in cooperation with the WG 'Precious Metals'
of the Committee of Chemists of GDMB

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Certification Report

Certified Reference Material

ERM®-EZ505

Electronic Scrap

January 2011

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Summary

This report describes preparation, analysis and certification of the electronic scrap reference material ERM®-EZ505.
The certified reference material can be used for chemical analysis of minor elements and traces in similar materials. It is intended for development, validation and quality control of analytical methods and procedures.
Certified reference material ERM®-EZ505 is available as powder with a particle size < 150 µm.

The following mass fractions and uncertainties have been certified:

Element	**Mass fraction** in %	**Uncertainty** in %
Cu	15.10	0.11
Ni	0.470	0.008
	in mg/kg	in mg/kg
Ag	692	13
Au	292	4
Be	68.8	2.3
In	91	7
Pd	90.5	2.4
Pt	8.5	0.8

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 19 laboratories which participated in the certification interlaboratory comparison.

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List of abbreviations
(if not explained elsewhere)

CRM	certified reference material
ERM	European reference material
ETAAS	electrothermal atomic absorption spectrometry
FAAS	flame atomic absorption spectrometry
ICP-MS	inductively coupled plasma mass spectrometry
ICP-OES	inductively coupled plasma optical emission spectrometry
M	arithmetic mean of means
n	number of accepted data sets
NAA	neutron activation analysis
SD	standard deviation of an individual data set
SD_M	standard deviation of the mean of means

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 is the outcome of the discussions within the German Gesellschaft für Bergbau, Metallurgie, Rohstoff- und Umwelttechnik (GDMB) and especially the working group „precious metals“ of the committee of chemists within GDMB. From this working group the needs are defined, since the members are potential users of the prepared CRMs. Secondly from this group the participating laboratories are recruited. Since all of these 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 materials is carried out on the basis of the relevant ISO-Guides [1-3], the „Guidelines for the production of BAM Reference Materials“ [4] and the “Technical Guidelines for the Production and Acceptance of a European Reference Material” [5].

Starting material for the preparation of CRM ERM®-EZ505 were approx. 40 kg of used mixed printed circuit boards.

2. Companies/laboratories involved

Preparation of the material:
- G + S Gesellschaft für Labortechnik und Probenaufbereitung mbH

Test for homogeneity:

- Allgemeine Gold- und Silberscheideanstalt AG
- W.C. Heraeus GmbH
- Institut für Materialprüfung Glörfeld GmbH
- BAM Bundesanstalt für Materialforschung und -prüfung

Participants in the certification interlaboratory comparison:

- Alfred Knight Int. Ltd, St. Helens (United Kingdom)
- Allgemeine Gold- und Silberscheideanstalt AG, Pforzheim (Germany)
- AMI Doduco GmbH, Pforzheim (Germany)
- Aurubis AG, Hamburg (Germany)
- BAM Bundesanstalt für Materialforschung und -prüfung, Berlin (Germany)
- Forschungsinstitut Edelmetalle & Metallchemie, Schwäbisch Gmünd (Germany)
- Horiba Scientific, Unterhaching, (Germany)
- Inspectorate International Ltd, Reno NV (United States)
- Inspectorate International Ltd, Witham (United Kingdom)
- Institut für Materialprüfung Glörfeld GmbH, Willich (Germany)
- Ledoux & Company, Teanec NJ (United States)
- Nickelhütte Aue GmbH, Aue (Germany)

- SAXONIA Edelmetallrecycling GmbH, Halsbrücke (Germany)
- Umicore AG & Co. KG, Hanau (Germany)
- Umicore Precious Metals, Hoboken (Belgium)
- Varian, Darmstadt (Germany)
- W.C. Heraeus GmbH, Hanau (Germany)
- Wieland Edelmetalle GmbH & Co, Pforzheim (Germany)
- WRC World Resources Company GmbH, Wurzen (Germany)

Statistical evaluation of the data
- BAM Bundesanstalt für Materialforschung und -prüfung, Berlin

3. Candidate material

Approx. 40 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. 40 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 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 250 bottles were filled.

4. Homogeneity testing

Three laboratories performed analyses for homogeneity testing. Each laboratory received five randomly chose bottles (Table 1). The laboratories were asked to analyse all elements once in four bottles and four times in the fifth bottle. Lab. 3 carried out additional analyses on all five bottles (3 to 6 single results depending on the element).

Table 1: Homogeneity testing, chosen bottles (in brackets: number of single results)

Laboratory-No.	**1**	**2**	**3**
Bottle-No.	3 (4)	10 (1)	122 (3-6)
	54 (1)	28 (1)	143 (3-6)
	111 (1)	48 (1)	159 (3-6)
	202 (1)	118 (4)	179 (3-6)
	222 (1)	147 (1)	241 (3-6)

From the data of each laboratory an estimator for the inhomogeneity contribution was calculated according to Eqs. 1 - 6.

$$MS_{bw,i} = \frac{\sum_{k=1}^{n_i} dev_{k,i}}{n_i - 1} \quad (1)$$

n_i = number of bottles tested in the ith laboratory ($k = 1 \ldots n_i$)

$$dev_{k,i} = p_{k,i} \cdot \left(M_{bottle_k} - M_{total,i}\right)^2 \quad (2)$$

with M being mean values (for the bottle or grand mean, respectively; bw means “between”, wi means “within”), and $p_{k,i}$ the number of single values obtained for bottle *k* in laboratory *i.*

$$MS_{wi,i} = \frac{\sum_{k=1}^{n_i^*} s_{k,i}^2}{n_i^*} \qquad (3)$$

$s_{k,i}$ = standard deviation of single values obtained on bottle *k*
n_i^* = number of estimates $s_{k,i}$ available in laboratory *i* (n_i^* = 1 for laboratories 1 and 2)

$MS_{bw,i} > MS_{wi,i}$:

$$s_{bb,i} = \sqrt{\frac{MS_{bw,i} - MS_{wi,i}}{f}} \qquad (4)$$

$MS_{bw,i} < MS_{wi,i}$:

$$s_{bb,i,\min} = \sqrt{\frac{MS_{wi,i}}{f}} \cdot \sqrt{\frac{2}{p_i - 1}} \qquad (5)$$

for laboratories 1 and 2 with an effective degree of freedom *f* = 1.4 and

$MS_{bw,i} < MS_{wi,i}$

$$s_{bb,i,\min} = \sqrt{\frac{MS_{wi,i}}{f}} \cdot \sqrt{\frac{2}{\sum_{k=1}^{n_i} p_{k,i} - n_i}} \qquad (6)$$

for laboratory 3 with *f* = 3.5. Note that in Equation 5 (for laboratories 1 and 2), the p_i refers to the number of replicate measurements taken from one single bottle, the only data providing $MS_{wi,i}$ estimates for these laboratories.

Table 2 Homogeneity testing, sample intake in g

Laboratory-No.	**1**	**2**	**3**
Element			
Cu	5	5	4.5
Ni	5	5	4.5
In	5	5	4.5
Ag	5	5	2.3
Au	5	5	2.3
Pd	5	5	2.3
Pt	5	5	2.3
Be	5	5	4.5

Comparing $s_{bb,i}$ with $s_{bb,i,min}$ the higher value was used as an estimator of $u_{bb,i}$ for laboratory *i,* and converted into a relative uncertainty contribution. The averaged relative estimator for inhomogeneity u_{bb} to be included in the uncertainty budget of the certified value in

accordance with [3] was then calculated by quadratic averaging of the resp. relative laboratory estimators. Table 3 shows the resp. estimators for inhomogeneity.

Since most of the data for inhomogeneity estimation were produced on the basis of a sample intake of 5 g, the minimum sample intake for the use of the certified reference material is 5 g.

