Bundesanstalt für Materialforschung und -prüfung (BAM)

in co-operation with the

Committee of Chemists of GDMB
Gesellschaft der Metallurgen und Bergleute e.V.

The Certification of Mass Fractions of Al, Ca, Fe, Mg, Si, Th, Ti, U, Hf, and Y (2015) and subsequent certification of Oxygen (2024) in Yttrium Stabilized Zirconium Oxide

ERM®-ED105

Certification report

Original certification: July 2015
Subsequent certification: January 2024

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Abstract

This report describes the preparation and certification of reference material ERM®-ED105, an yttrium stabilized zirconium oxide with certified impurity contents, carried out in co-operation with the Committee of Chemists of GDMB. The certified mass fractions and additional determined data are listed below (blue: determined in 2023)

Parameter	Mass fraction	
	Certified Value 1) in mg/kg	**Uncertainty 2) in mg/kg**
Aluminium	660	15
Calcium	242	9
Iron	95	9
Magnesium	12.9	1.7
Silicon	195	40
Thorium	112	17
Titanium	497	11
Uranium	292	19
	Certified Value 1) in %	**Uncertainty 2) in %**
Hafnium	1.535	0.024
Yttrium	6.11	0.09
Oxygen	25.2	0.4

1) The certified values are the means of 11-20 series of results (depending on the parameter) obtained by different laboratories. Up to 7 different analytical methods were used for the measurement of one parameter. The methods applied for determination of element mass fractions were calibrated using pure substances of definite stoichiometry or solutions prepared from them, thus achieving traceability to the International System of Units (SI).
2) The certified uncertainty is the expanded uncertainty estimated in accordance with the Guide to the Expression of Uncertainty in Measurement (GUM) with a coverage factor k = 2. It includes contributions from sample inhomogeneity and long term stability.

Additional material information

Parameter	Mass fraction 1) in mg/kg	Uncertainty 2) in mg/kg
Nitrogen	912	140
Phosphorus	< 75	

1) The value stated is based on 5 series of results obtained by different laboratories. Two different analytical methods were used for the measurement of the parameter.

Parameter		Mass fraction in %
Phase 1)	monoclinic	1.94
Particle size distribution 2)		**Particle size in µm**
	d_{10}	18.9
	d_{50}	33.3
	d_{90}	55.4

1) The measurements were carried out by X-ray powder diffraction using Rietveld method for evaluation.
2) The particle size distribution was determined by laser light diffraction method.

Contents

List of abbreviations
(if not explained elsewhere)

CGHE	Carrier gas hot extraction
DCArc-OES	Direct current arc optical emission spectrometry
ET AAS	Atomic absorption spectrometry with electrothermal atomization
ETV-ICP-OES	Inductively coupled plasma optical emission spectrometry with electrothermal vaporisation
FAAS	Flame atomic absorption spectrometry
ICP-OES	Inductively coupled plasma optical emission spectrometry
ICP-MS	Inductively coupled plasma mass spectrometry
INAA	Instrumental neutron activation analysis
SS ET AAS	Solid sampling electrothermal atomic absorption spectrometry
XRF	X-ray fluorescence spectrometry

M	mean of the laboratories' means
u_c	combined uncertainty of certified mass fraction
s_M	standard deviation of the accepted laboratory mean values of interlaboratory comparison for certification
n	number of accepted laboratory mean values of interlaboratory comparison for certification

1 Introduction

1.1 Scope

Zirconium oxide is a chemically inert material with a wide spread of technical applications. ZrO_2 adopts a monoclinic crystal structure at room temperature and transitions to tetragonal and cubic at higher temperatures. The volume expansion caused by the cubic to tetragonal to monoclinic transformation induces large stresses, and these stresses cause ZrO_2 to crack upon cooling from high temperatures. When the zirconia is blended with some other oxides, the tetragonal and/or cubic phases are stabilized. One of the effective dopants is yttrium oxide (Y_2O_3).
For technical applications zirconium oxide is often more useful in its 'stabilized' state. Upon heating, zirconia undergoes disruptive phase changes. In its stabilized state, phase changes are eliminated, and the resulting material has superior thermal, mechanical, and electrical properties.
The main use of zirconia is in the production of ceramics, with additional applications as a protective coating on particles of titanium dioxide pigments, as a refractory material, in insulation, abrasives and enamels. Stabilized zirconia is used in oxygen sensors and fuel cell membranes because it has the ability to allow oxygen ions to move freely through the crystal structure at high temperatures. This high ionic conductivity (combined with a low electronic conductivity) makes it one of the most useful electroceramics. Other applications are its use as tooth crowns, as a refractory e.g., in jet engines, as a thermal barrier coating in gas turbines and as a material for non-metallic knife blades.

1.2 Certification procedure

Certification of reference materials is carried out on the basis of the relevant ISO-Guides [1-3] and the „Guidelines for the production of BAM Reference Materials“ [4].
The yttrium stabilized zirconium oxide powder material was a commercial product (Amperit 827) taken from the customary production line of the producer H.C. Starck, Laufenburg (Germany) and was bottled into 400 bottles each containing ca. 47 g of the material. After homogeneity testing one bottle was distributed to each of the participants of the certification interlaboratory comparison. All participating laboratories were asked to carry out six independent determinations using an analytical method of their own choice. A technical discussion on analytical methods and on the results of the certification interlaboratory comparison took place during the biannual sessions of the working group "Special Materials" of GDMB.
The statistical evaluation of all analytical results (except O and N) was performed using the software program SoftCRM 1.2.2. [5]. After removal of technical and statistical outliers the certified values were calculated as means of the laboratory means reported from the participating laboratories of the interlaboratory comparison. The certified uncertainties were calculated taking into account the contributions from interlaboratory comparison and from inhomogeneity of the material.

2 Participating laboratories

2.1 Allocation and preparation of the material

- The material was produced by H.C. Starck, Laufenburg, Germany
- The material was bottled by Bundesanstalt für Materialforschung und -prüfung (BAM)

2.2 Homogeneity testing

The analytical investigations and all statistical evaluations for the homogeneity testing were carried out by BAM

2.3 Certification analysis

19 laboratories from four different countries participated in the original interlaboratory comparison for certification. In the subsequent interlaboratory comparison for O and N 9 laboratories took part. These laboratories were either involved in daily analysis of ceramics or had well known ability to analyse difficult materials by adequate analytical methods. Most of them already participated successfully in certification interlaboratory comparisons of other special materials CRMs. Therefore no preceding qualification round robin was carried out.

Bundesanstalt für Materialforschung und -prüfung (BAM), Berlin, Germany
Bruker AXS GmbH, Germany
BCRC, Mons, Belgium
ESK Ceramics GmbH & Co. KG, Kempten, Germany
Forschungsinstitut für anorganische Werkstoffe - Glas/Keramik GmbH, Höhr-Grenzhausen, Germany
Forschungszentrum Jülich, Jülich, Germany
GfE Fremat GmbH, Freiberg, Germany
H. C. Starck GmbH & Co. KG, Werk Goslar, Goslar, Germany
H. C. Starck GmbH & Co. KG, Werk Laufenburg, Laufenburg, Germany
Institute Jozef Stefan, Ljubljana, Slovenia
Karlsruher Institut für Technologie, Karlsruhe, Germany
Leibniz Institut für Kristallzüchtung, Berlin, Germany
Max-Planck-Institut für intelligente Systeme, Stuttgart, Germany
Osram AG, Augsburg, Germany
Plansee SE, Reutte, Austria
Revierlabor GmbH, Essen, Germany
Treibacher Industrie AG, Treibach-Althofen, Austria
Wolfram Bergbau und Hütten AG, St. Martin i.S., Austria
Umicore AG & Co. KG, Hanau, Germany

Determination of O and N

Bundesanstalt für Materialforschung und -prüfung (BAM), Berlin, Germany
Bruker AXS GmbH, Germany
Eltra GmbH, Haan, Germany
Horn & Co Analytics, Wenden-Hünsborn, Germany
IFW Dresden e.V., Dresden (Germany)
Karlsruher Institut für Technologie (KIT), Karlsruhe, Germany
LECO Instrumente GmbH, Berlin, Germany
Plansee SE, Reutte, Austria
revierlabor, Essen, Germany

2.4 Determination of additional material data

The determination of phases was performed by three of the participating laboratories using X-ray structure and phase analysis.
The particle size distribution was determined by BAM, Division 5.5 "Advanced Technical Ceramics".

3 Homogeneity investigation of the material

For homogeneity testing 15 bottles were representatively taken from a total of 400 bottles by a combination of random access and systematic selection (see Table 1). From each of the N = 15 bottles four subsamples were taken for analysis.

Tab. 1a: Selected bottles analysed for homogeneity of ERM®-ED105

10	32	65	95	122	156	169	215
255	276	282	309	326	370	394	

Tab. 1b: Selected bottles analysed for homogeneity of ERM®-ED105 (Si after fusion)

32	65	113	156	181	233	276	309	348	394

Tab. 1c: Selected bottles analysed for homogeneity of ERM®-ED105 (O, N)

10	56	62	112	169	181	255	276	326	394

The homogeneity investigations were performed after acid digestion of 200 mg of the material for 13 h in a high pressure asher DAB-III (Berghof GmbH, Eningen, Germany) with 5 ml of HNO_3 and 5 ml of H_2SO_4. Si was analysed after decomposition of 100 mg of the sample material by fusion. O (sample intake: 1-2 mg) and N (sample intake: 0.2 g) were determined using CGHE.
The measurements were carried out using ICP-OES (Al, Ca, Fe, Mg, Si, Ti, Hf and Y) or ICP-MS (U, Th). Aqueous calibration solutions were used for calibration. Resulting lack of trueness of results or of metrological traceability is not relevant, because a high precision is the only necessity in case of homogeneity investigation.
The estimates of analyte-specific inhomogeneity contributions u_{bb} to be included into the total uncertainty budget were calculated according to ISO Guide 35 [3] using Eq. (1) and Eq. (2):

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

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

with:

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

s_{bb} signifies the between-bottle standard deviation, whereas u^*_{bb} denotes the maximum heterogeneity that can potentially be hidden by an insufficient repeatability of the applied measurement method (which has to be considered as the minimum uncertainty contribution). In any case the larger of the two values was used as u_{bb}. Eq. (1) does not apply if MS_{within} is larger than MS_{among}.
The results of the homogeneity testing are listed in Appendix 1.

4 Stability of the material

Due to its chemical composition zirconium oxide is a very stable material not only at room temperature but also at higher temperatures. Therefore it is used for dental applications as well as for high temperature applications. Because of its chemical inertness and its stability at high temperatures no specific stability test was performed for ERM®-ED105.

5 Characterisation study

5.1 Analytical methods used for certification

19 laboratories (9 laboratories for O, N) participated in the certification interlaboratory comparison. For some elements part of the laboratories used more than one analytical method and therefore reported more than one dataset. The laboratories were asked to analyse six subsamples. They were free to choose any suitable method for analysis. Table 2 shows the analytical methods used by the participating laboratories.
All participating laboratories were instructed to use only calibrants prepared from pure metals or stoichiometric compounds or well checked commercial calibration solutions.

Table 2: Analytical procedures used by the participating laboratories

Lab-No.	Element.	Sample mass	Sample pretreatment/Calibration	Analytical method
1	Fe, Mg, Si, Ti, Th, U, P	0.3 g	Solid sampling technique, Mixing of 0.3 g of sample with 2.1 g of pure graphite. Calibration with pure metal solutions (Fe, Mg, Ti) or commercial solutions matrix matched with Zr, Hf, Y	ETV-ICP-OES
1	Al, Fe, Mg, Si	0.3 g	Solid sampling technique, Mixing of 0.3 g of sample with 2.1 g of pure graphite. Calibration with commercial multioxide standard in graphite	DCArc-OES
1	U, Th	0.2 g	Dissolution with HNO_3/H_2SO_4 in a DAB-III high pressure asher for 13 h Calibration with commercial solutions	ICP-MS
1	Al, Ca, Fe, Mg, Ti, Y	0.2 g	Dissolution with HNO_3/H_2SO_4 in a DAB-III high pressure asher for 13 h Calibration with solutions made from pure metals or oxides (Al, Ca (from $CaCO_3$) Fe, Mg, Ti, Y (from Y_2O_3)) or commercial solutions matrix matched with Zr, Y (not for determination of Y)	ICP-OES
1	Si, Hf, Y	0.1 g	Decomposition with 1 g of $K_2S_2O_7$ in a platinum crucible, dissolution of the melt with HCl Calibration with solutions made from pure oxide (Y_2O_3) or commercial solutions matrix matched with Zr and $K_2S_2O_7$	ICP-OES
1	O	1.5 mg	Sn-capsules, Ni-flux, Calibration with Fe_2O_3,	CGHE
1	N	0.2 g	Sn-capsules Calibration with N_2 (gas calibration),	CGHE
2	Al, Ca, Fe, Mg, Ti	0.1 g	Microwave assisted dissolution with $HNO_3/H_2O_2/HF$ Calibration with commercial solutions	ICP-OES
2	Hf, Y	0.3 g	Melted with 9.65 g of Li-tetraborate and 0.05 g Li-bromide Calibration with oxides, matrix matched	XRF
2	Th, U	0.1 g	Microwave assisted dissolution with $HNO_3/H_2O_2/HF$ Calibration with commercial solutions	ICP-MS
3	Al, Ca, Fe, Mg, Si, Ti, Hf, Y	0.05 g	Microwave assisted dissolution with $H_3BO_3/HNO_3/HF$ Calibration with commercial solutions	ICP-OES
3	Th, U	0.05 g	Microwave assisted dissolution with $H_3BO_3/HNO_3/HF$ Calibration with commercial solutions	ICP-MS

Lab-No.	Element.	Sample mass	Sample pretreatment/Calibration	Analytical method
4	Al, Ca, Fe, Mg, Si, Ti, Hf, Y	0.2 g	Dissolution with HNO_3/H_2SO_4 in a DAB-II high pressure asher for 24 h Calibration with commercial solutions	ICP-OES
4	Th, U	0.2 g	Dissolution with HNO_3/H_2SO_4 in a DAB-II high pressure asher for 24 h Calibration with commercial solutions	ICP-MS
5	Si	43 - 70 mg	Solid sampling technique Calibration with commercial solution	SS ET AAS
6	Al, Ca, Fe, Mg, Si, Ti, P, Hf, Y	0.1 g	Decomposition with 0.8 g of $Na_2B_4O_7$, dissolution of the melt with HNO_3 Calibration with commercial solutions, matrix matched with $Na_2B_4O_7$	ICP-OES
6	Si	0.1 g	Decomposition with 2 g of KOH, dissolution of the melt with H_2O Calibration with commercial solution	ET AAS
7	Al, Ca, Fe, Mg, Si, Ti, Hf	0.05 g	Microwave assisted dissolution with $H_3BO_3/HNO_3/HF$ Calibration with pure metal solutions oxides (Al, Ca (from $CaCO_3$) Fe, Hf, Mg, Si, Ti, Y) matrix matched with Zr, Y	ICP-OES
7	Y	0.05 g	Microwave assisted dissolution with $H_3BO_3/HNO_3/HF$ Calibration with pure metal solutions oxides (Al, Ca (from $CaCO_3$) Fe, Hf, Mg, Si, Ti, Y) matrix matched with Zr	ICP-OES
8	Al, Fe, Mg	0.5 g	Dissolution with HF/H_2SO_4 Calibration with commercial solutions matrix matched with Zr	FAAS
8	Ca	0.5 g	Dissolution with HF/H_2SO_4 Calibration with commercial solutions matrix matched with Zr	ICP-OES
8	Hf, Ti, Y	0.5 g	Borat-flux with Na-tetraborate Calibration with 84 matrix matched borate fluxes made of pure oxides	XRF
8	O	10 - 20 mg	Ni-capsules Calibration with ZrO_2 (25.97% O)	CGHE
8	N	10 - 20 mg	Ni-capsules Calibration with steel.RM (0.0104 % N)	CGHE
9	Al, Ca, Fe, Hf, Th, Mg, Ti, Y	0.1 g	Microwave assisted dissolution with $HNO_3/H_2O_2/HF$ Calibration with commercial solutions matrix matched with Zr	ICP-OES
10	Al, Ca, Fe, Mg, Ti, Hf, Y, P	0.5 g	Dissolution with HNO_3/H_2SO_4 in a DAB-III high pressure asher for 24 h Calibration with solutions made from pure salts	ICP-OES
10	Si, Hf, Y	0.05 g	Decomposition with 5 g of $NaKCO_3$ in a platinum crucible, dissolution of the melt with HCl Calibration with solutions made from pure salts	ICP-OES
10	Si	1 g	Decomposition with 2 ml Hf, distillation as H_2SiF_6 Calibration with solution made from pure salt	ICP-OES

