0.00200**	0.00084
L2	13(3)	ETV-ICP-OES	0.00404	0.00274	0.00379	0.00277	0.00222	0.00210	**0.00294**	0.00080
L3	6	ETV-ICP-OES	0.00274	0.00374	0.00287	0.00340	0.00385	0.00336	**0.00333**	0.00045
L4	10	ETV-ICP-OES	0.00444	0.00357	0.00378	0.00364	0.00425	0.00473	**0.00407**	0.00048
L5	13(1)	ETV-ICP-OES	0.00496	0.00459	0.00527	0.00498	0.00493	0.00464	**0.00489**	0.00025
L6	26	ETV-ICP-OES	0.00367	0.00373	0.00599	0.00631	0.00554	0.00418	**0.00490**	0.00118
L7[1)]	11	ETV-ICP-OES	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	**< 0.005**	
L8	13(2)	ETV-ICP-OES	0.00446	0.00376	0.00519	0.00554	0.00608	0.00519	**0.00504**	0.00082
L9	22	ETV-ICP-OES	0.00700	0.00880	0.00670	0.00610	0.00520	0.00660	**0.00673**	0.00119
L10	24	ETV-ICP-OES	0.01127	0.01064	0.01025	0.00911	0.00718	0.00907	**0.00959**	0.00146
L11[2)]	3	ETV-ICP-OES	<0.005	<0.005	<0.005	0.01042	<0.005	<0.005	**0.01042**	
L12[3)]	31	ETV-ICP-OES	0.02309	0.03951	0.02774	0.02332			**0.02842**	0.00770

[1)] Data given as below limit of quantification
[2)] Data not accepted on technical grounds
[3)] Outlier, dataset rejected

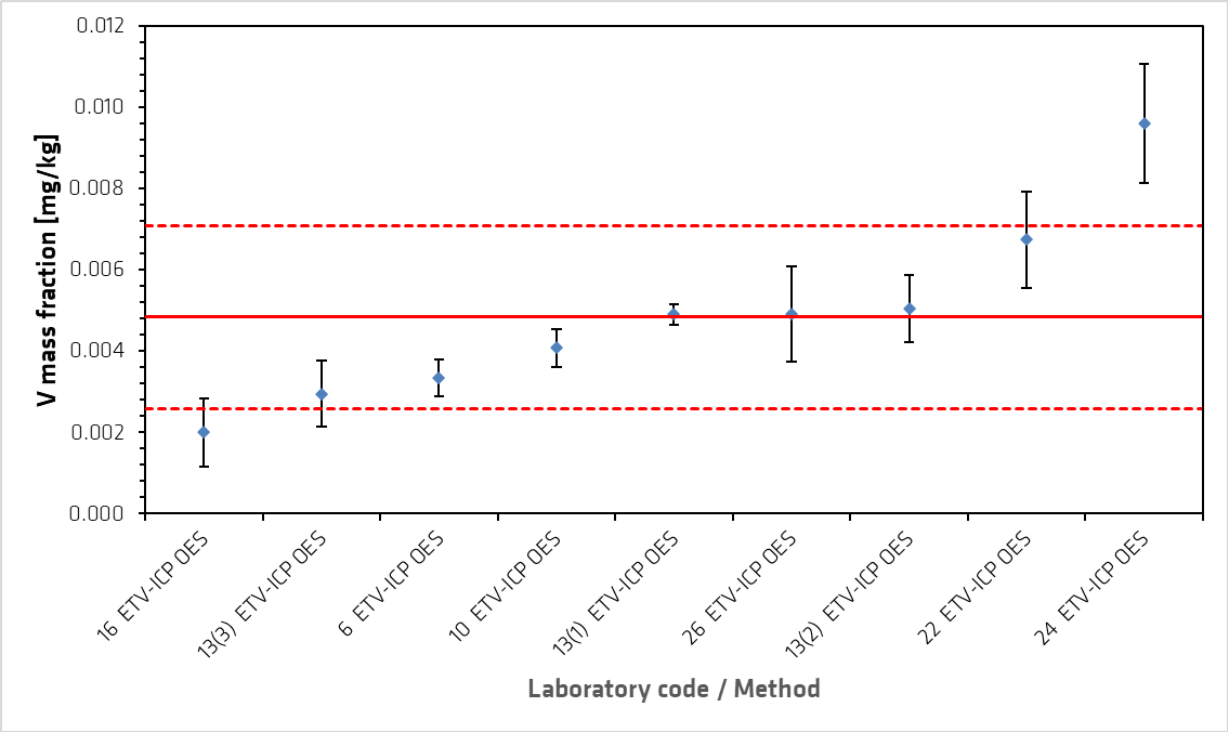

Figure B.27: Accepted laboratory means for V. Uncertainty bars represent the standard deviation of each lab's results. The solid red line represents the certified value (the mean of laboratory means), while the broken red lines give the standard deviation of the laboratories' means.

Table B.28: Individual results for W (values in mg/kg)
Data not used for certification is indicated by a grey background.

Lab No.	Lab code	Method	Sub-sample #1	Sub-sample #2	Sub-sample #3	Sub-sample #4	Sub-sample #5	Sub-sample #6	**Mean**	s_i
L1	9	k_0-NAA	0.00160	0.00200	0.00160	0.00200	0.00150	0.00190	**0.00177**	0.00023
L2	13(1)	ETV-ICP-OES	0.00281	0.00227	0.00362	0.00265	0.00193	0.00235	**0.00261**	0.00059
L3	6	ETV-ICP-OES	0.00302	0.00329	0.00229	0.00234	0.00206	0.00277	**0.00263**	0.00048
L4	13(2)	ETV-ICP-OES	0.00428	0.00320	0.00261	0.00179	0.00336	0.00319	**0.00307**	0.00083
L5	11	ETV-ICP-OES	0.00340	0.00590	0.00260	0.00280	0.00280	0.00850	**0.00433**	0.00238
L6	10	ETV-ICP-OES	0.00440	0.00424	0.00512	0.00596	0.00324	0.00384	**0.00447**	0.00096
L7	13(3)	ETV-ICP-OES	0.00464	0.00663	0.00412	0.00461	0.00385	0.00479	**0.00477**	0.00098
L8	16	ETV-ICP-OES	0.00103	0.00091	0.00235	0.00919	0.00429	0.01144	**0.00487**	0.00445
L9 [1)]	3	ETV-ICP-OES	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	**<0.1**	

[1)] Data given as below limit of quantification

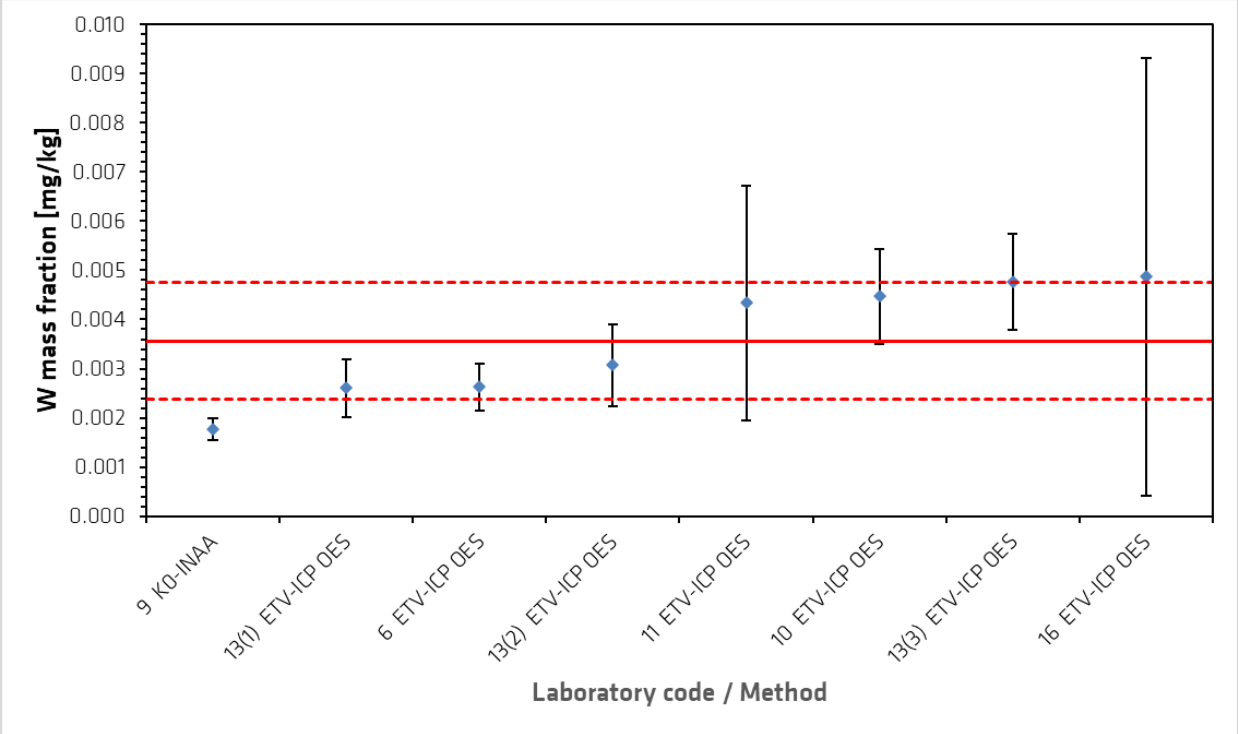

Figure B.28: Accepted laboratory means for W. Uncertainty bars represent the standard deviation of each lab's results. The solid red line represents the certified value (the mean of laboratory means), while the broken red lines give the standard deviation of the laboratories' means.

Table B.29: Individual results for Y(values in mg/kg)
Data not used for certification is indicated by a grey background.

