

Proficiency Tests

**DLA**

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**Evaluation Report**

proficiency test

**DLA 46/2017**

**Heavy Metals and Trace Elements:  
in Plant Food**

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**General Information on the proficiency test (PT)**

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<i>Vertraulichkeit</i> <i>Confidentiality</i>	<p>Die Teilnehmerergebnisse sind im EP-Bericht in anonymisierter Form mit Auswertenummern benannt. Daten einzelner Teilnehmer werden ausschließlich nach vorheriger Zustimmung des Teilnehmers an Dritte weitergegeben.  Participant result are named anonymously with evaluation numbers in the PT report. Data of individual participants will be passed on to third parties only with prior consent of the participant.</p>

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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 a common in commerce plant-based food potato powder (suitable for the production of mashed potatoes within a dietary nutrition "potato diet") from an European provider with natural contents of relevant elements. The raw material was crushed using a centrifugal mill (mesh < 500 µm) and afterwards mechanically homogenized. The composition is given in Table 1 below.

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

Table 1: Composition of DLA-Samples

Ingredients	Content
Potato-Powder Ingredients: Potatoes, E471, E304, E223, E100 Nutrition per 100 g: protein 8,3 g, salt 0,15 g, fat 0,6 g, carbohydrates 76 g	100 g/100g

**Note:** The metrological traceability of temperature, mass and volume during production of the PT samples is ensured by DAkkS calibrated reference materials.

### 2.1.1 Homogeneity

The **mixture homogeneity before bottling** was examined 8-fold by determination of copper by LC/MS (DIN EN ISO 17294-2). The repeatability standard deviation was 2,97% and thus within the range of repeatability standard deviations of comparable methods (e.g. ASU §64 L 00.00-144, s. 3.6.1). The results of homogeneity analysis are given in the documentation.

The calculation of the **repeatability standard deviations  $S_r$  of the participants** was also used as an indicator of homogeneity. For all parameters with more than 7 results they are in the range of 1,15% to 5,35%. Iron is an exception ( $S_r$  10,0%). Thus they were similar to the repeatability standard deviations of the corresponding official methods (e.g. ASU §64 L 00.00-144, s. 3.6.2) (see Tab. 3) [16-19]. The repeatability standard deviations of the participants' results are given in the documentation in the statistic data (see 4.1 and 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 line were 0,0458 mg/kg for copper and 0,302 µg/kg for cadmium and thus in the range of up to approx. 20% of the target standard deviation  $\sigma_{pt}$  or  $\sigma_{pt}'$  (s. 5.2 Homogeneity) and can therefore be regarded as low.

In case the criterion for sufficient homogeneity of the test items is not fulfilled the impact on the target standard deviation will be verified. If necessary the evaluation of results will be done considering the standard uncertainty of the assigned value by z'-scores (s. 3.8 and 3.11) [3].

### 2.1.2 Stability

The experience with various DLA reference materials showed good storage stability with respect to the durability of the sample (spoilage) and the content of the PT parameters for comparable food matrices and water activity ( $a_w$  value <0,5). The stability of the sample material is therefore given during the investigation period under consideration of given storage conditions.

## 2.2 Sample shipment and information to the test

Two portions of test material were sent to every participating laboratory in the 15<sup>th</sup> week of 2017. The testing method was optional. The tests should be finished at 26<sup>st</sup> May 2017 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 potato powder with a mostly natural content of 20 elements to be determined quantitatively.*

*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.*

**Please note the attached information on the proficiency test.**

(see documentation, section 5.4 Information on the PT)

## 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.

All 11 participants submitted their results 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.

***In the present evaluation statistic data for parameters with a minimum of 5 values were given for information also. It should be considered that the significance can be limited due to the low number of results.***

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  $\sigma_{pt}$  (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  $VK_R$  in percent of the mean is given as variation coefficient in the statistical data of participant for each parameter. The significance of  $VK_R$  is further explained in section 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]. Even if a result clearly deviates from the robust mean (e.g. factor >10) and has an influence on the robust statistics, a result can be excluded from statistical evaluation [3].

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  $\sigma_{pt}$  (= standard deviation for proficiency assessment) can be determined according to the following methods.

If an acceptable quotient  $S^*/\sigma_{pt}$  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 methods are applied by the participants. On the other hand the target standard deviation from the evaluation of precision data of an 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.

***For valuation of all elements in the present PT the target standard deviation according to the general model of Horwitz was applied (see 3.6.1).***

***Additionally for Al the standard uncertainty was considered by evaluating with z'-scores (see 3.8).***

***Due to the number of < 5 the results for Cr, Rb and S were not evaluated with z-scores.***

#### 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  $\sigma_R$  [6]. Later the model was modified by Thompson for certain concentration ranges [10]. The reproducibility standard deviation  $\sigma_R$  can be applied as the relative target standard deviation  $\sigma_{pt}$  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 Precision experiment

Using the reproducibility standard deviation  $\sigma_R$  and the repeatability standard deviation  $\sigma_r$  of a precision experiment (collaborative trial or proficiency test) the target standard deviation  $\sigma_{pt}$  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 relative repeatability standard deviations ( $RSD_r$ ) and relative reproducibility standard deviation ( $RSD_R$ ) given in Table 2 were determined in ring tests using the indicated methods.

The resulting target standard deviations  $\sigma_{pt}$ , which were identified there, were used to evaluate the results and to provide additional information for the statistical data.

**Table 2:** Relative repeatability standard deviations ( $RSD_r$ ) and relative reproducibility standard deviations ( $RSD_R$ ) according to selected evaluations of tests for precision and the resulting target standard deviation  $\sigma_{pt}$  [16-26]

Parameter	Matrix	Mean	$RSD_r$	$RSD_R$	$\sigma_{pt}$	Method / Literature
Al	Cocoa powder	205	3,25%	5,83%	5,36% <sup>1</sup>	ICP-MS [16]
	Cocoa powder	210	1,91%	8,71%	8,61%	ICP-OES [17]
As	Fish homogenate	1,6	4,6%	8,8%	8,18%	ICP-MS [18]
	Mussels	9,3	4,5%	13%	12,6%	ICP-MS [18]
Pb	Fish homogenate	2,1	5,0%	8%	7,18%	ICP-MS [18]
	Mussels	2,5	13%	16%	13,1%	ICP-MS [18]
Ca	Lobster	183	4,90%	6,31%	5,27%	ICP-OES [22]
	Children's food soy	6191	3,41%	7,97%	7,60% <sup>1</sup>	ICP-OES [22]
Cd	Fish homogenate	0,87	7,3%	11%	9,71% <sup>1</sup>	ICP-MS [18]
	Mussels	1,7	3,9%	9,5%	9,09%	ICP-MS [18]
Cr	Baby food	0,17	7,3%	19%	18,3%	GF-AAS [20]
	Rice powder	0,11	19,2%	35%	32,3% <sup>1</sup>	GF-AAS [20]
Cu	Lobster	16,40	5,72%	6,82%	5,49%	ICP-OES [22]
	Children's food soy	4,51	4,30%	11,06%	10,6% <sup>1</sup>	ICP-OES [22]
Fe	Lobster	12,1	6,45%	8,59%	7,28%	ICP-OES [22]
	Children's food soy	77	2,75%	6,98%	6,70% <sup>1</sup>	ICP-OES [22]
I	Codfish muscles	4,15	0,7%	8,9%	8,89%	ICP-MS (16)
	Soy food	1,26	3,7%	6,7%	6,17%	ICP-MS (16)
K	Lobster	871	3,63%	6,27%	5,71%	ICP-OES [22]
	Children's food soy	6733	4,08%	5,49%	4,67% <sup>1</sup>	ICP-OES [22]
Mn	Lobster	1,20	4,74%	7,95%	7,21%	ICP-OES [22]
	Children's food soy	2,19	4,67%	13,7%	13,3% <sup>1</sup>	ICP-OES [22]
Mg	Lobster	85	3,73%	8,63%	8,21%	ICP-OES [22]
	Children's food soy	599	4,30%	7,64%	7,01% <sup>1</sup>	ICP-OES [22]
Mo	Baby food	0,50	6,6%	21%	20,5%	GF-AAS [20]
	Rice powder	0,56	8,7%	20%	19,0% <sup>1</sup>	GF-AAS [20]
Na	Lobster	186	3,31%	6,60%	6,17%	ICP-OES [22]
	Children's food soy	2220	3,67%	4,89%	4,15% <sup>1</sup>	ICP-OES [22]
P	Lobster	973	3,16%	7,13%	6,78%	ICP-OES [22]
	Children's food soy	4129	3,45%	7,87%	7,48% <sup>1</sup>	ICP-OES [22]
S	Lobster	876	3,13%	7,54%	7,21%	ICP-OES [22]
	Children's food soy	1234	3,86%	10,71%	10,4%	ICP-OES [22]
Se	Katfish	1,797	9,85%	10,1%	7,31%	AAS [21]
	Rice	0,374	2,41%	11,8%	11,7%	AAS [21]
Zn	Lobster	13,9	4,63%	7,90%	7,19%	ICP-OES (22)
	Children's food soy	43,5	2,60%	6,89%	6,64%	ICP-OES (22)

<sup>1</sup> used in evaluation (s. chapter 4)

### 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 3 shows selected statistic data of participants results of present PT compared to PT results of previous years.

### 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 ( $\sigma_{pt}$ ) 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

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

The valid z-Score for each parameter is indicated as z-Score ( $\sigma_{pt}$ ). The value indicated as z-Score (Info) only obtains a informative character. The both z-Scores were calculated with the different target standard deviations in accordance with 3.6.

#### 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  $\geq 10$  results [3].

**Table 3:** Characteristics of the present PT (on grey) in comparison to previous PTs since 2016 (SD = standard deviation, CV = coefficient of variation)

Parameter	Matrix (Powder)	robust Mean [g/100g]	rob. SD (S*) [g/100g]	rel. SD (VK <sub>S*</sub> ) [%]	Quotient S*/σ <sub>pt</sub>	DLA-report
Al	Mussel-Fish	444	143	32,2%	2,2 <sup>1</sup>	DLA 58/2016
Al	Potatoes	0,527	0,283	53,7%	1,5 <sup>1</sup>	DLA 46/2017
B	Potatoes	3,88	0,689	17,8%	1,4	DLA 46/2017
Ba	Mussel-Fish	2,35	0,832	35,5%	1,8 <sup>1</sup>	DLA 58/2016
Ba	Potatoes	0,220	-	-	-	DLA 46/2017
Ca	Potatoes	238	12,0	5,04%	0,72	DLA 46/2017
Cd	Mussel-Fish	1,03	0,0616	5,97%	0,37	DLA 58/2016
Cd	Potatoes	0,0399	0,0029	7,30%	0,28	DLA 46/2017
Cr	Mussel-Fish	1,23	0,266	21,6%	1,4	DLA 58/2016
Cr	Potatoes	**	-	-	-	DLA 46/2017
Co	Mussel-Fish	0,586	0,0347	5,91%	0,34	DLA 58/2016
Co	Potatoes	0,0110	0,00223	20,3%	0,64	DLA 46/2017
Cu	Mussel-Fish	5,75	0,439	7,63%	0,62	DLA 58/2016
Cu	Potatoes	1,98	0,117	5,90%	0,41	DLA 46/2017
Fe	Mussel-Fish	305	22,1	7,24%	1,1	DLA 58/2016
Fe	Potatoes	15,0	1,22	8,10%	0,76	DLA 46/2017
K	Potatoes	13162	604	4,59%	1,2	DLA 46/2017
Mg	Potatoes	736	27,1	3,68%	0,62	DLA 46/2017
Mn	Mussel-Fish	8,79	0,696	7,93%	0,69	DLA 58/2016
Mn	Potatoes	3,66	0,327	8,9%	0,68	DLA 46/2017
Mo	Mussel-Fish	0,536	0,0400	7,45%	0,42	DLA 58/2016
Mo	Potatoes	0,197	0,0161	8,2%	0,40	DLA 46/2017
Na	Potatoes	195	13,7	7,03%	1,0	DLA 46/2017
Ni	Mussel-Fish	1,40	0,232	16,6%	1,1	DLA 58/2016
Ni	Potatoes	0,0398	0,00645	16,2%	0,62	DLA 46/2017
P	Potatoes	1451	49,1	3,38%	0,63	DLA 46/2017
S	Potatoes	1000	-	-	-	DLA 46/2017
Sr	Potatoes	0,720	0,0336	4,67%	0,28	DLA 46/2017
Rb	Mussel-Fish	**	-	-	-	DLA 58/2016
Rb	Potatoes	**	-	-	-	DLA 46/2017
Zn	Mussel-Fish	51,0	5,17	10,2%	1,1	DLA 58/2016
Zn	Potatoes	7,83	0,726	9,3%	0,79	DLA 46/2017

<sup>1</sup> with target standard deviation σ<sub>pt</sub>'

\*\* no statistical evaluation (< 7 or < 5 results)

### 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 ( $\sigma_{pt}$ ) 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  $\sigma_{pt}'$ .

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)

The variation coefficient (CV) 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 gives the relative variability within a data region. While a low CV, e.g. <5-10% can be taken as evidence for a homogeneous set of results, a CV 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 levels or the performance evaluation of the participating laboratories possibly can not be done [3].

### 3.10 Quotient $S^*/\sigma_{pt}$

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  $\sigma_{pt}$  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 of the assigned value

Every assigned value has a standard uncertainty that depends on the analytical method, differences between the analytical methods used, the test material, the number of participating laboratories (P) and on other factors. The standard uncertainty ( $U_{(x_{pt})}$ ) 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 assigned value needs not to be included in the interpretation of the results of the PT [3]. Values exceeding 0,3 imply, that the target standard deviation could be too low with respect to 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 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.

### Comments to the statistic data:

For Cr, Rb and S there were < 5 results, therefore no statistical evaluation could be done.

For Al, Ba, Co, Ni and Sr there were < 7 results, thus the significance of the statistical evaluation is limited due to the low number of results, especially for Al (s. below).

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

For information the target standard deviation using statistical data obtained from precision experiments (ASU §64 method) was additionally given, when available.

For Al the distribution of results showed an increased variability. The quotient  $S^*/\sigma_{pt}$  was clearly higher than 2,0. Thus the parameter was evaluated considering the standard uncertainty by z'-scores. The quotient  $S^*/\sigma_{pt}'$  was then 1,5 (s. Tab. 4).

For the other parameters the distribution of results showed a low to normal variability. The quotients  $S^*/\sigma_{pt}$  were all in the range of 0,3 to 1,5 (s. Tab. 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 increased for Al, B, Ba, Co, K and Na with > 0,3 (0,4 to 0,6). For all other parameters the quotient was low (< 0,3).

80% to 100% of results were in the regarding target range.

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:

<b>Statistic Data</b>
<i>Number of results</i>
<i>Number of outliers</i>
Mean
Median
Robust mean ( $X_{pt}$ )
Robust standard deviation ( $S^*$ )
<i>Number with m replicate measurements</i>
Repeatability standard deviation ( $S_r$ )
Coefficient of Variation ( $CV_r$ ) in %
Reproducibility standard deviation ( $S_R$ )
Coefficient of Variation ( $CV_R$ ) in %
<i>Target range:</i>
Target standard deviation $\sigma_{pt}$ or $\sigma_{pt}'$
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 ( $X_{pt} + 2\sigma_{pt}'$ ) *
Variation coefficient $V_K$ in %
Quotient $S^*/\sigma_{pt}$ or $S^*/\sigma_{pt}'$
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 table below, the results of the participating laboratories are formatted in 3 valid digits\*\*:

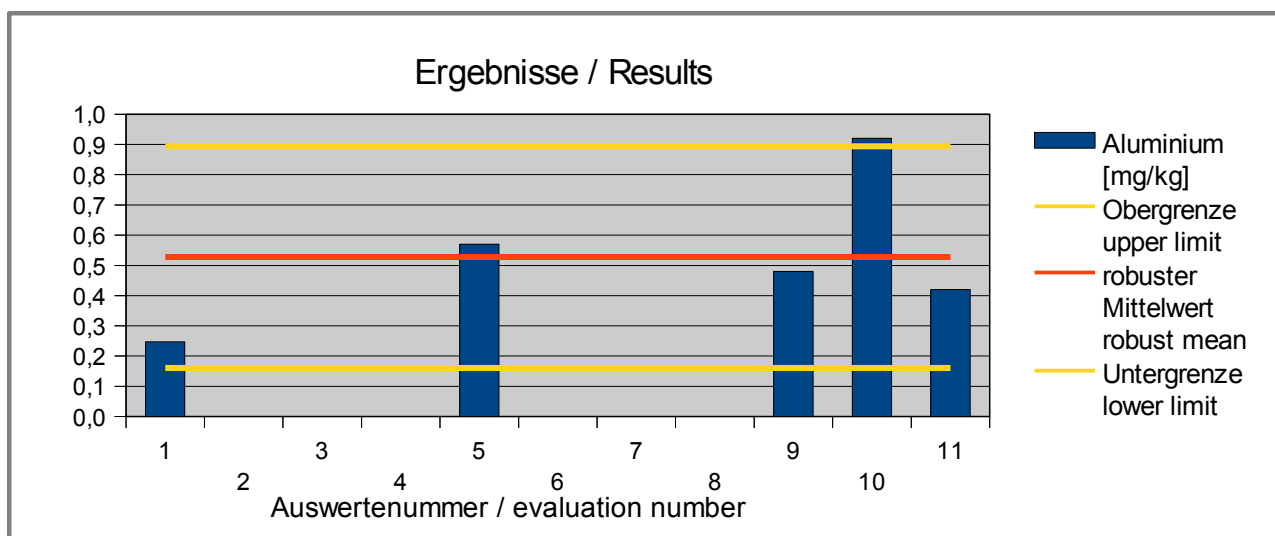
<b>Auswertenummer</b>	<b>Parameter [Einheit / Unit]</b>	<b>Abweichung</b>	<b>z-Score <math>\sigma_{pt}</math></b>	<b>z-Score (Info)</b>	<b>Hinweis</b>
<b>Evaluation number</b>		<b>Deviation</b>			<b>Remark</b>

\*\* In the documentation part, the results are given as they were transmitted by the participants.

**4.1 Al - Aluminium in mg/kg**

**Vergleichsuntersuchung / Proficiency Test**

<b>Statistic Data</b>	
Number of results	5
Number of outliers	0
Mean	0,527
Median	0,480
<b>Robust Mean (X)</b>	<b>0,527</b>
<b>Robust standard deviation (S*)</b>	<b>0,283</b>
Number with 2 replicates	4
Repeatability SD ( $S_r$ )	0,0263
Repeatability ( $CV_r$ )	5,07%
Reproducibility SD ( $S_R$ )	0,290
Reproducibility ( $CV_R$ )	55,9%
<i>Target range:</i>	
<b>Target standard deviation <math>\sigma_{pt}</math></b>	<b>0,183</b>
Target standard deviation (for Information)	0,0283
<b>lower limit of target range</b>	<b>0,161</b>
<b>upper limit of target range</b>	<b>0,894</b>
Quotient $S^*/\sigma_{pt}$	1,5
Standard uncertainty $U(X_{pt})$	0,158
Quotient $U(X_{pt})/\sigma_{pt}$	0,86
Results in the target range	4
Percent in the target range	80%



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

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

Auswertenummer	Aluminium [mg/kg]	Abweichung [mg/kg]	z'-Score* (σ <sub>pt</sub> )	z'-Score* (Info)	Hinweis
Evaluation number		Deviation [mg/kg]			Remark
1	0,247	-0,280	-1,5	-9,9	
2	< 0,300				
3					
4					
5	0,570	0,0426	0,2	1,5	
6	< 2,00				
7	< 0,600				
8	< 1,00				
9	0,480	-0,0474	-0,3	-1,7	
10	0,920	0,393	2,1	13,9	
11	0,420	-0,107	-0,6	-3,8	

\*Significance of statistical evaluation is limited for Al (s. p. 17)

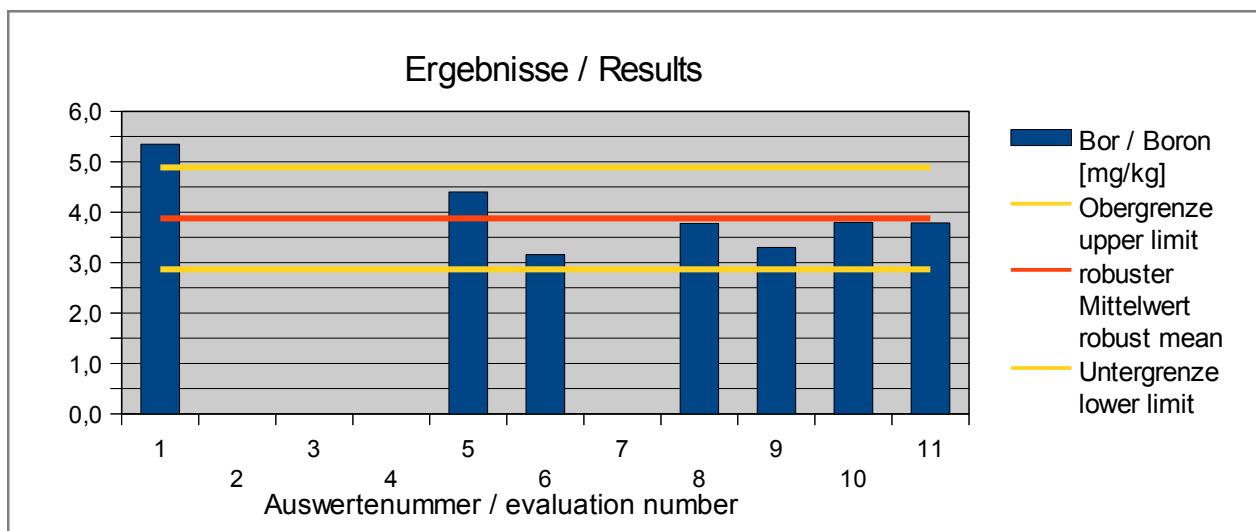


**Abb. / Fig. 2:** z'-Scores Aluminium

**4.2 B - Bor / Boron in mg/kg**

**Vergleichsuntersuchung / Proficiency Test**

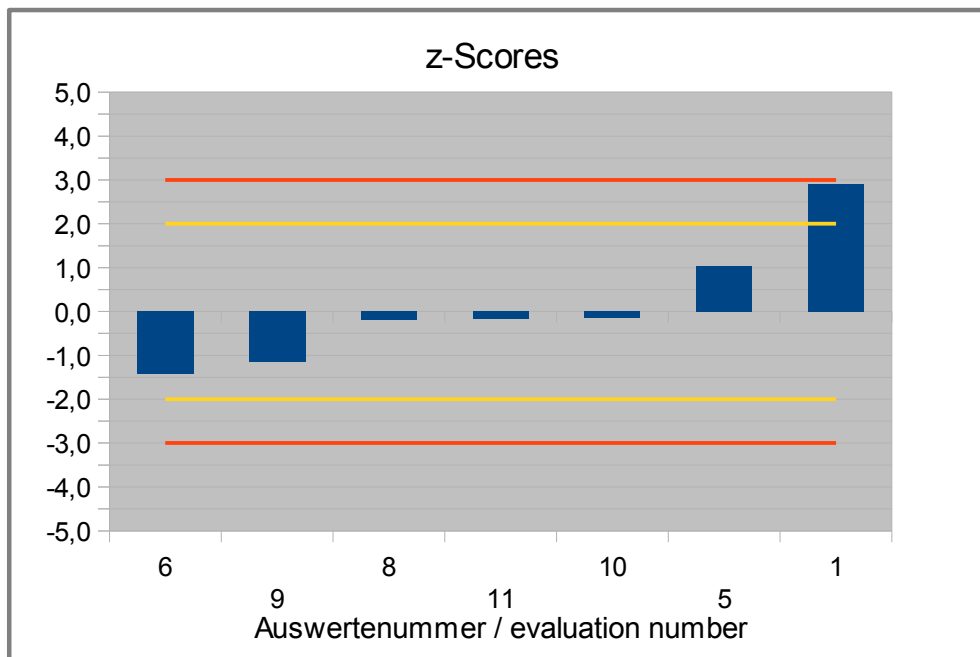
<b>Statistic Data</b>	
Number of results	7
Number of outliers	0
Mean	3,94
Median	3,79
<b>Robust Mean (X)</b>	<b>3,88</b>
<b>Robust standard deviation (S*)</b>	<b>0,689</b>
Number with 2 replicates	5
Repeatability SD ( $S_r$ )	0,224
Repeatability ( $CV_r$ )	5,64%
Reproducibility SD ( $S_R$ )	0,832
Reproducibility ( $CV_R$ )	21,0%
<i>Target range:</i>	
<b>Target standard deviation <math>\sigma_{pt}</math></b>	<b>0,506</b>
<b>lower limit of target range</b>	<b>2,86</b>
<b>upper limit of target range</b>	<b>4,89</b>
Quotient $S^*/\sigma_{pt}$	1,4
Standard uncertainty $U(x_{pt})$	0,326
Quotient $U(x_{pt})/\sigma_{pt}$	0,64
Results in the target range	6
Percent in the target range	86%