Lab-No.	Element.	Sample mass	Sample pretreatment/Calibration	Analytical method
11	Fe, Mg	0.2 g	Microwave assisted dissolution with $HNO_3/H_2SO_4/HCl$ Calibration with commercial solutions	ICP-MS
11	Ti, Hf, Y	0.2 g	Microwave assisted dissolution with $HNO_3/H_2SO_4/HCl$ Calibration with commercial solutions	ICP-OES
12	Al, Ca	0.5 g	Decomposition with 4 g of $Na_2B_4O_7$ and 0.5 g NaI, dissolution of the melt with HNO_3 Calibration with commercial solutions (standard addition)	FAAS
12	Al, Ca, Fe, Hf, Mg, Si, Th, Ti, U, Y, P	0.5 g	Decomposition with 4 g of $Na_2B_4O_7$ and 0.5 g NaI, dissolution of the melt with HNO_3 Calibration with commercial solutions (standard addition)	ICP-OES
14	Al, Ca, Fe, Hf, Mg, Th, Ti, U, Y, P	0.5 g	Dissolution with HF/HNO_3 Calibration with commercial solutions	ICP-OES
14	Si	0.2 g	Fusion with 2 g Na_2CO_3 and 1 g H_3BO_3, dissolution of the melt with HCl	ICP-OES
15	Al, Ca, Fe, Hf, Mg, Ti, Y	0.1 g	Microwave assisted dissolution with HNO_3/H_2SO_4 Calibration with commercial solutions matrix matched with Zr	ICP-OES
15	Th, U	0.1 g	Microwave assisted dissolution with HNO_3/H_2SO_4 Calibration with commercial solutions matrix matched with Zr	ICP-MS
15	Al, Ca, Fe, Hf, Si, Ti, Y			XRF
	O	20 - 60 mg	Sn-capsules, Sn-flux Calibration with TiO_2/ZrO_2	CGHE
	N	50 - 60 mg	Sn-capsules, Sn-flux Calibration with ELTRA 91100-1002-415F (steel)	CGHE
16	Al, Ca, Fe, Mg, Th, Ti, U, P	0.5 g	Decomposition with 1 g of $K_2S_2O_7$ in a platinum crucible, dissolution of the melt with HCl and tartaric acid Calibration with solutions made from pure oxide (Y_2O_3) or commercial solutions matrix matched with Zr, Y and $K_2S_2O_7$	ICP-OES
16	Hf, Ti, Y, Th. U	0.5 g	Borat-flux with 5 g Li-tetraborate Calibration with matched borate fluxes made of pure oxides	XRF
16	Si	1.4 g	Dissolution in HF (40%) Calibration with commercial solutions matrix matched with Zr	ICP-OES
16	O	30 - 50 mg	Ni-capsules, Sn/Ni-flux Calibration with ZrO_2 (LECO 502-140)	CGHE
16	N	0.1 g	Ni-capsules, Sn/Ni-flux Calibration with LECO steel samples	CGHE

Lab-No.	Element.	Sample mass	Sample pretreatment/Calibration	Analytical method
18	Al, Ca, Fe, Hf, Mg, Ti, Y	0.1 g	Microwave assisted dissolution with HCl/H_2SO_4 Calibration with commercial solutions (standard addition)	ICP-OES
18	Si	0.5 g	Borat-flux with 5 g Li-tetraborate	XRF
19	Hf	0.17 g		k_0-INAA
20	Si	0.15 g	Dissolution with HF/H_2SO_4 (4 subsamples) or pyrosulfate/ H_2SO_4 (2 subsamples) with subsequent distillation Calibration with commercial solutions	Spectrophotometry
21	O	0.01 g	Sn-capsules, Ni-flux Calibration with CRM JK47A	CGHE
21	N	0.01 g	Sn-capsules, Ni-flux Calibration with N_2 (gas calibration)	CGHE
22	O	20 mg	Calibration with ZrO_2	CGHE (ASTM E1019)
22	N	0.2 g	Calibration with KNO_3	CGHE (ASTM E1019)
23	O	0.015 g	Calibration with ZrO_2	CGHE (DIN EN 10276-2)
23	N	0.015 g	Calibration with ZrO_2	CGHE (DIN EN ISO 15351)
24	O	0.01 g	Ni/Sn-capsules Calibration with ZrO_2 (Alfa Aesar 99.975 %)	CGHE
24	N	0.01 g	Ni/Sn-capsules Calibration with N_2 (gas calibration)	CGHE
25	O	0.06 g	Calibration with ZrO_2 (LECO)	CGHE

The analytical results of the certification interlaboratory comparison are listed in Tables A2.1.1 to A2.11.1 in Appendix 2. These tables show the single results of each laboratory, the resp. laboratories' mean values together with the intralaboratory standard deviation (s_i).

5.2 Methods used for the determination of additional material data

Three laboratories carried out the quantitative phase analysis using high resolution X-ray powder diffraction. The evaluation of the diffraction data and the quantification was carried out using the Rietveld method. The tetragonal and cubic phases could not be determined due to signal overlapping. Table 3 shows the single results.

Table 3: Determination of the monoclinic phase of YSZO

	Mass fraction in %
Line no.	monoclinic
1	*1.8*
2	*2.0*
3	*2.01*
M:	*1.94*

The particle size distribution was determined by laser light diffraction method by one single laboratory.

6 Results and discussion

The statistical evaluation of the data was performed using the software program SoftCRM 1.2.2. [5]. The results of the statistical calculations are given in Appendix 2.

The analytical results of the certification interlaboratory comparison are listed in Table 4. The table shows the laboratories' means, the mean of the laboratories' means (*M*) together with the standard deviation of the laboratories' means (s_M), u_{bb} and the combined uncertainty u_c, calculated from the spread resulting from the certification interlaboratory comparison (u_{ilc}) and the uncertainty contributions from possible inhomogeneity of the material using Equation 3, as well as the expanded uncertainty *U* which is calculated from u_c by multiplication with the coverage factor $k = 2$. The mean of the laboratories' means is taken as certified value.

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

with

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

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

7 Instructions for use

7.1 Area of application

The main area of application is to check the trueness of results when one or more of the certified parameters in zirconium dioxide material are determined by a laboratory. Based on own results and on certified values the uncertainty of own measurements can be calculated.

7.2 Recommendations for correct sampling and sample preparation

To ensure a representative sub-sampling for the analysis the bottle containing the CRM should be shaken in different directions for about two minutes before taking the sub-sample. Each sub-sample has to be taken separately. According to the sub-sample masses taken for the homogeneity testing at least 200 mg of powder has to be weighed. The opening duration of the bottle should be as short as possible. The lid of the bottle containing a special sealing gasket should be locked tightly immediately after usage.

Table 4: Calculation of the certified values and their combined and expanded uncertainties (Mass fraction in mg/kg respectively % (Hf, Y, O, N))

No.	**Al**	**Ca**	Fe	Mg	Si	Th	Ti	U	Hf	Y	O	N
1	-	-	71.83	9.45	80.83	60.30	454.00	235.67	1.469	5.780		0.0667
2	614.33	219.33	73.30	9.50	97.83	90.50	463.00	251.83	1.474	5.984	24.89	0.0728
3	627.50	223.83	79.50	9.67	132.33	91.33	473.67	280.50	1.481	5.992	25.01	0.0814
4	635.00	230.33	80.90	10.02	150.33	95.33	474.00	283.55	1.482	5.998	25.05	0.0838
5	638.33	231.67	82.35	10.17	151.70	112.57	485.00	295.33	1.485	6.020	25.23	0.0920
6	641.67	235.00	82.67	10.92	152.20	114.70	494.67	297.17	1.497	6.024	25.24	0.0955
7	648.33	236.67	83.33	11.30	186.83	117.83	497.86	298.17	1.518	6.092	25.35	0.1160
8	649.00	238.33	83.33	11.33	192.00	121.50	498.33	300.80	1.537	6.093	25.44	0.1215
9	653.50	239.83	83.60	11.34	202.80	130.00	499.83	301.67	1.539	6.098	25.50	
10	655.50	240.67	85.83	12.00	204.20	131.00	500.17	333.33	1.540	6.118		
11	656.67	241.17	89.35	12.50	206.20	166.67	501.67	335.83	1.543	6.125		
12	660.23	243.67	89.68	12.67	206.33		506.67		1.554	6.127		
13	665.00	244.67	95.50	13.17	254.30		508.33		1.558	6.148		
14	671.17	246.67	103.33	13.33	272.50		516.00		1.560	6.155		
15	676.55	271.57	107.33	15.99	298.97		525.00		1.564	6.207		
16	695.00	280.00	111.67	18.33	326.68		526.67		1.567	6.237		
17	704.33	-	117.27	20.00	-		530.00		1.625	6.360		
18	726.30		119.83	20.17					1.630	6.368		
19	-		131.67									
20			137.50									
M:	659.91	241.56	95.49	12.88	194.75	111.98	497.34	292.17	1.535	6.107	25.21	0.0912
s_M:	28.41	15.88	19.28	3.47	68.44	27.65	21.80	29.78	0.048	0.138	0.21	0.0194

evaluation of uncertainty

	Al	Ca	Fe	Mg	Si	Th	Ti	U	Hf	Y	O	N
s_M	28.409	15.883	19.281	3.469	68.435	27.650	21.801	29.777	0.048	0.138	0.215	0.019
n	17	15	20	18	16	11	17	11	18	18	8	8
u_{bb}(rel.)	0.351	0.755	0.258	0.973	5.122	0.456	0.256	0.462	0.294	0.472	0.549	0.8113
u_{bb}	2.319	1.825	0.246	0.125	9.975	0.511	1.273	1.349	0.005	0.029	0.138	0.0007
u_c	7.270	4.489	4.318	0.827	19.804	8.352	5.438	9.079	0.012	0.044	0.158	0.0069
$U=2*u_c$	**14.540**	**8.977**	**8.637**	**1.655**	**39.609**	**16.705**	**10.877**	**18.158**	**0.024**	**0.087**	**0.316**	**0.014**

7.3 Recommendations for correct storage

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

7.4 Safety guidelines

Yttrium stabilized zirconium oxide is not known to be toxic. No hazardous effect is to be expected if the material is used under conditions usually adopted in analytical laboratories when handling finely dispersed powder materials.

8 References

[1] ISO Guide 31, Contents of certificates of reference materials, 2000

[2] ISO Guide 34, General requirements for the competence of reference material producers, 2009

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

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

9 Informative references

- Ma, X., Li, Y.; "Determination of trace impurities in high-purity zirconium dioxide by inductively coupled plasma atomic emission spectrometry using microwave-assisted digestion and wavelet transform-based correction procedure" in: Analytica Chimica Acta 579 (2006) 47-52
- Chen, S., Lu, D., Hu, Z., Wu, B.; "In-situ vaporization and matrix removal for the determination of rare earth impurities in zirconium dioxide by electrothermal vaporization inductively coupled plasma atomic emission spectrometry" in: Spectrochimica Acta, Part B, 60 (2005) 537-541

10 Information on and purchase of the CRM

Information and purchase is done by

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Each unit of ERM®-ED105 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.

11 Appendices

Appendix 1: Homogeneity testing

Appendix 2: Statistical evaluation of all results of interlaboratory comparison

Appendix 1: Homogeneity testing

Aluminium

Summary						
Bottle	*Number*	*Sum*	*Mean*	*Variance*		
10	4	2427.766853	606.9417133	1.79508487		
32	4	2435.712161	608.9280402	10.2308638		
65	4	2446.488453	611.6221134	95.5621425		
95	4	2385.105927	596.2764818	445.698721		
122	4	2420.304349	605.0760872	469.999243		
156	4	2430.065877	607.5164694	22.8762128		
169	4	2409.567912	602.3919779	27.8525216		
215	4	2448.819139	612.2047848	24.9410131		
255	4	2413.162364	603.2905909	50.7579085		
276	4	2430.359372	607.5898429	43.7743613		
282	4	2430.537812	607.634453	6.41964442		
309	4	2426.216686	606.5541714	33.0987366		
326	4	2413.065728	603.2664321	27.6484601		
370	4	2421.160109	605.2900272	1.21695701		
394	4	2430.42668	607.6066701	29.7470489		
			606.1459904			

ANOVA						
Source of variation	*sums of squares (SS)*	*degrees of freedom (df)*	*Mean squares (MS)*	*F-value*	*P-value*	*critical F-value*
Between groups	853.4834688	14	60.96310492	0.70798481	0.7547488	1.91824856
Within groups	3874.856758	45	86.10792796			
Total	4728.340227	59				

within-sd	9.27943576
effective n	4.00
s_bb	0
s_bb_min	2.13032539
u_bb	2.13032539
u_bb(rel.)	0.35145418

Calcium

Summary				
Bottle	*Number*	*Sum*	*Mean*	*Variance*
10	4	904.1448187	226.0362047	5.64744567
32	4	904.7089672	226.1772418	2.22010948
65	4	897.135489	224.2838722	4.09500272
95	4	893.7887609	223.4471902	131.149674
122	4	896.6092527	224.1523132	119.142968
156	4	897.9367706	224.4841926	1.39432442
169	4	849.9345118	212.483628	176.822298
215	4	904.3773505	226.0943376	4.16148768
255	4	892.2974038	223.074351	0.65681074
276	4	898.3555462	224.5888865	2.07265148
282	4	891.8073712	222.9518428	0.18392806
309	4	895.5387301	223.8846825	3.02297358
326	4	891.9090506	222.9772626	2.27488526
370	4	892.6381729	223.1595432	0.89475045
394	4	890.0899631	222.5224908	3.5064309
			223.354536	

ANOVA						
Source of variation	*sums of squares (SS)*	*degrees of freedom (df)*	*Mean squares (MS)*	*F-value*	*P-value*	*critical F-value*
Between groups	586.1799656	14	41.86999754	1.37355017	0.20556606	1.91824856
Within groups	1371.73722	45	30.48304933			
Total	1957.917186	59				

within-sd	5.5211457
effective	4.00
s_bb	1.6872276
s_bb_min	1.2675164
u_bb	1.6872276
u_bb(rel.)	0.7554033

Iron

Summary						
Bottle	*Number*	*Sum*	*Mean*	*Variance*		
10	4	316.1570747	79.03926868	0.53507274		
32	4	316.3966245	79.09915614	0.29509528		
65	4	316.9948213	79.24870533	1.22147574		
95	4	311.9577562	77.98943904	0.47054145		
122	4	312.0797208	78.01993019	0.34805973		
156	4	311.5935858	77.89839644	0.80483009		
169	4	314.8131902	78.70329755	0.70187564		
215	4	316.7391167	79.18477919	1.26180466		
255	4	315.5998243	78.89995608	1.31343389		
276	4	315.115857	78.77896424	0.65628157		
282	4	315.7609242	78.94023106	0.50416293		
309	4	311.3629205	77.84073012	0.63852086		
326	4	315.2729725	78.81824313	0.10220751		
370	4	314.5314608	78.6328652	1.40265385		
394	4	314.9828642	78.74571605	1.42645867		
			78.65597856			
ANOVA						
Source of variation	*sums of squares (SS)*	*degrees of freedom (df)*	*Mean squares (MS)*	*F-value*	*P-value*	*critical F-value*
Between groups	13.01708744	14	0.92979196	1.19382921	0.31286269	1.91824856
Within groups	35.04742388	45	0.778831642			
Total	48.06451132	59				
within-sd	0.882514386					
effective r	4.00					
s_bb	0.194268061					
s_bb_min	0.202603138					
u_bb	0.202603138					
u_bb(rel.)	0.257581359					

