

DLA
Dienstleistung
Lebensmittel
Analytik GbR

Evaluation Report
proficiency test

DLA 58/2016

Heavy Metals and Trace Elements
in Animal Food Supplement

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1. Introduction

The participation in proficiency testing schemes is an essential element of the quality-management-system of every laboratory testing food and feed, cosmetics and food contact materials. The implementation of proficiency tests enables the participating laboratories to prove their own analytical competence under realistic conditions. At the same time they receive valuable data regarding the verification and/or validation of the particular testing method [1, 5].

The purpose of DLA is to offer proficiency tests for selected parameters in concentrations with practical relevance.

Realisation and evaluation of the present proficiency test follows the technical requirements of DIN EN ISO/IEC 17043 (2010) and DIN ISO 13528:2009 / ISO 13528:2015 [2, 3].

2. Realisation

2.1 Test material

The test material is an common in commerce animal food supplement green lipped mussel powder (*Perna canaliculus*) with an addition of fish powder (coalfish / saithe, *Pollachius virens*). The raw materials were sieved and homogenized. The composition is given in Table 1 below.

Afterwards the samples were portioned to approximately 8 g into metallised PET film bags and chronologically numbered.

Table 1: Composition of DLA-Samples

Ingredients	Content
Green Lipped Mussel Powder Ingredients: Green lipped mussel (<i>Perna Canaliculus</i>) Nutrients per 100 g: Protein 53 g	57,6 g/100g
Fish-Powder Ingredients: Coalfish (<i>Pollachius virens</i>), cooked, dried, ground Nutrients per 100 g: Protein 87 g, salt 2,5 g, fat 5,0 g, carbohydrates 0 g	42,4 g/100g

2.1.1 Homogeneity

The **homogeneity of bottled numbered DLA-samples** was checked by 5fold determination of copper by ICP-OES (VDLUFA III, 10.8.2). The repeatability standard deviation of 6,12 % is in the range of the repeatability standard deviation of comparable methods (e.g. the German official method ASU §64 L 00.00-144, s. 3.6.2). The results of the homogeneity test are given in the documentation.

The calculation of the **repeatability standard deviation S_r of the participants** was also used as an indicator of homogeneity. For 12 of 17 elements it is < 5,0% and for all elements < 10%. Therefore these repeatability standard deviations are similar to precision data of the referring standardized methods (e.g. ASU §64 L 00.00-144, s. 3.6.2) (see Tab. 3) [16-26]. The repeatability standard deviations of the participants' results are given in the tables of statistic data (see 4.1 to 4.20).

Furthermore, the homogeneity was characterized by the **trend line function of participants' results for chronological bottled single samples**. The maximum deviations from the mean value of the trend lines for Al and Pb were below 30% of the target standard deviations σ_{pt}' and σ_{pt} , respectively (s. 5.2 homogeneity) and can therefore be regarded as low.

If the criteria for sufficient homogeneity of the test material are not fulfilled on a particular parameter, the impact on the target standard deviation is checked and optionally the evaluation of the results of the participants will be done using the z'-score considering the standard uncertainty of the assigned value (see 3.8 and 3.11) [3].

2.2 Sample shipment and information to the test

Two portions of test material were sent to every participating laboratory in the 17th week of 2016. The testing method was optional. The tests should be finished at 9th June 2016 the latest.

With the cover letter along with the sample shipment the following information was given to participants:

The two portions contain identical samples of a food supplement with green-lipped mussel powder and fish powder.

In general we recommend to homogenize a representative sample amount before analysis according to good laboratory practice, especially in case of low sample weights.

Note: Please indicate the applied hydrolization method and especially the hydrolization solutions, to ensure better comparability of results. It is also possible to submit several results for one element obtained by different hydrolization methods.

2.3 Submission of results

The participants submitted their results in standard forms, which have been handed out with the samples (by email).

The finally calculated concentrations of the parameter as average of duplicate determinations of both numbered samples were used for the statistical evaluation. For the calculation of the repeatability- and reproducibility standard deviation the single values of the double determination were used.

Queried and documented were single results, recovery and the used testing methods.

In case participants submitted several results for the same parameter obtained by different methods these results were evaluated with the same evaluation number with a letter as a suffix and indication of the related method.

From the 15 participants one participant submitted the results delayed in consultation with DLA. All other participants submitted the result in time.

3. Evaluation

3.1 Consensus value from participants (assigned value)

The robust mean of the submitted results was used as assigned value (X_{pt}) („consensus value from participants“) providing a normal distribution. The calculation was done according to algorithm A as described in annex C of ISO 13528 [3].

The condition is that the majority of the participants' results show a normal distribution or are distributed unimodal and symmetrically. To this end, an examination of the distribution is carried out, inter alia, using the kernel density estimate [3, 12].

In case there are indications for sources of higher variability such as a bimodal distribution of results, a cause analysis is performed. Frequently different analytical methods may cause an anomaly in results' distribution. If this is the case, separate evaluations with own assigned values (X_{pti}) are made whenever possible.

The statistical evaluation is carried out for all the parameters for a minimum of 7 values are present.

The actual measurement results will be drafted. Individual results, which are outside the specified measurement range of the participating laboratory (for example with the result > 25 mg/kg or $< 2,5$ mg/kg) or the indicating "0" will not be considered for the statistic evaluation [3].

3.2 Robust standard deviation

For comparison to the target standard deviation σ_{pt} (standard deviation for proficiency assessment) a robust standard deviation (S^*) was calculated. The calculation was done according to algorithm A as described in annex C of ISO 13528 [3].

3.3 Repeatability standard deviation

The repeatability standard deviation S_r is based on the laboratory's standard deviation of (outlier free) individual participant results, each under repeatability conditions, that means analyses was performed on the same sample by the same operator using the same equipment in the same laboratory within a short time. It characterizes the mean deviation of the results within the laboratories [3] and is used by DLA as an indication of the homogeneity of the sample material.

In case single results from participants are available the calculation of the repeatability standard deviation S_r , also known as standard deviation within laboratories S_w , is performed by: [3, 4].

The relative repeatability standard deviation as a percentage of the mean value is indicated as coefficient of variation CV_r in the table of statistical characteristics in the results section in case single results from participants are available.

3.4 Reproducibility standard deviation

The reproducibility standard deviation S_R represents a inter-laboratory estimate of the standard deviation for the determination of each parameter on the bases of (outlier free) individual participant results. It takes into account both the repeatability standard deviation S_r and the within-laboratory standard deviation S_s . Reproducibility standard deviations of PT's may differ from reproducibility standard deviations of ring trials, because the participating laboratories of a PT generally use different internal conditions and methods for determining the measured values.

In the present evaluation, the specification of the reproducibility standard deviation, therefore, does not refer to a specific method, but characterizes approximately the comparability of results between the laboratories, assumed the effect of homogeneity and stability of the sample are negligible.

In case single results from participants are available the calculation of the reproducibility standard deviation S_R is performed by: [3, 4].

The relative reproducibility standard deviation as a percentage of the mean value is indicated as coefficient of variation CV_R in the table of statistical characteristics in the results section in case single results from participants are available. Its meaning is explained in more detail in 3.9.

3.5 Exclusion of results and outliers

Before statistical evaluation obvious blunders, such as those with incorrect units, decimal point errors, and results for a another proficiency test item can be removed from the data set [2]. All results should be given at least with 2 significant digits. Specifying 3 significant digits is usually sufficient.

Results obtained by different analytical methods causing an increased variability and/or a bi- or multimodal distribution of results, are treated separately or could be excluded in case of too few numbers of results. For this results are checked by kernel density estimation [3, 12].

Results are identified as outliers by the use of robust statistics. If a value deviates from the robust mean by more than 3 times the robust standard deviation, it is classified as an outlier [3]. Detected outliers are stated for information only, when z-score are < -2 or > 2 . Due to the use of robust statistics outliers are not excluded, provided that no other reasons are present [3].

3.6 Target standard deviation (for proficiency assessment)

The target standard deviation of the assigned value σ_{pt} (= standard deviation for proficiency assessment) can be determined according to the following methods.

If an acceptable quotient S^*/σ_{pt} is present, the target standard deviation of the general model by Horwitz is preferably used for the proficiency assessment. It is usually suitable for evaluation of interlaboratory studies, where different analytical methods are applied by the participants. On the other hand the target standard deviation from the evaluation of precision data of a precision experiment is derived from collaborative studies with specified analytical methods.

In cases where both above-mentioned models are not suitable, the target standard deviation is determined based on values by perception, see under 3.6.3.

For information the z-scores of both models are given in the evaluation, if available.

In the present PT for valuation of all elements the target standard deviation according to the general model of Horwitz was applied (see 3.4.1).

Additionally for Al, Ba and I the standard uncertainty was considered by valuating with z'-scores (see 3.6).

3.6.1 General model (Horwitz)

Based on statistical characteristics obtained in numerous PTs for different parameters and methods Horwitz has derived a general model for estimating the reproducibility standard deviation σ_R [6]. Later the model was modified by Thompson for certain concentration ranges [10]. The reproducibility standard deviation σ_R can be applied as the relative target standard deviation σ_{pt} in % of the assigned values and calculated according to the following equations [3]. For this the assigned value X_{pt} is used for the concentration c .

Equations	Range of concentrations	corresponds to
$\sigma_R = 0,22c$	$c < 1,2 \times 10^{-7}$	$< 120 \mu\text{g}/\text{kg}$
$\sigma_R = 0,02c^{0,8495}$	$1,2 \times 10^{-7} \leq c \leq 0,138$	$\geq 120 \mu\text{g}/\text{kg}$
$\sigma_R = 0,01c^{0,5}$	$c > 0,138$	$> 13,8 \text{ g}/100\text{g}$

with c = mass content of analyte (as relative size, e.g. 1 mg/kg = 1 ppm = 10^{-6} kg/kg)

3.6.2 Value by precision experiment

Using the reproducibility standard deviation σ_R and the repeatability standard deviation σ_r of a precision experiment (collaborative trial or proficiency test) the target standard deviation σ_{pt} can be derived considering the number of replicate measurements m of participants in the present PT [3]:

$$\sigma_{pt} = \sqrt{\sigma_R^2 - \sigma_r^2 (m-1/m)}$$

The values given in Table 2 relative repeatability standard deviation (RSD_r) and relative reproducibility standard deviation (RSD_R) were determined in collaborative trials using the specified methods.

The in the table indicated resulting target standard deviations σ_{pt} were used for evaluation of the results.

For information the target standard deviations according to Horwitz are given additionally.

3.6.3 Value by perception

The target standard deviation for proficiency assessment can be set at a value that corresponds to the level of performance that the coordinator would wish laboratories to be able to achieve [3].

For the present evaluation the target standard deviation according to 3.6.1 was regarded suitable partly using the z'-scores.

Table 2: Relative repeatability standard deviations (RSD_r) and relative reproducibility standard deviations (RSD_R) from precision experiments and resulting target standard deviations σ_{pt} [16-26]

Parameter	Matrix	Mean values	RSD_r	RSD_R	σ_{pt}	Method / Literature
Al	cocoa powder	205	3,25%	5,83%	5,36%	ICP-MS [16]
	cocoa powder	210	1,91%	8,71%	8,61% ¹	ICP-OES [17]
As	fish homogenate	1,6	4,6%	8,8%	8,18%	ICP-MS [18]
	mussel	9,3	4,5%	13%	12,6% ¹	ICP-MS [18]
Pb	fish homogenate	2,1	5,0%	8%	7,18%	ICP-MS [18]
	mussel	2,5	13%	16%	13,1% ¹	ICP-MS [18]
Cd	fish homogenate	0,87	7,3%	11%	9,71%	ICP-MS [18]
	mussel	1,7	3,9%	9,5%	9,09% ¹	ICP-MS [18]
Cr	infant food	0,17	7,3%	19%	18,3% ¹	GF-AAS [20]
	rice powder	0,11	19,2%	35%	32,3%	GF-AAS [20]
Cu	lobster	16,40	5,72%	6,82%	5,49% ¹	ICP-OES [22]
	infant food soya	4,51	4,30%	11,06%	10,6%	ICP-OES [22]
Fe	lobster	12,1	6,45%	8,59%	7,28% ¹	ICP-OES [22]
	infant food soya	77	2,75%	6,98%	6,70%	ICP-OES [22]
I	cod fish muscle	4,15	0,7%	8,9%	8,89% ¹	ICP-MS (16)
	soya food	1,26	3,7%	6,7%	6,17%	ICP-MS (16)
Mn	lobster	1,20	4,74%	7,95%	7,21% ¹	ICP-OES [22]
	infant food soya	2,19	4,67%	13,7%	13,3%	ICP-OES [22]
Mo	infant food	0,50	6,6%	21%	20,5% ¹	GF-AAS [20]
	rice powder	0,56	8,7%	20%	19,0%	GF-AAS [20]
Se	wolffish	1,797	9,85%	10,1%	7,31% ¹	AAS [21]
	rice	0,374	2,41%	11,8%	11,7%	AAS [21]
Zn	lobster	13,9	4,63%	7,90%	7,19% ¹	ICP-OES (22)
	infant food soya	43,5	2,60%	6,89%	6,64%	ICP-OES (22)

¹ used in evaluation (s. chapter 4)

3.7 z-Score

To assess the results of the participants the z-score is used. It indicates about which multiple of the target standard deviation (σ_{pt}) the result (x_i) of the participant is deviating from the assigned value (X_{pt}) [3].

Participants' z-scores are derived from:

$$z_i = \frac{(x_i - X_{pt})}{\sigma_{pt}}$$

The requirements for the analytical performance are generally considered as fulfilled if

$$-2 \leq z \leq 2 .$$

3.7.1 Warning and action signals

In accordance with the norm ISO 13528 it is recommended that a result that gives rise to a z-score above 3,0 or below -3,0, shall be considered to give an "action signal" [3]. Likewise, a z-score above 2,0 or below -2,0 shall be considered to give a "warning signal". A single "action signal", or "warning signal" in two successive PT-rounds, shall be taken as evidence that an anomaly has occurred which requires investigation. For example a fault isolation or a root cause analysis through the examination of transmission error or an error in the calculation, in the trueness and precision must be performed and if necessary appropriate corrective measures should be applied [3].

In the figures of z-scores DLA gives the limits of warning and action signals as yellow and red lines respectively. According to ISO 13528 the signals are valid only in case of a number of ≥ 10 results [3].

3.8 z'-Score

The z'-score can be used for the valuation of the results of the participants, in cases the standard uncertainty has to be considered (s. 3.8). The z'-score represents the relation of the deviation of the result (x) of the participant from the respective consensus value (X) to the square root of quadrat sum of the target standard deviation ($\hat{\sigma}$) and the standard uncertainty ($U_{x_{pt}}$) [3].

The calculation is performed by:

$$z'_i = \frac{x_i - x_{pt}}{\sqrt{\sigma_{pt}^2 + u_{(x_{pt})}^2}}$$

If carried out an evaluation of the results by means of z 'score, we have defined below the expression in the denominator as a target standard deviation σ_{pt}' .

The requirements for the analytical performance are generally considered as fulfilled if

$$-2 \leq z' \leq 2 .$$

For warning and action signals see 3.7.1.

3.9 Reproducibility coefficient of variation (CV_R)

The coefficient of variation (CV_R) of the reproducibility (= relative reproducibility standard deviation) is calculated from the standard deviation and the mean as follows [4, 13]:

$$CV_R = \frac{S_R * 100}{X}$$

In contrast to the standard deviation as a measure of the absolute variability the CV_R gives the relative variability within a data region. While a low CV_R, e.g. <5-10% can be taken as evidence for a homogeneous set of results, a CV_R of more than 50% indicates a "strong inhomogeneity of statistical mass", so that the suitability for certain applications such as the assessment of exceeded maximum values or the performance evaluation of the participants possibly can not be done [3].

3.10 Quotient S^*/σ_{pt}

Following the HorRat-value the results of a proficiency-test (PT) can be considered convincing, if the quotient of robust standard deviation S^* and target standard deviation σ_{pt} does not exceed the value of 2. A value > 2 means an insufficient precision, i.e. the analytical method is too variable, or the variation between the test participants is higher than estimated. Thus the comparability of the results is not given [3].

3.11 Standard uncertainty

The consensus value has a standard uncertainty $U(X_{pt})$ that depends on the analytical method, differences between the analytical methods used, the test material, the number of participant laboratories (P) and perhaps on other factors. The standard uncertainty of the assigned value ($U(X_{pt})$) for this PT is calculated as follows [3]:

$$u_{(x_{pt})} = 1,25 \times \frac{s^*}{\sqrt{p}}$$

If $U(X_{pt}) \leq 0,3 \sigma_{pt}$ the standard uncertainty of the consensus value needs not to be included in the interpretation of the results of the PT [3]. A clear exceeded the value of 0,3 is an indication that the target standard deviation was possibly set too low for the standard uncertainty of the assigned value.

The quotient $U(X_{pt})/\sigma_{pt}$ is reported in the characteristics of the test.

4. Results

Comments to the distribution of the results:

The kernel density plots showed for all elements except barium nearly a normal distribution of results (figures see documentation 5.3). Partly slight shoulders and separate smaller peaks can be seen, which are due to individual values and outliers.

In the case of barium, a distribution of results with two peaks can be seen. However, the information provided by the participants on the methods gave no obvious indications of such an array of results. Therefore, the statistical analysis of all results for barium was carried out together taking into account the standard uncertainty by z'-score.

Comments to the statistic data:

For Rb, Ti and Y there were < 7 results, therefore no statistical evaluation could be done.

The target standard deviation was calculated for all other elements according to the model of Horwitz.

For Al, Ba and I the distribution of results showed an increased variability. The quotients S^*/σ_{pt} were clearly above 2,0. Valuation of the three elements was done considering the standard uncertainty by z'-score. The quotients S^*/σ_{pt} were in the range of 2,0 then (see table 4).

For the other elements the distribution of results showed a normal to low variability. The quotients S^*/σ_{pt} were all below 2,0, in part below 1,0 (see table 4).

The robust standard deviation as well as the repeatability and reproducibility standard deviations were in the range of established values for the applied methods (see 3.6.2).

The comparability of results is given.

The quotient $U(x_{pt})/\sigma_{pt}$ was low for As, Cd, Co, Cu, Mn, Mo and Pb (< 0,3). For Cr, Fe, Ni, Li and Zn the quotients were (slightly) increased in the range of 0,33 to 0,42 and for Se and V 0,58 and 0,56.

67% to 100% of results were in the target range.

Table 4: Selected statistic data of the present PT
(SD = standard deviation, CV = coefficient of variation)

Parameter	Matrix (Powder)	robust Mean [mg/kg]	rob. SD (S*) [mg/kg]	rel. SD (CV _{S*}) [%]	Quotient S*/ σ_{pt}	DLA- Report
Al	mussel-fish	444	143	32,2%	2,2 ¹	DLA 58/2016
As	mussel-fish	6,52	0,735	11,3%	0,93	DLA 58/2016
Ba	mussel-fish	2,35	0,832	35,5%	1,8 ¹	DLA 58/2016
Cd	mussel-fish	1,03	0,0616	5,97%	0,37	DLA 58/2016
Cr	mussel-fish	1,23	0,266	21,6%	1,4	DLA 58/2016
Co	mussel-fish	0,586	0,0347	5,91%	0,34	DLA 58/2016
Cu	mussel-fish	5,75	0,439	7,63%	0,62	DLA 58/2016
Fe	mussel-fish	305	22,1	7,24%	1,1	DLA 58/2016
I	mussel-fish	9,81	3,41	34,8%	1,9 ¹	DLA 58/2016
Li	mussel-fish	1,51	0,204	13,6%	0,90	DLA 58/2016
Mn	mussel-fish	8,79	0,696	7,93%	0,69	DLA 58/2016
Mo	mussel-fish	0,536	0,0400	7,45%	0,42	DLA 58/2016
Ni	mussel-fish	1,40	0,232	16,6%	1,1	DLA 58/2016
Pb	mussel-fish	0,311	0,0525	16,9%	0,89	DLA 58/2016
Rb	mussel-fish	**	-	-	-	DLA 58/2016
Se	mussel-fish	1,86	0,481	25,8%	1,8	DLA 58/2016
Ti	mussel-fish	**	-	-	-	DLA 58/2016
V	mussel-fish	1,40	0,318	22,7%	1,5	DLA 58/2016
Y	mussel-fish	**	-	-	-	DLA 58/2016
Zn	mussel-fish	51,0	5,17	10,2%	1,1	DLA 58/2016

¹ with target standard deviation σ_{pt}

** no statistical evaluation (< 7 results)

All following tables are anonymized. With the delivering of the evaluation-report the participants are informed about their individual evaluation-number.

In the first table the characteristics are listed:

Statistic Data
<i>Number of results</i>
<i>Number of outliers</i>
Mean
Median
Robust mean (X_{pt})
Robust standard deviation (S^*)
<i>Target range:</i>
Target standard deviation σ_{pt} or σ_{pt}'
Target standard deviation for information
lower limit of target range $(X_{pt} - 2\sigma_{pt})$ or $(X_{pt} - 2\sigma_{pt}')$ *
upper limit of target range $(X_{pt} + 2\sigma_{pt})$ or $(X_{pt} + 2\sigma_{pt}')$ *
Quotient S^*/σ_{pt} or S^*/σ_{pt}'
Standard uncertainty $U(X_{pt})$
Quotient $U(X_{pt})/\sigma_{pt}$ or $U(X_{pt})/\sigma_{pt}'$
<i>Number of results in the target range</i>
<i>Percent in the target range</i>

* Target range is calculated with z-score or z'-score

In the second table the individual results of the participating laboratories are listed:

Auswerte- nummer	Parameter [Einheit / Unit]	Abweichung	z-Score σ_{pt}	z-Score (Info)	Hinweis
Evaluation number		Deviation			Remark

4.1 Al - Aluminium in mg/kg

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	9
Number of outliers	0
Mean	441
Median	456
Robust Mean (X_{pt})	444
Robust standard deviation (S^*)	143
Number with 2 replicates	8
Repeatability SD (S_r)	14,1
Repeatability (CV_r)	3,09%
Reproducibility SD (S_R)	134
Reproducibility (CV_R)	29,4%
Target range:	
Target standard deviation σ_{pt}'	65,9
Target standard deviation (for Information)	38,2
lower limit of target range	313
upper limit of target range	576
Quotient S^*/σ_{pt}'	2,2
Standard uncertainty $U(X_{pt})$	59,5
Quotient $U(X_{pt})/\sigma_{pt}'$	0,90
Results in the target range	7
Percent in the target range	78%

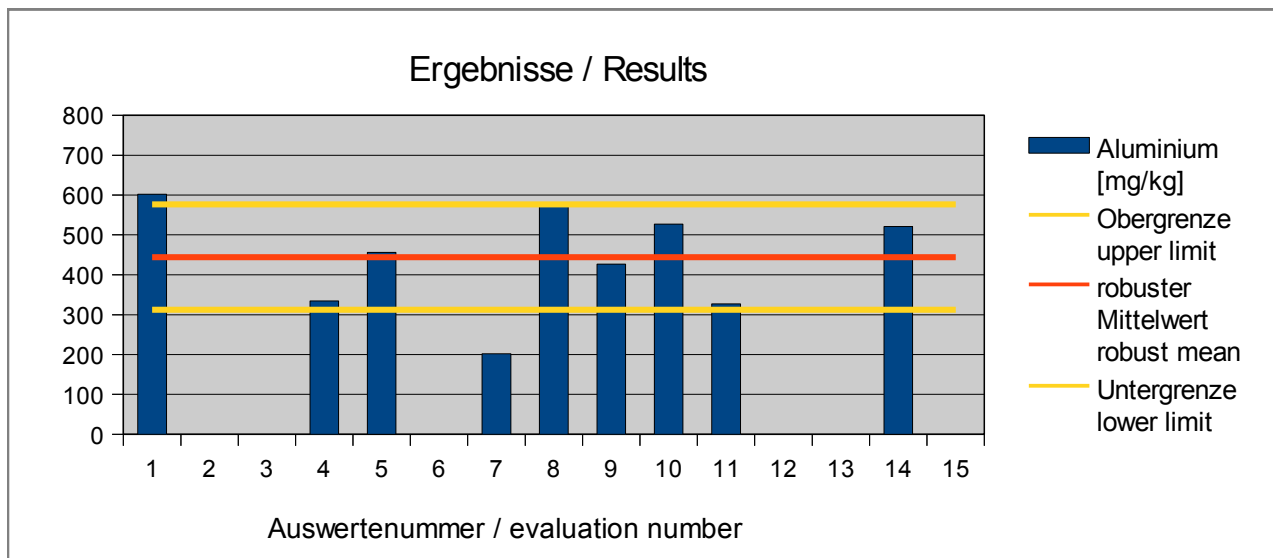


Abb. 1: Ergebnisse Aluminium / Fig. 1: Results Aluminium

Ergebnisse der Teilnehmer:
Results of Participants:

