Certification Report

Certified Reference Material

BAM-M505a

Electronic Scrap

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Summary

This report describes preparation, analysis and certification of the electronic scrap reference material BAM-M505a.
The certified reference material BAM-M505a is intended for the development, validation and quality control of analytical methods and procedures for the determination of main and trace components in electronic waste.
Certified reference material BAM-M505a is available as powder with a particle size < 150 µm.

The following mass fractions and uncertainties have been certified:

Element	Mass fraction [1)] in %	Uncertainty [2)] in %
Cu	16.76	0.04
Ni	0.694	0.008
Ag	0.0633	0.0008
Pb	1.13	0.05
Cr	0.980	0.017
Sn	0.469	0.015
	in mg/kg	**in mg/kg**
Au	52.4	0.9
Pd	48.0	0.8
Pt	5.7	0.4
As	372	20
Be	6.8	0.9
Cd	16.4	0.7

1) Unweighted mean value of the means of accepted sets of data (consisting of at least 5 but usually 6 single results), each set being obtained by a different laboratory and/or a different method of measurement.

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

This report contains detailed information on the preparation of the CRM as well as on homogeneity investigations and on the analytical methods used for certification analysis.
The certified values are based on the results of 15 laboratories which participated in the certification interlaboratory comparison. The mass fractions for In and Hg are given for information.

Element	Mass fraction in mg/kg	Uncertainty in mg/kg
In	43	6
Hg	< 5	

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List of abbreviations
(if not explained elsewhere)

CRM	certified reference material
CVAAS	cold vapour atomic absorption spectrometry
ETAAS	electrothermal atomic absorption spectrometry
FAAS	flame atomic absorption spectrometry
ICP-OES	inductively coupled plasma optical emission spectrometry
ICP-MS	inductively coupled plasma mass spectrometry
LIBS	laser-induced breakdown spectroscopy
SOES	spark optical emission spectrometry
XRF	X-ray fluorescence spectrometry
M	mean value
n	number of accepted data sets
s	standard deviation of an individual data set
s_M	standard deviation of laboratory means
s_{rel}	relative standard deviation
s_{ilc}	standard deviation from inter-laboratory comparison
$\bar{s}_i$	square root of mean of variances of data sets under repeatability conditions
M_i	single result

1. Introduction

Printed circuit boards are a source for precious metals of economic interest. The price for used printed circuit boards is related to their precious metals content and on the current price for metals like silver, gold, platinum and palladium. Resulting from the high price for precious metals, an accurate determination of these elements is necessary. To ensure this, certified reference materials either for calibration and quality control are required.

The idea to produce a reference material based on electronic scrap originally was the outcome of the discussions within the German Gesellschaft der Metallurgen und Bergleute (GDMB) and especially the working group „Precious Metals“ of the committee of chemists within GDMB. BAM-M505a replaces ERM-EB505 which is sold out and no longer available. From this working group the participating laboratories are recruited. Since all the laboratories are highly experienced with precious metals analysis there was no preceding round robin test for qualification. Most of the participants had an accreditation or a certification according to ISO 17025 or 9001.

Certification of reference material BAM-M505a was carried out on the basis of ISO 17034 [1] and the relevant ISO-Guides [2, 3].
Starting material for the preparation of CRM BAM-M505a were approx. 70 kg of used mixed printed circuit boards.

2. Companies/laboratories involved

Preparation of the material:
Institut für Materialprüfung Glörfeld GmbH, Willich (Germany)

Test for homogeneity:

- Bundesanstalt für Materialforschung und -prüfung (BAM)
- Participating laboratories in connection with characterisation of material

Participants in the certification interlaboratory comparison:

- Alfred Knight Int. Ltd, St. Helens (United Kingdom)
- Allgemeine Gold- und Silberscheideanstalt AG, Pforzheim (Germany)
- ALS Minerals Division, Prescot (United Kingdom)
- AnRec GmbH & Co. KG, Gelnhausen (Germany)
- Aurubis AG, Hamburg (Germany)
- Bundesanstalt für Materialforschung und -prüfung (BAM), Berlin (Germany)
- Forschungsinstitut Edelmetalle & Metallchemie, Schwäbisch Gmünd (Germany)
- Inspectorate International Ltd, Shanghai (China)
- Inspectorate International Ltd, Witham (United Kingdom)
- Institut für Materialprüfung Glörfeld GmbH, Willich (Germany)
- Ledoux & Company, Teanec NJ (United States)
- Petrographisches Labor, Seulingen (Germany)
- Umicore Precious Metals, Hoboken (Belgium)
- W.C. Heraeus GmbH, Hanau (Germany)
- WRC World Resources Company GmbH, Wurzen (Germany)

Statistical evaluation of the data

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

3. Candidate material

Approx. 70 kg of used mixed printed circuit boards were taken as initial material. These boards were doped with Be, In and Pt, ashed and melted with approx. 50 kg of pyrite (FeS_2). After milling and grinding the material was sieved to a particle size below 150 µm and homogenised thoroughly. The material was filled into 10 plastic containers. From each of these 10 containers three samples were taken for homogeneity testing.
The material was then bottled in 200 ml amber glass containers each filled with 200 g of material. The bottles were sealed with screw caps equipped with PE insert and with shrinking foil. In total 600 bottles were filled.

4. Homogeneity testing

All 30 samples taken for homogeneity testing were analysed using XRF. This method was only suitable for the elements Cu, Ag, Ni, Cr, Sn, and Pb. For all other elements XRF was not sensitive enough to determine their contents. The homogeneity contribution to the total uncertainty was calculated using a 1-way-ANOVA (see Annex 1).
For homogeneity investigation of the elements Au, Pd, Pt, As, Be, Cd and In all participating laboratories received two bottles of material from different containers. They were asked to carry out three determinations from each of the two bottles. With this data the homogeneity contribution to the total uncertainty was calculated using a 2-way-ANOVA (see Annex 1). In cases, where $s_{within} > s_{between}$ the inhomogeneity contribution to the combined uncertainty is set to zero.

5. Stability

There is no instability of the certified material to be expected (calcinated inorganic material) if the material is stored at ambient temperature. From the CRM ERM-EZ505, which was produced in the same way as BAM-M505a, there is no hint to any instability of the material.

6. Certification study

6.1 Analytical methods

15 laboratories participated in the certification interlaboratory comparison. Each laboratory received two randomly chosen bottles with approx. 80 g of powder.
The laboratories were told to analyse six subsamples, three from each of the two bottles. They were free to choose any suitable analytical method for analysis. Tables 1 to 14 show the analytical methods used by the participating laboratories.
For all analytical methods where a calibration was necessary this calibration was performed using liquid standard solutions. All participating laboratories were asked to use only standard solutions prepared from pure metals or stoichiometric compounds or well checked commercial calibration solutions.

Table 1: Analytical procedures for the determination of copper

Lab-No.	Sample mass	Sample pretreatment	Analytical method
1	0.25 g	$HCl/HNO_3/HBF_4$/microwave	ICP-OES, calibration with commercial monoelement standard solution
3	0.2 g	Dissolution with aqua regia	ICP-OES, calibration with gravimetrically prepared monoelement standard solution (Cu 99.99 %)
4	0.5 g	Dissolution with acid, thiocyanate separation	ICP-OES, matrix matched calibration with commercial monoelement standard solution
5	5 g	Dissolution with HNO_3/H_2SO_4	Electrogravimetry
6	0.5 g	Dissolution with aqua regia, microwave	ICP-OES, calibration with gravimetrically prepared monoelement standard solution (Cu 99.999 %) with Sc as internal standard
7	2 g	Dissolution with aqua regia, Na_2O_2	ICP-OES, calibration with commercial monoelement standard solution with Sc as internal standard
8	4 g	Dissolution with HNO_3/H_2SO_4	Electrogravimetry
9	n.a.	Acid digestion	Electrogravimetry
10	2-2.5 g	Dissolution with aqua regia/H_2SO_4/HF	Electrogravimetry
11	0.5 g/ 5 g	Alkaline fusion with Na_2O_2, leaching with HCl	ICP-OES, calibration with gravimetrically prepared monoelement standard solution
13	n.a.	Acid digestion, precipitation of interferences	Electrogravimetry
15	2 g	Dissolution in $HBr/Br_2/H_2SO_4$, separation of Ag with NaCl	Electrogravimetry
16	0.2 g	Dissolution with aqua regia	ICP-OES, calibration with gravimetrically prepared monoelement standard solution (Cu metal)

Table 2: Analytical procedures for the determination of nickel

Lab-No.	Sample mass	Sample pretreatment	Analytical method
1	0.25 g	$HCl/HNO_3/HBF_4$/microwave	ICP-OES, calibration with commercial monoelement standard solution
3	0.2 g	Dissolution with aqua regia	ICP-OES, calibration with gravimetrically prepared monoelement standard solution (Ni 99.99 %)
5	2.5 g	Dissolution with HCl/HNO_3, decomposition of residue with Na_2O_2	ICP-OES
6	0.5 g	Dissolution with aqua regia, microwave	ICP-OES, calibration with gravimetrically prepared monoelement standard solution (Ni 99.999 %) with Sc as internal standard
7	2 g	Dissolution with aqua regia, Na_2O_2	ICP-OES, calibration with commercial monoelement standard solution with Sc as internal standard
8	2 g	Dissolution with HNO_3/H_2SO_4/HCl	ICP-OES, calibration with commercial monoelement standard solution
9	n.a.	Fusion with Na_2O_2	ICP-OES
10	0.5 g	Alkaline fusion, dissolution with HCl	ICP-OES, calibration with commercial monoelement standard solution
13	n.a.	Acid digestion	ICP-OES, calibration with commercial monoelement standard solution
15	0.5 g	Fusion with Na_2O_2, dissolution with HCl	ICP-OES, matrix matched calibration with commercial monoelement standard solution
16	0.5 g	Dissolution with aqua regia	ICP-OES, matrix matched calibration with commercial standard solution

Table 3: Analytical procedures for the determination of silver

Lab-No.	Sample mass	Sample pretreatment	Analytical method
1	5 g	Melting with PbO-mix and decanting	SOES, calibration with self-prepared calibration samples
3	2.5 g	Dissolution with aqua regia	ICP-OES, calibration with gravimetrically prepared monoelement standard solution (Ag 99.99 %)
4	2.5 g	Fire assay, palladium collection	ICP-OES, matrix matched calibration with commercial monoelement standard solution
5	10 g	Fire assay, lead collection	ICP-OES
6	2 g	Fire assay, palladium collection	ICP-OES, calibration with gravimetrically prepared monoelement standard (Ag 99.99 %) with Sc as internal standard
7	0.5 g	Dissolution with aqua regia, microwave	ICP-OES, calibration with commercial monoelement standard solution with Y as internal standard
8	7.5 g	Fire assay, lead collection	ICP-OES, calibration with gravimetrically prepared monoelement standard solution (Ag 99.999 %)
9	n.a.	Fire assay	Gravimetry
10	5 g	Fire assay, lead collection	Gravimetry
11	0.5 g/ 5 g	Alkaline fusion with Na_2O_2, leaching with HCl	ICP-OES, calibration with gravimetrically prepared monoelement standard solution
13	n.a.	Alkaline fusion with Na_2O_2	FAAS, matrix matched calibration with commercial monoelement standard solution
15	7.5 g	Fire assay, lead collection	ICP-OES, calibration with gravimetrically prepared monoelement standard solution (Ag metal) with Y, Sc as internal standard
16	0.5 g	Dissolution with aqua regia	ICP-OES, matrix matched calibration with commercial standard solution

Table 4: Analytical procedures for the determination of lead

Lab-No.	Sample mass	Sample pretreatment	Analytical method
1	0.25 g	$HCl/HNO_3/HBF_4$/microwave	ICP-OES, calibration with commercial monoelement standard solution
3	0.2 g	Dissolution with aqua regia	ICP-OES, matrix matched calibration with commercial monoelement standard solution
5	2.5 g	Dissolution with HCl/HNO_3, decomposition of residue with Na_2O_2	ICP-OES
6	0.5 g	Dissolution with aqua regia, microwave	ICP-OES, calibration with gravimetrically prepared monoelement standard solution
7	2 g	Dissolution with aqua regia, Na_2O_2	ICP-OES, calibration with commercial monoelement standard solution with Sc as internal standard
8	1 g	Acid digestion	ICP-OES, calibration with gravimetrically prepared monoelement standard solution (Pb 99.999 %)
9	n.a.	Fusion with Na_2O_2	ICP-OES
10	0.5 g	Alkaline fusion, dissolution with HCl	ICP-OES, calibration with commercial monoelement standard solution
13	n.a.	Acid digestion	ICP-OES, matrix matched calibration with commercial monoelement standard solution
15	0.5 g	Fusion with Na_2O_2, dissolution with HCl	ICP-OES, matrix matched calibration with commercial monoelement standard solution
16	0.5 g	Dissolution with aqua regia	ICP-OES, matrix matched calibration with commercial standard solution

Table 5: Analytical procedures for the determination of chromium

Lab-No.	Sample mass	Sample pretreatment	Analytical method
1	0.25 g	$HCl/HNO_3/HBF_4$/microwave	ICP-OES, calibration with commercial monoelement standard solution
3	0.2 g	Dissolution with aqua regia	ICP-OES, matrix matched calibration with commercial monoelement standard solution
5	2.5 g	Dissolution with HCl/HNO_3, decomposition of residue with Na_2O_2	ICP-OES
7	2 g	Dissolution with aqua regia, Na_2O_2	ICP-OES, calibration with commercial monoelement standard solution with Y as internal standard
8	2 g	Dissolution with $HNO_3/H_2SO_4/HCl$	ICP-OES, calibration with commercial monoelement standard solution
9	n.a.	Fusion with Na_2O_2	ICP-OES
10	0.5 g	Alkaline fusion, dissolution with HCl	ICP-OES, calibration with commercial monoelement standard solution
13	n.a.	Peroxide fusion	ICP-OES, matrix matched calibration with commercial monoelement standard solution
15	0.5 g	Fusion with Na_2O_2, dissolution with HCl	ICP-OES, matrix matched calibration with commercial monoelement standard solution