Lab No.	Lab code	Method	Sub-sample #1	Sub-sample #2	Sub-sample #3	Sub-sample #4	Sub-sample #5	Sub-sample #6	**Mean**	s_i
L1	13(1)	ETV-ICP-OES	0.00007	0.00008	0.00007	0.00007	0.00009	0.00007	**0.000075**	0.000009
L2	13(2)	ETV-ICP-OES	0.00015	0.00013	0.00008	0.00011	0.00008	0.00007	**0.000103**	0.000032
L3	13(3)	ETV-ICP-OES	0.00027	0.00015	0.00013	0.00011	0.00009	0.00012	**0.000145**	0.000067
L4	24	ETV-ICP-OES	0.00023	0.00021	0.00023	0.00018	0.00021	0.00012	**0.000197**	0.000042
L5	6	ETV-ICP-OES	0.00034	0.00028	0.00030	0.00028	0.00026	0.00027	**0.000289**	0.000026
L6	26	ETV-ICP-OES	0.00037	0.00029	0.00043	0.00037	0.00027	0.00032	**0.000342**	0.000059
L7 [1)]	22	ETV-ICP-OES	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	**<0.001**	

[1)] Data given as below limit of quantification

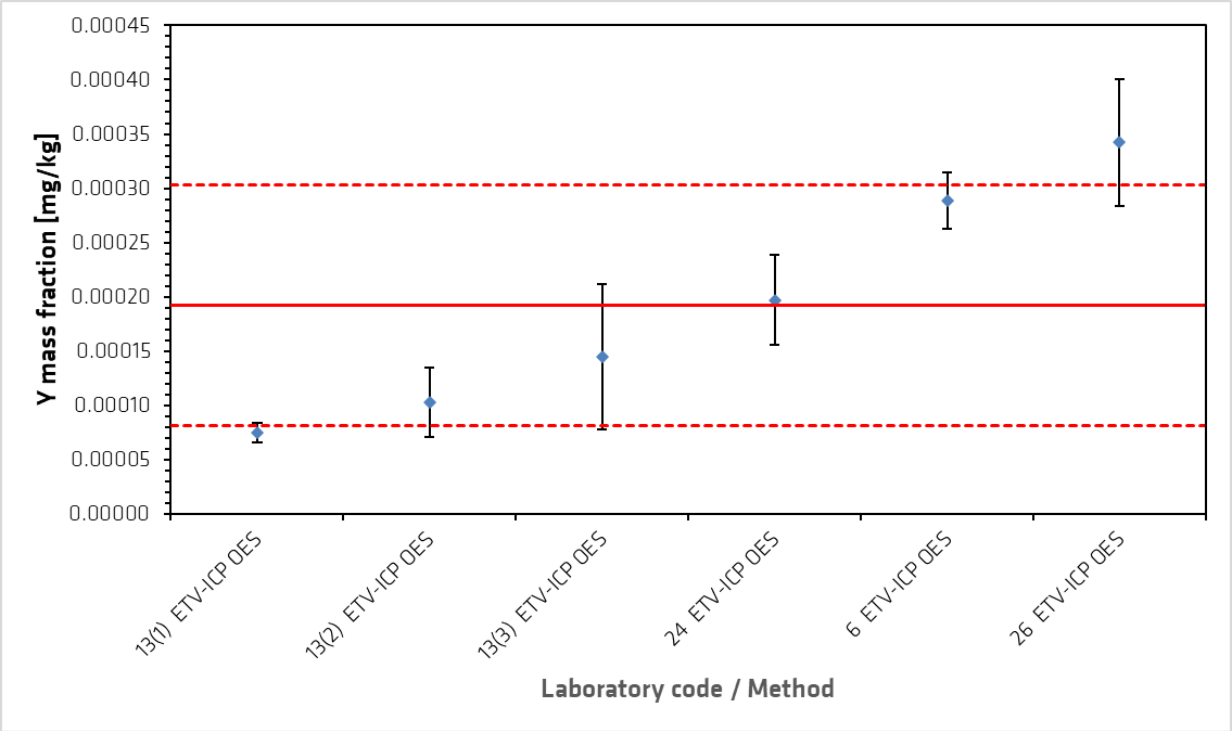

Figure B.29: Accepted laboratory means for Y. Uncertainty bars represent the standard deviation of each lab's results. The solid red line represents the certified value (the mean of laboratory means), while the broken red lines give the standard deviation of the laboratories' means.

Table B.30: Individual results for Zn (values in mg/kg)
Data not used for certification is indicated by a grey background.

Lab No.	Lab code	Method	Sub-sample #1	Sub-sample #2	Sub-sample #3	Sub-sample #4	Sub-sample #5	Sub-sample #6	Mean	s_i
L1	13(2)	ETV-ICP-OES	0.00435	0.00486	0.00529	0.00377	0.00373	0.01355	**0.00593**	0.00379
L2	6	ETV-ICP-OES	0.00656	0.00964	0.00620	0.00556	0.00969	0.00589	**0.00726**	0.00189
L3	11	ETV-ICP-OES	0.00760	0.00630	0.00710		0.00840	0.00880	**0.00764**	0.00100
L4	10	ETV-ICP-OES	0.00923	0.00977	0.00655	0.00892	0.00604	0.00785	**0.00806**	0.00151
L5	13(1)	ETV-ICP-OES	0.00897	0.00987	0.00833	0.00744	0.00843	0.01090	**0.00899**	0.00123
L6	13(3)	ETV-ICP-OES	0.00671	0.01405	0.01265	0.00551	0.01264	0.00663	**0.00970**	0.00380
L7	16	ETV-ICP-OES	0.01241	0.01499	0.00999	0.00744	0.01036	0.00803	**0.01054**	0.00281
L8	22	ETV-ICP-OES	0.00900	0.01860	0.01040	0.00920	0.01060	0.00780	**0.01093**	0.00389
L9	28	SS-ET AAS	0.01159	0.00895	0.00896	0.01018	0.01661	0.01119	**0.01125**	0.00285
L10	3	ETV-ICP-OES	0.01851	0.01594	0.00600	0.01397	0.02693	0.01464	**0.01600**	0.00681
L11	26	ETV-ICP-OES	0.01799	0.02239	0.02395	0.01939	0.02332	0.02234	**0.02156**	0.00235
L12	24	ETV-ICP-OES	0.00982	0.02130	0.00805	0.05430	0.01854	0.05599	**0.02800**	0.02163
L13	27	INAA		0.05300	0.05000	0.02700	0.01900	0.02400	**0.03460**	0.01573
L14 [3)]	31	ETV-ICP-OES	0.04259	0.04733	0.05245	0.05535			**0.04943**	0.00564
L15 [1)]	9	k_0-INAA	< 0.08	< 0.03	< 0.06	< 0.06	<0.04	<0.03	**< 0.08**	

[1)] Data given as below limit of quantification
[3)] Outlier, dataset rejected

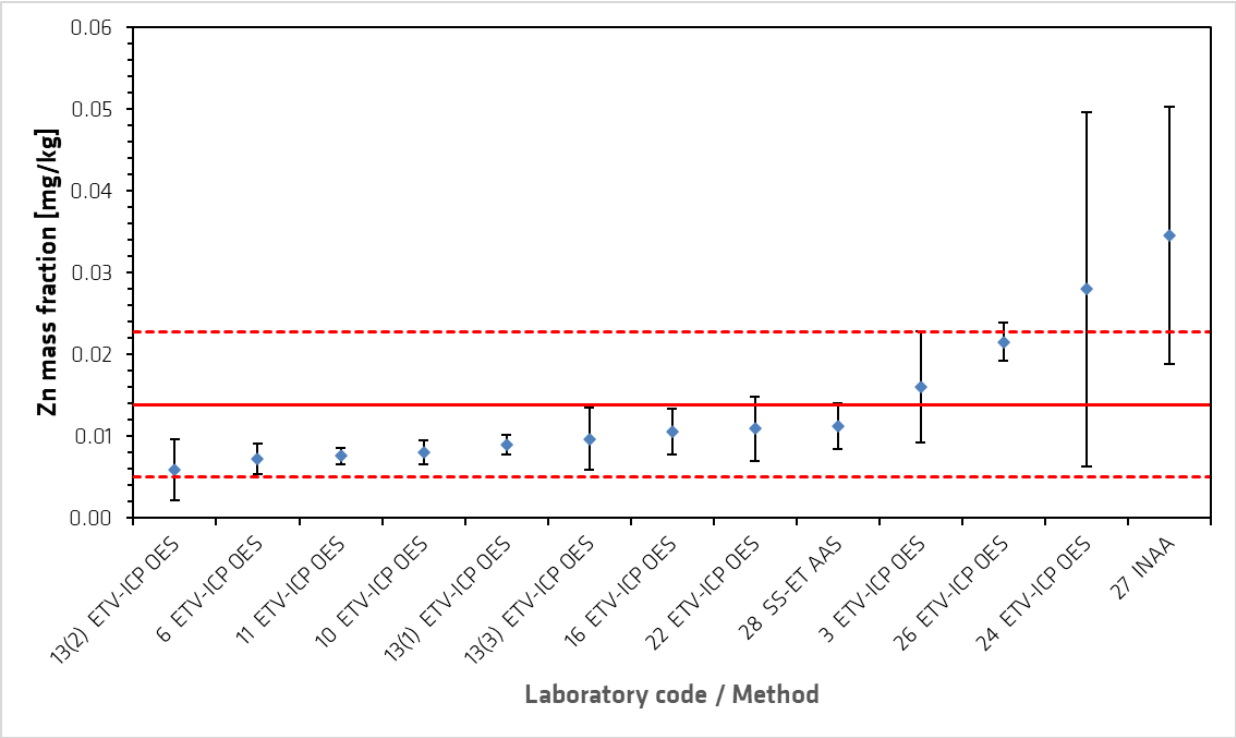

Figure B.30: Accepted laboratory means for Zn. Uncertainty bars represent the standard deviation of each lab's results. The solid red line represents the certified value (the mean of laboratory means), while the broken red lines give the standard deviation of the laboratories' means.