Auswertenummer	Aluminium [mg/kg]	Abweichung [mg/kg]	z'-Score (σ _{pt})	z-Score (Info)	Hinweis
Evaluation number		Deviation [mg/kg]			Remark
1	602	157,54	2,4	4,1	
2					
3					
4	334,7	-109,76	-1,7	-2,9	
5	455,95	11,49	0,2	0,3	
6					
7	202	-242,46	-3,7	-6,3	
8	575,2	130,74	2,0	3,4	
9	427	-17,46	-0,3	-0,5	
10	527	82,54	1,3	2,2	
11	326,95	-117,51	-1,8	-3,1	
12					
13					
14	521	76,54	1,2	2,0	
15					



Abb. 2: z'-Scores Aluminium
Fig. 2: z'-Scores Aluminium

4.2 As - Arsen / Arsenic in mg/kg

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	17
Number of outliers	2
Mean	5,92
Median	6,64
Robust Mean (X_{pt})	6,52
Robust standard deviation (S^*)	0,735
Number with 2 replicates	13
Repeatability SD (S_r)	0,305
Repeatability (CV_r)	4,71%
Reproducibility SD (S_R)	0,764
Reproducibility (CV_R)	11,8%
<i>Target range:</i>	
Target standard deviation σ_{pt}	0,786
Target standard deviation (for Information)	0,821
lower limit of target range	4,94
upper limit of target range	8,09
Quotient S^*/σ_{pt}	0,93
Standard uncertainty $U(X_{pt})$	0,223
Quotient $U(X_{pt})/\sigma_{pt}$	0,28
Results in the target range	14
Percent in the target range	82%

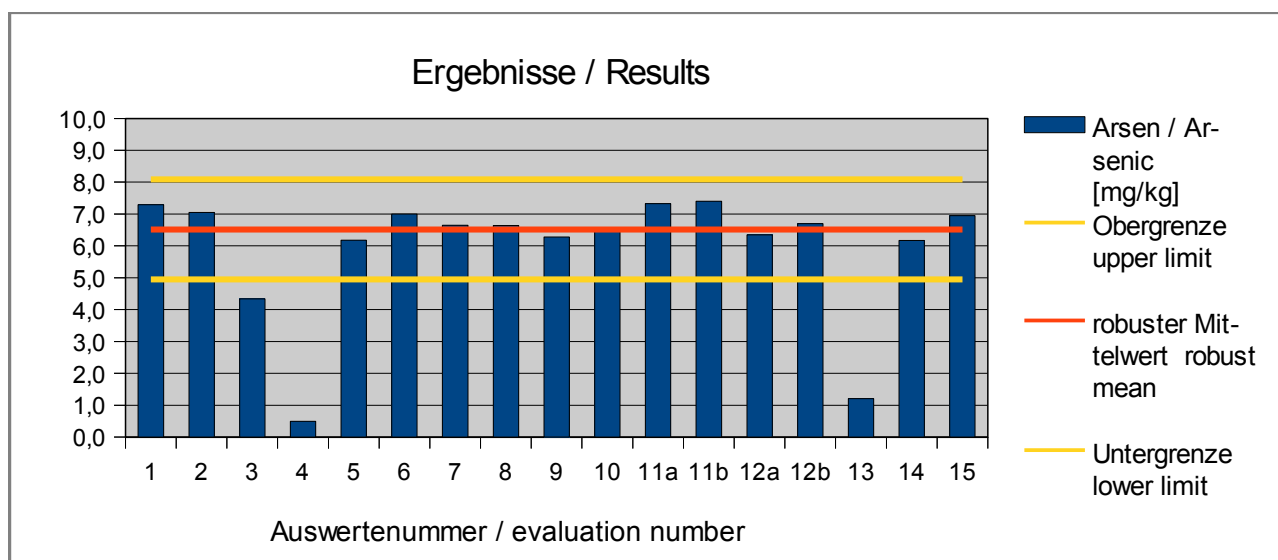


Abb. 3: Ergebnisse Arsen / **Fig. 3:** Results Arsenic

**Ergebnisse der Teilnehmer:
Results of Participants:**

Auswertenummer Evaluation number	Arsen / Arsenic [mg/kg]	Abweichung [mg/kg] Deviation [mg/kg]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	7,29	0,77	1,0	0,9	
2	7,05	0,53	0,7	0,6	
3	4,34	-2,18	-2,8	-2,7	
4	0,496	-6,02	-7,7	-7,3	Ausreisser / Outlier
5	6,1765	-0,34	-0,4	-0,4	
6	7,0	0,48	0,6	0,6	
7	6,65	0,13	0,2	0,2	
8	6,644	0,13	0,2	0,2	
9	6,28	-0,24	-0,3	-0,3	
10	6,55	0,03	0,0	0,0	
11a	7,325	0,81	1,0	1,0	
11b	7,3985	0,88	1,1	1,1	
12a	6,35	-0,17	-0,2	-0,2	
12b	6,7	0,18	0,2	0,2	
13	1,21	-5,31	-6,7	-6,5	Ausreisser / Outlier
14	6,17	-0,35	-0,4	-0,4	
15	6,946	0,43	0,5	0,5	

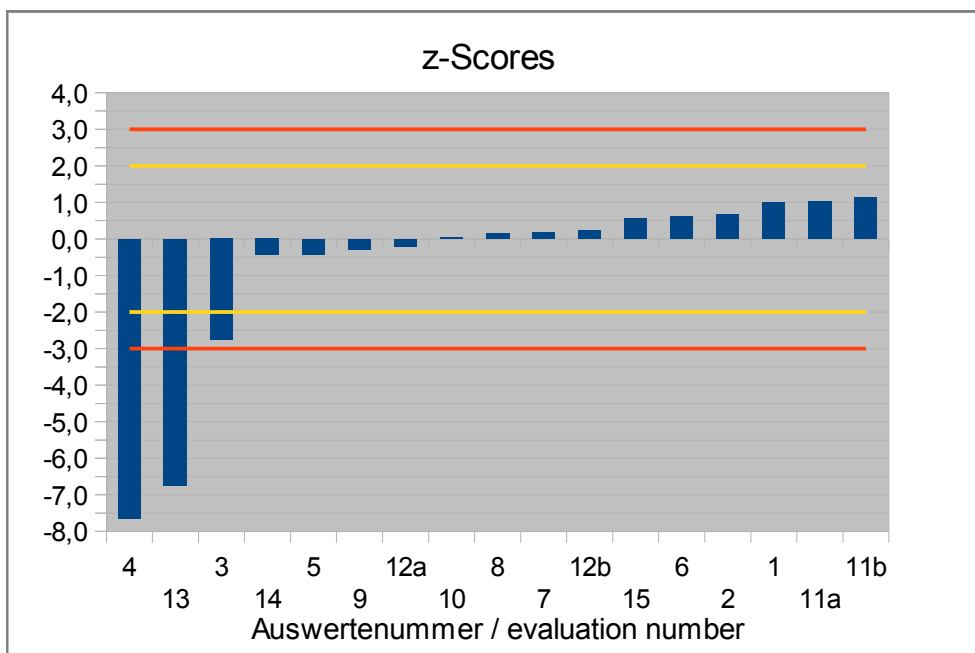


Abb. 4: Z-Scores Arsen
Fig. 4: Z-Scores Arsenic

4.3 Ba - Barium in mg/kg

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	10
Number of outliers	0
Mean	2,35
Median	2,13
Robust Mean (X_{pt})	2,35
Robust standard deviation (S^*)	0,832
Number with 2 replicates	8
Repeatability SD (S_r)	0,215
Repeatability (CV_r)	8,65%
Reproducibility SD (S_R)	0,760
Reproducibility (CV_R)	30,6%
Target range:	
Target standard deviation σ_{pt}'	0,466
lower limit of target range	1,41
upper limit of target range	3,28
Quotient S^*/σ_{pt}'	1,8
Standard uncertainty $U(X_{pt})$	0,329
Quotient $U(X_{pt})/\sigma_{pt}'$	0,71
Results in the target range	8
Percent in the target range	80%

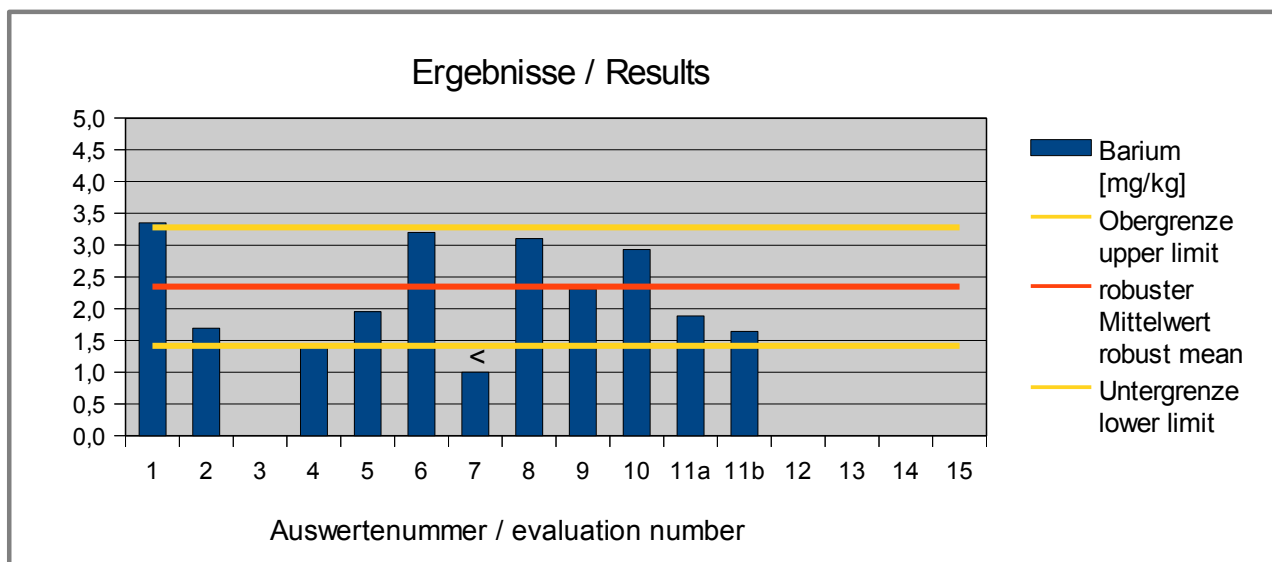


Abb. 5: Ergebnisse Barium / Fig. 5: Results Barium

**Ergebnisse der Teilnehmer:
Results of Participants:**

Auswertenummer Evaluation number	Barium [mg/kg]	Abweichung [mg/kg] Deviation [mg/kg]	z'-Score (σ_{pt})	Hinweis Remark
1	3,35	1,00	2,2	
2	1,69	-0,66	-1,4	
3				
4	1,396	-0,95	-2,0	
5	1,951	-0,39	-0,8	
6	3,2	0,85	1,8	
7	< 1			
8	3,102	0,76	1,6	
9	2,31	-0,04	-0,1	
10	2,93	0,58	1,3	
11a	1,885	-0,46	-1,0	
11b	1,641	-0,70	-1,5	
12				
13				
14				
15				

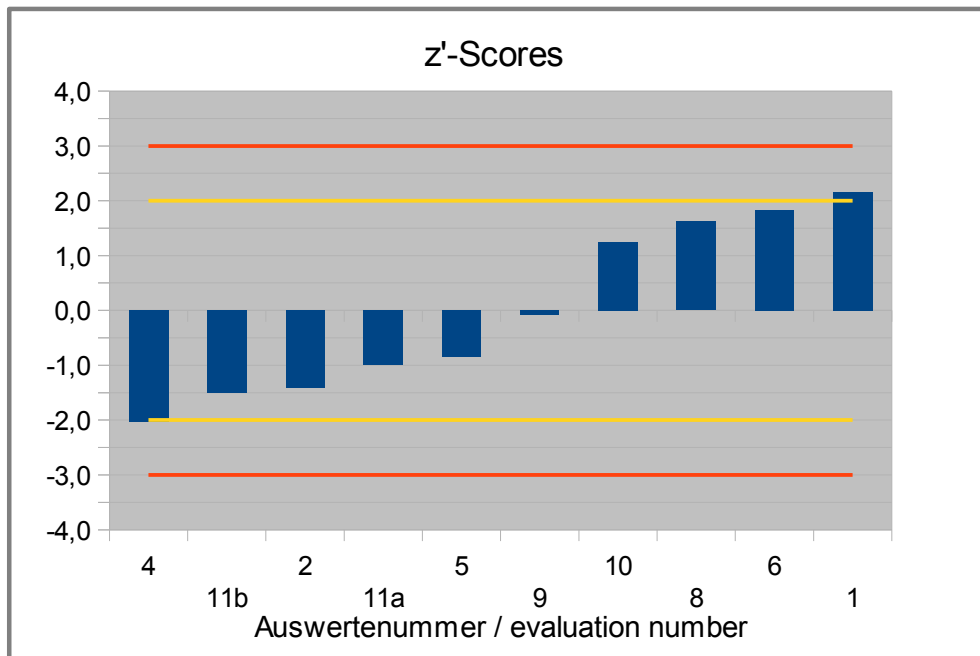


Abb. 6: z'-Scores Barium
Fig. 6: z'-Scores Barium

4.4 Cd - Cadmium in mg/kg

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	15
Number of outliers	0
Mean	1,03
Median	1,03
Robust Mean (X_{pt})	1,03
Robust standard deviation (S^*)	0,0616
Number with 2 replicates	13
Repeatability SD (S_r)	0,0268
Repeatability (CV_r)	2,63%
Reproducibility SD (S_R)	0,052
Reproducibility (CV_R)	5,13%
<i>Target range:</i>	
Target standard deviation σ_{pt}	0,164
Target standard deviation (for Information)	0,094
lower limit of target range	0,704
upper limit of target range	1,36
Quotient S^*/σ_{pt}	0,37
Standard uncertainty $U(X_{pt})$	0,0199
Quotient $U(X_{pt})/\sigma_{pt}$	0,12
Results in the target range	15
Percent in the target range	100%

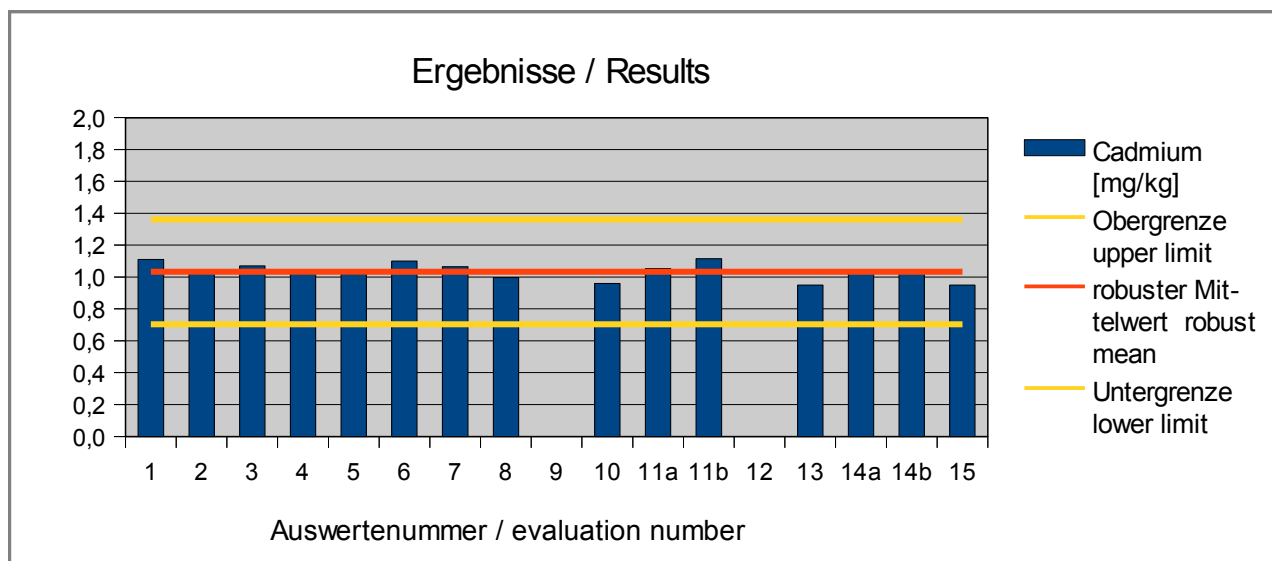


Abb. 7: Ergebnisse Cadmium / **Fig. 7:** Results Cadmium

**Ergebnisse der Teilnehmer:
Results of Participants:**

Auswertenummer Evaluation number	Cadmium [mg/kg]	Abweichung [mg/kg] Deviation [mg/kg]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	1,11	0,077	0,5	0,8	
2	1,02	-0,013	-0,1	-0,1	
3	1,07	0,037	0,2	0,4	
4	1,02	-0,013	-0,1	-0,1	
5	1,018	-0,015	-0,1	-0,2	
6	1,1	0,067	0,4	0,7	
7	1,065	0,032	0,2	0,3	
8	0,9968	-0,036	-0,2	-0,4	
9					
10	0,96	-0,073	-0,4	-0,8	
11a	1,054	0,021	0,1	0,2	
11b	1,116	0,083	0,5	0,9	
12					
13	0,95	-0,083	-0,5	-0,9	
14a	1,03	-0,003	0,0	0,0	
14b	1,03	-0,003	0,0	0,0	
15	0,949	-0,084	-0,5	-0,9	

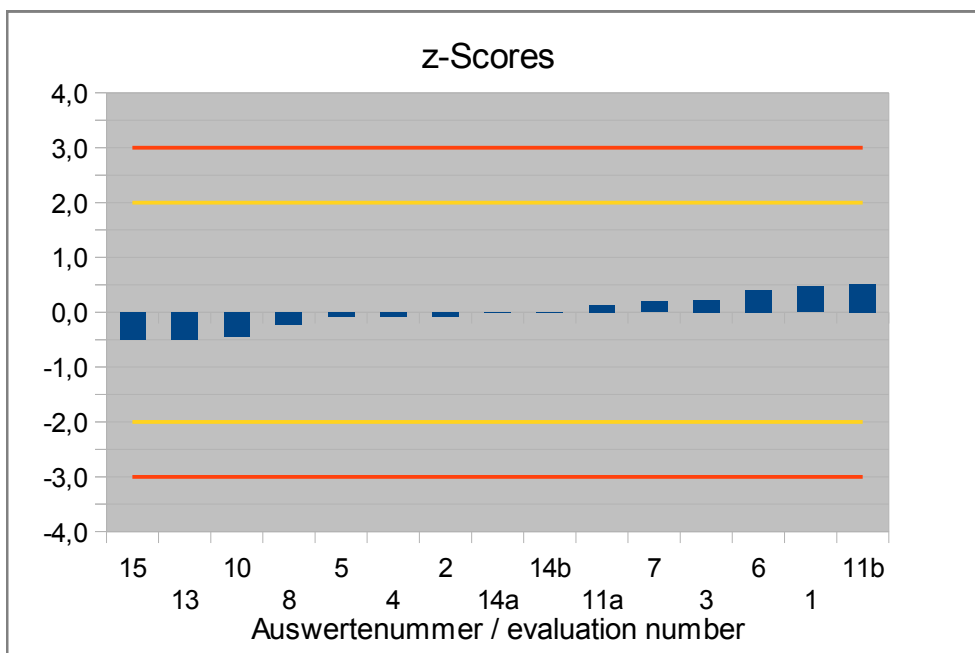


Abb. 8: Z-Scores Cadmium

Fig. 8: Z-Scores Cadmium

4.5 Cr - Chrom / Chromium in mg/kg

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	17
Number of outliers	0
Mean	1,22
Median	1,23
Robust Mean (X_{pt})	1,23
Robust standard deviation (S^*)	0,266
Number with 2 replicates	15
Repeatability SD (S_r)	0,0432
Repeatability (CV_r)	3,51%
Reproducibility SD (S_R)	0,323
Reproducibility (CV_R)	26,2%
Target range:	
Target standard deviation σ_{pt}	0,191
Target standard deviation (for Information)	0,225
lower limit of target range	0,849
upper limit of target range	1,61
Quotient S^*/σ_{pt}	1,4
Standard uncertainty $U(X_{pt})$	0,0806
Quotient $U(X_{pt})/\sigma_{pt}$	0,42
Results in the target range	13
Percent in the target range	76%

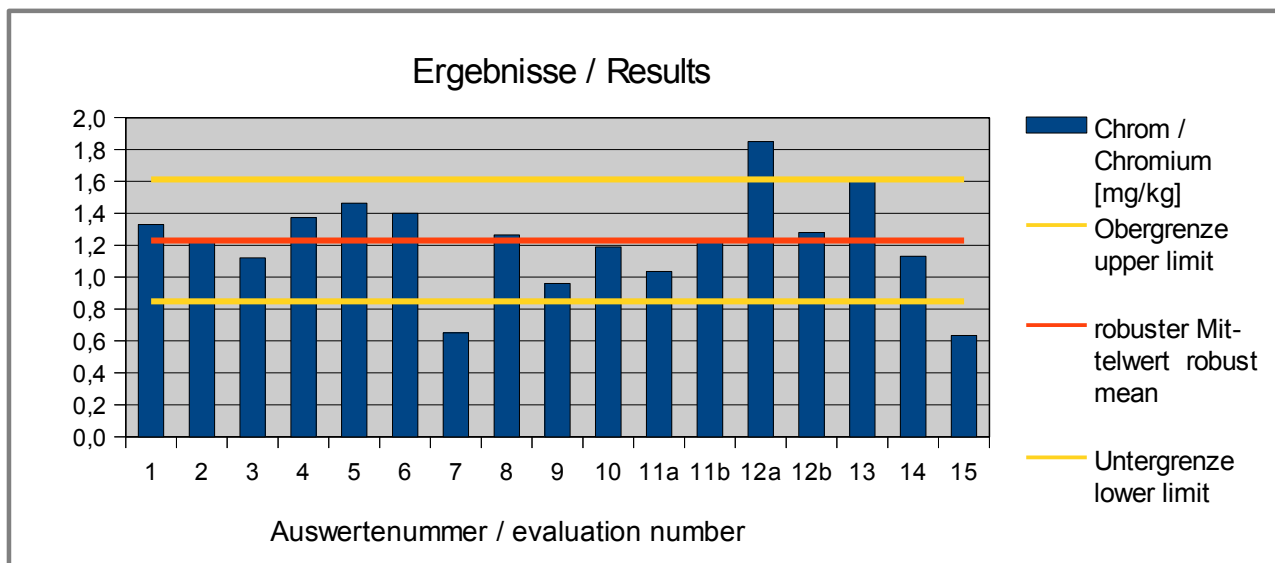


Abb. 9: Ergebnisse Chrom / Fig. 9: Results Chromium

**Ergebnisse der Teilnehmer:
Results of Participants:**

Auswertenummer Evaluation number	Chrom / Chromium [mg/kg]	Abweichung [mg/kg] Deviation [mg/kg]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	1,33	0,100	0,5	0,4	
2	1,22	-0,010	-0,1	0,0	
3	1,12	-0,110	-0,6	-0,5	
4	1,374	0,144	0,8	0,6	
5	1,4635	0,233	1,2	1,0	
6	1,4	0,170	0,9	0,8	
7	0,651	-0,579	-3,0	-2,6	
8	1,265	0,035	0,2	0,2	
9	0,96	-0,270	-1,4	-1,2	
10	1,19	-0,040	-0,2	-0,2	
11a	1,0365	-0,194	-1,0	-0,9	
11b	1,231	0,001	0,0	0,0	
12a	1,85	0,620	3,2	2,8	
12b	1,28	0,050	0,3	0,2	
13	1,62	0,390	2,0	1,7	
14	1,13	-0,100	-0,5	-0,4	
15	0,634	-0,60	-3,1	-2,7	