**Abb. / Fig. 3:** Ergebnisse Bor / Results Boron

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

Auswertenummer Evaluation number	Bor / Boron [mg/kg]	Abweichung [mg/kg] Deviation [mg/kg]	z-Score ( $\sigma_{pt}$ )	Hinweis Remark
1	5,35	1,47	2,9	
2				
3				
4				
5	4,40	0,523	1,0	
6	3,16	-0,721	-1,4	
7				
8	3,78	-0,0966	-0,2	
9	3,30	-0,577	-1,1	
10	3,80	-0,0766	-0,2	
11	3,79	-0,0866	-0,2	



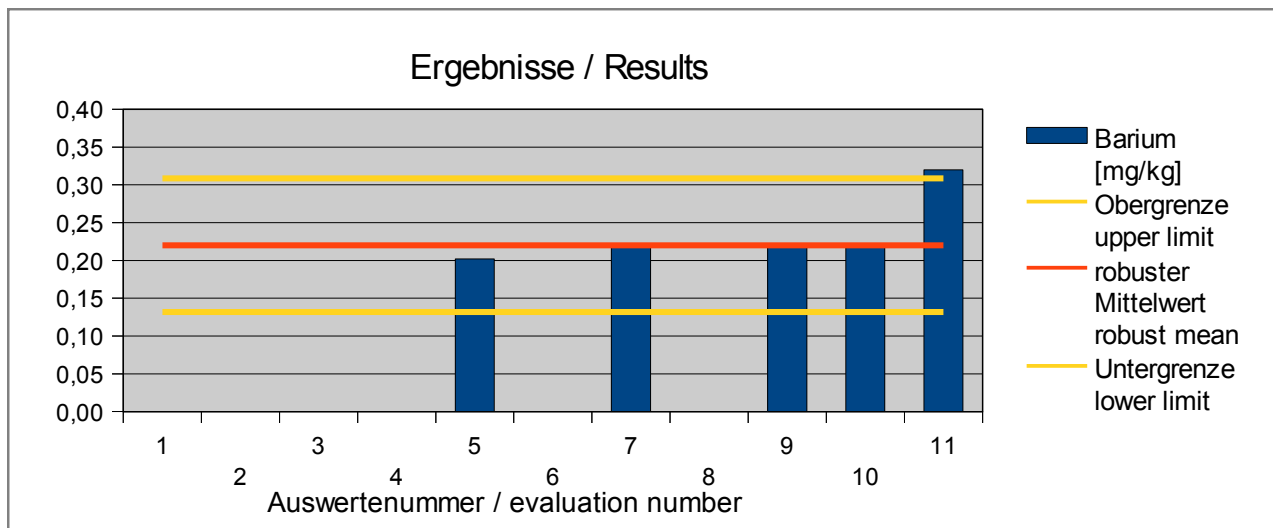
**Abb. / Fig. 4:** z-Scores Bor / Boron

**4.3 Ba - Barium in mg/kg**

**Vergleichsuntersuchung / Proficiency Test**

Statistic Data	
Number of results	5
Number of outliers	0
Mean	0,236
Median	0,220
<b>Robust Mean (X)</b>	<b>0,220</b>
<b>Robust standard deviation (S*)</b>	<b>-</b>
Number with 2 replicates	3
Repeatability SD ( $S_r$ )	0,0135
Repeatability ( $CV_r$ )	5,38%
Reproducibility SD ( $S_R$ )	0,0559
Reproducibility ( $CV_R$ )	22,2%
Target range:	
<b>Target standard deviation <math>\sigma_{pt}</math></b>	<b>0,0442</b>
<b>lower limit of target range</b>	<b>0,132</b>
<b>upper limit of target range</b>	<b>0,308</b>
Quotient $S_R/\sigma_{pt}$ <sup>1</sup>	1,3
Standard uncertainty $U(X_{pt})$ <sup>1</sup>	0,0312
Quotient $U(X_{pt})/\sigma_{pt}$	0,71
Results in the target range	4
Percent in the target range	80%

<sup>1</sup> calculated with the reproducibility standard deviation ( $S_R$ )



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

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

Auswertenummer	Barium [mg/kg]	Abweichung [mg/kg]	z-Score (σ <sub>pt</sub> )	Hinweis
Evaluation number		Deviation [mg/kg]		Remark
1				
2				
3				
4				
5	0,202	-0,0180	-0,4	
6	< 3,86			
7	0,220	0,0000	0,0	
8	< 0,500			
9	0,220	0,0000	0,0	
10	0,220	0,0000	0,0	
11	0,320	0,100	2,3	



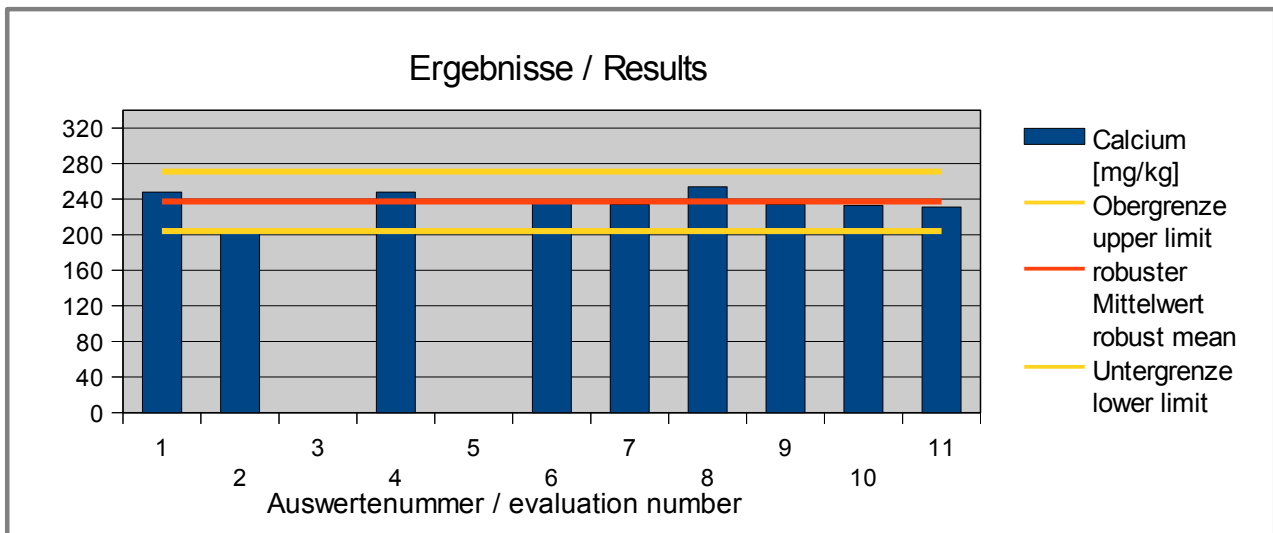
Abb. / Fig. 6: z-Scores Barium



**4.4 Ca - Calcium in mg/kg**

**Vergleichsuntersuchung / Proficiency Test**

<b>Statistic Data</b>	
Number of results	9
Number of outliers	0
Mean	236
Median	234
<b>Robust Mean (X)</b>	<b>238</b>
<b>Robust standard deviation (S*)</b>	<b>12,0</b>
Number with 2 replicates	9
Repeatability SD ( $S_r$ )	7,79
Repeatability ( $CV_r$ )	3,31%
Reproducibility SD ( $S_R$ )	15,7
Reproducibility ( $CV_R$ )	6,65%
<i>Target range:</i>	
<b>Target standard deviation <math>\sigma_{pt}</math></b>	<b>16,7</b>
Target standard deviation (for Information)	17,5
<b>lower limit of target range</b>	<b>204</b>
<b>upper limit of target range</b>	<b>271</b>
Quotient $S^*/\sigma_{pt}$	0,72
Standard uncertainty $U(x_{pt})$	5,01
Quotient $U(x_{pt})/\sigma_{pt}$	0,30
Results in the target range	8
Percent in the target range	89%



**Abb. / Fig. 7:** Ergebnisse Calcium / Results Caclium

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

Auswertenummer Evaluation number	Calcium [mg/kg]	Abweichung [mg/kg] Deviation [mg/kg]	z-Score ( $\sigma_{pt}$ )	z-Score (Info)	Hinweis Remark
1	248	10,4	0,6	0,6	
2	203	-34,6	-2,1	-2,0	
3					
4	248	10,4	0,6	0,6	
5					
6	237	-0,852	-0,1	0,0	
7	234	-3,60	-0,2	-0,2	
8	254	16,4	1,0	0,9	
9	234	-3,60	-0,2	-0,2	
10	233	-4,60	-0,3	-0,3	
11	231	-6,60	-0,4	-0,4	

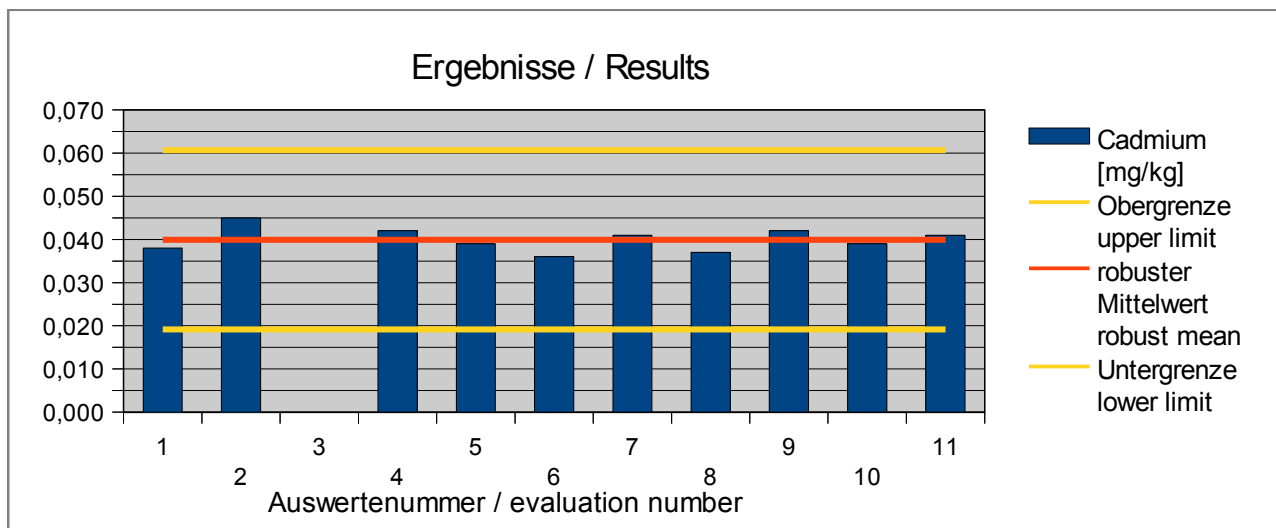


**Abb. / Fig. 8:** Z-Scores Calcium

**4.5 Cd - Cadmium in mg/kg**

**Vergleichsuntersuchung / Proficiency Test**

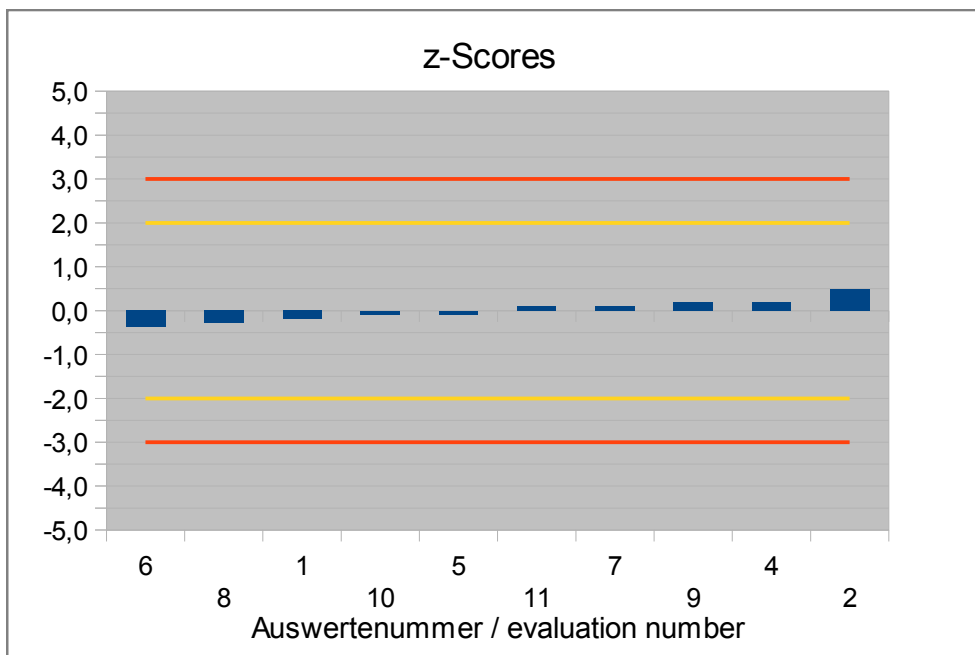
<b>Statistic Data</b>	
Number of results	10
Number of outliers	0
Mean	0,0400
Median	0,0400
<b>Robust Mean (X)</b>	<b>0,0399</b>
<b>Robust standard deviation (S*)</b>	<b>0,00291</b>
Number with 2 replicates	9
Repeatability SD ( $S_r$ )	0,00203
Repeatability ( $CV_r$ )	5,08%
Reproducibility SD ( $S_R$ )	0,00311
Reproducibility ( $CV_R$ )	7,79%
<i>Target range:</i>	
<b>Target standard deviation <math>\sigma_{pt}</math></b>	<b>0,0104</b>
Target standard deviation (for Information)	0,00388
<b>lower limit of target range</b>	<b>0,0192</b>
<b>upper limit of target range</b>	<b>0,0607</b>
Quotient $S^*/\sigma_{pt}$	0,28
Standard uncertainty $U(x_{pt})$	0,00115
Quotient $U(x_{pt})/\sigma_{pt}$	0,11
Results in the target range	10
Percent in the target range	100%



**Abb. / Fig. 9:** Ergebnisse Cadmium / Results Cadmium

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

Auswertenummer	Cadmium [mg/kg]	Abweichung [mg/kg]	z-Score (σ <sub>pt</sub> )	z-Score (Info)	Hinweis
Evaluation number		Deviation [mg/kg]			Remark
1	0,0380	-0,00193	-0,2	-0,5	
2	0,0450	0,00507	0,5	1,3	
3					
4	0,0420	0,00207	0,2	0,5	
5	0,0390	-0,000930	-0,1	-0,2	
6	0,0360	-0,00393	-0,4	-1,0	
7	0,0410	0,00107	0,1	0,3	
8	0,0370	-0,00293	-0,3	-0,8	
9	0,0420	0,00207	0,2	0,5	
10	0,0390	-0,000930	-0,1	-0,2	
11	0,0410	0,00107	0,1	0,3	

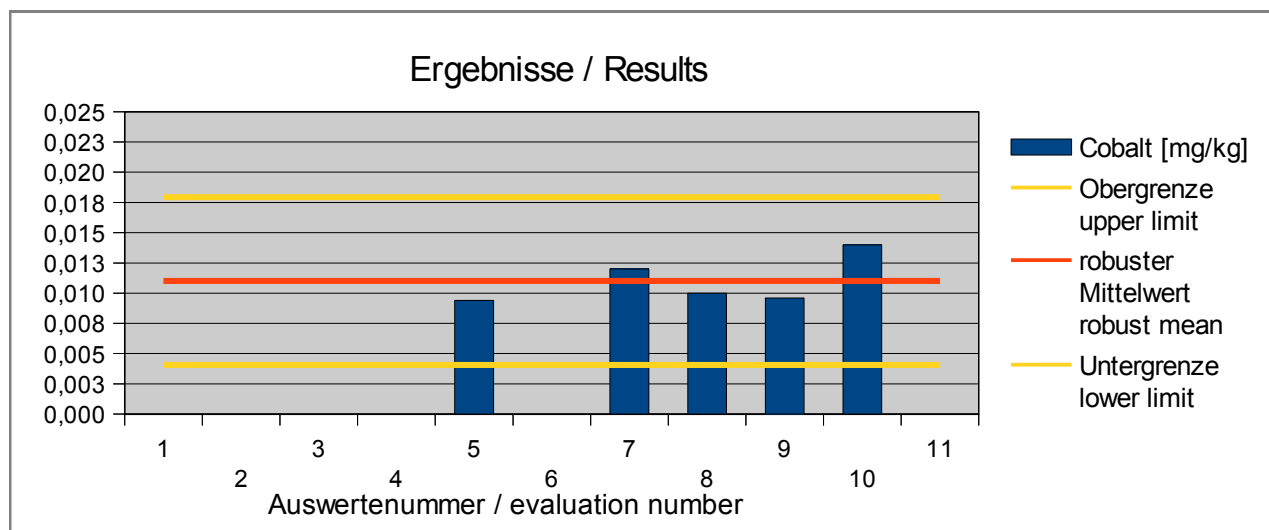


**Abb. / Fig. 10:** Z-Scores Cadmium

**4.6 Co - Cobalt in mg/kg**

**Vergleichsuntersuchung / Proficiency Test**

<b>Statistic Data</b>	
Number of results	5
Number of outliers	0
Mean	0,0110
Median	0,0100
<b>Robust Mean (X)</b>	<b>0,0110</b>
<b>Robust standard deviation (S*)</b>	<b>0,00223</b>
Number with 2 replicates	5
Repeatability SD ( $S_r$ )	0,00350
Repeatability ( $CV_r$ )	35,5%
Reproducibility SD ( $S_R$ )	0,00422
Reproducibility ( $CV_R$ )	42,8%
<i>Target range:</i>	
<b>Target standard deviation <math>\sigma_{pt}</math></b>	<b>0,00347</b>
<b>lower limit of target range</b>	<b>0,00406</b>
<b>upper limit of target range</b>	<b>0,0179</b>
Quotient $S^*/\sigma_{pt}$	0,64
Standard uncertainty $U(X_{pt})$	0,00125
Quotient $U(X_{pt})/\sigma_{pt}$	0,36
Results in the target range	5
Percent in the target range	100%



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

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

Auswertenummer	Cobalt [mg/kg]	Abweichung [mg/kg]	z-Score	Hinweis
Evaluation number		Deviation [mg/kg]	( $\sigma_{pt}$ )	Remark
1				
2				
3				
4	< 0,0500			
5	0,00940	-0,00160	-0,5	
6	< 0,160			
7	0,0120	0,00100	0,3	
8	0,0100	-0,00100	-0,3	
9	0,00960	-0,00140	-0,4	
10	0,0140	0,00300	0,9	
11	< 0,0100			



Abb. / Fig. 12: z-Scores Cobalt

**4.7 Cr - Chrom / Chromium in mg/kg****Vergleichsuntersuchung / Proficiency Test**

<b>Statistic Data</b>	
<i>Number of results</i>	4
<i>Number of outliers</i>	0
Mean	0,0423
Median	0,0400
<b>Robust Mean (X)</b>	<b>0,0423</b>
<b>Robust standard deviation (S*)</b>	<b>0,0104</b>
<i>Number with 2 replicates</i>	
Repeatability SD ( $S_r$ )	
Repeatability ( $CV_r$ )	
Reproducibility SD ( $S_R$ )	
Reproducibility ( $CV_R$ )	
<i>Target range:</i>	
<b>Target standard deviation <math>\sigma_{pt}</math></b>	
Target standard deviation (for Information)	
<b>lower limit of target range</b>	
<b>upper limit of target range</b>	
<i>Quotient <math>S^*/\sigma_{pt}</math></i>	
<i>Standard uncertainty <math>U_{(X_{pt})}</math></i>	
<i>Quotient <math>U_{(X_{pt})}/\sigma_{pt}</math></i>	
<i>Results in the target range</i>	
<i>Percent in the target range</i>	