Magnesium

Summary						
Bottle	*Number*	*Sum*	*Mean*	*Variance*		
10	4	46.345529	11.58638225	0.00458526		
32	4	45.85360575	11.46340144	0.00988073		
65	4	45.48659835	11.37164959	0.01918291		
95	4	44.65978183	11.16494546	0.08391177		
122	4	44.43690084	11.10922521	0.00929126		
156	4	44.69213807	11.17303452	0.00230184		
169	4	44.87481835	11.21870459	0.0050749		
215	4	45.64058267	11.41014567	0.00324107		
255	4	45.24988169	11.31247042	0.01492865		
276	4	45.06035514	11.26508878	0.00326546		
282	4	45.30505269	11.32626317	0.00046896		
309	4	45.27324418	11.31831104	0.00804268		
326	4	44.97038921	11.2425973	0.00336792		
370	4	45.14592428	11.28648107	0.0061484		
394	4	45.44083737	11.36020934	0.00856498		
			11.30726066			
ANOVA						
Source of variation	*sums of squares (SS)*	*degrees of freedom (df)*	*Mean squares (MS)*	*F-value*	*P-value*	*critical F-value*
Between groups	0.848224295	14	0.06058745	4.98643547	1.7917E-05	1.91824856
Within groups	0.546770383	45	0.012150453			
Total	1.394994678	59				
within-sd	0.110229093					
effective n	4.00					
s_bb	0.110042034					
s_bb_min	0.025305831					
u_bb	0.110042034					
u_bb(rel.)	0.973197992					

Silicon

Summary						
Bottle	*Number*	*Sum*	*Mean*	*Variance*		
Zeile 1	4	432.770919	108.1927297	38.3983733		
Zeile 2	4	397.361695	99.34042374	6.80275048		
Zeile 3	4	405.9489821	101.4872455	19.7477115		
Zeile 4	3	303.5454822	101.1818274	101.764528		
Zeile 5	4	457.3247545	114.3311886	74.2802997		
Zeile 6	4	398.3117494	99.57793734	18.8980472		
Zeile 7	4	450.1180738	112.5295184	53.107936		
Zeile 8	4	433.7268425	108.4317106	80.6894165		
Zeile 9	4	458.4040157	114.6010039	44.5916592		
Zeile 10	4	458.6215678	114.6553919	15.7030531		
			107.4328977			
ANOVA						
Source of variation	*sums of squares (SS)*	*degrees of freedom (df)*	*Mean squares (MS)*	*F-value*	*P-value*	*critical F-value*
Between groups	1481.115373	9	164.5683747	3.78712337	0.00296425	2.22287383
Within groups	1260.186798	29	43.45471716			
Total	2741.30217	38				
within-sd	6.5920192					
effective n	4.00					
s_bb	5.50258252					
s_bb_min	1.68906541					
u_bb	5.50258252					
u_bb(rel.)	5.12187853					

Titanium

Summary						
Bottle	*Number*	*Sum*	*Mean*	*Variance*		
10	4	1937.652972	484.413243	2.76572898		
32	4	1947.412198	486.8530494	1.84536127		
65	4	1945.676994	486.4192484	18.5615845		
95	4	1930.209825	482.5524563	6.64534881		
122	4	1935.854481	483.9636204	15.7574291		
156	4	1938.03526	484.508815	9.38286714		
169	4	1930.685832	482.671458	0.82292874		
215	4	1953.480458	488.3701145	3.44919866		
255	4	1930.312601	482.5781502	6.8150008		
276	4	1942.650252	485.662563	15.9438937		
282	4	1941.16453	485.2911326	2.1222783		
309	4	1938.616248	484.6540621	11.4329093		
326	4	1926.753659	481.6884148	8.26435626		
370	4	1931.372119	482.8430296	6.18205436		
394	4	1938.116392	484.529098	3.33258147		
			484.4665637			
ANOVA						
Source of variation	*sums of squares (SS)*	*degrees of freedom (df)*	*Mean squares (MS)*	*F-value*	*P-value*	*critical F-value*
Between groups	191.8370657	14	13.70264755	1.81374273	0.06623956	1.91824856
Within groups	339.970564	45	7.554901422			
Total	531.8076297	59				
within-sd	2.74861809					
effective n	4.00					
s_bb	1.23973244					
s_bb_min	0.63101368					
u_bb	1.23973244					
u_bb(rel.)	0.25589639					

Hafnium

Summary						
Bottle	*Number*	*Sum*	*Mean*	*Variance*		
10	4	5.628783636	1.407195909	3.1644E-05		
32	4	5.64065594	1.410163985	5.8513E-05		
65	4	5.670330528	1.417582632	4.9954E-05		
95	4	5.647721503	1.411930376	5.6785E-05		
122	4	5.642392959	1.41059824	5.5114E-06		
156	4	5.644067725	1.411016931	2.3797E-05		
169	4	5.619226547	1.404806637	4.9207E-05		
215	4	5.679367662	1.419841915	4.1099E-05		
255	4	5.672766624	1.418191656	9.2271E-05		
276	4	5.630005074	1.407501269	0.00016774		
282	4	5.658160383	1.414540096	8.9056E-06		
309	4	5.709635879	1.42740897	0.00010049		
326	4	5.64634641	1.411586603	3.1019E-05		
370	4	5.65531521	1.413828803	0.00013681		
394	4	5.657269777	1.414317444	6.9789E-05		
			1.413367431			

ANOVA						
Source of variation	*sums of squares (SS)*	*degrees of freedom (df)*	*Mean squares (MS)*	*F-value*	*P-value*	*critical F-value*
Between groups	0.001828376	14	0.000130598	2.12118899	0.02877061	1.91824856
Within groups	0.002770579	45	6.15684E-05			
Total	0.004598955	59				

within-sd	0.00784655
effective n	4.00
s_bb	0.00415421
s_bb_min	0.00180137
u_bb	0.00415421
u_bb(rel.)	0.29392285

Yttrium

Summary						
Bottle	*Number*	*Sum*	*Mean*	*Variance*		
10	4	24.17643343	6.044108356	0.00333101		
32	4	23.96294617	5.990736541	0.00546814		
65	4	24.25700076	6.06425019	0.00117509		
95	4	24.25914983	6.064787459	0.00308103		
122	4	24.06896405	6.017241011	0.00047782		
156	4	24.14054013	6.035135034	0.00055759		
169	4	24.08393464	6.020983659	0.00089225		
215	4	24.39743812	6.099359531	0.00214287		
255	4	24.4049097	6.101227425	0.0019827		
276	4	24.28345972	6.07086493	0.00032532		
282	4	24.15154021	6.037885052	0.00120806		
309	4	24.56616783	6.141541957	0.00380714		
326	4	24.1163395	6.029084874	0.00330919		
370	4	24.23123116	6.057807791	0.00932109		
394	4	24.1700881	6.042522024	0.0012574		
			6.054502389			
ANOVA						
Source of variation	*sums of squares (SS)*	*degrees of freedom (df)*	*Mean squares (MS)*	*F-value*	*P-value*	*critical F-value*
Between groups	0.081510334	14	0.005822167	2.27803921	0.01870948	1.91824856
Within groups	0.115010094	45	0.00255578			
Total	0.196520428	59				
within-sd	0.05055472					
effective r	4.00					
s_bb	0.02857616					
s_bb_min	0.01160609					
u_bb	0.02857616					
u_bb(rel.)	0.47198192					

Thorium

Summary						
Bottle	*Number*	*Sum*	*Mean*	*Variance*		
10	4	445.083386	111.270847	1.05911019		
32	4	449.314034	112.328509	0.26529854		
65	4	448.540881	112.13522	0.43790031		
95	4	443.422718	110.85568	1.91434879		
122	4	442.842641	110.71066	0.24890767		
156	4	444.779395	111.194849	1.50635492		
169	4	448.887879	112.22197	0.33173074		
215	4	450.459885	112.614971	0.66669241		
255	4	448.183029	112.045757	1.66600398		
276	4	442.428523	110.607131	0.45427794		
282	4	449.053887	112.263472	1.6163752		
309	4	450.603743	112.650936	1.68142244		
326	4	450.794664	112.698666	0.38852261		
370	4	448.415895	112.103974	2.40056585		
394	4	446.224644	111.556161	1.18449072		
			111.817253			

ANOVA						
Source of variation	*sums of squares (SS)*	*degrees of freedom (df)*	*Mean squares (MS)*	*F-value*	*P-value*	*critical F-value*
Between groups	29.3430754	14	2.09593396	1.98704366	0.04148986	1.91824856
Within groups	47.4660069	45	1.05480015			
Total	76.8090823	59				
within-sd	1.027035					
effective r	4.00					
s_bb	0.51018					
s_bb_min	0.235781					
u_bb	0.51018					
u_bb(rel.)	0.456262					

Uranium

Summary						
Bottle	*Number*	*Sum*	*Mean*	*Variance*		
10	4	1121.94581	280.486453	18.0486084		
32	4	1122.2228	280.555701	32.2197406		
65	4	1120.24777	280.061943	1.42133831		
95	4	1113.6345	278.408624	0.80480165		
122	4	1120.00126	280.000315	1.26259895		
156	4	1124.27989	281.069971	0.85232958		
169	4	1122.47643	280.619106	0.62562862		
215	4	1134.81784	283.70446	3.26630133		
255	4	1130.84089	282.710223	2.87321773		
276	4	1127.5168	281.879201	1.19120651		
282	4	1125.21499	281.303748	6.01505009		
309	4	1136.43308	284.10827	0.54666244		
326	4	1136.74264	284.18566	3.98365727		
370	4	1130.64863	282.662158	0.98949913		
394	4	1133.1282	283.28205	1.73804126		
			281.669192			
ANOVA						
Source of variation	*sums of squares (SS)*	*degrees of freedom (df)*	*Mean squares (MS)*	*F-value*	*P-value*	*critical F-value*
Between groups	165.491589	14	11.8208278	2.33802081	0.01586622	1.91824856
Within groups	227.516046	45	5.05591213			
Total	393.007635	59				
within-sd	2.248536					
effective r	4.00					
s_bb	1.300473					
s_bb_min	0.516207					
u_bb	1.300473					
u_bb(rel.)	0.461702					

Oxygen

Summary						
Bottle	*Number*	*Sum*	*Mean*	*Variance*		
10	3	73.23743	24.41247667	0.69236568		
62	3	73.92373	24.64124333	0.10867102		
112	3	75.7268	25.24226667	0.16479905		
181	3	73.39176	24.46392	0.04131152		
255	3	74.02061	24.67353667	0.11360441		
326	3	75.04059	25.01353	0.15053634		
394	3	73.56356	24.52118667	0.05487194		
56	3	74.82035	24.94011667	0.31848352		
169	3	74.74883	24.91627667	0.03963397		
276	3	73.97323	24.65774333	0.06621613		
			24.74822967			
ANOVA						
Source of variation	*sums of squares (SS)*	*degrees of freedom (df)*	*Mean squares (MS)*	*F-value*	*P-value*	*critical F-value*
Between groups	1.949522169	9	0.216613574	1.23744284	0.32792722	2.39281411
Within groups	3.500987159	20	0.175049358			
Total	5.450509327	29				
within-sd	0.418389					
effective n	3.00					
s_bb	0.11770615					
s_bb_min	0.13583749					
u_bb	0.13583749	135.837486				
u_bb(rel.)	0.548877586					

Nitrogen

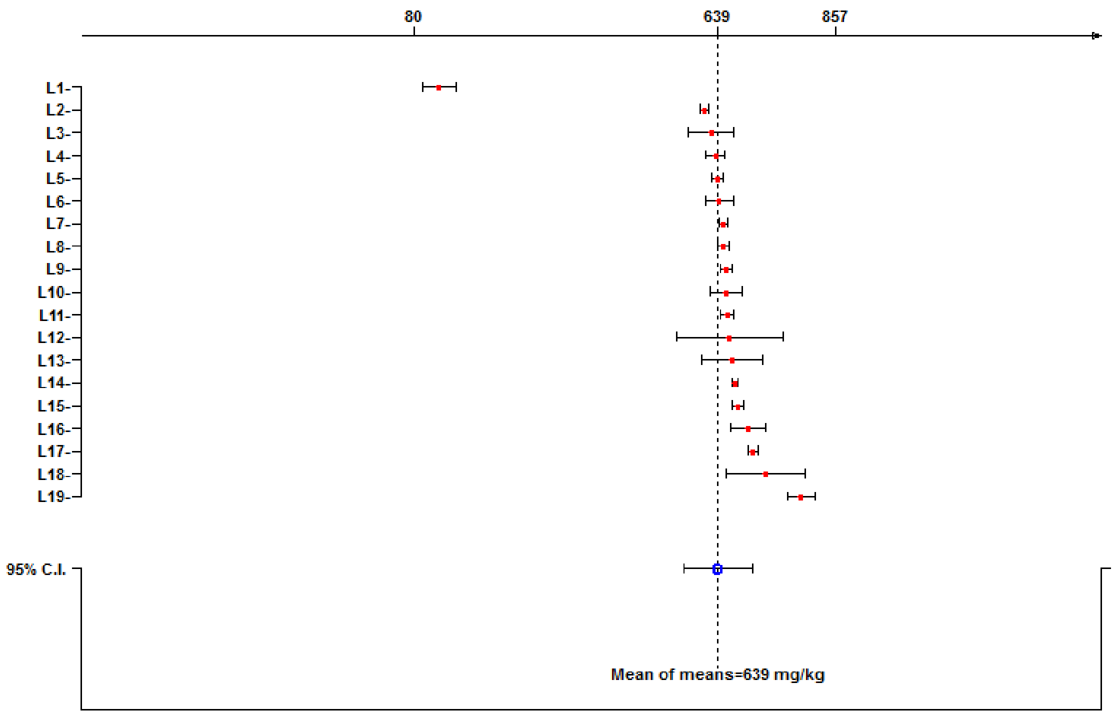
Summary						
Bottle	*Number*	*Sum*	*Mean*	*Variance*		
10	3	0.20316	0.06772	3.4032E-06		
62	3	0.19768	0.065893333	4.1426E-06		
112	3	0.19751	0.065836667	8.3333E-08		
181	3	0.20313	0.06771	1.3671E-06		
255	3	0.19451	0.064836667	8.7184E-06		
326	3	0.20391	0.06797	2.401E-07		
394	3	0.19891	0.066303333	7.9633E-08		
56	3	0.19632	0.06544	9.777E-07		
169	3	0.20008	0.066693333	2.2102E-06		
276	3	0.20203	0.067343333	6.4542E-06		
			0.066574667			
ANOVA						
Source of variation	*sums of squares (SS)*	*degrees of freedom (df)*	*Mean squares (MS)*	*F-value*	*P-value*	*critical F-value*
Between groups	3.16297E-05	9	3.51442E-06	1.26981504	0.31154667	2.39281411
Within groups	5.53532E-05	20	2.76766E-06			
Total	8.69829E-05	29				
within-sd	0.00166363					
effective n	3.00					
s_bb	0.00049892					
s_bb_min	0.00054013					
u_bb	0.00054013	0.5401268				
u_bb(rel.)	0.811309835					

Appendix 2: Statistical evaluation of all results of interlaboratory comparison

Tab. A2.1.1: Results of aluminium determination (values in mg/kg)

Lab. No.	Lab. No. (graph)	Mathod	Sample #1	Sample #2	Sample #3	Sample #4	Sample #5	Sample #6	Mean (mg/kg)	s_i
2	L1	XRF	130	140	100	160	80	140	125.0	29.5
15	L2	ICP OES	616	613	613	625	603	616	614.3	7.1
16	L3	ICP OES	630	590	640	650			627.5	26.3
15	L4	XRF	642	625	666	617	630	630	635.0	17.2
3	L5	ICP OES	640	650	630	630	630	650	638.3	9.8
12	L6	FAAS	680	610	650	630	650	630	641.7	24.0
6	L7	ICP OES	650	640	660	640	650	650	648.3	7.5
7	L8	ICP OES	642	641	636	660	656	659	649.0	10.5
2	L9	ICP OES	663	662	656	639	658	643	653.5	10.1
12	L10	ICP OES	610	651	694	674	657	647	655.5	28.2
18	L11	ICP OES	660	670	650	670	650	640	656.7	12.1
1	L12	DC ARC OES	490	642	716	751	715	648	660.2	93.6
9	L13	ICP OES	720	690	630	590	720	640	665.0	53.2
1	L14	ICP OES	670	676	674	666	675	666	671.2	4.5
10	L15	ICP OES	668	687	669	691	677	667	676.6	10.6
17	L16	ICP OES	720	680	710	730	680	650	695.0	30.2
4	L17	ICP OES	704	695	695	705	715	712	704.3	8.3
14	L18	ICP OES	688	857	750	672	703	688	726.3	69.4
8	L19	FAAS	791	802	782	789	833	760	792.8	24.1

Diagram of means and 95% confidence intervals (to Tab. A2.1.1)



Tab. A2.1.2: Results of statistical tests (aluminium)

Number of data sets	19
Snedecor-F-Test and Bartlett-Test	Pooling not allowed
Dixon (α = 0.05)	Lab. 2, Lab. 19
Dixon (α = 0.01)	Lab. 2
Nalimov (α = 0.05)	Lab. 2
Nalimov (α = 0.01)	Lab. 2
Grubbs (α = 0.05)	Lab. 2
Grubbs (α = 0.01)	Lab. 2
Grubbs Pair (α = 0.05)	---
Grubbs Pair (α = 0.01)	---
Cochran (α = 0.05)	Lab. 1 (DCArc), Lab. 9, Lab. 14
Cochran (α = 0.01)	Lab. 1 (DCArc), Lab. 9, Lab. 14
Kolmogorov-Smirnov-Lilliefors Test (α = 0.05)	Distribution: not normal
Kolmogorov-Smirnov-Lilliefors Test (α = 0.01)	Distribution: not normal
Skewness & Kurtosis Test (α = 0.05)	Distribution: not normal
Skewness & Kurtosis Test (α = 0.01)	Distribution: not normal

The results of Laboratory 2 were withdrawn by the resp. laboratory as technical outlier after discussion.