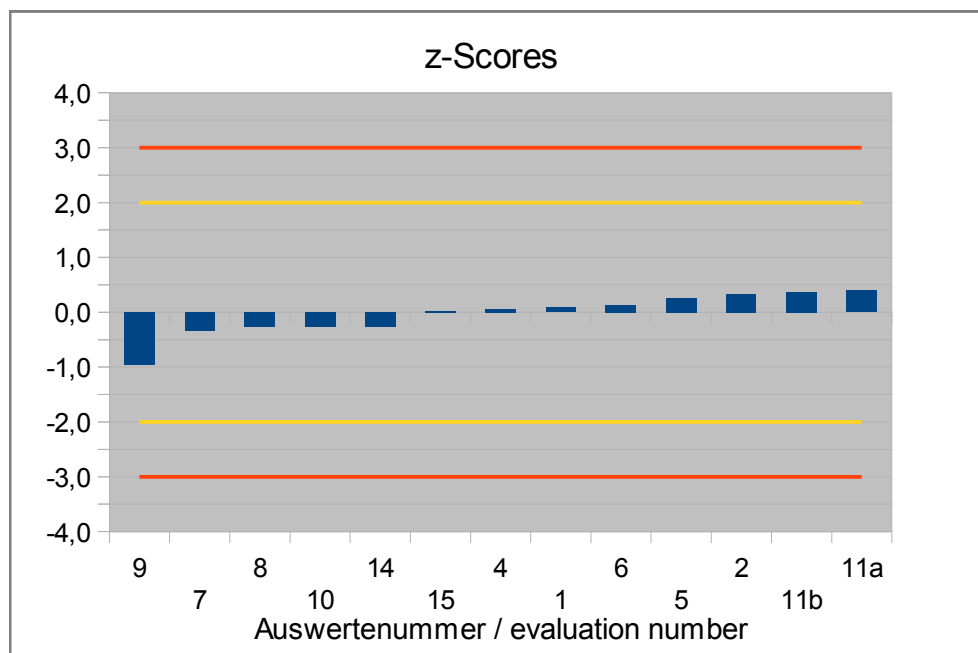


Abb. 10: Z-Scores Chrom
Fig. 10: Z-Scores Chromium

4.6 Co - Kobalt / Cobalt in mg/kg

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	13
Number of outliers	0
Mean	0,583
Median	0,592
Robust Mean (X_{pt})	0,586
Robust standard deviation (S^*)	0,0347
Number with 2 replicates	11
Repeatability SD (S_r)	0,0104
Repeatability (CV_r)	1,81%
Reproducibility SD (S_R)	0,0379
Reproducibility (CV_R)	6,60%
Target range:	
Target standard deviation σ_{pt}	0,102
lower limit of target range	0,383
upper limit of target range	0,790
Quotient S^*/σ_{pt}	0,34
Standard uncertainty $U(X_{pt})$	0,0120
Quotient $U(X_{pt})/\sigma_{pt}$	0,12
Results in the target range	13
Percent in the target range	100%

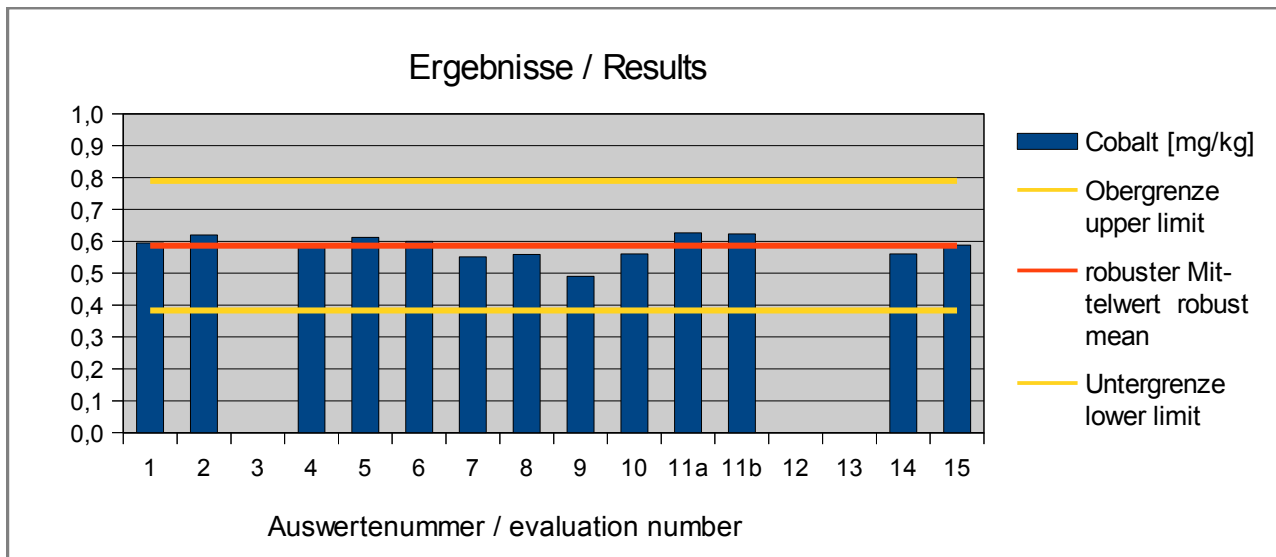


Abb. 11: Ergebnisse Kobalt / Fig. 11: Results Cobalt

**Ergebnisse der Teilnehmer:
Results of Participants:**

Auswertenummer Evaluation number	Cobalt [mg/kg]	Abweichung [mg/kg] Deviation [mg/kg]	z-Score (σ_{pt})	Hinweis Remark
1	0,595	0,009	0,1	
2	0,62	0,034	0,3	
3				
4	0,592	0,006	0,1	
5	0,6125	0,026	0,3	
6	0,6	0,014	0,1	
7	0,551	-0,035	-0,3	
8	0,5589	-0,027	-0,3	
9	0,49	-0,096	-0,9	
10	0,56	-0,026	-0,3	
11a	0,6265	0,040	0,4	
11b	0,6235	0,037	0,4	
12				
13				
14	0,56	-0,026	-0,3	
15	0,588	0,002	0,0	

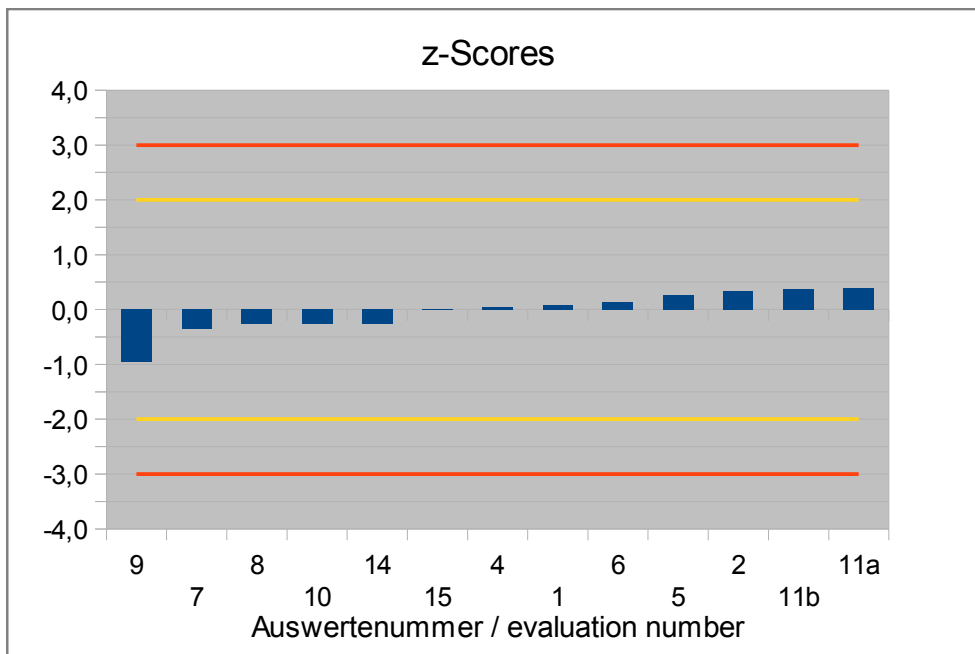


Abb. 12: Z-Scores Kobalt
Fig. 12: Z-Scores Cobalt

4.7 Cu - Kupfer / Copper in mg/kg

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	16
Number of outliers	0
Mean	5,77
Median	5,76
Robust Mean (X_{pt})	5,75
Robust standard deviation (S^*)	0,439
Number with 2 replicates	14
Repeatability SD (S_r)	0,438
Repeatability (CV_r)	7,68%
Reproducibility SD (S_R)	0,665
Reproducibility (CV_R)	11,6%
Target range:	
Target standard deviation σ_{pt}	0,707
Target standard deviation (for Information)	0,316
lower limit of target range	4,34
upper limit of target range	7,17
Quotient S^*/σ_{pt}	0,62
Standard uncertainty $U(X_{pt})$	0,137
Quotient $U(X_{pt})/\sigma_{pt}$	0,19
Results in the target range	16
Percent in the target range	100%

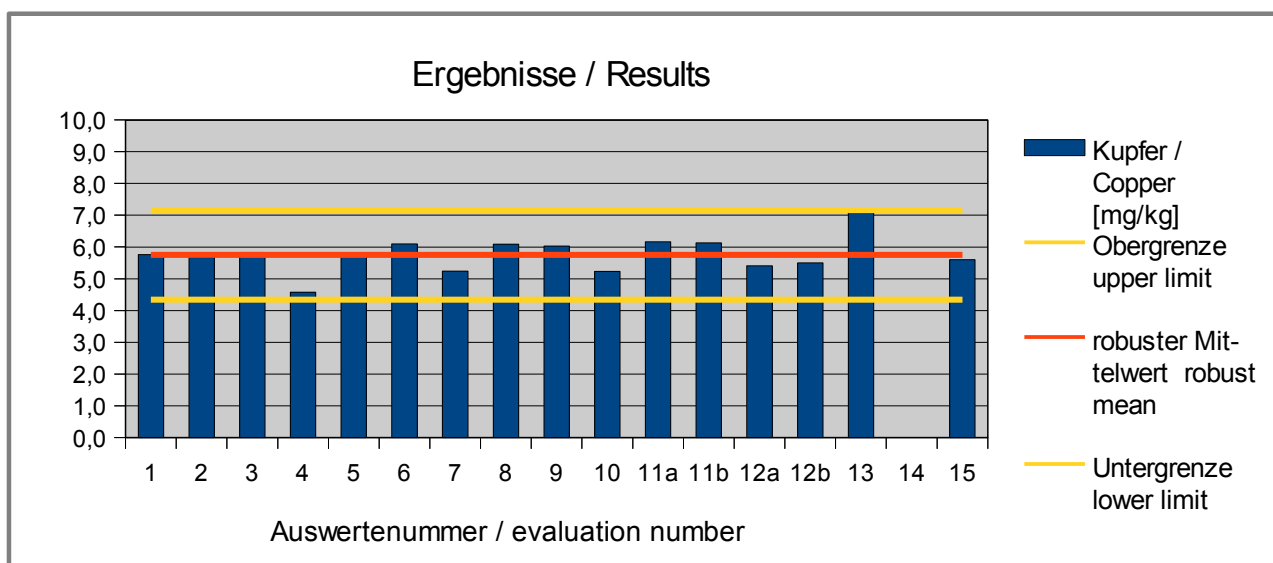


Abb. 13: Ergebnisse Kupfer / Fig. 13: Results Copper

**Ergebnisse der Teilnehmer:
Results of Participants:**

Auswertenummer Evaluation number	Kupfer / Copper [mg/kg]	Abweichung [mg/kg] Deviation [mg/kg]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	5,76	0,009	0,0	0,0	
2	5,67	-0,081	-0,1	-0,3	
3	5,83	0,079	0,1	0,3	
4	4,578	-1,173	-1,7	-3,7	
5	5,7565	0,006	0,0	0,0	
6	6,1	0,349	0,5	1,1	
7	5,24	-0,511	-0,7	-1,6	
8	6,09	0,339	0,5	1,1	
9	6,03	0,279	0,4	0,9	
10	5,23	-0,521	-0,7	-1,6	
11a	6,165	0,414	0,6	1,3	
11b	6,1325	0,382	0,5	1,2	
12a	5,41	-0,341	-0,5	-1,1	
12b	5,5	-0,251	-0,4	-0,8	
13	7,2	1,449	2,0	4,6	
14					
15	5,597	-0,15	-0,2	-0,5	

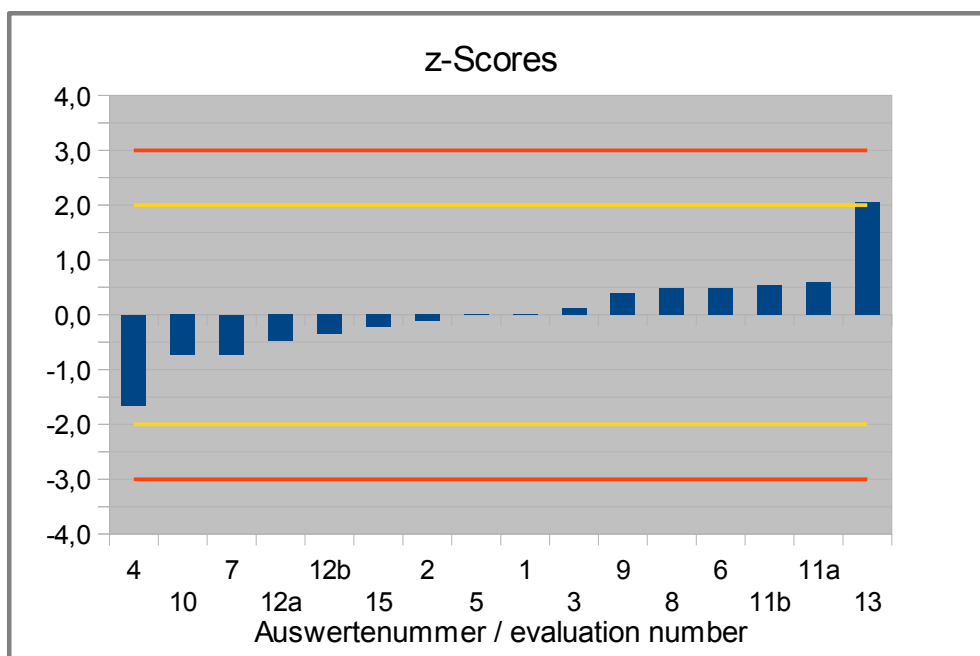


Abb. 14: Z-Scores Kupfer

Fig. 14: Z-Scores Copper

4.8 Fe - Eisen / Iron in mg/kg

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	16
Number of outliers	1
Mean	300
Median	310
Robust Mean (X_{pt})	305
Robust standard deviation (S^*)	22,1
Number with 2 replicates	13
Repeatability SD (S_r)	8,27
Repeatability (CV_r)	2,71%
Reproducibility SD (S_R)	27,9
Reproducibility (CV_R)	9,2%
Target range:	
Target standard deviation σ_{pt}	20,6
Target standard deviation (for Information)	22,2
lower limit of target range	264
upper limit of target range	346
Quotient S^*/σ_{pt}	1,1
Standard uncertainty $U(X_{pt})$	6,91
Quotient $U(X_{pt})/\sigma_{pt}$	0,33
Results in the target range	12
Percent in the target range	75%

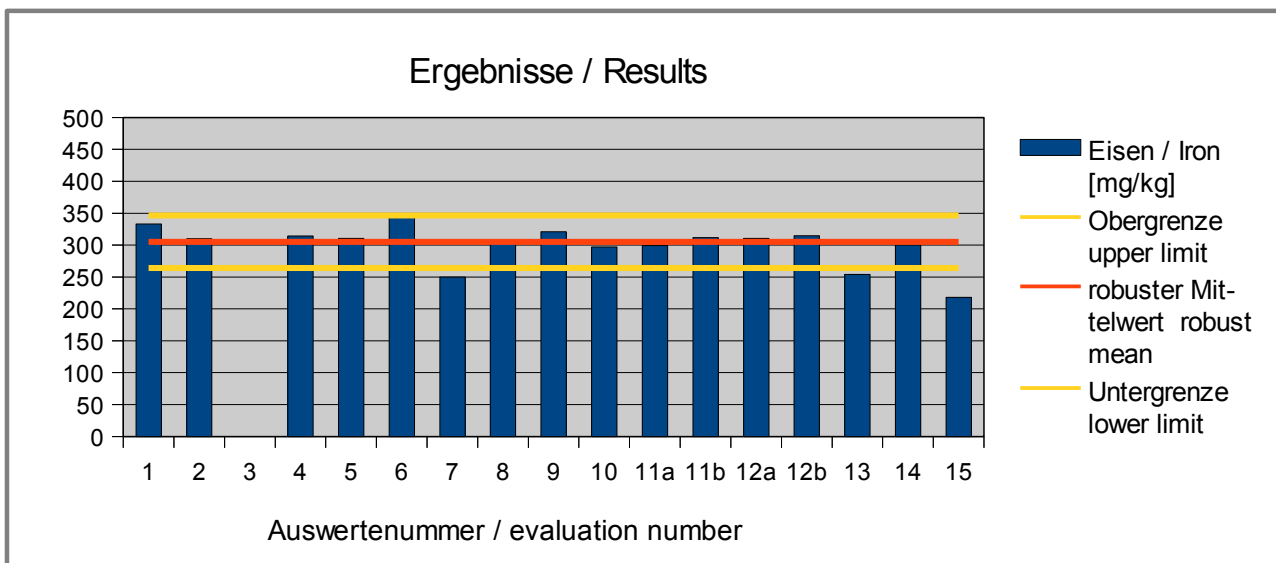


Abb. 14: Ergebnisse Eisen / Fig. 14: Results Iron

**Ergebnisse der Teilnehmer:
Results of Participants:**

Auswertenummer	Eisen / Iron [mg/kg]	Abweichung [mg/kg]	z-Score (σ _{pt})	z-Score (Info)	Hinweis
Evaluation number		Deviation [mg/kg]			Remark
1	333	27,839	1,3	1,3	
2	310	4,839	0,2	0,2	
3					
4	314,325	9,164	0,4	0,4	
5	310,65	5,489	0,3	0,2	
6	348	42,839	2,1	1,9	
7	250	-55,161	-2,7	-2,5	
8	304,5	-0,661	0,0	0,0	
9	321	15,839	0,8	0,7	
10	297	-8,161	-0,4	-0,4	
11a	299,4	-5,761	-0,3	-0,3	
11b	311,65	6,489	0,3	0,3	
12a	310,6	5,439	0,3	0,2	
12b	314,62	9,459	0,5	0,4	
13	254	-51,161	-2,5	-2,3	
14	300	-5,161	-0,3	-0,2	
15	217,97	-87,19	-4,2	-3,9	Ausreisser / Outlier

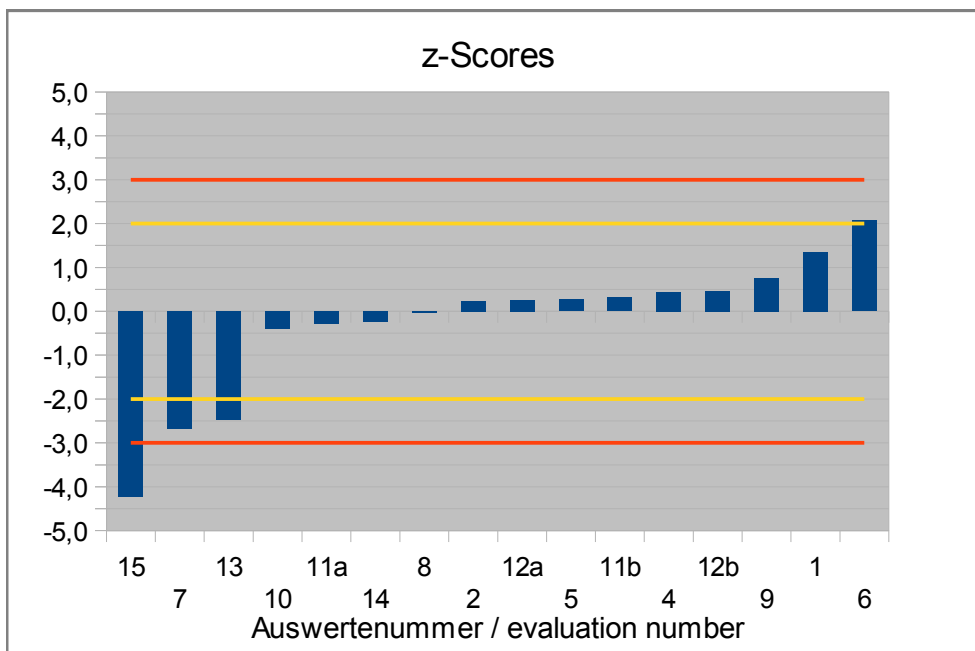


Abb. 15: Z-Scores Eisen

Fig. 15: Z-Scores Iron

4.9 I - Iod / Iodine in mg/kg

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	9
Number of outliers	0
Mean	9,22
Median	10,5
Robust Mean (X_{pt})	9,81
Robust standard deviation (S^*)	3,41
Number with 2 replicates	8
Repeatability SD (S_r)	0,322
Repeatability (CV_r)	3,56%
Reproducibility SD (S_R)	4,34
Reproducibility (CV_R)	48,0%
Target range:	
Target standard deviation σ_{pt}'	1,81
Target standard deviation (for Information)	0,87
lower limit of target range	6,20
upper limit of target range	13,4
Quotient S^*/σ_{pt}'	1,9
Standard uncertainty $U(X_{pt})$	1,42
Quotient $U(X_{pt})/\sigma_{pt}'$	0,79
Results in the target range	6
Percent in the target range	67%

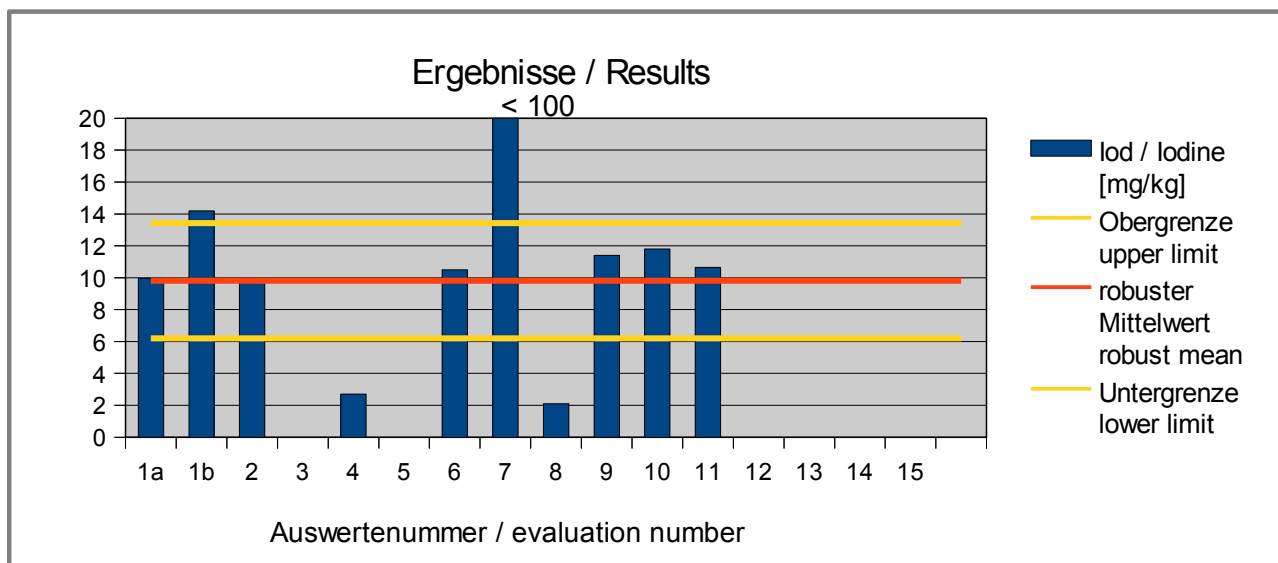


Abb. 16: Ergebnisse Iod / Fig. 16: Results Iodine

**Ergebnisse der Teilnehmer:
Results of Participants:**

Auswertenummer	Iod / Iodine [mg/kg]	Abweichung [mg/kg]	z'-Score (σ _{pt})	z-Score (Info)	Hinweis
Evaluation number		Deviation [mg/kg]			Remark
1a	9,96	0,151	0,1	0,2	
1b	14,2	4,391	2,4	5,0	
2	9,7	-0,109	-0,1	-0,1	
3					
4	2,697	-7,112	-3,9	-8,2	
5					
6	10,5	0,691	0,4	0,8	
7	< 100				
8	2,1	-7,709	-4,3	-8,8	
9	11,4	1,591	0,9	1,8	
10	11,8	1,991	1,1	2,3	
11	10,647	0,838	0,5	1,0	
12					
13					
14					
15					