*Due to the low number of results <5 no statistical evaluation was done.*

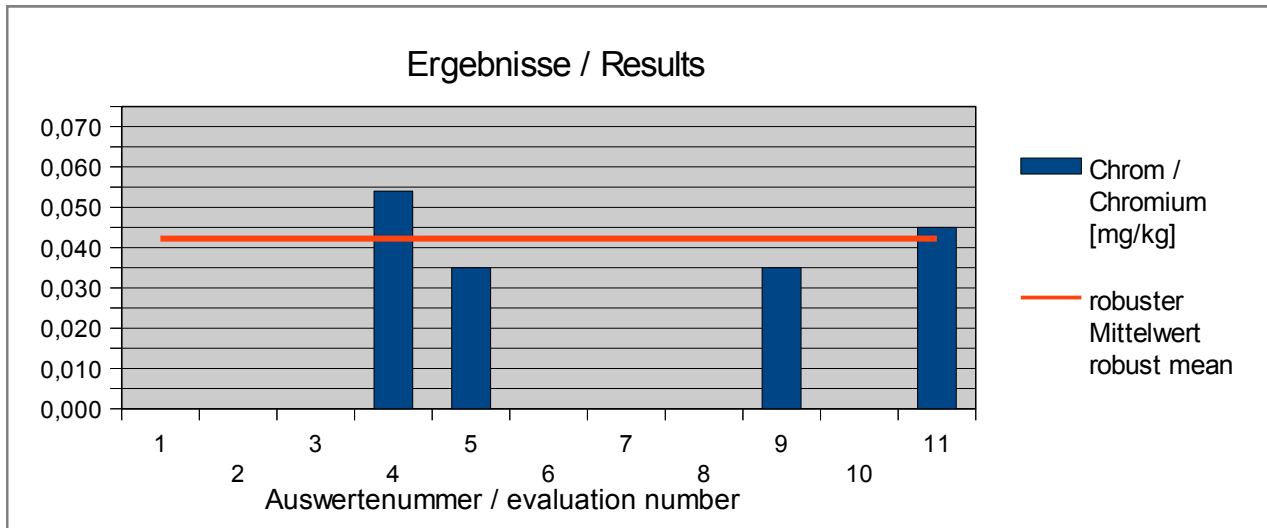


Abb. / Fig. 13: Ergebnisse Chrom / Results Chromium

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

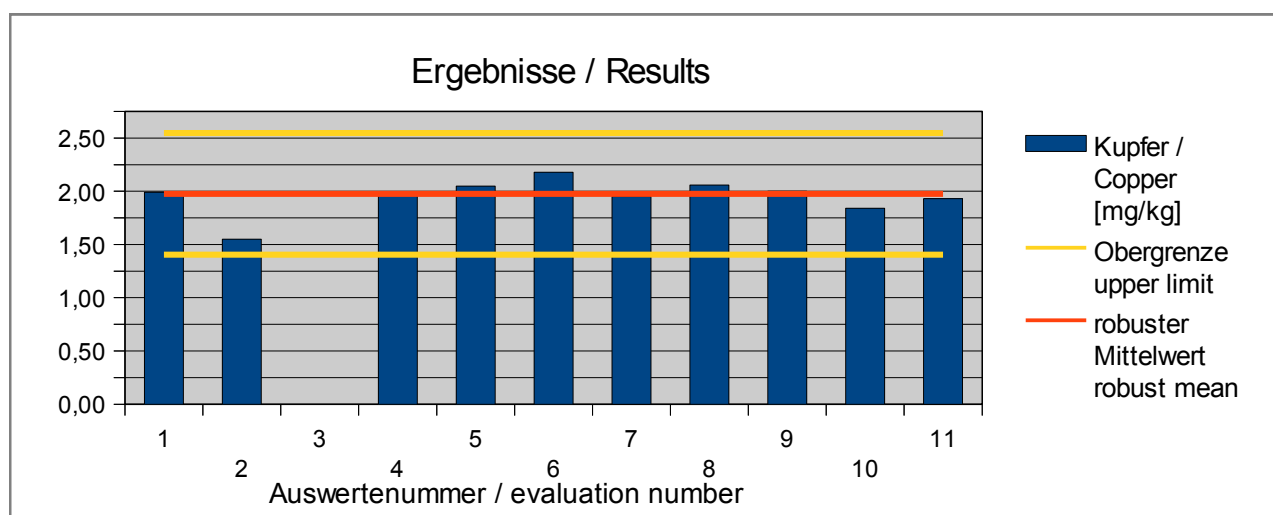
Auswertenummer Evaluation number	Chrom / Chromium [mg/kg]	Abweichung [mg/kg] Deviation [mg/kg]	z-Score ( $\sigma_{pt}$ )	z-Score (Info)	Hinweis Remark
1					
2	< 0,0600				
3					
4	0,0540	0,0118			
5	0,0350	-0,00725			
6	< 0,420				
7	< 0,0600				
8	< 0,100				
9	0,0350	-0,00725			
10	< 0,0500				
11	0,0450	0,00275			



**4.8 Cu - Kupfer / Copper in mg/kg**

**Vergleichsuntersuchung / Proficiency Test**

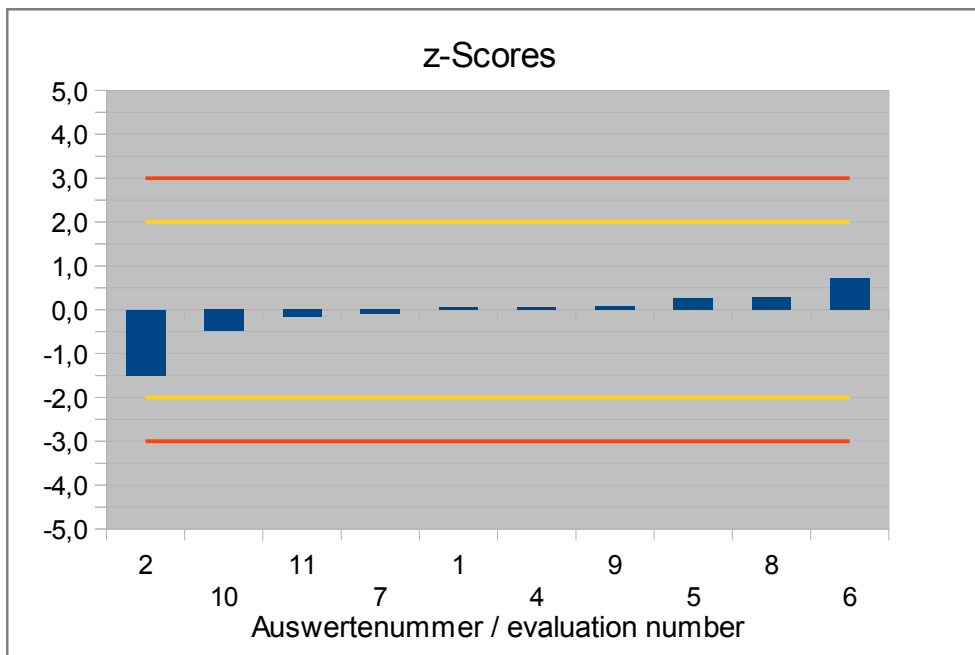
<b>Statistic Data</b>	
Number of results	10
Number of outliers	0
Mean	1,95
Median	1,99
<b>Robust Mean (X)</b>	<b>1,98</b>
<b>Robust standard deviation (S*)</b>	<b>0,117</b>
Number with 2 replicates	9
Repeatability SD ( $S_r$ )	0,0659
Repeatability ( $CV_r$ )	3,39%
Reproducibility SD ( $S_R$ )	0,180
Reproducibility ( $CV_R$ )	9,27%
<i>Target range:</i>	
<b>Target standard deviation <math>\sigma_{pt}</math></b>	<b>0,285</b>
Target standard deviation (for Information)	0,210
<b>lower limit of target range</b>	<b>1,41</b>
<b>upper limit of target range</b>	<b>2,55</b>
Quotient $S^*/\sigma_{pt}$	0,41
Standard uncertainty $U(x_{pt})$	0,0464
Quotient $U(x_{pt})/\sigma_{pt}$	0,16
Results in the target range	10
Percent in the target range	100%



**Abb. / Fig. 14:** Ergebnisse Kupfer / Results Copper

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

Auswertenummer Evaluation number	Kupfer / Copper [mg/kg]	Abweichung [mg/kg] Deviation [mg/kg]	z-Score ( $\sigma_{pt}$ )	z-Score (Info)	Hinweis Remark
1	1,99	0,0137	0,0	0,1	
2	1,55	-0,426	-1,5	-2,0	
3					
4	1,99	0,0137	0,0	0,1	
5	2,05	0,0737	0,3	0,4	
6	2,18	0,204	0,7	1,0	
7	1,95	-0,0263	-0,1	-0,1	
8	2,06	0,0837	0,3	0,4	
9	2,00	0,0237	0,1	0,1	
10	1,84	-0,136	-0,5	-0,6	
11	1,93	-0,0463	-0,2	-0,2	

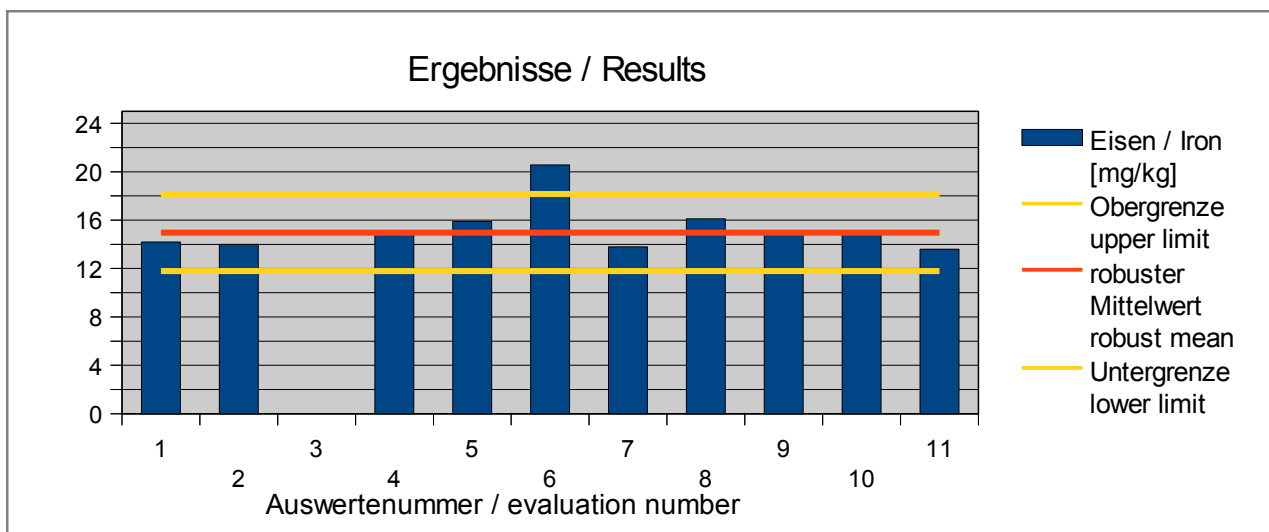


**Abb. / Fig. 15:** z-Scores Kupfer / Copper

**4.9 Fe - Eisen / Iron in mg/kg**

**Vergleichsuntersuchung / Proficiency Test**

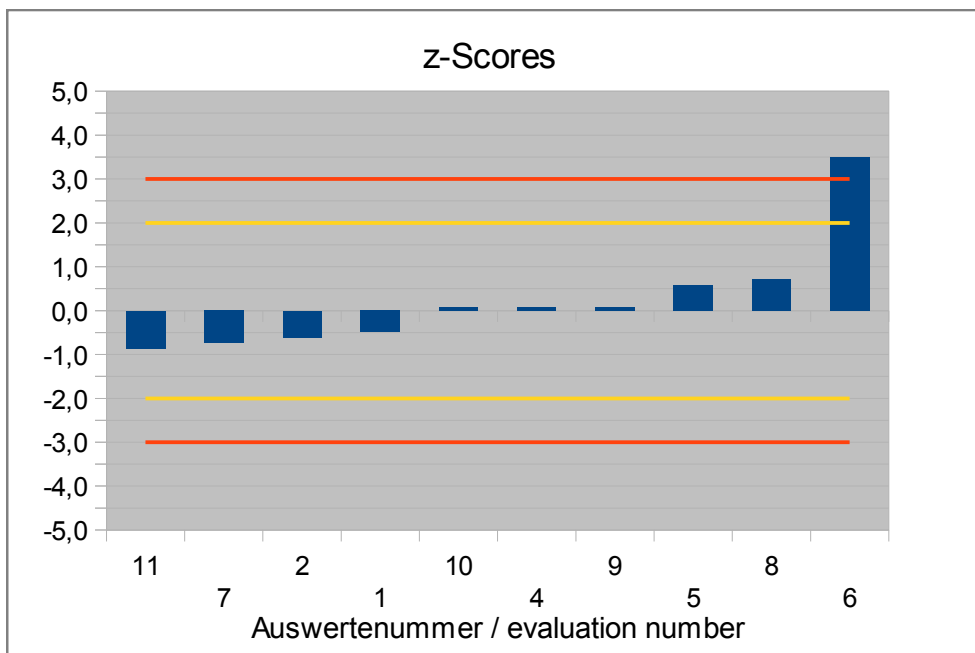
<b>Statistic Data</b>	
Number of results	10
Number of outliers	1
Mean	15,3
Median	15,1
<b>Robust Mean (X)</b>	<b>15,0</b>
<b>Robust standard deviation (S*)</b>	<b>1,22</b>
Number with 2 replicates	9
Repeatability SD ( $S_r$ )	1,53
Repeatability ( $CV_r$ )	10,0%
Reproducibility SD ( $S_R$ )	2,40
Reproducibility ( $CV_R$ )	15,7%
<i>Target range:</i>	
<b>Target standard deviation <math>\sigma_{pt}</math></b>	<b>1,59</b>
Target standard deviation (for Information)	1,00
<b>lower limit of target range</b>	<b>11,8</b>
<b>upper limit of target range</b>	<b>18,2</b>
Quotient $S^*/\sigma_{pt}$	0,76
Standard uncertainty $U(x_{pt})$	0,481
Quotient $U(x_{pt})/\sigma_{pt}$	0,30
Results in the target range	9
Percent in the target range	90%



**Abb. / Fig. 16:** Ergebnisse Eisen / Results Iron

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

Auswertenummer	Eisen / Iron [mg/kg]	Abweichung [mg/kg]	z-Score (σ <sub>pt</sub> )	z-Score (Info)	Hinweis
Evaluation number		Deviation [mg/kg]			Remark
1	14,2	-0,772	-0,5	-0,8	
2	14,0	-0,972	-0,6	-1,0	
3					
4	15,1	0,128	0,1	0,1	
5	15,9	0,928	0,6	0,9	
6	20,6	5,59	3,5	5,6	Ausreisser / Outlier
7	13,8	-1,17	-0,7	-1,2	
8	16,1	1,15	0,7	1,1	
9	15,1	0,128	0,1	0,1	
10	15,1	0,128	0,1	0,1	
11	13,6	-1,37	-0,9	-1,4	

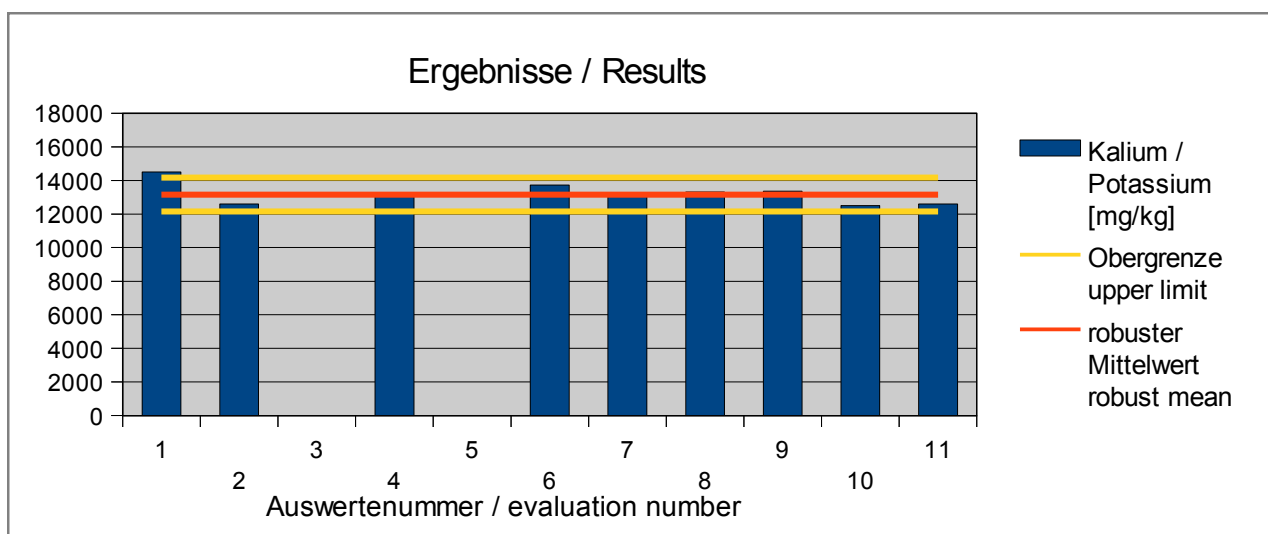


**Abb. / Fig. 17:** Z-Scores Eisen / Iron

**4.10 K - Kalium / Potassium in mg/kg**

**Vergleichsuntersuchung / Proficiency Test**

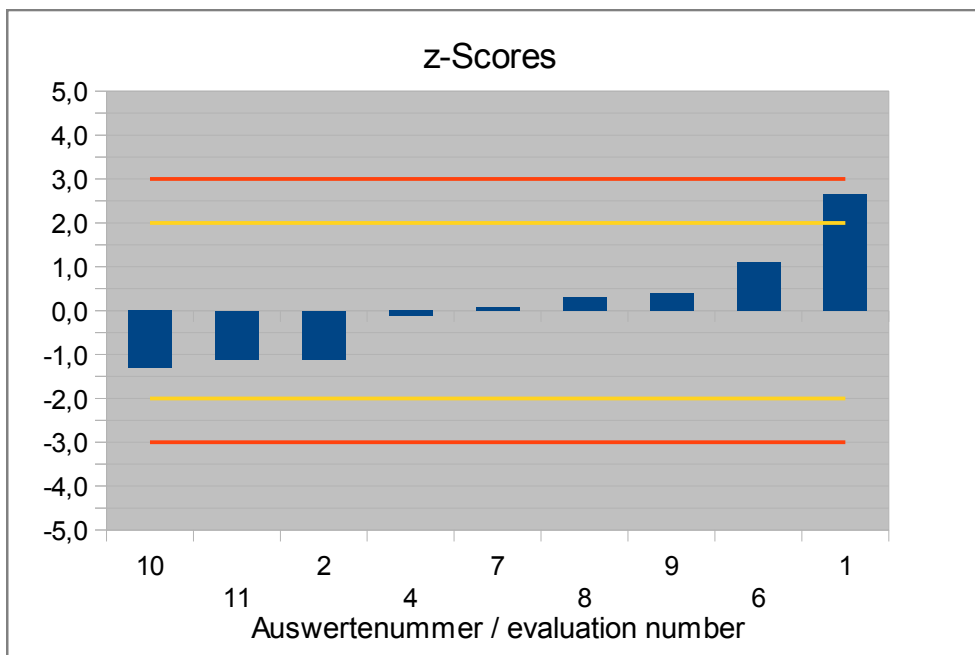
<b>Statistic Data</b>	
Number of results	9
Number of outliers	0
Mean	13200
Median	13200
<b>Robust Mean (X)</b>	<b>13200</b>
<b>Robust standard deviation (S*)</b>	<b>604</b>
Number with 2 replicates	9
Repeatability SD ( $S_r$ )	151
Repeatability ( $CV_r$ )	1,15%
Reproducibility SD ( $S_R$ )	631
Reproducibility ( $CV_R$ )	4,78%
<i>Target range:</i>	
<b>Target standard deviation <math>\sigma_{pt}</math></b>	<b>505</b>
Target standard deviation (for Information)	615
<b>lower limit of target range</b>	<b>12200</b>
<b>upper limit of target range</b>	<b>14200</b>
Quotient $S^*/\sigma_{pt}$	1,2
Standard uncertainty $U(x_{pt})$	252
Quotient $U(x_{pt})/\sigma_{pt}$	0,50
Results in the target range	8
Percent in the target range	89%



**Abb. / Fig. 18:** Ergebnisse Kalium / Results Potassium

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

Auswertenummer Evaluation number	Kalium / Potassium [mg/kg]	Abweichung [mg/kg] Deviation [mg/kg]	z-Score ( $\sigma_{pt}$ )	z-Score (Info)	Hinweis Remark
1	14500	1338	2,6	2,2	
2	12600	-562	-1,1	-0,9	
3					
4	13100	-61,9	-0,1	-0,1	
5					
6	13720	558	1,1	0,9	
7	13200	38,1	0,1	0,1	
8	13311	149	0,3	0,2	
9	13358	196	0,4	0,3	
10	12500	-662	-1,3	-1,1	
11	12600	-562	-1,1	-0,9	

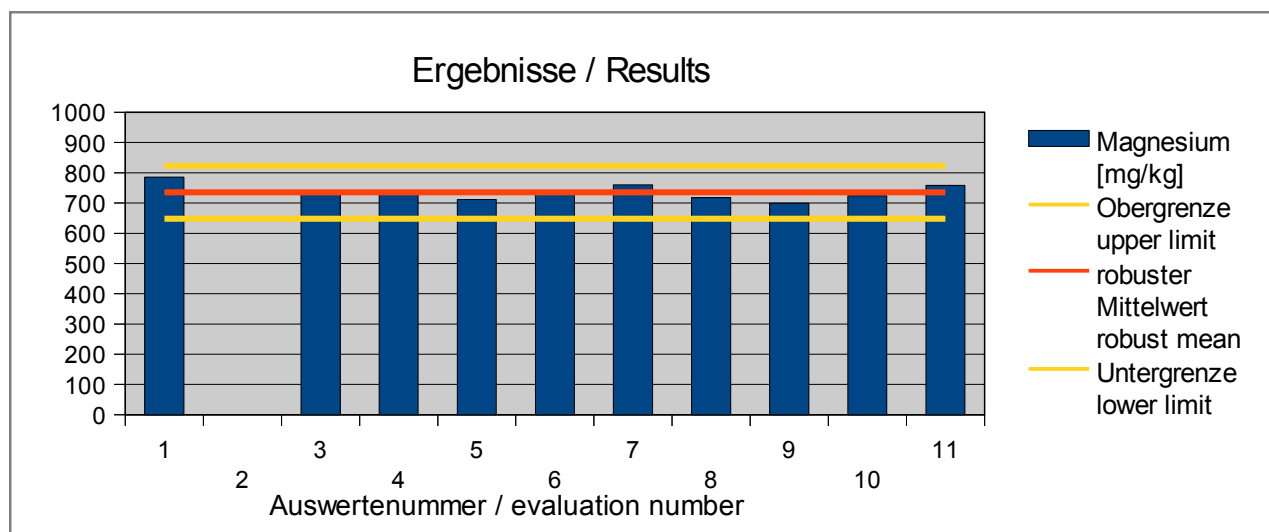


**Abb. / Fig. 19:** Z-Scores Kalium / Potassium

**4.11 Mg - Magnesium in mg/kg**

**Vergleichsuntersuchung / Proficiency Test**

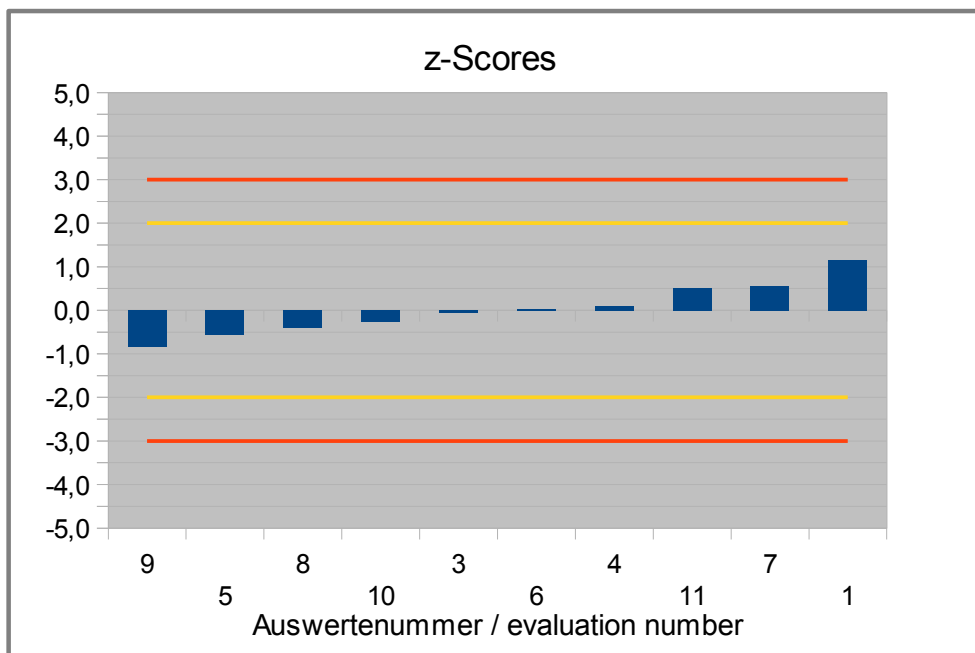
<b>Statistic Data</b>	
Number of results	10
Number of outliers	0
Mean	737
Median	735
<b>Robust Mean (X)</b>	<b>736</b>
<b>Robust standard deviation (S*)</b>	<b>27,1</b>
Number with 2 replicates	9
Repeatability SD ( $S_r$ )	13,8
Repeatability ( $CV_r$ )	1,86%
Reproducibility SD ( $S_R$ )	27,5
Reproducibility ( $CV_R$ )	3,71%
<i>Target range:</i>	
<b>Target standard deviation <math>\sigma_{pt}</math></b>	<b>43,6</b>
Target standard deviation (for Information)	51,6
<b>lower limit of target range</b>	<b>648</b>
<b>upper limit of target range</b>	<b>823</b>
Quotient $S^*/\sigma_{pt}$	0,62
Standard uncertainty $U(x_{pt})$	10,7
Quotient $U(x_{pt})/\sigma_{pt}$	0,25
Results in the target range	10
Percent in the target range	100%