Table B.31: Individual results for Zr (values in mg/kg)
Data not used for certification is indicated by a grey background.

Lab No.	Lab code	Method	Sub-sample #1	Sub-sample #2	Sub-sample #3	Sub-sample #4	Sub-sample #5	Sub-sample #6	**Mean**	s_i
L1	10	ETV-ICP-OES	0.00021	0.00050	0.00044	0.00106	0.00062	0.00058	**0.00057**	0.00028
L2 [1)]	22	ETV-ICP-OES	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	**<0.001**	
L3	24	ETV-ICP-OES	0.00134	0.00085	0.00168	0.00159	0.00046	0.00084	**0.00113**	0.00049
L4	13(1)	ETV-ICP-OES	0.00142	0.00134	0.00157	0.00172	0.00124	0.00164	**0.00149**	0.00019
L5	6	ETV-ICP-OES	0.00130	0.00133	0.00195	0.00153	0.00128	0.00166	**0.00151**	0.00026
L6	13(3)	ETV-ICP-OES	0.00268	0.00184	0.00247	0.00110	0.00142	0.00172	**0.00187**	0.00061
L7	13(2)	ETV-ICP-OES	0.00185	0.00173	0.00231	0.00223	0.00174	0.00200	**0.00198**	0.00025
L8	26	ETV-ICP-OES	0.00234	0.00131	0.00215	0.00202	0.00113	0.00315	**0.00202**	0.00074
L9	16	ETV-ICP-OES	0.00295	0.00170	0.00202	0.00256	0.00197	0.00234	**0.00226**	0.00045
L10 [1)]	11	ETV-ICP-OES	< 0.004	< 0.004	< 0.004	< 0.004	< 0.004	< 0.004	**< 0.004**	
L11 [2)]	3	ETV-ICP-OES	<0.01	<0.01	<0.01	0.02533	0.01319	<0.01	**0.01926**	0.00858
L12 [2)]	31	ETV-ICP-OES	<0.009	0.02822	<0.009	0.02738			**0.02780**	0.00059

[1)] Data given as below limit of quantification
[2)] Data not accepted on technical grounds

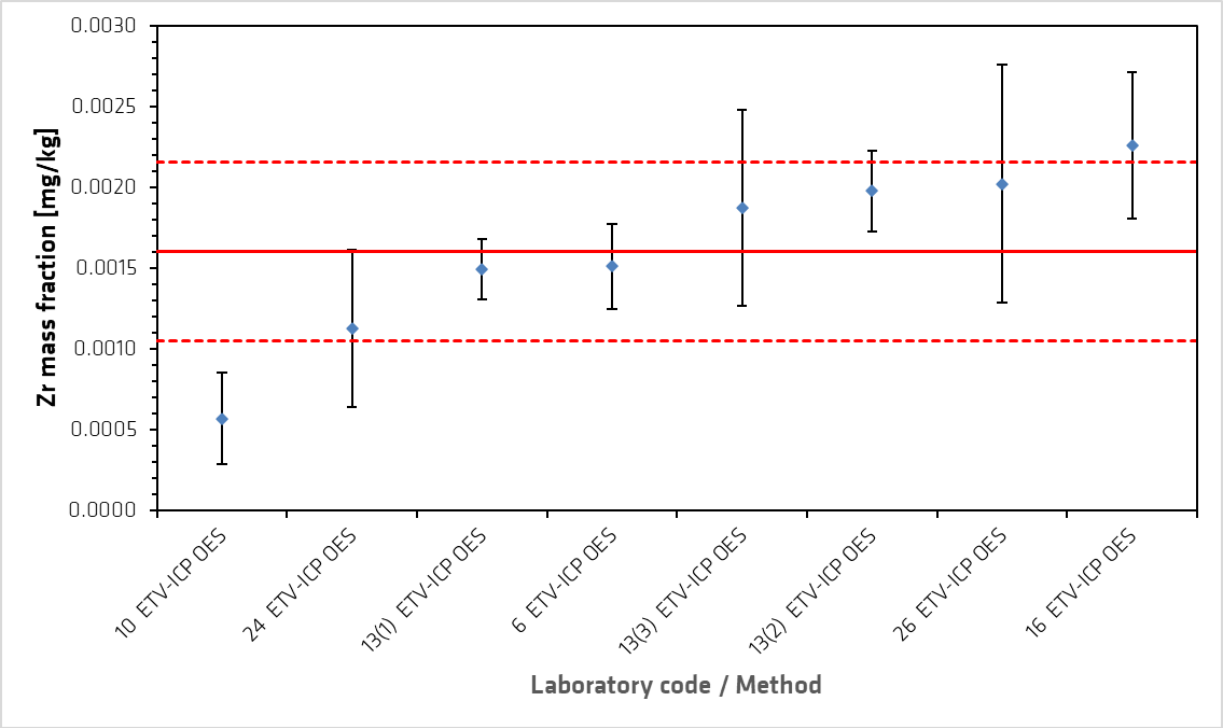

Figure B.31: Accepted laboratory means for Zr. Uncertainty bars represent the standard deviation of each lab's results. The solid red line represents the certified value (the mean of laboratory means), while the broken red lines give the standard deviation of the laboratories' means.

Table B.32: Individual results for additionally reported parameters (values in mg/kg)

Parameter	Lab No.	Lab code	Method	Sub-sample #1	Sub-sample #2	Sub-sample #3	Sub-sample #4	Sub-sample #5	Sub-sample #6	Mean
Au	L1	9	k_0-INAA	< 0.00002	< 0.00002	< 0.00003	< 0.00002	< 0.00002	< 0.00002	**< 0.00003**
Bi	L1	13(1)	ETV-ICP-OES	0.00095	0.00297	0.00250	0.00278	0.00282	0.00184	**0.00231**
	L2	13(2)	ETV-ICP-OES	0.00078	0.00239	0.00159	0.00088	0.00036	0.00156	**0.00126**
Cs	L1	27	INAA	0.0003	0.0002	0.0002	0.0003	<0.0003	<0.0003	**<0.0004**
	L2	9	k_0-INAA	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002	**<0.002**
Eu	L1	9	k_0-INAA	< 0.00013	< 0.00015	< 0.00015	<0.00015	< 0.00016	< 0.00017	**< 0.00017**
	L2	27	INAA	<0.0003	<0.0003	<0.0002	<0.0002	<0.0002	<0.0002	**<0.0003**
Hg	L1	9	DM-AAS	0.00698	0.00715	0.00719	0.00718	0.00714	0.00719	**0.00715**
	L2	9	k_0-INAA	0.0046	0.0058	0 0036	0.0048	0.0046	0 0056	**0.0049**
La	L1	27	INAA	0.0005	0.0010	0.0013	0.0147	0.0004	0.0007	**0.0031**
Rb	L1	27	INAA	<0.004	<0.005	<0.006	<0.006	<0.006	<0.005	**<0.006**
Re	L1	9	k_0-INAA	0.00051	0.00050	0.00046	0.00048	0.00059	0.00049	**0.00051**
Sc	L1	6	ETV-ICP-OES	0.00005	0.00002	0.00001	0.00006	0.00002	0.00005	**0.00004**
	L2	9	k_0-INAA	< 0.00014	< 0.00016	< 0.00016	< 0.00018	< 0.00014	< 0.00012	**< 0.00018**
	L3	27	INAA	0.00033	0.00017	0.00018	0.00023	0.00017	0.00015	**0.00021**
Se	L1	27	INAA	0.0023	<0.0024	<0.0024	0.0015	<0.0042	<0.0023	**<0.0042**
Sm	L1	9	k_0-INAA	<0.00005	< 0 00005	< 0.00005	< 0.00005	< 0.00005	< 0.00005	**< 0.00005**
Sn	L1	13(2)	ETV-ICP-OES	0.00263	0.00133	0.00052	0.00181	0.00222	0.00120	**0.00162**
	L2	13(1)	ETV-ICP-OES	0.00453	0.00483	0.00358	0.00314	0.00638	0.00291	**0.00423**
	L3	13(3)	ETV-ICP-OES	0.00898	0.01888	0.01461	0.01851	0.02435	0.02177	**0.01785**
Ta	L1	9	k_0-INAA	< 0.0004	< 0.0008	<0.0009	<0.0009	< 0.0004	< 0.0002	**<0.0009**
	L2	10	ETV-ICP-OES	0.00086	0.00016	0.00081	0.00061	0.00044	0.00036	**0.00054**
Tb	L1	9	k_0-INAA	< 0.0007	< 0.00001	< 0.0008	< 0.0008	< 0.0007	<0.0006	**< 0.0008**
Te	L1	13(1)	ETV-ICP-OES	0.00013	0.00040	0.00046	0.00006	0.00041	0.00021	**0.00028**
	L2	13(3)	ETV-ICP-OES	0.00136	0.00169	0.00058	0.00052	0.00026	0.00131	**0.00095**
	L3	6	ETV-ICP-OES	0.00171	0.00181	0.00217	0.00214	0.00232	0.00142	**0.00193**
Th	L1	9	k_0-INAA	< 0.0009	<0.0010	<0.0011	< 0.0011	< 0.0010	< 0.0008	**< 0.0011**
U	L1	9	k_0-INAA	< 0.0007	< 0.0008	< 0.0008	<0.0007	< 0.0008	< 0.0008	**< 0.0008**