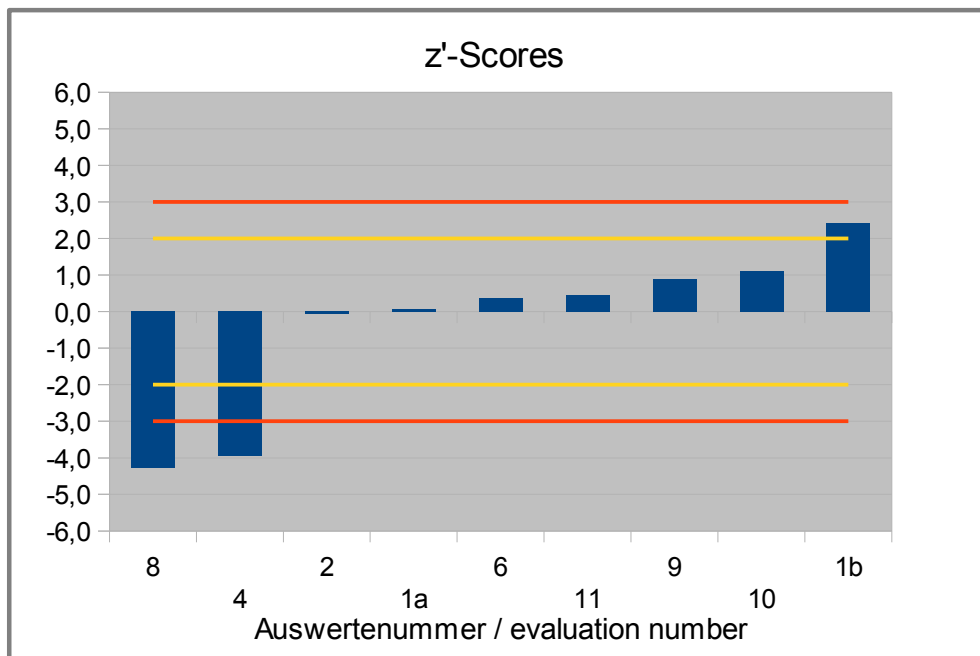


Abb. 17: z'-Scores Iod

Fig. 17: z'-Scores Iodine

4.10 Li - Lithium in mg/kg

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	9
Number of outliers	2
Mean	2,68
Median	1,45
Robust Mean (X_{pt})	1,51
Robust standard deviation (S^*)	0,204
Number with 2 replicates	6
Repeatability SD (S_r)	0,134
Repeatability (CV_r)	9,37%
Reproducibility SD (S_R)	0,136
Reproducibility (CV_R)	9,49%
Target range:	
Target standard deviation σ_{pt}	0,227
lower limit of target range	1,06
upper limit of target range	1,96
Quotient S^*/σ_{pt}	0,90
Standard uncertainty $U(X_{pt})$	0,085
Quotient $U(X_{pt})/\sigma_{pt}$	0,38
Results in the target range	7
Percent in the target range	78%

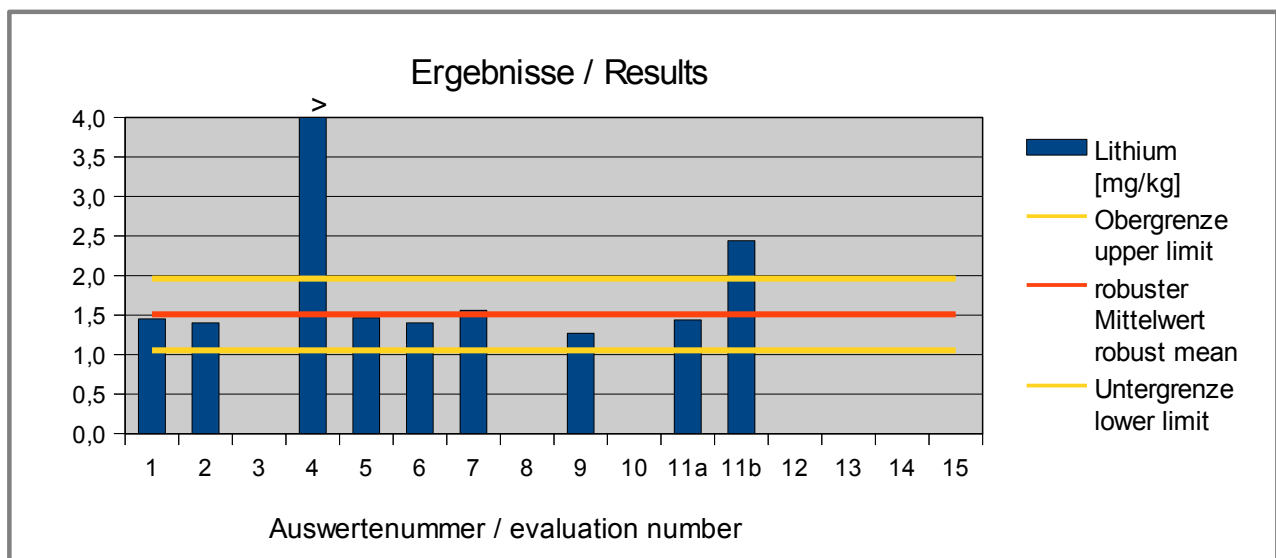


Abb. 18: Ergebnisse Lithium / Fig. 18: Results Lithium

**Ergebnisse der Teilnehmer:
Results of Participants:**

Auswertenummer	Lithium [mg/kg]	Abweichung [mg/kg]	z-Score	Hinweis
Evaluation number		Deviation [mg/kg]	(σ_{pt})	Remark
1	1,45	-0,059	-0,3	
2	1,4	-0,109	-0,5	
3				
4	11,72	10,211	45,0	Ausreisser / Outlier
5	1,466	-0,043	-0,2	
6	1,4	-0,109	-0,5	
7	1,56	0,051	0,2	
8				
9	1,27	-0,239	-1,1	
10				
11a	1,4395	-0,069	-0,3	
11b	2,442	0,933	4,1	Ausreisser / Outlier
12				
13				
14				
15				
16				



Abb. 19: Z-Scores Lithium

Fig. 19: Z-Scores Lithium

4.11 Mn - Mangan / Manganese in mg/kg

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	17
Number of outliers	0
Mean	8,75
Median	8,73
Robust Mean (X_{pt})	8,79
Robust standard deviation (S^*)	0,696
Number with 2 replicates	15
Repeatability SD (S_r)	0,308
Repeatability (CV_r)	3,53%
Reproducibility SD (S_R)	0,838
Reproducibility (CV_R)	9,59%
Target range:	
Target standard deviation σ_{pt}	1,01
Target standard deviation (for Information)	0,633
lower limit of target range	6,76
upper limit of target range	10,8
Quotient S^*/σ_{pt}	0,69
Standard uncertainty $U(X_{pt})$	0,211
Quotient $U(X_{pt})/\sigma_{pt}$	0,21
Results in the target range	17
Percent in the target range	100%

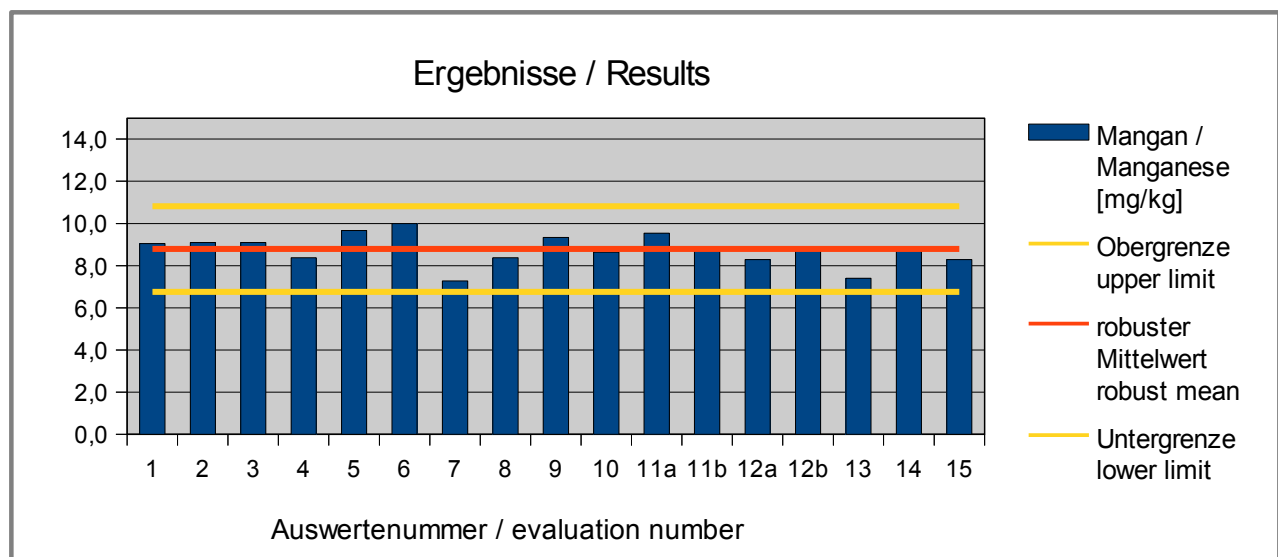


Abb. 20: Ergebnisse Mangan / **Fig. 20:** Results Manganese

**Ergebnisse der Teilnehmer:
Results of Participants:**

Auswertenummer Evaluation number	Mangan / Manganese [mg/kg]	Abweichung [mg/kg] Deviation [mg/kg]	z-Score (σ _{pt})	z-Score (Info)	Hinweis Remark
1	9,04	0,254	0,3	0,4	
2	9,1	0,314	0,3	0,5	
3	9,1	0,314	0,3	0,5	
4	8,3755	-0,410	-0,4	-0,6	
5	9,66	0,874	0,9	1,4	
6	10	1,214	1,2	1,9	
7	7,275	-1,511	-1,5	-2,4	
8	8,381	-0,405	-0,4	-0,6	
9	9,34	0,554	0,5	0,9	
10	8,64	-0,146	-0,1	-0,2	
11a	9,54	0,754	0,7	1,2	
11b	8,7025	-0,083	-0,1	-0,1	
12a	8,29	-0,496	-0,5	-0,8	
12b	8,86	0,074	0,1	0,1	
13	7,4	-1,386	-1,4	-2,2	
14	8,73	-0,056	-0,1	-0,1	
15	8,287	-0,50	-0,5	-0,8	

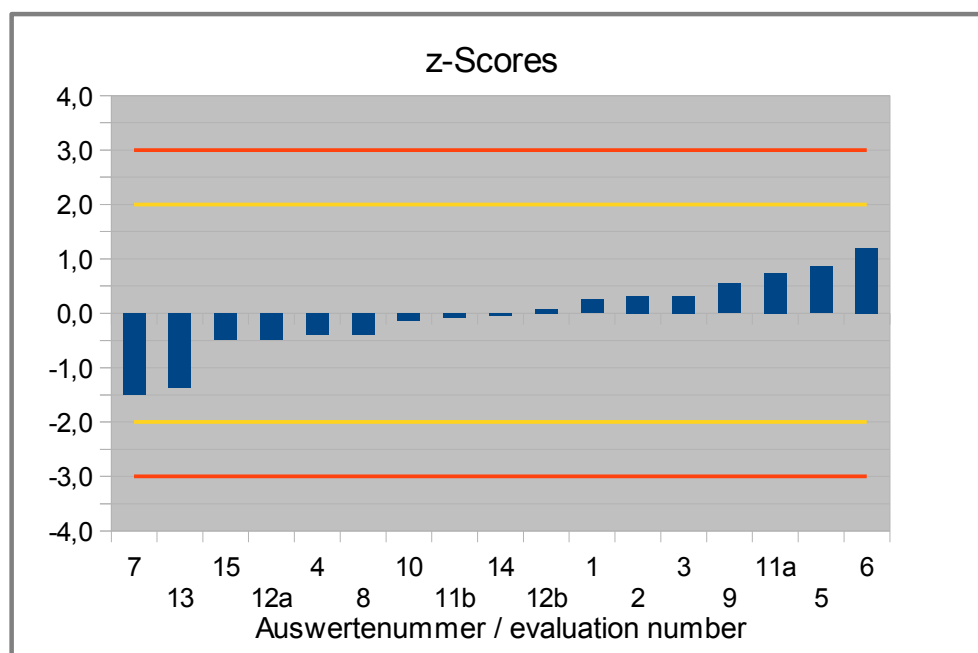


Abb. 21: Z-Scores Mangan
Fig. 21: Z-Scores Manganese

4.12 Mo - Molybdän / Molybdenum in mg/kg

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	11
Number of outliers	1
Mean	0,565
Median	0,540
Robust Mean (X_{pt})	0,536
Robust standard deviation (S^*)	0,0400
Number with 2 replicates	8
Repeatability SD (S_r)	0,0241
Repeatability (CV_r)	4,69%
Reproducibility SD (S_R)	0,0456
Reproducibility (CV_R)	8,86%
Target range:	
Target standard deviation σ_{pt}	0,0942
Target standard deviation (for Information)	0,1098
lower limit of target range	0,348
upper limit of target range	0,725
Quotient S^*/σ_{pt}	0,42
Standard uncertainty $U(X_{pt})$	0,0151
Quotient $U(X_{pt})/\sigma_{pt}$	0,16
Results in the target range	10
Percent in the target range	91%

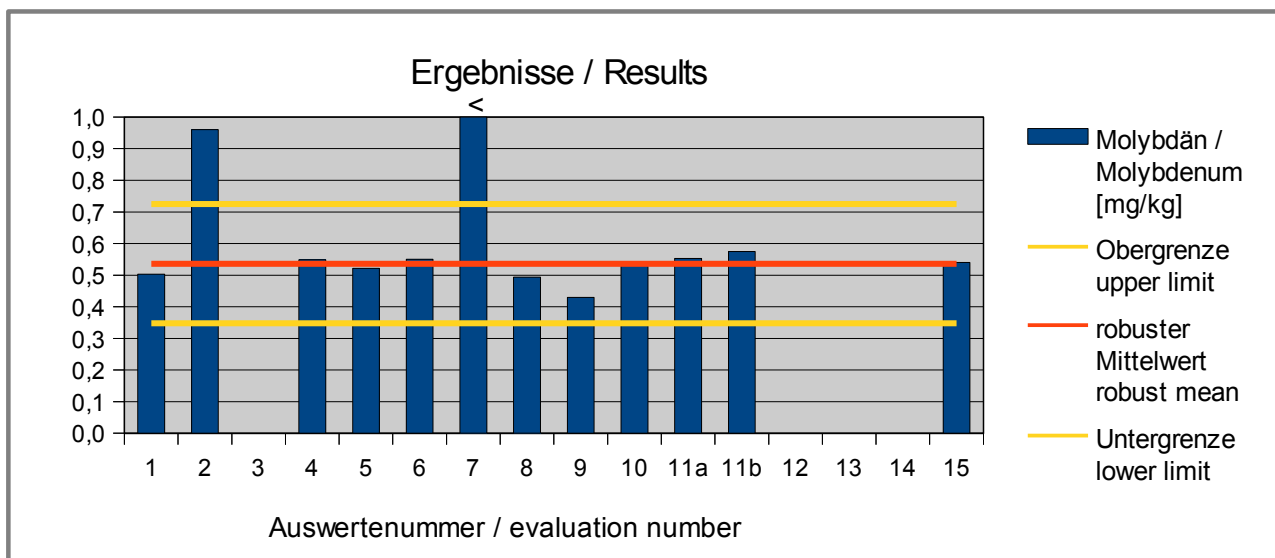


Abb. 22: Ergebnisse Molybdän / Fig. 22: Results Molybdenum

Ergebnisse der Teilnehmer:
Results of Participants:

Auswertenummer	Molybdän / Molybdenum [mg/kg]	Abweichung [mg/kg]	z-Score (σ_{pt})	z-Score (Info)	Hinweis
Evaluation number		Deviation [mg/kg]			Remark
1	0,503	-0,033	-0,4	-0,3	
2	0,96	0,424	4,5	3,9	Ausreisser / Outlier
3					
4	0,549	0,013	0,1	0,1	
5	0,522	-0,014	-0,2	-0,1	
6	0,55	0,014	0,1	0,1	
7	< 1				
8	0,4938	-0,042	-0,4	-0,4	
9	0,43	-0,106	-1,1	-1,0	
10	0,54	0,004	0,0	0,0	
11a	0,5525	0,016	0,2	0,1	
11b	0,575	0,039	0,4	0,4	
12					
13					
14					
15	0,54	0,004	0,0	0,0	

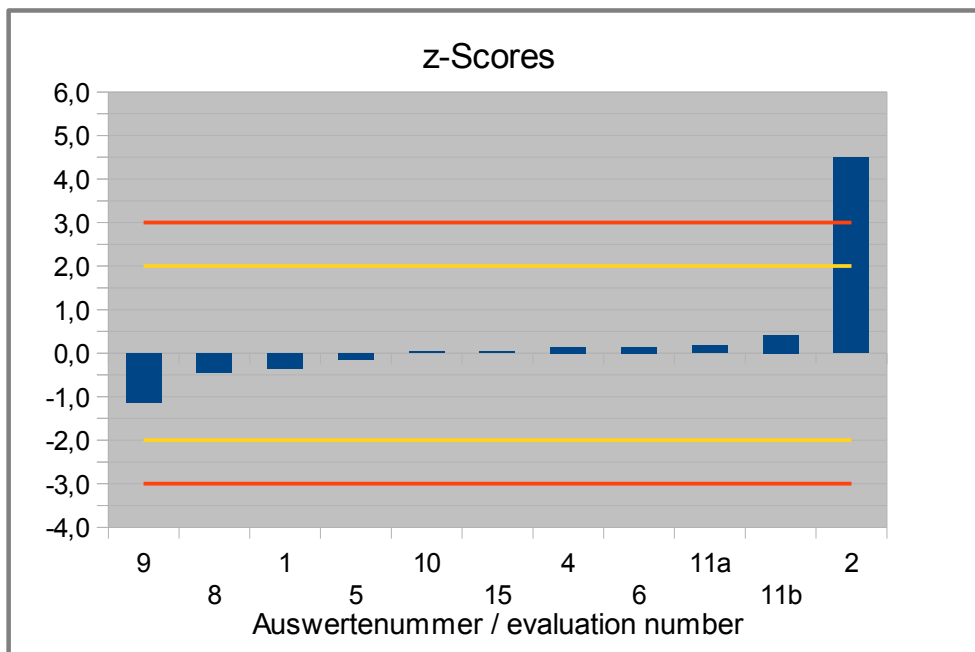


Abb. 23: Z-Scores Molybdän
Fig. 23: Z-Scores Molybdenum

4.13 Ni - Nickel in mg/kg

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	17
Number of outliers	0
Mean	1,40
Median	1,38
Robust Mean (X_{pt})	1,40
Robust standard deviation (S^*)	0,232
Number with 2 replicates	15
Repeatability SD (S_r)	0,0614
Repeatability (CV_r)	4,40%
Reproducibility SD (S_R)	0,219
Reproducibility (CV_R)	15,7%
Target range:	
Target standard deviation σ_{pt}	0,213
lower limit of target range	0,972
upper limit of target range	1,82
Quotient S^*/σ_{pt}	1,1
Standard uncertainty $U(X_{pt})$	0,0703
Quotient $U(X_{pt})/\sigma_{pt}$	0,33
Results in the target range	17
Percent in the target range	100%

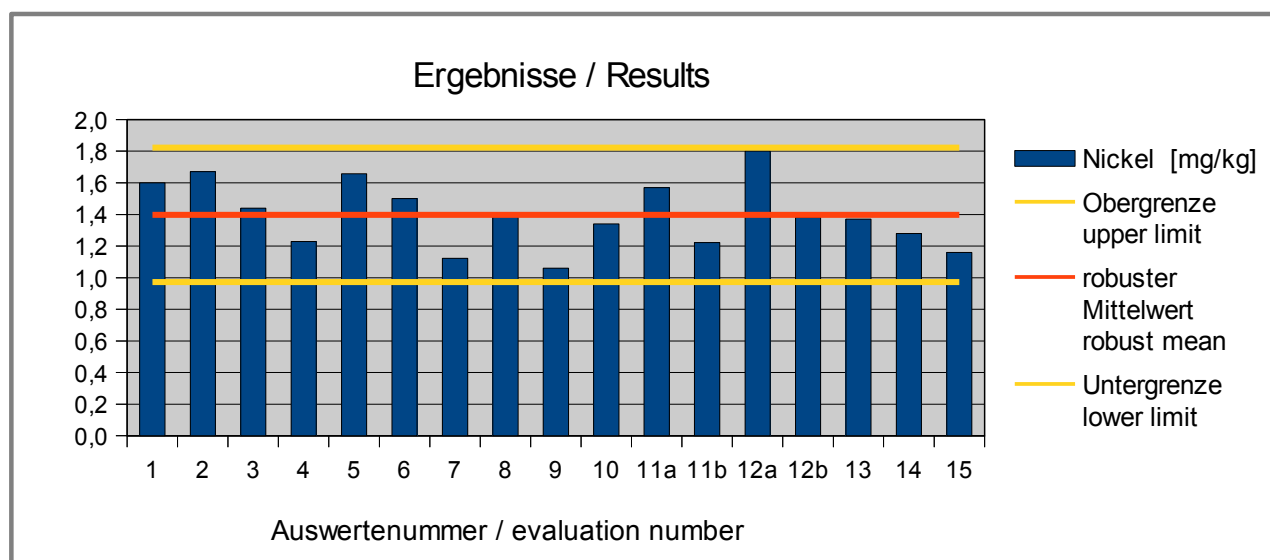


Abb. 24: Ergebnisse Nickel / **Fig. 24:** Results Nickel

**Ergebnisse der Teilnehmer:
Results of Participants:**

Auswertenummer	Nickel [mg/kg]	Abweichung [mg/kg]	z-Score	Hinweis
Evaluation number		Deviation [mg/kg]	(σ_{pt})	Remark
1	1,6	0,203	1,0	
2	1,67	0,273	1,3	
3	1,44	0,043	0,2	
4	1,229	-0,168	-0,8	
5	1,657	0,260	1,2	
6	1,5	0,103	0,5	
7	1,122	-0,275	-1,3	
8	1,41	0,013	0,1	
9	1,06	-0,337	-1,6	
10	1,34	-0,057	-0,3	
11a	1,57	0,173	0,8	
11b	1,222	-0,175	-0,8	
12a	1,8	0,403	1,9	
12b	1,38	-0,017	-0,1	
13	1,37	-0,027	-0,1	
14	1,28	-0,117	-0,6	
15	1,159	-0,24	-1,1	

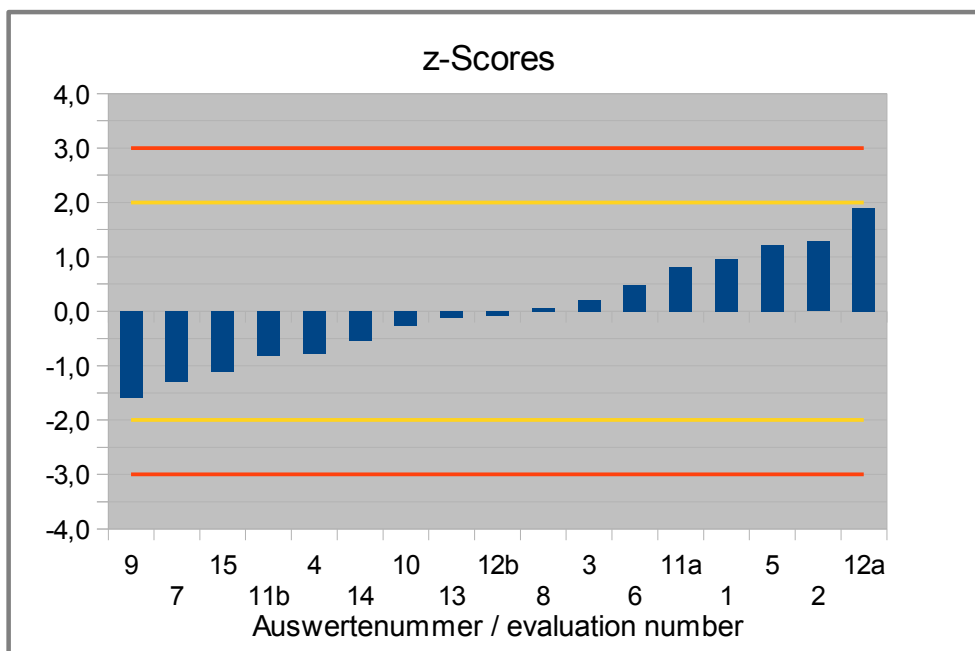


Abb. 25: Z-Scores Nickel
Fig. 25: Z-Scores Nickel

4.14 Pb - Blei / Lead in mg/kg

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	16
Number of outliers	1
Mean	0,300
Median	0,329
Robust Mean (X_{pt})	0,311
Robust standard deviation (S^*)	0,0525
Number with 2 replicates	13
Repeatability SD (S_r)	0,0117
Repeatability (CV_r)	3,81%
Reproducibility SD (S_R)	0,058
Reproducibility (CV_R)	19,0%
<i>Target range:</i>	
Target standard deviation σ_{pt}	0,0592
Target standard deviation (for Information)	0,0407
lower limit of target range	0,192
upper limit of target range	0,429
Quotient S^*/σ_{pt}	0,89
Standard uncertainty $U(X_{pt})$	0,0164
Quotient $U(X_{pt})/\sigma_{pt}$	0,28
Results in the target range	14
Percent in the target range	88%