**Abb. / Fig. 20:** Ergebnisse Magnesium / Results Magnesium

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

Auswertenummer	Magnesium [mg/kg]	Abweichung [mg/kg]	z-Score (σ <sub>pt</sub> )	z-Score (Info)	Hinweis
Evaluation number		Deviation [mg/kg]		(Info)	Remark
1	786	50,3	1,2	1,0	
2					
3	733	-2,88	-0,1	-0,1	
4	740	4,34	0,1	0,1	
5	712	-24,1	-0,6	-0,5	
6	737	1,24	0,0	0,0	
7	760	24,3	0,6	0,5	
8	718	-17,7	-0,4	-0,3	
9	699	-36,7	-0,8	-0,7	
10	724	-11,7	-0,3	-0,2	
11	758	22,3	0,5	0,4	



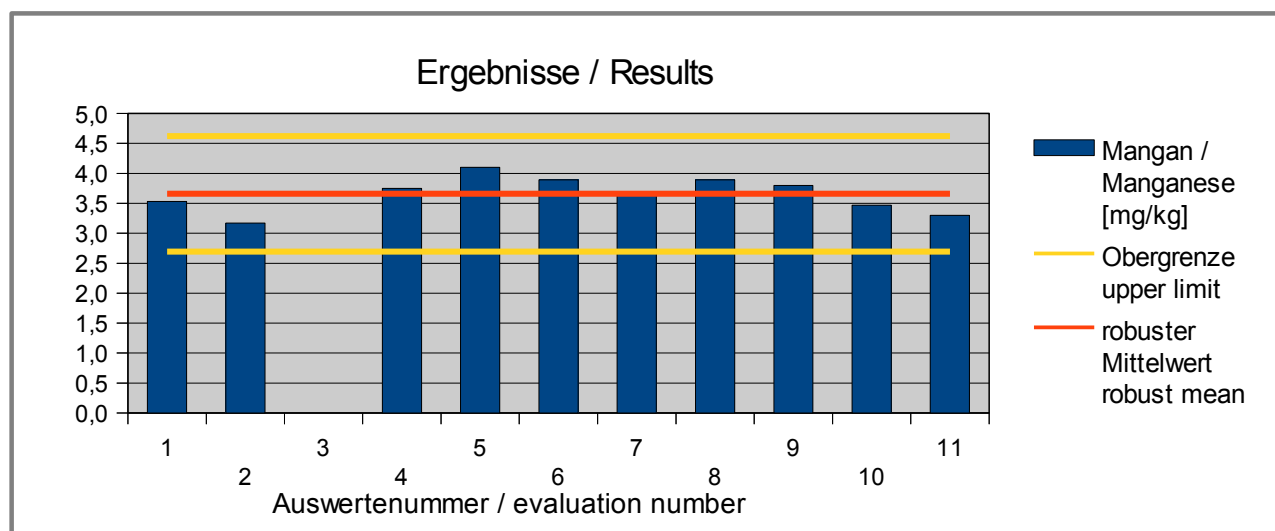
**Abb. / Fig. 21:** Z-Scores Magnesium



**4.12 Mn – Mangan / Manganese in mg/kg**

**Vergleichsuntersuchung / Proficiency Test**

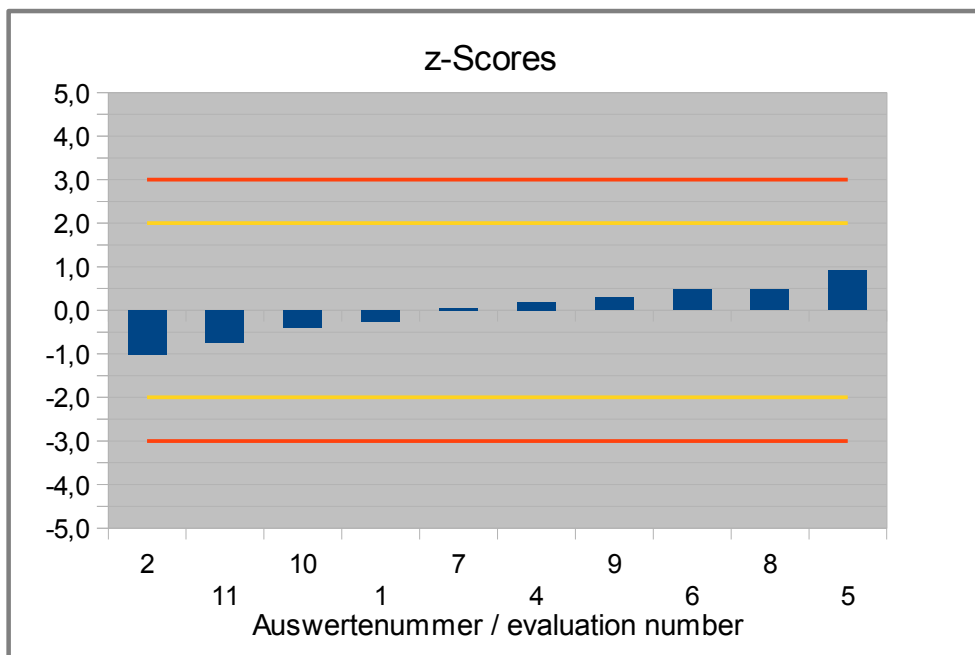
<b>Statistic Data</b>	
Number of results	10
Number of outliers	0
Mean	3,66
Median	3,72
<b>Robust Mean (X)</b>	<b>3,66</b>
<b>Robust standard deviation (S*)</b>	<b>0,327</b>
Number with 2 replicates	9
Repeatability SD ( $S_r$ )	0,0514
Repeatability ( $CV_r$ )	1,43%
Reproducibility SD ( $S_R$ )	0,262
Reproducibility ( $CV_R$ )	7,26%
<i>Target range:</i>	
<b>Target standard deviation <math>\sigma_{pt}</math></b>	<b>0,482</b>
Target standard deviation (for Information)	0,486
<b>lower limit of target range</b>	<b>2,70</b>
<b>upper limit of target range</b>	<b>4,62</b>
Quotient $S^*/\sigma_{pt}$	0,68
Standard uncertainty $U(x_{pt})$	0,129
Quotient $U(x_{pt})/\sigma_{pt}$	0,27
Results in the target range	10
Percent in the target range	100%



**Abb. / Fig. 22:** Ergebnisse Mangan / Results Manganese

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

Auswertenummer Evaluation number	Mangan / Manganese [mg/kg]	Abweichung [mg/kg] Deviation [mg/kg]	z-Score ( $\sigma_{pt}$ )	z-Score (Info)	Hinweis Remark
1	3,53	-0,129	-0,3	-0,3	
2	3,17	-0,489	-1,0	-1,0	
3					
4	3,75	0,091	0,2	0,2	
5	4,10	0,441	0,9	0,9	
6	3,89	0,231	0,5	0,5	
7	3,69	0,031	0,1	0,1	
8	3,89	0,231	0,5	0,5	
9	3,80	0,141	0,3	0,3	
10	3,47	-0,189	-0,4	-0,4	
11	3,30	-0,359	-0,7	-0,7	

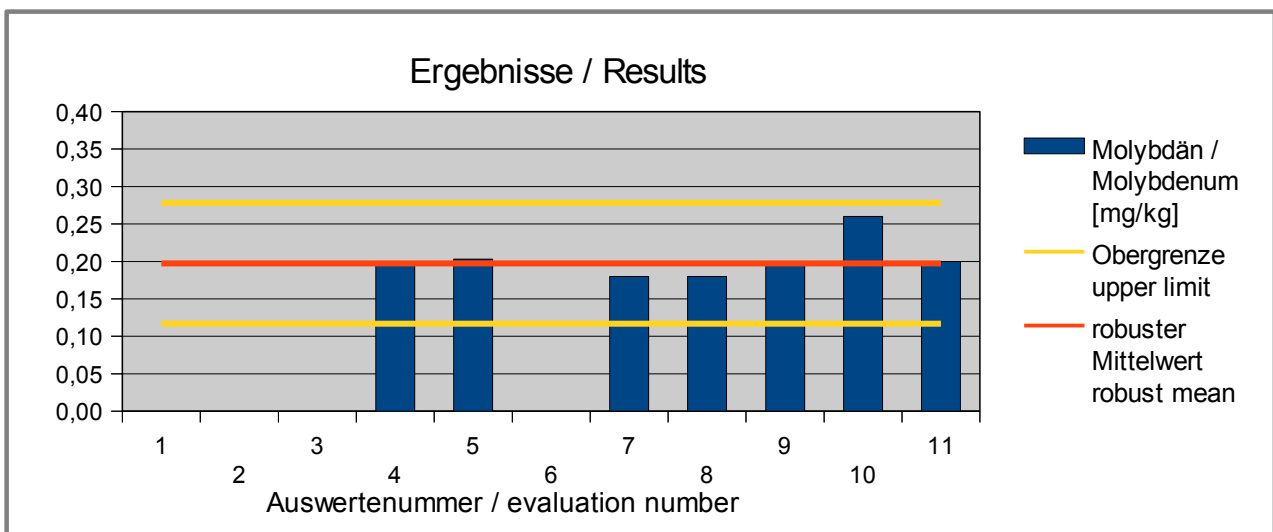


**Abb. / Fig. 23:** z-Scores Mangan / Manganese

**4.13 Mo – Molybdän / Molybdenum in mg/kg**

**Vergleichsuntersuchung / Proficiency Test**

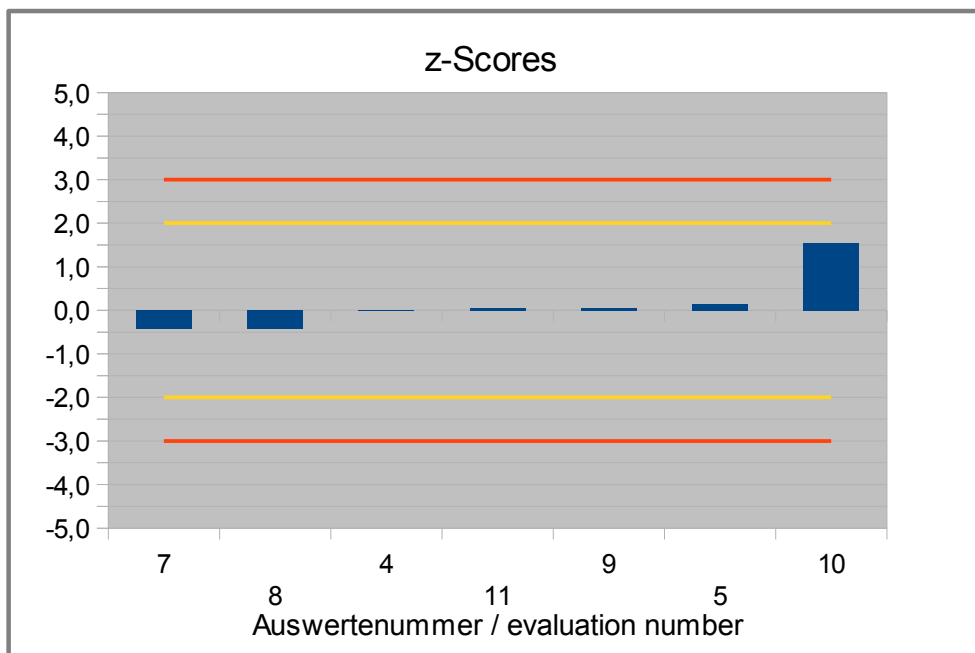
<b>Statistic Data</b>	
Number of results	7
Number of outliers	0
Mean	0,203
Median	0,200
<b>Robust Mean (X)</b>	<b>0,197</b>
<b>Robust standard deviation (S*)</b>	<b>0,0161</b>
Number with 2 replicates	6
Repeatability SD (S <sub>r</sub> )	0,00580
Repeatability (CV <sub>r</sub> )	2,86%
Reproducibility SD (S <sub>R</sub> )	0,0298
Reproducibility (CV <sub>R</sub> )	14,7%
<i>Target range:</i>	
<b>Target standard deviation <math>\sigma_{pt}</math></b>	<b>0,0403</b>
Target standard deviation (for Information)	0,0376
<b>lower limit of target range</b>	<b>0,117</b>
<b>upper limit of target range</b>	<b>0,278</b>
Quotient $S^*/\sigma_{pt}$	0,40
Standard uncertainty $U(x_{pt})$	0,00762
Quotient $U(x_{pt})/\sigma_{pt}$	0,19
Results in the target range	7
Percent in the target range	100%



**Abb. / Fig. 24:** Ergebnisse Molybdän / Results Molybdenum

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

Auswertenummer	Molybdän / Molybdenum [mg/kg]	Abweichung [mg/kg]	z-Score (σ <sub>pt</sub> )	z-Score (Info)	Hinweis
Evaluation number		Deviation [mg/kg]			Remark
1					
2					
3					
4	0,198	0,000558	0,0	0,0	
5	0,203	0,00556	0,1	0,1	
6	< 1,48				
7	0,180	-0,0174	-0,4	-0,5	
8	0,180	-0,0174	-0,4	-0,5	
9	0,200	0,00256	0,1	0,1	
10	0,260	0,0626	1,6	1,7	
11	0,200	0,00256	0,1	0,1	

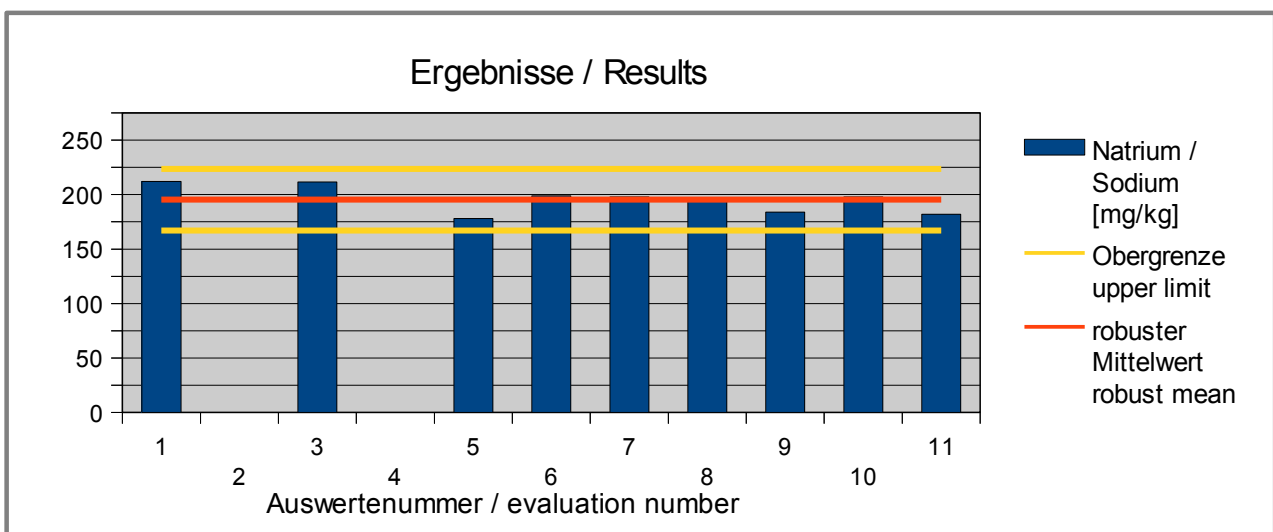


**Abb. / Fig. 25:** z-Scores Molybdän / Molybdenum

**4.14 Na - Natrium / Sodium in mg/kg**

**Vergleichsuntersuchung / Proficiency Test**

<b>Statistic Data</b>	
Number of results	9
Number of outliers	0
Mean	195
Median	198
<b>Robust Mean (X)</b>	<b>195</b>
<b>Robust standard deviation (S*)</b>	<b>13,7</b>
Number with 2 replicates	8
Repeatability SD ( $S_r$ )	3,16
Repeatability ( $CV_r$ )	1,60%
Reproducibility SD ( $S_R$ )	11,1
Reproducibility ( $CV_R$ )	5,63%
<i>Target range:</i>	
<b>Target standard deviation <math>\sigma_{pt}</math></b>	<b>14,1</b>
Target standard deviation (for Information)	8,09
<b>lower limit of target range</b>	<b>167</b>
<b>upper limit of target range</b>	<b>224</b>
Quotient $S^*/\sigma_{pt}$	1,0
Standard uncertainty $U(x_{pt})$	5,72
Quotient $U(x_{pt})/\sigma_{pt}$	0,40
Results in the target range	9
Percent in the target range	100%



**Abb. / Fig. 26:** Ergebnisse Natrium / Results Sodium

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

Auswertenummer Evaluation number	Natrium / Sodium [mg/kg]	Abweichung [mg/kg] Deviation [mg/kg]	z-Score ( $\sigma_{pt}$ )	z-Score (Info)	Hinweis Remark
1	212	16,7	1,2	2,1	
2					
3	212	16,3	1,2	2,0	
4					
5	178	-17,2	-1,2	-2,1	
6	199	3,61	0,3	0,4	
7	198	2,71	0,2	0,3	
8	195	-0,286	0,0	0,0	
9	184	-11,3	-0,8	-1,4	
10	198	2,71	0,2	0,3	
11	182	-13,3	-0,9	-1,6	

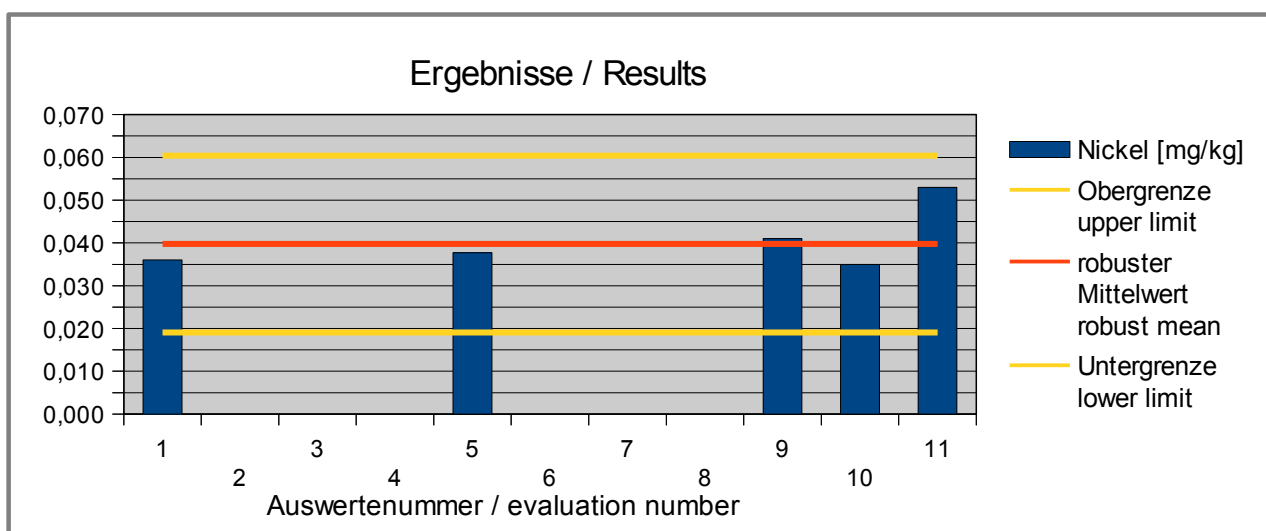


**Abb. / Fig. 27:** Z-Scores Natrium / Sodium

**4.15 Ni - Nickel in mg/kg**

**Vergleichsuntersuchung / Proficiency Test**

<b>Statistic Data</b>	
Number of results	5
Number of outliers	0
Mean	0,0405
Median	0,0377
<b>Robust Mean (X)</b>	<b>0,0398</b>
<b>Robust standard deviation (S*)</b>	<b>0,00645</b>
Number with 2 replicates	4
Repeatability SD ( $S_r$ )	0,00366
Repeatability ( $CV_r$ )	8,95%
Reproducibility SD ( $S_R$ )	0,00854
Reproducibility ( $CV_R$ )	20,9%
<i>Target range:</i>	
<b>Target standard deviation <math>\sigma_{pt}</math></b>	<b>0,0103</b>
<b>lower limit of target range</b>	<b>0,0191</b>
<b>upper limit of target range</b>	<b>0,0604</b>
Quotient $S^*/\sigma_{pt}$	0,62
Standard uncertainty $U(X_{pt})$	0,00360
Quotient $U(X_{pt})/\sigma_{pt}$	0,35
Results in the target range	5
Percent in the target range	100%



**Abb. / Fig. 28:** Ergebnisse Nickel / Results Nickel

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

Auswertenummer	Nickel [mg/kg]	Abweichung [mg/kg]	z-Score	Hinweis
Evaluation number		Deviation [mg/kg]	( $\sigma_{pt}$ )	Remark
1	0,0360	-0,00375	-0,4	
2	< 0,0600			
3				
4	< 0,100			
5	0,0377	-0,00205	-0,2	
6	< 2,00			
7	< 0,200			
8	< 0,100			
9	0,0410	0,00125	0,1	
10	0,0350	-0,00475	-0,5	
11	0,0530	0,0132	1,3	