Table 6: Analytical procedures for the determination of tin

Lab-No.	Sample mass	Sample pretreatment	Analytical method
1	0.25 g	$HCl/HNO_3/HBF_4$/microwave	ICP-OES, calibration with commercial monoelement standard solution
3	0.2 g	Dissolution with aqua regia	ICP-OES, matrix matched calibration with commercial monoelement standard solution
5	2.5 g	Dissolution with HCl/HNO_3, decomposition of residue with Na_2O_2	ICP-OES
6	0.5 g	Dissolution with aqua regia, microwave	ICP-OES, calibration with gravimetrically prepared monoelement standard solution (Sn 99.999 %) with Sc as internal standard
7	2 g	Dissolution with aqua regia, Na_2O_2	ICP-OES, calibration with commercial monoelement standard solution with Sc as internal standard
8	2 g	Dissolution with $HNO_3/H_2SO_4/HCl$	ICP-OES, calibration with commercial monoelement standard solution
9	n.a.	Fusion with Na_2O_2	ICP-OES
10	0.5 g	Alkaline fusion, dissolution with HCl	ICP-OES, calibration with commercial monoelement standard solution
13	n.a.	Peroxide fusion	ICP-OES, matrix matched calibration with commercial monoelement standard solution
15	0.5 g	Fusion with Na_2O_2, dissolution with HCl	ICP-OES, matrix matched calibration with commercial monoelement standard solution
16	0.5 g	Dissolution with aqua regia	ICP-OES, matrix matched calibration with commercial standard solution

Table 7: Analytical procedures for the determination of gold

Lab-No.	Sample mass	Sample pretreatment	Analytical method
1	2.5 g	Melting with PbO-mix and Ag, cupellation, dissolving	ICP-OES, calibration with gravimetrically prepared monoelement standard solution
3	3 g	Fire assay with lead	ICP-OES, calibration with gravimetrically prepared monoelement standard solution (Au 99.99 %)
4	2.5 g	Fire assay, palladium collection	ICP-OES, matrix matched calibration with commercial monoelement standard
5	10 g	Fire assay	ICP-OES
6	2 g	Fire assay, silver collection	ICP-OES, calibration with gravimetrically prepared monoelement standard solution (Au 99.999 %) with Sc as internal standard
8	7.5 g	Fire assay, lead collection	ICP-OES, calibration with gravimetrically prepared monoelement standard solution (Au 99.99 %)
9	n.a.	Fire assay	Gravimetry
10	5 g	Fire assay, lead collection	Gravimetry
11	0.5 g/ 5 g	Fire assay with PbO, dissolution in aqua regia	ICP-OES, calibration with gravimetrically prepared monoelement standard solution
13	n.a.	Fire assay	Gravimetry
15	7.5 g	Fire assay, silver collection	ICP-OES, calibration with gravimetrically prepared monoelement standard solution (Au metal) with Y, Sc as internal standard
16	1 g	Fire assay, collection with lead	ICP-OES, matrix matched calibration with commercial standard solution

Table 8: Analytical procedures for the determination of palladium

Lab-No.	Sample mass	Sample pretreatment	Analytical method
1	2.5 g	Melting with PbO-mix and Ag, cupellation, dissolving	ICP-OES, calibration with gravimetrically prepared monoelement standard solution
3	3 g	Fire assay with lead	ICP-OES, calibration with gravimetrically prepared monoelement standard solution (Pd 99.99 %)
4	2.5 g	Fire assay, gold collection	ICP-OES, matrix matched calibration with commercial monoelement standard solution
5	10 g	Fire assay	ICP-OES
6	2 g	Fire assay, silver collection	ICP-OES, calibration with gravimetrically prepared monoelement standard solution (Pd 99.97 %) with Sc as internal standard
8	7.5 g	Fire assay, lead collection	ICP-OES, calibration with gravimetrically prepared monoelement standard solution (Pd 99.99 %)
9	n.a.	Fire assay	ICP-OES
10	5 g	Fire assay, lead collection	ICP-OES, calibration with commercial monoelement standard solution
11	0.5 g/ 5 g	Fire assay with PbO, dissolution in aqua regia	ICP-OES, calibration with gravimetrically prepared monoelement standard solution
13	n.a.	Fire assay	ICP-OES, matrix matched calibration with commercial monoelement standard solution
15	7.5 g	Fire assay, silver collection	ICP-OES, calibration with gravimetrically prepared monoelement standard solution (Pd metal) with Y, Sc as internal standard
16	1 g	Fire assay, collection with lead	ICP-OES, matrix matched calibration with commercial standard solution

Table 9: Analytical procedures for the determination of platinum

Lab-No.	Sample mass	Sample pretreatment	Analytical method
1	2.5 g	Melting with PbO-mix and Ag, cupellation, dissolving	ICP-OES, calibration with gravimetrically prepared monoelement standard solution
3	3 g	Fire assay with lead	ICP-OES, calibration with gravimetrically prepared monoelement standard solution (Pt 99.99 %)
4	2.5 g	Fire assay, gold collection	ICP-OES, matrix matched calibration with commercial monoelement standard solution
5	10 g	Fire assay	ICP-OES
6	2 g	Fire assay, gold collection	ICP-OES, calibration with gravimetrically prepared monoelement standard solution (Pt 99.98 %)
8	7.5 g	Fire assay, lead collection	ICP-OES, calibration with gravimetrically prepared monoelement standard solution (Pt 99.99 %)
9	n.a.	Fire assay	ICP-OES
10	5 g	Fire assay, lead collection	ICP-OES, calibration with commercial monoelement standard solution
11	0.5 g/ 5 g	Fire assay with PbO, dissolution in aqua regia	ICP-OES, calibration with gravimetrically prepared monoelement standard solution
13	n.a.	Fire assay	ICP-OES, matrix matched calibration with commercial monoelement standard solution
15	7.5 g	Fire assay, silver collection	ICP-OES, calibration with gravimetrically prepared monoelement standard solution (Pt metal) with Y, Sc as internal standard
16	1 g	Fire assay, collection with lead	ICP-OES, matrix matched calibration with commercial standard solution

Table 10: Analytical procedures for the determination of arsenic

Lab-No.	Sample mass	Sample pretreatment	Analytical method
1	0.25 g	$HCl/HNO_3/HBF_4$/microwave	ICP-OES, calibration with commercial monoelement standard solution
3	1 g	Dissolution with aqua regia	ICP-OES, matrix matched calibration with commercial monoelement standard solution
5	2.5 g	Dissolution with HCl/HNO_3, decomposition of residue with Na_2O_2	ICP-OES, matrix matched calibration with commercial monoelement standard solution
	1 g	Dissolution with HCl/HNO_3, decomposition of residue with Na_2O_2	ICP-MS, matrix matched calibration with commercial monoelement standard solution
6	0.5 g	Dissolution with aqua regia, microwave	ICP-OES, calibration with gravimetrically prepared monoelement standard solution with Sc as internal standard
7	0.5 g	Dissolution with aqua regia, microwave	ICP-OES, calibration with commercial monoelement standard solution with Y as internal standard
9	n.a.	Acid digestion	ICP-OES
10	0.5 g	Alkaline fusion, dissolution with HCl	ICP-OES, calibration with commercial monoelement standard solution
13	n.a.	Acid digestion	ICP-OES, matrix matched calibration with commercial monoelement standard solution
15	2 g	Dissolution with HNO_3/HF, fuming of the residue with H_2SO_4	ICP-OES, matrix matched calibration with commercial monoelement standard solution
16	0.5 g	Dissolution with aqua regia	ICP-OES, matrix matched calibration with commercial standard solution
20	0.5 g	Dissolution with aqua regia, microwave	ICP-OES, matrix matched calibration with commercial monoelement standard solution

Table 11: Analytical procedures for the determination of beryllium

Lab-No.	Sample mass	Sample pretreatment	Analytical method
1	0.25 g	$HCl/HNO_3/HBF_4$/microwave	ICP-OES, calibration with commercial monoelement standard solution
3	1 g	Dissolution with aqua regia	ICP-OES, matrix matched calibration with commercial monoelement standard solution
5	2.5 g	Dissolution with HCl/HNO_3, decomposition of residue with Na_2O_2	ICP-OES, matrix matched calibration with commercial monoelement standard solution
	1 g	Dissolution with HCl/HNO_3, decomposition of residue with Na_2O_2	ICP-MS, matrix matched calibration with commercial monoelement standard solution
6	0.5 g	Dissolution with aqua regia, microwave	ICP-OES, calibration with gravimetrically prepared monoelement standard solution with Sc as internal standard
7	0.5 g	Dissolution with aqua regia, microwave	ICP-OES, calibration with commercial monoelement standard solution with Y as internal standard
9	n.a.	Acid digestion	ICP-OES
10	0.5 g	Alkaline fusion, dissolution with HCl	ICP-OES, calibration with commercial monoelement standard solution
15	2 g	Dissolution with HNO_3/HF, fuming of the residue with H_2SO_4	ICP-OES, matrix matched calibration with commercial monoelement standard solution
16	0.5 g	Dissolution with aqua regia	ICP-OES, matrix matched calibration with commercial standard solution
20	0.5 g	Dissolution with aqua regia, microwave	ICP-OES, matrix matched calibration with commercial monoelement standard solution

Table 12: Analytical procedures for the determination of cadmium

Lab-No.	Sample mass	Sample pretreatment	Analytical method
1	0.25 g	$HCl/HNO_3/HBF_4$/microwave	ICP-MS, calibration with commercial monoelement standard solution
3	1 g	Dissolution with aqua regia	ICP-OES, matrix matched calibration with commercial monoelement standard solution
5	2.5 g	Dissolution with HCl/HNO_3, decomposition of residue with Na_2O_2	ICP-OES, matrix matched calibration with commercial monoelement standard solution
	1 g	Dissolution with HCl/HNO_3, decomposition of residue with Na_2O_2	ICP-MS, matrix matched calibration with commercial monoelement standard solution
6	0.5 g	Dissolution with aqua regia, microwave	ICP-OES, calibration with gravimetrically prepared monoelement standard solution with Sc as internal standard
7	0.5 g	Dissolution with aqua regia, microwave	ICP-OES, calibration with commercial monoelement standard solution with Y as internal standard
9	n.a.	Fusion with Na_2O_2	ICP-OES
10	0.5 g	Alkaline fusion, dissolution with HCl	ICP-OES, calibration with commercial monoelement standard solution
13	n.a.	Acid digestion	ICP-OES, matrix matched calibration with commercial monoelement standard solution
15	2 g	Dissolution with HNO_3/HF, fuming of the residue with H_2SO_4	ICP-OES, matrix matched calibration with commercial monoelement standard solution
16	0.5 g	Dissolution with aqua regia	ICP-OES, matrix matched calibration with commercial standard solution
20	0.5 g	Dissolution with aqua regia, microwave	ICP-OES, matrix matched calibration with commercial monoelement standard solution

Table 13: Analytical procedures for the determination of indium

Lab-No.	Sample mass	Sample pretreatment	Analytical method
1	0.25 g	$HCl/HNO_3/HBF_4$/microwave	ICP-OES, calibration with commercial monoelement standard solution
3	1 g	Dissolution with aqua regia	ICP-OES, matrix matched calibration with commercial monoelement standard solution
5	2.5 g	Dissolution with HCl/HNO_3, decomposition of residue with Na_2O_2	ICP-OES, matrix matched calibration with commercial monoelement standard solution
	1 g	Dissolution with HCl/HNO_3, decomposition of residue with Na_2O_2	ICP-MS, matrix matched calibration with commercial monoelement standard solution
6	0.5 g	Dissolution with aqua regia, microwave	ICP-OES, calibration with gravimetrically prepared monoelement standard solution with Sc as internal standard
7	0.5 g	Dissolution with aqua regia, microwave	ICP-OES, calibration with commercial monoelement standard solution with Y as internal standard
9	n.a.	Acid digestion	ICP-OES
13	n.a.	Acid digestion	ICP-OES, matrix matched calibration with commercial monoelement standard solution
15	2 g	Dissolution with HNO_3/HF, fuming of the residue with H_2SO_4	ICP-OES, matrix matched calibration with commercial monoelement standard solution
16	0.5 g	Dissolution with aqua regia	ICP-OES, matrix matched calibration with commercial standard solution
20	0.5 g	Dissolution with aqua regia, microwave	ICP-OES, matrix matched calibration with commercial monoelement standard solution
22	3 g	Preparation of an epoxy resin tablet	LIBS, calibration with commercial standard

Table 14: Analytical procedures for the determination of mercury

Lab-No.	Sample mass	Sample pretreatment	Analytical method
1	0.25 g	$HCl/HNO_3/HBF_4$/microwave	ICP-OES, calibration with commercial monoelement standard solution
3	1 g	Dissolution with aqua regia	ICP-OES, matrix matched calibration with commercial monoelement standard solution
5	2.5 g	Dissolution with HCl/HNO_3, decomposition of residue with Na_2O_2	ICP-OES, matrix matched calibration with commercial monoelement standard solution
5	1 g	Dissolution with HCl/HNO_3, decomposition of residue with Na_2O_2	ICP-MS, matrix matched calibration with commercial monoelement standard solution
7	0.5 g	Dissolution with aqua regia, microwave	ICP-OES, calibration with commercial monoelement standard solution with Y as internal standard
9	n.a.	Acid digestion	ICP-OES
10	5 g	Acid digestion with HCl/Br_2	ICP-OES, calibration with commercial monoelement standard solution
13	n.a.	Acid digestion	ICP-OES, matrix matched calibration with commercial monoelement standard solution
15	0.2 g	Acid digestion, microwave	Cold vapour AAS, calibration with commercial monoelement standard solution
20	50 mg		Direct mercury analyser

6.2 Analytical results and statistical evaluation

The analytical results of the certification interlaboratory comparison are listed in Tables 15 to 28. These tables show the single results (M_i) of each laboratory, the resp. laboratories' mean values (M) together with the innerlaboratory standard deviation (s) and in addition the mean standard deviation ($\overline{s}$) of all laboratories. The values highlighted in blue are the results from the first bottle, the values highlighted in red are the results from the second bottle. The continuous line marks the certified value (mean od the laboratories' means), the broken lines mark the standard deviation, calculated from the laboratories' means.

In the related figures for each laboratory its mean value and single standard deviation is given.