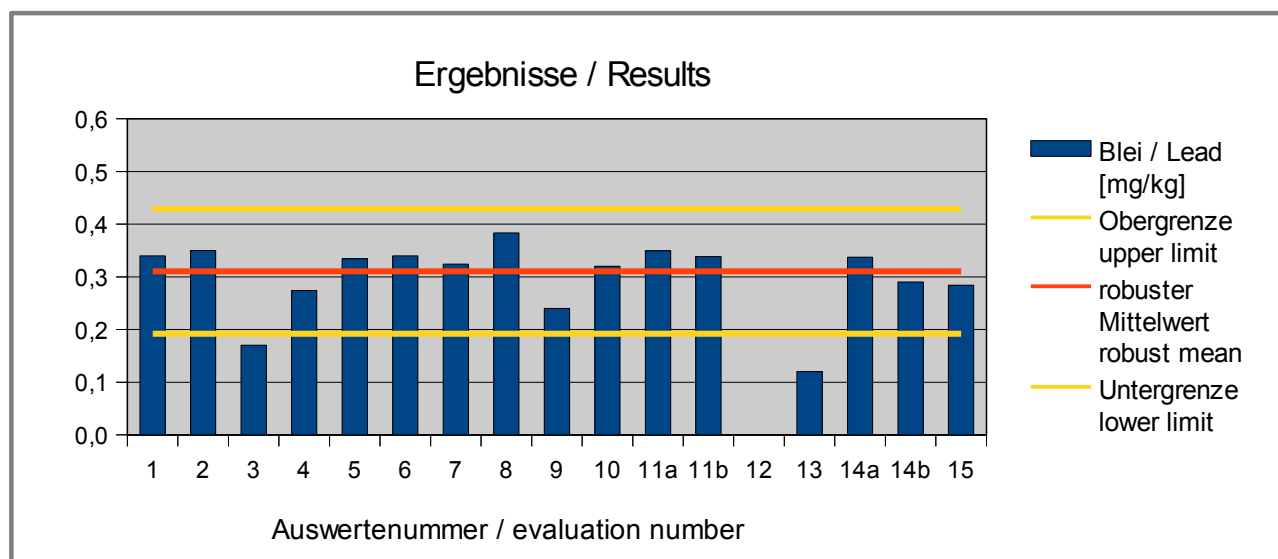


Abb. 26: Ergebnisse Blei / Fig. 26: Results Lead

**Ergebnisse der Teilnehmer:
Results of Participants:**

Auswertenummer Evaluation number	Blei / Lead [mg/kg]	Abweichung [mg/kg] Deviation [mg/kg]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	0,34	0,029	0,5	0,7	
2	0,35	0,039	0,7	1,0	
3	0,17	-0,141	-2,4	-3,5	
4	0,274	-0,037	-0,6	-0,9	
5	0,334	0,023	0,4	0,6	
6	0,34	0,029	0,5	0,7	
7	0,324	0,013	0,2	0,3	
8	0,3832	0,073	1,2	1,8	
9	0,24	-0,071	-1,2	-1,7	
10	0,32	0,009	0,2	0,2	
11a	0,3495	0,039	0,7	1,0	
11b	0,3385	0,028	0,5	0,7	
12					
13	0,12	-0,191	-3,2	-4,7	Ausreisser / Outlier
14a	0,337	0,026	0,4	0,7	
14b	0,29	-0,021	-0,3	-0,5	
15	0,284	-0,03	-0,4	-0,7	

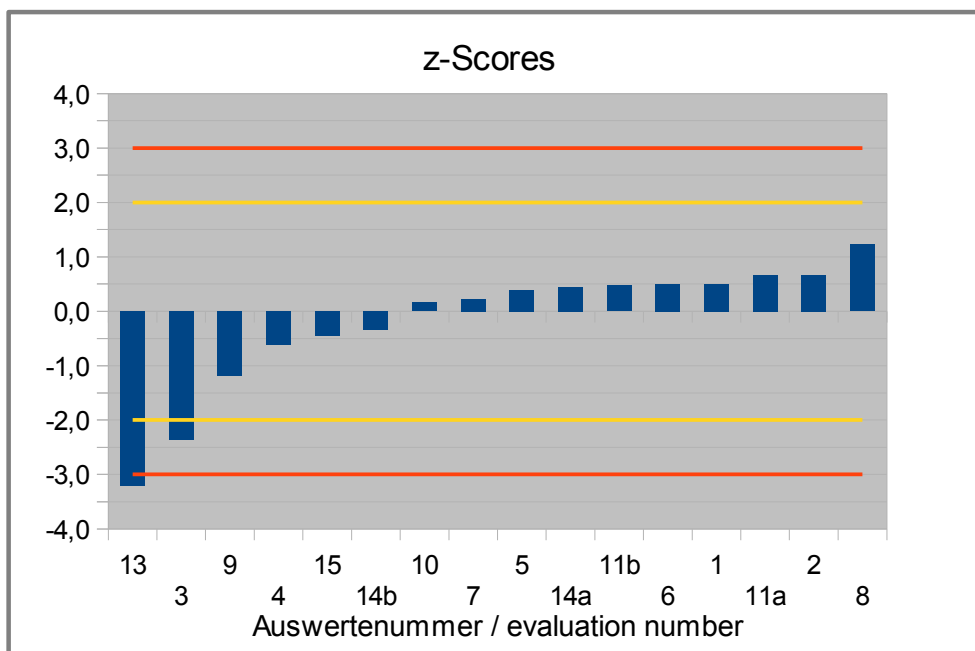


Abb. 27: Z-Scores Blei
Fig. 27: Z-Scores Lead

4.15 Rb - Rubidium in mg/kg**Vergleichsuntersuchung / Proficiency Test**

Statistic Data	All*	ICP-MS*	RFA*
<i>Number of results</i>	6	4	2
<i>Number of outliers</i>	-	-	-
Mean	3,11	3,77	1,78
Median	3,61	3,85	
Robust Mean (X)	3,11	3,77	
Robust standard deviation (S*)	1,19	0,28	
<i>Number with 2 replicates</i>			
Repeatability SD (S_r)			
Repeatability (CV_r)			
Reproducibility SD (S_R)			
Reproducibility (CV_R)			
<i>Target range:</i>			
Target standard deviation σ_{pt}			
Target standard deviation (for Information)			
lower limit of target range			
upper limit of target range			
<i>Quotient S^*/σ_{pt}</i>			
<i>Standard uncertainty $U_{(X_{pt})}$</i>			
<i>Quotient $U_{(X_{pt})}/\sigma_{pt}$</i>			
<i>Results in the target range</i>			
<i>Percent in the target range</i>			

* Methods see documentation

Due to the low number of results <7 no statistical evaluation was done

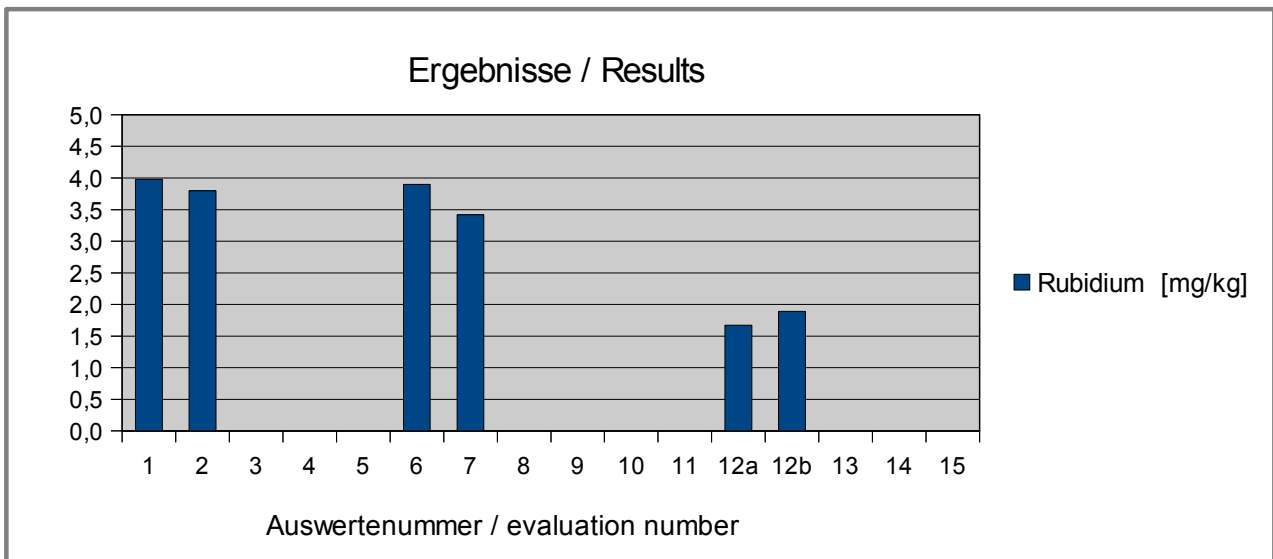


Abb. 28: Ergebnisse Rubidium / Fig. 28: Results Rubidium

Ergebnisse der Teilnehmer:
Results of Participants:

Auswertenummer Evaluation number	Rubidium [mg/kg]	Abweichung [mg/kg] Deviation [mg/kg]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	3,98				ICP-MS
2	3,8				ICP-MS
3					
4					
5					
6	3,9				ICP-MS
7	3,418				ICP-MS
8					
9					
10					
11					
12a	1,67				RFA
12b	1,89				RFA
13					
14					
15					

4.16 Se - Selen / Selenium in mg/kg

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	16
Number of outliers	0
Mean	1,90
Median	1,79
Robust Mean (X_{pt})	1,86
Robust standard deviation (S^*)	0,481
Number with 2 replicates	14
Repeatability SD (S_r)	0,172
Repeatability (CV_r)	9,22%
Reproducibility SD (S_R)	0,704
Reproducibility (CV_R)	37,6%
<i>Target range:</i>	
Target standard deviation σ_{pt}	0,272
Target standard deviation (for Information)	0,136
lower limit of target range	1,32
upper limit of target range	2,41
Quotient S^*/σ_{pt}	1,8
Standard uncertainty $U(X_{pt})$	0,150
Quotient $U(X_{pt})/\sigma_{pt}$	0,55
Results in the target range	13
Percent in the target range	81%

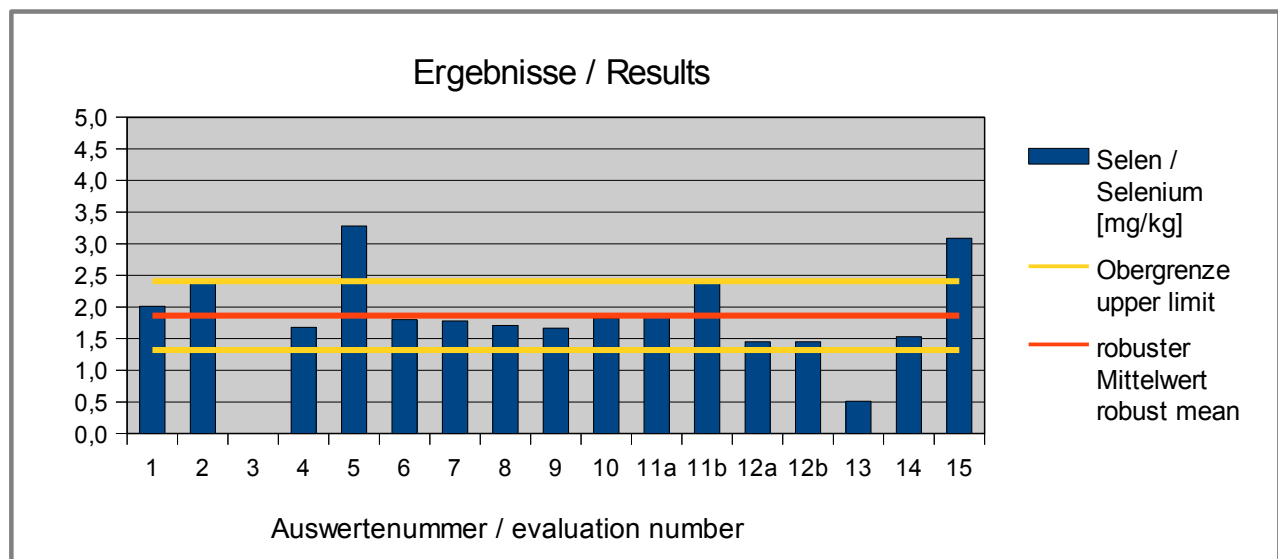


Abb. 29: Ergebnisse Selen / Fig. 29: Results Selenium

**Ergebnisse der Teilnehmer:
Results of Participants:**

Auswertenummer Evaluation number	Selen / Selenium [mg/kg]	Abweichung [mg/kg] Deviation [mg/kg]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	2,01	0,145	0,5	1,1	
2	2,4	0,535	2,0	3,9	
3					
4	1,678	-0,187	-0,7	-1,4	
5	3,281	1,416	5,2	10,4	
6	1,8	-0,065	-0,2	-0,5	
7	1,778	-0,087	-0,3	-0,6	
8	1,707	-0,158	-0,6	-1,2	
9	1,667	-0,198	-0,7	-1,5	
10	1,82	-0,045	-0,2	-0,3	
11a	1,8495	-0,015	-0,1	-0,1	
11b	2,3835	0,519	1,9	3,8	
12a	1,45	-0,415	-1,5	-3,0	
12b	1,45	-0,415	-1,5	-3,0	
13	0,51	-1,355	-5,0	-9,9	
14	1,53	-0,335	-1,2	-2,5	
15	3,089	1,22	4,5	9,0	

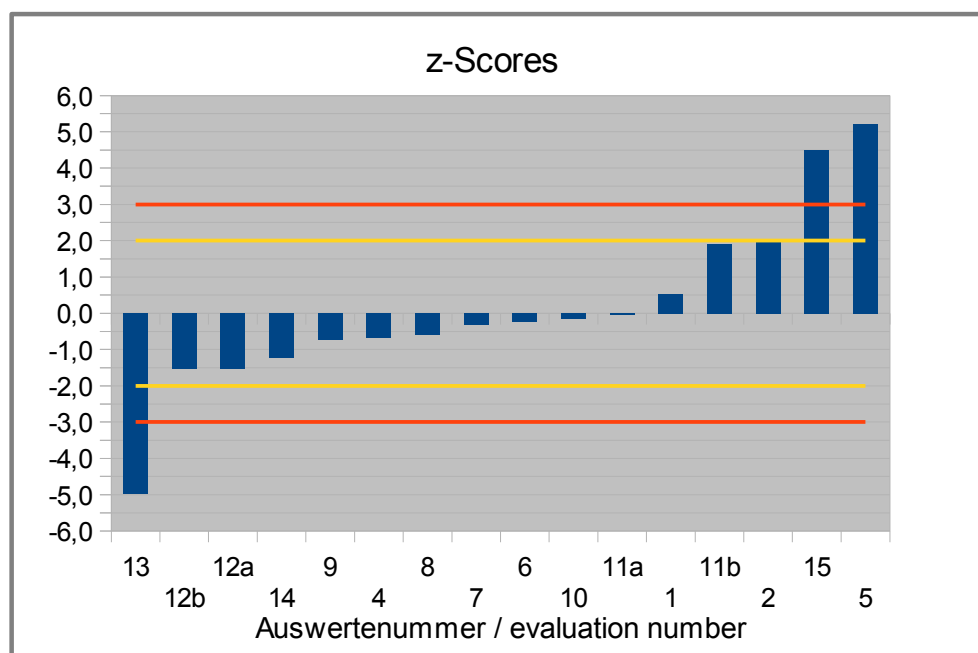


Abb. 30: Z-Scores Selen
Fig. 30: Z-Scores Selenium

4.17 Ti - Titan / Titanium in mg/kg**Vergleichsuntersuchung / Proficiency Test**

Statistic Data	
<i>Number of results</i>	6
<i>Number of outliers</i>	0
Mean	21,6
Median	22,9
Robust Mean (X)	21,9
Robust standard deviation (S*)	8,92
<i>Number with 2 replicates</i>	
Repeatability SD (S_r)	
Repeatability (CV_r)	
Reproducibility SD (S_R)	
Reproducibility (CV_R)	
<i>Target range:</i>	
Target standard deviation σ_{pt}	
Target standard deviation (for Information)	
lower limit of target range	
upper limit of target range	
<i>Quotient S^*/σ_{pt}</i>	
<i>Standard uncertainty $U_{(X_{pt})}$</i>	
<i>Quotient $U_{(X_{pt})}/\sigma_{pt}$</i>	
<i>Results in the target range</i>	
<i>Percent in the target range</i>	

Due to the low number of results <7 no statistical evaluation was done

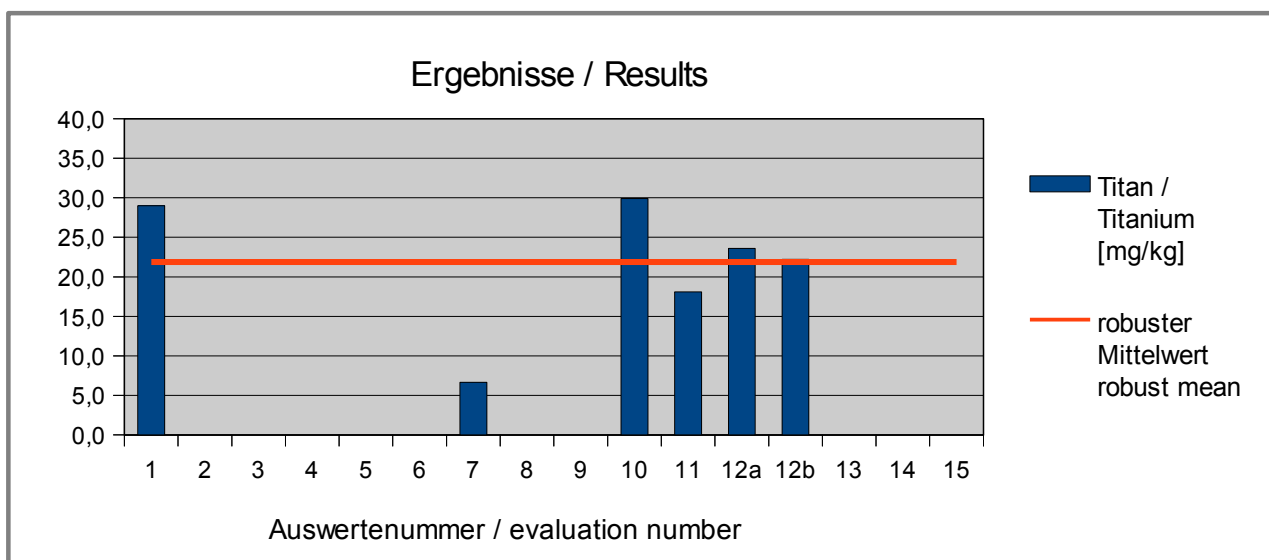


Abb. 31: Ergebnisse Titan / Fig. 31: Results Titanium

Ergebnisse der Teilnehmer:
Results of Participants:

Auswertenummer Evaluation number	Titan / Titanium [mg/kg]	Abweichung [mg/kg] Deviation [mg/kg]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	29	7,1			
2					
3					
4					
5					
6					
7	6,65	-15,2			
8					
9					
10	29,9	8,0			
11	18,1	-3,8			
12a	23,6	1,7			
12b	22,21	0,3			
13					
14					
15					
16					

4.18 V - Vanadium in mg/kg

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	11
Number of outliers	0
Mean	1,41
Median	1,40
Robust Mean (X_{pt})	1,40
Robust standard deviation (S^*)	0,318
Number with 2 replicates	9
Repeatability SD (S_r)	0,0255
Repeatability (CV_r)	1,83%
Reproducibility SD (S_R)	0,242
Reproducibility (CV_R)	17,4%
Target range:	
Target standard deviation σ_{pt}	0,213
lower limit of target range	0,977
upper limit of target range	1,83
Quotient S^*/σ_{pt}	1,5
Standard uncertainty $U(X_{pt})$	0,120
Quotient $U(X_{pt})/\sigma_{pt}$	0,56
Results in the target range	9
Percent in the target range	82%

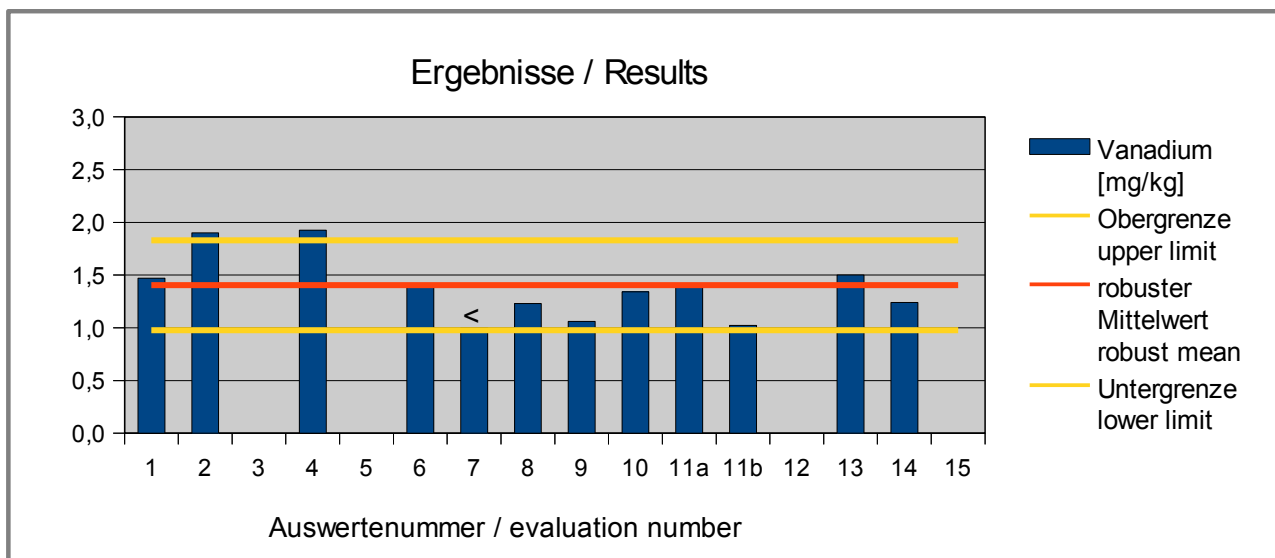


Abb. 32: Ergebnisse Vanadium / Fig. 32: Results Vanadium

**Ergebnisse der Teilnehmer:
Results of Participants:**

Auswertenummer Evaluation number	Vanadium [mg/kg]	Abweichung [mg/kg] Deviation [mg/kg]	z-Score (σ_{pt})	Hinweis Remark
1	1,47	0,067	0,3	
2	1,9	0,497	2,3	
3				
4	1,925	0,522	2,4	
5				
6	1,4	-0,003	0,0	
7	< 1			
8	1,23	-0,173	-0,8	
9	1,06	-0,343	-1,6	
10	1,34	-0,063	-0,3	
11a	1,42	0,017	0,1	
11b	1,0215	-0,382	-1,8	
12				
13	1,5	0,097	0,5	
14	1,24	-0,163	-0,8	
15				