Annex C: Outcome of statistical tests on results from the ILC

Tab. C.1: Outcome of statistical tests on results obtained for Ag

Element			Ag
Run of evaluation program			Run 1
Number of data sets			4
Testing for outlying Lab means	Dixon test outlier	$\alpha = 0.05$	none
		$\alpha = 0.01$	none
	Nalimov test outlier	$\alpha = 0.05$	none
		$\alpha = 0.01$	none
	Grubbs test outlier	$\alpha = 0.05$	none
		$\alpha = 0.01$	none
Testing for outlying Lab variances	Cochran test outlier	$\alpha = 0.05$	Lab 10
		$\alpha = 0.01$	Lab 10
Normality of Lab means distribution:	Kolmogorov-Smirnov-Lilliefors test	$\alpha = 0.05$	normal
		$\alpha = 0.01$	normal
	Skewness & Kurtosis test	$\alpha = 0.05$	Insufficient data
		$\alpha = 0.01$	Insufficient data
Snedecor F-test and Bartlett test show that pooling is:			not allowed
Decision concluded			Run 1 accepted, no outlier removed

* 2 from 6 datasets submitted were not included into the statistical evaluation (cf. Table B.1)

Tab. C.2: Outcome of statistical tests on results obtained for Al

Element			Al
Run of evaluation program			Run 1
Number of data sets			13
Testing for outlying Lab means	Dixon test outlier	$\alpha = 0.05$	---
		$\alpha = 0.01$	---
	Nalimov test outlier	$\alpha = 0.05$	Lab 22
		$\alpha = 0.01$	---
	Grubbs test outlier	$\alpha = 0.05$	---
		$\alpha = 0.01$	---
Testing for outlying Lab variances	Cochran test outlier	$\alpha = 0.05$	Lab 3
		$\alpha = 0.01$	Lab 3
Normality of Lab means distribution:	Kolmogorov-Smirnov-Lilliefors test	$\alpha = 0.05$	normal
		$\alpha = 0.01$	normal
	Skewness & Kurtosis test	$\alpha = 0.05$	not normal
		$\alpha = 0.01$	not normal
Snedecor F-test and Bartlett test show that pooling is:			not allowed
Decision concluded			Run 1 accepted

Tab. C.3: Outcome of statistical tests on results obtained for As

Element			As
Run of evaluation program			Run 1
Number of data sets			5*
Testing for outlying Lab means	Dixon test outlier	α = 0.05	---
		α = 0.01	---
	Nalimov test outlier	α = 0.05	Lab 13(2)
		α = 0.01	---
	Grubbs test outlier	α = 0.05	---
		α = 0.01	---
Testing for outlying Lab variances	Cochran test outlier	α = 0.05	Lab 10
		α = 0.01	Lab 10
Normality of Lab means distribution:	Kolmogorov-Smirnov-Lilliefors test	α = 0.05	normal
		α = 0.01	normal
	Skewness & Kurtosis test	α = 0.05	Insufficient data
		α = 0.01	Insufficient data
Snedecor F-test and Bartlett test show that pooling is:			not allowed
Decision concluded			Outlier Lab 10 removed

* 1 from 6 datasets submitted was not included into the statistical evaluation (cf. Table B.3)

(continued)

Element			As
Run of evaluation program			Run 2 after removal of Lab 10
Number of data sets			4
Testing for outlying Lab means	Dixon test outlier	α = 0.05	---
		α = 0.01	---
	Nalimov test outlier	α = 0.05	Lab 13(2)
		α = 0.01	---
	Grubbs test outlier	α = 0.05	---
		α = 0.01	---
Testing for outlying Lab variances	Cochran test outlier	α = 0.05	---
		α = 0.01	---
Normality of Lab means distribution:	Kolmogorov-Smirnov-Lilliefors test	α = 0.05	normal
		α = 0.01	normal
	Skewness & Kurtosis test	α = 0.05	Insufficient data
		α = 0.01	Insufficient data
Snedecor F-test and Bartlett test show that pooling is:			not allowed
Decision concluded			Run 2 accepted, the outlier Lab 13(2) was not removed

Tab. C.4: Outcome of statistical tests on results obtained for B

Element			B
Run of evaluation program			Run 1
Number of data sets			7*
Testing for outlying Lab means	Dixon test outlier	α = 0.05	---
		α = 0.01	---
	Nalimov test outlier	α = 0.05	---
		α = 0.01	---
	Grubbs test outlier	α = 0.05	---
		α = 0.01	---
Testing for outlying Lab variances	Cochran test outlier	α = 0.05	---
		α = 0.01	---
Normality of Lab means distribution:	Kolmogorov-Smirnov-Lilliefors test	α = 0.05	normal
		α = 0.01	normal
	Skewness & Kurtosis test	α = 0.05	normal
		α = 0.01	normal
Snedecor F-test and Bartlett test show that pooling is:			not allowed
Decision concluded			Run 1 accepted

* 1 from 8 datasets submitted was not included into the statistical evaluation (cf. Table B.5)

Tab. C.5: Outcome of statistical tests on results obtained for Ba

Element			Ba
Run of evaluation program			Run 1
Number of data sets			10
Testing for outlying Lab means	Dixon test outlier	α = 0.05	Lab 31
		α = 0.01	Lab 31
	Nalimov test outlier	α = 0.05	Lab 31
		α = 0.01	Lab 31
	Grubbs test outlier	α = 0.05	Lab 31
		α = 0.01	Lab 31
Testing for outlying Lab variances	Cochran test outlier	α = 0.05	---
		α = 0.01	---
Normality of Lab means distribution:	Kolmogorov-Smirnov-Lilliefors test	α = 0.05	not normal
		α = 0.01	not normal
	Skewness & Kurtosis test	α = 0.05	not normal
		α = 0.01	not normal
Snedecor F-test and Bartlett test show that pooling is:			not allowed
Decision concluded			Outlier Lab 31 removed

* 3 from 13 datasets submitted were not included into the statistical evaluation (cf. Table B.6)

(continued)

Element			Ba
Run of evaluation program			Run 2 after removal of Lab 31
Number of data sets			9
Testing for outlying Lab means	Dixon test outlier	α = 0.05	---
		α = 0.01	---
	Nalimov test outlier	α = 0.05	---
		α = 0.01	---
	Grubbs test outlier	α = 0.05	---
		α = 0.01	---
Testing for outlying Lab variances	Cochran test outlier	α = 0.05	---
		α = 0.01	---
Normality of Lab means distribution:	Kolmogorov-Smirnov-Lilliefors test	α = 0.05	normal
		α = 0.01	normal
	Skewness & Kurtosis test	α = 0.05	normal
		α = 0.01	normal
Snedecor F-test and Bartlett test show that pooling is:			not allowed
Decision concluded			Run 2 accepted

Tab. C.6: Outcome of statistical tests on results obtained for Be

Element			**Be**
Run of evaluation program			Run 1
Number of data sets			7
Testing for outlying Lab means	Dixon test outlier	$\alpha = 0.05$	---
		$\alpha = 0.01$	---
	Nalimov test outlier	$\alpha = 0.05$	Lab 13(1)
		$\alpha = 0.01$	---
	Grubbs test outlier	$\alpha = 0.05$	---
		$\alpha = 0.01$	---
	Paired Grubbs test outliers	$\alpha = 0.05$	Lab 10, Lab 16
		$\alpha = 0.01$	Lab 10, Lab 16
Testing for outlying Lab variances	Cochran test outlier	$\alpha = 0.05$	Lab 16, Lab 10, Lab 26
		$\alpha = 0.01$	Lab 13(1)
Normality of Lab means distribution:	Kolmogorov-Smirnov-Lilliefors test	$\alpha = 0.05$	normal
		$\alpha = 0.01$	normal
	Skewness & Kurtosis test	$\alpha = 0.05$	not normal
		$\alpha = 0.01$	normal
Snedecor F-test and Bartlett test show that pooling is:			not allowed
Decision concluded			Datasets of Lab 10 and 16 rejected as paired Grubbs outlier ($\alpha = 0.01$)

(continued)

Element			**Be**
Run of evaluation program			Run 2
Number of data sets			5
Testing for outlying Lab means	Dixon test outlier	$\alpha = 0.05$	Lab 26
		$\alpha = 0.01$	---
	Nalimov test outlier	$\alpha = 0.05$	Lab 26
		$\alpha = 0.01$	Lab 26
	Grubbs test outlier	$\alpha = 0.05$	Lab 26
		$\alpha = 0.01$	Lab 26
Testing for outlying Lab variances	Cochran test outlier	$\alpha = 0.05$	Lab 26
		$\alpha = 0.01$	Lab 26
Normality of Lab means distribution:	Kolmogorov-Smirnov-Lilliefors test	$\alpha = 0.05$	not normal
		$\alpha = 0.01$	normal
	Skewness & Kurtosis test	$\alpha = 0.05$	Insufficient data
		$\alpha = 0.01$	Insufficient data
Snedecor F-test and Bartlett test show that pooling is:			not allowed
Decision concluded			Run 2 accepted, the outlier Lab 26 was not removed

Tab. C.7: Outcome of statistical tests on results obtained for Ca

Element			Ca
Run of evaluation program			Run 1
Number of data sets			13
Testing for outlying Lab means	Dixon test outlier	$\alpha = 0.05$	Lab 22
		$\alpha = 0.01$	Lab 22
	Nalimov test outlier	$\alpha = 0.05$	Lab 22
		$\alpha = 0.01$	Lab 22
	Grubbs test outlier	$\alpha = 0.05$	Lab 22
		$\alpha = 0.01$	Lab 22
Testing for outlying Lab variances	Cochran test outlier	$\alpha = 0.05$	Lab 22, Lab 3
		$\alpha = 0.01$	Lab 22, Lab 3
Normality of Lab means distribution:	Kolmogorov-Smirnov-Lilliefors test	$\alpha = 0.05$	not normal
		$\alpha = 0.01$	normal
	Skewness & Kurtosis test	$\alpha = 0.05$	not normal
		$\alpha = 0.01$	not normal
Snedecor F-test and Bartlett test show that pooling is:			not allowed
Decision concluded			Outlier Lab 22 removed