Table 3: Estimated inhomogeneity, u_{bb} ($u_{bb,abs}$ for Cu, Ni in %, for all other elements in mg/kg)

Element	$u_{bb,rel}$	$u_{bb,abs}$
Cu	0.27 %	0.0406
Ni	0.47 %	0.00221
In	2.32 %	2.11
Ag	0.76 %	5.25
Au	0.43 %	1.26
Pd	0.88 %	0.797
Pt	3.60 %	0.307
Be	0.78 %	0.537

5. Stability

There is no instability of the certified material to be expected (calcinated inorganic material) if the material is stored at ambient temperature. To be sure that no oxidation of the matrix appears, the mass of three individual bottles will be checked regularily every six month. An expiry date of 10 years is given.

6. Certification study

6.1 Analytical methods

19 laboratories participated in the certification interlaboratory comparison. Each laboratory received one randomly chosen bottle with approx. 200 g of powder. Before analysis the material had to be dried at 105 °C for at least 8 hours.
The laboratories were told to analyse six subsamples. They were free to choose any suitable analytical method for analysis. Tables 5 to 12 show the analytical methods used by the participating laboratories. It is noticeable that often the sample intake is below 5 g, the sample intake used for homogeneity testing. The spread of single results from laboratories using less than 5 g for analysis gave no hint that the material was not homogeneous (n.a. means not available).
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 5: Analytical procedures for the determination of copper

Method (Abrev.)	Lab-No.	Sample mass	Sample pretreatment	Analytical method
I	6	1 g	Aqua regia/microwave	ICP OES with Sc as internal standard
E	4	3 g		Electrogravimetry
E	15	n.a.	Dissolution with HNO_3/H_2SO_4, fuming with Br_2/HBr	Electrogravimetry
I	16, 18	n.a.	Dissolution with aqua regia	ICP OES
E	19	2 g	Dissolution with aqua regia, fuming with H_2SO_4	Electrogravimetry
E	1	n.a.	3 x acid dissolution, 3 x dissolution with aqua regia	Electrogravimetry X-ray fluorescence
I	14	0.2 g	Dissolution with HNO_3	ICP OES with Y as internal standard
E	10	2 g	Dissolution in acid mixture	Electrogravimetry
A	13	1 g	Fusion with Na_2O_2 and Na/K-carbonate, dissolution in HCl	FAAS
I	3	n.a.	n.a.	ICP OES bracketing
I	7	4.5 g	HCl-digestion/HNO_3/Na_2O_2 fusion	ICP OES
E	5	n.a.	Dissolution in HNO_3, Addition of HCl and HF, fuming with H_2SO_4	Electrogravimetry

Table 6: Analytical procedures for the determination of nickel

Method (Abrev.)	Lab-No.	Sample mass	Sample pretreatment	Analytical method
I	6	1 g	Aqua regia/microwave	ICP OES with Sc as internal standard,
I	4	1 g	Dissolution in acids	ICP OES
I	15	n.a.	Dissolution with HNO_3/H_2SO_4, fuming with Br_2/HBr	ICP OES
I	16, 18	n.a.	Dissolution with aqua regia	ICP OES
I	1	0.25 g	Microwave dissolution	ICP OES
X	1	n.a.	Melting with $K_2S_2O_7$	X-ray fluorescence
I	9	0.4 g	Dissolution with HCl/aqua regia, melting with Na_2O	ICP OES
I	14	0.2 g	Dissolution with HNO_3	ICP OES with Y as internal standard
A	10	2 g	Dissolution in acid mixture	FAAS
A	13	1 g	Fusion with Na_2O_2 and Na/K-carbonate, dissolution in HCl	FAAS
I	3	n.a.	n.a.	ICP OES bracketing
I	7	4.5 g	HCl-digestion/HNO_3/Na_2O_2 fusion	ICP OES
I	5	n.a.	Acid dissolution	ICP OES

Table 7: Analytical procedures for the determination of beryllium

Method (Abrev.)	Lab-No.	Sample mass	Sample pretreatment	Analytical method
I	6	1 g	Aqua regia/microwave	ICP OES with Sc as internal standard,
E	15	n.a.	HNO_3/HF/microwave	ETAAS
I	16	n.a.	Dissolution with aqua regia	ICP OES
I	19	2 g	Dissolution with aqua regia, fuming with H_2SO_4, decomposition of residue with Na_2O_2	ICP OES
I	1	0.25 g	Microwave dissolution	ICP OES
I	9	1 g	Dissolution with aqua regia	ICP OES
I	14	2 g	Dissolution with HNO_3	ICP OES with Y as internal standard
A	10	1 g	Dissolution with acid mixture/ alkaline fusion	FAAS
I	3	n.a.	n.a.	ICP OES bracketing
I	5	n.a.	Acid dissolution	ICP OES

Table 8: Analytical procedures for the determination of indium

Method (Abrev.)	Lab-No.	Sample mass	Sample pretreatment	Analytical method
I	6	1 g	Aqua regia/microwave	ICP OES with Sc as internal standard,
E	15	n.a.	HNO_3/HF/microwave	ETAAS
I	16	n.a.	Dissolution with aqua regia	ICP OES
I	19	2 g	Dissolution with aqua regia, fuming with H_2SO_4, decomposition of residue with Na_2O_2	ICP OES
I	1	0.25 g	Microwave dissolution	ICP OES
I	10	1 g	Dissolution in acid mixture	ICP OES
I	5	n.a.	Acid dissolution	ICP OES
I	3	5 g	Dissolution in HCl/HNO_3/HF	ICP OES
NAA	20	0.1 g		Neutron activation analysis
I	21	5 g	Dissolution in HCl/HNO_3/HF	ICP OES
I	22	5 g	Dissolution in HCl/HNO_3/HF	ICP OES
IMS	20	5 g	Dissolution in HCl/HNO_3/HF	ICP-MS

Table 9: Analytical procedures for the determination of gold

Method (Abrev.)	Lab-No.	Sample mass	Sample pretreatment	Analytical method
I	6	1 g	Fire assay with lead, dissolution in aqua regia	ICP OES with Sc as internal standard,
G	4	10 g	Fire assay with lead	Gravimetry
I	2	n.a.	Fire assay with lead, dissolution in aqua regia	ICP OES with Sc as internal standard,
I	15	5 g	Fire assay with lead	ICP OES
I	16	n.a.	Melting with nitrate	ICP OES
I	18	n.a.	Fire assay with lead	ICP OES
O	1	n.a.	Fire assay with lead	Spark-OES
I	1	n.a.	Fire assay, lead collection, dissolution in aqua regia	ICP OES
I	14	10 g	Fire assay, collection with Cu_2O, dissolution in aqua regia	ICP OES with Y as internal standard
I	10	10 g	Fire assay, collection with lead	ICP OES
G	13	5 g	Fire assay with litharge flux/ dilution in HNO_3	Gravimetry
I	3	n.a.	n.a.	ICP OES bracketing
I	7	2.3 g	Fire assay, collection with lead	ICP OES
I	5	n.a.	Fire assay, collection with lead	ICP OES, In as internal standard

Table 10: Analytical procedures for the determination of silver

Method (Abrev.)	Lab-No.	Sample mass	Sample pretreatment	Analytical method
I	6	1 g	Fire assay with lead, dissolution in aqua regia	ICP OES with Sc as internal standard,
I	2	n.a.	Fire assay with lead, dissolution in aqua regia	ICP OES with Sc as internal standard,
I	15	5 g	Fire assay with lead	ICP OES
I	16	n.a.	Melting with nitrate	ICP OES
I	18	n.a.	Fire assay with lead	ICP OES
O	1	n.a.	Fire assay with lead	Spark-OES
A	10	1 g	Dissolution in acid mixture	FAAS
A	13	1 g	Fusion with Na_2O_2 and Na/K-carbonate, dissolution in HCl	FAAS
I	7	2.3 g	Fire assay, collection with lead	ICP OES
I	5	n.a.	Fire assay, collection with lead	ICP OES, In as internal standard