Tab. A2.1.3: Results of statistical tests (aluminium, without withdrawn result of Lab. 2)

Number of data sets	18
Snedecor-F-Test and Bartlett-Test	Pooling not allowed
Dixon (α = 0.05)	Lab. 8
Dixon (α = 0.01)	---
Nalimov (α = 0.05)	Lab. 8
Nalimov (α = 0.01)	Lab. 8
Grubbs (α = 0.05)	Lab. 8
Grubbs (α = 0.01)	Lab. 8
Grubbs Pair (α = 0.05)	---
Grubbs Pair (α = 0.01)	---
Cochran (α = 0.05)	Lab. 1 (DCArc), Lab. 9, Lab. 14
Cochran (α = 0.01)	Lab. 1 (DCArc), Lab. 9, Lab. 14
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: not normal
Skewness & Kurtosis Test (α = 0.01)	Distribution: not normal

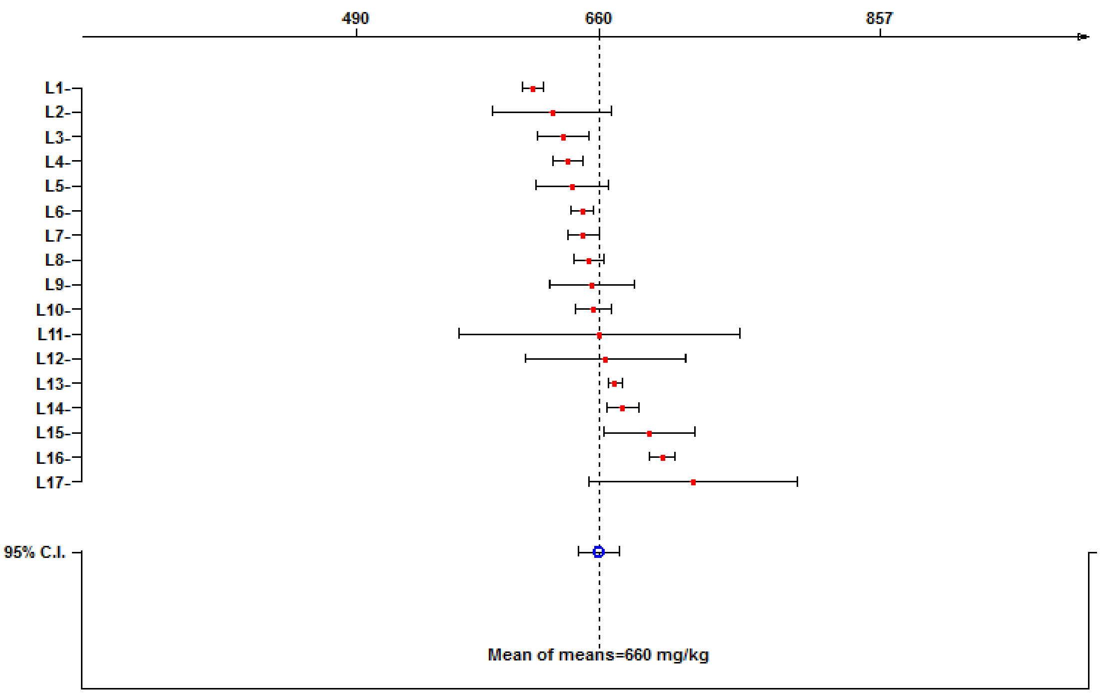
The results of Laboratory 8 were removed.

Tab. A2.1.4: Results of statistical tests (aluminium, without removed result of Lab. 8)

Number of data sets	17
Snedecor-F-Test and Bartlett-Test	Pooling not allowed
Dixon (α = 0.05)	---
Dixon (α = 0.01)	---
Nalimov (α = 0.05)	Lab. 14
Nalimov (α = 0.01)	---
Grubbs (α = 0.05)	---
Grubbs (α = 0.01)	---
Grubbs Pair (α = 0.05)	---
Grubbs Pair (α = 0.01)	---
Cochran (α = 0.05)	Lab. 1 (DCArc), Lab. 9, Lab. 14
Cochran (α = 0.01)	Lab. 1 (DCArc), Lab. 9, Lab. 14
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 results of Laboratory 14 were not removed.

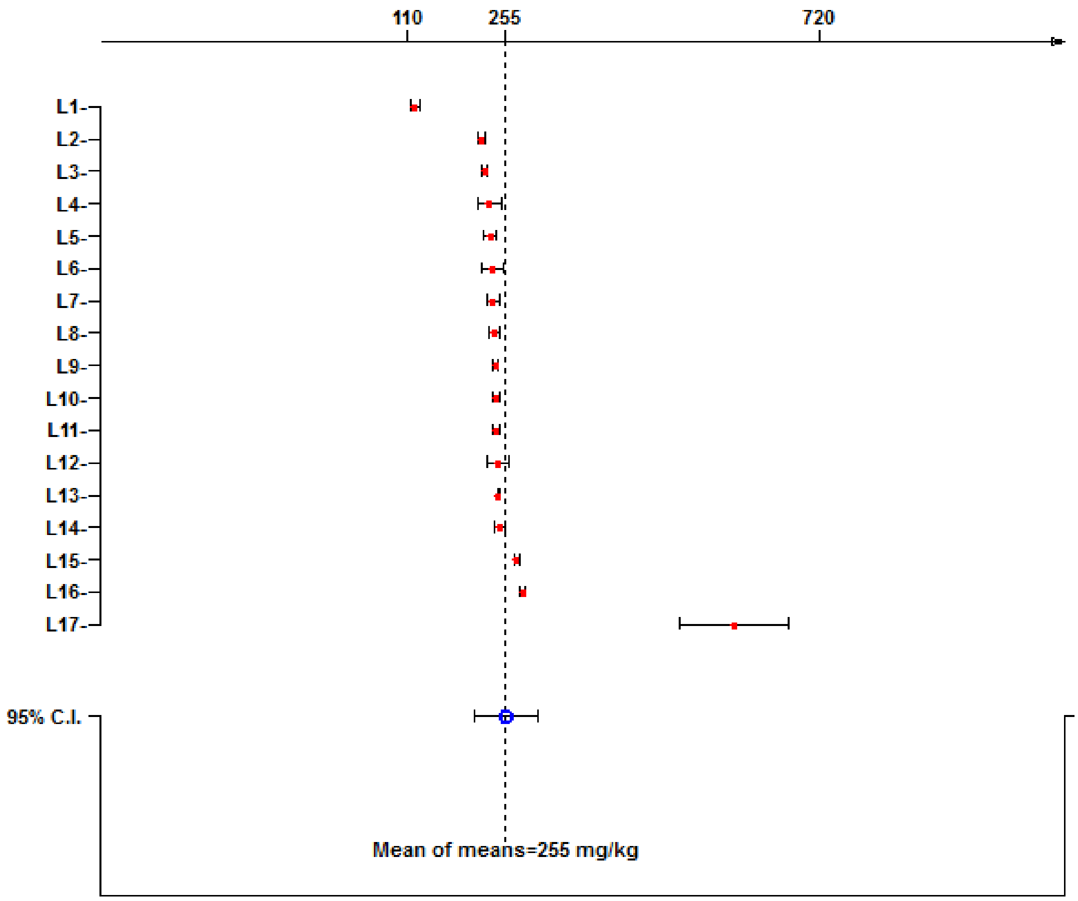
Diagram of accepted means and 95% confidence intervals (aluminium)



Tab. A2.2.1: Results of calcium determination (values in mg/kg)

Lab. No.	Lab. No. (graph)	Mathod	Sample #1	Sample #2	Sample #3	Sample #4	Sample #5	Sample #6	Mean (mg/kg)	s_i
6	L1	ICP OES	110	120	120	120	120	130	120.0	6.3
12	L2	ICP OES	214	220	227	226	217	212	219.3	6.2
15	L3	ICP OES	227	224	229	226	217	220	223.8	4.5
12	L4	FAAS	216	227	232	213	234	260	230.3	16.8
8	L5	ICP OES	244	231	225	221	231	238	231.7	8.4
16	L6	ICP OES	230	230	250	230			235.0	10.0
9	L7	ICP OES	240	230	250	230	240	230	236.7	8.2
3	L8	ICP OES	230	240	250	240	230	240	238.3	7.5
4	L9	ICP OES	243	244	243	239	236	234	239.8	4.2
2	L10	ICP OES	245	245	236	232	238	248	240.7	6.3
7	L11	ICP OES	244	239	236	250	243	235	241.2	5.6
15	L12	XRF	236	233	233	273	239	248	243.7	15.4
1	L13	ICP OES	246	245	245	243	246	243	244.7	1.4
17	L14	ICP OES	250	240	250	240	260	240	246.7	8.2
10	L15	ICP OES	272	273	272	274	272	266	271.6	2.7
14	L16	ICP OES	276	277	279	285	278	285	280.0	4.0
18	L17	ICP OES	720	640	590	570	540	500	593.3	77.9

Diagram of means and 95% confidence intervals (to Tab. A2.2.1)



Tab. A2.2.2: Results of statistical tests (calcium)

Number of data sets	17
Snedecor-F-Test and Bartlett-Test	Pooling not allowed
Dixon (α = 0.05)	Lab. 6, Lab. 18
Dixon (α = 0.01)	Lab. 6, Lab. 18
Nalimov (α = 0.05)	Lab. 18
Nalimov (α = 0.01)	Lab. 18
Grubbs (α = 0.05)	Lab. 18
Grubbs (α = 0.01)	Lab. 18
Grubbs Pair (α = 0.05)	---
Grubbs Pair (α = 0.01)	---
Cochran (α = 0.05)	Lab. 12, Lab. 15, Lab. 18
Cochran (α = 0.01)	Lab. 12, Lab. 15, Lab. 18
Kolmogorov-Smirnov-Lilliefors Test (α = 0.05)	Distribution: not normal
Kolmogorov-Smirnov-Lilliefors Test (α = 0.01)	Distribution: not normal
Skewness & Kurtosis Test (α = 0.05)	Distribution: not normal
Skewness & Kurtosis Test (α = 0.01)	Distribution: not normal

The results of Laboratory 18 were removed as obviousy erroneous values.

Tab. A2.2.3: Results of statistical tests (calcium, without removed result of Lab. 18)

Number of data sets	16
Snedecor-F-Test and Bartlett-Test	Pooling not allowed
Dixon (α = 0.05)	Lab. 6, Lab. 14
Dixon (α = 0.01)	Lab. 6
Nalimov (α = 0.05)	Lab. 6
Nalimov (α = 0.01)	Lab. 6
Grubbs (α = 0.05)	Lab. 6
Grubbs (α = 0.01)	Lab. 6
Grubbs Pair (α = 0.05)	---
Grubbs Pair (α = 0.01)	---
Cochran (α = 0.05)	Lab. 12, Lab. 15
Cochran (α = 0.01)	Lab. 12, Lab. 15
Kolmogorov-Smirnov-Lilliefors Test (α = 0.05)	Distribution: not normal
Kolmogorov-Smirnov-Lilliefors Test (α = 0.01)	Distribution: not normal
Skewness & Kurtosis Test (α = 0.05)	Distribution: not normal
Skewness & Kurtosis Test (α = 0.01)	Distribution: not normal

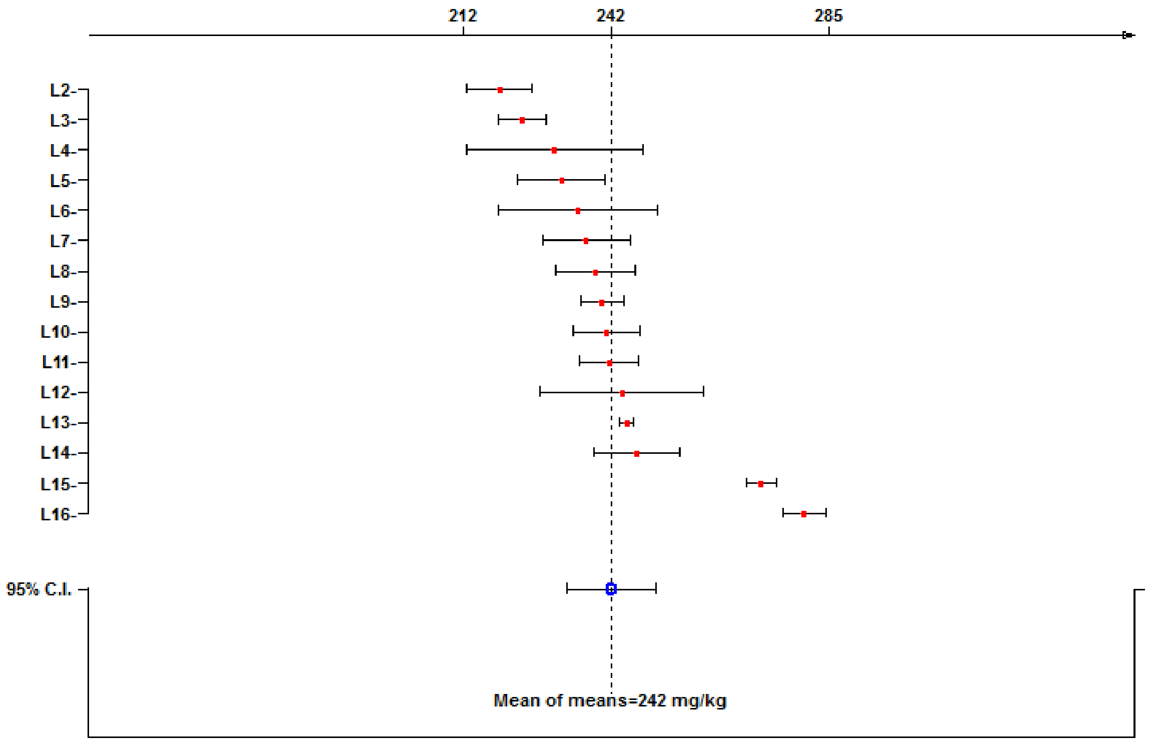
The results of Laboratory 6 were removed.

Tab. A2.2.4: Results of statistical tests (calcium, without removed result of Lab. 6)

Number of data sets	15
Snedecor-F-Test and Bartlett-Test	Pooling not allowed
Dixon (α = 0.05)	Lab. 14
Dixon (α = 0.01)	Lab. 14
Nalimov (α = 0.05)	Lab. 14, Lab. 10
Nalimov (α = 0.01)	Lab. 14
Grubbs (α = 0.05)	Lab. 14
Grubbs (α = 0.01)	---
Grubbs Pair (α = 0.05)	---
Grubbs Pair (α = 0.01)	---
Cochran (α = 0.05)	Lab. 12, Lab. 15
Cochran (α = 0.01)	Lab. 12, Lab. 15
Kolmogorov-Smirnov-Lilliefors Test (α = 0.05)	Distribution: not normal
Kolmogorov-Smirnov-Lilliefors Test (α = 0.01)	Distribution: normal
Skewness & Kurtosis Test (α = 0.05)	Distribution: not normal
Skewness & Kurtosis Test (α = 0.01)	Distribution: normal

The results of Laboratories 14 und 10 were not removed.