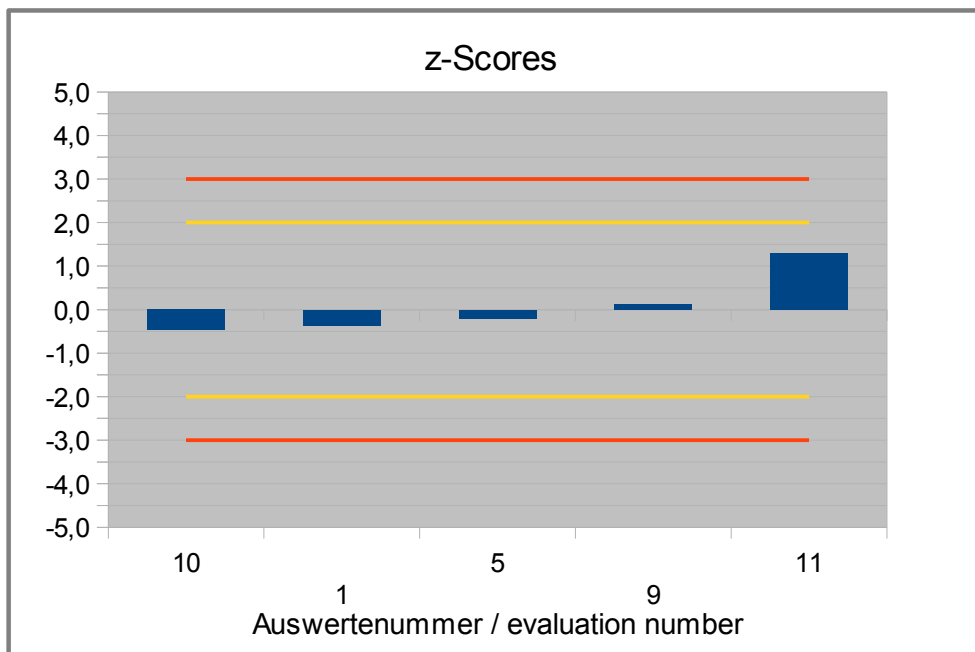


Abb. / Fig. 29: z-Scores Nickel



**4.16 P - Phosphor / Phosphorus in mg/kg**

**Vergleichsuntersuchung / Proficiency Test**

<b>Statistic Data</b>	
Number of results	7
Number of outliers	0
Mean	1450
Median	1450
<b>Robust Mean (X)</b>	<b>1450</b>
<b>Robust standard deviation (S*)</b>	<b>49,1</b>
Number with 2 replicates	7
Repeatability SD (S <sub>r</sub> )	24,0
Repeatability (CV <sub>r</sub> )	1,65%
Reproducibility SD (S <sub>R</sub> )	47,3
Reproducibility (CV <sub>R</sub> )	3,26%
<i>Target range:</i>	
<b>Target standard deviation <math>\sigma_{pt}</math></b>	<b>77,6</b>
Target standard deviation (for Information)	109
<b>lower limit of target range</b>	<b>1300</b>
<b>upper limit of target range</b>	<b>1610</b>
Quotient $S^*/\sigma_{pt}$	0,63
Standard uncertainty $U(x_{pt})$	23,2
Quotient $U(x_{pt})/\sigma_{pt}$	0,30
Results in the target range	7
Percent in the target range	100%

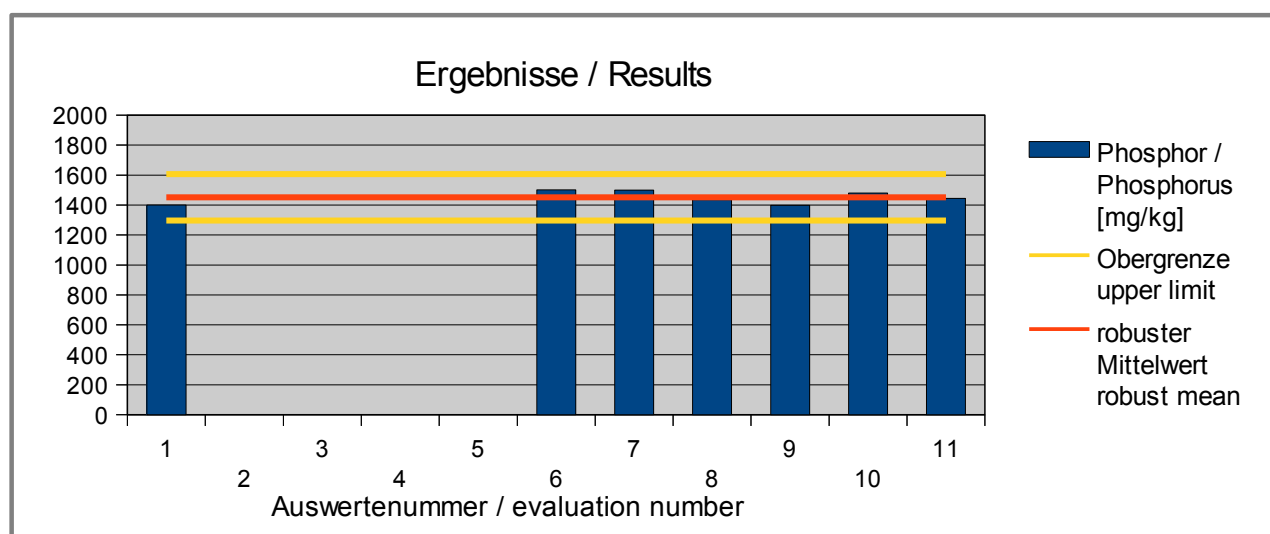


Abb. / Fig. 30: Ergebnisse Phosphor / Results Phosphorus

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

Auswertenummer	Phosphor / Phosphorus [mg/kg]	Abweichung [mg/kg]	z-Score (σ <sub>pt</sub> )	z-Score (Info)	Hinweis
Evaluation number		Deviation [mg/kg]			Remark
1	1400	-51,3	-0,7	-0,5	
2					
3					
4					
5					
6	1501	49,7	0,6	0,5	
7	1499	47,7	0,6	0,4	
8	1435	-16,3	-0,2	-0,1	
9	1399	-52,3	-0,7	-0,5	
10	1480	28,7	0,4	0,3	
11	1445	-6,29	-0,1	-0,1	



**Abb. / Fig. 31:** Z-Scores Phosphor / Phosphorus

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

<b>Statistic Data</b>	
<i>Number of results</i>	4
<i>Number of outliers</i>	0
Mean	2,76
Median	2,76
<b>Robust Mean (X)</b>	<b>2,76</b>
<b>Robust standard deviation (S*)</b>	<b>0,0558</b>
<i>Number with 2 replicates</i>	
Repeatability SD ( $S_r$ )	
Repeatability ( $CV_r$ )	
Reproducibility SD ( $S_R$ )	
Reproducibility ( $CV_R$ )	
<i>Target range:</i>	
<b>Target standard deviation <math>\sigma_{pt}</math></b>	
Target standard deviation (for Information)	
<b>lower limit of target range</b>	
<b>upper limit of target range</b>	
<i>Quotient <math>S^*/\sigma_{pt}</math></i>	
<i>Standard uncertainty <math>U(X_{pt})</math></i>	
<i>Quotient <math>U(X_{pt})/\sigma_{pt}</math></i>	
<i>Results in the target range</i>	
<i>Percent in the target range</i>	

Due to the low number of results <5 no statistical evaluation was done.

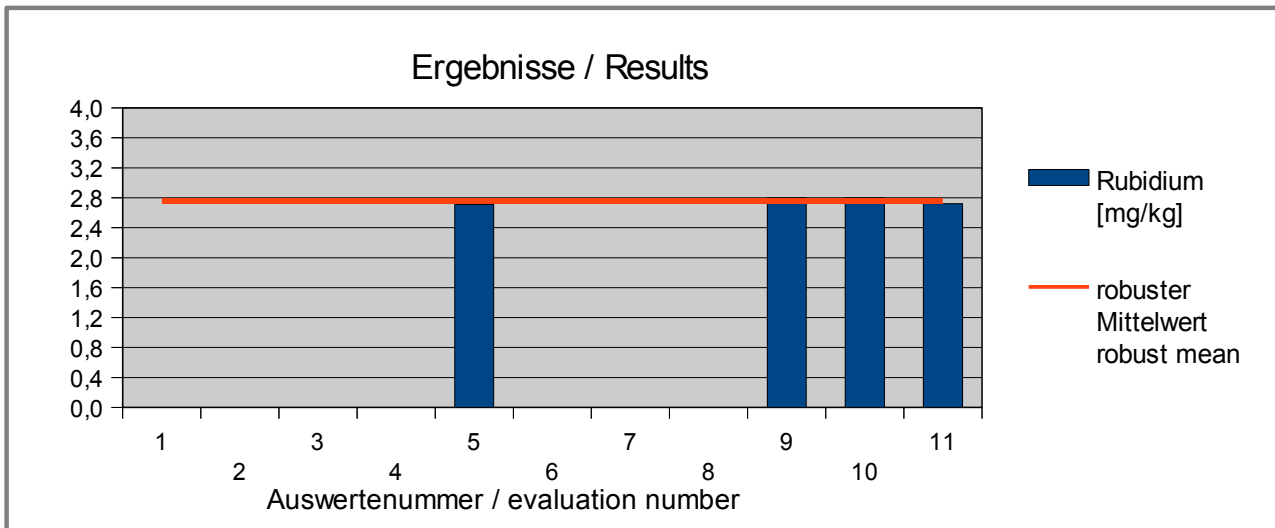


Abb. / Fig. 32: Ergebnisse Rubidium / Results Rubidium

Ergebnisse der Teilnehmer:  
Results of Participants:

Auswertenummer Evaluation number	Rubidium [mg/kg]	Abweichung [mg/kg] Deviation [mg/kg]	z-Score ( $\sigma_{pt}$ )	z-Score (Info)	Hinweis Remark
1					
2					
3					
4					
5	2,71	-0,0475			
6	< 2,00				
7					
8					
9	2,80	0,0425			
10	2,80	0,0425			
11	2,72	-0,0375			

**4.18 S - Schwefel / Sulfur in mg/kg****Vergleichsuntersuchung / Proficiency Test**

<b>Statistic Data</b>	
<i>Number of results</i>	4
<i>Number of outliers</i>	0
Mean	1000
Median	1100
<b>Robust Mean (X)</b>	<b>1000</b>
<b>Robust standard deviation (S*)</b>	<b>838</b>
<i>Number with 2 replicates</i>	
Repeatability SD ( $S_r$ )	
Repeatability ( $CV_r$ )	
Reproducibility SD ( $S_R$ )	
Reproducibility ( $CV_R$ )	
<i>Target range:</i>	
<b>Target standard deviation <math>\sigma_{pt}</math></b>	
Target standard deviation (for Information)	
<b>lower limit of target range</b>	
<b>upper limit of target range</b>	
<i>Quotient <math>S^*/\sigma_{pt}</math></i>	
<i>Standard uncertainty <math>U(x_{pt})</math></i>	
<i>Quotient <math>U(x_{pt})/\sigma_{pt}</math></i>	
<i>Results in the target range</i>	
<i>Percent in the target range</i>	

Due to the low number of results < 5 no statistical evaluation was done.

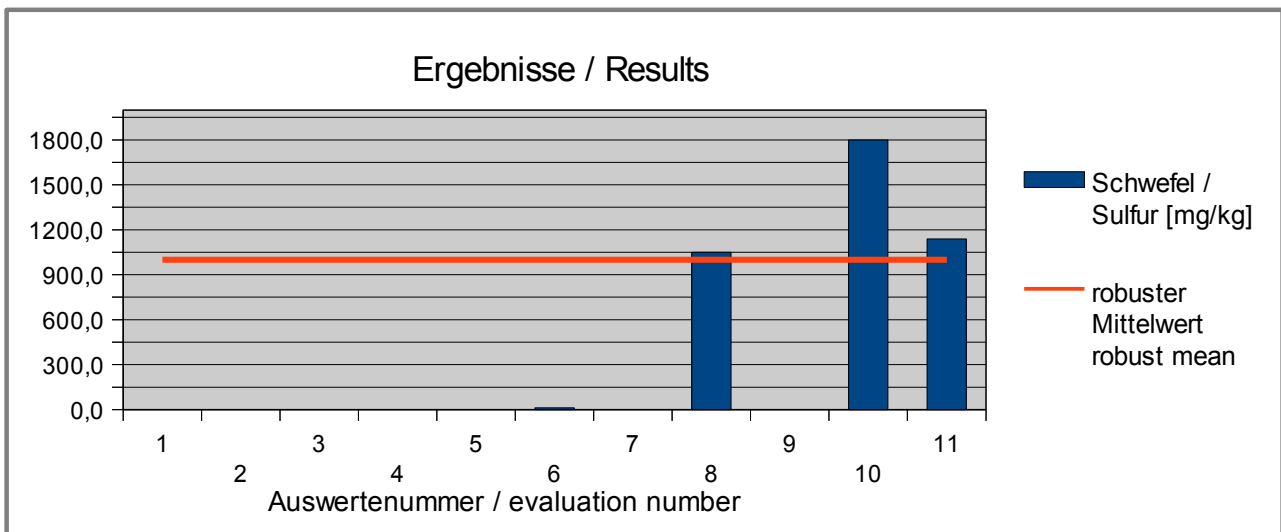


Abb. / Fig. 33: Ergebnisse Schwefel / Results Sulfur

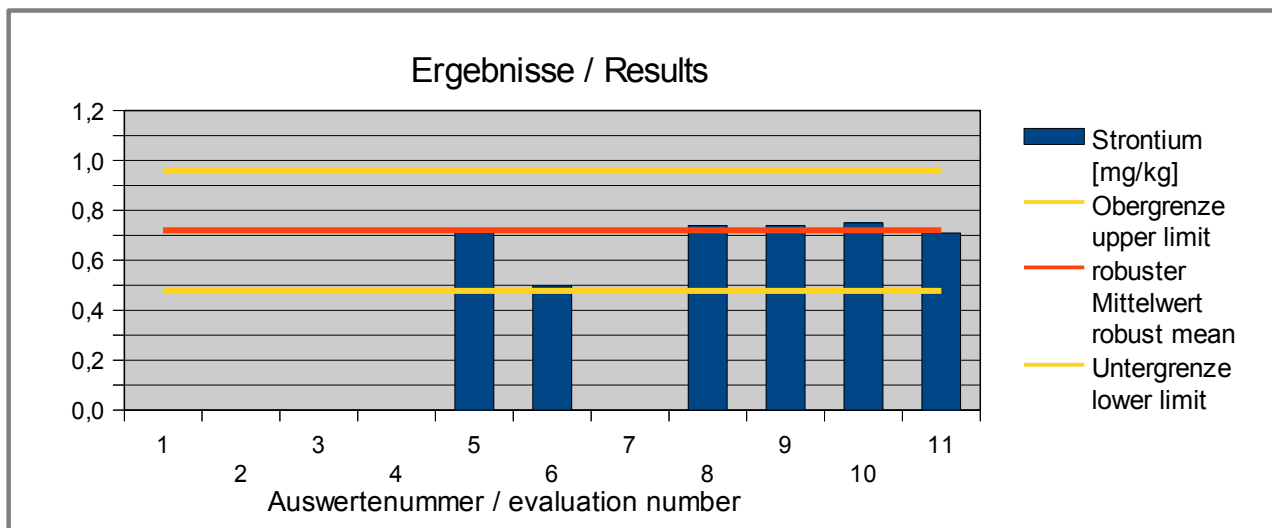
Ergebnisse der Teilnehmer:  
Results of Participants:

Auswertenummer Evaluation number	Schwefel / Sulfur [mg/kg]	Abweichung [mg/kg] Deviation [mg/kg]	z-Score ( $\sigma_{pt}$ )	z-Score (Info)	Hinweis Remark
1					
2					
3					
4					
5					
6	11,7	-988			unit failure?
7					
8	1050	49,8			
9					
10	1800	800			
11	1139	139			

**4.19 Sr - Strontium in mg/kg**

**Vergleichsuntersuchung / Proficiency Test**

<b>Statistic Data</b>	
Number of results	6
Number of outliers	0
Mean	0,691
Median	0,725
<b>Robust Mean (X)</b>	<b>0,720</b>
<b>Robust standard deviation (S*)</b>	<b>0,0336</b>
Number with 2 replicates	5
Repeatability SD ( $S_r$ )	0,00425
Repeatability ( $CV_r$ )	0,620%
Reproducibility SD ( $S_R$ )	0,107
Reproducibility ( $CV_R$ )	15,5%
<i>Target range:</i>	
<b>Target standard deviation <math>\sigma_{pt}</math></b>	<b>0,121</b>
<b>lower limit of target range</b>	<b>0,478</b>
<b>upper limit of target range</b>	<b>0,962</b>
Quotient $S^*/\sigma_{pt}$	0,28
Standard uncertainty $U(X_{pt})$	0,0172
Quotient $U(X_{pt})/\sigma_{pt}$	0,14
Results in the target range	6
Percent in the target range	100%



**Abb. / Fig. 34:** Ergebnisse Strontium / Results Strontium

Ergebnisse der Teilnehmer:  
Results of Participants:

Auswertenummer	Strontium [mg/kg]	Abweichung [mg/kg]	z-Score ( $\sigma_{pt}$ )	Hinweis
Evaluation number		Deviation [mg/kg]		Remark
1				
2				
3				
4				
5	0,710	-0,0100	-0,1	
6	0,498	-0,223	-1,8	
7				
8	0,740	0,0200	0,2	
9	0,740	0,0200	0,2	
10	0,750	0,0300	0,2	
11	0,710	-0,0100	-0,1	

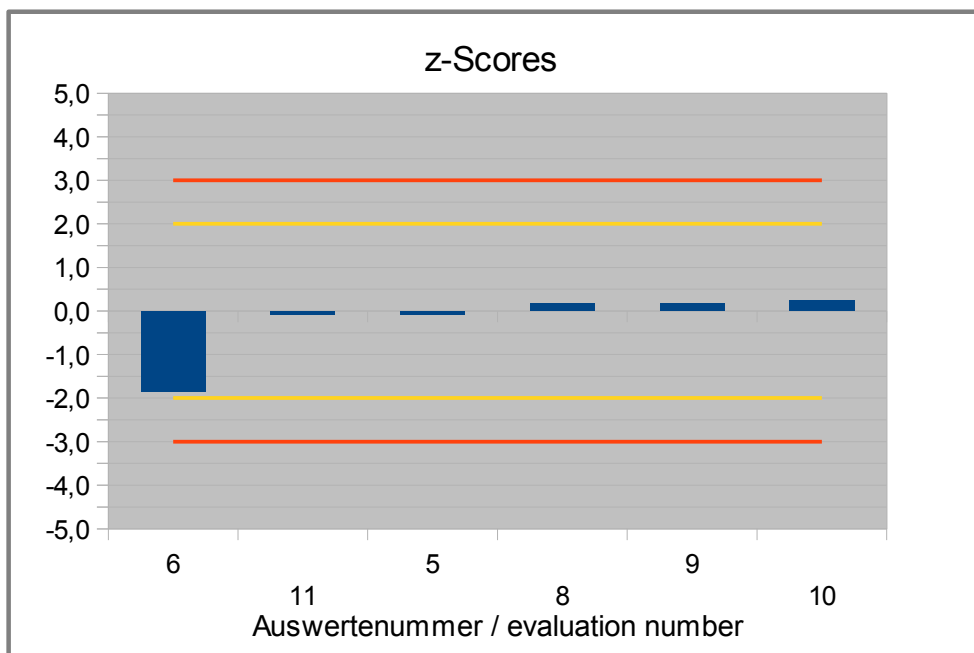


Abb. / Fig. 35: Z-Scores Strontium



**4.20 Zn - Zink / Zinc in mg/kg**

**Vergleichsuntersuchung / Proficiency Test**

<b>Statistic Data</b>	
Number of results	10
Number of outliers	0
Mean	7,83
Median	7,85
<b>Robust Mean (X)</b>	<b>7,83</b>
<b>Robust standard deviation (S*)</b>	<b>0,726</b>
Number with 2 replicates	9
Repeatability SD ( $S_r$ )	0,421
Repeatability ( $CV_r$ )	5,35%
Reproducibility SD ( $S_R$ )	0,734
Reproducibility ( $CV_R$ )	9,34%
<i>Target range:</i>	
<b>Target standard deviation <math>\sigma_{pt}</math></b>	<b>0,919</b>
Target standard deviation (for Information)	0,520
<b>lower limit of target range</b>	<b>5,99</b>
<b>upper limit of target range</b>	<b>9,67</b>
Quotient $S^*/\sigma_{pt}$	0,79
Standard uncertainty $U(x_{pt})$	0,287
Quotient $U(x_{pt})/\sigma_{pt}$	0,31
Results in the target range	10
Percent in the target range	100%

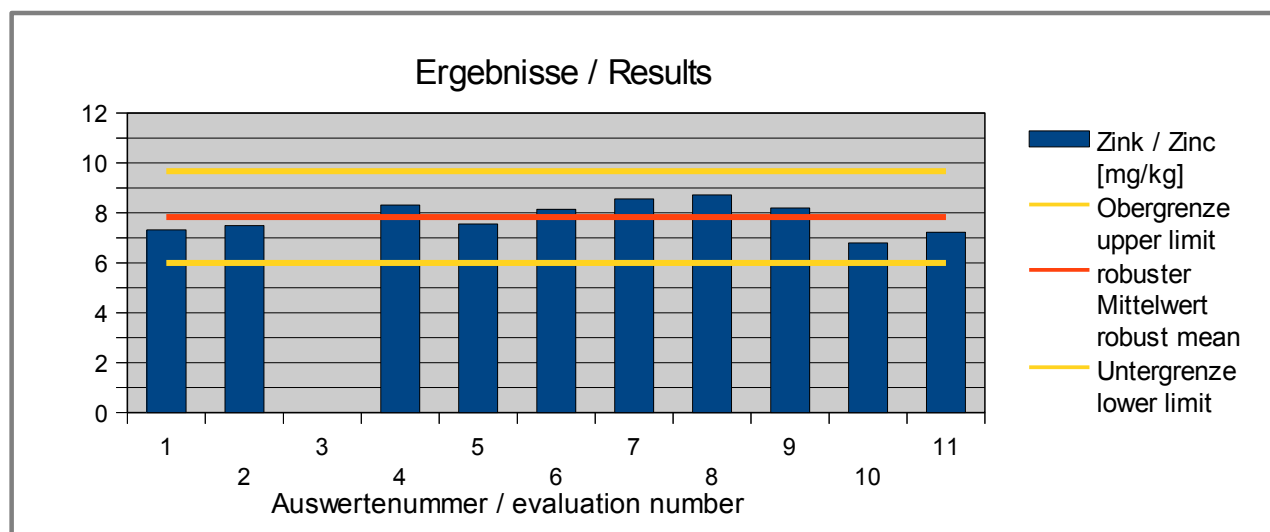
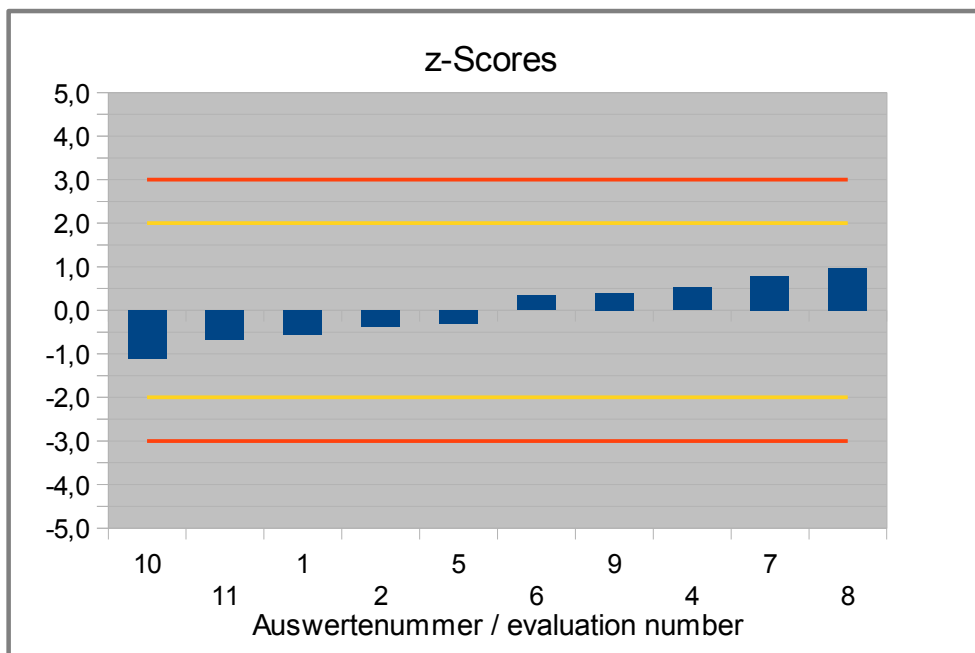


Abb. / Fig. 36: Ergebnisse Zink / Results Zinc

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

Auswertenummer Evaluation number	Zink / Zinc [mg/kg]	Abweichung [mg/kg] Deviation [mg/kg]	z-Score ( $\sigma_{pt}$ )	z-Score (Info)	Hinweis Remark
1	7,32	-0,513	-0,6	-1,0	
2	7,49	-0,343	-0,4	-0,7	
3					
4	8,32	0,487	0,5	0,9	
5	7,55	-0,283	-0,3	-0,5	
6	8,15	0,316	0,3	0,6	
7	8,56	0,727	0,8	1,4	
8	8,72	0,887	1,0	1,7	
9	8,20	0,367	0,4	0,7	
10	6,80	-1,03	-1,1	-2,0	
11	7,22	-0,613	-0,7	-1,2	



**Abb. / Fig. 37:** z-Scores Zink / Zinc

## 5. Documentation

### 5.1 Details by the participants

Note: Information given in German were translated by DLA to the best of our knowledge (without guarantee of correctness).