Table 15: Results for Cu

Lab.	16	7	13	8	5	15	4	10	9	3	11	6	1		
M_i [%]	16.63	16.71	16.735	16.706	16.73	16.745	16.655	16.77	16.79	16.836	16.840	16.808	16.855		*n*
	16.65	16.72	16.763	16.746	16.74	16.740	16.696	16.76	16.79	16.836	16.800	16.899	16.808		13
	16.57	16.75	16.696	16.769	16.80	16.735	16.867	16.80		16.792	16.772	16.808	16.771		
			16.733												
	16.63	16.72	16.713	16.676	16.65	16.735	16.733	16.77	16.83	16.763	16.938	16.903	16.996		
	16.61	16.72	16.773	16.719	16.72	16.740	16.723	16.75	16.79	16.751		16.829	16.768		
	16.62	16.69	16.711	16.786	16.77	16.725	16.850	16.76		16.831		16.826	16.921		
			16.733												
M(B1)	*16.617*	*16.727*	*16.732*	*16.740*	*16.757*	*16.740*	*16.739*	*16.777*	*16.790*	*16.821*	*16.804*	*16.838*	*16.811*		
M(B2)	*16.620*	*16.710*	*16.733*	*16.727*	*16.713*	*16.733*	*16.768*	*16.760*	*16.810*	*16.782*	*16.938*	*16.853*	*16.895*		
M [%]	**16.62**	**16.72**	**16.73**	**16.73**	**16.74**	**16.74**	**16.75**	**16.77**	**16.80**	**16.80**	**16.84**	**16.85**	**16.85**		**16.76**
s [%]	0.027	0.019	0.026	0.041	0.051	0.007	0.086	0.017	0.020	0.038	0.072	0.044	0.091	s_M [%]	0.064
														$\bar{s}_i$ [%]	0.049
s_{rel}	0.0016	0.0012	0.0016	0.0025	0.0030	0.0004	0.0051	0.0010	0.0012	0.0023	0.0043	0.0026	0.0054		0.0038

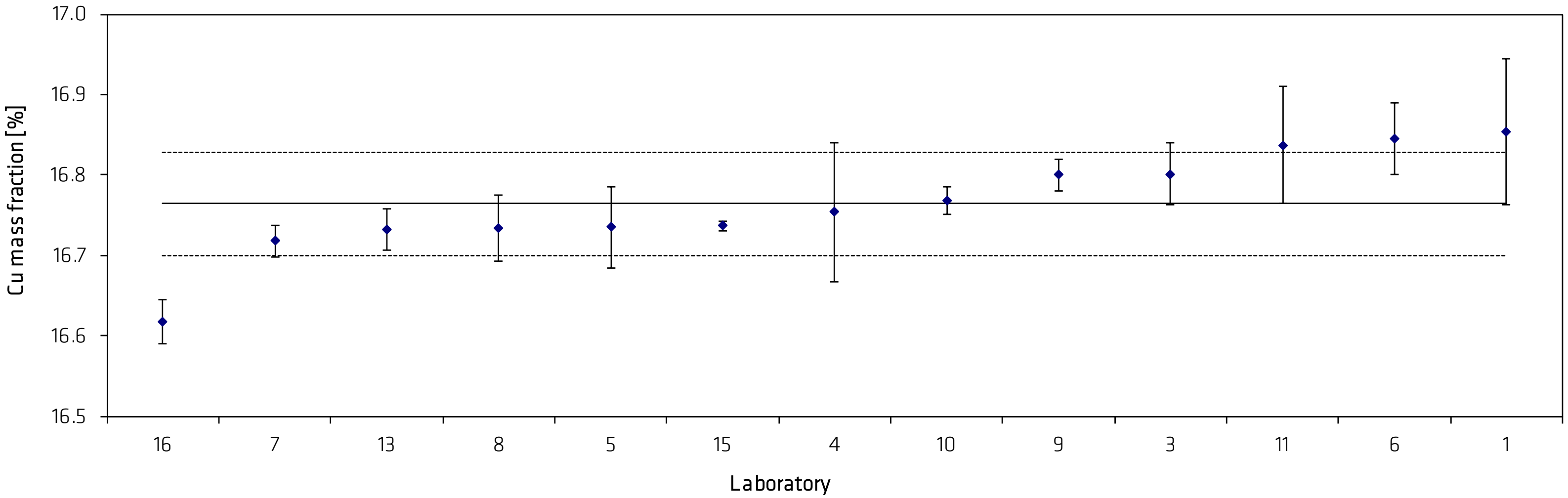


Table 16: Results for Ni

Lab.	6	13	8	16	5	10/R	1	15	9	3	7		
M_i [%]	0.676	0.681	0.689	0.688	0.69	0.69	0.698	0.702	0.703	0.70	0.711		*n*
	0.680	0.703	0.694	0.692	0.70	0.70	0.698	0.688	0.702	0.69	0.704		11
	0.678	0.676	0.688	0.695	0.69	0.69		0.688		0.70	0.708		
	0.675	0.696	0.684	0.699	0.70	0.70	0.699	0.707	0.687	0.70	0.710		
	0.679	0.682	0.684	0.694	0.69	0.69	0.698	0.707	0.703	0.71	0.712		
	0.673	0.649	0.683	0.689	0.69	0.70		0.697		0.71	0.708		
		0.681											
M(B1)	*0.678*	*0.687*	*0.690*	*0.692*	*0.693*	*0.693*	*0.698*	*0.693*	*0.703*	*0.697*	*0.708*		
M(B2)	*0.676*	*0.677*	*0.683*	*0.694*	*0.693*	*0.697*	*0.699*	*0.704*	*0.695*	*0.707*	*0.710*		
***M* [%]**	**0.677**	**0.681**	**0.687**	**0.693**	**0.693**	**0.695**	**0.698**	**0.698**	**0.699**	**0.702**	**0.709**		**0.694**
s [%]	0.0026	0.0171	0.0043	0.0041	0.0052	0.0055	0.0006	0.0087	0.0078	0.0075	0.0029	s_M [%]	0.0092
												$\bar{s}_i$ [%]	0.0073
s_{rel}	0.0039	0.0251	0.0062	0.0059	0.0074	0.0079	0.0008	0.0125	0.0112	0.0107	0.0040		0.0133

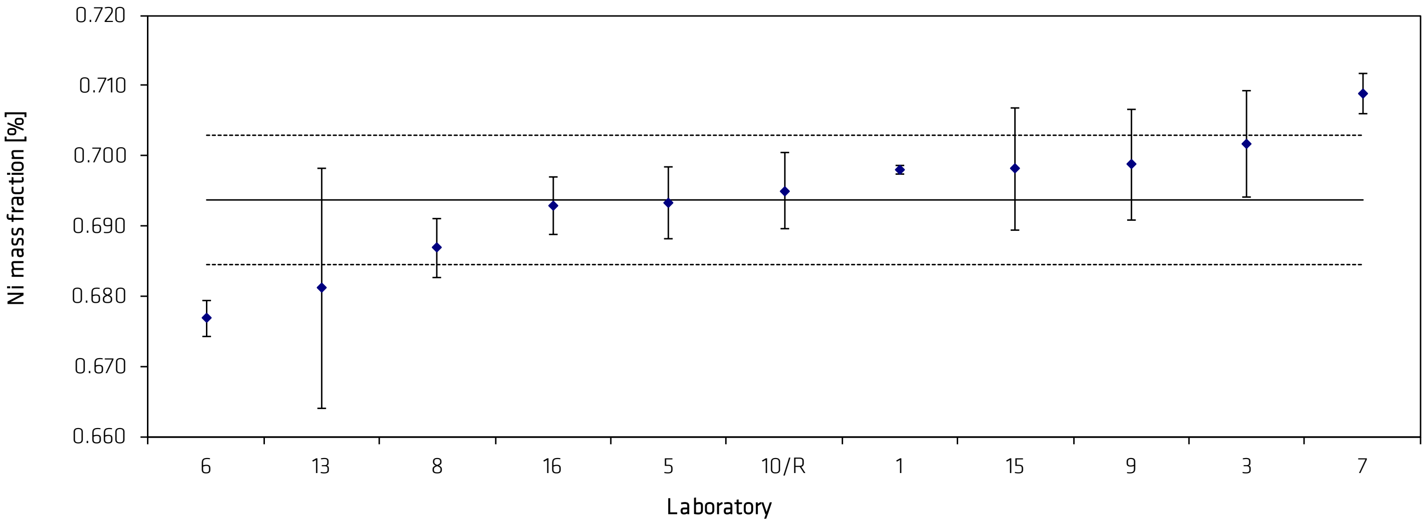


Table 17: Results for Ag

Lab.	13	9	16	4(R)	8	5	1	11	7	10	3	6	15		
M_i [%]	0.061	0.0629	0.062	0.062	0.0623	0.0629	0.0639	0.062	0.0629	0.0644	0.0640	0.0654	0.066		*n*
	0.062	0.0635	0.062	0.062	0.0617	0.0639	0.0627		0.0633	0.0635	0.0640	0.0648	0.066		13
	0.061	0.0615	0.062	0.062	0.0621	0.0624	0.0639		0.0646	0.0633	0.0640	0.0647	0.066		
	0.061	0.0613	0.063	0.063	0.0628	0.0635	0.0634	0.065	0.0632	0.0643	0.0650	0.0649	0.067		
	0.061	0.0602	0.060	0.062	0.0624	0.0641	0.0634		0.0644	0.0634	0.0640	0.0649	0.066		
	0.062	0.0608	0.062	0.062	0.0625	0.0637	0.0635		0.0643	0.0651	0.0640	0.0649	0.066		
M(B1)	*0.0614*	*0.0626*	*0.0620*	*0.0621*	*0.0620*	*0.0631*	*0.0635*	*0.0623*	*0.0636*	*0.0637*	*0.0640*	*0.0650*	*0.0660*		
M(B2)	*0.0616*	*0.0608*	*0.0617*	*0.0624*	*0.0626*	*0.0638*	*0.0634*	*0.0652*	*0.0640*	*0.0643*	*0.0643*	*0.0649*	*0.0663*		
M [%]	**0.0615**	**0.0617**	**0.0618**	**0.0623**	**0.0623**	**0.0634**	**0.0635**	**0.0637**	**0.0638**	**0.0640**	**0.0642**	**0.0649**	**0.0662**		**0.0633**
s [%]	0.0003	0.0013	0.0010	0.0002	0.0004	0.0006	0.0004	0.0020	0.0007	0.0007	0.0004	0.0002	0.0004	s_M [%]	0.0014
														$\bar{s}_i$ [%]	0.0008
s_{rel}	0.0055	0.0204	0.0159	0.0038	0.0060	0.0102	0.0070	0.0319	0.0115	0.0112	0.0064	0.0037	0.0062		0.0217

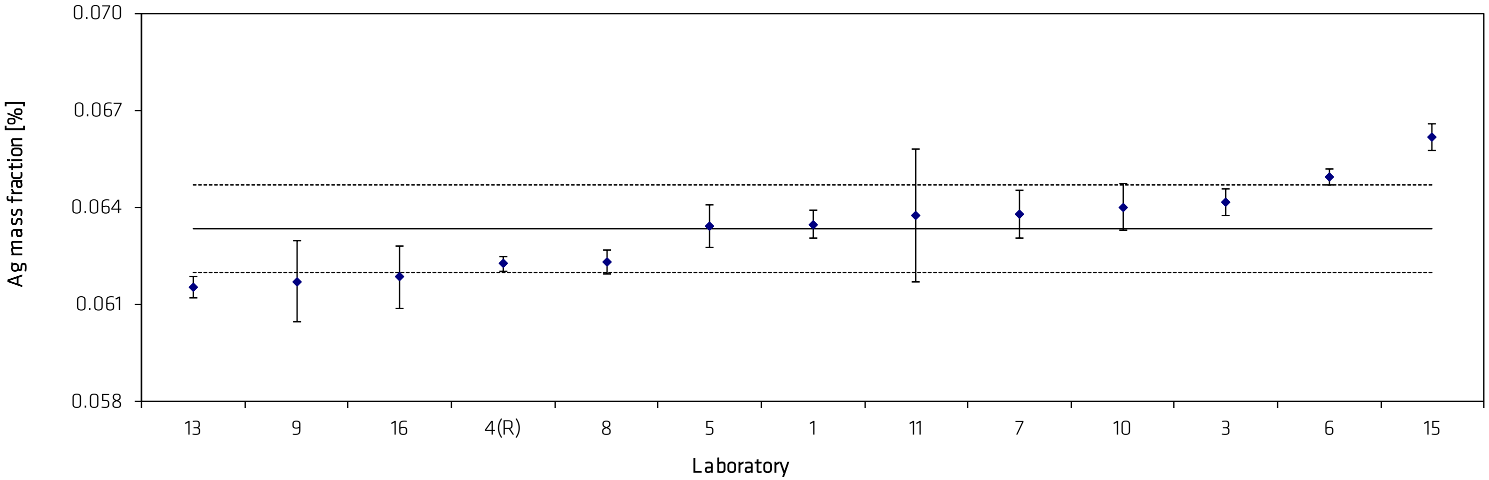


Table 18: Results for Pb

Lab.	5	10	7	15	13	8	9	16	1	6	3		
M_i [%]	1.05	1.05	1.024	1.102	1.137	1.143	1.140	1.172	1.194	1.204	1.216		*n*
	1.02	1.03	1.047	1.096	1.155	1.141	1.140	1.178	1.178	1.192	1.223		11
	1.03	1.04	1.035	1.079	1.141	1.142		1.181		1.191	1.231		
	1.01	1.05	1.055	1.109	1.136	1.140	1.147	1.170	1.197	1.189	1.241		
	1.02	1.03	1.054	1.117	1.148	1.145	1.154	1.170	1.150	1.204	1.233		
	1.01	1.04	1.051	1.112	1.120	1.143		1.175		1.193	1.239		
M(B1)	*1.033*	*1.040*	*1.035*	*1.092*	*1.144*	*1.142*	*1.140*	*1.177*	*1.186*	*1.196*	*1.223*		
M(B2)	*1.013*	*1.040*	*1.053*	*1.113*	*1.135*	*1.143*	*1.151*	*1.172*	*1.173*	*1.195*	*1.238*		
M [%]	**1.023**	**1.040**	**1.044**	**1.103**	**1.140**	**1.142**	**1.145**	**1.174**	**1.180**	**1.196**	**1.231**		**1.129**
s [%]	0.015	0.009	0.012	0.014	0.012	0.002	0.007	0.005	0.022	0.007	0.009	s_M [%]	0.068
												$\bar{s}_i$ [%]	0.012
s_{rel}	0.0147	0.0086	0.0118	0.0124	0.0103	0.0015	0.0059	0.0038	0.0185	0.0056	0.0077		0.061