Table 11: Analytical procedures for the determination of palladium

Method (Abrev.)	Lab-No.	Sample mass	Sample pretreatment	Analytical method
I	6	1 g	Fire assay with lead, dissolution in aqua regia	ICP OES with Sc as internal standard,
I	4	10 g	Fire assay with lead	ICP OES
I	2	n.a.	Fire assay with lead, dissolution in aqua regia	ICP OES with Sc as internal standard,
I	15	5 g	Fire assay with lead	ICP OES
I	16	n.a.	Melting with nitrate	ICP OES
I	18	n.a.	Fire assay with lead	ICP OES
I	19	5 g	Fire assay, Cu-collection	ICP OES with internal standard
O	1	n.a.	Fire assay with lead	Spark-OES
I	1	n.a.	Fire assay, lead collection, dissolution in aqua regia	ICP OES
I	9	5 g	Fire assay with lead, dissolution in aqua regia	ICP OES
I	14	10 g	Fire assay, collection with Cu_2O, dissolution in aqua regia	ICP OES with Y as internal standard
I	10	10 g	Fire assay, collection with lead	ICP OES
I	13	5 g	Fire assay with litharge flux/ dilution in HNO_3	ICP OES
I	3	5 g	Fire assay, nitrate fusion	ICP OES
I	7	2.3 g	Fire assay, collection with lead	ICP OES
I	5	n.a.	Fire assay, collection with lead	ICP OES, In as internal standard

Table 12: Analytical procedures for the determination of platinum

Method (Abrev.)	Lab-No.	Sample mass	Sample pretreatment	Analytical method
I	6	1 g	Fire assay with lead, dissolution in aqua regia	ICP OES with Sc as internal standard,
I	4	10 g	Fire assay with lead	ICP OES
I	2	n.a.	Fire assay with lead, dissolution in aqua regia	ICP OES with Sc as internal standard,
I	15	5 g	Fire assay with lead	ICP OES
I	16	n.a.	Nitrate fusion	ICP OES
I	18	n.a.	Fire assay with lead	ICP OES
I	19	2 g	Fire assay, Pb-collection	ICP OES with internal standard
O	1	n.a.	Fire assay with lead	Spark-OES
I	1	n.a.	Fire assay, lead collection, dissolution in aqua regia	ICP OES
I	9	5 g	Fire assay with lead, dissolution in aqua regia	ICP OES
I	14	10 g	Fire assay, collection with Cu_2O, dissolution in aqua regia	ICP OES with Y as internal standard
I	10	10 g	Fire assay, collection with lead	ICP OES
I	13	5 g	Fire assay with litharge flux/ dilution in HNO_3	ICP OES
I	3	5 g	Fire assay, nitrate fusion	ICP OES
I	7	2.3 g	Fire assay, collection with lead	ICP OES
I	5	n.a.	Fire assay, collection with lead	ICP OES, In as internal standard

6.2 Analytical results and statistical evaluation

The analytical results of the certification interlaboratory comparison are listed in Tables 15 to 22. These tables show the single results (EW) of each laboratory, the resp. laboratories' mean values (MW) together with the innerlaboratory standard deviation (s) and in addition the mean standard deviation ($\overline{s}$) of all laboratories. The continous line markes 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.

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

Copper:

Number of data sets	13
Snedecor-F-Test and Bartlett-Test	Pooling not allowed
Dixon (a = 0.05)	---
Dixon (a = 0.01)	---
Nalimov (a = 0.05)	Laboratory 14
Nalimov (a = 0.01)	---
Grubbs (a = 0.05)	---
Grubbs (a = 0.01)	---
Grubbs Pair (a = 0.05)	---
Grubbs Pair (a = 0.01)	---
Cochran	---
Kolmogorov-Smirnov-Lilliefors Test (a = 0.05)	Distribution: normal
Kolmogorov-Smirnov-Lilliefors Test (a = 0.01)	Distribution: normal

The outlying value (Lab. 14) was not removed.

Nickel:

Number of data sets	13
Snedecor-F-Test and Bartlett-Test	Pooling not allowed
Dixon (a = 0.05)	---
Dixon (a = 0.01)	---
Nalimov (a = 0.05)	Laboratory 6
Nalimov (a = 0.01)	---
Grubbs (a = 0.05)	---
Grubbs (a = 0.01)	---
Grubbs Pair (a = 0.05)	---
Grubbs Pair (a = 0.01)	---
Cochran	---
Kolmogorov-Smirnov-Lilliefors Test (a = 0.05)	Distribution: normal
Kolmogorov-Smirnov-Lilliefors Test (a = 0.01)	Distribution: normal

The outlying value (Lab. 6) was not removed.

Indium:

Number of data sets	12
Snedecor-F-Test and Bartlett-Test	Pooling not allowed
Dixon (a = 0.05)	---
Dixon (a = 0.01)	---
Nalimov (a = 0.05)	---
Nalimov (a = 0.01)	---
Grubbs (a = 0.05)	---
Grubbs (a = 0.01)	---
Grubbs Pair (a = 0.05)	---
Grubbs Pair (a = 0.01)	---
Cochran	---
Kolmogorov-Smirnov-Lilliefors Test (a = 0.05)	Distribution: not normal
Kolmogorov-Smirnov-Lilliefors Test (a = 0.01)	Distribution: normal

Beryllium:

Number of data sets	10
Snedecor-F-Test and Bartlett-Test	Pooling not allowed
Dixon (a = 0.05)	---
Dixon (a = 0.01)	---
Nalimov (a = 0.05)	Laboratory 10
Nalimov (a = 0.01)	---
Grubbs (a = 0.05)	---
Grubbs (a = 0.01)	---
Grubbs Pair (a = 0.05)	---
Grubbs Pair (a = 0.01)	---
Cochran	---
Kolmogorov-Smirnov-Lilliefors Test (a = 0.05)	Distribution: normal
Kolmogorov-Smirnov-Lilliefors Test (a = 0.01)	Distribution: normal

The outlying value (Lab. 10) was not removed.

Silver:

Number of data sets	12
Snedecor-F-Test and Bartlett-Test	Pooling not allowed
Dixon (a = 0.05)	---
Dixon (a = 0.01)	---
Nalimov (a = 0.05)	Laboratory 2
Nalimov (a = 0.01)	---
Grubbs (a = 0.05)	---
Grubbs (a = 0.01)	---
Grubbs Pair (a = 0.05)	---
Grubbs Pair (a = 0.01)	---
Cochran	---
Kolmogorov-Smirnov-Lilliefors Test (a = 0.05)	Distribution: normal
Kolmogorov-Smirnov-Lilliefors Test (a = 0.01)	Distribution: normal

The outlying value (Lab. 2) was not removed.

Palladium:

Number of data sets	16
Snedecor-F-Test and Bartlett-Test	Pooling not allowed
Dixon (a = 0.05)	---
Dixon (a = 0.01)	---
Nalimov (a = 0.05)	Laboratory 19
Nalimov (a = 0.01)	---
Grubbs (a = 0.05)	---
Grubbs (a = 0.01)	---
Grubbs Pair (a = 0.05)	---
Grubbs Pair (a = 0.01)	---
Cochran	---
Kolmogorov-Smirnov-Lilliefors Test (a = 0.05)	Distribution: normal
Kolmogorov-Smirnov-Lilliefors Test (a = 0.01)	Distribution: normal

The outlying value (Lab. 19) was not removed.

Gold:

Number of data sets	11
Snedecor-F-Test and Bartlett-Test	Pooling not allowed
Dixon (a = 0.05)	---
Dixon (a = 0.01)	---
Nalimov (a = 0.05)	Laboratory 10
Nalimov (a = 0.01)	---
Grubbs (a = 0.05)	---
Grubbs (a = 0.01)	---
Grubbs Pair (a = 0.05)	---
Grubbs Pair (a = 0.01)	---
Cochran	---
Kolmogorov-Smirnov-Lilliefors Test (a = 0.05)	Distribution: normal
Kolmogorov-Smirnov-Lilliefors Test (a = 0.01)	Distribution: normal

The outlying value (Lab. 10) was not removed.