(continued)

Element			Ca
Run of evaluation program			Run 2 after removal of Lab 22
Number of data sets			12
Testing for outlying Lab means	Dixon test outlier	$\alpha = 0.05$	---
		$\alpha = 0.01$	---
	Nalimov test outlier	$\alpha = 0.05$	Lab 13(2)
		$\alpha = 0.01$	---
	Grubbs test outlier	$\alpha = 0.05$	---
		$\alpha = 0.01$	---
Testing for outlying Lab variances	Cochran test outlier	$\alpha = 0.05$	Lab 3
		$\alpha = 0.01$	Lab 3
Normality of Lab means distribution:	Kolmogorov-Smirnov-Lilliefors test	$\alpha = 0.05$	normal
		$\alpha = 0.01$	normal
	Skewness & Kurtosis test	$\alpha = 0.05$	normal
		$\alpha = 0.01$	normal
Snedecor F-test and Bartlett test show that pooling is:			not allowed
Decision concluded			Run 2 accepted, the outlier Lab 13(2) was not removed

Tab. C.8: Outcome of statistical tests on results obtained for Cd

Element			Cd
Run of evaluation program			Run 1
Number of data sets			7*
Testing for outlying Lab means	Dixon test outlier	α = 0.05	Lab 16
		α = 0.01	Lab 16
	Nalimov test outlier	α = 0.05	Lab 16
		α = 0.01	Lab 16
	Grubbs test outlier	α = 0.05	Lab 16
		α = 0.01	Lab 16
Testing for outlying Lab variances	Cochran test outlier	α = 0.05	Lab 16, Lab 10, Lab 26
		α = 0.01	Lab 16, Lab 10, Lab 26
Normality of Lab means distribution:	Kolmogorov-Smirnov-Lilliefors test	α = 0.05	not normal
		α = 0.01	not normal
	Skewness & Kurtosis test	α = 0.05	not normal
		α = 0.01	not normal
Snedecor F-test and Bartlett test show that pooling is:			not allowed
Decision concluded			Outlier Lab 16 removed

* 1 from 8 datasets submitted was not included into the statistical evaluation (cf. Table B.10)

(continued)

Element			Cd
Run of evaluation program			Run 2 after removal of Lab 16
Number of data sets			6
Testing for outlying Lab means	Dixon test outlier	α = 0.05	---
		α = 0.01	---
	Nalimov test outlier	α = 0.05	---
		α = 0.01	---
	Grubbs test outlier	α = 0.05	---
		α = 0.01	---
Testing for outlying Lab variances	Cochran test outlier	α = 0.05	Lab 10, Lab 26
		α = 0.01	Lab 10, Lab 26
Normality of Lab means distribution:	Kolmogorov-Smirnov-Lilliefors test	α = 0.05	normal
		α = 0.01	normal
	Skewness & Kurtosis test	α = 0.05	Insufficient data
		α = 0.01	Insufficient data
Snedecor F-test and Bartlett test show that pooling is:			not allowed
Decision concluded			Run 2 accepted

Tab. C.9: Outcome of statistical tests on results obtained for Co

Element			Co
Run of evaluation program			Run 1
Number of data sets			10*
Testing for outlying Lab means	Dixon test outlier	α = 0.05	Lab 27
		α = 0.01	Lab 27
	Nalimov test outlier	α = 0.05	Lab 27
		α = 0.01	Lab 27
	Grubbs test outlier	α = 0.05	Lab 27
		α = 0.01	Lab 27
Testing for outlying Lab variances	Cochran test outlier	α = 0.05	Lab 27, Lab 16, Lab 24, Lab 26
		α = 0.01	Lab 27, Lab 16, Lab 24
Normality of Lab means distribution:	Kolmogorov-Smirnov-Lilliefors test	α = 0.05	not normal
		α = 0.01	not normal
	Skewness & Kurtosis test	α = 0.05	not normal
		α = 0.01	not normal
Snedecor F-test and Bartlett test show that pooling is:			not allowed
Decision concluded			Outlier Lab 27 removed

* 4 from 14 datasets submitted were not included into the statistical evaluation (cf. Table B.11)

(continued)

Element			Co
Run of evaluation program			Run 2 after removal of Lab 27
Number of data sets			9
Testing for outlying Lab means	Dixon test outlier	α = 0.05	---
		α = 0.01	---
	Nalimov test outlier	α = 0.05	Lab 16
		α = 0.01	---
	Grubbs test outlier	α = 0.05	---
		α = 0.01	---
Testing for outlying Lab variances	Cochran test outlier	α = 0.05	Lab 16, Lab 24, Lab 26
		α = 0.01	Lab 16, Lab 24
Normality of Lab means distribution:	Kolmogorov-Smirnov-Lilliefors test	α = 0.05	not normal
		α = 0.01	not normal
	Skewness & Kurtosis test	α = 0.05	not normal
		α = 0.01	normal
Snedecor F-test and Bartlett test show that pooling is:			not allowed
Decision concluded			Run 2 accepted, the outlier Lab 16 was not removed

Tab. C.10: Outcome of statistical tests on results obtained for Cr

Element			Cr
Run of evaluation program			Run 1
Number of data sets			14*
Testing for outlying Lab means	Dixon test outlier	α = 0.05	---
		α = 0.01	---
	Nalimov test outlier	α = 0.05	Lab 31
		α = 0.01	Lab 31
	Grubbs test outlier	α = 0.05	Lab 31
		α = 0.01	---
Testing for outlying Lab variances	Cochran test outlier	α = 0.05	Lab 28, Lab 9, Lab 27
		α = 0.01	Lab 9, Lab 27
Normality of Lab means distribution:	Kolmogorov-Smirnov-Lilliefors test	α = 0.05	not normal
		α = 0.01	not normal
	Skewness & Kurtosis test	α = 0.05	not normal
		α = 0.01	not normal
Snedecor F-test and Bartlett test show that pooling is:			not allowed
Decision concluded			Outlier Lab 31 removed

* 1 from 15 datasets submitted was not included into the statistical evaluation (cf. Table B.11)

(continued)

Element			Cr
Run of evaluation program			Run 2 after removal of Lab 31
Number of data sets			13
Testing for outlying Lab means	Dixon test outlier	α = 0.05	Lab 9
		α = 0.01	---
	Nalimov test outlier	α = 0.05	Lab 9
		α = 0.01	Lab 9
	Grubbs test outlier	α = 0.05	Lab 9
		α = 0.01	Lab 9
Testing for outlying Lab variances	Cochran test outlier	α = 0.05	Lab 28, Lab 9, Lab 27
		α = 0.01	Lab 9, Lab 27
Normality of Lab means distribution:	Kolmogorov-Smirnov-Lilliefors test	α = 0.05	not normal
		α = 0.01	not normal
	Skewness & Kurtosis test	α = 0.05	not normal
		α = 0.01	not normal
Snedecor F-test and Bartlett test show that pooling is:			not allowed
Decision concluded			Run 2 accepted, the outlier Lab 9 was not removed

Tab. C.11: Outcome of statistical tests on results obtained for Cu

Element			Cu
Run of evaluation program			Run 1
Number of data sets			10*
Testing for outlying Lab means	Dixon test outlier	α = 0.05	---
		α = 0.01	---
	Nalimov test outlier	α = 0.05	Lab 16
		α = 0.01	---
	Grubbs test outlier	α = 0.05	---
		α = 0.01	---
Testing for outlying Lab variances	Cochran test outlier	α = 0.05	Lab 26, Lab 16
		α = 0.01	Lab 26, Lab 16
Normality of Lab means distribution:	Kolmogorov-Smirnov-Lilliefors test	α = 0.05	not normal
		α = 0.01	normal
	Skewness & Kurtosis test	α = 0.05	normal
		α = 0.01	normal
Snedecor F-test and Bartlett test show that pooling is:			not allowed
Decision concluded			Run 1 accepted, the outlier Lab 16 was not removed

* 3 from 13 datasets submitted were not included into the statistical evaluation (cf. Table B.12)

Tab. C.12: Outcome of statistical tests on results obtained for Fe

Element			Fe
Run of evaluation program			Run 1
Number of data sets			14
Testing for outlying Lab means	Dixon test outlier	α = 0.05	Lab 27
		α = 0.01	Lab 27
	Nalimov test outlier	α = 0.05	Lab 27
		α = 0.01	Lab 27
	Grubbs test outlier	α = 0.05	Lab 27
		α = 0.01	Lab 27
Testing for outlying Lab variances	Cochran test outlier	α = 0.05	Lab 3, Lab 27
		α = 0.01	Lab 3, Lab 27
Normality of Lab means distribution:	Kolmogorov-Smirnov-Lilliefors test	α = 0.05	normal
		α = 0.01	normal
	Skewness & Kurtosis test	α = 0.05	not normal
		α = 0.01	not normal
Snedecor F-test and Bartlett test show that pooling is:			not allowed
Decision concluded			Outlier Lab 27 removed

(continued)