#### 5.1.1 Primary Data and analytical Methods

Parameter	Teilnehmer	Einheit	Proben-Nr. 1	Proben-Nr. 2	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis 1	Ergebnis 2	Bestimmungsgrenze	Inkl. WF	Wiederfindungsrate [%]
Analyte	Participant	Unit	Sample No. 1	Sample No. 2	Date of analysis	Result (Mean)	Result 1	Result 2	Limit of determination	Incl. RR	Recovery rate [%]
Aluminium	1	mg/kg	30	66	03/05	0,247	0,214	0,279	5ppb		
	2	mg/kg	28	68	20.05.17	< 0,30	< 0,30	< 0,30	0,3	no	
	3	mg/kg	21	75							
	4	mg/kg	20	76							
	5	mg/kg	17	-	26.04.17	0,57	0,57		0,14	no	
	6	mg/kg	8	88	05.05.17	< 2,000	< 2,00	< 2,00	2	no	---
	7	mg/kg	32	64	10.05.	<0,6	<0,6	<0,6	0,6	no	
	8	mg/kg	19	77		<1	<1	<1	<1	no	
	9	mg/kg	18	78	20.04.17	0,48	0,48	0,48	1	no	
	10	mg/kg	10	86	17.05.17	0,92	0,91	0,94	0,05	no	
	11	mg/kg	46	50	31.05.17	0,42	0,43	0,41	0,1	no	-

Parameter	Teilnehmer	Einheit	Proben-Nr. 1	Proben-Nr. 2	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis 1	Ergebnis 2	Bestimmungsgrenze	Inkl. WF	Wiederfindungsrate [%]
Analyte	Participant	Unit	Sample No. 1	Sample No. 2	Date of analysis	Result (Mean)	Result 1	Result 2	Limit of determination	Incl. RR	Recovery rate [%]
<b>Barium</b>	1	mg/kg	30	66							
	2	mg/kg	28	68							
	3	mg/kg	21	75							
	4	mg/kg	20	76							
	5	mg/kg	17	-	26.04.17	0,202	0,202		0,011	no	
	6	mg/kg	8	88	05.05.17	< 3,860	< 3,86	< 3,86	3,86	no	81
	7	mg/kg	32	64	10.05.	0,22	0,22	0,23	0,09	no	
	8	mg/kg	19	77		<0,5	<0,5	<0,5	<0,5	no	
	9	mg/kg	18	78	20.04.17	0,22	0,22	0,22	0,1	no	
	10	mg/kg	10	86	17.05.17	0,22	0,21	0,22	0,02	no	
	11	mg/kg	46	50	31.05.17	0,32	0,33	0,3	0,1	no	-

Parameter	Teilnehmer	Einheit	Proben-Nr. 1	Proben-Nr. 2	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis 1	Ergebnis 2	Bestimmungsgrenze	Inkl. WF	Wiederfindungsrate [%]
Analyte	Participant	Unit	Sample No. 1	Sample No. 2	Date of analysis	Result (Mean)	Result 1	Result 2	Limit of determination	Incl. RR	Recovery rate [%]
<b>Bor / Boron</b>	1	mg/kg	30	66	17/05	5,35	5,57	5,13	5ppb		
	2	mg/kg	28	68							
	3	mg/kg	21	75							
	4	mg/kg	20	76							
	5	mg/kg	17	-	26.04.17	4,4	4,4		0,07	no	
	6	mg/kg	8	88	05.05.17	3,156	3,176	3,136	2	no	---
	7	mg/kg	32	64	not analyzed						
	8	mg/kg	19	77		3,78	3,79	3,76	<1	no	
	9	mg/kg	18	78	20.04.17	3,3	3,3	3,3	0,6	no	
	10	mg/kg	10	86	17.05.17	3,8	4,06	3,51	0,1	no	
	11	mg/kg	46	50	31.05.17	3,79	3,76	3,82	1	no	-

Parameter	Teilnehmer	Einheit	Proben-Nr. 1	Proben-Nr. 2	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis 1	Ergebnis 2	Bestimmungsgrenze	Inkl. WF	Wiederfindungsrate [%]
Analyte	Participant	Unit	Sample No. 1	Sample No. 2	Date of analysis	Result (Mean)	Result 1	Result 2	Limit of determination	Incl. RR	Recovery rate [%]
<b>Calcium</b>	1	mg/kg	30	66	04/05	248	235	260	5ppb		
	2	mg/kg	28	68	20.05.17	203	207	199	30	no	
	3	mg/kg	21	75							
	4	mg/kg	20	76	20.04.17	248	249,5	247		no	
	5	mg/kg	17	-							
	6	mg/kg	8	88	08.05.17	236,75	242,1	231,4	82,08	no	94
	7	mg/kg	32	64	09.05.	234	234	235	18	no	
	8	mg/kg	19	77		254	254	253	<100	no	
	9	mg/kg	18	78	20.04.17	234	232	235		no	
	10	mg/kg	10	86	17.05.17	233	231	235	10	no	
	11	mg/kg	46	50	31.05.17	231	239	223	5	no	-

Parameter	Teilnehmer	Einheit	Proben-Nr. 1	Proben-Nr. 2	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis 1	Ergebnis 2	Bestimmungsgrenze	Inkl. WF	Wiederfindungsrate [%]
Analyte	Participant	Unit	Sample No. 1	Sample No. 2	Date of analysis	Result (Mean)	Result 1	Result 2	Limit of determination	Incl. RR	Recovery rate [%]
Cadmium	1	mg/kg	30	66	11/05	0,038	0,036	0,039	5ppb		
	2	mg/kg	28	68	20.05.17	0,045	0,045	0,044	0,006	no	114
	3	mg/kg	21	75							
	4	mg/kg	20	76	20.04.17	0,042	0,041	0,0425		no	
	5	mg/kg	17	-	26.04.17	0,039	0,039		0,0002	no	
	6	mg/kg	8	88	05.05.17	0,036	0,038	0,034	0,003	no	102
	7	mg/kg	32	64	10.05.	0,041	0,041	0,042	0,03	no	
	8	mg/kg	19	77		0,037	0,036	0,038	<0,005	no	
	9	mg/kg	18	78	20.04.17	0,042	0,041	0,042	0,001	no	
	10	mg/kg	10	86	17.05.17	0,039	0,040	0,038	0,002	no	
	11	mg/kg	46	50	31.05.17	0,041	0,038	0,044	0,01	no	-

Parameter	Teilnehmer	Einheit	Proben-Nr. 1	Proben-Nr. 2	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis 1	Ergebnis 2	Bestimmungsgrenze	Inkl. WF	Wiederfindungsrate [%]
Analyte	Participant	Unit	Sample No. 1	Sample No. 2	Date of analysis	Result (Mean)	Result 1	Result 2	Limit of determination	Incl. RR	Recovery rate [%]
<b>Cobalt</b>	1	mg/kg	30	66							
	2	mg/kg	28	68							
	3	mg/kg	21	75							
	4	mg/kg	20	76	20.04.17	< 0,050	<0,050	<0,050		no	
	5	mg/kg	17	-	26.04.17	0,0094	0,0094		0,00022	no	
	6	mg/kg	8	88	05.05.17	< 0,160	< 0,16	< 0,16	0,16	yes	121
	7	mg/kg	32	64	10.05.	0,012	0,013	0,01	0,03	no	
	8	mg/kg	19	77		0,01	0,008	0,011	<0,005	no	
	9	mg/kg	18	78	20.04.17	0,0096	0,0096	0,0095	0,003	no	
	10	mg/kg	10	86	17.05.17	0,014	0,012	0,016	0,005	no	
	11	mg/kg	46	50	31.05.17	<0,01	<0,01	<0,01	0,01	no	-



Parameter	Teilnehmer	Einheit	Proben-Nr. 1	Proben-Nr. 2	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis 1	Ergebnis 2	Bestimmungsgrenze	Inkl. WF	Wiederfindungsrate [%]
Analyte	Participant	Unit	Sample No. 1	Sample No. 2	Date of analysis	Result (Mean)	Result 1	Result 2	Limit of determination	Incl. RR	Recovery rate [%]
<b>Kupfer / Copper</b>	1	mg/kg	30	66	03/05	1,99	2,02	1,95	5ppb		
	2	mg/kg	28	68	20.05.17	1,55	1,57	1,53	0,06	no	101
	3	mg/kg	21	75							
	4	mg/kg	20	76	20.04.17	1,99	1,96	2,01		no	
	5	mg/kg	17	-	26.04.17	2,05	2,05		0,03	no	
	6	mg/kg	8	88	05.05.17	2,18	2,305	2,055	1	no	87
	7	mg/kg	32	64	10.05.	1,95	1,94	1,97	0,06	no	
	8	mg/kg	19	77		2,06	2,07	2,05	<0,4	no	
	9	mg/kg	18	78	20.04.17	2	2	2	0,1	no	
	10	mg/kg	10	86	17.05.17	1,84	1,85	1,83	0,05	no	
	11	mg/kg	46	50	31.05.17	1,93	1,96	1,89	0,1	no	-

Parameter	Teilnehmer	Einheit	Proben-Nr. 1	Proben-Nr. 2	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis 1	Ergebnis 2	Bestimmungsgrenze	Inkl. WF	Wiederfindungsrate [%]
Analyte	Participant	Unit	Sample No. 1	Sample No. 2	Date of analysis	Result (Mean)	Result 1	Result 2	Limit of determination	Incl. RR	Recovery rate [%]
Chrom / Chromium	1	mg/kg	30	66							
	2	mg/kg	28	68	20.05.17	< 0,060	< 0,060	< 0,060	0,06	no	97
	3	mg/kg	21	75							
	4	mg/kg	20	76	20.04.17	0,054	0,055	0,0535		no	
	5	mg/kg	17	-	26.04.17	0,035	0,035		0,0022	no	
	6	mg/kg	8	88	09.05.17	< 0,420	< 0,42	< 0,42	0,42	no	84
	7	mg/kg	32	64	10.05.	<0,06	<0,06	<0,06	0,06	no	
	8	mg/kg	19	77		<0,10	<0,10	<0,10	<0,10	no	
	9	mg/kg	18	78	20.04.17	0,035	0,035	0,035	0,06	no	
	10	mg/kg	10	86	17.05.17	<0,05	<0,05	<0,05	0,05	no	
	11	mg/kg	46	50	31.05.17	0,045	0,044	0,045	0,01	no	-

Parameter	Teilnehmer	Einheit	Proben-Nr. 1	Proben-Nr. 2	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis 1	Ergebnis 2	Bestimmungsgrenze	Inkl. WF	Wiederfindungsrate [%]
Analyte	Participant	Unit	Sample No. 1	Sample No. 2	Date of analysis	Result (Mean)	Result 1	Result 2	Limit of determination	Incl. RR	Recovery rate [%]
Eisen / Iron	1	mg/kg	30	66	03/05	14,2	14,7	13,7	5ppb		
	2	mg/kg	28	68	20.05.17	14	14,2	13,7	0,3	no	99
	3	mg/kg	21	75							
	4	mg/kg	20	76	20.04.17	15,1	15,15	15		no	
	5	mg/kg	17	-	26.04.17	15,9	15,9		0,073	no	
	6	mg/kg	8	88	08.05.17	20,56	23,76	17,36	1,06	no	97
	7	mg/kg	32	64	05.05.	13,8	13,7	13,9	3	no	
	8	mg/kg	19	77		16,12	16,05	16,18	<1	no	
	9	mg/kg	18	78	20.04.17	15,1	15,1	15,1	0,6	no	
	10	mg/kg	10	86	17.05.17	15,1	15	15,2	0,2	no	
	11	mg/kg	46	50	31.05.17	13,6	13,6	13,5	0,5	no	-

Parameter	Teilnehmer	Einheit	Proben-Nr. 1	Proben-Nr. 2	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis 1	Ergebnis 2	Bestimmungsgrenze	Inkl. WF	Wiederfindungsrate [%]
Analyte	Participant	Unit	Sample No. 1	Sample No. 2	Date of analysis	Result (Mean)	Result 1	Result 2	Limit of determination	Incl. RR	Recovery rate [%]
<b>Kalium / Potassium</b>	1	mg/kg	30	66	04/05	14500	14400	14500	5ppb		
	2	mg/kg	28	68	20.05.17	12600	12600	12600	300	no	100
	3	mg/kg	21	75							
	4	mg/kg	20	76	20.04.17	13100	13220	12970		no	
	5	mg/kg	17	-							
	6	mg/kg	8	88	08.05.17	13720	13500	13940	69,24	no	94
	7	mg/kg	32	64	09.05.	13200	13224	13175	18	no	
	8	mg/kg	19	77		13311	13241	13380	<100	no	
	9	mg/kg	18	78	20.04.17	13358	13463	13253		no	
	10	mg/kg	10	86	17.05.17	12500	12600	12400	100	no	
	11	mg/kg	46	50	31.05.17	12600	12700	12500	5	no	-

Parameter	Teilnehmer	Einheit	Proben-Nr. 1	Proben-Nr. 2	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis 1	Ergebnis 2	Bestimmungsgrenze	Inkl. WF	Wiederfindungsrate [%]
Analyte	Participant	Unit	Sample No. 1	Sample No. 2	Date of analysis	Result (Mean)	Result 1	Result 2	Limit of determination	Incl. RR	Recovery rate [%]
<b>Magnesium</b>	1	mg/kg	30	66	04/05	786	785	787	5ppb		
	2	mg/kg	28	68							
	3	mg/kg	21	75	15.05.17	732,78	733,45	732,12			
	4	mg/kg	20	76	20.04.17	740	741,5	738		no	
	5	mg/kg	17	-	09.05.17	711,6	711,6		0,048	no	
	6	mg/kg	8	88	08.05.17	736,9	726,5	747,3	90,62	no	94
	7	mg/kg	32	64	09.05.	760	762	758	6	no	
	8	mg/kg	19	77		718	708	728	<100	no	
	9	mg/kg	18	78	20.04.17	699	697	701		no	
	10	mg/kg	10	86	17.05.17	724	733	718	10	no	
	11	mg/kg	46	50	31.05.17	758	782	734	5	no	-

Parameter	Teilnehmer	Einheit	Proben-Nr. 1	Proben-Nr. 2	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis 1	Ergebnis 2	Bestimmungsgrenze	Inkl. WF	Wiederfindungsrate [%]
Analyte	Participant	Unit	Sample No. 1	Sample No. 2	Date of analysis	Result (Mean)	Result 1	Result 2	Limit of determination	Incl. RR	Recovery rate [%]
<b>Mangan / Manganese</b>	1	mg/kg	30	66	03/05	3,53	3,61	3,44	5ppb		
	2	mg/kg	28	68	20.05.17	3,17	3,19	3,14	0,06	no	94
	3	mg/kg	21	75							
	4	mg/kg	20	76	20.04.17	3,75	3,71	3,785		no	
	5	mg/kg	17	-	26.04.17	4,1	4,1		0,01	no	
	6	mg/kg	8	88	08.05.17	3,8895	3,884	3,895	0,64	yes	79
	7	mg/kg	32	64	09.05.	3,69	3,71	3,67	0,03	no	
	8	mg/kg	19	77		3,89	3,88	3,9	<0,2	no	
	9	mg/kg	18	78	20.04.17	3,8	3,8	3,8	0,1	no	
	10	mg/kg	10	86	17.05.17	3,47	3,51	3,45	0,05	no	
	11	mg/kg	46	50	31.05.17	3,3	3,33	3,26	0,1	no	-

Parameter	Teilnehmer	Einheit	Proben-Nr. 1	Proben-Nr. 2	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis 1	Ergebnis 2	Bestimmungsgrenze	Inkl. WF	Wiederfindungsrate [%]
Analyte	Participant	Unit	Sample No. 1	Sample No. 2	Date of analysis	Result (Mean)	Result 1	Result 2	Limit of determination	Incl. RR	Recovery rate [%]
<b>Molybdän / Molybdenum</b>	1	mg/kg	30	66							
	2	mg/kg	28	68							
	3	mg/kg	21	75							
	4	mg/kg	20	76	20.04.17	0,198	0,197	0,199		no	
	5	mg/kg	17	-	26.04.17	0,203	0,203		0,012	no	
	6	mg/kg	8	88	08.05.17	< 1,480	< 1,48	< 1,48	1,48	yes	72
	7	mg/kg	32	64	10.05.	0,18	0,18	0,18	0,03	no	
	8	mg/kg	19	77		0,18	0,17	0,19	<0,01	no	
	9	mg/kg	18	78	20.04.17	0,2	0,2	0,2	0,06	no	
	10	mg/kg	10	86	17.05.17	0,26	0,26	0,26	0,05	no	
	11	mg/kg	46	50	31.05.17	0,2	0,2	0,2	0,01	no	-

Parameter	Teilnehmer	Einheit	Proben-Nr. 1	Proben-Nr. 2	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis 1	Ergebnis 2	Bestimmungsgrenze	Inkl. WF	Wiederfindungsrate [%]
Analyte	Participant	Unit	Sample No. 1	Sample No. 2	Date of analysis	Result (Mean)	Result 1	Result 2	Limit of determination	Incl. RR	Recovery rate [%]
<b>Natrium / Sodium</b>	1	mg/kg	30	66	03/05	212	215	208	5ppb		
	2	mg/kg	28	68							
	3	mg/kg	21	75	15.05.17	211,57	210,69	212,45			
	4	mg/kg	20	76							
	5	mg/kg	17	-	09.05.17	178,1	178,1		0,59	no	
	6	mg/kg	8	88	08.05.17	198,9	199,6	198,2	51,78	no	84
	7	mg/kg	32	64	09.05.	198	197	198	9	no	
	8	mg/kg	19	77		195	197	192	<100	no	
	9	mg/kg	18	78	20.04.17	184	184	184		no	
	10	mg/kg	10	86	17.05.17	198	200	196	10	no	
	11	mg/kg	46	50	31.05.17	182	186	178	5	no	-



Parameter	Teilnehmer	Einheit	Proben-Nr. 1	Proben-Nr. 2	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis 1	Ergebnis 2	Bestimmungsgrenze	Inkl. WF	Wiederfindungsrate [%]
Analyte	Participant	Unit	Sample No. 1	Sample No. 2	Date of analysis	Result (Mean)	Result 1	Result 2	Limit of determination	Incl. RR	Recovery rate [%]
Nickel	1	mg/kg	30	66	03/05	0,036	0,036	0,035	5ppb		
	2	mg/kg	28	68	20.05.17	< 0,060	< 0,060	< 0,060	0,06	no	98
	3	mg/kg	21	75							
	4	mg/kg	20	76	20.04.17	<0,10	<0,10	<0,10		no	
	5	mg/kg	17	-	26.04.17	0,0377	0,0377		0,0026	no	
	6	mg/kg	8	88	05.05.17	< 2,000	< 2,00	< 2,00	2	yes	76
	7	mg/kg	32	64	10.05.	<0,2	<0,2	<0,2	0,2	no	
	8	mg/kg	19	77		<0,10	<0,10	<0,10	<0,10	no	
	9	mg/kg	18	78	20.04.17	0,041	0,042	0,039	0,06	no	
	10	mg/kg	10	86	17.05.17	0,035	0,033	0,037	0,01	no	
	11	mg/kg	46	50	31.05.17	0,053	0,057	0,048	0,01	no	-

Parameter	Teilnehmer	Einheit	Proben-Nr. 1	Proben-Nr. 2	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis 1	Ergebnis 2	Bestimmungsgrenze	Inkl. WF	Wiederfindungsrate [%]
Analyte	Participant	Unit	Sample No. 1	Sample No. 2	Date of analysis	Result (Mean)	Result 1	Result 2	Limit of determination	Incl. RR	Recovery rate [%]
Phosphor / Phosphorus	1	mg/kg	30	66	04/05	1400	1400	1400	5ppb		
	2	mg/kg	28	68							
	3	mg/kg	21	75							
	4	mg/kg	20	76							
	5	mg/kg	17	-							
	6	mg/kg	8	88	05.05.17	1501	1484	1518	15,44	no	95
	7	mg/kg	32	64	09.05.	1499	1467	1531	9	no	
	8	mg/kg	19	77		1435	1450	1419	<100	no	
	9	mg/kg	18	78	20.04.17	1399	1393	1405		no	
	10	mg/kg	10	86	17.05.17	1480	1505	1469	20	no	
	11	mg/kg	46	50	31.05.17	1445	1455	1435	5	no	-