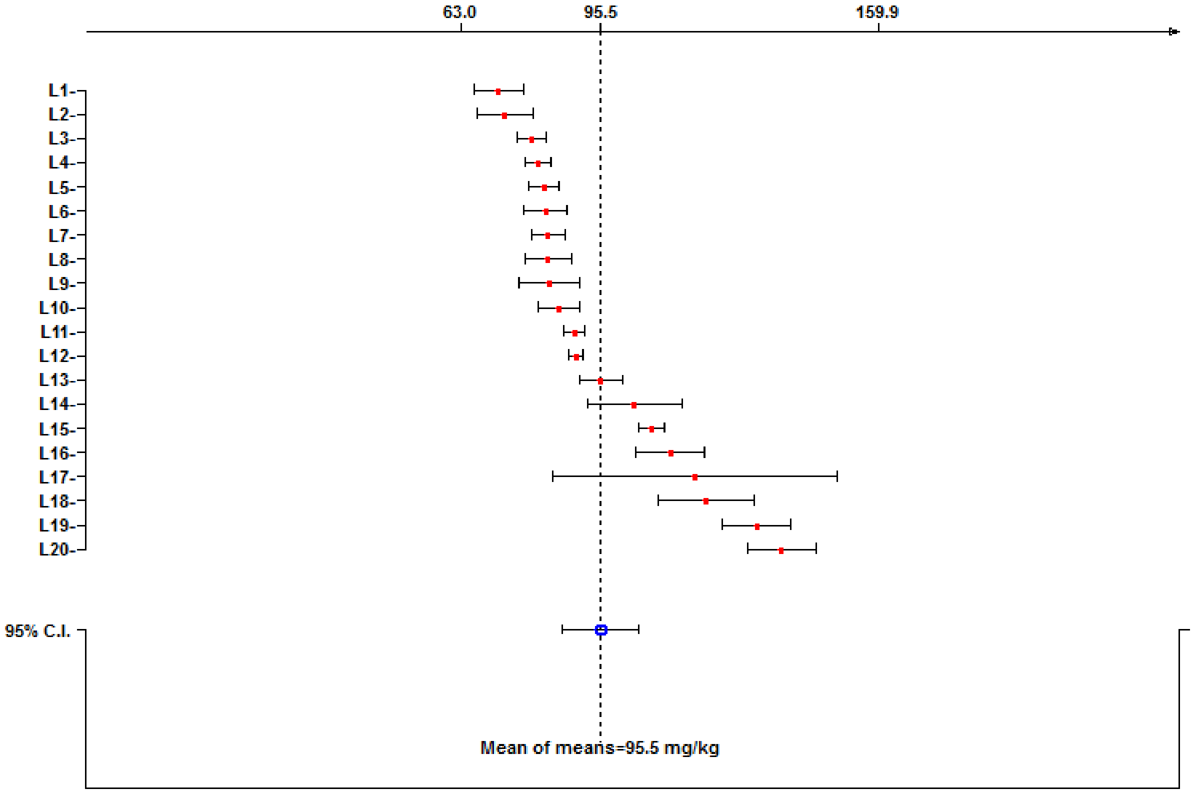
Diagram of accepted means and 95% confidence intervals (calcium)



Tab. A2.3.1: Results of iron determination (values in mg/kg)

Lab. No.	Lab. No. (graph)	Mathod	Sample #1	Sample #2	Sample #3	Sample #4	Sample #5	Sample #6	Mean (mg/kg)	s_i
17	L1	ICP OES	80.0	72.0	72.0	72.0	72.0	63.0	71.8	5.4
14	L2	ICP OES	79.0	70.0	81.0	76.0	67.0	67.0	73.3	6.2
15	L3	XRF	77.0	77.0	76.0	84.0	82.0	81.0	79.5	3.3
1	L4	ETV-ICP OES	82.3	81.9	76.2	84.2	81.4	79.4	80.9	2.8
15	L5	ICP OES	86.7	81.4	82.3	85.2	77.4	81.1	82.4	3.3
11	L6	ICP-MS	91.0	86.0	78.0	80.0	80.0	81.0	82.7	4.9
2	L7	ICP OES	83.0	85.0	90.0	80.0	81.0	81.0	83.3	3.7
3	L8	ICP OES	80.0	80.0	90.0	80.0	80.0	90.0	83.3	5.2
17	L9	F AAS	84.0	79.0	93.0	80.0	82.0		83.6	5.6
4	L10	ICP OES	88.0	83.0	94.0	82.0	82.0	86.0	85.8	4.7
10	L11	ICP OES	88.4	89.6	87.4	90.8	92.9	87.0	89.4	2.2
1	L12	ICP OES	90.9	89.3	88.3	92.0	88.6	89.0	89.7	1.5
12	L13	ICP OES	93.0	92.0	96.0	104.0	97.0	91.0	95.5	4.8
9	L14	ICP OES	120.0	100.0	100.0	110.0	100.0	90.0	103.3	10.3
7	L15	ICP OES	111.0	105.0	105.0	105.0	110.0	108.0	107.3	2.7
18	L16	ICP OES	110.0	120.0	120.0	110.0	110.0	100.0	111.7	7.5
1	L17	DC ARC OES	86.4	86.2	137.8	137.0	159.9	96.3	117.3	31.6
8	L18	FAAS	121.0	131.0	117.0	110.0	133.0	107.0	119.8	10.7
6	L19	ICP OES	120.0	130.0	140.0	130.0	140.0	130.0	131.7	7.5
16	L20	ICP OES	140.0	130.0	140.0	140.0			137.5	5.0

Diagram of means and 95% confidence intervals (to Tab. A2.3.1)



Tab. A2.3.2: Results of statistical tests (iron)

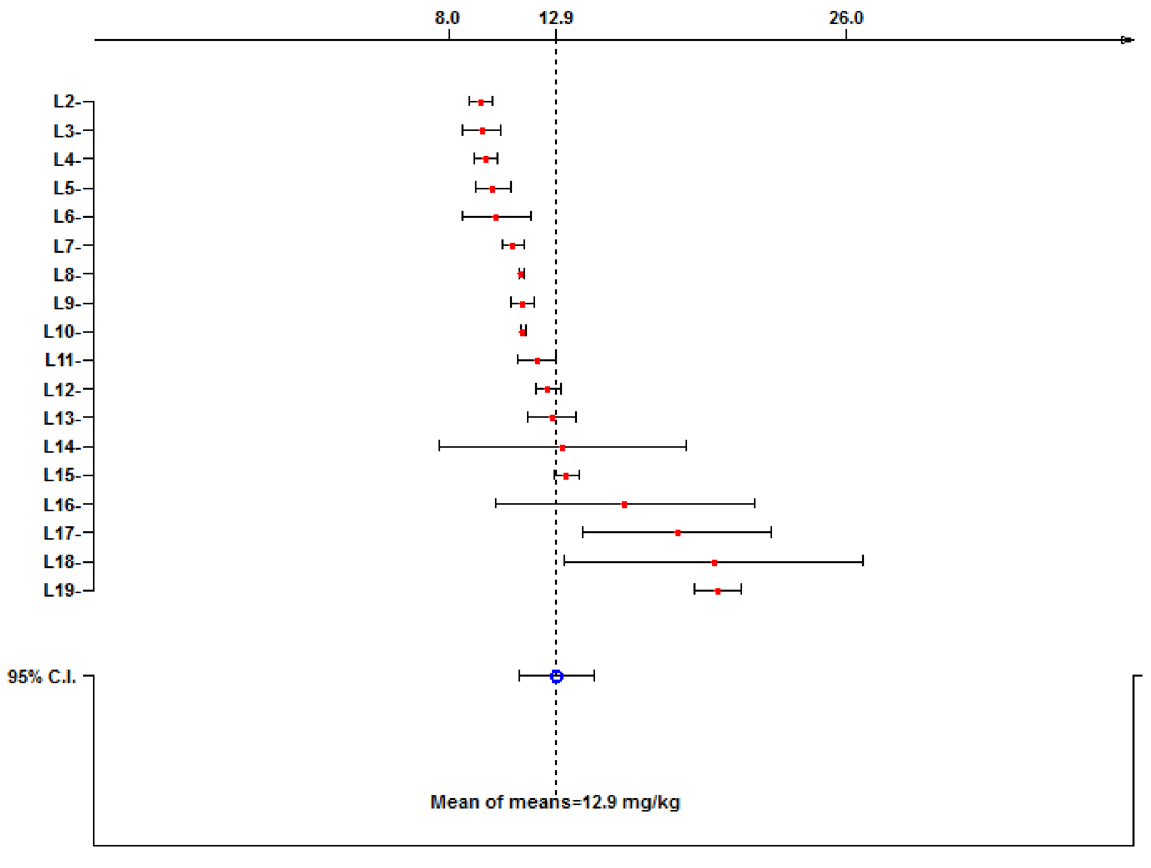
Number of data sets	20
Snedecor-F-Test and Bartlett-Test	Pooling not allowed
Dixon (α = 0.05)	---
Dixon (α = 0.01)	---
Nalimov (α = 0.05)	Lab. 16
Nalimov (α = 0.01)	---
Grubbs (α = 0.05)	---
Grubbs (α = 0.01)	---
Grubbs Pair (α = 0.05)	---
Grubbs Pair (α = 0.01)	---
Cochran (α = 0.05)	Lab. 1 (DCArc), Lab. 9, Lab. 8
Cochran (α = 0.01)	Lab. 1 (DCArc)
Kolmogorov-Smirnov-Lilliefors Test (α = 0.05)	Distribution: not normal
Kolmogorov-Smirnov-Lilliefors Test (α = 0.01)	Distribution: normal
Skewness & Kurtosis Test (α = 0.05)	Distribution: not normal
Skewness & Kurtosis Test (α = 0.01)	Distribution: normal

The results of Laboratory 16 were not removed.

Tab. A2.4.1: Results of magnesium determination (values in mg/kg)

Lab. No.	Lab. No. (graph)	Mathod	Sample #1	Sample #2	Sample #3	Sample #4	Sample #5	Sample #6	Mean (mg/kg)	s_i	H.W. CI (95%)
7	L2	ICP OES	10,0	9,1	9,0	10,1	9,4	9,1	9,5	0,48	0,51
17	L3	ICP OES	10,0	8,0	9,0	10,0	10,0	10,0	9,5	0,84	0,88
2	L4	ICP OES	10,0	10,0	10,0	9,0	9,0	10,0	9,7	0,52	0,54
1	L5	ETV-ICP OES	10,0	11,4	9,4	10,2	9,6	9,5	10,0	0,74	0,78
11	L6	ICP-MS	12,0	12,0	10,0	9,0	9,0	9,0	10,2	1,47	1,54
15	L7	ICP OES	10,6	10,8	11,5	10,3	11,3	11,0	10,9	0,44	0,47
1	L8	ICP OES	11,4	11,3	11,4	11,3	11,3	11,1	11,3	0,11	0,11
4	L9	ICP OES	12,0	12,0	11,0	11,0	11,0	11,0	11,3	0,52	0,54
10	L10	ICP OES	11,3	11,5	11,3	11,4	11,4	11,2	11,3	0,08	0,08
17	L11	F AAS	13,0	12,0	11,0	12,0	12,0	---	12,0	0,71	0,74
12	L12	ICP OES	13,0	12,0	12,0	13,0	12,0	13,0	12,5	0,55	0,57
3	L13	ICP OES	13,0	14,0	11,0	13,0	13,0	12,0	12,7	1,03	1,08
14	L14	ICP OES	15,0	23,0	8,0	11,0	11,0	11,0	13,2	5,31	5,57
9	L15	ICP OES	13,0	13,0	13,0	13,0	14,0	14,0	13,3	0,52	0,54
1	L16	DC Arc OES	8,3	17,8	20,0	18,9	---	14,9	16,0	4,68	4,91
18	L17	ICP OES	20,0	20,0	15,0	25,0	15,0	15,0	18,3	4,08	4,29
16	L18	ICP OES	19,0	16,0	19,0	26,0			20,0	4,24	4,45
8	L19	FAAS	22,0	20,0	20,0	19,0	20,0	20,0	20,2	0,98	1,03

Diagram of means and 95% confidence intervals (to Tab. A2.4.1)



Tab. A2.4.2: Results of statistical tests (magnesium)

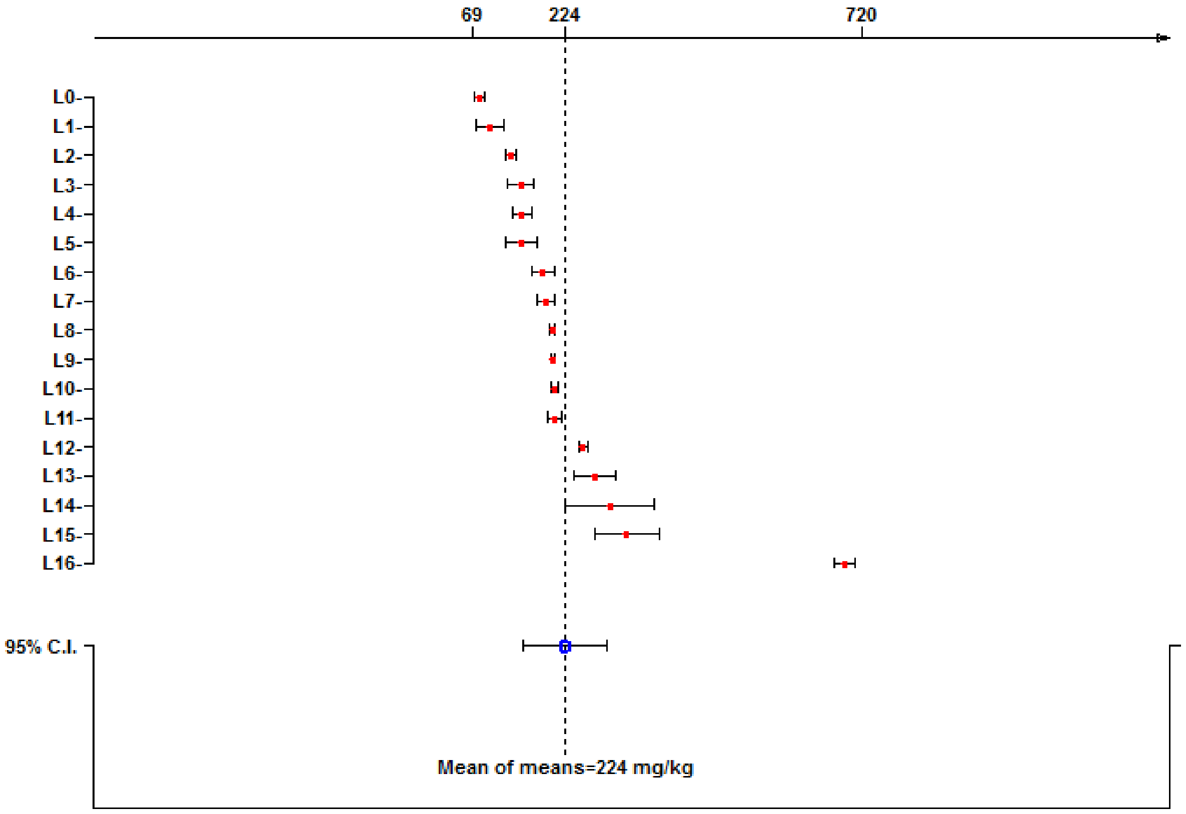
Number of data sets	18
Snedecor-F-Test and Bartlett-Test	Pooling not allowed
Dixon (α = 0.05)	---
Dixon (α = 0.01)	---
Nalimov (α = 0.05)	Labs. 8, 16
Nalimov (α = 0.01)	---
Grubbs (α = 0.05)	---
Grubbs (α = 0.01)	---
Grubbs Pair (α = 0.05)	---
Grubbs Pair (α = 0.01)	---
Cochran (α = 0.05)	Lab. 1 (DCArc), Labs. 11, 14, 16, 18
Cochran (α = 0.01)	Lab. 1 (DCArc), Labs. 11, 14, 16, 18
Kolmogorov-Smirnov-Lilliefors Test (α = 0.05)	Distribution: not normal
Kolmogorov-Smirnov-Lilliefors Test (α = 0.01)	Distribution: normal
Skewness & Kurtosis Test (α = 0.05)	Distribution: not normal
Skewness & Kurtosis Test (α = 0.01)	Distribution: normal

The oulying results were not removed.