Element			Fe
Run of evaluation program			Run 2 after removal of Lab 27
Number of data sets			13
Testing for outlying Lab means	Dixon test outlier	α = 0.05	---
		α = 0.01	---
	Nalimov test outlier	α = 0.05	Lab 28, Lab 3
		α = 0.01	---
	Grubbs test outlier	α = 0.05	---
		α = 0.01	---
Testing for outlying Lab variances	Cochran test outlier	α = 0.05	Lab 3
		α = 0.01	Lab 3
Normality of Lab means distribution:	Kolmogorov-Smirnov-Lilliefors test	α = 0.05	normal
		α = 0.01	normal
	Skewness & Kurtosis test	α = 0.05	normal
		α = 0.01	normal
Snedecor F-test and Bartlett test show that pooling is:			not allowed
Decision concluded			Outlier Lab 3 removed

(continued)

Element			Fe
Run of evaluation program			Run 3 after removal of Lab 3
Number of data sets			12
Testing for outlying Lab means	Dixon test outlier	$\alpha = 0.05$	---
		$\alpha = 0.01$	---
	Nalimov test outlier	$\alpha = 0.05$	Lab 28
		$\alpha = 0.01$	Lab 28
	Grubbs test outlier	$\alpha = 0.05$	---
		$\alpha = 0.01$	---
Testing for outlying Lab variances	Cochran test outlier	$\alpha = 0.05$	---
		$\alpha = 0.01$	---
Normality of Lab means distribution:	Kolmogorov-Smirnov-Lilliefors test	$\alpha = 0.05$	normal
		$\alpha = 0.01$	normal
	Skewness & Kurtosis test	$\alpha = 0.05$	normal
		$\alpha = 0.01$	normal
Snedecor F-test and Bartlett test show that pooling is:			not allowed
Decision concluded			Run 3 accepted, the outlier Lab 28 was not removed

Tab. C.13: Outcome of statistical tests on results obtained for K

Element			K
Run of evaluation program			Run 1
Number of data sets			9*
Testing for outlying Lab means	Dixon test outlier	$\alpha = 0.05$	---
		$\alpha = 0.01$	---
	Nalimov test outlier	$\alpha = 0.05$	Lab 22
		$\alpha = 0.01$	---
	Grubbs test outlier	$\alpha = 0.05$	---
		$\alpha = 0.01$	---
Testing for outlying Lab variances	Cochran test outlier	$\alpha = 0.05$	Lab 10, Lab 22
		$\alpha = 0.01$	Lab 10, Lab 22
Normality of Lab means distribution:	Kolmogorov-Smirnov-Lilliefors test	$\alpha = 0.05$	normal
		$\alpha = 0.01$	normal
	Skewness & Kurtosis test	$\alpha = 0.05$	normal
		$\alpha = 0.01$	normal
Snedecor F-test and Bartlett test show that pooling is:			not allowed
Decision concluded			Run 1 accepted, the outlier Lab 22 was not removed

* 3 from 12 datasets submitted were not included into the statistical evaluation (cf. Table B.13)

Tab. C.14: Outcome of statistical tests on results obtained for Li

Element			Li
Run of evaluation program			Run 1
Number of data sets			9*
Testing for outlying Lab means	Dixon test outlier	α = 0.05	Lab 13(1)
		α = 0.01	---
	Nalimov test outlier	α = 0.05	Lab 13(1)
		α = 0.01	Lab 13(1)
	Grubbs test outlier	α = 0.05	Lab 13(1)
		α = 0.01	Lab 13(1)
Testing for outlying Lab variances	Cochran test outlier	α = 0.05	Lab 13(1), Lab 10, Lab 26, Lab 13(3), Lab 22, Lab 24
		α = 0.01	Lab 13(1), Lab 26, Lab 13(3), Lab 22, Lab 24
Normality of Lab means distribution:	Kolmogorov-Smirnov-Lilliefors test	α = 0.05	not normal
		α = 0.01	normal
	Skewness & Kurtosis test	α = 0.05	not normal
		α = 0.01	not normal
Snedecor F-test and Bartlett test show that pooling is:			not allowed
Decision concluded			Outlier Lab 13(1) removed

* 1 from 10 datasets submitted was not included into the statistical evaluation (cf. Table B.14)

(continued)

Element			Li
Run of evaluation program			Run 2 after removal of Lab 13(1)
Number of data sets			8
Testing for outlying Lab means	Dixon test outlier	α = 0.05	---
		α = 0.01	---
	Nalimov test outlier	α = 0.05	---
		α = 0.01	---
	Grubbs test outlier	α = 0.05	---
		α = 0.01	---
Testing for outlying Lab variances	Cochran test outlier	α = 0.05	Lab 10, Lab 26, Lab 13(3), Lab 22, Lab 24
		α = 0.01	Lab 26, Lab 22, Lab 24
Normality of Lab means distribution:	Kolmogorov-Smirnov-Lilliefors test	α = 0.05	normal
		α = 0.01	normal
	Skewness & Kurtosis test	α = 0.05	normal
		α = 0.01	normal
Snedecor F-test and Bartlett test show that pooling is:			not allowed
Decision concluded			Run 2 accepted

Tab. C.15: Outcome of statistical tests on results obtained for Mg

Element			Mg
Run of evaluation program			Run 1
Number of data sets			13
Testing for outlying Lab means	Dixon test outlier	α = 0.05	---
		α = 0.01	---
	Nalimov test outlier	α = 0.05	Lab 6
		α = 0.01	---
	Grubbs test outlier	α = 0.05	---
		α = 0.01	---
Testing for outlying Lab variances	Cochran test outlier	α = 0.05	Lab 3
		α = 0.01	Lab 3
Normality of Lab means distribution:	Kolmogorov-Smirnov-Lilliefors test	α = 0.05	normal
		α = 0.01	normal
	Skewness & Kurtosis test	α = 0.05	normal
		α = 0.01	normal
Snedecor F-test and Bartlett test show that pooling is:			not allowed
Decision concluded			Run 1 accepted, the outlier Lab 6 was not removed

Tab. C.16: Outcome of statistical tests on results obtained for Mn

Element			Mn
Run of evaluation program			Run 1
Number of data sets			11*
Testing for outlying Lab means	Dixon test outlier	α = 0.05	Lab 31
		α = 0.01	Lab 31
	Nalimov test outlier	α = 0.05	Lab 31
		α = 0.01	Lab 31
	Grubbs test outlier	α = 0.05	Lab 31
		α = 0.01	Lab 31
Testing for outlying Lab variances	Cochran test outlier	α = 0.05	Lab 31, Lab 28
		α = 0.01	Lab 31
Normality of Lab means distribution:	Kolmogorov-Smirnov-Lilliefors test	α = 0.05	not normal
		α = 0.01	not normal
	Skewness & Kurtosis test	α = 0.05	not normal
		α = 0.01	not normal
Snedecor F-test and Bartlett test show that pooling is:			not allowed
Decision concluded			Outlier Lab 31 removed

* 2 from 13 datasets submitted were not included into the statistical evaluation (cf. Table B.16)

(continued)

Element			Mn
Run of evaluation program			Run 2 after removal of Lab 31
Number of data sets			10
Testing for outlying Lab means	Dixon test outlier	α = 0.05	---
		α = 0.01	---
	Nalimov test outlier	α = 0.05	---
		α = 0.01	---
	Grubbs test outlier	α = 0.05	---
		α = 0.01	---
Testing for outlying Lab variances	Cochran test outlier	α = 0.05	Lab 28
		α = 0.01	---
Normality of Lab means distribution:	Kolmogorov-Smirnov-Lilliefors test	α = 0.05	normal
		α = 0.01	normal
	Skewness & Kurtosis test	α = 0.05	normal
		α = 0.01	normal
Snedecor F-test and Bartlett test show that pooling is:			not allowed
Decision concluded			Run 2 accepted

Tab. C.17: Outcome of statistical tests on results obtained for Mo

Element			Mo
Run of evaluation program			Run 1
Number of data sets			8*
Testing for outlying Lab means	Dixon test outlier	α = 0.05	---
		α = 0.01	---
	Nalimov test outlier	α = 0.05	---
		α = 0.01	---
	Grubbs test outlier	α = 0.05	---
		α = 0.01	---
Testing for outlying Lab variances	Cochran test outlier	α = 0.05	Lab 26
		α = 0.01	Lab 26
Normality of Lab means distribution:	Kolmogorov-Smirnov-Lilliefors test	α = 0.05	normal
		α = 0.01	normal
	Skewness & Kurtosis test	α = 0.05	normal
		α = 0.01	normal
Snedecor F-test and Bartlett test show that pooling is:			not allowed
Decision concluded			Run 1 accepted

* 2 from 10 datasets submitted were not included into the statistical evaluation (cf. Table B.17)

Tab. C.18: Outcome of statistical tests on results obtained for Na

Element			Na
Run of evaluation program			Run 1
Number of data sets			13
Testing for outlying Lab means	Dixon test outlier	$\alpha = 0.05$	---
		$\alpha = 0.01$	---
	Nalimov test outlier	$\alpha = 0.05$	Lab 27
		$\alpha = 0.01$	Lab 27
	Grubbs test outlier	$\alpha = 0.05$	Lab 27
		$\alpha = 0.01$	---
Testing for outlying Lab variances	Cochran test outlier	$\alpha = 0.05$	Lab 23, Lab 27, Lab 26, Lab 16
		$\alpha = 0.01$	Lab 23, Lab 27, Lab 26, Lab 16
Normality of Lab means distribution:	Kolmogorov-Smirnov-Lilliefors test	$\alpha = 0.05$	not normal
		$\alpha = 0.01$	not normal
	Skewness & Kurtosis test	$\alpha = 0.05$	not normal
		$\alpha = 0.01$	normal
Snedecor F-test and Bartlett test show that pooling is:			not allowed
Decision concluded			Outlier Lab 27 removed

(continued)