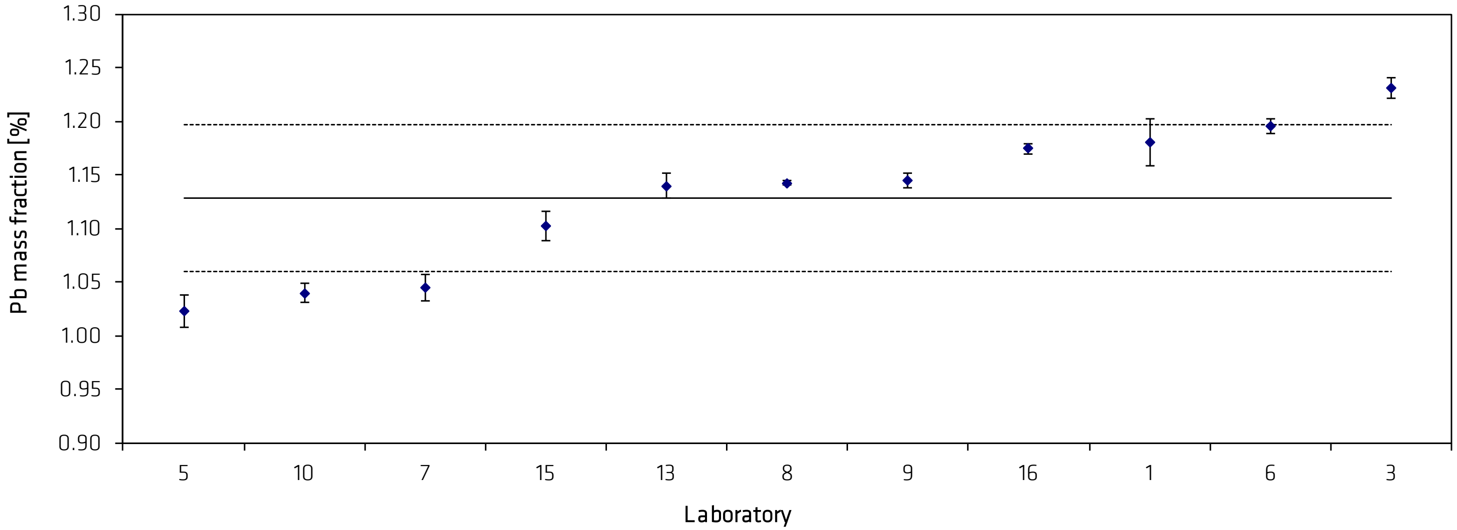


Table 19: Results for Cr

Lab.	10	8	13	3	5	9	15	7	1		
M_i [%]	0.95	0.959	0.951	0.995	0.97	0.979	1.001	0.996	1.063		*n*
	0.95	0.962	0.968	0.979	0.99	0.972	0.984	1.001			9
	0.95	0.939	0.978	0.967	0.98		0.986	0.995			
	0.95	0.959	0.966	0.972	0.98	0.976	0.991	0.995	1.021		
	0.95	0.941	0.972	0.971	0.97	0.995	1.001	1.003	1.011		
	0.94	0.947	0.965	0.965	0.98		0.983	1.000			
M(B1)	*0.950*	*0.954*	*0.965*	*0.980*	*0.980*	*0.975*	*0.990*	*0.997*	*1.063*		
M(B2)	*0.947*	*0.949*	*0.967*	*0.969*	*0.977*	*0.985*	*0.992*	*0.999*	*1.016*		
M [%]	**0.948**	**0.951**	**0.966**	**0.975**	**0.978**	**0.980**	**0.991**	**0.998**	**1.032**		**0.980**
s [%]	0.0041	0.0101	0.0090	0.0112	0.0075	0.0100	0.0082	0.0034	0.0277	s_M [%]	0.0254
										$\bar{s}_i$ [%]	0.0122
s_{rel}	0.0043	0.0107	0.0094	0.0115	0.0077	0.0102	0.0083	0.0035	0.0268		0.0259

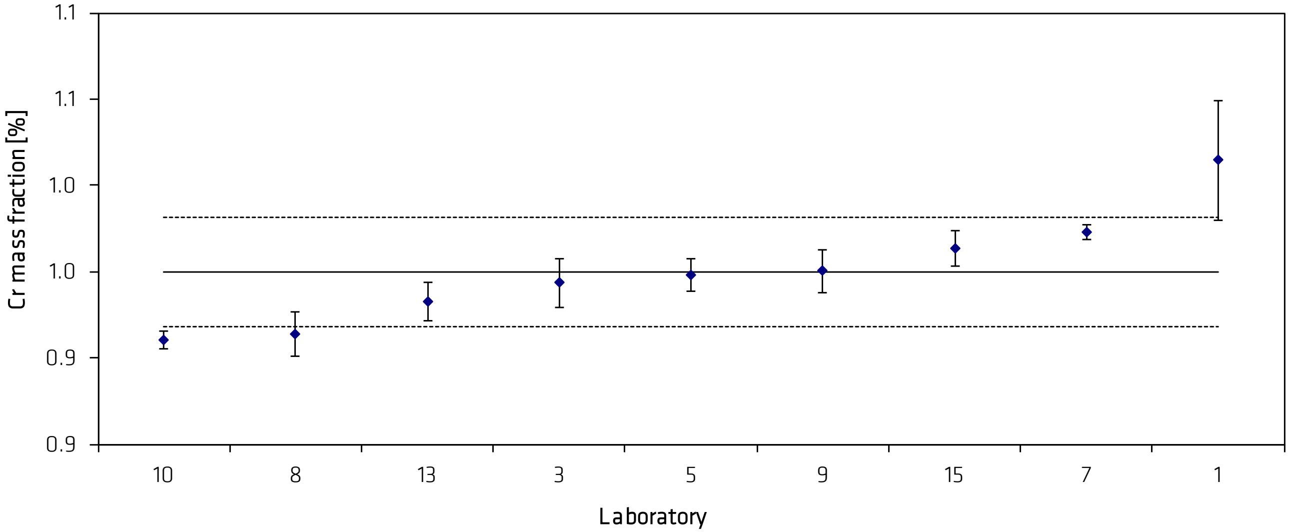


Table 20: Results for Sn

Lab.	5	10	7	13	9/R	8	15	16	3	1	6		
M_i [%]	0.44	0.43	0.456	0.451	0.470	0.470	0.476	0.477	0.480	0.501	0.505		*n*
	0.44	0.43	0.452	0.463	0.450	0.474	0.481	0.482	0.478	0.501	0.501		11
	0.44	0.43	0.455	0.461	0.460	0.466	0.474	0.481	0.480		0.501		
	0.38	0.43	0.456	0.463		0.469	0.475	0.481	0.480	0.496	0.503		
	0.44	0.43	0.454	0.461		0.471	0.477	0.478	0.485	0.503	0.499		
	0.44	0.44	0.455	0.457		0.470	0.475	0.481	0.484		0.503		
M(B1)	*0.440*	*0.430*	*0.454*	*0.458*	*0.460*	*0.470*	*0.477*	*0.480*	*0.479*	*0.501*	*0.502*		
M(B2)	*0.420*	*0.433*	*0.455*	*0.461*		*0.470*	*0.476*	*0.480*	*0.483*	*0.500*	*0.502*		
***M* [%]**	**0.430**	**0.432**	**0.455**	**0.459**	**0.460**	**0.470**	**0.476**	**0.480**	**0.481**	**0.500**	**0.502**		**0.468**
s [%]	0.0245	0.0041	0.0015	0.0045	0.0100	0.0025	0.0025	0.0020	0.0027	0.0027	0.0022	s_M [%]	0.0238
												$\bar{s}_i$ [%]	0.0084
s_{rel}	0.0570	0.0095	0.0033	0.0098	0.0217	0.0053	0.0053	0.0042	0.0057	0.0055	0.0043		0.0509

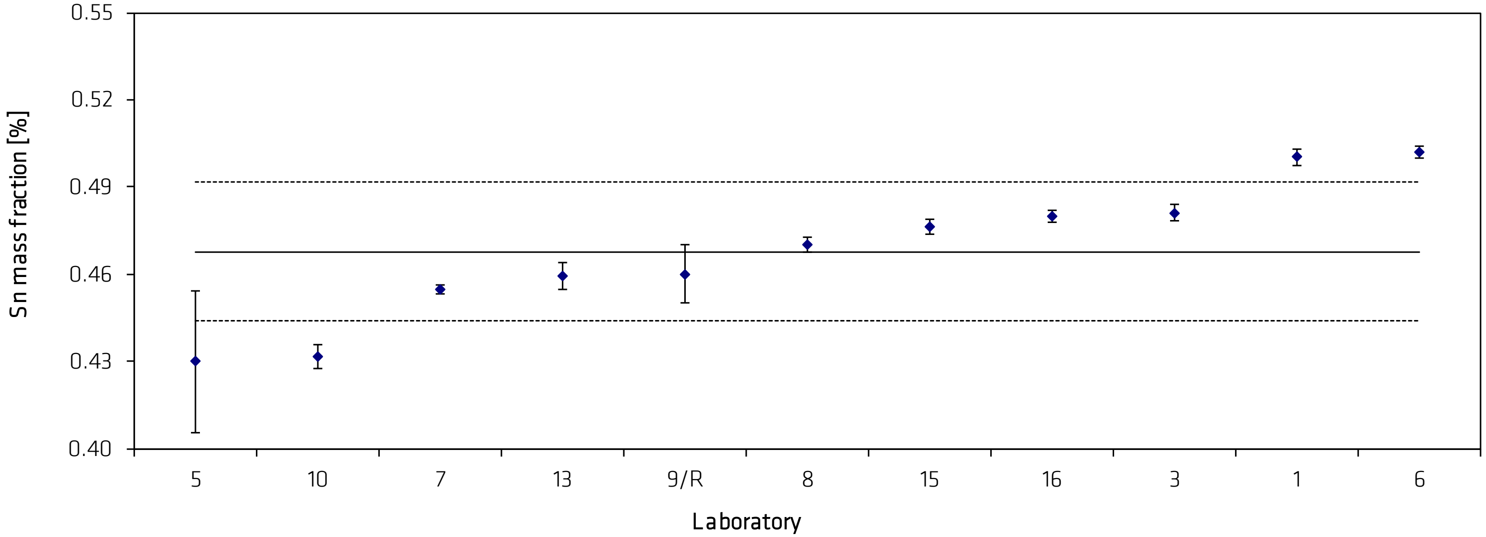


Table 21: Results for Au

Lab.	8	5	1	16	10	15	9	13	11	4	6	3		
M_i [mg/kg]	49.9	51.3	50.6	61.7	49.5	50.8	53.8	52.2	51.9	53.5	53.3	56.9		*n*
	49.4	51.5	53.4	42.3	52.5	52.3	52.8	53.0	52.0	53.7	55.1	54.7		12
	50.5	51.0	49.7	51.3	50.2	53.3	50.1	52.9	53.6	51.7	55.6	56.6		
	51.3	49.7	49.1	50.1	52.6	50.6	50.6	52.3	52.9	54.9	53.8	56.8		
	50.7	49.8	51.6	50.4	53.1	51.1	53.0	52.7	54.2	53.4	55.8	53.4		
	51.6	50.3	52.7	53.5	53.3	53.5	53.0	53.1		53.7	55.1	53.4		
M(B1)	*49.9*	*51.3*	*51.2*	*51.8*	*50.7*	*52.1*	*52.2*	*52.7*	*52.5*	*53.0*	*54.6*	*56.0*		
M(B2)	*51.2*	*49.9*	*51.1*	*51.3*	*53.0*	*51.7*	*52.2*	*52.7*	*53.6*	*54.0*	*54.9*	*54.5*		
M [mg/kg]	**50.6**	**50.6**	**51.2**	**51.6**	**51.9**	**51.9**	**52.2**	**52.7**	**52.9**	**53.5**	**54.8**	**55.3**		**52.4**
s [mg/kg]	0.83	0.77	1.66	6.26	1.61	1.28	1.49	0.39	1.00	1.03	1.02	1.69	s_M [mg/kg]	1.50
													$\bar{s}_i$ [mg/kg]	2.16
s_{rel}	0.016	0.015	0.033	0.121	0.031	0.025	0.029	0.007	0.019	0.019	0.019	0.031		0.03

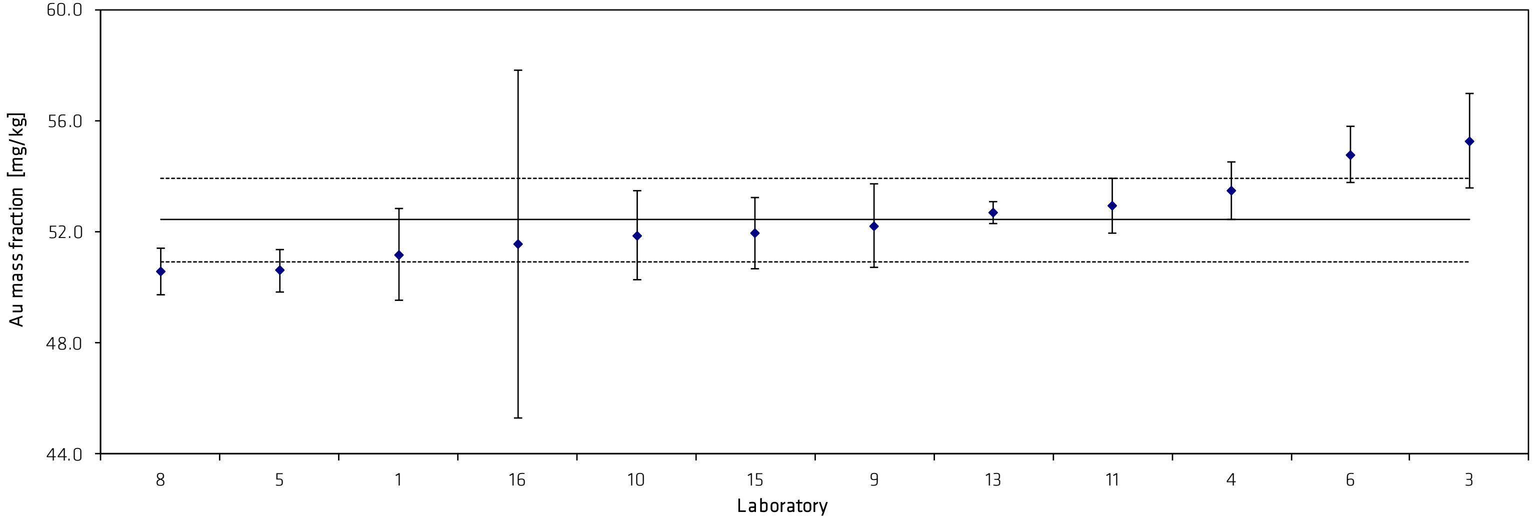


Table 22: Results for Pd

Lab.	15	4	11	9	8	13	10	1	5	16	3	6		
M_i [mg/kg]	46.7	46.5	47.0	48	47.0	47.1	47	48.0	48.7	49.7	49.0	50.4		*n*
	46.4	46.1	46.9	46	47.0	47.7	47	48.4	48.4	45.5	49.7	50.2		12
	46.7	45.2	46.3	48	47.4	48.1	48	48.1	49.5	50.8	50.0	50.5		
	46.4	47.7	46.7	47	47.7	47.3	49	48.1	48.4	49.9	50.7	50.3		
	46.6	46.6	46.9	47	47.6	47.8	48	48.2	48.3	48.4	50.0	50.6		
	46.3	47.8		47	47.7	47.5	48	47.9	48.4	48.6	50.1	50.5		
M(B1)	*46.6*	*45.9*	*46.7*	*47.3*	*47.1*	*47.6*	*47.3*	*48.2*	*48.9*	*48.7*	*49.6*	*50.4*		
M(B2)	*46.4*	*47.4*	*46.8*	*47.0*	*47.7*	*47.5*	*48.3*	*48.0*	*48.4*	*49.0*	*50.2*	*50.5*		
M [mg/kg]	**46.5**	**46.7**	**46.7**	**47.2**	**47.4**	**47.6**	**47.8**	**48.1**	**48.6**	**48.8**	**49.9**	**50.4**		**48.0**
s [mg/kg]	0.17	0.99	0.30	0.75	0.33	0.36	0.75	0.18	0.45	1.85	0.55	0.14	s_M [mg/kg]	1.25
													$\bar{s}_i$ [mg/kg]	0.73
s_{rel}	0.004	0.021	0.006	0.016	0.007	0.008	0.016	0.004	0.009	0.038	0.011	0.003		0.026