Platinum:

Number of data sets	15
Snedecor-F-Test and Bartlett-Test	Pooling not allowed
Dixon (a = 0.05)	---
Dixon (a = 0.01)	---
Nalimov (a = 0.05)	---
Nalimov (a = 0.01)	---
Grubbs (a = 0.05)	---
Grubbs (a = 0.01)	---
Grubbs Pair (a = 0.05)	---
Grubbs Pair (a = 0.01)	---
Cochran	---
Kolmogorov-Smirnov-Lilliefors Test (a = 0.05)	Distribution: normal
Kolmogorov-Smirnov-Lilliefors Test (a = 0.01)	Distribution: normal

The certified mass fractions of all elements were calculated as mean of the accepted data sets. These values are given in Table 13.
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.

$$u_{\text{combined}} = \sqrt{\frac{s_{\text{ilc}}^2}{n} + u_{bb}^2} \qquad (7)$$

with

$\frac{s_{ilc}^2}{n}$: spread resulting from interlaboratory comparison

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

Table 13: Uncertainty calculation

	MW	***n***	s_{ilc}	u_{bb}
copper	15.10 %	14	0.11 %	0.041 %
nickel	0.47 %	14	0.01 %	0.00221 %
silver	692 mg/kg	13	13 mg/kg	5.26 mg/kg
gold	292 mg/kg	15	5 mg/kg	1.26 mg/kg
beryllium	68.8 mg/kg	10	3.1 mg/kg	0.54 mg/kg
indium	91 mg/kg	11	8.9 mg/kg	2.11 mg/kg
palladium	90.5 mg/kg	16	3.6 mg/kg	0.8 mg/kg
platinum	8.5 mg/kg	16	0.67 mg/kg	0.31 mg/kg

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

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

Table 14 shows the certified mass fractions and their resp. expanded uncertainties.

Table 14: Certified values of CRM ERM®-EZ505

Element	**Mass fraction** in %	**Uncertainty** in %
Cu	15.10	0.11
Ni	0.470	0.008
	in mg/kg	in mg/kg
Ag	692	13
Au	292	4
Be	68.8	2.3
In	91	7
Pd	90.5	2.4
Pt	8.5	0.8

Lab./Meth.	14/I	13/A	16/I(R)	15/E	18/I	1/E+X	5/E	7/I	4/E	19/E	10/E	3/I	6/I		Ges.
EW [%]	14.89	14.96	14.98	15.03	15.07	15.13	15.09	15.13	15.14	15.14	15.21	15.21	15.29		N
	14.85	14.99	14.99	15.04	14.99	15.08	15.14	15.15	15.16	15.20	15.21	15.22	15.38		13
	14.92	15.05	14.99	15.00	15.03	15.08	15.07	15.08	15.16	15.20	15.16	15.25	15.27		
	14.84	14.84	15.01	15.01	15.06	15.09	15.09	15.14	15.17	15.18	15.24	15.18	15.24		
	14.93	14.99	15.06	15.08	15.08	15.02	15.14	15.14	15.16	15.20	15.20	15.17	15.29		
	14.94	15.07	14.99	15.05	15.11	15.14	15.09	15.15	15.11	15.19	15.19	15.19	15.35		
MW [%]	**14.90**	**14.98**	**15.01**	**15.04**	**15.06**	**15.09**	**15.10**	**15.13**	**15.15**	**15.19**	**15.20**	**15.20**	**15.30**		**15.10**
s[%]	0.042	0.081	0.031	0.029	0.042	0.043	0.029	0.026	0.022	0.023	0.026	0.029	0.052		0.109
$\bar{s}$ [%]															0.037
s_{rel}	0.003	0.005	0.002	0.002	0.003	0.003	0.002	0.002	0.001	0.002	0.002	0.002	0.003		0.007
	15.1	15.1	15.1	15.1	15.1	15.1	15.1	15.1	15.1	15.1	15.1	15.1	15.1		
	15.0	15.0	15.0	15.0	15.0	15.0	15.0	15.0	15.0	15.0	15.0	15.0	15.0		
	15.2	15.2	15.2	15.2	15.2	15.2	15.2	15.2	15.2	15.2	15.2	15.2	15.2		

Extraction with Aqua Regia

HCl/Aqua Regia/Na2O2

HNO3/H2SO4, Fuming with Br2/HBr

Aqua Regia/Fuming with H2SO4/Na2O2-melt

HNO3

Decomposition with Na-peroxide/Na-/K-carbonate

HNO3/HCl/HF/Fuming with H2SO4

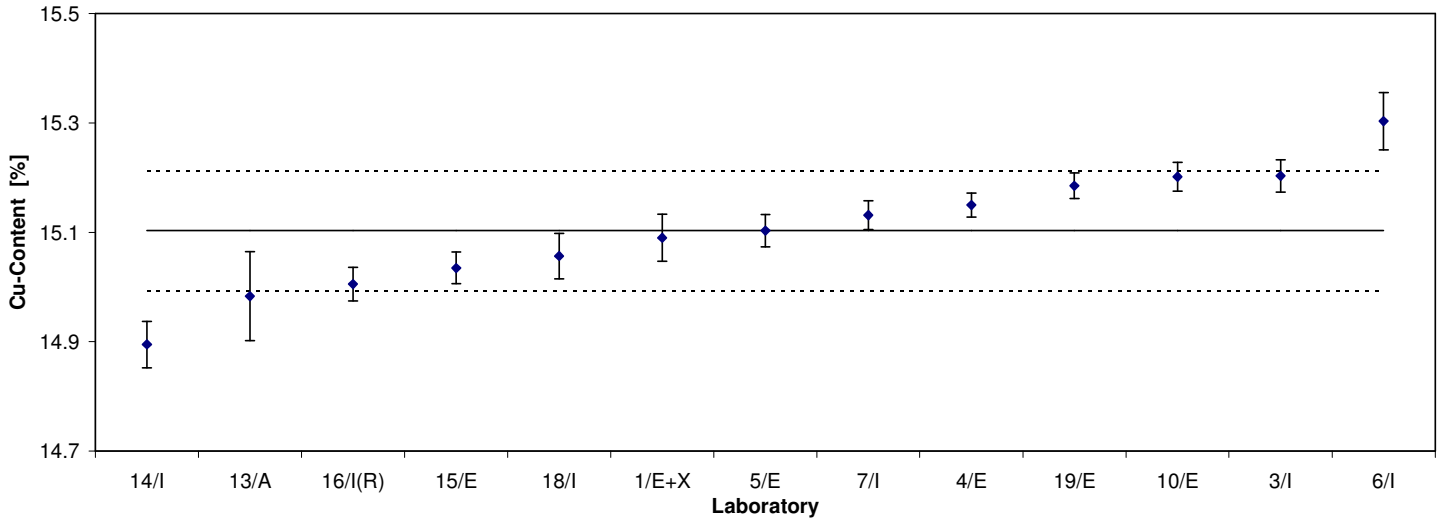

Table 15: Results for copper

Lab./Meth.	14/I	15/I	13/A	5/I	1/I	7/I	18/I	3/I	1/X	9/I	4/I	10/A	16/I	6/I		Ges.
EW [%]	0.455	0.457	0.465	0.464	0.460	0.466	0.468	0.469	0.467	0.475	0.470	0.481	0.48	0.492		N
	0.453	0.454	0.454	0.466	0.470	0.470	0.464	0.472	0.481	0.474	0.472	0.488	0.48	0.495		13
	0.457	0.454	0.444	0.462	0.470	0.467	0.469	0.469	0.469	0.475	0.477	0.472	0.48	0.490		
	0.446	0.456	0.466	0.468	0.470	0.469	0.474	0.473	0.472	0.473	0.476	0.472	0.48	0.497		
	0.454	0.461	0.471	0.471	0.465	0.468	0.472	0.469		0.473	0.473	0.479	0.48	0.491		
	0.454	0.456	0.467	0.468	0.467	0.467	0.470	0.471		0.473	0.481	0.469	0.48	0.492		
MW [%]	**0.453**	**0.456**	**0.461**	**0.467**	**0.467**	**0.468**	**0.470**	**0.471**	**0.472**	**0.474**	**0.475**	**0.477**	**0.480**	**0.493**		**0.470**
s[%]	0.0038	0.0026	0.0101	0.0032	0.0040	0.0015	0.0034	0.0018	0.0062	0.0010	0.0040	0.0071	0.0000	0.0026		0.010
$\bar{s}$ [%]																0.004
s_{rel}	0.0083	0.0057	0.0220	0.0069	0.0086	0.0031	0.0073	0.0037	0.0131	0.0021	0.0084	0.0150	0.0000	0.0054		0.021
	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5		
	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5		
	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5		