Tab. A2.5.1: Results of silicon determination (values in mg/kg)

Lab. No.	Lab. No. (graph)	Mathod	Sample #1	Sample #2	Sample #3	Sample #4	Sample #5	Sample #6	Mean (mg/kg)	s_i
4	L0	ICP OES	86.0	87.0	73.0	78.0	69.0	92.0	80.8	8.93
3	L1	ICP OES	135.0	90.0	74.0	79.0	110.0	99.0	97.8	22.43
1	L2	ICP OES	129.0	118.0	141.0	130.0	137.0	139.0	132.3	8.52
7	L3	ICP OES	124.0	145.0	134.0	161.0	182.0	156.0	150.3	20.68
20	L4	Photometry	160.0	150.0	130.0	160.0	140.0	170.0	151.7	14.72
1	L5	ETV-ICP OES	183.7	142.0	129.9	156.6	178.2	122.8	152.2	25.11
17	L6	ICP OES	177.0	160.0	184.0	187.0	197.0	216.0	186.8	18.86
6	L7	ET AAS	186.0	176.0	208.0	195.0	181.0	206.0	192.0	13.22
10	L8	ICP OES	202.0	204.9	200.1	209.5	203.9	196.5	202.8	4.44
10	L9	ICP OES	202.9	204.1	205.1	206.5	200.5	205.8	204.1	2.19
17	L10	ICP OES	202.0	201.0	209.0	210.0	209.0		206.2	4.32
12	L11	ICP OES	219.0	217.0	198.0	208.0	209.0	187.0	206.3	12.06
5	L12	SS ET AAS	258.7	253.6	265.6	246.0	244.5	257.7	254.3	8.07
16	L13	ICP OES	300.0	250.0	260.0	280.0			272.5	22.17
15	L14	XRF	336.2	274.6	288.8	318.4	181.7	394.1	299.0	71.08
1	L15	DC Arc OES	293.4	327.2	348.0	370.2	380.0	241.3	326.7	52.13
18	L16	XRF	690.0	720.0	690.0	680.0	670.0	690.0	690.0	16.73

Diagram of means and 95% confidence intervals (to Tab. A2.5.1)



Tab. A2.5.2: Results of statistical tests (silicon)

Number of data sets	17
Snedecor-F-Test and Bartlett-Test	Pooling not allowed
Dixon (α = 0.05)	Lab. 18
Dixon (α = 0.01)	Lab. 18
Nalimov (α = 0.05)	Lab. 18
Nalimov (α = 0.01)	Lab. 18
Grubbs (α = 0.05)	Lab. 18
Grubbs (α = 0.01)	Lab. 18
Grubbs Pair (α = 0.05)	---
Grubbs Pair (α = 0.01)	---
Cochran (α = 0.05)	Lab. 1 (DCArc), Lab. 15
Cochran (α = 0.01)	Lab. 1 (DCArc), Lab. 15
Kolmogorov-Smirnov-Lilliefors Test (α = 0.05)	Distribution: not normal
Kolmogorov-Smirnov-Lilliefors Test (α = 0.01)	Distribution: not normal
Skewness & Kurtosis Test (α = 0.05)	Distribution: not normal
Skewness & Kurtosis Test (α = 0.01)	Distribution: not normal

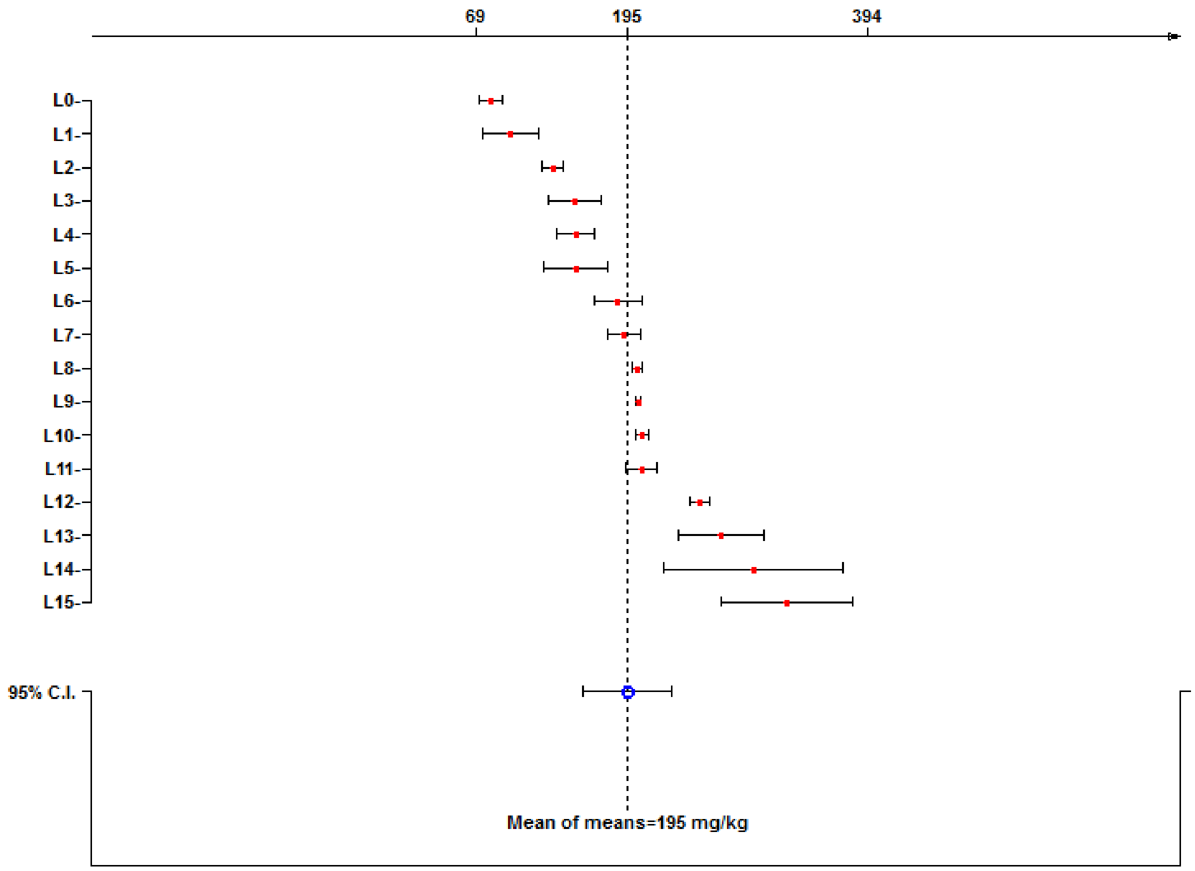
The results of Laboratory 18 were removed.

Tab. A2.5.3: Results of statistical tests (silicon, without removed result of Lab. 18)

Number of data sets	16
Snedecor-F-Test and Bartlett-Test	Pooling not allowed
Dixon (α = 0.05)	---
Dixon (α = 0.01)	---
Nalimov (α = 0.05)	Lab. 1 (DCArc)
Nalimov (α = 0.01)	---
Grubbs (α = 0.05)	---
Grubbs (α = 0.01)	---
Grubbs Pair (α = 0.05)	---
Grubbs Pair (α = 0.01)	---
Cochran (α = 0.05)	Lab. 1 (DCArc), Lab. 15
Cochran (α = 0.01)	Lab. 1 (DCArc), Lab. 15
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 results of Laboratory 1 (DCArc) were not removed.

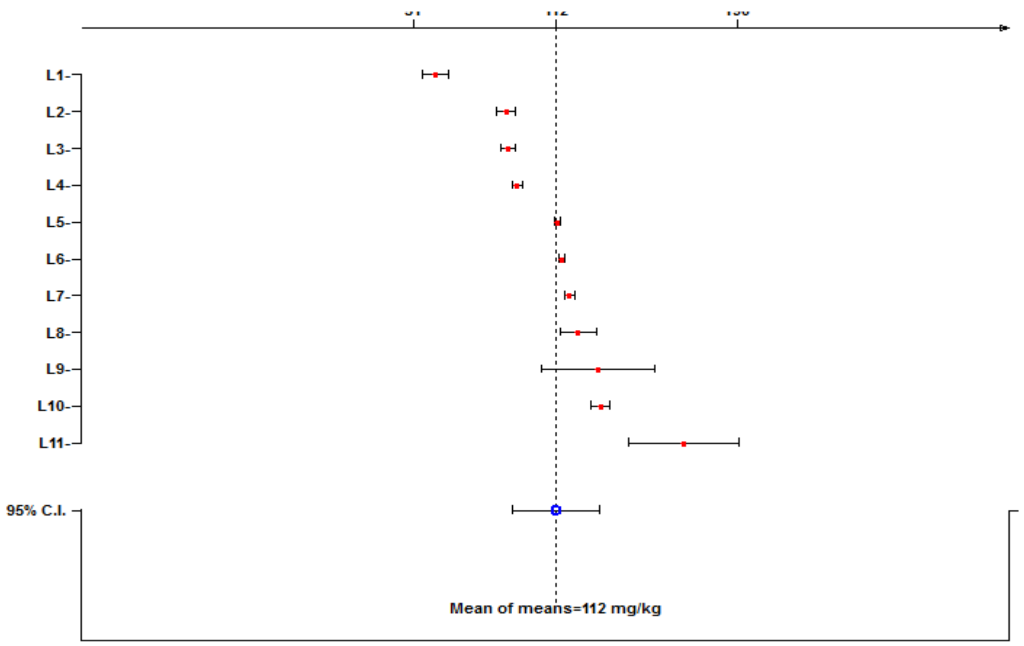
Diagram of accepted means and 95% confidence intervals (silicon)



Tab. A2.6.1: Results of thorium determination (values in mg/kg)

Lab. No.	Lab. No. (graph)	Mathod	Sample #1	Sample #2	Sample #3	Sample #4	Sample #5	Sample #6	Mean (mg/kg)	s_i
14	L1	ICP OES	51.0	60.0	58.0	65.0	65.0	63.0	60.3	5.35
3	L2	ICP-MS	96.0	90.0	87.0	94.0	88.0	88.0	90.5	3.67
12	L3	ICP OES	88.0	95.0	92.0	92.0	88.0	93.0	91.3	2.80
2	L4	ICP-MS	93.0	95.0	95.0	94.0	98.0	97.0	95.3	1.86
1	L5	ICP-MS	113.4	112.2	113.8	111.4	113.2	111.4	112.6	1.05
19	L6	K_0-INAA	116.0	116.0	115.0	114.0	113.0	114.0	114.7	1.21
4	L7	ICP-MS	119.0	120.0	120.0	116.0	116.0	116.0	117.8	2.04
1	L8	ETV-ICP OES	129.0	132.0	115.0	120.0	119.0	114.0	121.5	7.40
17	L9	ICP OES	122.0	177.0	125.0	120.0	119.0	117.0	130.0	23.19
15	L10	ICP-MS	130.0	127.0	135.0	132.0	135.0	127.0	131.0	3.63
17	L11	XRF	140.0	140.0	170.0	190.0	190.0	170.0	166.7	22.51

Diagram of means and 95% confidence intervals (to Tab. A2.6.1)



Tab. A2.6.2: Results of statistical tests (thorium)

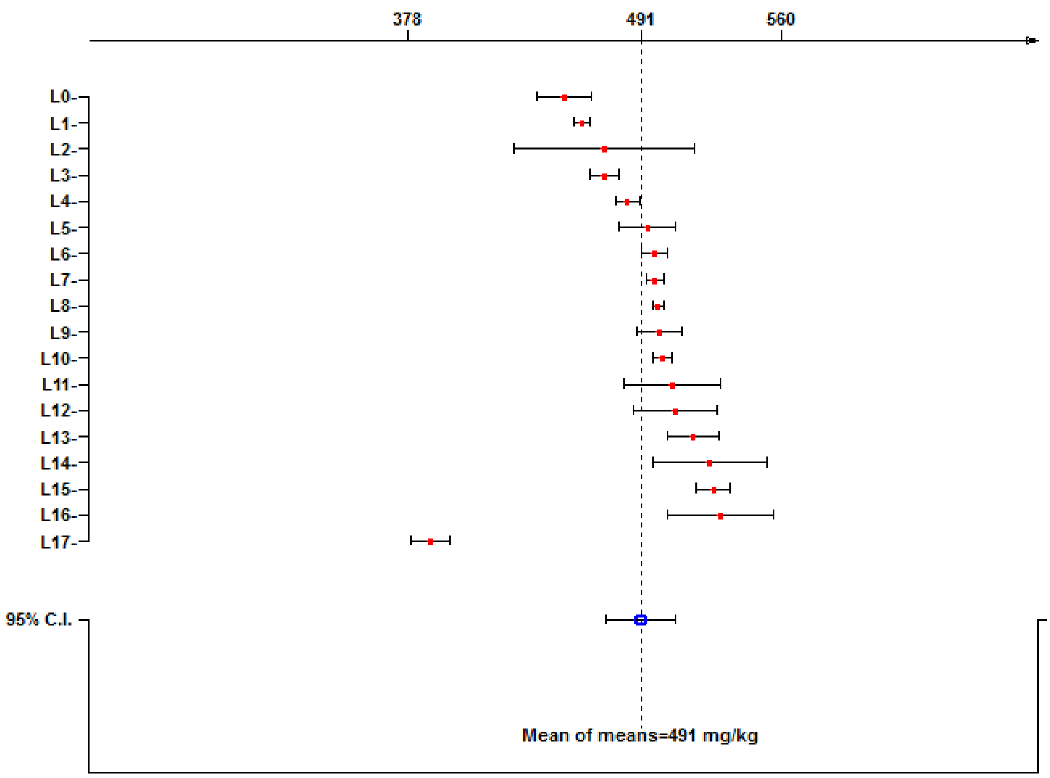
Number of data sets	11
Snedecor-F-Test and Bartlett-Test	Pooling not allowed
Dixon (α = 0.05)	---
Dixon (α = 0.01)	---
Nalimov (α = 0.05)	Lab. 14, Lab. 17
Nalimov (α = 0.01)	---
Grubbs (α = 0.05)	---
Grubbs (α = 0.01)	---
Grubbs Pair (α = 0.05)	---
Grubbs Pair (α = 0.01)	---
Cochran (α = 0.05)	Lab. 1 (ETV), Labs. 9, 14, 17
Cochran (α = 0.01)	Lab. 1 (ETV), Labs. 9, 17
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 results of Laboratories 14 and 17 were not removed.

Tab. A2.7.1: Results of titanium determination (values in mg/kg)

Lab. No.	Lab. No. (graph)	Mathod	Sample #1	Sample #2	Sample #3	Sample #4	Sample #5	Sample #6	Mean (mg/kg)	s_i
14	L17	ICP OES	400	378	378	394	390	393	388.8	9
11	L0	ICP OES	460	463	447	455	467	432	454.0	13
15	L1	ICP OES	465	462	462	469	458	462	463.0	4
1	L2	ETV-ICP OES	441	555	454	481	451	460	473.7	42
2	L3	ICP OES	479	479	478	465	477	466	474.0	7
3	L4	ICP OES	480	490	490	480	490	480	485.0	5
12	L5	ICP OES	507	499	498	499	496	469	494.7	13
10	L6	ICP OES	493	504	495	507	494	495	497.9	6
18	L7	ICP OES	500	500	500	500	500	490	498.3	4
1	L8	ICP OES	501	498	504	500	499	497	499.8	2
7	L9	ICP OES	491	492	489	512	508	509	500.2	11
4	L10	ICP OES	496	497	502	506	507	502	501.7	5
8	L11	XRF	515	505	505	545	480	490	506.7	23
9	L12	ICP OES	520	510	530	480	520	490	508.3	19
15	L13	XRF	540	511	519	510	510	511	516.0	12
16	L14	ICP OES	540	500	530	530			525.0	17
6	L15	ICP OES	540	520	520	520	530	530	526.7	8
17	L16	ICP OES	540	500	540	540	560	500	530.0	24

Diagram of means and 95% confidence intervals (to Tab. A2.7.1)



Tab. A2.7.2: Results of statistical tests (titanium)

Number of data sets	18
Snedecor-F-Test and Bartlett-Test	Pooling not allowed
Dixon (α = 0.05)	Lab. 14
Dixon (α = 0.01)	---
Nalimov (α = 0.05)	Lab. 14
Nalimov (α = 0.01)	Lab. 14
Grubbs (α = 0.05)	Lab. 14
Grubbs (α = 0.01)	Lab. 14
Grubbs Pair (α = 0.05)	---
Grubbs Pair (α = 0.01)	---
Cochran (α = 0.05)	Lab. 1 (ETV), Labs. 6, 8, 9, 17
Cochran (α = 0.01)	Lab. 1 (ETV)
Kolmogorov-Smirnov-Lilliefors Test (α = 0.05)	Distribution: not normal
Kolmogorov-Smirnov-Lilliefors Test (α = 0.01)	Distribution: normal
Skewness & Kurtosis Test (α = 0.05)	Distribution: not normal
Skewness & Kurtosis Test (α = 0.01)	Distribution: not normal