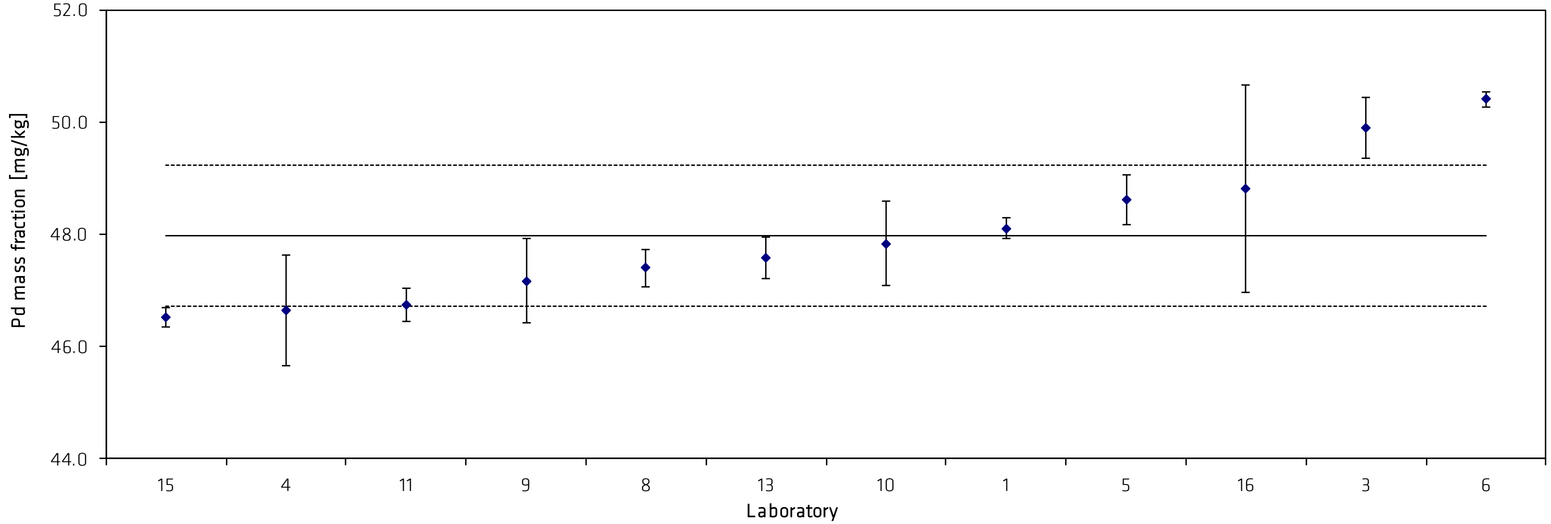


Table 23: Results for Pt

Lab.	1	16	10	8	9	13	5	11	6	3	15	4		
M_i [mg/kg]	4.9	6.0	4	5.4	6	6.5	5.6	6.1	6.1	6.3	6.5	6.5		*n*
	5.5	4.6	5	5.5	5	6.6	6.1	6.0	6.1	6.2	5.8	6.1		12
	5.2	5.4	5	5.7	5	5.9	6.6	5.4	6.1	6.3	5.8	7.1		
	4.4	5.4	5	5.4	6	5.5	5.7	5.9	6.1	6.6	6.2	6.4		
	5.2	3.5	5	5.4	6	5.7	5.6	6.3	6.2	6.4	6.1	5.8		
	4.1	4.7	6	5.6	6	5.1	5.8		6.1	5.8	7.2	6.8		
M(B1)	*5.2*	*5.3*	*4.7*	*5.5*	*5.3*	*6.3*	*6.1*	*5.8*	*6.1*	*6.2*	*6.0*	*6.6*		
M(B2)	*4.6*	*4.5*	*5.3*	*5.5*	*6.0*	*5.4*	*5.7*	*6.1*	*6.1*	*6.3*	*6.5*	*6.3*		
M [mg/kg]	**4.9**	**4.9**	**5.0**	**5.5**	**5.7**	**5.9**	**5.9**	**5.9**	**6.1**	**6.3**	**6.3**	**6.5**		**5.7**
s [mg/kg]	0.54	0.87	0.63	0.13	0.52	0.59	0.39	0.34	0.03	0.27	0.53	0.47	s_M [mg/kg]	0.55
													$\bar{s}_i$ [mg/kg]	0.49
s_{rel}	0.110	0.177	0.126	0.023	0.091	0.101	0.066	0.057	0.004	0.043	0.084	0.073		0.095

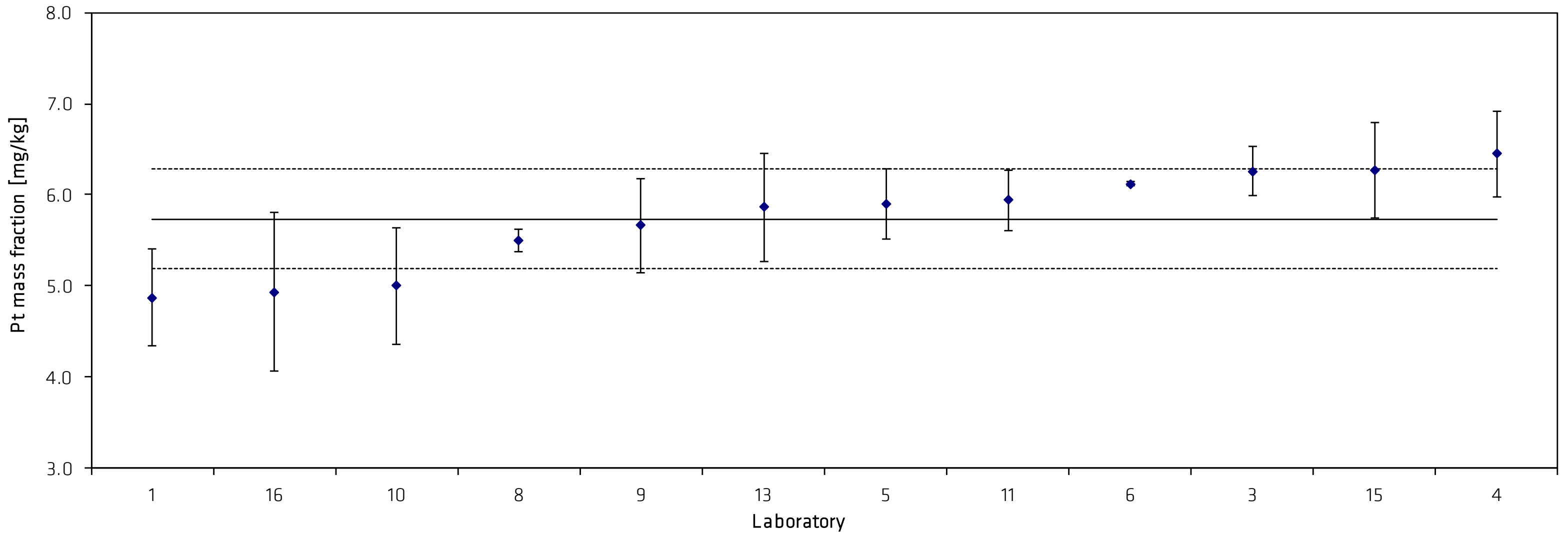


Table 24: Results for As

Lab.	3	1	15	20	16	5/2	5/1	13	10	9	7	6		
M_i [mg/kg]	321.2	326.2	343.4	344.3	376	387	380	384	383	402	400.2	415.7		*n*
	318.9	326.5	339.4	349.9	373	376	382	393	388	401	401.2	428.2		12
	320.5		335.9	351.5	373		384	381	384	402	405.2	417.2		
								384						
	318.8	320.9	334.2	342.5	375	379	382	392	383	398	399.0	437.7		
	318.6		358.0	339.5	374	360	382	380	383	400	409.6	439.8		
	322.0		333.6	340.7	376		382	365	386	397	406.8	437.4		
								384						
M(B1)	*320.2*	*326.4*	*339.6*	*348.6*	*374.0*	*381.5*	*382.0*	*386.0*	*385.0*	*401.7*	*402.2*	*420.4*		
M(B2)	*319.8*	*320.9*	*342.0*	*340.9*	*375.0*	*369.5*	*382.0*	*379.0*	*384.0*	*398.3*	*405.1*	*438.3*		
M [mg/kg]	320.0	324.5	340.8	344.7	374.5	375.5	382.0	382.7	384.5	400.0	403.7	429.4		371.9
s [mg/kg]	1.4	3.2	9.2	4.9	1.4	11.3	1.3	9.3	2.1	2.1	4.2	10.7	s_M [mg/kg]	33.2
													$\bar{s}_i$ [mg/kg]	6.3
s_{rel}	0.004	0.010	0.027	0.014	0.004	0.030	0.003	0.024	0.005	0.005	0.010	0.025		0.09

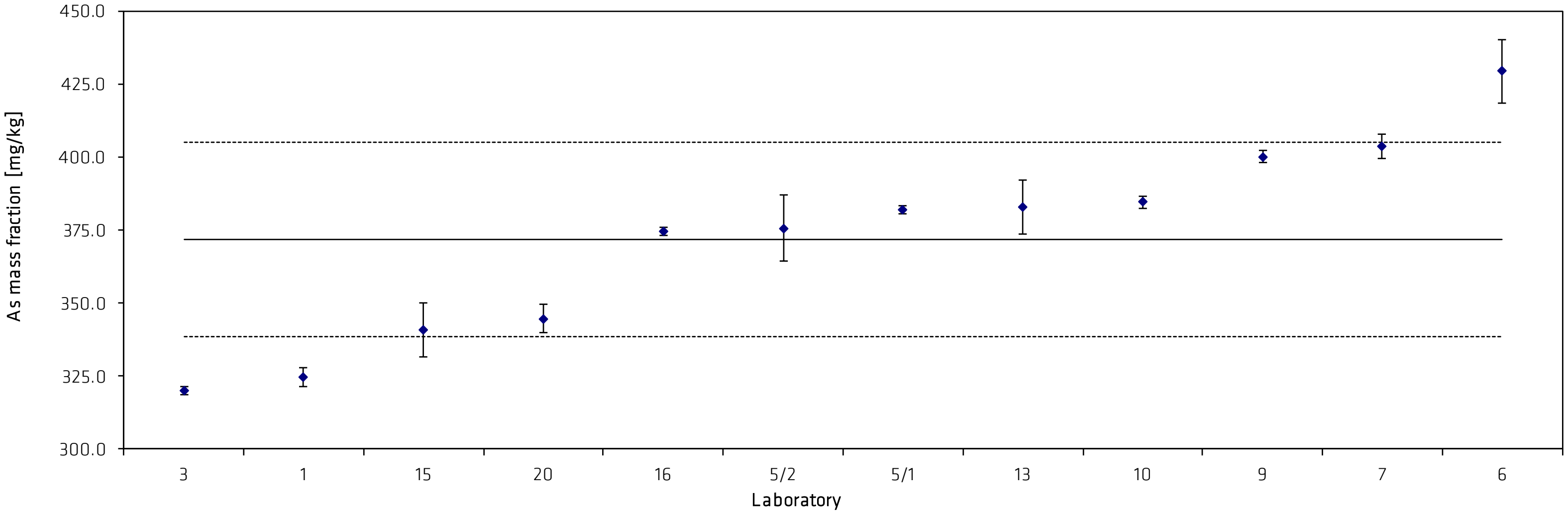


Table 25: Results for Be

Lab.	5/2	20	16	3	15	6	9/R	7	5/1	1/R	10		
M_i [mg/kg]	4.9	5.3	6.0	6.1	6.43	6.7	6.9	7.2	9.9	9.3	< 20		*n*
	5.1	5.3	5.9	6.5	6.57	6.6	7.0	7.0	8.5	9.4	< 20		10
		5.3	6.3	6.5	6.70	6.6	6.9	6.8	8.5	9.7	< 20		
	5.0	5.6	6.0	6.4	6.35	6.6		7.4	8.6	9.5	< 20		
	5.4	5.4	6.0	6.7	6.60	6.7		7.4	8.7	9.2	< 20		
		5.5	6.1	6.5	6.26	6.7		7.3	8.7		< 20		
M(B1)	*5.0*	*5.3*	*6.1*	*6.4*	*6.6*	*6.6*	*6.9*	*7.0*	*9.0*	*9.5*	*< 20*		
M(B2)	*5.2*	*5.5*	*6.0*	*6.5*	*6.4*	*6.7*		*7.4*	*8.7*	*9.4*	*< 20*		
M [mg/kg]	**5.1**	**5.4**	**6.1**	**6.4**	**6.5**	**6.7**	**6.9**	**7.2**	**8.8**	**9.4**	**< 20**		**6.8**
s [mg/kg]	0.22	0.13	0.14	0.20	0.17	0.04	0.06	0.24	0.54	0.18		s_M [mg/kg]	1.37
												$\bar{s}_i$ [mg/kg]	0.22
s_{rel}	0.042	0.024	0.023	0.032	0.026	0.007	0.008	0.033	0.061	0.019			0.20