Extraction with Aqua Regia

HCl/Aqua Regia/Na2O2

HNO3/H2SO4, Fuming with Br2/HBr

Aqua Regia/Fuming with H2SO4/Na2O2-melt

HNO3

Decomposition with Na-Peroxid/Na-/K-Carbonat

Acid mixture

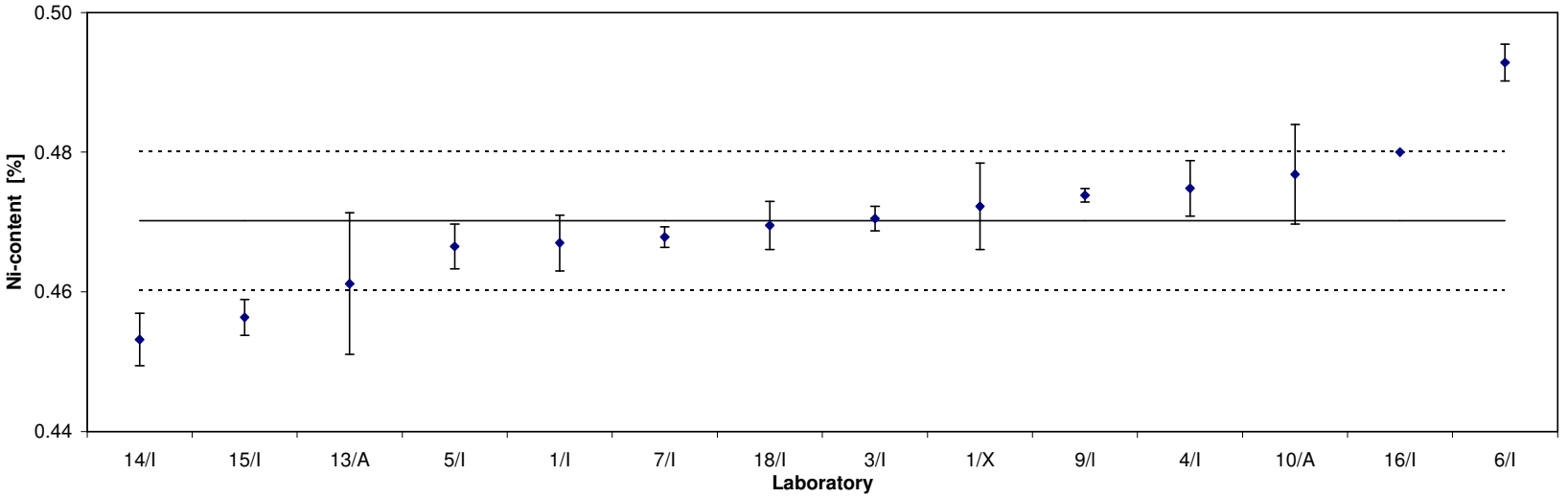

Table 16: Results for nickel

Lab./Meth.	15/EA	6/I	20/NAA	16/I	3/I	20MS	22/I	1/I	21/MS	5/I	19/I	10/I		Ges.
EW [µg/g]	76.8	79.3	81.3	79.5	79.1	81.0	90	96	92.7	100.0	102.8	103.0		N
	77.6	78.4	82.1	79.9	78.9	81.5	89	93	97.4	98.0	103.1	103.4		12
	77.8	79.5	80.5	80.8	85.1	82.6	91	93	92.4	96.5	101.4	104.8		
	75.6	79.4	80.0	84.7	83.9			91	93.7	97.5	101.2	104.4		
	80.1	80.1	81.2	82.2	84.2			90	95.4	98.5	101.1	104.3		
	78.3	80.2	81.6	83.0	78.9			89	95.4	96.5	103.1	104.0		
MW [µg/g]	**77.7**	**79.5**	**81.1**	**81.7**	**81.7**	**81.7**	**90.0**	**92.0**	**94.5**	**97.8**	**102.1**	**104.0**		**90.7**
s[µg/g]	1.51	0.65	0.76	1.99	3.00	0.82	1.00	2.53	1.91	1.33	0.98	0.67		8.88
$\bar{s}$ [µg/g]														1.43
s_{rel}	0.02	0.01	0.01	0.02	0.04	0.01	0.01	0.03	0.02	0.01	0.01	0.01		0.10
	90.7	90.7	90.7	90.7	90.7	90.7	90.7	90.7	90.7	90.7	90.7	90.7		
	81.8	81.8	81.8	81.8	81.8	81.8	81.8	81.8	81.8	81.8	81.8	81.8		
	99.5	99.5	99.5	99.5	99.5	99.5	99.5	99.5	99.5	99.5	99.5	99.5		

Extraction with Aqua Regia
Acid mixture
HCl/HNO3/HF
Aqua Regia/Fuming with H2SO4/Na2O2-melt
Microwave HNO3/HF