Parameter	Teilnehmer	Einheit	Proben-Nr. 1	Proben-Nr. 2	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis 1	Ergebnis 2	Bestimmungsgrenze	Inkl. WF	Wiederfindungsrate [%]
Analyte	Participant	Unit	Sample No. 1	Sample No. 2	Date of analysis	Result (Mean)	Result 1	Result 2	Limit of determination	Incl. RR	Recovery rate [%]
Rubidium	1	mg/kg	30	66							
	2	mg/kg	28	68							
	3	mg/kg	21	75							
	4	mg/kg	20	76							
	5	mg/kg	17	-	26.04.17	2,71	2,71		0,17	no	
	6	mg/kg	8	88	10.05.17	< 2,000	< 2,00	< 2,00	2	no	---
	7	mg/kg	32	64	not analyzed						
	8	mg/kg	19	77							
	9	mg/kg	18	78	20.04.17	2,8	2,8	2,7		no	
	10	mg/kg	10	86	17.05.17	2,8	2,84	2,76	0,1	no	
	11	mg/kg	46	50	31.05.17	2,72	2,73	2,71	0,1	no	-

Parameter	Teilnehmer	Einheit	Proben-Nr. 1	Proben-Nr. 2	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis 1	Ergebnis 2	Bestimmungsgrenze	Inkl. WF	Wiederfindungsrate [%]
Analyte	Participant	Unit	Sample No. 1	Sample No. 2	Date of analysis	Result (Mean)	Result 1	Result 2	Limit of determination	Incl. RR	Recovery rate [%]
<b>Schwefel / Sulfur</b>	1	mg/kg	30	66							
	2	mg/kg	28	68							
	3	mg/kg	21	75							
	4	mg/kg	20	76							
	5	mg/kg	17	-							
	6	mg/kg	8	88	05.05.17	11,745	12,17	11,32	10	no	97
	7	mg/kg	32	64	not analyzed						
	8	mg/kg	19	77		1050	1011	1089	<100	no	
	9	mg/kg	18	78		n.b.	n.b.	n.b.			
	10	mg/kg	10	86	17.05.17	1800	1800	1700	500	no	
	11	mg/kg	46	50	31.05.17	1139	1146	1132	5	no	-

Parameter	Teilnehmer	Einheit	Proben-Nr. 1	Proben-Nr. 2	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis 1	Ergebnis 2	Bestimmungsgrenze	Inkl. WF	Wiederfindungsrate [%]
Analyte	Participant	Unit	Sample No. 1	Sample No. 2	Date of analysis	Result (Mean)	Result 1	Result 2	Limit of determination	Incl. RR	Recovery rate [%]
<b>Strontium</b>	1	mg/kg	30	66							
	2	mg/kg	28	68							
	3	mg/kg	21	75							
	4	mg/kg	20	76							
	5	mg/kg	17	-	26.04.17	0,71	0,71		0,005	no	
	6	mg/kg	8	88	10.05.17	0,4975	0,493	0,502	0,4	yes	50
	7	mg/kg	32	64	not analyzed						
	8	mg/kg	19	77		0,74	0,74	0,74	<0,5	no	
	9	mg/kg	18	78	20.04.17	0,74	0,74	0,74	0,3	no	
	10	mg/kg	10	86	17.05.17	0,75	0,75	0,74	0,05	no	
	11	mg/kg	46	50	31.05.17	0,71	0,71	0,71	0,1	no	-

Parameter	Teilnehmer	Einheit	Proben-Nr. 1	Proben-Nr. 2	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis 1	Ergebnis 2	Bestimmungsgrenze	Inkl. WF	Wiederfindungsrate [%]
Analyte	Participant	Unit	Sample No. 1	Sample No. 2	Date of analysis	Result (Mean)	Result 1	Result 2	Limit of determination	Incl. RR	Recovery rate [%]
<b>Zink / Zinc</b>	1	mg/kg	30	66	03/05	7,32	7,41	7,23	5ppb		
	2	mg/kg	28	68	20.05.17	7,49	7,6	7,37	0,3	no	82
	3	mg/kg	21	75							
	4	mg/kg	20	76	20.04.17	8,32	8,335	8,295		no	
	5	mg/kg	17	-	26.04.17	7,55	7,55		0,03	no	
	6	mg/kg	8	88	08.05.17	8,1485	9,015	7,282	1,3	no	97
	7	mg/kg	32	64	05.05.	8,56	8,48	8,65	0,9	no	
	8	mg/kg	19	77		8,72	8,64	8,79	<0,5	no	
	9	mg/kg	18	78	20.04.17	8,2	8,2	8,2	4	no	
	10	mg/kg	10	86	17.05.17	6,8	6,9	6,7	0,1	no	
	11	mg/kg	46	50	31.05.17	7,22	7,26	7,17	0,1	no	-

## 5.1.2 Analytical Methods

Parameter	Teilnehmer	Methodenbeschreibung	Homogenisierung	Einwaage	digestion: Methode	digestion: Lösung	Kalibrierverfahren/Referenzmaterial	Methode akkreditiert nach ISO/IEC 17025	Sonstige Hinweise
Analyte	Participant	Method description	Homogenisation	sample weight	Digestion: Method	Digestion: Solution	Calibration / reference material	Method accredited via ISO/IEC 17025	Further remarks
Aluminium	1	AOAC 993.14		*	Microwave	5% HNO <sub>3</sub>	NIST	no	* approx. 0.5 g sample weight for double determination
	2	DIN EN ISO 11885		0,5 g	Microwave pressure digest.	aqua regia	SPS-SW1	yes	
	3								
	4								
	5			0.5 g	Microwave digestion	8 mL HNO <sub>3</sub> + 2 mL H <sub>2</sub> O <sub>2</sub>	External Calibration with internal Standard: Germanium	no	
	6	internal Method	no	0,99g / 1,00g	ASU L 00.00 – 19/1		---	yes	
	7	Al with ICP-MS after Microwave digestion	mixing	1,0 g	Microwave	HNO <sub>3</sub>	ext. With IS In	yes	
	8	DIN EN ISO 11885		0,5 g	Microwave pressure digestion	HNO <sub>3</sub> , H <sub>2</sub> O <sub>2</sub> , H <sub>2</sub> O		yes	
	9	ICP-MS	no	450 mg	Microwave	HNO <sub>3</sub> /H <sub>2</sub> O <sub>2</sub>	1;3 Kalib. (0,505-2,04 mg/l)	yes	1 (Lichen IAEA-336)
	10	EN 15763	no	400	Microwave pressure digestion	HNO <sub>3</sub>	Linear	yes	
	11	ICP-MS	yes	0,5 g	Microwave	HNO <sub>3</sub> / H <sub>2</sub> O <sub>2</sub>	external	yes	-

Parameter	Teilnehmer	Methodenbeschreibung	Homogenisierung	Einwaage	Aufschluss: Methode	Aufschluss: Lösung	Kalibrierverfahren/Referenzmaterial	Methode akkreditiert nach ISO/IEC 17025	Sonstige Hinweise
Analyte	Participant	Method description	Homogenisation	sample weight	Digestion: Method	Digestion: Solution	Calibration / reference material	Method accredited via ISO/IEC 17025	Further remarks
Bor /Boron	1	AOAC 993.14		*	Microwave	5% H NO3	NIST	no	
	2								
	3								
	4								
	5			0.5 g	Microwave digestion	8 mL HNO3 + 2 mL H2O2	External Calibration with internal Standard: Germanium	no	
	6	internal Method	no	0,99g / 1,00g	ASU L 00.00 – 19/1		---	yes	
	7								
	8	DIN EN ISO 11885		0,5 g	Microwave pressure digestion	HNO3, H2O2, H2O		yes	
	9	ICP-MS	no	450 mg	Microwave	HNO3/H2O2	4; 5 Calib. (0,02-0,4 mg/l)	yes	2 (Citrus Leafs NCS ZC)
	10	EN 15763	no	400	Microwave pressure digestion	HNO3	Linear	yes	
	11	ICP-OES	yes	0,5 g	Microwave	HNO3 / H2O2	external	yes	-



Parameter	Teilnehmer	Methodenbeschreibung	Homogenisierung	Einwaage	Aufschluss: Methode	Aufschluss: Lösung	Kalibrierverfahren/Referenzmaterial	Methode akkreditiert nach ISO/IEC 17025	Sonstige Hinweise
Analyte	Participant	Method description	Homogenisation	sample weight	Digestion: Method	Digestion: Solution	Calibration / reference material	Method accredited via ISO/IEC 17025	Further remarks
Barium	1								
	2								
	3								
	4								
	5			0,5 g	Microwave digestion	8 mL HNO3 + 2 mL H2O2	External Calibration with internal Standard: Indium	no	
	6	internal Method	no	0,99g / 1,00g	ASU L 00.00 – 19/1		LGC7162	yes	
	7	Ba with ICP-MS after Microwave digestion	mixing	0,2 g	Microwave	HNO3	ext. mit IS In	no	
	8	DIN EN ISO 11885		0,5 g	Microwave pressure digestion	HNO3, H2O2, H2O		no	
	9	ICP-MS	no	450 mg	Microwave	HNO3/H2O2	1;2 Calib. (0,002-0,04 mg/l)	yes	3 (OYSTER Tissue NIST 1566b)
	10	EN 15763	no	400	Microwave pressure digestion	HNO3	Linear	yes	
	11	ICP-MS	yes	0,5 g	Microwave	HNO3 / H2O2	external Calibration with internal Standard: Indium	yes	-

Parameter	Teilnehmer	Methodenbeschreibung	Homogenisierung	Einwaage	Aufschluss: Methode	Aufschluss: Lösung	Kalibrierverfahren/ Referenzmaterial	Methode akkreditiert nach ISO/IEC 17025	Sonstige Hinweise
Analyte	Participant	Method description	Homogenisation	sample weight	Digestion: Method	Digestion: Solution	Calibration / reference material	Method accredited via ISO/IEC 17025	Further remarks
Calcium	1	AOAC 993.14		*	Microwave	5% HNO <sub>3</sub>	NIST	yes	
	2	DIN EN ISO 11885		0,5 g	Microwave pressure digestion.	aqua regia	SPS-SW1	yes	
	3								
	4	ICP-OES	man. Mixing	0,5 g					
	5								
	6	ASU L 00.00 – 144	no	0,99g / 1,00g	ASU L 00.00 – 19/1		ERM-CE278k	yes	
	7	Ca with ICP-MS after Microwave digestion	mixing	0,2 g	Microwave	HNO <sub>3</sub>	ext. by IS In	yes	
	8	DIN EN ISO 11885		0,5 g	Microwave pressure digestion	HNO <sub>3</sub> , H <sub>2</sub> O <sub>2</sub> , H <sub>2</sub> O		yes	
	9	ICP-MS	no	450 mg	Microwave	HNO <sub>3</sub> /H <sub>2</sub> O <sub>2</sub>	2;3 Calib.(1-10 mg/l)	yes	4 (Apple Leafs 1515)
	10	EN 15763	no	400	Microwave pressure digestion	HNO <sub>3</sub>	Linear	yes	
	11	ICP-OES	yes	0,5 g	Microwave	HNO <sub>3</sub> / H <sub>2</sub> O <sub>2</sub>	external	yes	-

Parameter	Teilnehmer	Methodenbeschreibung	Homogenisierung	Einwaage	Aufschluss: Methode	Aufschluss: Lösung	Kalibrierverfahren/ Referenzmaterial	Methode akkreditiert nach ISO/IEC 17025	Sonstige Hinweise
Analyte	Participant	Method description	Homogenisation	sample weight	Digestion: Method	Digestion: Solution	Calibration / reference material	Method accredited via ISO/IEC 17025	Further remarks
Cadmium	1	AOAC 993.14		*	Microwave	5% HNO <sub>3</sub>	NIST	yes	
	2	DIN EN ISO 11885		0,5 g	Microwave pressure digestion.	aqua regia	SPS-SW1	yes	
	3								
	4	ICP-MS	man. Mixing	0,5 g					
	5			0.5 g	Microwave digestion	8 mL HNO <sub>3</sub> + 2 mL H <sub>2</sub> O <sub>2</sub>	External Calibration with internal Standard: Germanium	no	
	6	internal Method	no	0,99g / 1,00g	ASU L 00.00 – 19/1		ERM-CE278k	yes	
	7	Cd with ICP-MS after Microwave digestion	mixing	0,2 g	Microwave	HNO <sub>3</sub>	ext. With IS In	yes	
	8	DIN EN ISO 17294		0,5 g	Microwave pressure digestion	HNO <sub>3</sub> , H <sub>2</sub> O <sub>2</sub> , H <sub>2</sub> O		yes	
	9	ICP-MS	no	450 mg	Microwave	HNO <sub>3</sub> /H <sub>2</sub> O <sub>2</sub>	2;3;4 Calib. (00,0001-0,04 mg/l)	yes	5 (Tomato Leafs 1573a)
	10	EN 15763	no	400	Microwave pressure digestion	HNO <sub>3</sub>	Linear	yes	
	11	ICP-MS	yes	0,5 g	Microwave	HNO <sub>3</sub> / H <sub>2</sub> O <sub>2</sub>	external	yes	-

Parameter	Teilnehmer	Methodenbeschreibung	Homogenisierung	Einwaage	Aufschluss: Methode	Aufschluss: Lösung	Kalibrierverfahren/ Referenzmaterial	Methode akkreditiert nach ISO/IEC 17025	Sonstige Hinweise
Analyte	Participant	Method description	Homogenisation	sample weight	Digestion: Method	Digestion: Solution	Calibration / reference material	Method accredited via ISO/IEC 17025	Further remarks
Cobalt	1								
	2								
	3								
	4	ICP-MS	man. Mixing	0,5 g					
	5			0,5 g	Microwave digestion	8 mL HNO3 + 2 mL H2O2	External Calibration with internal Standard: Germanium	no	
	6	internal Method	no	0,99g / 1,00g	ASU L 00.00 – 19/1		LGC7162	yes	
	7	Co with ICP-MS after Microwave digestion	mixing	0,2 g	Microwave	HNO3	ext. With IS In	no	
	8	DIN EN ISO 17294		0,5 g	Microwave pressure digestion	HNO3, H2O2, H2O		no	
	9	ICP-MS	no	450 mg	Microwave	HNO3/H2O2	1;2;3 Calib. (0,0001-0,04 mg/l)	yes	
	10	EN 15763	no	400	Microwave pressure digestion	HNO3	Linear	yes	
	11	ICP-MS	yes	0,5 g	Microwave	HNO3 / H2O2	external	yes	-

Parameter	Teilnehmer	Methodenbeschreibung	Homogenisierung	Einwaage	Aufschluss: Methode	Aufschluss: Lösung	Kalibrierverfahren/Referenzmaterial	Methode akkreditiert nach ISO/IEC 17025	Sonstige Hinweise
Analyte	Participant	Method description	Homogenisation	sample weight	Digestion: Method	Digestion: Solution	Calibration / reference material	Method accredited via ISO/IEC 17025	Further remarks
Chrom / Chromium	1								
	2	DIN EN ISO 11885		0,5 g	Microwave-pressure digest.	aqua regia	SPS-SW2	yes	
	3								
	4	ICP-MS	man. Mixing	0,5 g					
	5			0,5 g	Microwave digestion	8 mL HNO3 + 2 mL H2O2	External Calibration with internal Standard: Germanium	no	
	6	internal Method	no	0,99g / 1,00g	ASU L 00.00 – 19/1		LGC7162	yes	
	7	Cr with ICP-MS after Microwave digestion	mixing	1,0 g	Microwave	HNO3	ext. With IS In	yes	
	8	DIN EN ISO 17294		0,5 g	Microwave pressure digestion	HNO3, H2O2, H2O		yes	
	9	ICP-MS	no	450 mg	Microwave	HNO3/H2O2	1;2 Calib. (0,0001-0,04 mg/l)	yes	
	10	EN 15763	no	400	Microwave pressure digestion	HNO3	Linear	yes	
	11	ICP-MS	yes	0,5 g	Microwave	HNO3 / H2O2	external	yes	-

Parameter	Teilnehmer	Methodenbeschreibung	Homogenisierung	Einwaage	Aufschluss: Methode	Aufschluss: Lösung	Kalibrierverfahren/ Referenzmaterial	Methode akkreditiert nach ISO/IEC 17025	Sonstige Hinweise
Analyte	Participant	Method description	Homogenisation	sample weight	Digestion: Method	Digestion: Solution	Calibration / reference material	Method accredited via ISO/IEC 17025	Further remarks
Kupfer / Copper	1	AOAC 993.14		*	Microwave	5% H NO3	NIST	yes	
	2	DIN EN ISO 11885		0,5 g	Microwave pressure digestion	aqua regia	SPS-SW1	yes	
	3								
	4	ICP-MS	man. Mixing	0,5 g					
	5			0,5 g	Microwave digestion	8 mL HNO3 + 2 mL H2O2	External Calibration with internal Standard: Germanium	no	
	6	ASU L 00.00 – 144	no	0,99g / 1,00g	ASU L 00.00 – 19/1		ERM-CE278k	yes	
	7	Cu with ICP-MS after Microwave digestion	mixing	0,2 g	Microwave	HNO3	ext. With IS In	yes	
	8	DIN EN ISO 11885		0,5 g	Microwave pressure digestion	HNO3, H2O2, H2O		yes	
	9	ICP-MS	no	450 mg	Microwave	HNO3/H2O2	1;2 Calib. (0,0001-0,04 mg/l)	yes	
	10	EN 15763	no	400	Microwave pressure digestion	HNO3	Linear	yes	
	11	ICP-MS	yes	0,5 g	Microwave	HNO3 / H2O2	external	yes	-

Parameter	Teilnehmer	Methodenbeschreibung	Homogenisierung	Einwaage	Aufschluss: Methode	Aufschluss: Lösung	Kalibrierverfahren/Referenzmaterial	Methode akkreditiert nach ISO/IEC 17025	Sonstige Hinweise
Analyte	Participant	Method description	Homogenisation	sample weight	Digestion: Method	Digestion: Solution	Calibration / reference material	Method accredited via ISO/IEC 17025	Further remarks
Eisen / Iron	1	AOAC 993.14		*	Microwave	5% HNO <sub>3</sub>	NIST	yes	
	2	DIN EN ISO 11885		0,5 g	Microwave pressure digestion.	aqua regia	SPS-SW1	yes	
	3								
	4	ICP-MS	man. Mixing	0,5 g					
	5			0.5 g	Microwave digestion	8 mL HNO <sub>3</sub> + 2 mL H <sub>2</sub> O <sub>2</sub>	External Calibration with internal Standard: Germanium	no	
	6	ASU L 00.00 – 144	no	0,99g / 1,00g	ASU L 00.00 – 19/1		LGC7162	yes	
	7	Fe with ICP-MS after Microwave digestion	mixing	0,2 g	Microwave	HNO <sub>3</sub>	ext. With IS In	yes	
	8	DIN EN ISO 11885		0,5 g	Microwave pressure digestion	HNO <sub>3</sub> , H <sub>2</sub> O <sub>2</sub> , H <sub>2</sub> O		yes	
	9	ICP-MS	no	450 mg	Microwave	HNO <sub>3</sub> /H <sub>2</sub> O <sub>2</sub>	1;2 Calib. (0,55-2,4 mg/l)	yes	
	10	EN 15763	no	400	Microwave pressure digestion	HNO <sub>3</sub>	Linear	yes	
	11	ICP-OES	yes	0,5 g	Microwave	HNO <sub>3</sub> / H <sub>2</sub> O <sub>2</sub>	external	yes	-

Parameter	Teilnehmer	Methodenbeschreibung	Homogenisierung	Einwaage	Aufschluss: Methode	Aufschluss: Lösung	Kalibrierverfahren/ Referenzmaterial	Methode akkreditiert nach ISO/IEC 17025	Sonstige Hinweise
Analyte	Participant	Method description	Homogenisation	sample weight	Digestion: Method	Digestion: Solution	Calibration / reference material	Method accredited via ISO/IEC 17025	Further remarks
<b>Kalium / Potassium</b>	1	AOAC 993.14		*	Microwave	5% H NO3	NIST	yes	
	2	DIN EN ISO 11885		0,5 g	Microwave pressure digest.	aqua regia		yes	
	3								
	4	FI-AAS	man. Mixing	0,5 g					
	5								
	6	ASU L 00.00 – 144	no	0,99g / 1,00g	ASU L 00.00 – 19/1		LGC7162	yes	
	7	K with ICP-MS after Microwave digestion	mixing	0,2 g	Microwave	HNO3	ext. With IS In	yes	
	8	DIN EN ISO 11885		0,5 g	Microwave pressure digestion	HNO3, H2O2, H2O		yes	
	9	ICP-MS	no	450 mg	Microwave	HNO3/H2O2	1;2 Calib. (1-10 mg/l)	yes	
	10	EN 15763	no	400	Microwave pressure digestion	HNO3	Linear	yes	
	11	ICP-OES	yes	0,5 g	Microwave	HNO3 / H2O2	external	yes	-



Parameter	Teilnehmer	Methodenbeschreibung	Homogenisierung	Einwaage	Aufschluss: Methode	Aufschluss: Lösung	Kalibrierverfahren/ Referenzmaterial	Methode akkreditiert nach ISO/IEC 17025	Sonstige Hinweise
Analyte	Participant	Method description	Homogenisation	sample weight	Digestion: Method	Digestion: Solution	Calibration / reference material	Method accredited via ISO/IEC 17025	Further remarks
Magnesium	1	AOAC 993.14		*	Microwave	5% HNO <sub>3</sub>	NIST	yes	
	2								
	3	UNI EN 15505:2008		300 mg	HNO <sub>3</sub> + H <sub>2</sub> O <sub>2</sub> in MW oven		Mg solution 0,1% Fluka code 42992 / batch BCBN4821V	yes	AA Flame Atomization
	4	ICP-OES	man. Mixing	0,5 g					
	5			0,5 g	Microwave digestion	8 mL HNO <sub>3</sub> + 2 mL H <sub>2</sub> O <sub>2</sub>	External Calibration with internal Standard: Germanium	no	
	6	ASU L 00.00 – 144	no	0,99g / 1,00g	ASU L 00.00 – 19/1		LGC7162	yes	
	7	Mg with ICP-MS after Microwave digestion	mixing	0,2 g	Microwave	HNO <sub>3</sub>	ext. With IS In	yes	
	8	DIN EN ISO 11885		0,5 g	Microwave pressure digestion	HNO <sub>3</sub> , H <sub>2</sub> O <sub>2</sub> , H <sub>2</sub> O		yes	
	9	ICP-MS	no	450 mg	Microwave	HNO <sub>3</sub> /H <sub>2</sub> O <sub>2</sub>	2;3;4 Calib.(1-6 mg/l)	yes	
	10	EN 15763	no	400	Microwave pressure digestion	HNO <sub>3</sub>	Linear	yes	
	11	ICP-OES	yes	0,5 g	Microwave	HNO <sub>3</sub> / H <sub>2</sub> O <sub>2</sub>	external	yes	-