Element			Na
Run of evaluation program			Run 2 after removal of Lab 27
Number of data sets			12
Testing for outlying Lab means	Dixon test outlier	$\alpha = 0.05$	---
		$\alpha = 0.01$	---
	Nalimov test outlier	$\alpha = 0.05$	Lab 16
		$\alpha = 0.01$	---
	Grubbs test outlier	$\alpha = 0.05$	---
		$\alpha = 0.01$	---
Testing for outlying Lab variances	Cochran test outlier	$\alpha = 0.05$	Lab 23, Lab 26, Lab 16
		$\alpha = 0.01$	Lab 23, Lab 26, Lab 16
Normality of Lab means distribution:	Kolmogorov-Smirnov-Lilliefors test	$\alpha = 0.05$	not normal
		$\alpha = 0.01$	normal
	Skewness & Kurtosis test	$\alpha = 0.05$	normal
		$\alpha = 0.01$	normal
Snedecor F-test and Bartlett test show that pooling is:			not allowed
Decision concluded			Run 2 accepted, the outlier Lab 16 was not removed

Tab. C.19: Outcome of statistical tests on results obtained for Ni

Element			Ni
Run of evaluation program			Run 1
Number of data sets			13
Testing for outlying Lab means	Dixon test outlier	$\alpha = 0.05$	---
		$\alpha = 0.01$	---
	Nalimov test outlier	$\alpha = 0.05$	Lab 3
		$\alpha = 0.01$	---
	Grubbs test outlier	$\alpha = 0.05$	---
		$\alpha = 0.01$	---
Testing for outlying Lab variances	Cochran test outlier	$\alpha = 0.05$	Lab 3
		$\alpha = 0.01$	Lab 3
Normality of Lab means distribution:	Kolmogorov-Smirnov-Lilliefors test	$\alpha = 0.05$	not normal
		$\alpha = 0.01$	normal
	Skewness & Kurtosis test	$\alpha = 0.05$	not normal
		$\alpha = 0.01$	normal
Snedecor F-test and Bartlett test show that pooling is:			not allowed
Decision concluded			Run 1 accepted, the outlier Lab 3 was not removed

Tab. C.20: Outcome of statistical tests on results obtained for P

Element			P
Run of evaluation program			Run 1
Number of data sets			7
Testing for outlying Lab means	Dixon test outlier	$\alpha = 0.05$	Lab 26
		$\alpha = 0.01$	---
	Nalimov test outlier	$\alpha = 0.05$	Lab 26
		$\alpha = 0.01$	Lab 26
	Grubbs test outlier	$\alpha = 0.05$	Lab 26
		$\alpha = 0.01$	---
Testing for outlying Lab variances	Cochran test outlier	$\alpha = 0.05$	---
		$\alpha = 0.01$	---
Normality of Lab means distribution:	Kolmogorov-Smirnov-Lilliefors test	$\alpha = 0.05$	normal
		$\alpha = 0.01$	normal
	Skewness & Kurtosis test	$\alpha = 0.05$	not normal
		$\alpha = 0.01$	normal
Snedecor F-test and Bartlett test show that pooling is:			not allowed
Decision concluded			Outlier Lab 26 removed

(continued)

Element			P
Run of evaluation program			Run 2 after removal of Lab 26
Number of data sets			6
Testing for outlying Lab means	Dixon test outlier	$\alpha = 0.05$	---
		$\alpha = 0.01$	---
	Nalimov test outlier	$\alpha = 0.05$	---
		$\alpha = 0.01$	---
	Grubbs test outlier	$\alpha = 0.05$	---
		$\alpha = 0.01$	---
Testing for outlying Lab variances	Cochran test outlier	$\alpha = 0.05$	Lab 16
		$\alpha = 0.01$	---
Normality of Lab means distribution:	Kolmogorov-Smirnov-Lilliefors test	$\alpha = 0.05$	normal
		$\alpha = 0.01$	normal
	Skewness & Kurtosis test	$\alpha = 0.05$	Insufficient data
		$\alpha = 0.01$	Insufficient data
Snedecor F-test and Bartlett test show that pooling is:			not allowed
Decision concluded			Run 2 accepted

Tab. C.21: Outcome of statistical tests on results obtained for Pb

Element			Pb
Run of evaluation program			Run 1
Number of data sets			9*
Testing for outlying Lab means	Dixon test outlier	$\alpha = 0.05$	---
		$\alpha = 0.01$	---
	Nalimov test outlier	$\alpha = 0.05$	Lab 16
		$\alpha = 0.01$	Lab 16
	Grubbs test outlier	$\alpha = 0.05$	Lab 16
		$\alpha = 0.01$	---
Testing for outlying Lab variances	Cochran test outlier	$\alpha = 0.05$	Lab 16, Lab 22, Lab 26
		$\alpha = 0.01$	Lab 16, Lab 22, Lab 26
Normality of Lab means distribution:	Kolmogorov-Smirnov-Lilliefors test	$\alpha = 0.05$	normal
		$\alpha = 0.01$	normal
	Skewness & Kurtosis test	$\alpha = 0.05$	not normal
		$\alpha = 0.01$	normal
Snedecor F-test and Bartlett test show that pooling is:			not allowed
Decision concluded			Outlier Lab 16 removed

* 2 from 11 datasets submitted were not included into the statistical evaluation (cf. Table B.21)

(continued)

Element			Pb
Run of evaluation program			Run 2 after removal of Lab 16
Number of data sets			8
Testing for outlying Lab means	Dixon test outlier	$\alpha = 0.05$	---
		$\alpha = 0.01$	---
	Nalimov test outlier	$\alpha = 0.05$	---
		$\alpha = 0.01$	---
	Grubbs test outlier	$\alpha = 0.05$	---
		$\alpha = 0.01$	---
Testing for outlying Lab variances	Cochran test outlier	$\alpha = 0.05$	Lab 22, Lab 26
		$\alpha = 0.01$	Lab 22, Lab 26
Normality of Lab means distribution:	Kolmogorov-Smirnov-Lilliefors test	$\alpha = 0.05$	normal
		$\alpha = 0.01$	normal
	Skewness & Kurtosis test	$\alpha = 0.05$	normal
		$\alpha = 0.01$	normal
Snedecor F-test and Bartlett test show that pooling is:			not allowed
Decision concluded			Run 2 accepted

Tab. C.22: Outcome of statistical tests on results obtained for S

Element			S
Run of evaluation program			Run 1
Number of data sets			9
Testing for outlying Lab means	Dixon test outlier	α = 0.05	---
		α = 0.01	---
	Nalimov test outlier	α = 0.05	Lab 10
		α = 0.01	Lab 10
	Grubbs test outlier	α = 0.05	Lab 10
		α = 0.01	---
Testing for outlying Lab variances	Cochran test outlier	α = 0.05	---
		α = 0.01	---
Normality of Lab means distribution:	Kolmogorov-Smirnov-Lilliefors test	α = 0.05	normal
		α = 0.01	normal
	Skewness & Kurtosis test	α = 0.05	normal
		α = 0.01	normal
Snedecor F-test and Bartlett test show that pooling is:			not allowed
Decision concluded			Outlier Lab 10 removed

(continued)

Element			S
Run of evaluation program			Run 2 after removal of Lab 10
Number of data sets			8
Testing for outlying Lab means	Dixon test outlier	α = 0.05	---
		α = 0.01	---
	Nalimov test outlier	α = 0.05	Lab 16
		α = 0.01	---
	Grubbs test outlier	α = 0.05	---
		α = 0.01	---
Testing for outlying Lab variances	Cochran test outlier	α = 0.05	---
		α = 0.01	---
Normality of Lab means distribution:	Kolmogorov-Smirnov-Lilliefors test	α = 0.05	normal
		α = 0.01	normal
	Skewness & Kurtosis test	α = 0.05	normal
		α = 0.01	normal
Snedecor F-test and Bartlett test show that pooling is:			not allowed
Decision concluded			Run 2 accepted, the outlier Lab 16 was not removed

Tab. C.23: Outcome of statistical tests on results obtained for Sb

Element			Sb
Run of evaluation program			Run 1
Number of data sets			7*
Testing for outlying Lab means	Dixon test outlier	α = 0.05	Lab 13(2)
		α = 0.01	Lab 13(2)
	Nalimov test outlier	α = 0.05	Lab 13(2)
		α = 0.01	Lab 13(2)
	Grubbs test outlier	α = 0.05	Lab 13(2)
		α = 0.01	Lab 13(2)
Testing for outlying Lab variances	Cochran test outlier	α = 0.05	Lab 13(2)
		α = 0.01	---
Normality of Lab means distribution:	Kolmogorov-Smirnov-Lilliefors test	α = 0.05	not normal
		α = 0.01	not normal
	Skewness & Kurtosis test	α = 0.05	not normal
		α = 0.01	not normal
Snedecor F-test and Bartlett test show that pooling is:			not allowed
Decision concluded			Run 1 accepted, the outlier Lab 13(2) was not removed

* 1 from 8 datasets submitted was not included into the statistical evaluation (cf. Table B.23)

Tab. C.24: Outcome of statistical tests on results obtained for Si

Element			Si
Run of evaluation program			Run 1
Number of data sets			11*
Testing for outlying Lab means	Dixon test outlier	α = 0.05	---
		α = 0.01	---
	Nalimov test outlier	α = 0.05	---
		α = 0.01	---
	Grubbs test outlier	α = 0.05	---
		α = 0.01	---
Testing for outlying Lab variances	Cochran test outlier	α = 0.05	---
		α = 0.01	---
Normality of Lab means distribution:	Kolmogorov-Smirnov-Lilliefors test	α = 0.05	normal
		α = 0.01	normal
	Skewness & Kurtosis test	α = 0.05	normal
		α = 0.01	normal
Snedecor F-test and Bartlett test show that pooling is:			not allowed
Decision concluded			Run 1 accepted

* 1 from 12 datasets submitted was not included into the statistical evaluation (cf. Table B.24)