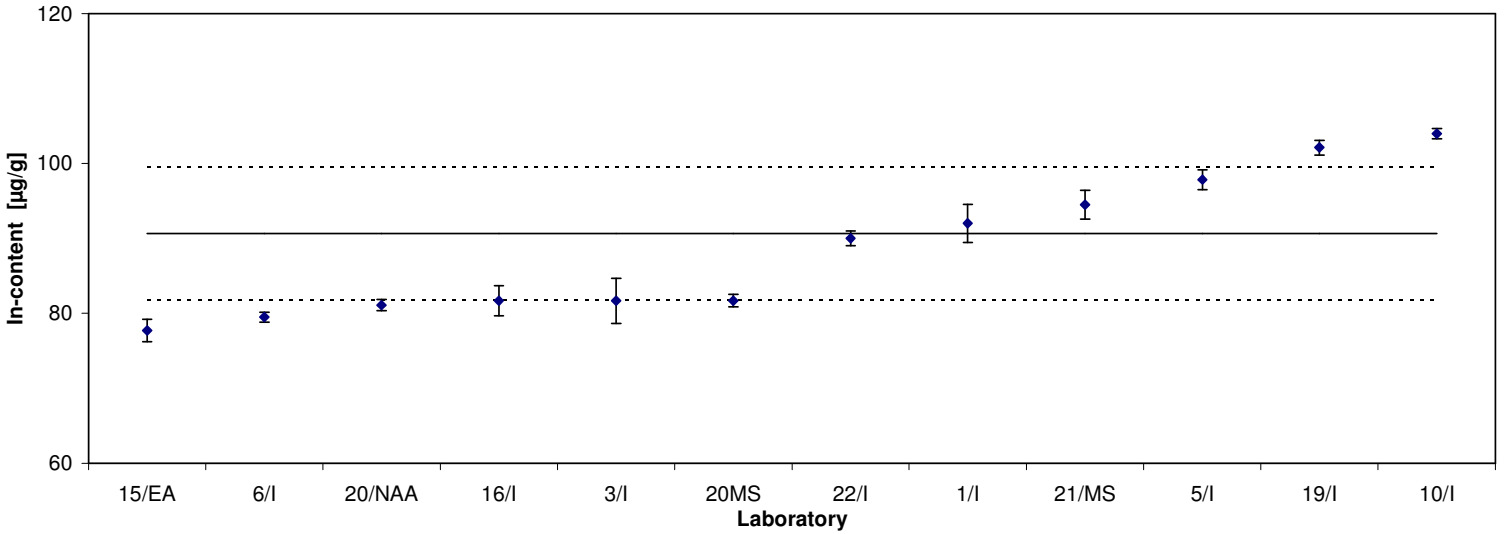

Table 17: Results for indium

Lab./Meth.	16/I	15/I	13/A	10/A	5/I	3/I	1/O	18/I	20/A	6/I	7/I	2/I		Ges.
EW [µg/g]	663.4	673.3	677.4	680.2	687.0	690.1	704	697	726	680.3	693	730		N
	668.5	680.1	678.5	685.3	680.0	692.5	686	692	710	686.1	710	693		12
	677.8	683.4	677.0	680.4	680.0	685.2	692	714	680	664.0	700	699		
	692.2	680.9	688.7	679.6	683.0	693.4	704	704	684	719.1	710	719		
	661.5	687.1	688.6	689.4	686.5	688.1	693	712	695	743.1		740		
			684.8	682.6	687.5	685.4	688	671	708	716.7		709		
MW [µg/g]	**672.7**	**680.9**	**682.5**	**682.9**	**684.0**	**689.1**	**694.5**	**698.3**	**700.5**	**701.6**	**703.3**	**715.0**		**692.1**
s[µg/g]	12.61	5.08	5.54	3.81	3.48	3.49	7.79	15.83	17.43	29.54	8.30	18.12		12.08
$\bar{s}$ [µg/g]														10.92
s_{rel}	0.02	0.01	0.01	0.01	0.01	0.01	0.01	0.02	0.02	0.04	0.01	0.03		0.02
	692.1	692.1	692.1	692.1	692.1	692.1	692.1	692.1	692.1	692.1	692.1	692.1		
	680.0	680.0	680.0	680.0	680.0	680.0	680.0	680.0	680.0	680.0	680.0	680.0		
	704.2	704.2	704.2	704.2	704.2	704.2	704.2	704.2	704.2	704.2	704.2	704.2		

Extraction with Aqua Regia | HCl/Aqua Regia/Na2O2 | Acid mixture

Lead collection | Decomposition with Na-Peroxid/Na-/K-Carbonat

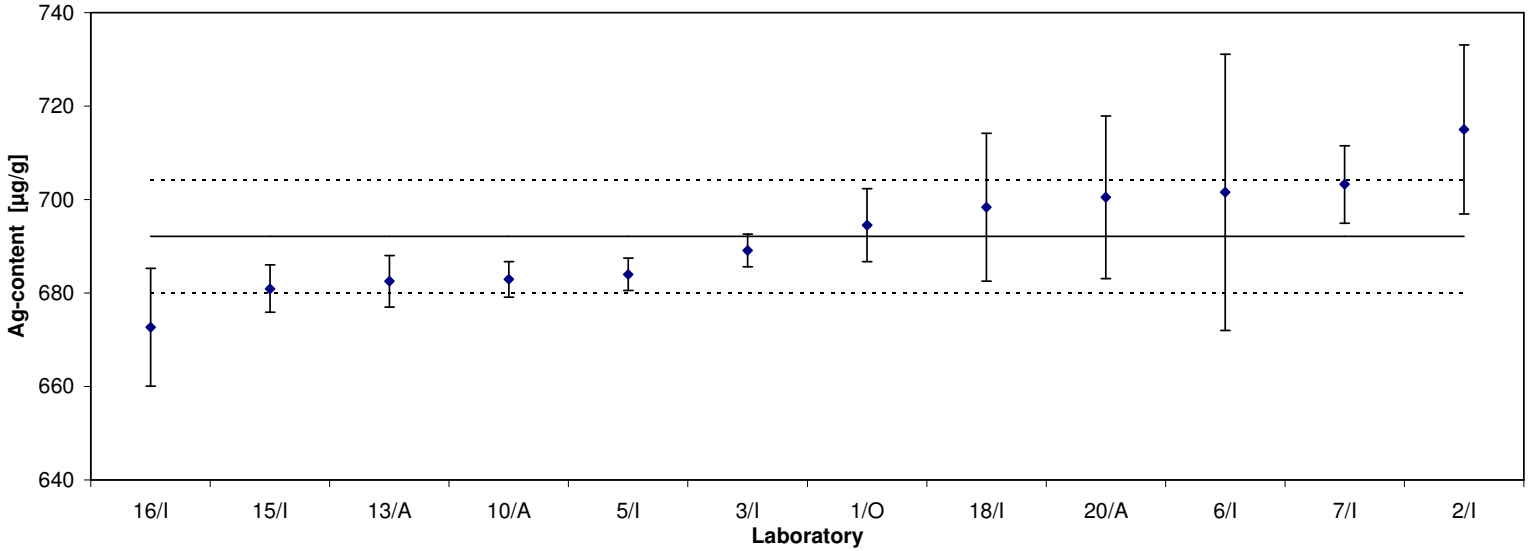

Table 18: Results for silver

Lab./Meth.	10/I	14/I	1/I	4/G	15/I	7/I	3/I	2/I	1/O	5/I	18/I	16/I	13/G	6/I		Ges.
EW [µg/g]	284.6	286	289	286.9	291.2	288.7	293.1	296	293	293.5	298	289.5	297.4	300.2		N
	286.1	286	284	290.6	290.6	292.3	289.6	291	288	293.8	293	295.6	299.3	300.2		14
	285.1	286	290	286.3	289.9	290.8	292.1	287	295	292.9	295	289.4	294.8	301.5		
	286.2	286	284	289.0	289.9	290.7	295.0	293	290	293.8	295	300.8	298.8	296.9		
	284.8	284	290	290.4	290.4		289.1	293	294	293.1	292	298.4	298.1	297.7		
	282.9	288	285	289.4	291.3		292.5	[314]	295	293.5	297	297.5	297.7	299.8		
MW [µg/g]	**285.0**	**286.0**	**287.0**	**288.8**	**290.5**	**290.6**	**291.9**	**292.0**	**292.5**	**293.4**	**295.0**	**295.2**	**297.7**	**299.4**		**291.8**
s[µg/g]	1.20	1.26	2.97	1.79	0.61	1.48	2.22	3.32	2.88	0.37	2.28	4.76	1.58	1.73		4.23
$\bar{s}$ [µg/g]																2.03
s_{rel}	0.00	0.00	0.01	0.01	0.00	0.01	0.01	0.01	0.01	0.00	0.01	0.02	0.01	0.01		0.01
	291.8	291.8	291.8	291.8	291.8	291.8	291.8	291.8	291.8	291.8	291.8	291.8	291.8	291.8		
	287.5	287.5	287.5	287.5	287.5	287.5	287.5	287.5	287.5	287.5	287.5	287.5	287.5	287.5		
	296.0	296.0	296.0	296.0	296.0	296.0	296.0	296.0	296.0	296.0	296.0	296.0	296.0	296.0		