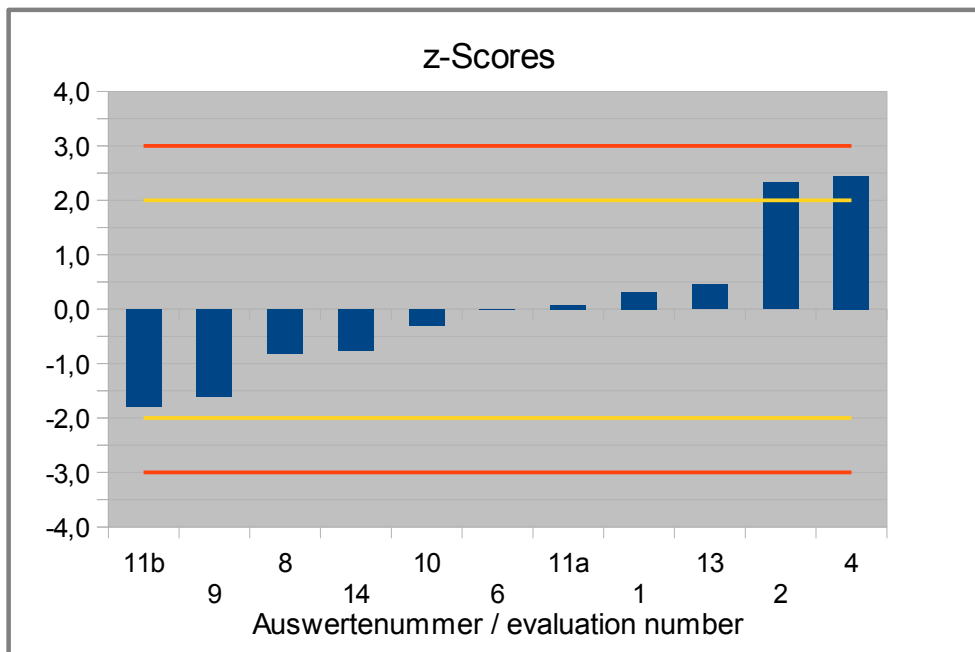


Abb. 33: Z-Scores Vanadium
Fig. 33: Z-Scores Vanadium

4.19 Y - Yttrium in mg/kg**Vergleichsuntersuchung / Proficiency Test**

Statistic Data	
<i>Number of results</i>	2
<i>Number of outliers</i>	0
Mean	0,10
Median	0,10
Robust Mean (X)	0,10
Robust standard deviation (S*)	0,010
<i>Number with 2 replicates</i>	
Repeatability SD (S_r)	
Repeatability (CV_r)	
Reproducibility SD (S_R)	
Reproducibility (CV_R)	
<i>Target range:</i>	
Target standard deviation σ_{pt}	
Target standard deviation (for Information)	
lower limit of target range	
upper limit of target range	
<i>Quotient S^*/σ_{pt}</i>	
<i>Standard uncertainty $U_{(X_{pt})}$</i>	
<i>Quotient $U_{(X_{pt})}/\sigma_{pt}$</i>	
<i>Results in the target range</i>	
<i>Percent in the target range</i>	

Due to the low number of results <7 no statistical evaluation was done

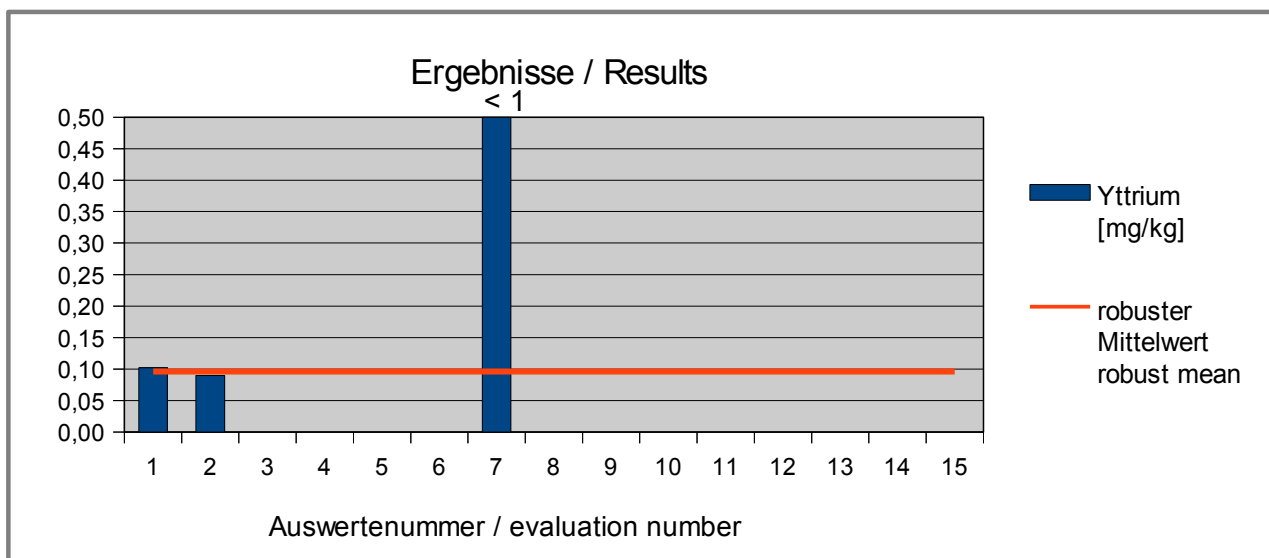


Abb. 34: Ergebnisse Yttrium / Fig. 34: Results Yttrium

Ergebnisse der Teilnehmer:

Results of Participants:

Auswertenummer	Yttrium [mg/kg]	Abweichung [mg/kg]	z-Score	z-Score	Hinweis
Evaluation number		Deviation [mg/kg]	(σ_{pt})	(Info)	Remark
1	0,102	0,006	0,3	0,3	
2	0,09	-0,006	-0,3	-0,3	
3					
4					
5					
6					
7	< 1				
8					
9					
10					
11					
12					
13					
14					
15					

4.20 Zn - Zink / Zinc in mg/kg

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	15
Number of outliers	0
Mean	51,1
Median	50,1
Robust Mean (X_{pt})	51,0
Robust standard deviation (S^*)	5,17
Number with 2 replicates	15
Repeatability SD (S_r)	2,94
Repeatability (CV_r)	5,74%
Reproducibility SD (S_R)	5,86
Reproducibility (CV_R)	11,5%
Target range:	
Target standard deviation σ_{pt}	4,51
Target standard deviation (for Information)	3,67
lower limit of target range	42,0
upper limit of target range	60,0
Quotient S^*/σ_{pt}	1,1
Standard uncertainty $U(X_{pt})$	1,67
Quotient $U(X_{pt})/\sigma_{pt}$	0,37
Results in the target range	13
Percent in the target range	87%

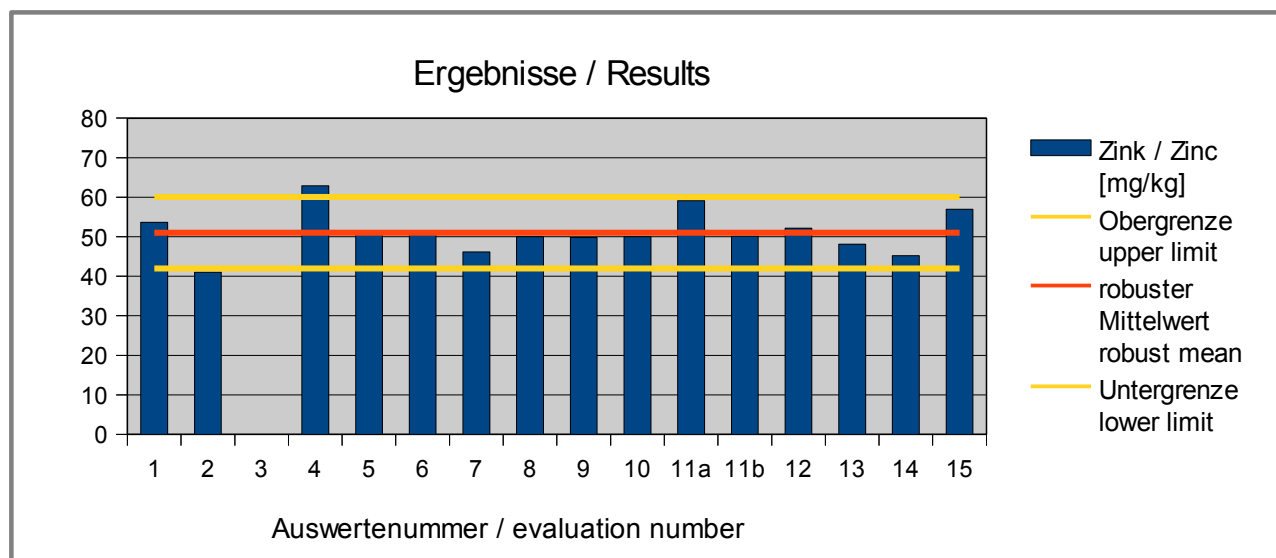


Abb. 35: Ergebnisse Zink / Fig. 35: Results Zinc

**Ergebnisse der Teilnehmer:
Results of Participants:**

Auswertenummer Evaluation number	Zink / Zinc [mg/kg]	Abweichung [mg/kg] Deviation [mg/kg]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	53,6	2,61	0,6	0,7	
2	41	-9,99	-2,2	-2,7	
3					
4	62,855	11,87	2,6	3,2	
5	51,07	0,08	0,0	0,0	
6	51	0,01	0,0	0,0	
7	46,16	-4,83	-1,1	-1,3	
8	50,01	-0,98	-0,2	-0,3	
9	49,8	-1,19	-0,3	-0,3	
10	50	-0,99	-0,2	-0,3	
11a	59,065	8,08	1,8	2,2	
11b	50,11	-0,88	-0,2	-0,2	
12	52,15	1,16	0,3	0,3	
13	48,1	-2,89	-0,6	-0,8	
14	45,21	-5,78	-1,3	-1,6	
15	56,952	5,96	1,3	1,6	

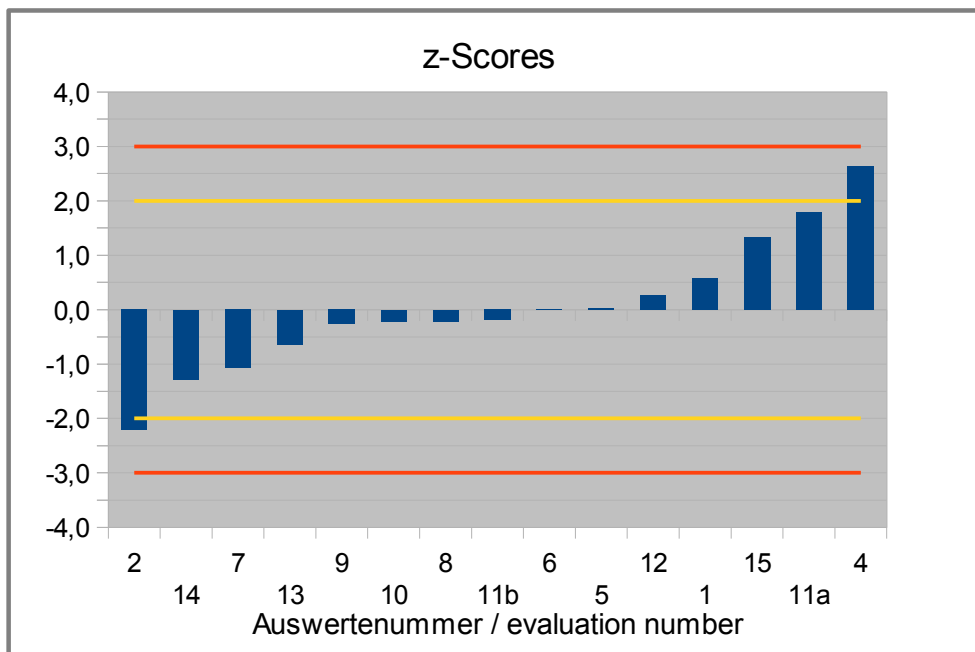


Abb. 36: Z-Scores Zink
Fig. 36: Z-Scores Zinc

5. Documentation

5.1 Primary data

Parameter	Teilnehmer	Einheit	Proben-Nr. A	Proben-Nr. B	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis A	Ergebnis B	Inkl. WF	Wiederfindungsrate [%]
Analyte	Participant	Unit	Sample No. A	Sample No. B	Date of analysis	Result (Mean)	Result A	Result B	Incl. RR	Recovery rate [%]
Al	1	mg/kg	07	36	03.06.16	602	597	607	no	-
	2	mg/kg	2	31	25.05.16				yes / no	100
	3	mg/kg	34	52						
	4	mg/kg	44	60	17.05.16	334,7	359,9	309,5	no	
	5	mg/kg	18	57	02.05.16	455,95	465,5	446,4		
	6	mg/kg	10	72						
	7	mg/kg	9	47	23.05.16	202	201	203	no	
	8	mg/kg	22	71	10.05.16	575,2	573,5	576,9	no	
	9	mg/kg	27	45	03.06.	427	425	429	no	
	10	mg/kg	39	68	19.05.	527	523	531	no	
	11	mg/kg	40	15	09.05.16	326,95	292,7 / 361,2		no	100
	12	mg/kg	38	65	-	-	-	-	-	-
	13	mg/kg	12	58						
	14	mg/kg	24	63	24.05.	521	516,5	525,4	no	90-110
	15	mg/kg	19	61						

Parameter	Teilnehmer	Einheit	Proben-Nr. A	Proben-Nr. B	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis A	Ergebnis B	Inkl. WF	Wiederfindungsrate [%]
Analyte	Participant	Unit	Sample No. A	Sample No. B	Date of analysis	Result (Mean)	Result A	Result B	Incl. RR	Recovery rate [%]
As	1	mg/kg	07	36	03.06.16	7,29	7,04	7,55	no	
	2	mg/kg	2	31	25.05.16	7,05	6,95	7,22	yes / no	100
	3	mg/kg	34	52	04.06.	4,34	4,86	3,82	no	117
	4	mg/kg	44	60	17.05.16	0,5	0,49	0,5	no	
	5	mg/kg	18	57	02.05.16	6,18	6,15	6,2		
	6	mg/kg	10	72	28.04.16	7	7,4	6,6	no	
	7	mg/kg	9	47	04.05.16	6,65	6,41	6,89	no	
	8	mg/kg	22	71	06.06.16	6,64	6,66	6,62	yes	110
	9	mg/kg	27	45	26.05.	6,28	6,31	6,24	no	
	10	mg/kg	39	68	17.05.	6,55	6,71	6,39	no	
	11a	mg/kg	40	15	10.05.16	7,33	7,47 / 7,18		no	100
	11b	mg/kg	40	15	09.05.16	7,4	7,296 / 7,501		no	100
	12a	mg/kg	38	65	18.05.16	6,35	6,34	6,35	no	-
	12b	mg/kg	38	65	23.05.16	6,7	6,73	6,67	no	-
	13	mg/kg	12	58	12.05.16	1,21	1,22	1,2	no	
14	mg/kg	24	63	24.05.	6,17	6,23	6,11	no	90-110	
15	mg/kg	19	61	11.05.16	6,95	6,97	6,93			

Parameter	Teilnehmer	Einheit	Proben-Nr. A	Proben-Nr. B	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis A	Ergebnis B	Inkl. WF	Wiederfindungsrate [%]
Analyte	Participant	Unit	Sample No. A	Sample No. B	Date of analysis	Result (Mean)	Result A	Result B	Incl. RR	Recovery rate [%]
Ba	1	mg/kg	07	36	03.06.16	3,35	3,37	3,32	no	
	2	mg/kg	2	31	25.05.16	1,69	1,62	1,77	yes / no	100
	3	mg/kg	34	52						
	4	mg/kg	44	60	17.05.16	1,4	1,51	1,28	no	
	5	mg/kg	18	57	02.05.16	1,95	2,11	1,8		
	6	mg/kg	10	72	28.04.16	3,2	3,5	2,8	no	
	7	mg/kg	9	47	23.05.16	<1	<1	<1	no	
	8	mg/kg	22	71	07.06.16	3,1	3,11	3,1	yes	107
	9	mg/kg	27	45	03.06.	2,31	2,17	2,45	no	
	10	mg/kg	39	68	25.05.	2,93	2,93	2,92	no	
	11a	mg/kg	40	15	10.05.2016	1,885		1,63 / 2,013	no	100
	11b	mg/kg	40	15	09.05.16	1,64	1,391 / 1,891		no	100
	12	mg/kg	38	65	-	-	-	-		-
	13	mg/kg	12	58						
	14	mg/kg	24	63						
15	mg/kg	19	61							

Parameter	Teilnehmer	Einheit	Proben-Nr. A	Proben-Nr. B	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis A	Ergebnis B	Inkl. WF	Wiederfindungsrate [%]
Analyte	Participant	Unit	Sample No. A	Sample No. B	Date of analysis	Result (Mean)	Result A	Result B	Incl. RR	Recovery rate [%]
Cd	1	mg/kg	07	36	03.06.16	1,11	1,11	1,11	no	
	2	mg/kg	2	31	25.05.16	1,02	1,03	1,01	yes / no	100
	3	mg/kg	34	52	04.06.	1,07	1,08	1,06	no	101
	4	mg/kg	44	60	17.05.16	1,02	1,03	1,01	no	
	5	mg/kg	18	57	02.05.16	1,02	1,02	1,01		
	6	mg/kg	10	72	28.04.16	1,1	1	1,1	no	
	7	mg/kg	9	47	04.05.16	1,07	1,04	1,09	no	
	8	mg/kg	22	71	06.06.16	1	0,99	1	yes	110
	9	mg/kg	27	45						
	10	mg/kg	39	68	17.05.	0,96	0,98	0,94	no	
	11a	mg/kg	40	15	10.05.16	1,05		1,074 / 1,034	no	100
	11b	mg/kg	40	15	09.05.16	1,12	1,134 / 1,098		no	100
	12	mg/kg	38	65	-	-	-	-	-	-
	13	mg/kg	12	58	18.05.16	0,95	0,97	0,92	no	
	14a	mg/kg	24	63	24.05.	1,03	1,03	1,03	no	100-105
14b	mg/kg	24	63	13.05.	1,03	1,02	1,04	no	90-110	
15	mg/kg	19	61	11.05.16	0,95	0,95	0,95			

Parameter	Teilnehmer	Einheit	Proben-Nr. A	Proben-Nr. B	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis A	Ergebnis B	Inkl. WF	Wiederfindungsrate [%]
Analyte	Participant	Unit	Sample No. A	Sample No. B	Date of analysis	Result (Mean)	Result A	Result B	Incl. RR	Recovery rate [%]
Co	1	mg/kg	07	36	03.06.16	0,6	0,6	0,6	no	
	2	mg/kg	2	31	25.05.16	0,62	0,62	0,63	yes / no	100
	3	mg/kg	34	52						
	4	mg/kg	44	60	17.05.16	0,59	0,6	0,59	no	
	5	mg/kg	18	57	02.05.16	0,61	0,62	0,61		
	6	mg/kg	10	72	28.04.16	0,60	0,58	0,61	no	
	7	mg/kg	9	47	04.05.16	0,55	0,54	0,57	no	
	8	mg/kg	22	71	06.06.16	0,56	0,56	0,55	no	
	9	mg/kg	27	45	26.05.	0,49	0,49	0,49	no	
	10	mg/kg	39	68	06.06.	0,56	0,56	0,55	no	
	11a	mg/kg	40	15	10.05.16	0,63		0,636 / 0,617	no	100
	11b	mg/kg	40	15	09.05.16	0,62	0,627 / 0,62		no	100
	12	mg/kg	38	65	-	-	-	-	-	-
	13	mg/kg	12	58						
	14	mg/kg	24	63	24.05.	0,56	0,57	0,56	no	80
15	mg/kg	19	61	11.05.16	0,59	0,58	0,59			

Parameter	Teilnehmer	Einheit	Proben-Nr. A	Proben-Nr. B	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis A	Ergebnis B	Inkl. WF	Wiederfindungsrate [%]
Analyte	Participant	Unit	Sample No. A	Sample No. B	Date of analysis	Result (Mean)	Result A	Result B	Incl. RR	Recovery rate [%]
Cr	1	mg/kg	07	36	03.06.16	1,33	1,31	1,34	no	
	2	mg/kg	2	31	25.05.16	1,22	1,17	1,27	yes / no	100
	3	mg/kg	34	52	04.06.	1,12	1,12	1,11	no	106
	4	mg/kg	44	60	17.05.16	1,37	1,39	1,36	no	
	5	mg/kg	18	57	02.05.16	1,46	1,46	1,47		
	6	mg/kg	10	72	28.04.16	1,4	1,4	1,4	no	
	7	mg/kg	9	47	04.05.16	0,65	0,66	0,65	no	
	8	mg/kg	22	71	06.06.16	1,27	1,27	1,26	no	
	9	mg/kg	27	45	26.05.	0,96	0,93	0,98	no	
	10	mg/kg	39	68	17.05.	1,19	1,19	1,19	no	
	11a	mg/kg	40	15	10.05.16	1,04		0,979 / 1,094	no	100
	11b	mg/kg	40	15	09.05.16	1,23	1,184 / 1,278		no	100
	12a	mg/kg	38	65	18.05.16	1,85	1,85	1,85	no	-
	12b	mg/kg	38	65	23.05.16	1,28	1,18	1,38	no	-
	13	mg/kg	12	58	18.05.16	1,62	1,61	1,64	no	
14	mg/kg	24	63	24.05.	1,13	1,13	1,12	no	90-110	
15	mg/kg	19	61	11.05.16	0,63	0,62	0,65			

Parameter	Teilnehmer	Einheit	Proben-Nr. A	Proben-Nr. B	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis A	Ergebnis B	Inkl. WF	Wiederfindungsrate [%]
Analyte	Participant	Unit	Sample No. A	Sample No. B	Date of analysis	Result (Mean)	Result A	Result B	Incl. RR	Recovery rate [%]
Cu	1	mg/kg	07	36	03.06.16	5,76	5,7	5,83	no	
	2	mg/kg	2	31	25.05.16	5,67	5,62	5,72	yes / no	100
	3	mg/kg	34	52	04.06.	5,83	5,64	6,01	no	96
	4	mg/kg	44	60	17.05.16	4,58	5,68	3,47	no	
	5	mg/kg	18	57	02.05.16	5,76	5,74	5,78		
	6	mg/kg	10	72	28.04.16	6,1	5,9	6,2	no	
	7	mg/kg	9	47	23.05.16	5,24	5,32	5,16	no	
	8	mg/kg	22	71	06.06.16	6,09	6,2	5,98	no	
	9	mg/kg	27	45	03.06.	6,03	6,11	5,95	no	
	10	mg/kg	39	68	17.05.	5,23	5,3	5,15	no	
	11a	mg/kg	40	15	10.05.16	6,17		6,31 / 6,02	no	100
	11b	mg/kg	40	15	09.05.16	6,13	6,211 / 6,054		no	100
	12a	mg/kg	38	65	18.05.16	5,41	5,33	5,48	no	-
	12b	mg/kg	38	65	23.05.16	5,5	5,45	5,54	no	-
	13	mg/kg	12	58	18.05.16	7,2	7,1	7,3	no	
14	mg/kg	24	63							
15	mg/kg	19	61	11.05.16	5,6	5,52	5,67			

Parameter	Teilnehmer	Einheit	Proben-Nr. A	Proben-Nr. B	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis A	Ergebnis B	Inkl. WF	Wiederfindungsrate [%]
Analyte	Participant	Unit	Sample No. A	Sample No. B	Date of analysis	Result (Mean)	Result A	Result B	Incl. RR	Recovery rate [%]
Fe	1	mg/kg	07	36	03.06.16	333	330	336	no	
	2	mg/kg	2	31	25.05.16	310	304	315	yes / no	100
	3	mg/kg	34	52						
	4	mg/kg	44	60	17.05.16	314,33	326,45	302,2	no	
	5	mg/kg	18	57	02.05.16	310,65	309,8	311,5		
	6	mg/kg	10	72	28.04.16	348	363	334	no	
	7	mg/kg	9	47	23.05.16	250	248	251	no	
	8	mg/kg	22	71	10.05.16	304,5	303	305,9	no	
	9	mg/kg	27	45	03.06.	321	321	320	no	
	10	mg/kg	39	68	17.05.	297	303	291	no	
	11a	mg/kg	40	15	10.05.16	299,4		291,3 / 307,5	no	100
	11b	mg/kg	40	15	09.05.16	311,65	306,4 / 316,9		no	100
	12a	mg/kg	38	65	18.05.16	310,6	309,49	311,71	no	-
	12b	mg/kg	38	65	23.05.16	314,62	313,6	315,64	no	-
	13	mg/kg	12	58	21.05.16	254	253	255	no	
14	mg/kg	24	63	24.05.	300	301,7	298,1	no	90-110	
15	mg/kg	19	61	11.05.16	217,97	209,59	226,36			