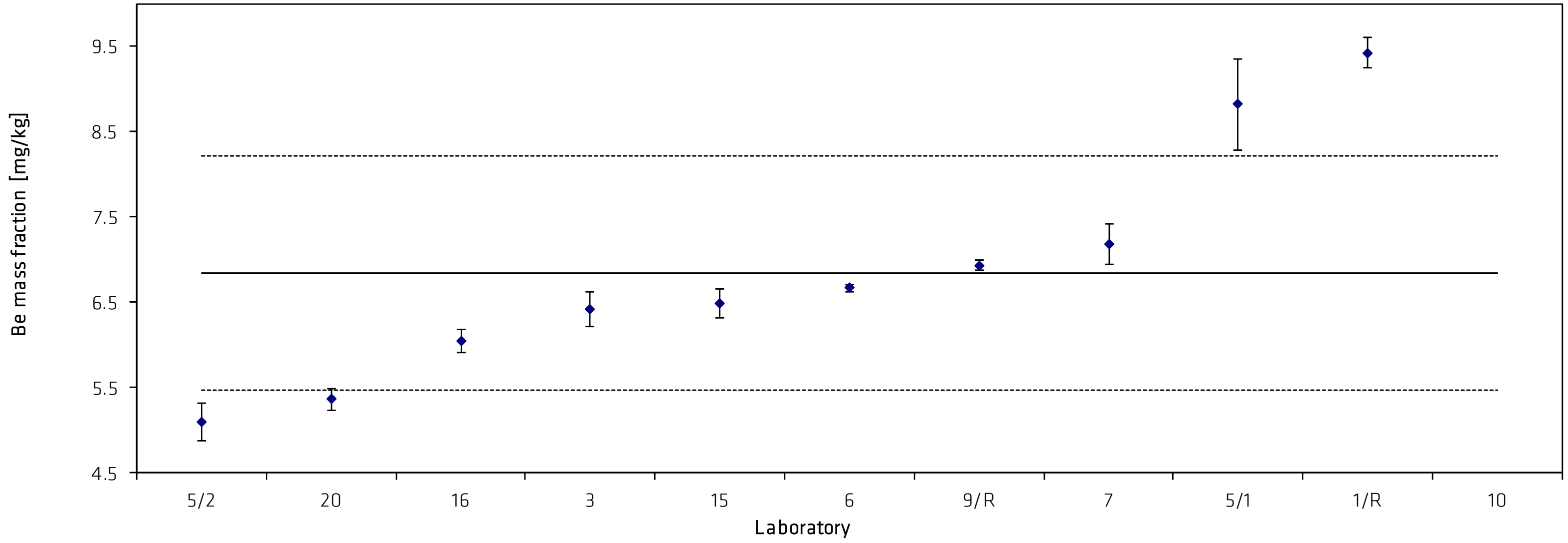


Table 26: Results for Cd

Lab.	20	15	3	13	5/1	16	6	9/R	5/2	7	1	10		
M_i [mg/kg]	14.2	14.6	16.0	16.4	16.4	16.8	15.7	16.8	17.4	17.6	17.0	< 20		*n*
	14.2	14.7	16.2	16.8	16.5	17.1	16.6	17.0	17.4	17.2	18.7	< 20		11
	14.3	14.5	16.3	16.6	16.5	17.0	16.0	16.9		17.8	18.6	< 20		
				16.3										
	14.4	14.3	16.2	16.3	16.4	16.7	17.0		17.0	17.9	18.5	< 20		
	14.4	14.4	16.2	16.3	16.3	15.6	17.6		16.2	18.5	17.5	< 20		
	14.4	14.4	16.1	15.7	16.4	17.7	18.3			17.2	17.4	< 20		
				16.1										
M(B1)	*14.2*	*14.6*	*16.2*	*16.6*	*16.5*	*17.0*	*16.1*	*16.9*	*17.4*	*17.5*	*18.1*	*< 20*		
M(B2)	*14.4*	*14.4*	*16.1*	*16.1*	*16.4*	*16.7*	*17.6*		*16.6*	*17.9*	*17.8*	*< 20*		
M [mg/kg]	**14.3**	**14.5**	**16.2**	**16.3**	**16.4**	**16.8**	**16.9**	**16.9**	**17.0**	**17.7**	**18.0**	**< 20**		**16.4**
s [mg/kg]	0.10	0.16	0.09	0.33	0.08	0.69	0.97	0.10	0.57	0.49	0.73		s_M [mg/kg]	1.15
													$\bar{s}_i$ [mg/kg]	0.49
s_{rel}	0.007	0.011	0.006	0.020	0.005	0.041	0.058	0.006	0.033	0.028	0.041			0.070

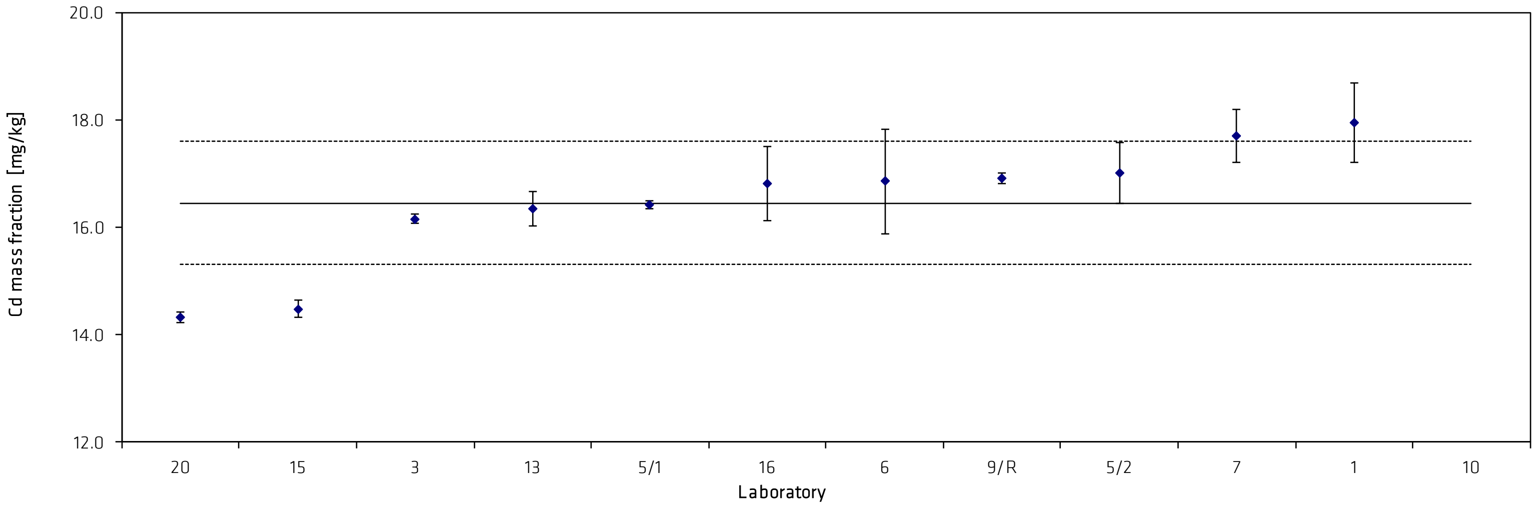


Table 27: Results for In

Lab.	6	3	15	20	5/2	1	16	5/1	13	7	9	22		
M_i [mg/kg]	32.6	37.5	39.1	39.2	42.2	39.4	41.0	44.3	46.1	51.5	56.0	60.2		*n*
	35.3	37.7	39.1	38.8	42.1	39.6	42.7	40.8	44.0	52.1	58.0	56.0		12
	31.8	38.1	38.3	39.3			42.7	48.4	45.3	50.1	56.0	59.0		
									44.9					
	35.4	37.9	38.3	38.9	36.7	40.0	38.7	38.2	40.2	50.4	54.0	61.3		
	33.5	38.3	38.1	38.9	36.4		38.5	38.2	40.2	50.7	57.0	61.0		
	34.4	37.9	38.5	39.3			38.6	38.4	41.4	50.3	54.0	58.4		
									40.5					
M(B1)	*33.2*	*37.8*	*38.8*	*39.1*	*42.2*	*39.5*	*42.1*	*44.5*	*45.1*	*51.2*	*56.7*	*58.4*		
M(B2)	*34.5*	*38.0*	*38.3*	*39.0*	*36.6*	*40.0*	*38.6*	*38.3*	*40.6*	*50.4*	*55.0*	*60.2*		
M [mg/kg]	**33.8**	**37.9**	**38.6**	**39.1**	**39.4**	**39.7**	**40.4**	**41.4**	**42.8**	**50.8**	**55.8**	**59.3**		**43.2**
s [mg/kg]	1.46	0.27	0.44	0.23	3.24	0.31	2.03	4.18	2.50	0.79	1.60	1.95	s_M [mg/kg]	7.81
													$\bar{s}_i$ [mg/kg]	2.00
s_{rel}	0.043	0.007	0.012	0.006	0.082	0.008	0.050	0.101	0.058	0.016	0.029	0.033		0.18

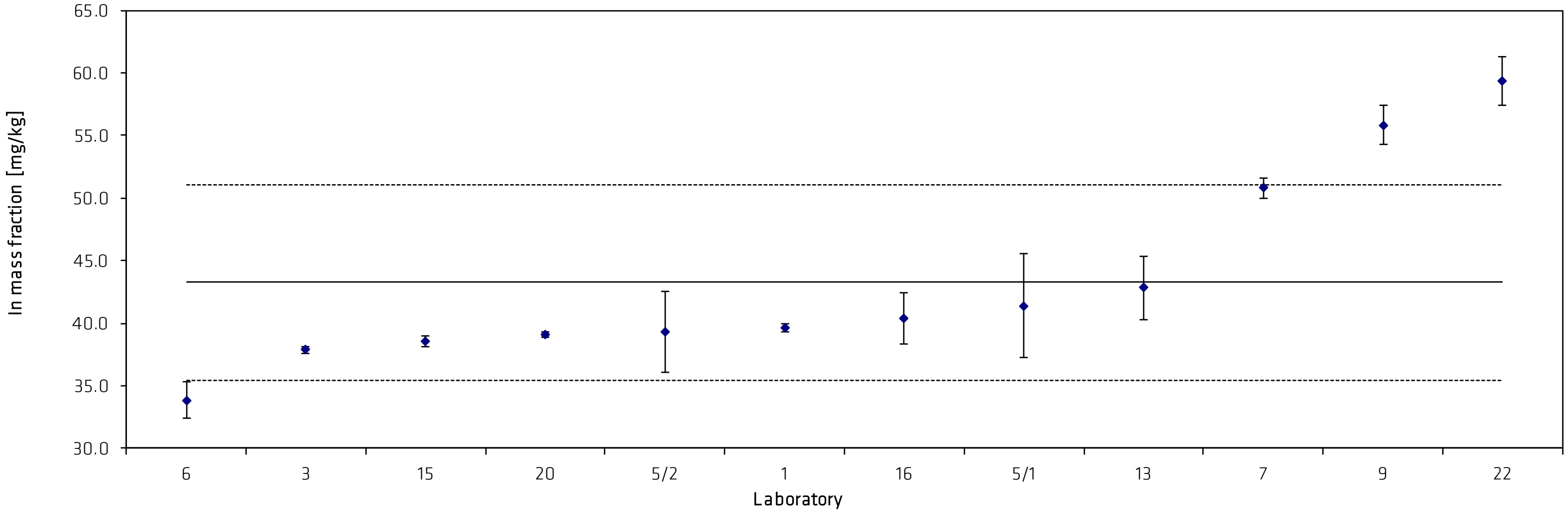
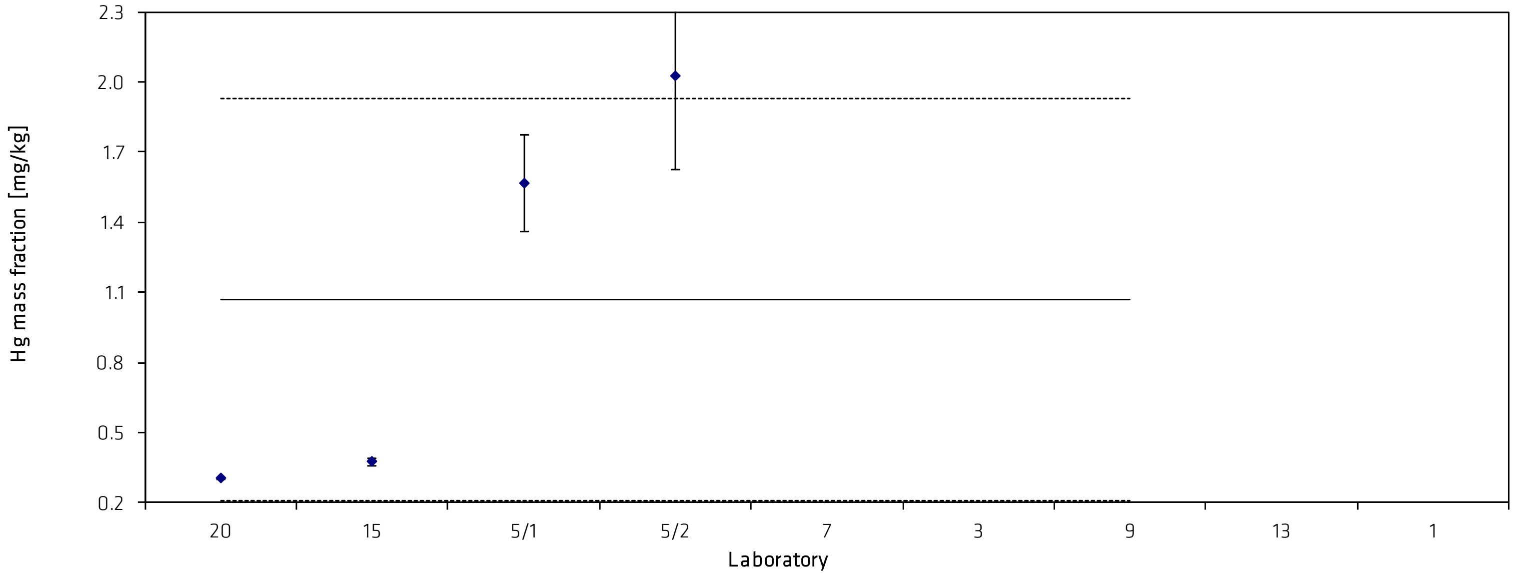