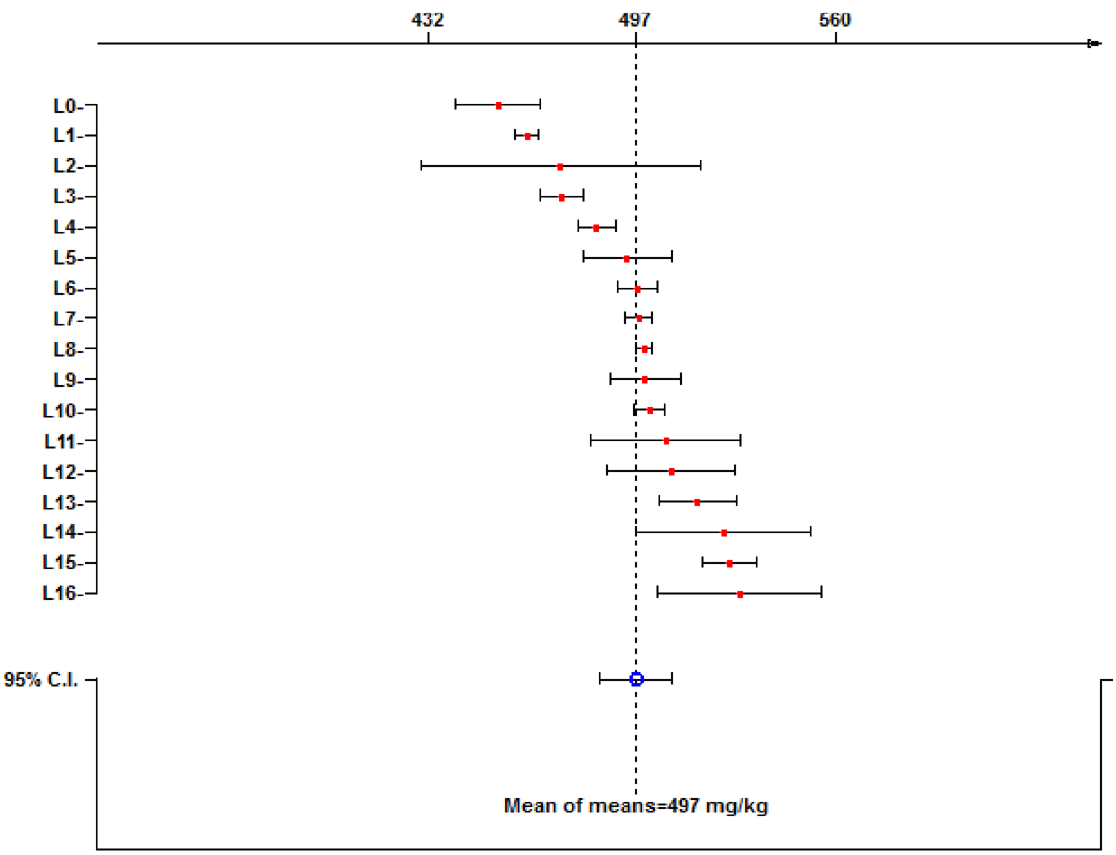
The results of Laboratory 14 were removed.

Tab. A2.7.3: Results of statistical tests (without removed result of Lab. 14)

Number of data sets	17
Snedecor-F-Test and Bartlett-Test	Pooling not allowed
Dixon (α = 0.05)	---
Dixon (α = 0.01)	---
Nalimov (α = 0.05)	Lab. 11
Nalimov (α = 0.01)	---
Grubbs (α = 0.05)	---
Grubbs (α = 0.01)	---
Grubbs Pair (α = 0.05)	---
Grubbs Pair (α = 0.01)	---
Cochran (α = 0.05)	Lab. 1 (ETV), Labs. 6, 8, 9, 17
Cochran (α = 0.01)	Lab. 1 (ETV)
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 results of Laboratory 11 were not removed.

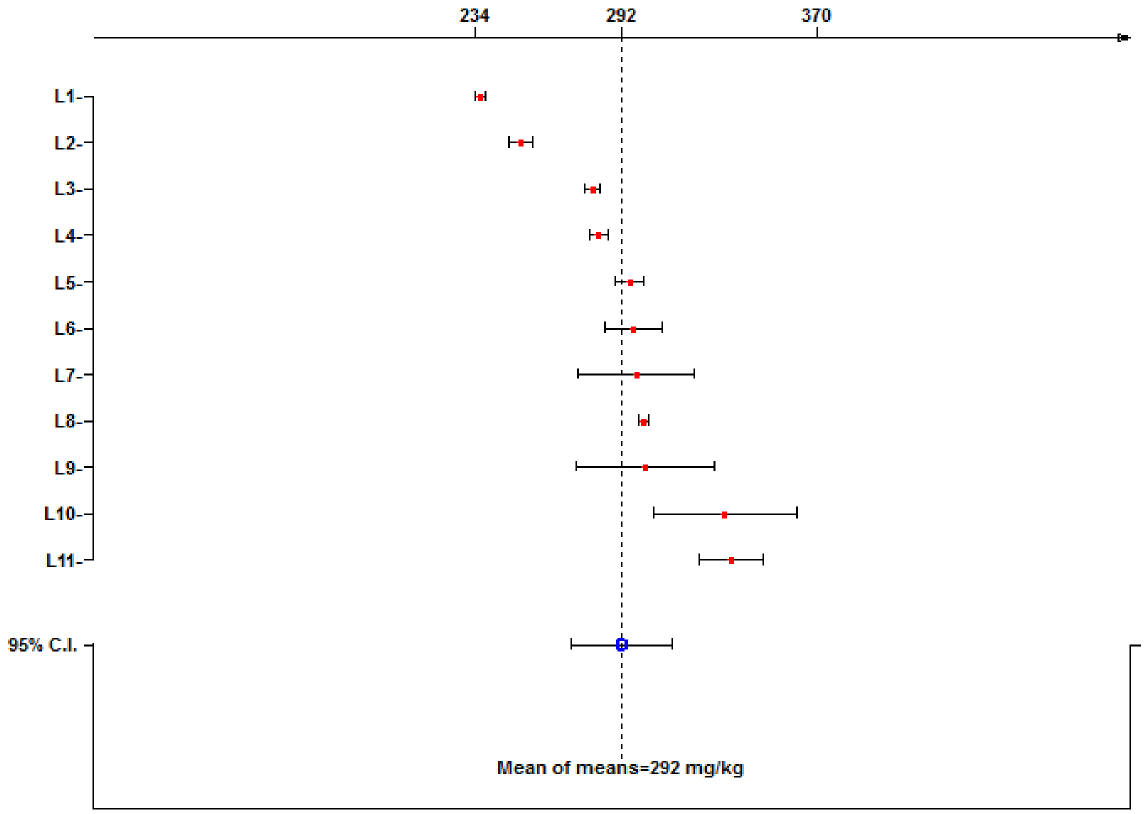
Diagram of accepted means and 95% confidence intervals (titanium)



Tab. A2.8.1: Results of uranium determination (values in mg/kg)

Lab. No.	Lab. No. (graph)	Mathod	Sample #1	Sample #2	Sample #3	Sample #4	Sample #5	Sample #6	Mean (mg/kg)	s_i
3	L1	ICP-MS	236	236	239	234	235	234	235.7	1.86
2	L2	ICP-MS	247	251	250	248	257	258	251.8	4.62
14	L3	ICP OES	282	280	282	284	279	276	280.5	2.81
1	L4	ICP-MS	286	282	288	280	285	281	283.6	3.27
4	L5	ICP-MS	300	301	300	292	290	289	295.3	5.57
12	L6	ICP OES	299	286	303	314	296	285	297.2	10.91
1	L7	ETV-ICP OES	301	332	292	306	294	264	298.2	22.08
19	L8	K_0-INAA	302	303	303	299	300	298	300.8	2.14
17	L9	ICP OES	340	290	330	280	290	280	301.7	26.39
17	L10	XRF	310	300	370	330	360	330	333.3	27.33
15	L11	ICP-MS	332	320	339	341	355	328	335.8	12.09

Diagram of means and 95% confidence intervals (to Tab. A2.8.1)



Tab. A2.8.2: Results of statistical tests (uranium)

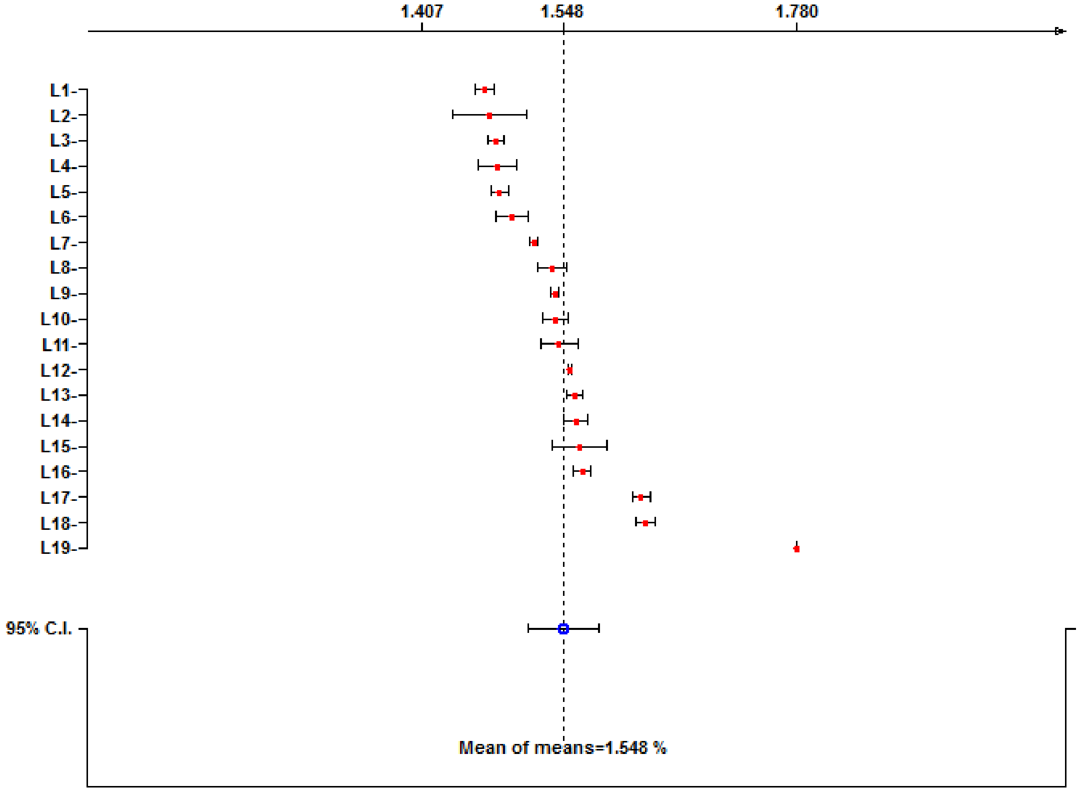
Number of data sets	11
Snedecor-F-Test and Bartlett-Test	Pooling not allowed
Dixon (α = 0.05)	---
Dixon (α = 0.01)	---
Nalimov (α = 0.05)	Lab. 3
Nalimov (α = 0.01)	---
Grubbs (α = 0.05)	---
Grubbs (α = 0.01)	---
Grubbs Pair (α = 0.05)	---
Grubbs Pair (α = 0.01)	---
Cochran (α = 0.05)	Lab. 1 (ETV), Lab. 17 (ICP), Lab. 17 (XRF), Labs. 12, 16
Cochran (α = 0.01)	Lab. 1 (ETV), Lab. 17 (ICP), Labs. 12, 16
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 results of Laboratory 3 were not removed.

Tab. A2.9.1: Results of hafnium determination (values in mg/kg)

Lab. No.	Lab. No. (graph)	Mathod	Sample #1	Sample #2	Sample #3	Sample #4	Sample #5	Sample #6	Mean (mg/kg)	s_i
7	L1	ICP OES	1.458	1.475	1.461	1.476	1.480	1.464	1.469	0.009
14	L2	ICP OES	1.471	1.503	1.491	1.407	1.493	1.481	1.474	0.035
8	L3	XRF	1.4935	1.4830	1.4805	1.4745	1.4805	1.4730	1.481	0.007
3	L4	ICP OES	1.456	1.485	1.472	1.509	1.478	1.492	1.482	0.018
19	L5	K_0-INAA	1.488	1.498	1.486	1.477	1.483	1.475	1.485	0.008
1	L6	ICP OES	1.477	1.519	1.506	1.486	1.492	1.503	1.497	0.015
15	L7	ICP OES	1.52	1.52	1.52	1.52	1.51	1.52	1.518	0.004
9	L8	ICP OES	1.53	1.56	1.52	1.54	1.53	1.54	1.537	0.014
11	L9	ICP OES	1.540	1.538	1.532	1.543	1.541	1.541	1.539	0.004
10	L10	ICP OES	1.52	1.54	1.54	1.56	1.54	1.54	1.540	0.013
12	L11	ICP OES	1.56	1.54	1.55	1.55	1.51	1.55	1.543	0.018
15	L12	XRF	1.554	1.553	1.558	1.554	1.552	1.554	1.554	0.002
17	L13	XRF	1.55	1.55	1.56	1.57	1.56	1.56	1.558	0.008
18	L14	ICP OES	1.56	1.58	1.55	1.55	1.56	1.56	1.560	0.011
4	L15	ICP OES	1.560	1.602	1.591	1.544	1.547	1.542	1.564	0.026
2	L16	XRF	1.56	1.56	1.56	1.57	1.58	1.57	1.567	0.008
6	L17	ICP OES	1.62	1.63	1.63	1.61	1.63	1.63	1.625	0.008
10	L18	ICP OES	1.62	1.63	1.64	1.63	1.62	1.64	1.630	0.009
16	L19	ICP OES	1.78	1.78	1.78	1.78			1.780	0.000

Diagram of means and 95% confidence intervals (to Tab. A2.9.1)



Tab. A2.9.2: Results of statistical tests (hafnium)

Number of data sets	19
Snedecor-F-Test and Bartlett-Test	Pooling not allowed
Dixon (α = 0.05)	Lab. 16
Dixon (α = 0.01)	---
Nalimov (α = 0.05)	Lab. 16
Nalimov (α = 0.01)	Lab. 16
Grubbs (α = 0.05)	Lab. 16
Grubbs (α = 0.01)	Lab. 16
Grubbs Pair (α = 0.05)	---
Grubbs Pair (α = 0.01)	---
Cochran (α = 0.05)	Labs. 4, 14
Cochran (α = 0.01)	Labs. 4, 14
Kolmogorov-Smirnov-Lilliefors Test (α = 0.05)	Distribution: not normal
Kolmogorov-Smirnov-Lilliefors Test (α = 0.01)	Distribution: not normal
Skewness & Kurtosis Test (α = 0.05)	Distribution: not normal
Skewness & Kurtosis Test (α = 0.01)	Distribution: not normal

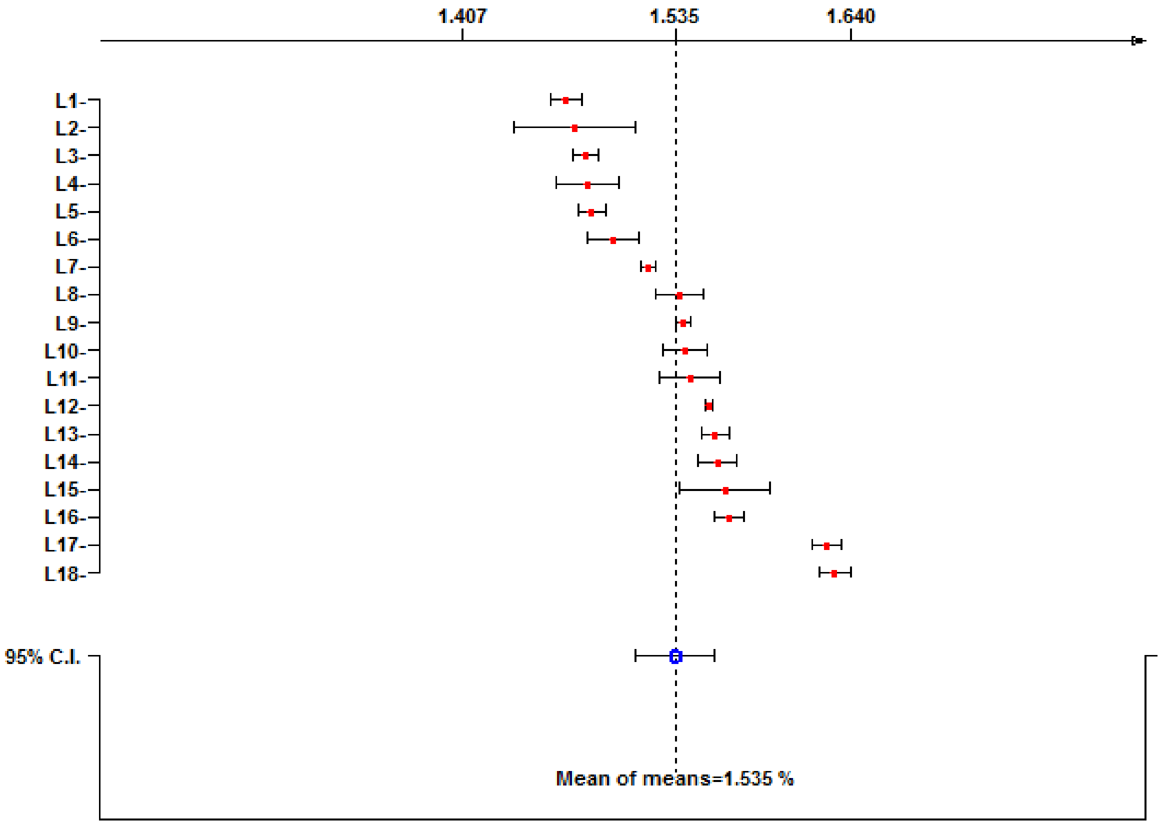
The results of Laboratory 16 were removed.