Lead collection Cu2O-melt Silver collection HCl/Aqua Regia/Na2O2

Nitrate-melt

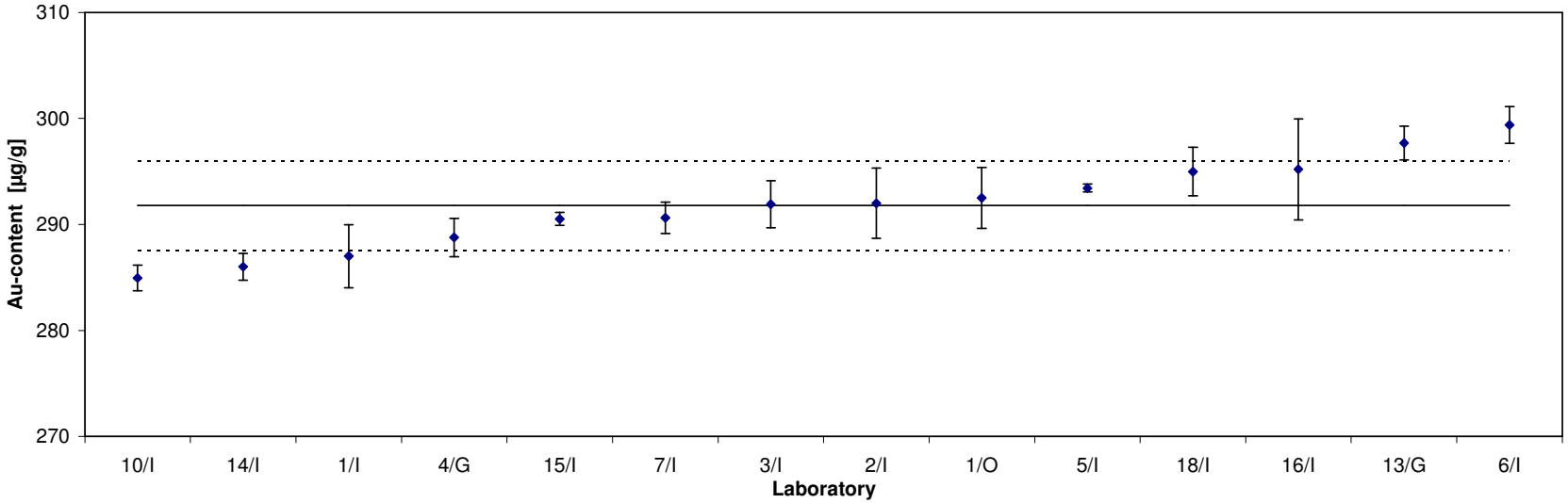

Table 19: Results for gold

Lab./Meth.	15/I	9/I	16/I	14/I	13/I	18/I	2/I	4/I	3/I	1/I	7/I	5/I	1/O	10/I	6/I	19/I		Ges.
EW [µg/g]	86	88.0	85.9	86	85.7	87	86.5	89.5	90.1	94.6	90.3	91.8	95.8	93.6	99.8	95.1		N
	86	88.5	91.7	89	89.5	91	91.0	89.9	92.4	92.7	92.1	92.5	92.5	93.1	95.5	96.8		16
	87	89.8	84.8	86	88.2	89	87.0	92.1	91.1	94.2	93.4	92.0	89.5	93.9	97.9	97.9		
	85	82.8	85.3	83	92.4	90	90.3	90.0	89.1	87.2	91.5	91.6	92.7	94.2	95.2	100.7		
	87	83.7	85.8	86	83.1	85	86.4	90.2	90.8	91.1	91.4	91.5	94.2	92.2	94.0	99.8		
	85	84.6	85.1	89	87.8	92	95.2	90.5	92.1	88.1	91.3	91.2	91.3	91.6	95.9	99.9		
MW [µg/g]	**86.0**	**86.2**	**86.4**	**86.5**	**87.8**	**89.0**	**89.4**	**90.4**	**90.9**	**91.3**	**91.7**	**91.8**	**92.7**	**93.1**	**96.4**	**98.4**		**90.5**
s[µg/g]	0.78	2.89	2.61	2.26	3.18	2.61	3.47	0.91	1.23	3.11	1.03	0.45	2.20	1.02	2.10	2.15		3.59
$\bar{s}$ [µg/g]																		2.00
s_{rel}	0.01	0.03	0.03	0.03	0.04	0.03	0.04	0.01	0.01	0.03	0.01	0.00	0.02	0.01	0.02	0.02		0.04
	90.5	90.5	90.5	90.5	90.5	90.5	90.5	90.5	90.5	90.5	90.5	90.5	90.5	90.5	90.5	90.5		
	86.9	86.9	86.9	86.9	86.9	86.9	86.9	86.9	86.9	86.9	86.9	86.9	86.9	86.9	86.9	86.9		
	94.1	94.1	94.1	94.1	94.1	94.1	94.1	94.1	94.1	94.1	94.1	94.1	94.1	94.1	94.1	94.1		

Lead collection | Cu2O-melt | Silver collection | HCl/Aqua Regia/Na2O2 | Nitrate-melt | copper collection

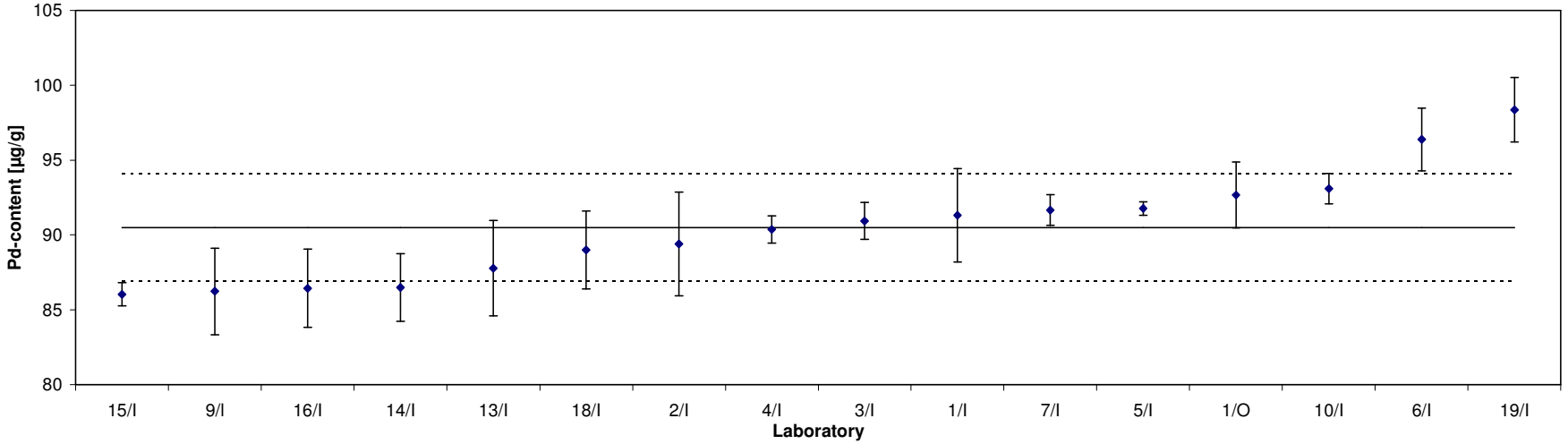

Table 20: Results for palladium

Lab./Meth.	19/I	9/I	16/I	2/I	15/I	1/I	1/O	5/I	4/I	6/I	3/I	7/I	13/I	10/I	18/I	14/I		Ges.
EW [µg/g]	7.49	7.45	6.9	9.2	8.5	8.7	10.4	8.30	8.7	8.2	8.1	9.2	8.6	9.49	10			N
	7.43	7.53	7.7	7.8	8.1	9.1	6.2	8.39	8.6	8.4	9.3	9.2	10.4	9.32	10			15
	8.03	7.92	7.4	9.0	8.3	8.4	10.4	8.51	8.7	9.1	8.2	9.4	8.4	9.34	10			
	7.06	7.30	8.1	6.0	8.2	7.8	6.7	8.48	8.4	9.0	8.9	9.5	11.3	9.40	9			
	7.21	7.60	8.3	6.7	8.0	8.0	9.9	8.50	8.5	8.4	9.6	8.8	8.0	9.28	9			
	7.67	7.84	8.0	9.2	8.0	7.5	7.0	8.47	8.6	8.9	8.6	9.3	9.3	9.30	10			
MW [µg/g]	**7.48**	**7.61**	**7.73**	**7.98**	**8.18**	**8.25**	**8.43**	**8.44**	**8.58**	**8.65**	**8.78**	**9.23**	**9.33**	**9.36**	**9.67**	**< 10**		**8.51**
s[µg/g]	0.344	0.235	0.516	1.386	0.194	0.596	1.997	0.081	0.117	0.368	0.598	0.242	1.280	0.078	0.516			0.670
$\bar{s}$ [µg/g]																		0.570
s_{rel}	0.046	0.031	0.067	0.174	0.024	0.072	0.237	0.010	0.014	0.043	0.068	0.026	0.137	0.008	0.053			0.079
	8.5	8.5	8.5	8.5	8.5	8.5	8.5	8.5	8.5	8.5	8.5	8.5	8.5	8.5	8.5	8.5		
	7.8	7.8	7.8	7.8	7.8	7.8	7.8	7.8	7.8	7.8	7.8	7.8	7.8	7.8	7.8	7.8		
	9.2	9.2	9.2	9.2	9.2	9.2	9.2	9.2	9.2	9.2	9.2	9.2	9.2	9.2	9.2	9.2		