Table 28: Results for Hg

Lab.	20	15	5/1	5/2	7	3	9	13	1	10		
M_i [mg/kg]	0.30	0.40	1.60	2.00	< 1	< 5	< 5	< 5	< 5.02	< 20		*n*
	0.31	0.36	1.60	2.60	< 1	< 5	< 5	< 5	< 5.02	< 20		4
	0.31	0.38	1.40		< 1	< 5	< 5			< 20		
	0.30	0.38	1.90	1.80	< 1	< 5	< 5	< 5	< 5.02	< 20		
	0.31	0.37	1.30	1.70	< 1	< 5	< 5	< 5		< 20		
	0.30	0.36	1.60		< 1	< 5	< 5			< 20		
M(B1)	*0.30*	*0.38*	*1.53*	*2.30*	*< 1*	*< 5*	*< 5*		*< 5.02*	*< 20*		
M(B2)	*0.31*	*0.37*	*1.60*	*1.75*	*< 1*	*< 5*	*< 5*		*< 5.02*	*< 20*		
M [mg/kg]	**0.30**	**0.37**	**1.57**	**2.03**	**< 1**	**< 5**	**< 5**	**< 5**	**< 5.02**	**< 20**		**1.07**
s [mg/kg]	0.004	0.016	0.207	0.403							s_M [mg/kg]	0.862
											$\bar{s}_i$ [mg/kg]	0.151
s_{rel}	0.014	0.044	0.132	0.199								0.807



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

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

	Cu	Ni
Number of data sets	13	11
Scheffe's test (data compatible?)	yes	yes
Snedecor-F-Test and Bartlett-Test	Pooling not allowed	Pooling not allowed
Dixon (α = 0.05)	---	---
Dixon (α = 0.01)	---	---
Nalimov (α = 0.05)	Lab. 16	Lab. 6
Nalimov (α = 0.01)	---	---
Grubbs (α = 0.05)	---	---
Grubbs (α = 0.01)	---	---
Grubbs Pair (α = 0.05)	---	---
Grubbs Pair (α = 0.01)	---	---
Cochran	---	---
Kolmogorov-Smirnov-Lilliefors Test (α = 0.05)	Distribution: normal	Distribution: normal
Kolmogorov-Smirnov-Lilliefors Test (α = 0.01)	Distribution: normal	Distribution: normal
Skewness & Kurtosis Test (α = 0.05)	Distribution: normal	Distribution: normal
Skewness & Kurtosis Test (α = 0.01)	Distribution: normal	Distribution: normal

The stragglers (Lab. 16; Cu; Lab. 6, Ni) were not removed.

Tab. 30: Outcome of statistical tests on the results obtained for Ag and Pb

	Ag	Pb
Number of data sets	13	11
Scheffe's test (data compatible?)	yes	yes
Snedecor-F-Test and Bartlett-Test	Pooling not allowed	Pooling not allowed
Dixon (α = 0.05)	---	---
Dixon (α = 0.01)	---	---
Nalimov (α = 0.05)	Lab. 15	---
Nalimov (α = 0.01)	---	---
Grubbs (α = 0.05)	---	---
Grubbs (α = 0.01)	---	---
Grubbs Pair (α = 0.05)	---	---
Grubbs Pair (α = 0.01)	---	---
Cochran	---	---
Kolmogorov-Smirnov-Lilliefors Test (α = 0.05)	Distribution: normal	Distribution: normal
Kolmogorov-Smirnov-Lilliefors Test (α = 0.01)	Distribution: normal	Distribution: normal
Skewness & Kurtosis Test (α = 0.05)	Distribution: normal	Distribution: normal
Skewness & Kurtosis Test (α = 0.01)	Distribution: normal	Distribution: normal

The straggler (Lab. 15; Ag) was not removed.

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

	Cr	Sn
Number of data sets	9	11
Scheffe's test (data compatible?)	yes	yes
Snedecor-F-Test and Bartlett-Test	Pooling not allowed	Pooling not allowed
Dixon (α = 0.05)	---	---
Dixon (α = 0.01)	---	---
Nalimov (α = 0.05)	Lab. 1	---
Nalimov (α = 0.01)	---	---
Grubbs (α = 0.05)	---	---
Grubbs (α = 0.01)	---	---
Grubbs Pair (α = 0.05)	---	---
Grubbs Pair (α = 0.01)	---	---
Cochran	---	---
Kolmogorov-Smirnov-Lilliefors Test (α = 0.05)	Distribution: normal	Distribution: normal
Kolmogorov-Smirnov-Lilliefors Test (α = 0.01)	Distribution: normal	Distribution: normal
Skewness & Kurtosis Test (α = 0.05)	Distribution: normal	Distribution: normal
Skewness & Kurtosis Test (α = 0.01)	Distribution: normal	Distribution: normal

The straggler (Lab. 1; Cr) was not removed.

Tab. 32: Outcome of statistical tests on the results obtained for Au and Pd

	Au	Pd
Number of data sets	12	12
Scheffe's test (data compatible?)	yes	yes
Snedecor-F-Test and Bartlett-Test	Pooling not allowed	Pooling not allowed
Dixon (α = 0.05)	---	---
Dixon (α = 0.01)	---	---
Nalimov (α = 0.05)	Lab. 3	Lab. 6
Nalimov (α = 0.01)	---	---
Grubbs (α = 0.05)	---	---
Grubbs (α = 0.01)	---	---
Grubbs Pair (α = 0.05)	---	---
Grubbs Pair (α = 0.01)	---	---
Cochran	---	---
Kolmogorov-Smirnov-Lilliefors Test (α = 0.05)	Distribution: normal	Distribution: normal
Kolmogorov-Smirnov-Lilliefors Test (α = 0.01)	Distribution: normal	Distribution: normal
Skewness & Kurtosis Test (α = 0.05)	Distribution: normal	Distribution: normal
Skewness & Kurtosis Test (α = 0.01)	Distribution: normal	Distribution: normal

The stragglers (Lab. 3; Au; Lab. 6, Pd) were not removed.

Tab. 33: Outcome of statistical tests on the results obtained for Pt and As

	Pt	As
Number of data sets	12	12
Scheffe's test (data compatible?)	yes	yes
Snedecor-F-Test and Bartlett-Test	Pooling not allowed	Pooling not allowed
Dixon (α = 0.05)	---	---
Dixon (α = 0.01)	---	---
Nalimov (α = 0.05)	---	---
Nalimov (α = 0.01)	---	---
Grubbs (α = 0.05)	---	---
Grubbs (α = 0.01)	---	---
Grubbs Pair (α = 0.05)	---	---
Grubbs Pair (α = 0.01)	---	---
Cochran	---	---
Kolmogorov-Smirnov-Lilliefors Test (α = 0.05)	Distribution: normal	Distribution: normal
Kolmogorov-Smirnov-Lilliefors Test (α = 0.01)	Distribution: normal	Distribution: normal
Skewness & Kurtosis Test (α = 0.05)	Distribution: normal	Distribution: normal
Skewness & Kurtosis Test (α = 0.01)	Distribution: normal	Distribution: normal

Tab. 34: Outcome of statistical tests on the results obtained for Be and In (the "<" value for Be was not included into the calculations)

	Be	In
Number of data sets	10	12
Scheffe's test (data compatible?)	yes	Pooling not allowed
Snedecor-F-Test and Bartlett-Test	Pooling not allowed	---
Dixon (α = 0.05)	---	---
Dixon (α = 0.01)	Lab. 1	Lab. 22
Nalimov (α = 0.05)	---	---
Nalimov (α = 0.01)	---	---
Grubbs (α = 0.05)	---	---
Grubbs (α = 0.01)	---	---
Grubbs Pair (α = 0.05)	---	---
Grubbs Pair (α = 0.01)	---	---
Cochran	---	---
Kolmogorov-Smirnov-Lilliefors Test (α = 0.05)	Distribution: normal	Distribution: normal
Kolmogorov-Smirnov-Lilliefors Test (α = 0.01)	Distribution: normal	Distribution: normal
Skewness & Kurtosis Test (α = 0.05)	Distribution: normal	Distribution: normal
Skewness & Kurtosis Test (α = 0.01)	Distribution: normal	Distribution: normal

The stragglers (Lab. 1, Be; Lab. 22, In) were not removed.

Tab. 35: Outcome of statistical tests on the results obtained for Cd (the “<” value was not included into the calculations)

	Cd
Number of data sets	11
Scheffe’s test (data compatible?)	yes
Snedecor-F-Test and Bartlett-Test	Pooling not allowed
Dixon (α = 0.05)	---
Dixon (α = 0.01)	---
Nalimov (α = 0.05)	Lab. 20
Nalimov (α = 0.01)	---
Grubbs (α = 0.05)	---
Grubbs (α = 0.01)	---
Grubbs Pair (α = 0.05)	---
Grubbs Pair (α = 0.01)	---
Cochran	---
Kolmogorov-Smirnov-Lilliefors Test (α = 0.05)	Distribution: normal
Kolmogorov-Smirnov-Lilliefors Test (α = 0.01)	Distribution: normal
Skewness & Kurtosis Test (α = 0.05)	Distribution: normal
Skewness & Kurtosis Test (α = 0.01)	Distribution: normal

The straggler (Lab. 20) was not removed.

The certified mass fractions of all elements were calculated as mean of the accepted data sets. These values are given in Table 36.

The resp. combined uncertainties were calculated from the spread resulting from the certification interlaboratory comparison and the uncertainty contribution from possible inhomogeneity of the material using Equation 7.

$$u_{combined} = \sqrt{\frac{s_M^2}{n} + u_{bb}^2} \quad (7)$$

with

$\frac{s_M^2}{n}$: uncertainty contribution resulting from interlaboratory comparison

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

Table 36: Uncertainty calculation

	uncertainty contribution from						uncertainty contribution from			
	M	*n*	s_M	u_{ilc}	u_{bb}	*u* (comb)	*U*	homogeneity test (rel. %)		
	%		%	%	%	%	%			
Cu	16.7800	12	0.0643	0.0186	0.00317	0.0188	0.0377		0.0002	
Ni	0.6940	11	0.0092	0.0028	0.00061	0.0028	0.0057		0.0009	
Ag	0.0633	13	0.0014	0.0004	0.00012	0.0004	0.00081		0.0019	
Pb	1.129	11	0.0680	0.0205	0.00057	0.0205	0.04102		0.0005	
Cr	0.980	9	0.0254	0.0085	0.00035	0.0085	0.0169		0.0004	
Sn	0.468	11	0.0238	0.0072	0.00019	0.0072	0.01436		0.0004	
	mg/kg		mg/kg	mg/kg	mg/kg	mg/kg	mg/kg			
Au	52.4	12	1.50	0.4324	0.00000	0.4324	0.8649		0.0000	
Pd	47.98	12	1.25	0.3617	0.00000	0.3617	0.7234		0.0000	
Pt	5.73	12	0.55	0.1576	0.00000	0.1576	0.3152		0.0000	
As	371.9	12	33.22	9.5898	0.00000	9.5898	19.1796		0.0000	
Be	6.8	10	1.37	1.5540	0.00000	0.4332	0.8665		0.0000	
Cd	16.4	11	1.15	0.3467	0.00000	0.3467	0.693		0.0000	
In	43.2	12	7.81	2.2546	1.70366	2.8259	5.6517		0.0394	

The expanded uncertainties *U* are calculated by multiplication of $u_{combined}$ with a coverage factor of $k = 2$:

$$U = 2 \cdot u_{\mathrm{combined}} = 2 \cdot \sqrt{\frac{s_{\mathrm{ilc}}^2}{n} + u_{\mathrm{bb}}^2} \qquad (8)$$

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

7. Instructions for users

The certified reference material BAM-M505a is intended for the development, validation and quality control of analytical methods and procedures for the determination of main and trace components in electronic waste.
The material is stable, it has to be stored in a dry and clean atmosphere.

8. Metrological traceability

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

9. Information on and purchase of the CRM

Certified reference material BAM-M505a is supplied by
Bundesanstalt für Materialforschung und -prüfung (BAM)
Division 1.6 „Inorganic Reference Materials"
Richard-Willstätter-Str. 11, D-12489 Berlin, Germany
Phone +49 (0)30 - 8104 2061
Fax: +49 (0)30 – 8104 72061
E-Mail: sales.crm@bam.de

Each bottle of BAM-M505a will be distributed together with a detailed certificate containing the certified values and their uncertainties, the mean values and standard deviations of all accepted data sets and information on the analytical methods used and the names of the participating laboratories.
Information on certified reference materials can be obtained from BAM:
https://www.bam.de.
Tel. +49 30 8104 1111.

10. References

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

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

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

[4] Bonas G, Zervou M, Papaeoannou T, Lees M: Accred Qual Assur (2003) 8:101-107

[5] DIN 1333:1992-02 Zahlenangaben

Annex 1: Homogeneity test

Cu:

		16.513	16.482	16.489		
		16.528	16.533	16.494		
		16.503	16.507	16.564		
		16.573	16.521	16.552		
		16.522	16.585	16.531		
		16.515	16.566	16.52		
		16.514	16.459	16.488		
		16.482	16.549	16.449		
		16.469	16.512	16.473		
		16.54	16.491	16.492		

Bottle	*Number*	*Sum*	*Mean*	*Variance*
1	3	49.484	16.4946667	0.00026433
2	3	49.555	16.5183333	0.00045033
3	3	49.574	16.5246667	0.00116433
4	3	49.646	16.5486667	0.00068433
5	3	49.638	16.546	0.001161
6	3	49.601	16.5336667	0.00079033
7	3	49.461	16.487	0.000757
8	3	49.48	16.4933333	0.00259633
9	3	49.454	16.4846667	0.00056433
10	3	49.523	16.5076667	0.00078433

ANOVA

Source of variation	*sums of squares (SS)*	*degrees of freedom (df)*	*Mean squares (MS)*	*F-value*	*P-value*	*critical F-value*
Between groups	0.01552613	9	0.00172513	1.87174603	0.11652922	2.39281411
Within groups	0.01843333	20	0.00092167			
Total	0.03395947	29				

Homogeneous.	Contribution to combined uncertainty:				0.00311692	
			relative:		0.00018875	

Ni:

		0.602	0.6	0.606		
		0.611	0.604	0.602		
		0.61	0.606	0.604		
		0.604	0.607	0.607		
		0.599	0.605	0.605		
		0.612	0.602	0.599		
		0.599	0.607	0.604		
		0.596	0.612	0.601		
		0.596	0.608	0.596		
		0.607	0.595	0.6		

Bottle	*Number*	*Sum*	*Mean*	*Variance*
1	3	1.808	0.6027	9.3333E-06
2	3	1.817	0.6057	2.2333E-05
3	3	1.82	0.6067	9.3333E-06
4	3	1.818	0.6060	3E-06
5	3	1.809	0.6030	0.000012
6	3	1.813	0.6043	4.6333E-05
7	3	1.81	0.6033	1.6333E-05
8	3	1.809	0.6030	6.7E-05
9	3	1.8	0.6000	4.8E-05
10	3	1.802	0.6007	3.6333E-05