Tab. A2.9.3: Results of statistical tests (hafnium, without removed result of Lab. 16)

Number of data sets	18
Snedecor-F-Test and Bartlett-Test	Pooling not allowed
Dixon (α = 0.05)	---
Dixon (α = 0.01)	---
Nalimov (α = 0.05)	Lab. 6, 10
Nalimov (α = 0.01)	---
Grubbs (α = 0.05)	---
Grubbs (α = 0.01)	---
Grubbs Pair (α = 0.05)	---
Grubbs Pair (α = 0.01)	---
Cochran (α = 0.05)	Labs. 4, 14
Cochran (α = 0.01)	Labs. 4, 14
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 results of Laboratories 6 and 10 were not removed.

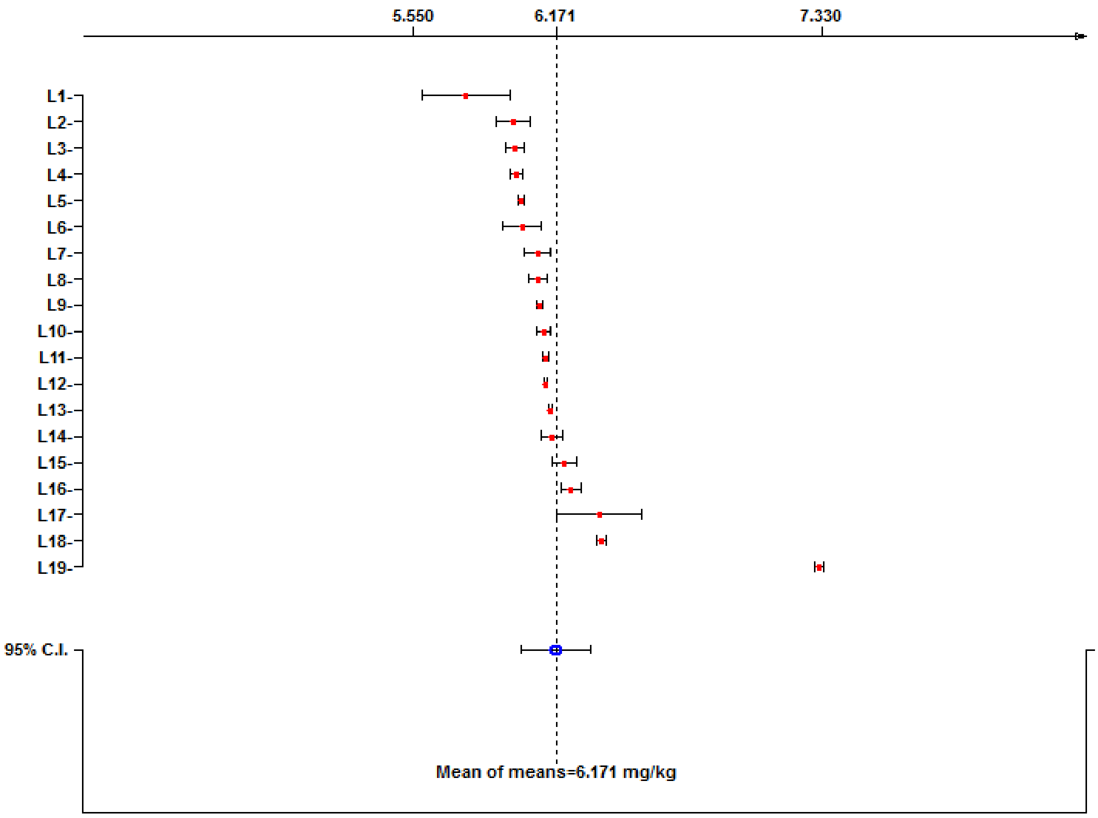
Diagram of accepted means and 95% confidence intervals (hafnium)



Tab. A2.10.1: Results of yttrium determination (values in mg/kg)

Lab. No.	Lab. No. (graph)	Mathod	Sample #1	Sample #2	Sample #3	Sample #4	Sample #5	Sample #6	Mean (mg/kg)	s_i
14	L1	ICP OES	5.55	5.89	5.98	5.55	5.89	5.82	5.780	0.185
4	L2	ICP OES	5.970	6.016	5.992	6.017	5.856	6.055	5.984	0.069
1	L3	ICP OES (1)	5.971	6.038	6.004	5.996	6.015	5.931	5.992	0.037
15	L4	ICP OES	5.96	6.00	6.02	6.02	5.98	6.01	5.998	0.024
17	L5	XRF	6.01	6.03	6.03	6.03	6.02	6.00	6.020	0.013
3	L6	ICP OES	6.011	6.166	6.012	5.930	6.052	5.971	6.024	0.081
12	L7	ICP OES	6.13	6.08	6.11	6.10	5.99	6.14	6.092	0.054
9	L8	ICP OES	6.08	6.16	6.07	6.10	6.09	6.06	6.093	0.036
7	L9	ICP OES	6.114	6.106	6.113	6.090	6.083	6.084	6.098	0.014
1	L10	ICP OES (2)	6.078	6.154	6.137	6.126	6.112	6.100	6.118	0.027
15	L11	XRF	6.123	6.115	6.147	6.124	6.113	6.128	6.125	0.012
11	L12	ICP OES	6.122	6.116	6.131	6.130	6.123	6.141	6.127	0.009
8	L13	XRF	6.1420	6.1515	6.1420	6.1565	6.1470	6.1475	6.148	0.006
10	L14	ICP OES	6.14	6.22	6.13	6.20	6.11	6.13	6.155	0.044
10	L15	ICP OES	6.18	6.22	6.24	6.20	6.27	6.13	6.207	0.049
2	L16	XRF	6.25	6.29	6.26	6.17	6.24	6.21	6.237	0.042
18	L17	ICP OES	6.30	6.68	6.17	6.27	6.42	6.32	6.360	0.176
6	L18	ICP OES	6.380	6.350	6.400	6.360	6.370	6.350	6.368	0.019
16	L19	ICP OES	7.33	7.30	7.32	7.31			7.315	0.013

Diagram of means and 95% confidence intervals (to Tab. A2.10.1)



Tab. A2.10.2: Results of statistical tests (yttrium)

Number of data sets	19
Snedecor-F-Test and Bartlett-Test	Pooling not allowed
Dixon (α = 0.05)	Lab. 16
Dixon (α = 0.01)	Lab. 16
Nalimov (α = 0.05)	Lab. 16
Nalimov (α = 0.01)	Lab. 16
Grubbs (α = 0.05)	Lab. 16
Grubbs (α = 0.01)	Lab. 16
Grubbs Pair (α = 0.05)	---
Grubbs Pair (α = 0.01)	---
Cochran (α = 0.05)	Labs. 3, 4, 14, 18
Cochran (α = 0.01)	Labs. 3, 4, 14, 18
Kolmogorov-Smirnov-Lilliefors Test (α = 0.05)	Distribution: not normal
Kolmogorov-Smirnov-Lilliefors Test (α = 0.01)	Distribution: not normal
Skewness & Kurtosis Test (α = 0.05)	Distribution: not normal
Skewness & Kurtosis Test (α = 0.01)	Distribution: not normal

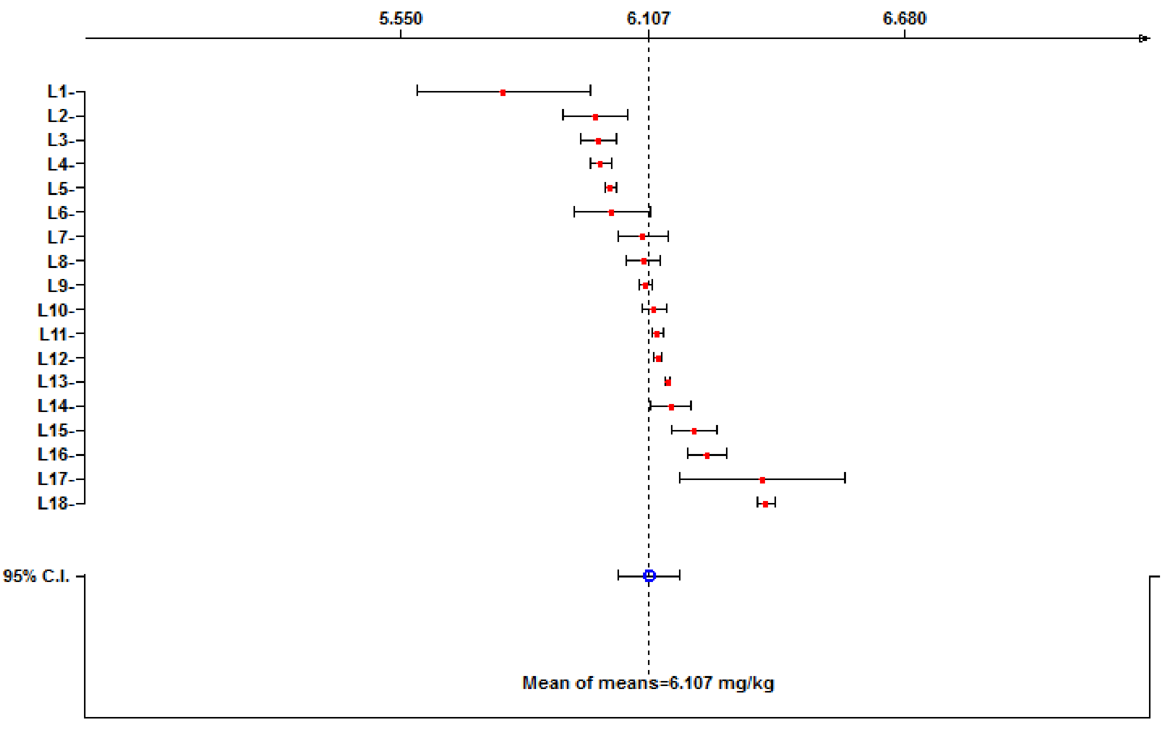
The results of Laboratory 16 were removed.

Tab. A2.10.3: Results of statistical tests (yttrium, without removed result of Lab. 16)

Number of data sets	18
Snedecor-F-Test and Bartlett-Test	Pooling not allowed
Dixon (α = 0.05)	---
Dixon (α = 0.01)	---
Nalimov (α = 0.05)	Lab. 6, 14
Nalimov (α = 0.01)	---
Grubbs (α = 0.05)	---
Grubbs (α = 0.01)	---
Grubbs Pair (α = 0.05)	---
Grubbs Pair (α = 0.01)	---
Cochran (α = 0.05)	Labs. 3, 4, 14, 18
Cochran (α = 0.01)	Labs. 3, 14, 18
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

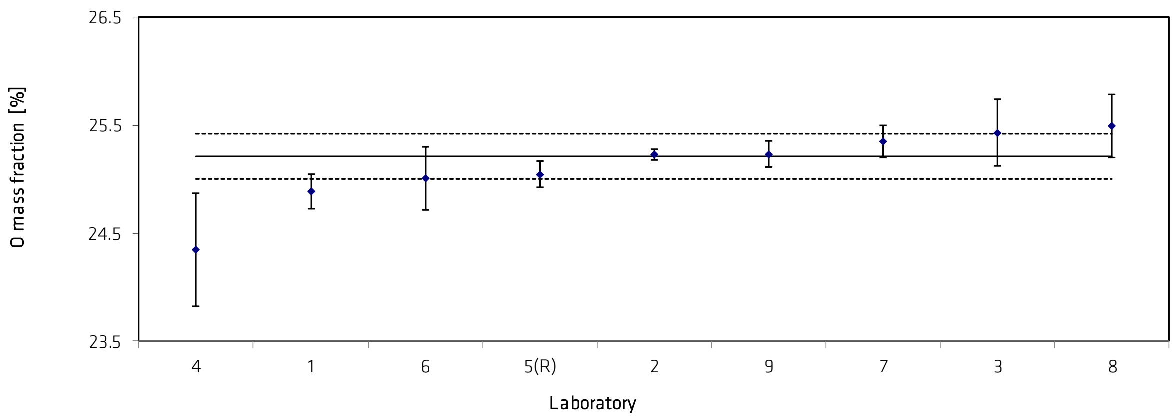
The results of Laboratories 6 and 14 were not removed.

Diagram of accepted means and 95% confidence intervals (yttrium)



Tab. A2.11: Results of oxygen determination (values in %)

Lab./Meth.	4	1	6	5(R)	2	9	7	3	8		
M_i [%]	23.30	24.73	24.97	24.90	25.22	25.03	25.58	25.37	25.14		*n*
	24.60	24.72	25.22	24.90	25.27	25.34	25.30	25.06	25.93		9
	24.50	24.83	25.29	25.20	25.25	25.22	25.43	25.67	25.54		
	24.40	25.07	24.56	25.10	25.20	25.34	25.41	25.41	25.36		
	24.70	25.08	24.77	25.10	25.29	25.31	25.25	25.21	25.30		
	24.60	24.91	25.24	25.10	25.15	25.18	25.15	25.90	25.70		
M [%]	24.35	24.89	25.01	25.05	25.23	25.24	25.35	25.44	25.50		25.21
s [%]	0.52	0.16	0.30	0.12	0.05	0.12	0.15	0.31	0.29	s_M [%]	0.21
										$\bar{s}_i$ [%]	0.20843
s_{rel}	0.02154	0.00640	0.01187	0.00489	0.00202	0.00484	0.00600	0.01206	0.01139		0.00851
	ZrO2	ZrO2	Fe2O3	ZrO2	ZrO2	ZrO2	ZrO2 (Alpha)	JK 47	ZrO2		



Tab. A2.12: Results of nitrogen determination (values in %)

Lab./Meth.	6	7	5	1	9	4	3	8		
M_i [%]	0.0692	0.0724	0.08140	0.0905	0.0876	0.0963	0.1156	0.1211		*n*
	0.0643	0.0737	0.08190	0.0802	0.0932	0.0988	0.1152	0.1181		8
	0.0668	0.0642	0.08140	0.0821	0.0933	0.0993	0.1158	0.1226		
	0.0654	0.0729	0.08150	0.0845	0.0923	0.1020	0.1172	0.1121		
	0.0649	0.0700	0.08090	0.0839	0.0947	0.0898	0.1154	0.1347		
	0.0669	0.0838	0.08120	0.0819	0.0908	0.0866	0.1165	0.1205		
	0.0693									
M [%]	**0.0667**	**0.0728**	**0.0814**	**0.0838**	**0.0920**	**0.0955**	**0.1160**	**0.1215**		**0.0912**
s [%]	0.0020	0.0064	0.0003	0.0036	0.0025	0.0060	0.0008	0.0074	s_M [%]	0.0194
									$\bar{s}_i$ [%]	0.00441
s_{rel}	0.02978	0.08754	0.00407	0.04304	0.02712	0.06283	0.00648	0.06123		0.21288
	N2	N2	Stahl-ZRM	Stahl-ZRM	KNO3	ZrO2	N2	Eltra-RM