Tab. C.25: Outcome of statistical tests on results obtained for Sr

Element			Sr
Run of evaluation program			Run 1
Number of data sets			7*
Testing for outlying Lab means	Dixon test outlier	α = 0.05	Lab 22
		α = 0.01	---
	Nalimov test outlier	α = 0.05	Lab 22
		α = 0.01	Lab 22
	Grubbs test outlier	α = 0.05	Lab 22
		α = 0.01	Lab 22
Testing for outlying Lab variances	Cochran test outlier	α = 0.05	Lab 22
		α = 0.01	Lab 22
Normality of Lab means distribution:	Kolmogorov-Smirnov-Lilliefors test	α = 0.05	not normal
		α = 0.01	normal
	Skewness & Kurtosis test	α = 0.05	not normal
		α = 0.01	not normal
Snedecor F-test and Bartlett test show that pooling is:			not allowed
Decision concluded			Outlier Lab 22 removed

* 1 from 8 datasets submitted was not included into the statistical evaluation (cf. Table B.25)

(continued)

Element			Sr
Run of evaluation program			Run 2 after removal of Lab 22
Number of data sets			6
Testing for outlying Lab means	Dixon test outlier	α = 0.05	Lab 26
		α = 0.01	---
	Nalimov test outlier	α = 0.05	Lab 26
		α = 0.01	Lab 26
	Grubbs test outlier	α = 0.05	Lab 26
		α = 0.01	---
Testing for outlying Lab variances	Cochran test outlier	α = 0.05	---
		α = 0.01	---
Normality of Lab means distribution:	Kolmogorov-Smirnov-Lilliefors test	α = 0.05	normal
		α = 0.01	normal
	Skewness & Kurtosis test	α = 0.05	Insufficient data
		α = 0.01	Insufficient data
Snedecor F-test and Bartlett test show that pooling is:			not allowed
Decision concluded			Run 2 accepted, the outlier Lab 26 was not removed

Tab. C.26: Outcome of statistical tests on results obtained for Ti

Element			Ti
Run of evaluation program			Run 1
Number of data sets			9*
Testing for outlying Lab means	Dixon test outlier	α = 0.05	---
		α = 0.01	---
	Nalimov test outlier	α = 0.05	Lab 26
		α = 0.01	---
	Grubbs test outlier	α = 0.05	---
		α = 0.01	---
Testing for outlying Lab variances	Cochran test outlier	α = 0.05	Lab 24, Lab 26
		α = 0.01	Lab 24, Lab 26
Normality of Lab means distribution:	Kolmogorov-Smirnov-Lilliefors test	α = 0.05	not normal
		α = 0.01	not normal
	Skewness & Kurtosis test	α = 0.05	not normal
		α = 0.01	normal
Snedecor F-test and Bartlett test show that pooling is:			not allowed
Decision concluded			Run 1 accepted, the outlier Lab 26 was not removed

* 3 from 12 datasets submitted were not included into the statistical evaluation (cf. Table B.26)

Tab. C.27: Outcome of statistical tests on results obtained for V

Element			V
Run of evaluation program			Run 1
Number of data sets			10*
Testing for outlying Lab means	Dixon test outlier	α = 0.05	Lab 31
		α = 0.01	Lab 31
	Nalimov test outlier	α = 0.05	Lab 31
		α = 0.01	Lab 31
	Grubbs test outlier	α = 0.05	Lab 31
		α = 0.01	Lab 31
	Paired Grubbs test outliers	α = 0.05	Lab 31
		α = 0.01	Lab 31
Testing for outlying Lab variances	Cochran test outlier	α = 0.05	Lab 31
		α = 0.01	Lab 31
Normality of Lab means distribution:	Kolmogorov-Smirnov-Lilliefors test	α = 0.05	not normal
		α = 0.01	not normal
	Skewness & Kurtosis test	α = 0.05	not normal
		α = 0.01	not normal
Snedecor F-test and Bartlett test show that pooling is:			not allowed
Decision concluded			Outlier Lab 31 removed

* 2 from 12 datasets submitted were not included into the statistical evaluation (cf. Table B.27)

(continued)

Element			V
Run of evaluation program			Run 2 after removal of Lab 31
Number of data sets			9
Testing for outlying Lab means	Dixon test outlier	α = 0.05	---
		α = 0.01	---
	Nalimov test outlier	α = 0.05	Lab 24
		α = 0.01	---
	Grubbs test outlier	α = 0.05	---
		α = 0.01	---
Testing for outlying Lab variances	Cochran test outlier	α = 0.05	---
		α = 0.01	---
Normality of Lab means distribution:	Kolmogorov-Smirnov-Lilliefors test	α = 0.05	normal
		α = 0.01	normal
	Skewness & Kurtosis test	α = 0.05	normal
		α = 0.01	normal
Snedecor F-test and Bartlett test show that pooling is:			not allowed
Decision concluded			Run 2 accepted, the outlier Lab 24 was not removed

Tab. C.28: Outcome of statistical tests on results obtained for W

Element			W
Run of evaluation program			Run 1
Number of data sets			8*
Testing for outlying Lab means	Dixon test outlier	α = 0.05	---
		α = 0.01	---
	Nalimov test outlier	α = 0.05	---
		α = 0.01	---
	Grubbs test outlier	α = 0.05	---
		α = 0.01	---
Testing for outlying Lab variances	Cochran test outlier	α = 0.05	Lab 16, Lab 11
		α = 0.01	Lab 16, Lab 11
Normality of Lab means distribution:	Kolmogorov-Smirnov-Lilliefors test	α = 0.05	normal
		α = 0.01	normal
	Skewness & Kurtosis test	α = 0.05	normal
		α = 0.01	normal
Snedecor F-test and Bartlett test show that pooling is:			not allowed
Decision concluded			Run 1 accepted

* 1 from 9 datasets submitted was not included into the statistical evaluation (cf. Table B.28)

Tab. C.29: Outcome of statistical tests on results obtained for Y

Element			Y
Run of evaluation program			Run 1
Number of data sets			6
Testing for outlying Lab means	Dixon test outlier	α = 0.05	---
		α = 0.01	---
	Nalimov test outlier	α = 0.05	---
		α = 0.01	---
	Grubbs test outlier	α = 0.05	---
		α = 0.01	---
Testing for outlying Lab variances	Cochran test outlier	α = 0.05	---
		α = 0.01	---
Normality of Lab means distribution:	Kolmogorov-Smirnov-Lilliefors test	α = 0.05	---
		α = 0.01	---
	Skewness & Kurtosis test	α = 0.05	Insufficient data
		α = 0.01	Insufficient data
Snedecor F-test and Bartlett test show that pooling is:			not allowed
Decision concluded			Run 1 accepted

* 1 from 7 datasets submitted was not included into the statistical evaluation (cf. Table B.29)

Tab. C.30: Outcome of statistical tests on results obtained for Zn

Element			Zn
Run of evaluation program			Run 1
Number of data sets			14*
Testing for outlying Lab means	Dixon test outlier	α = 0.05	---
		α = 0.01	---
	Nalimov test outlier	α = 0.05	Lab 31
		α = 0.01	Lab 31
	Grubbs test outlier	α = 0.05	Lab 31
		α = 0.01	---
Testing for outlying Lab variances	Cochran test outlier	α = 0.05	Lab 24, Lab 27, Lab 3, Lab 31
		α = 0.01	Lab 24, Lab 27
Normality of Lab means distribution:	Kolmogorov-Smirnov-Lilliefors test	α = 0.05	not normal
		α = 0.01	not normal
	Skewness & Kurtosis test	α = 0.05	not normal
		α = 0.01	not normal
Snedecor F-test and Bartlett test show that pooling is:			not allowed
Decision concluded			Outlier Lab 31 removed

* 1 from 15 datasets submitted were not included into the statistical evaluation (cf. Table B.30)

(continued)

Element			Zn
Run of evaluation program			Run 2 after removal of Lab 31
Number of data sets			13
Testing for outlying Lab means	Dixon test outlier	α = 0.05	---
		α = 0.01	---
	Nalimov test outlier	α = 0.05	Lab 27
		α = 0.01	Lab 27
	Grubbs test outlier	α = 0.05	Lab 27
		α = 0.01	---
Testing for outlying Lab variances	Cochran test outlier	α = 0.05	Lab 24, Lab 27, Lab 3
		α = 0.01	Lab 24, Lab 27, Lab 3
Normality of Lab means distribution:	Kolmogorov-Smirnov-Lilliefors test	α = 0.05	not normal
		α = 0.01	not normal
	Skewness & Kurtosis test	α = 0.05	not normal
		α = 0.01	not normal
Snedecor F-test and Bartlett test show that pooling is:			not allowed
Decision concluded			Run 2 accepted, the outlier Lab 27 was not removed

Tab. C.31: Outcome of statistical tests on results obtained for Zr

Element			Zr
Run of evaluation program			Run 1
Number of data sets			8*
Testing for outlying Lab means	Dixon test outlier	α = 0.05	---
		α = 0.01	---
	Nalimov test outlier	α = 0.05	Lab 1
		α = 0.01	---
	Grubbs test outlier	α = 0.05	---
		α = 0.01	---
Testing for outlying Lab variances	Cochran test outlier	α = 0.05	---
		α = 0.01	---
Normality of Lab means distribution:	Kolmogorov-Smirnov-Lilliefors test	α = 0.05	normal
		α = 0.01	normal
	Skewness & Kurtosis test	α = 0.05	normal
		α = 0.01	normal
Snedecor F-test and Bartlett test show that pooling is:			not allowed
Decision concluded			Run 1 accepted, the outlier Lab 1 was not removed

* 4 from 12 datasets submitted were not included into the statistical evaluation (cf. Table B.31)