ANOVA

Source of variation	*sums of squares (SS)*	*degrees of freedom (df)*	*Mean squares (MS)*	*F-value*	*P-value*	*critical F-value*
Between groups	0.00012947	9	1.4385E-05	0.53278464	0.83366015	2.39281411
Within groups	0.00054	20	0.000027			
Total	0.00066947	29				

Homogeneous.	Contribution to combined uncertainty:				0.00053348	
			relative:		0.00088393	

Ag:

		0.059	0.057	0.058		
		0.057	0.059	0.059		
		0.057	0.058	0.059		
		0.059	0.058	0.057		
		0.058	0.059	0.057		
		0.057	0.057	0.058		
		0.058	0.059	0.057		
		0.058	0.057	0.058		
		0.056	0.059	0.059		
		0.06	0.059	0.058		

Bottle	*Number*	*Sum*	*Mean*	*Variance*
1	3	0.174	0.0580	1E-06
2	3	0.175	0.0583	1.3333E-06
3	3	0.174	0.0580	1E-06
4	3	0.174	0.0580	1E-06
5	3	0.174	0.0580	1E-06
6	3	0.172	0.0573	3.3333E-07
7	3	0.174	0.0580	1E-06
8	3	0.173	0.0577	3.3333E-07
9	3	0.174	0.0580	3E-06
10	3	0.177	0.0590	1E-06

ANOVA

Source of variation	*sums of squares (SS)*	*degrees of freedom (df)*	*Mean squares (MS)*	*F-value*	*P-value*	*critical F-value*
Between grc	4.9667E-06	9	5.5185E-07	0.501683502	0.85606416	2.39281411
Within grouџ	2.2E-05	20	0.0000011			
Total	2.6967E-05	29				

Homogeneous.		Contribution to combined uncertainty:			0.000108	
				relative:	0.001855	

Pb:

		0.695	0.695	0.693		
		0.694	0.696	0.695		
		0.69	0.699	0.697		
		0.69	0.697	0.696		
		0.697	0.702	0.7		
		0.696	0.695	0.699		
		0.694	0.693	0.695		
		0.693	0.688	0.7		
		0.698	0.691	0.7		
		0.7	0.698	0.695		

Bottle	*Number*	*Sum*	*Mean*	*Variance*
1	3	2.083	0.6943	1.3333E-06
2	3	2.085	0.6950	0.000001
3	3	2.086	0.6953	2.2333E-05
4	3	2.083	0.6943	1.4333E-05
5	3	2.099	0.6997	6.3333E-06
6	3	2.09	0.6967	4.3333E-06
7	3	2.082	0.6940	0.000001
8	3	2.081	0.6937	3.6333E-05
9	3	2.089	0.6963	2.2333E-05
10	3	2.093	0.6977	6.3333E-06

ANOVA

Source of variation	*sums of squares (SS)*	*degrees of freedom (df)*	*Mean squares (MS)*	*F-value*	*P-value*	*critical F-value*
Between groups	9.6967E-05	9	1.0774E-05	0.93147614	0.51982188	2.39281411
Within groups	0.00023133	20	1.1567E-05			
Total	0.0003283	29				

Homogeneous.	Contribution to combined uncertainty:	0.00034918
	relative:	0.0005019

Cr:

		0.99	0.989	0.988		
		0.991	0.987	0.991		
		0.997	0.989	0.98		
		0.993	0.989	0.99		
		0.986	0.986	0.991		
		0.986	0.984	0.985		
		0.982	0.987	0.987		
		0.987	0.995	0.993		
		0.994	0.993	0.991		
		0.984	0.985	0.986		

Bottle	*Number*	*Sum*	*Mean*	*Variance*
1	3	2.967	0.989	0.000001
2	3	2.969	0.98966667	5.3333E-06
3	3	2.966	0.98866667	7.2333E-05
4	3	2.972	0.99066667	4.3333E-06
5	3	2.963	0.98766667	8.3333E-06
6	3	2.955	0.985	0.000001
7	3	2.956	0.98533333	8.3333E-06
8	3	2.975	0.99166667	1.7333E-05
9	3	2.978	0.99266667	2.3333E-06
10	3	2.955	0.985	0.000001

ANOVA

Source of variation	*sums of squares (SS)*	*degrees of freedom (df)*	*Mean squares (MS)*	*F-value*	*P-value*	*critical F-value*
Between group	0.0002068	9	2.2978E-05	1.89377289	0.11238273	2.39281411
Within groups	0.00024267	20	1.2133E-05			
Total	0.00044947	29				

Homogeneous.		Contribution to combined uncertainty:			0.00035763	
				relative:	0.00036177	

Sn:

		0.481	0.476	0.478		
		0.478	0.48	0.477		
		0.477	0.478	0.479		
		0.48	0.481	0.479		
		0.48	0.476	0.48		
		0.478	0.479	0.477		
		0.478	0.478	0.478		
		0.478	0.477	0.48		
		0.476	0.483	0.48		
		0.48	0.482	0.478		

Bottle	*Number*	*Sum*	*Mean*	*Variance*
1	3	1.435	0.4783	6.3333E-06
2	3	1.435	0.4783	2.3333E-06
3	3	1.434	0.4780	0.000001
4	3	1.44	0.4800	0.000001
5	3	1.436	0.4787	5.3333E-06
6	3	1.434	0.4780	0.000001
7	3	1.434	0.4780	0
8	3	1.435	0.4783	2.3333E-06
9	3	1.439	0.4797	1.2333E-05
10	3	1.44	0.4800	4E-06

ANOVA

Source of variation	*sums of squares (SS)*	*degrees of freedom (df)*	*Mean squares (MS)*	*F-value*	*P-value*	*critical F-value*
Between groups	1.8533E-05	9	2.0593E-06	0.57736241	0.79997327	2.39281411
Within groups	7.1333E-05	20	3.5667E-06			
Total	8.9867E-05	29				

Homogeneous. Contribution to combined uncertainty: 0.0001939

relative: 0.00040502

Estimate of inhomogeneity from the ILC (2way ANOVA)

comments

Element:	**Au**												
unit:	mg/kg												
	8	5	1	16	10	15	9	13	11	4	6	3	7
Bottle 1	49.9	51.3	50.64	61.7	49.5	50.8	53.8	52.17	51.94	53.5	53.252	56.87	66.3
	49.4	51.5	53.35	42.3	52.5	52.3	52.8	53.02	51.99	53.7	55.099	54.65	65.9
	50.5	51	49.68	51.3	50.2	53.3	50.1	52.87	53.63	51.7	55.55	56.55	62.5
Bottle 2	51.3	49.7	49.12	50.1	52.6	50.6	50.6	52.265	52.91	54.9	53.803	56.8	62
	50.7	49.8	51.63	50.4	53.1	51.1	53	52.66	54.22	53.4	55.829	53.35	63.6
	51.6	50.3	52.65	53.5	53.3	53.5	53	53.12	53.565	53.7	55.053	53.35	65.9

Source of variation	*sums of squares (SS)*	*degrees of freedom (df)*	*Mean squares (MS)*	*F-value*	*P-value*	comments
Sample	0.112532754	1	0.112532754	0.021311729	0.884497392	
Column	938.0111226	12	78.16759355	14.80357074	8.41965E-13	large deviations between labs (as expected)
Interaction	21.73227719	12	1.811023099	0.342976	0.976682923	
Error	274.5766502	52	5.280320196			
Total	1234.432583	77				

inhomogeneity contribution: 0 — repeatability is much worse than between-sample variation

Element: **Pt**

unit: mg/kg

	1	16	10	8	9	13	5	11	6	3	15	4	7
Bottle 1	4.878	6	4	5.4	6	6.45	5.6	6.1	6.099	6.27	6.5	6.5	9.6
	5.465	4.6	5	5.5	5	6.64	6.1	6	6.093	6.19	5.8	6.1	9.4
	5.21	5.4	5	5.7	5	5.87	6.6	5.41	6.106	6.28	5.8	7.1	9.7
Bottle 2	4.385	5.4	5	5.4	6	5.49	5.7	5.88	6.136	6.62	6.2	6.4	10.9
	5.227	3.5	5	5.4	6	5.66	5.6	6.31	6.164	6.39	6.1	5.8	9.8
	4.09	4.7	6	5.6	6	5.08	5.8	6.095	6.131	5.8	7.2	6.8	9.9

Source of	*sums of*	*degrees of*	*Mean squares*	*F-value*	*P-value*	
Sample	0.008266782	1	0.008266782	0.041348385	0.839660568	
Column	115.1470003	12	9.595583357	47.99471801	8.31929E-24	large deviations between labs
Interaction	5.458785718	12	0.45489881	2.275290547	0.020734089	
Error	10.39635933	52	0.199929987			
Total	131.0104121	77				

inhomogeneity contribution: 0 — repeatability is much worse than between-sample variation

Element: **Pd**

unit: mg/kg

	15	4	11	9	8	13	10	1	5	16	3	6	7
Bottle 1	46.7	46.5	46.97	48	47	47.14	47	47.98	48.7	49.7	48.97	50.39	52.2
	46.4	46.1	46.93	46	47	47.67	47	48.4	48.4	45.5	49.72	50.196	52.2
	46.7	45.2	46.27	48	47.4	48.12	48	48.13	49.5	50.8	49.98	50.468	52.7
Bottle 2	46.4	47.7	46.65	47	47.7	47.25	49	48.1	48.4	49.9	50.65	50.306	52.6
	46.6	46.6	46.92	47	47.6	47.78	48	48.16	48.3	48.4	50	50.576	50.9
	46.3	47.8	46.785	47	47.7	47.51	48	47.88	48.4	48.6	50.05	50.484	53.9

Source of	*sums of*	*degrees of*	*Mean squares*	*F-value*	*P-value*	
Sample	1.007996013	1	1.007996013	1.729705726	0.194218658	
Column	212.6358462	12	17.71965385	30.40665471	2.80713E-19	large deviations between labs
Interaction	5.490582154	12	0.457548513	0.785146243	0.662954867	
Error	30.30330067	52	0.582755782			
Total	249.437725	77				

inhomogeneity contribution: 0 — repeatability is much worse than between-sample variation

Element:	**As**								
unit:	mg/kg								
	3	15	20	16	13	10	9	7	6
Bottle 1	321.222	343.371	344.3	376	384	383	402	400.2	415.746
	318.92	339.422	349.9	373	393	388	401	401.2	428.242
	320.54	335.935	351.5	373	381	384	402	405.2	417.213
Bottle 2	318.84	334.23	342.5	375	392	383	398	399	437.743
	318.58	358.025	339.5	374	380	383	400	409.6	439.797
	322	333.624	340.7	376	365	386	397	406.8	437.376
Source of	*sums of*	*degrees of*	*Mean squares*	*F-value*	*P-value*				
Sample	3.84213363	1	3.84213363	0.124992598	0.725743564				
Column	57935.8867	8	7241.985838	235.596861	1.02655E-28				
Interaction	680.0696447	8	85.00870559	2.765509992	0.01717969				
Error	1106.600016	36	30.73888933						
Total	59726.39849	53							
inhomogeneity contribution:		0		repeatability is much worse than between-sample variation					

Element:	**Be**							
unit:	mg/kg							
	20	16	3	15	6	7	9	1/R
Bottle 1	5.25	6	6.06	6.434	6.7	7.2	9	9.3
	5.28	5.9	6.48	6.572	6.601	7	9	9.44
	5.25	6.3	6.51	6.699	6.638	6.8	9	9.65
Bottle 2	5.56	6	6.35	6.347	6.647	7.4	10	9.52
	5.36	6	6.66	6.596	6.703	7.4	9	9.19
	5.48	6.1	6.45	6.257	6.705	7.3	9	9.355
Source of	*sums of*	*degrees of*	*Mean squares*	*F-value*	*P-value*			
Sample	0.111747	1	0.111747	2.827481071	0.102399634			
Column	88.56565733	7	12.65223676	320.1335154	9.18738E-28			
Interaction	0.412687333	7	0.058955333	1.491718695	0.205554117			
Error	1.264696	32	0.03952175					
Total	90.35478767	47						
inhomogeneity contribution:		0		repeatability is worse than between-sample variation				

Element:	**Cd**								
unit:	mg/kg								
	20	15	3	13	16	6	7	1	9
Bottle 1	14.2	14.603	16.04	16.43	16.8	15.665	17.6	17	
	14.2	14.734	16.2	16.83	17.1	16.591	17.2	18.7	
	14.3	14.48	16.27	16.63	17	16.036	17.8	18.6	
Bottle 2	14.4	14.287	16.16	16.33	16.7	16.978	17.9	18.5	20
	14.4	14.401	16.22	16.33	15.6	17.597	18.5	17.5	21
	14.4	14.383	16.06	15.73	17.7	18.272	17.2	17.4	21

Source of	*sums of*	*degrees of*	*Mean squares*	*F-value*	*P-value*
Sample	0.078327521	1	0.078327521	0.33303836	0.567913619
Column	75.11008898	7	10.73001271	45.62260872	7.14147E-15
Interaction	4.326579313	7	0.618082759	2.62800694	0.029016887
Error	7.526102	32	0.235190688		
Total	87.04109781	47			

inhomogeneity contribution: 0 — repeatability is much worse than between-sample variation

Element:	**In**								
unit:	mg/kg								
	6	3	15	20	16	13	7	9	22/LIPS
Bottle 1	32.584	37.5	39.099	39.2	41	46.1	51.5	56	60.17
	35.26	37.7	39.129	38.8	42.7	44	52.05	58	56.04
	31.798	38.1	38.311	39.3	42.7	45.3	50.05	56	59.03
Bottle 2	35.416	37.9	38.261	38.9	38.7	40.2	50.35	54	61.28
	33.537	38.27	38.093	38.9	38.5	40.2	50.65	57	60.98
	34.436	37.88	38.49	39.3	38.6	41.4	50.25	54	58.44

Source of	*sums of*	*degrees of*	*Mean squares*	*F-value*	*P-value*
Sample	10.21641007	1	10.21641007	9.333930187	0.004218976
Column	3776.407054	8	472.0508818	431.2757557	2.30215E-33
Interaction	52.31186226	8	6.538982782	5.974154164	6.92467E-05
Error	39.40363333	36	1.09454537		
Total	3878.33896	53			

inhomogeneity contribution:	1.743737815
rel inhom contribution:	0.039375953