Parameter	Teilnehmer	Einheit	Proben-Nr. A	Proben-Nr. B	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis A	Ergebnis B	Inkl. WF	Wiederfindungsrate [%]
Analyte	Participant	Unit	Sample No. A	Sample No. B	Date of analysis	Result (Mean)	Result A	Result B	Incl. RR	Recovery rate [%]
I	1a	mg/kg	07	36	03.06.16	9,96	10,1	9,82	no	
	1b	mg/kg	07	36	03.06.16	14,2	14,8	13,6	no	-
	2	mg/kg	2	31	25.05.16	9,7	9,64	9,76	yes / no	100
	3	mg/kg	34	52						
	4	mg/kg	44	60	17.05.16	2,7	2,62	2,77	no	
	5	mg/kg	18	57						
	6	mg/kg	10	72	28.04.16	10,5	10,5	10,5	no	
	7	mg/kg	9	47	03.05.16	<100	<100	<100	no	
	8	mg/kg	22	71	28.04.16	2,1	2,12	2,08	no	
	9	mg/kg	27	45	12.05.	11,4	11,44	11,36	no	
	10	mg/kg	39	68	18.05.	11,8	11,9	11,6	no	
	11	mg/kg	40	15	04.05.16	10,65	10,905 / 10,388			
	12	mg/kg	38	65	-	-	-	-	-	-
	13	mg/kg	12	58						
	14	mg/kg	24	63						
15	mg/kg	19	61							

Parameter	Teilnehmer	Einheit	Proben-Nr. A	Proben-Nr. B	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis A	Ergebnis B	Inkl. WF	Wiederfindungsrate [%]
Analyte	Participant	Unit	Sample No. A	Sample No. B	Date of analysis	Result (Mean)	Result A	Result B	Incl. RR	Recovery rate [%]
Li	1	mg/kg	07	36	03.06.16	1,45	1,44	1,45	no	
	2	mg/kg	2	31	25.05.16	1,4	1,32	1,47	yes / no	100
	3	mg/kg	34	52						
	4	mg/kg	44	60	17.05.16	11,72	11,77	11,68	no	
	5	mg/kg	18	57	02.05.16	1,47	1,31	1,63		
	6	mg/kg	10	72	28.04.16	1,4	1,6	1,3	no	
	7	mg/kg	9	47	23.05.16	1,56	1,55	1,57	no	
	8	mg/kg	22	71						
	9	mg/kg	27	45	26.05.	1,27	1,27	1,26	no	
	10	mg/kg	39	68		not analysed				
	11a	mg/kg	40	15	10.05.16	1,44		1,513 / 1,366	no	100
	11b	mg/kg	40	15		2,44	2,407 / 2,477		no	100
	12	mg/kg	38	65	-	-	-	-	-	-
	13	mg/kg	12	58						
	14	mg/kg	24	63						
15	mg/kg	19	61							

Parameter	Teilnehmer	Einheit	Proben-Nr. A	Proben-Nr. B	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis A	Ergebnis B	Inkl. WF	Wiederfindungsrate [%]
Analyte	Participant	Unit	Sample No. A	Sample No. B	Date of analysis	Result (Mean)	Result A	Result B	Incl. RR	Recovery rate [%]
Mn	1	mg/kg	07	36	03.06.16	9,04	9,03	9,05	no	
	2	mg/kg	2	31	25.05.16	9,1	8,94	9,2	yes / no	100
	3	mg/kg	34	52	04.06.	9,1	8,71	9,55	no	104
	4	mg/kg	44	60	17.05.16	8,38	8,82	7,93	no	
	5	mg/kg	18	57	02.05.16	9,66	9,6	9,72		
	6	mg/kg	10	72	28.04.16	10	11	10	no	
	7	mg/kg	9	47	23.05.16	7,28	7,22	7,33	no	
	8	mg/kg	22	71	06.06.16	8,38	8,37	8,4	no	
	9	mg/kg	27	45	03.06.	9,34	9,43	9,25	no	
	10	mg/kg	39	68	23.05.	8,64	8,62	8,66	no	
	11a	mg/kg	40	15	10.05.16	9,54		9,71 / 9,37	no	100
	11b	mg/kg	40	15		8,7	8,709 / 8,696		no	100
	12a	mg/kg	38	65	18.05.16	8,29	8,17	8,4	no	-
	12b	mg/kg	38	65	23.05.16	8,86	8,74	8,98	no	-
	13	mg/kg	12	58	31.05.16	7,4	7,6	7,3	no	
14	mg/kg	24	63	24.05.	8,73	8,66	8,79	no	90-110	
15	mg/kg	19	61	11.05.16	8,29	8,34	8,23			

Parameter	Teilnehmer	Einheit	Proben-Nr. A	Proben-Nr. B	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis A	Ergebnis B	Inkl. WF	Wiederfindungsrate [%]
Analyte	Participant	Unit	Sample No. A	Sample No. B	Date of analysis	Result (Mean)	Result A	Result B	Incl. RR	Recovery rate [%]
Mo	1	mg/kg	07	36	03.06.16	0,5	0,5	0,51	no	
	2	mg/kg	2	31	25.05.16	0,96	1,01	0,92	yes / no	100
	3	mg/kg	34	52						
	4	mg/kg	44	60	17.05.16	0,55	0,59	0,5	no	
	5	mg/kg	18	57	02.05.16	0,52	0,52	0,53		
	6	mg/kg	10	72	28.04.16	0,55	0,57	0,54	no	
	7	mg/kg	9	47	23.05.16	<1	<1	<1	no	
	8	mg/kg	22	71	06.06.16	0,49	0,49	0,49	no	
	9	mg/kg	27	45	26.05.	0,43	0,42	0,43	no	
	10	mg/kg	39	68	19.05.	0,54	0,54	0,53	no	
	11a	mg/kg	40	15	10.05.16	0,55		0,553 / 0,552	no	100
	11b	mg/kg	40	15		0,58	0,601 / 0,549		no	100
	12	mg/kg	38	65	-	-	-	-	-	-
	13	mg/kg	12	58						
	14	mg/kg	24	63						
15	mg/kg	19	61	11.05.16	0,54	0,54	0,54			

Parameter	Teilnehmer	Einheit	Proben-Nr. A	Proben-Nr. B	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis A	Ergebnis B	Inkl. WF	Wiederfindungsrate [%]
Analyte	Participant	Unit	Sample No. A	Sample No. B	Date of analysis	Result (Mean)	Result A	Result B	Incl. RR	Recovery rate [%]
Ni	1	mg/kg	07	36	03.06.16	1,6	1,59	1,61	no	
	2	mg/kg	2	31	25.05.16	1,67	1,56	1,78	yes / no	100
	3	mg/kg	34	52	04.06.	1,44	1,39	1,48	no	113
	4	mg/kg	44	60	17.05.16	1,23	1,32	1,14	no	
	5	mg/kg	18	57	02.05.16	1,66	1,68	1,64		
	6	mg/kg	10	72	28.04.16	1,5	1,4	1,5	no	
	7	mg/kg	9	47	04.05.16	1,12	1,09	1,15	no	
	8	mg/kg	22	71	06.06.16	1,41	1,42	1,4	no	
	9	mg/kg	27	45	26.05.	1,06	1,05	1,07	no	
	10	mg/kg	39	68	17.05.	1,34	1,35	1,32	no	
	11a	mg/kg	40	15	10.05.16	1,57		1,59 / 1,57	no	100
	11b	mg/kg	40	15		1,22	1,212 / 1,232		no	100
	12a	mg/kg	38	65	18.05.16	1,8	1,81	1,79	no	-
	12b	mg/kg	38	65	23.05.16	1,38	1,41	1,35	no	-
	13	mg/kg	12	58	19.05.16	1,37	1,35	1,39	no	
14	mg/kg	24	63	24.05.	1,28	1,29	1,27	no	90-110	
15	mg/kg	19	61	11.05.16	1,16	1,15	1,17			

Parameter	Teilnehmer	Einheit	Proben-Nr. A	Proben-Nr. B	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis A	Ergebnis B	Inkl. WF	Wiederfindungsrate [%]
Analyte	Participant	Unit	Sample No. A	Sample No. B	Date of analysis	Result (Mean)	Result A	Result B	Incl. RR	Recovery rate [%]
Pb	1	mg/kg	07	36	03.06.16	0,34	0,34	0,34	no	
	2	mg/kg	2	31	25.05.16	0,35	0,36	0,37	yes / no	100
	3	mg/kg	34	52	04.06.	0,17	0,18	0,15	no	115
	4	mg/kg	44	60	17.05.16	0,27	0,26	0,29	no	
	5	mg/kg	18	57	02.05.16	0,33	0,34	0,33		
	6	mg/kg	10	72	28.04.16	0,34	0,34	0,34	no	
	7	mg/kg	9	47	04.05.16	0,32	0,33	0,32	no	
	8	mg/kg	22	71	06.06.16	0,38	0,39	0,38	no	
	9	mg/kg	27	45	26.05.	0,24	0,23	0,25	no	
	10	mg/kg	39	68	17.05.	0,32	0,33	0,31	no	
	11a	mg/kg	40	15	10.05.16	0,35		0,36 / 0,3495	no	100
	11b	mg/kg	40	15		0,34	0,329 / 0,348		no	100
	12	mg/kg	38	65	-	-	-	-	-	-
	13	mg/kg	12	58	19.05.16	0,12	12	0,12	no	
	14a	mg/kg	24	63	24.05.	0,34	0,34	0,34	no	90-110
14b	mg/kg	24	63	12.05.	0,29	0,3	0,29	no	90-110	
15	mg/kg	19	61	11.05.16	0,28	0,28	0,29			

Parameter	Teilnehmer	Einheit	Proben-Nr. A	Proben-Nr. B	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis A	Ergebnis B	Inkl. WF	Wiederfindungsrate [%]
Analyte	Participant	Unit	Sample No. A	Sample No. B	Date of analysis	Result (Mean)	Result A	Result B	Incl. RR	Recovery rate [%]
Rb	1	mg/kg	07	36	03.06.16	3,98	4	3,96	no	
	2	mg/kg	2	31	25.05.16	3,8	3,62	3,97	yes / no	100
	3	mg/kg	34	52						
	4	mg/kg	44	60	17.05.16					
	5	mg/kg	18	57						
	6	mg/kg	10	72	28.04.16	3,9	4	3,9	no	
	7	mg/kg	9	47	04.05.16	3,42	3,38	3,45	no	
	8	mg/kg	22	71						
	9	mg/kg	27	45						
	10	mg/kg	39	68		not analysed				
	11	mg/kg	40	15	10.05.16					
	12a	mg/kg	38	65	18.05.16	1,67	1,68	1,65	no	-
	12b	mg/kg	38	65	23.05.16	1,89	1,91	1,87	no	-
	13	mg/kg	12	58						
	14	mg/kg	24	63						
15	mg/kg	19	61							

Parameter	Teilnehmer	Einheit	Proben-Nr. A	Proben-Nr. B	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis A	Ergebnis B	Inkl. WF	Wiederfindungsrate [%]
Analyte	Participant	Unit	Sample No. A	Sample No. B	Date of analysis	Result (Mean)	Result A	Result B	Incl. RR	Recovery rate [%]
Se	1	mg/kg	07	36	03.06.16	2,01	2,01	2,01	no	
	2	mg/kg	2	31	25.05.16	2,4	2,01	2,84	yes / no	100
	3	mg/kg	34	52						
	4	mg/kg	44	60	17.05.16	1,68	1,69	1,67	no	
	5	mg/kg	18	57	02.05.16	3,28	3,15	3,42		
	6	mg/kg	10	72	28.04.16	1,8	1,9	1,7	no	
	7	mg/kg	9	47	04.05.16	1,78	1,76	1,79	no	
	8	mg/kg	22	71	31.05.16	1,71	1,7	1,72	no	
	9	mg/kg	27	45	13.05.	1,67	1,69	1,65	no	
	10	mg/kg	39	68	18.05.	1,82	1,82	1,82	no	
	11a	mg/kg	40	15	10.05.16	1,85		1,839 / 1,86	no	100
	11b	mg/kg	40	15		2,38	2,524 / 2,243		no	100
	12a	mg/kg	38	65	18.05.16	1,45	1,45	1,45	no	-
	12b	mg/kg	38	65	23.05.16	1,45	1,45	1,44	no	-
	13	mg/kg	12	58	24.05.16	0,51	0,51	0,51	no	
14	mg/kg	24	63	24.05.	1,53	1,51	1,54	no	90-100	
15	mg/kg	19	61	11.05.16	3,09	3,17	3,01			

Parameter	Teilnehmer	Einheit	Proben-Nr. A	Proben-Nr. B	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis A	Ergebnis B	Inkl. WF	Wiederfindungsrate [%]
Analyte	Participant	Unit	Sample No. A	Sample No. B	Date of analysis	Result (Mean)	Result A	Result B	Incl. RR	Recovery rate [%]
Ti	1	mg/kg	07	36	03.06.16	29	29,6	28,3	no	
	2	mg/kg	2	31	25.05.16				yes / no	100
	3	mg/kg	34	52						
	4	mg/kg	44	60	17.05.16					
	5	mg/kg	18	57						
	6	mg/kg	10	72						
	7	mg/kg	9	47	26.05.16	6,65	6,59	6,71	no	
	8	mg/kg	22	71						
	9	mg/kg	27	45						
	10	mg/kg	39	68	25.05.	29,9	30,2	29,6	no	
	11	mg/kg	40	15	10.05.16	18,1	16,03 / 20,17		no	100
	12a	mg/kg	38	65	18.05.16	23,6	23,34	23,85	no	-
	12b	mg/kg	38	65	23.05.16	22,21	23,76	20,65	no	-
	13	mg/kg	12	58						
	14	mg/kg	24	63						
15	mg/kg	19	61							

Parameter	Teilnehmer	Einheit	Proben-Nr. A	Proben-Nr. B	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis A	Ergebnis B	Inkl. WF	Wiederfindungsrate [%]
Analyte	Participant	Unit	Sample No. A	Sample No. B	Date of analysis	Result (Mean)	Result A	Result B	Incl. RR	Recovery rate [%]
V	1	mg/kg	07	36	03.06.16	1,47	1,48	1,46	no	
	2	mg/kg	2	31	25.05.16	1,9	1,86	1,86	yes / no	100
	3	mg/kg	34	52						
	4	mg/kg	44	60	17.05.16	1,93	1,93	1,92	no	
	5	mg/kg	18	57						
	6	mg/kg	10	72	28.04.16	1,4	1,5	1,4	no	
	7	mg/kg	9	47	23.05.16	<1	<1	<1	no	
	8	mg/kg	22	71	06.06.16	1,23	1,24	1,22	no	
	9	mg/kg	27	45	26.05.	1,06	1,04	1,07	no	
	10	mg/kg	39	68	20.05.	1,34	1,33	1,34	no	
	11a	mg/kg	40	15	10.05.16	1,42		1,35 / 1,49	no	100
	11b	mg/kg	40	15		1,02	0,955 / 1,088		no	100
	12	mg/kg	38	65	-	-	-	-	-	-
	13	mg/kg	12	58	12.05.16	1,5	1,5	1,5	no	
	14	mg/kg	24	63	24.05.	1,24	1,24	1,24	no	90-110
15	mg/kg	19	61							

Parameter	Teilnehmer	Einheit	Proben-Nr. A	Proben-Nr. B	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis A	Ergebnis B	Inkl. WF	Wiederfindungsrate [%]
Analyte	Participant	Unit	Sample No. A	Sample No. B	Date of analysis	Result (Mean)	Result A	Result B	Incl. RR	Recovery rate [%]
Y	1a	mg/kg	07	36	03.06.16	0,1	0,1	0,1	no	
	1b	mg/kg	07	36	25.05.16	0,09	0,08	0,1	yes / no	100
	2	mg/kg	2	31						
	3	mg/kg	34	52	17.05.16					
	4	mg/kg	44	60						
	5	mg/kg	18	57						
	6	mg/kg	10	72	03.05.16	<1	<1	<1	no	
	7	mg/kg	9	47						
	8	mg/kg	22	71						
	9	mg/kg	27	45		not analysed				
	10	mg/kg	39	68	10.05.16					
	11	mg/kg	40	15	-	-	-	-	-	-
	12	mg/kg	38	65						
	13	mg/kg	12	58						
	14	mg/kg	24	63						
15	mg/kg	19	61	03.06.16	0,1	0,1	0,1	no		

Parameter	Teilnehmer	Einheit	Proben-Nr. A	Proben-Nr. B	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis A	Ergebnis B	Inkl. WF	Wiederfindungsrate [%]
Analyte	Participant	Unit	Sample No. A	Sample No. B	Date of analysis	Result (Mean)	Result A	Result B	Incl. RR	Recovery rate [%]
Zn	1	mg/kg	07	36	03.06.16	53,6	55	52,3	no	
	2	mg/kg	2	31	25.05.16	41	39,9	42,2	yes / no	100
	3	mg/kg	34	52						
	4	mg/kg	44	60	17.05.16	62,86	70,39	55,33	no	
	5	mg/kg	18	57	02.05.16	51,07	50,93	51,21		
	6	mg/kg	10	72	28.04.16	51	52	49	no	
	7	mg/kg	9	47	23.05.16	46,16	46,35	45,97	no	
	8	mg/kg	22	71	10.05.16	50,01	49,87	50,14	no	
	9	mg/kg	27	45	03.06.	49,8	50,1	49,6	no	
	10	mg/kg	39	68	17.05.	50	51,2	48,7	no	
	11	mg/kg	40	15	10.05.16	59,07	59,27 / 58,86			
	12a	mg/kg	38	65	18.05.16	50,11	50,14	50,08	no	-
	12b	mg/kg	38	65	23.05.16	52,15	52,28	52,01	no	-
	13	mg/kg	12	58	31.05.16	48,1	48,7	47,4	no	
	14	mg/kg	24	63	24.05.	45,21	45,82	44,59	no	85-90
15	mg/kg	19	61	11.05.16	56,95	56,91	57			

5.2 Homogeneity

5.2.1 Homogeneity of bottled PT-samples

Homogeneity test of copper by ICP-OES (VDLUFA III, 10.8.2):

Independent samples	mg/kg
1	5,96
2	6,03
3	5,21
4	5,57
5	5,97

Mean 5,75
Repeatability Standard Deviation 0,352 6,12%

5.2.2 Comparison of sample numbers / test results and trend line

By comparison of the increasing sample numbers and the measurement results, the homogeneity of the chronological bottled PT item can be characterized with the help of the trend line function:

Al - Aluminium	
Target standard deviation σ_{pt}'	65,9
Sample numbers	7 - 71
Total numbers of samples	16
Slope:	-0,0388
Trend line range	455,93 - 455,31 mg/kg
Deviation trend line	455,62 ± 0,310 mg/kg
Percent of σ_{pt}'	0,47 %

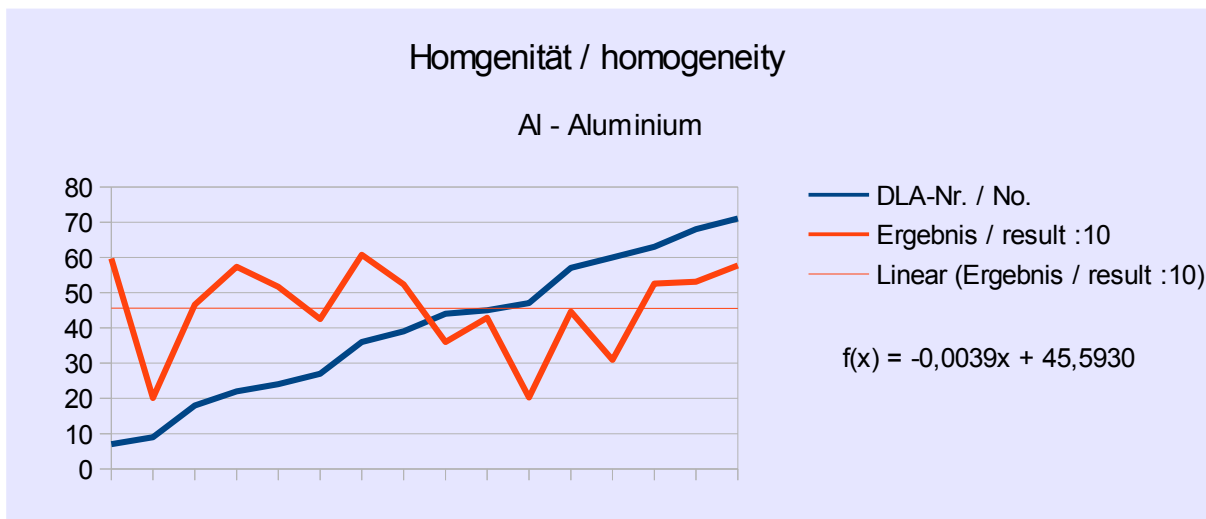


Abb. 37: Trendfunktion Probennummern / Al Ergebnisse (1/10 dargestellt)
Fig. 37: trend line function sample number / Al results (x 10 shown)

Pb - Lead	
Target standard deviation σ_{pt}	0,0592
Sample numbers	2 - 72
Total numbers of samples	24
Slope:	-0,0012
Trend line range	0,3234 - 0,2956 mg/kg
Deviation trend line	0,3095 \pm 0,0139 mg/kg
Percent of σ_{pt}	23,5 %

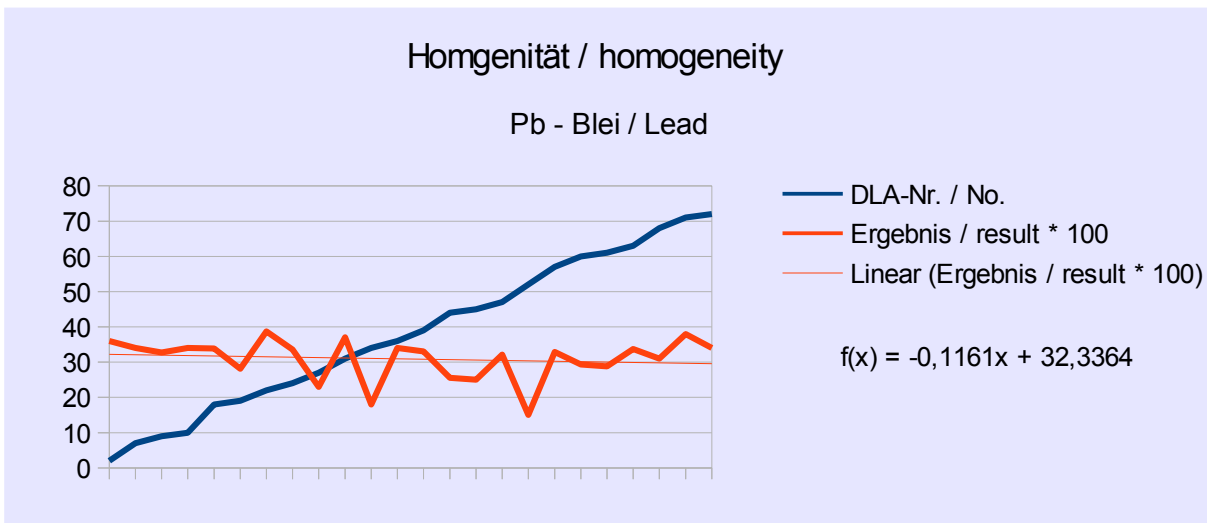


Abb. 38: Trendfunktion Probennummern / Pb Ergebnisse (*100 dargestellt)
Fig. 38: trend line function sample number / Pb results (*100 shown)

5.3 Kernel Density Plots of Results

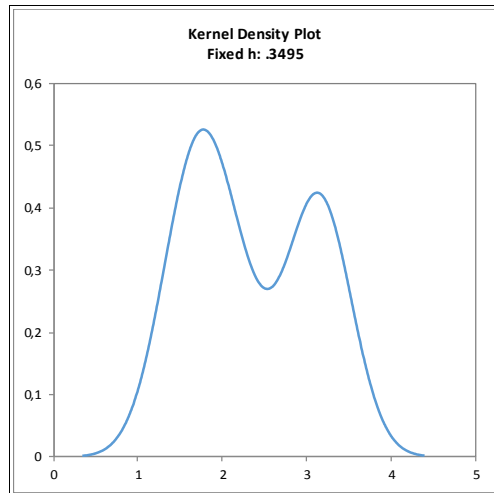
Abbildungen:

Kerndichte-Schätzungen der Teilnehmerergebnisse (mit $h = 0,75 \times \sigma_{pt}$ von X_{pt})

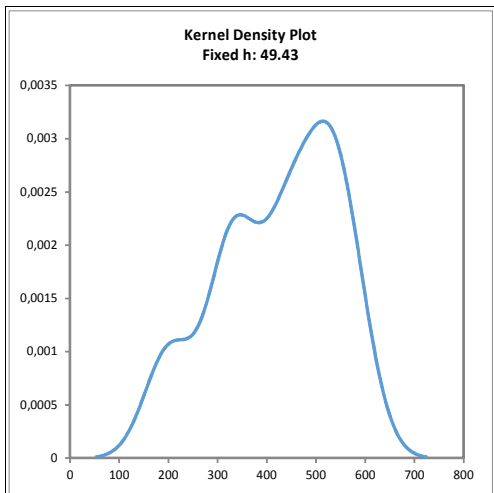
Figures:

Kernel density plots of participants' results (with $h = 0,75 \times \sigma_{pt}$ of X_{pt})

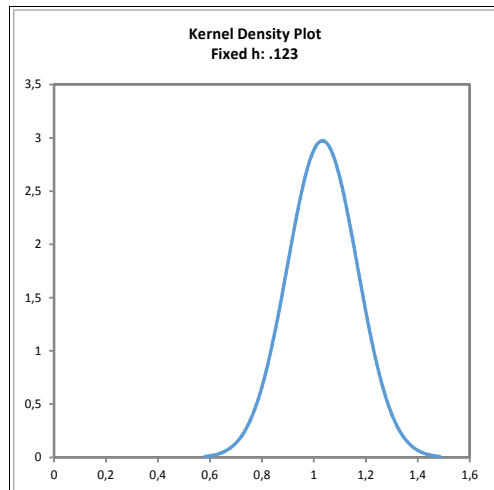
Ba - Barium



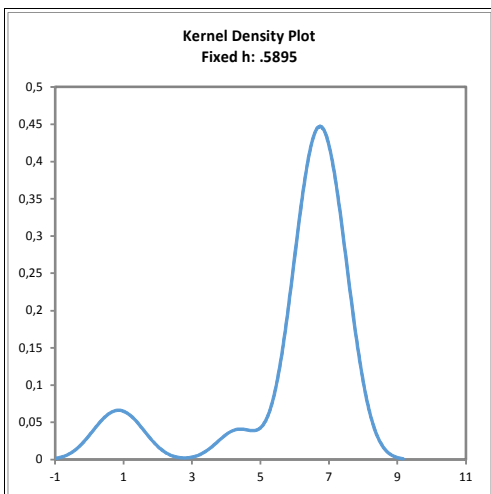
Al - Aluminium



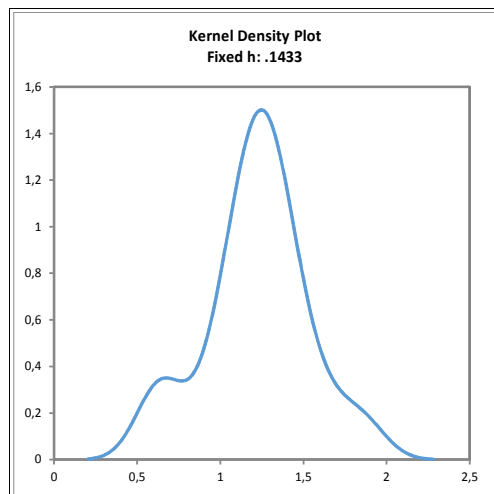
Cd - Cadmium



As - Arsen / Arsenic



Cr - Chrom / Chromium



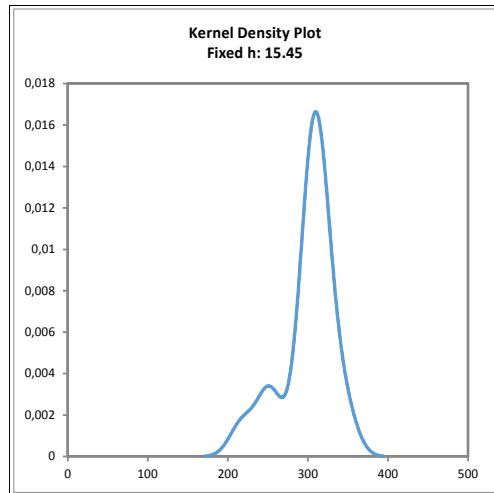
Abbildungen:

Kerndichte-Schätzungen der Teilnehmerergebnisse (mit $h = 0,75 \times \sigma_{pt}$ von X_{pt})

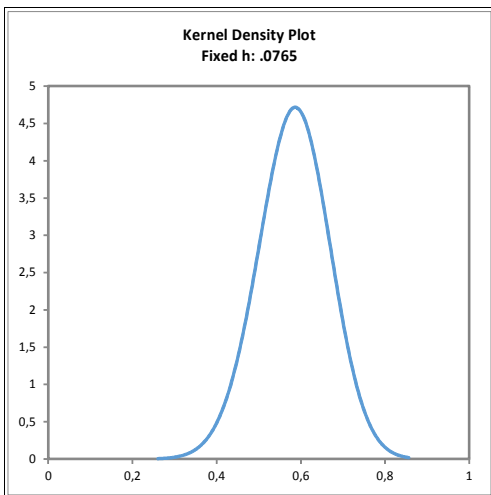
Figures:

Kernel density plots of participants' results (with $h = 0,75 \times \sigma_{pt}$ of X_{pt})

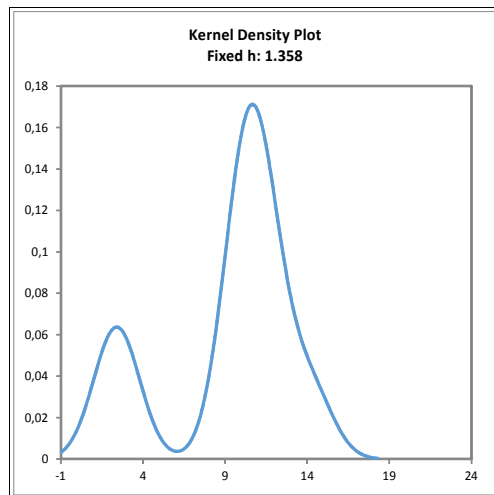
Fe - Eisen / Iron



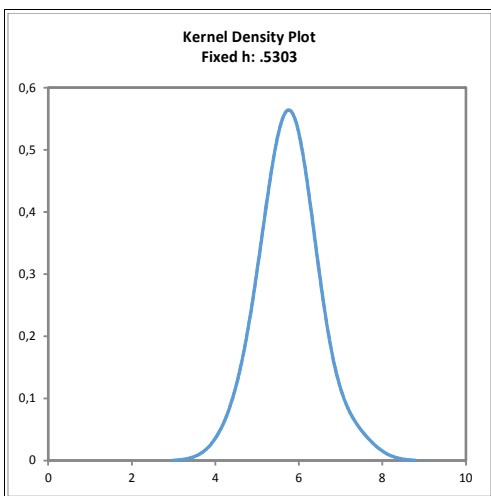
Co - Kobalt / Cobalt



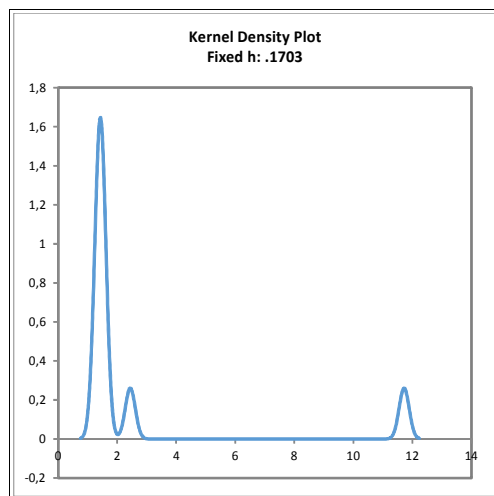
I - Iod / Iodine



Cu - Kupfer / Copper



Li - Lithium



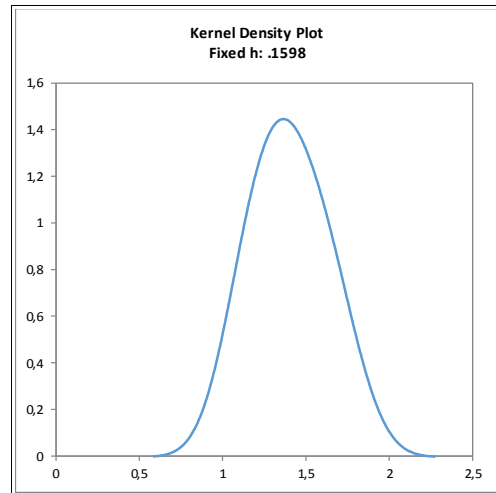
Abbildungen:

Kerndichte-Schätzungen der Teilnehmerergebnisse (mit $h = 0,75 \times \sigma_{pt}$ von X_{pt})

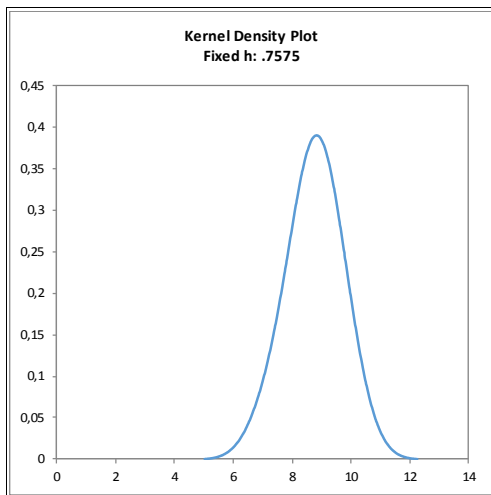
Figures:

Kernel density plots of participants' results (with $h = 0,75 \times \sigma_{pt}$ of X_{pt})

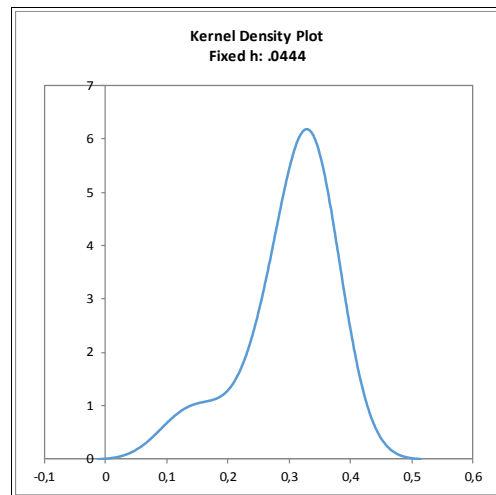
Ni - Nickel



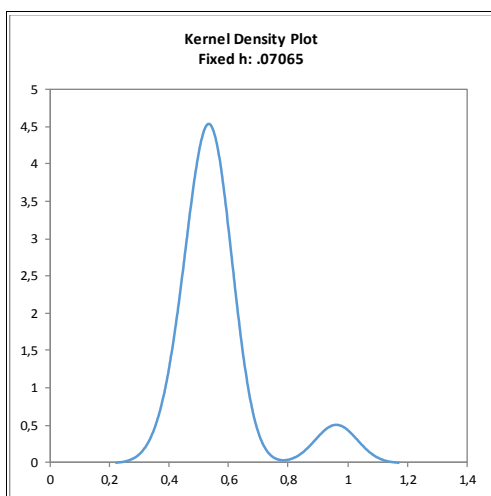
Mn - Mangan / Manganese



Pb - Blei / Lead



Mo - Molybdän / Molybdenum



Rb - Rubidium

< 8 Ergebnisse
< 8 Results

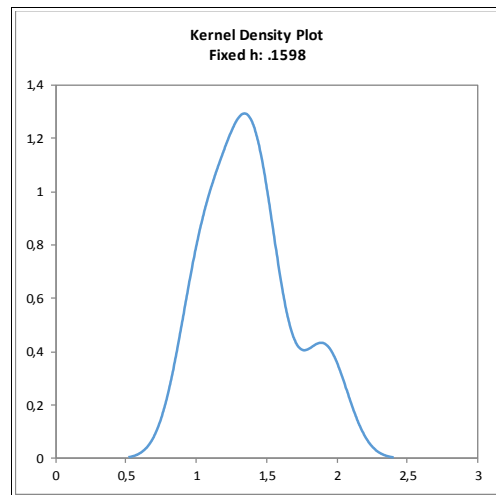
Abbildungen:

Kerndichte-Schätzungen
der Teilnehmerergebnisse
(mit $h = 0,75 \times \sigma_{pt}$ von X_{pt})

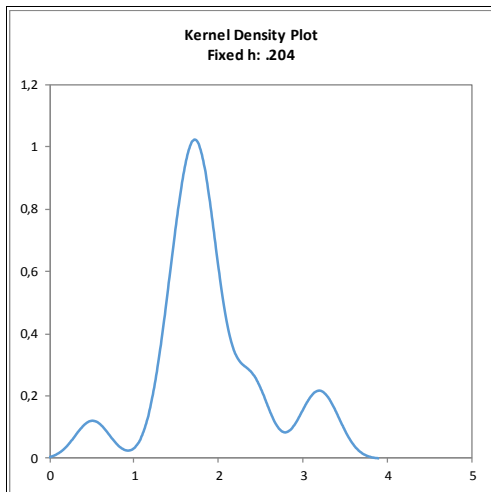
Figures:

Kernel density plots
of participants' results
(with $h = 0,75 \times \sigma_{pt}$ of X_{pt})

V - Vanadium



Se - Selen / Selenium



Y - Yttrium

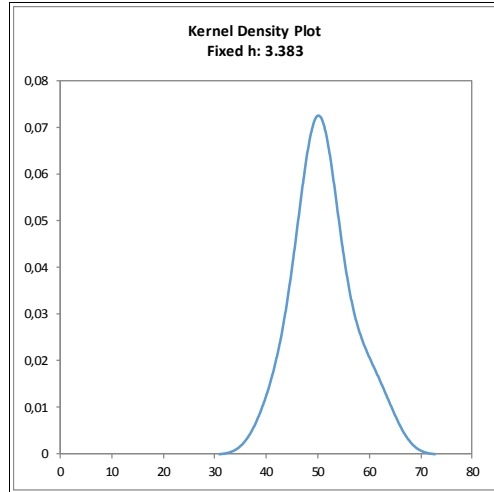
< 8 Ergebnisse

< 8 Results

Ti - Titan / Titanium

Zn - Zink / Zinc

< 8 Ergebnisse
< 8 Results



5.4 Analytical Methods

Details by the participants

Teilnehmer	Elemente	Methodenbeschreibung	Homogenisierung	Einwaage	Aufschlussmethode	Aufschlusslösung	Referenzmaterial	Kalibrierverfahren	Methode akkreditiert	Sonstige Hinweise
Participant	Elements	Method description	Homogenization	Sample weight	Hydrolization	Hydrolization solution	Referencematerial	Calibration	Method accredited	Further remarks
1	Al, Fe	ICP-OES	yes	0,5 g	microwave	HNO3/H2O2		extern	yes	
1	As, Ba, Cd, Co, Cr, Cu, Li, Mn, Mo, Ni, Pb, Rb, Se, Ti, V, Y, Zn	ICP-MS	yes	0,5 g	microwave	HNO3/H2O2		extern	yes	
1a	I	DIN EN 15111	yes	0,5 g	Extraktion mit TMAH			extern	yes	
1b	I	Photometry	yes	0,05 g	Schöninger-combustion; photometric determination of iodine in combustion solution		ERM BD-151	extern	yes	
2	Al, As, Ba, Cd, Co, Cr, Cu, Fe, I, Li, Mn, Mo, Ni, Pb, Rb, Se, Ti, V, Y, Zn	EN 15763: Food stuff – determination of traces of elements after pressure digestion	yes	400 mg	microwave pressure digestion			linear	yes	
3	As	DIN EN 14627 ASU L 00.00-19/6	shaking	0,5 g	ASU L 00.00-19/6	aqua regia	Plankon BCR-414	multiple points	yes	
3	Cd, Pb	DIN EN 14083 ASU L 00.00-19/3	shaking	0,5 g	ASU L 00.00-19/3	H2O2/HNO3	Plankon BCR-414	multiple points	yes	
3	Cr	DIN EN 1233 E 10-4	shaking	0,5 g	ASU L 00.00-19/3	H2O2/HNO3	Plankon BCR-414	multiple points	yes	
3	Cu	DIN EN 14084	shaking	0,5 g	ASU L 00.00-19/3	H2O2/HNO3	Plankon BCR-414	multiple points	yes	
3	Mn	DIN 38406 E 33-2	shaking	0,5 g	ASU L 00.00-19/3	H2O2/HNO3	Plankon BCR-414	multiple points	yes	
3	Ni	DIN 38406-E 11-2	shaking	0,5 g	ASU L 00.00-19/3	H2O2/HNO3	Plankon BCR-414	multiple points	yes	
4									yes	
5	Al, Ba, Cd, Co, Cr, Cu, Fe, Li, Mn, Mo, Ni, Pb, Rb, Zn	DIN EN ISO 17294-2	shaking	0,4	HNO3, H2O2		yes	6-Punkt-Kal.	yes	
6	As, Ba, Cd, Co, Cr, Cu, Fe, Li, Mn, Mo, Ni, Pb, Rb, Se, V, Zn	ICPMS after microwave pressure digestion	IKA-mill MF10	0,2-0,5 g	Mikrowellenpressure digestion (HNO3/H2O2)	HNO3	Rice flour, peach leaves, lichen	Standard-calibration	yes	
6	I	ICPMS after microwave pressure digestion	IKA-mill MF10	0,5 g	Mikrowellenpressure digestion (HNO3/H2O2)	TMAH	milk powder	Standard-calibration	yes	
7	Al, Ba, Cu, Fe, I, Li, Mn, Mo, Ti, V, Y, Zn	ICPAES		0,5g	Digiprep®	HNO3+H2O2			no	
7	As, Cd, Co, Cr, Ni, Pb, Rb, Se	ICPMS		0,5g	Digiprep®	HNO3+H2O2			yes	
8	Al, Fe, Zn	DIN EN ISO 11885	stirred with spatula	0,5g	pressure digestion 300 °C	H2O/HNO3/H2O2	plant, water	5-Point, Standard calibration	yes	
8	As, Ba, Cd, Co, Cr, Cu, I, Mn, Mo, Ni, Pb, V	DIN EN ISO 17294-2	stirred with spatula	0,5g	pressure digestion 300 °C	H2O/HNO3/H2O2	plant, water	3-Point, Standard calibration	yes	
8	Se	§ 64 LFGB L 00.00-19	stirred with spatula	0,5g	pressure digestion 300 °C	H2O/HNO3/H2O2	plant, water	7-Point, Standard calibration	yes	

Teilnehmer	Elemente	Methodenbeschreibung	Homogenisierung	Einwaage	Aufschlussmethode	Aufschlusslösung	Referenzmaterial	Kalibrierverfahren	Methode akkreditiert	Sonstige Hinweise
Participant	Elements	Method description	Homogenization	Sample weight	Hydrolization	Hydrolization solution	Referencematerial	Calibration	Method accredited	Further remarks
9	Al, Ba, Cu, Fe, Mn, Zn	DIN EN ISO 11885 (E22) (09.2009)	yes	0,5 g	microwave digestion	HNO ₃ / H ₂ O ₂	yes		yes	
9	As, Co, Cr, Li, Mo, Ni, Pb, V	DIN EN ISO 17294 - E 29 (02.2005)	yes	0,5 g	microwave digestion	HNO ₃ / H ₂ O ₂	yes		yes	
9	I	DIN EN 15111 (06.2007)	yes	0,5 g	TMAH-Extract	TMAH	yes		yes	
9	Se	DIN 38405 (D 23) (10.1194)	yes	0,5 g	microwave digestion	HNO ₃ / H ₂ O ₂	yes		yes	
10	Al, As, Ba, Cd, Co, Cr, Cu, Fe, Mn, Mo, Ni, Pb, Se, Ti, V, Zn	by ICP-MS after microwave digestion	mixing	0,2 g	microwave	HNO ₃	Al, Cr, Ni, Zn: tea leaves (TL); As, Ba, Cd, Mn, Mo, Se: bush branches and leaves (NCS); Pb: Soya flour	ext. by IS In	yes	
10	I	Iod (ICP-MS), DIN EN 15111, 2007-06 (mod.)	mixing	0,2 g	microwave	TMAH	milk powder	ext. by IS Te	yes	
11a	As, Ba, Cd, Co, Cr, Cu, Fe, Li, Mn, Mo, Ni, Pb, Se, V	ICP-MS	Grindomix	0,59 g	microwave	HN03 + H2O2	PK food supplement		As, Cd, Cu, Fe, Pb: yes; Ba, Cr, Li, Mn, Mo, Ni, Se, V: no	
11b	Al, As, Ba, Cd, Co, Cr, Cu, Fe, Li, Mn, Mo, Ni, Pb, Se, Ti, V, Zn	ICP-OES	Grindomix	0,59 g	microwave	HN03 + H2O2	PK food supplement		Al, Zn: yes	
11	I	ICPMS		160 mg	TMAH	4-5 % TMAH	milk powder	0 - 5 - 20 -50 - 100 - 200 ppb	no	Jens Sloth
12a	As, Cr, Cu, Fe, Mn, Ni, Rb, Se, Ti, Zn	total X-ray fluorescence analysis in-house method	mixing by hand	100 mg	wet grinding, ball mill	20%ige HNO ₃	Roth, Multi-Element	ISTD (Ga)	no	
12b	As, Cd, Cu, Fe, Mn, Ni, Rb, Se, Ti, Zn	total X-ray fluorescence analysis in-house method	ball mill, homogenisator	400 mg	acid digestion	65%ige HNO ₃ und H ₂ O ₂	Roth, Multi-Element	ISTD (Ga)	no	
13	As, Cd, Cr, Cu, Ni, Pb, V	DIN EN ISO 15586 E4		ca. 1 g	pressure digestion	HNO ₃	none	5 Point	yes	
13	Fe	DIN 38406 E-32		ca. 1 g	pressure digestion	HNO ₃	none	5 Point	yes	
13	Mn	DIN 38406 E33-1		ca. 1 g	pressure digestion	HNO ₃	none	5 Point	yes	
14a	Al, As, Cd, Co, Cr, Fe, Mn, Ni, Pb, Se, V, Zn	ICP-MS after microwave digestion	mechanical mixing	80-120 mg	microwave digestion	no additional digestion	DORM-4, IAEA-407, DLA 41/2015	5-Point	yes	
14b	Cd, Pb	GF-ZAAS after microwave digestion	mechanical mixing	80-120 mg	microwave digestion	no additional digestion	DORM 5	5-Point	yes	
15	As, Cd, Co, Cr, Cu, Fe, Mn, Mo, Ni, Pb, Se, Zn	AA 53, ICP-MS	ball mill	1g	microwave	HNO ₃ + H ₂ O ₂	NIST Standard Reference Material 3280		yes	

6. Index of participant laboratories in alphabetical order

Teilnehmer / Participant	Ort / Town	Land / Country
		Germany
		Germany
		Germany
		Germany
		BELGIUM
		Germany
		Germany
		BELGIUM
		Germany
		Germany
		Germany
		Germany
		Germany
		Germany
		Germany
		Germany

[Die Adressdaten der Teilnehmer wurden für die allgemeine Veröffentlichung des Auswerte-Berichts nicht angegeben.]

[The address data of the participants were deleted for publication of the evaluation report.]

7. Index of references

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17. ASU §64 L 00.00-158 (2016-2): Bestimmung von Aluminium in Lebensmitteln mit der optischen Emmissionsspektrometrie mit induktiv gekoppeltem Plasma (ICP-OES)
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19. ASU §64 L 00.00-19/2: Bestimmung von Eisen, Kupfer, Mangan und Zink mit der Atomabsorptionsspektrometrie (AAS) in der Flamme
20. ASU §64 L 00.00-19/3 / DIN EN 14083: Bestimmung von Blei, Cadmium, Chrom und Molybdän mit Graphitofen-Atomabsorptionsspektrometrie (GFAAS) nach Druckaufschluss / Foodstuffs. Determination of trace elements. Determination of lead, cadmium, chromium and molybdenum by graphite furnace atomic

- absorption spectrometry (GFAAS) after pressure digestion
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