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Lead collection Cu2O-melt Silver collection HCl/Aqua Regia/Na2O2 Nitrate-melt

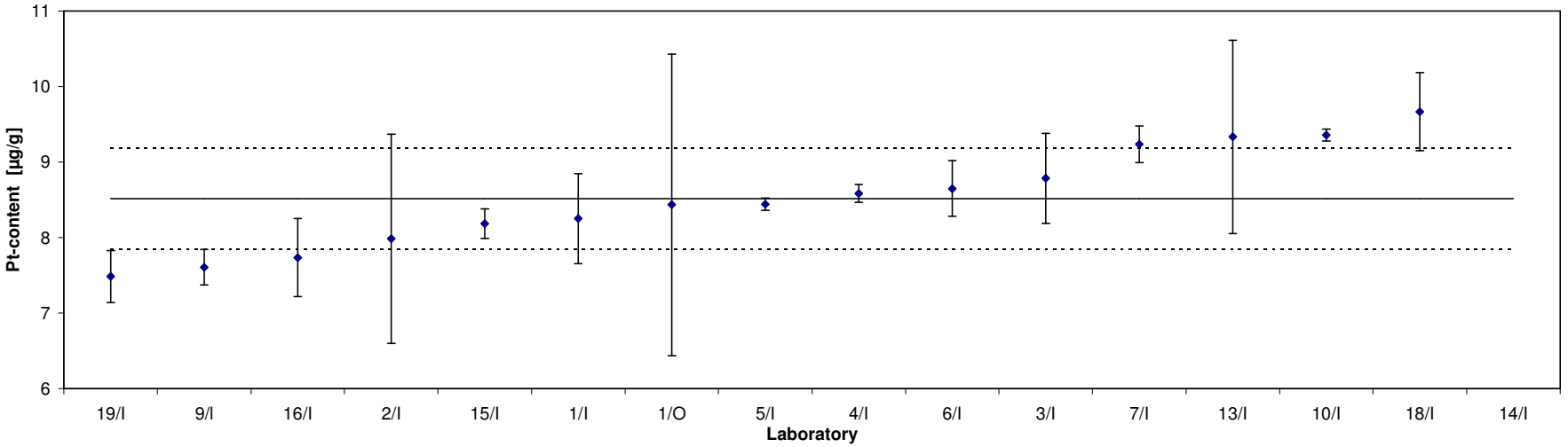

Table 21: Results for platinum

Lab./Meth.	19/I	15/EA	14/I	1/I	6/I	16/I	5/I	3/I	9/I	10/A		Ges.
EW [µg/g]	63.30	63.0	67.1	66	67.2	68.6	68.5	68.9	69.81	75.25		N
	64.30	68.2	66.8	66	67.7	69.1	69.5	71.2	70.32	74.03		10
	62.60	65.3	65.6	67	67.3	68.9	69.0	69.5	70.88	74.61		
	62.50	65.0	66.7	67	66.7	70.1	70.0	68.9	69.60	76.79		
	63.00	65.7	65.7	66	67.9	68.8	70.0	70.3	70.49	72.48		
	63.90	67.9	66.3	70	67.7	68.6	70.0	68.9	69.53	74.56		
MW [µg/g]	**63.27**	**65.85**	**66.37**	**67.00**	**67.41**	**69.02**	**69.50**	**69.62**	**70.11**	**74.62**		**68.83**
s[µg/g]	0.717	1.944	0.612	1.549	0.444	0.564	0.632	0.952	0.542	1.417		3.063
$\bar{s}$ [µg/g]												0.937
s_{rel}	0.011	0.030	0.009	0.023	0.007	0.008	0.009	0.014	0.008	0.019		0.045
	68.8	68.8	68.8	68.8	68.8	68.8	68.8	68.8	68.8	68.8		
	65.8	65.8	65.8	65.8	65.8	65.8	65.8	65.8	65.8	65.8		
	71.9	71.9	71.9	71.9	71.9	71.9	71.9	71.9	71.9	71.9		

Extraction with Aqua Regia
Microwave HNO3/HF
acid mixture/alkaline melt
Aqua Regia/Fuming with H2SO4/Na2O2-melt
HNO3
acid mixture

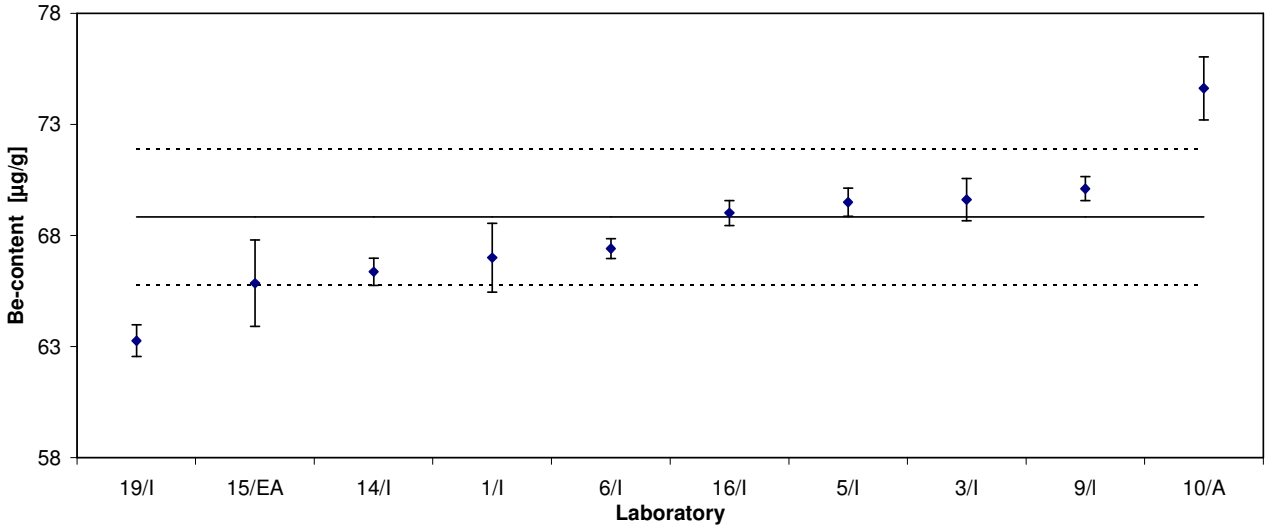

Table 22: Results for beryllium

7. Instructions for users

The certified reference material ERM®-EZ505 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.
Before analysis the material has to be dried at 105 °C for approx. 8 h. The minimum sample intake is 5 g.
The material is stable, it has to be stored in a dry and clean atmosphere.

8. Literature

[1] ISO Guide 31, Contents of certificates of reference materials, 1981

[2] ISO Guide 34, General requirements for the competence of reference material producers, 2000

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

[4] Guidelines for the production of BAM Reference Materials, 2006

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

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

9. Information on and purchase of the CRM

Information and purchase is done by

BAM Bundesanstalt für Materialforschung und -prüfung
Fachgruppe 1.1: Anorganisch-chemische Analytik, Referenzmaterialien
Richard-Willstätter-Straße 11, 12489 Berlin
Phone +49 (0)30 - 8104 2061 or 1119
Fax: +49 (0)30 - 8104 1117
E-Mail: sales.crm@bam.de

Each bottle of ERM®-EZ505 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, Tel. +49 30 8104 1111.