Parameter	Teilnehmer	Methodenbeschreibung	Homogenisierung	Einwaage	Aufschluss: Methode	Aufschluss: Lösung	Kalibrierverfahren/ Referenzmaterial	Methode akkreditiert nach ISO/IEC 17025	Sonstige Hinweise
Analyte	Participant	Method description	Homogenisation	sample weight	Digestion: Method	Digestion: Solution	Calibration / reference material	Method accredited via ISO/IEC 17025	Further remarks
Mangan / Manganese	1	AOAC 993.14		*	Microwave	5% HNO <sub>3</sub>	NIST	yes	
	2	DIN EN ISO 11885		0,5 g	Microwave pressure digestion.	aqua regia	SPS-SW1	yes	
	3								
	4	ICP-MS	man. Mixing	0,5 g					
	5			0.5 g	Microwave digestion	8 mL HNO <sub>3</sub> + 2 mL H <sub>2</sub> O <sub>2</sub>	External Calibration with internal Standard: Germanium	no	
	6	ASU L 00.00 – 144	no	0,99g / 1,00g	ASU L 00.00 – 19/1		LGC7162	yes	
	7	Mn with ICP-MS after Microwave digestion	mixing	0,2 g	Microwave	HNO <sub>3</sub>	ext. With IS In	yes	
	8	DIN EN ISO 11885		0,5 g	Microwave pressure digestion	HNO <sub>3</sub> , H <sub>2</sub> O <sub>2</sub> , H <sub>2</sub> O		yes	
	9	ICP-MS	no	450 mg	Microwave	HNO <sub>3</sub> /H <sub>2</sub> O <sub>2</sub>	1;2 Calib. (0,03-0,24 mg/l)	yes	
	10	EN 15763	no	400	Microwave pressure digestion	HNO <sub>3</sub>	Linear	yes	
	11	ICP-MS	yes	0,5 g	Microwave	HNO <sub>3</sub> / H <sub>2</sub> O <sub>2</sub>	external	yes	-

Parameter	Teilnehmer	Methodenbeschreibung	Homogenisierung	Einwaage	Aufschluss: Methode	Aufschluss: Lösung	Kalibrierverfahren/Referenzmaterial	Methode akkreditiert nach ISO/IEC 17025	Sonstige Hinweise
Analyte	Participant	Method description	Homogenisation	sample weight	Digestion: Method	Digestion: Solution	Calibration / reference material	Method accredited via ISO/IEC 17025	Further remarks
Molybdän / Molybdenum	1								
	2								
	3								
	4	ICP-MS	man. Mixing	0,5 g					
	5			0.5 g	Microwave digestion	8 mL HNO3 + 2 mL H2O2	External Calibration with internal Standard: Rhodium	no	
	6	internal Method	no	0,99g / 1,00g	ASU L 00.00 – 19/1		LGC7162	yes	
	7	Mo with ICP-MS after Microwave digestion	mixing	0,2 g	Microwave	HNO3	ext. With IS In	yes	
	8	DIN EN ISO 17294		0,5 g	Microwave pressure digestion	HNO3, H2O2, H2O		no	
	9	ICP-MS	no	450 mg	Microwave	HNO3/H2O2	2;3;4 Calib. (0,001-0,04 mg/l)	yes	
	10	EN 15763	no	400	Microwave pressure digestion	HNO3	Linear	yes	
	11	ICP-MS	yes	0,5 g	Microwave	HNO3 / H2O2	external	yes	-

Parameter	Teilnehmer	Methodenbeschreibung	Homogenisierung	Einwaage	Aufschluss: Methode	Aufschluss: Lösung	Kalibrierverfahren/ Referenzmaterial	Methode akkreditiert nach ISO/IEC 17025	Sonstige Hinweise
Analyte	Participant	Method description	Homogenisation	sample weight	Digestion: Method	Digestion: Solution	Calibration / reference material	Method accredited via ISO/IEC 17025	Further remarks
Natrium /Sodium	1	AOAC 993.14		*	Microwave	5% H NO3	NIST	no	
	2								
	3	UNI EN 15505:2008		300 mg	HNO3 + H2O2 in MW Oven		Na solution 0,1% Fluka code 05201 / batch BCBT3122	yes	AA Flame-Atomization
	4								
	5			0.5 g	Microwave digestion	8 mL HNO3 + 2 mL H2O2	External Calibration with internal Standard: Rhodium	no	
	6	ASU L 00.00 – 144	no	0,99g / 1,00g	ASU L 00.00 – 19/1		ERM-CE278k	yes	
	7	Na with ICP-MS after Microwave digestion	mixing	0,2 g	Microwave	HNO3	ext. With IS In	yes	
	8	DIN EN ISO 11885		0,5 g	Microwave pressure digestion	HNO3, H2O2, H2O		yes	
	9	ICP-MS	no	450 mg	Microwave	HNO3/H2O2	1;2;3 Calib. (1-10 mg/l)	yes	
	10	EN 15763	no	400	Microwave pressure digestion	HNO3	Linear	yes	
	11	ICP-OES	yes	0,5 g	Microwave	HNO3 / H2O2	external	yes	-

Parameter	Teilnehmer	Methodenbeschreibung	Homogenisierung	Einwaage	Aufschluss: Methode	Aufschluss: Lösung	Kalibrierverfahren/ Referenzmaterial	Methode akkreditiert nach ISO/IEC 17025	Sonstige Hinweise
Analyte	Participant	Method description	Homogenisation	sample weight	Digestion: Method	Digestion: Solution	Calibration / reference material	Method accredited via ISO/IEC 17025	Further remarks
Nickel	1	AOAC 993.14		*	Microwave	5% H NO3	NIST	no	
	2	DIN EN ISO 11885		0,5 g	Microwave pressure digest.	aqua regia	SPS-SW1	yes	
	3								
	4	ICP-MS	man. Mixing	0,5 g					
	5			0.5 g	Microwave digestion	8 mL HNO3 + 2 mL H2O2	External Calibration with internal Standard: Germanium	no	
	6	internal Method	no	0,99g / 1,00g	ASU L 00.00 – 19/1		LGC7162	yes	
	7	Ni with ICP-MS after Microwave digestion	mixing	1,0 g	Microwave	HNO3	ext. With IS In	yes	
	8	DIN EN ISO 17294		0,5 g	Microwave pressure digestion	HNO3, H2O2, H2O		yes	
	9	ICP-MS	no	450 mg	Microwave	HNO3/H2O2	4;5 Calib. (0,001-0,04 mg/l)	yes	
	10	EN 15763	no	400	Microwave pressure digestion	HNO3	Linear	yes	
	11	ICP-MS	yes	0,5 g	Microwave	HNO3 / H2O2	external	yes	-

Parameter	Teilnehmer	Methodenbeschreibung	Homogenisierung	Einwaage	Aufschluss: Methode	Aufschluss: Lösung	Kalibrierverfahren/ Referenzmaterial	Methode akkreditiert nach ISO/IEC 17025	Sonstige Hinweise
Analyte	Participant	Method description	Homogenisation	sample weight	Digestion: Method	Digestion: Solution	Calibration / reference material	Method accredited via ISO/IEC 17025	Further remarks
Phosphor / Phosphorus	1	AOAC 993.14		*	Microwave	5% H NO3	NIST	yes	
	2								
	3								
	4								
	5								
	6	ASU L 00.00 – 144	no	0,99g / 1,00g	ASU L 00.00 – 19/1		LGC7162	yes	
	7	P with ICP-MS after Microwave digestion	mixing	0,2 g	Microwave	HNO3	ext. With IS In	no	
	8	DIN EN ISO 11885		0,5 g	Microwave pressure digestion	HNO3, H2O2, H2O		yes	
	9	ICP-MS	no	450 mg	Microwave	HNO3/H2O2	1;2;4 Calib. (0,5-8 mg/l)	yes	
	10	EN 15763	no	400	Microwave pressure digestion	HNO3	Linear	yes	
	11	ICP-OES	yes	0,5 g	Microwave	HNO3 / H2O2	external	yes	-

Parameter	Teilnehmer	Methodenbeschreibung	Homogenisierung	Einwaage	Aufschluss: Methode	Aufschluss: Lösung	Kalibrierverfahren/ Referenzmaterial	Methode akkreditiert nach ISO/IEC 17025	Sonstige Hinweise
Analyte	Participant	Method description	Homogenisation	sample weight	Digestion: Method	Digestion: Solution	Calibration / reference material	Method accredited via ISO/IEC 17025	Further remarks
Rubidium	1								
	2								
	3								
	4								
	5			0.5 g	Microwave digestion	8 mL HNO3 + 2 mL H2O2	External Calibration with internal Standard: Indium	no	
	6	internal Method	no	0,99g / 1,00g	ASU L 00.00 – 19/1		---	yes	
	7				Microwave				
	8				Microwave pressure digestion				
	9	ICP-MS	no	450 mg	Microwave	HNO3/H2O2	1;2;4 Calib. (0,0001-0,04 mg/l)	yes	
	10	EN 15763	no	400	Microwave pressure digestion	HNO3	Linear	yes	
	11	ICP-MS	yes	0,5 g	Microwave	HNO3 / H2O2	external	yes	-

Parameter	Teilnehmer	Methodenbeschreibung	Homogenisierung	Einwaage	Aufschluss: Methode	Aufschluss: Lösung	Kalibrierverfahren/Referenzmaterial	Methode akkreditiert nach ISO/IEC 17025	Sonstige Hinweise	
Analyte	Participant	Method description	Homogenisation	sample weight	Digestion: Method	Digestion: Solution	Calibration / reference material	Method accredited via ISO/IEC 17025	Further remarks	
Schwefel / Sulfur	1									
	2									
	3									
	4									
	5									
	6	ASU L 00.00 – 144	no	0,99g / 1,00g	ASU L 00.00 – 19/1			LGC7162	yes	
	7				Microwave					
	8	DIN EN ISO 11885		0,5 g	Microwave pressure digestion	HNO3, H2O2, H2O			yes	
	9	ICP-MS			Microwave					
	10	EN 15763	no	400	Microwave pressure digestion	HNO3	Linear		yes	
	11	ICP-OES	yes	0,5 g	Microwave	HNO3 / H2O2	external		yes	-



Parameter	Teilnehmer	Methodenbeschreibung	Homogenisierung	Einwaage	Aufschluss: Methode	Aufschluss: Lösung	Kalibrierverfahren/ Referenzmaterial	Methode akkreditiert nach ISO/IEC 17025	Sonstige Hinweise
Analyte	Participant	Method description	Homogenisation	sample weight	Digestion: Method	Digestion: Solution	Calibration / reference material	Method accredited via ISO/IEC 17025	Further remarks
Strontium	1								
	2								
	3								
	4								
	5			0.5 g	Microwave digestion	8 mL HNO <sub>3</sub> + 2 mL H <sub>2</sub> O <sub>2</sub>	External Calibration with internal Standard: Indium	no	
	6	internal Method	no	0,99g / 1,00g	ASU L 00.00 – 19/1		LGC7162	yes	
	7				Microwave				
	8	DIN EN ISO 11885		0,5 g	Microwave pressure digestion	HNO <sub>3</sub> , H <sub>2</sub> O <sub>2</sub> , H <sub>2</sub> O		no	
	9	ICP-MS	no	450 mg	Microwave	HNO <sub>3</sub> /H <sub>2</sub> O <sub>2</sub>	1;2;4 Calib. (0,0001-0,04 mg/l)	yes	
	10	EN 15763	no	400	Microwave pressure digestion	HNO <sub>3</sub>	Linear	yes	
	11	ICP-MS	yes	0,5 g	Microwave	HNO <sub>3</sub> / H <sub>2</sub> O <sub>2</sub>	external	yes	-

Parameter	Teilnehmer	Methodenbeschreibung	Homogenisierung	Einwaage	Aufschluss: Methode	Aufschluss: Lösung	Kalibrierverfahren/ Referenzmaterial	Methode akkreditiert nach ISO/IEC 17025	Sonstige Hinweise
Analyte	Participant	Method description	Homogenisation	sample weight	Digestion: Method	Digestion: Solution	Calibration / reference material	Method accredited via ISO/IEC 17025	Further remarks
Znk / Zinc	1	AOAC 993.14		*	Microwave	5% HNO <sub>3</sub>	NIST	yes	
	2	DIN EN ISO 11885		0,5 g	Microwave pressure digestion.	aqua regia	SPS-SW1	yes	
	3								
	4	ICP-OES	man. Mixing	0,5 g					
	5			0.5 g	Microwave digestion	8 mL HNO <sub>3</sub> + 2 mL H <sub>2</sub> O <sub>2</sub>	External Calibration with internal Standard: Germanium	no	
	6	ASU L 00.00 – 144	no	0,99g / 1,00g	ASU L 00.00 – 19/1		ERM-CE278k	yes	
	7	Zn with ICP-MS after Microwave digestion	mixing	0,2 g	Microwave	HNO <sub>3</sub>	ext. With IS In	yes	
	8	DIN EN ISO 11885		0,5 g	Microwave pressure digestion	HNO <sub>3</sub> , H <sub>2</sub> O <sub>2</sub> , H <sub>2</sub> O		yes	
	9	ICP-MS	no	450 mg	Microwave	HNO <sub>3</sub> /H <sub>2</sub> O <sub>2</sub>	1;2 Calib. (0,01-0,4 mg/l)	yes	
	10	EN 15763	no	400	Microwave pressure digestion	HNO <sub>3</sub>	Linear	yes	
	11	ICP-MS	yes	0,5 g	Microwave	HNO <sub>3</sub> / H <sub>2</sub> O <sub>2</sub>	external	yes	-

## 5.2 Homogeneity

### 5.2.1 Homogeneity of bottled PT-samples

Homogeneity test of copper by ICP-MS:

Independant Samples	mg/kg
1	1,93
2	1,94
3	2,01
4	2,05
5	1,88
6	1,92
7	1,95
8	1,89

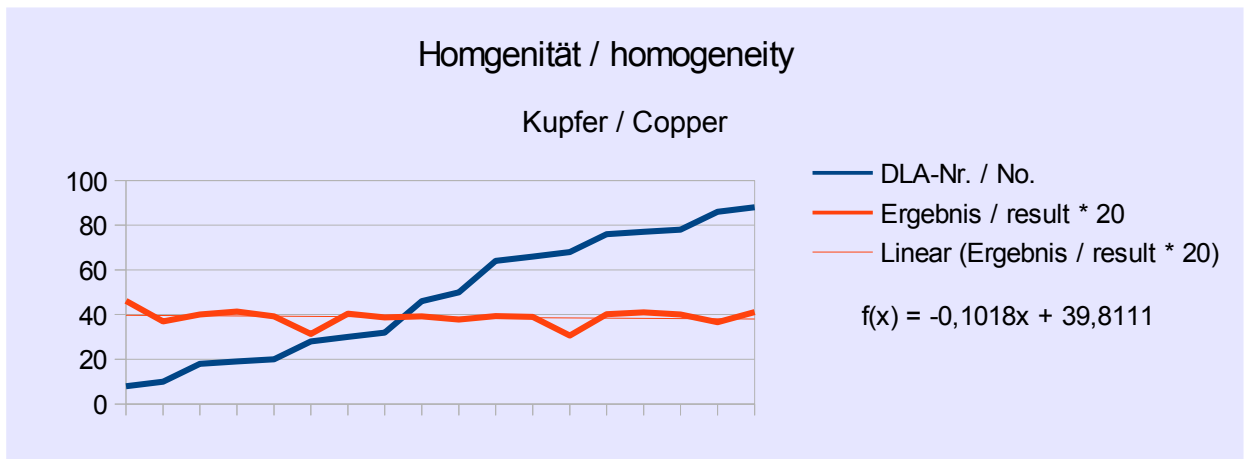
General Mean                                    1,95  
 Repeatability standard deviation    0,0578    2,97%

### 5.2.2 Comparison of sample numbers / test results and trend line

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

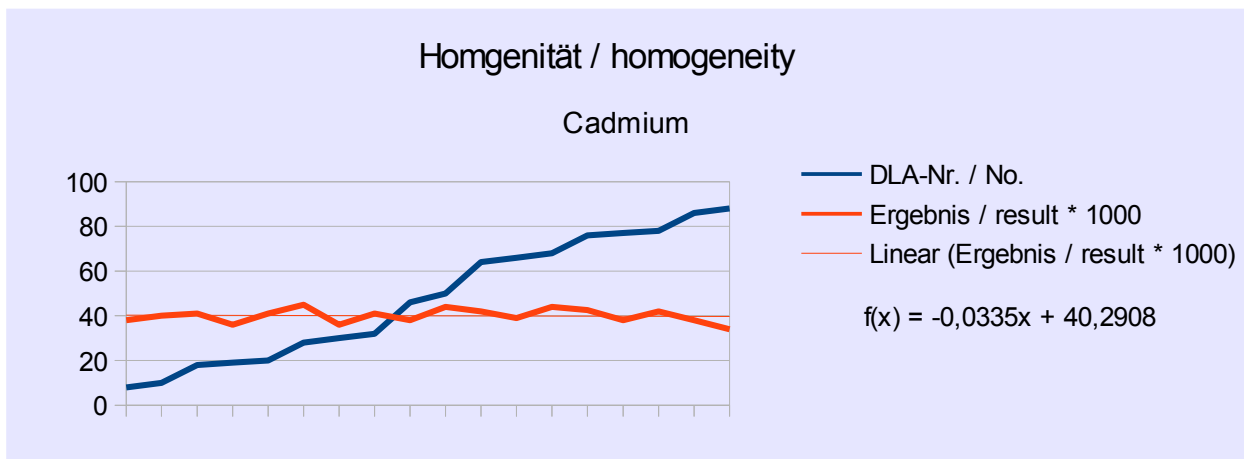
<b>Copper</b>	
Target standard deviation $\sigma_{pt}$	0,285                                    mg/kg
Sample numbers	8 - 88
Total numbers of samples	18
Slope	-0,005090
Trend line range	1,99                                    -                                    1,90                                    mg/kg
Deviation trend line	1,94 $\pm$ 0,0458                                    mg/kg
<b>Percent of <math>\sigma_{pt}</math></b>	16,1                                    %

<b>Cadmium</b>	
Target standard deviation $\sigma_{pt}$	0,0104                                    mg/kg
Sample numbers	8 - 88
Total numbers of samples	18
Slope	-0,00003350
Trend line range	0,0403                                    -                                    0,0397                                    mg/kg
Deviation trend line	0,0400 $\pm$ 0,000302                                    mg/kg
<b>Percent of <math>\sigma_{pt}</math></b>	2,90                                    %



**Abb./Fig. 38:**

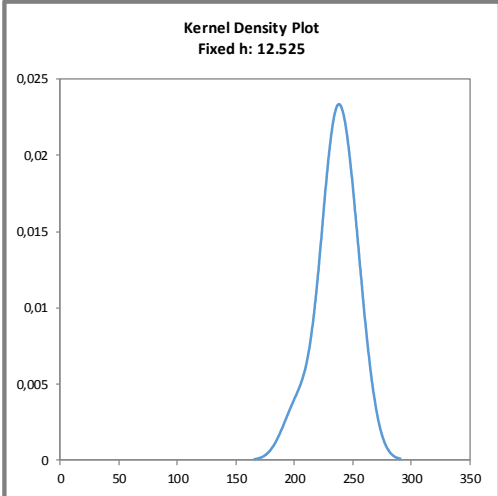
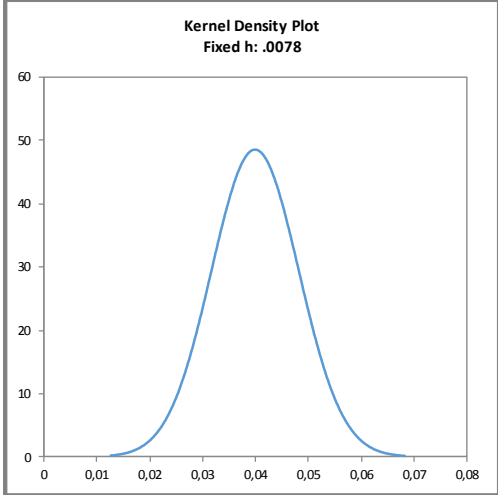
Trendfunktion Probennummern vs. Ergebnisse (1\*20 dargestellt)  
trend line function sample number vs. results (1\*20 shown)

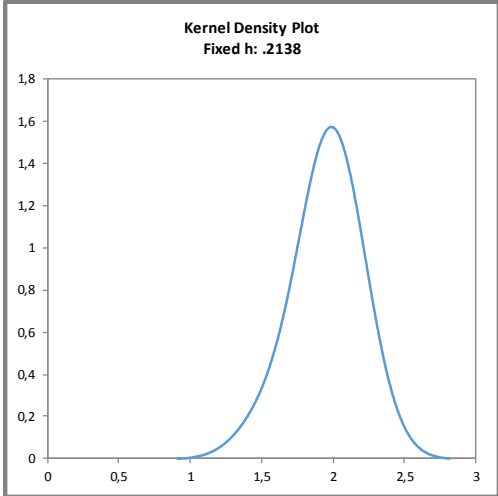
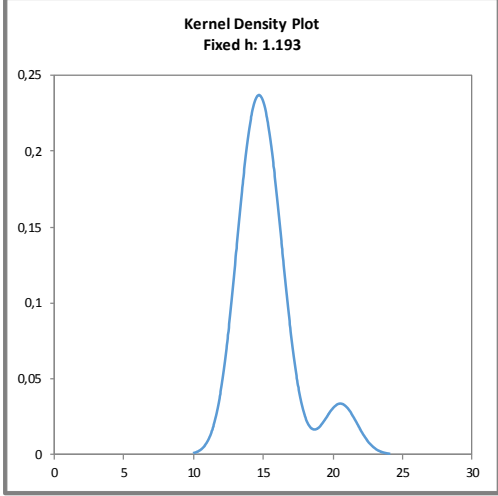
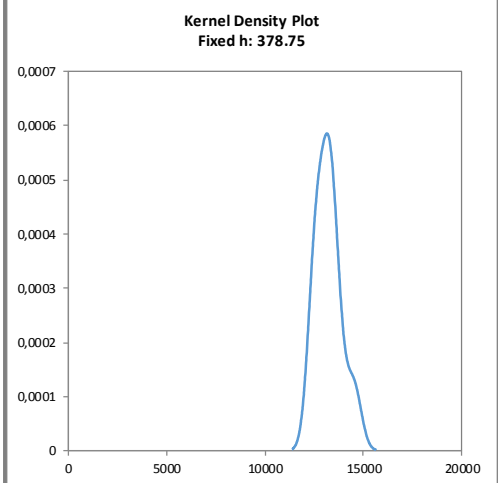


**Abb./Fig. 39:**

Trendfunktion Probennummern vs. Ergebnisse (1\*1000 dargestellt)  
trend line function sample number vs. results (1\*1000 shown)

**5.3 Kernel Density Plots of Results**

<p><b>Abbildungen:</b> Kerndichte-Schätzungen der Teilnehmerergebnisse (mit <math>h = 0,75 \times \sigma_{pt}</math> von <math>X_{pt}</math>)</p> <p><b>Figures:</b> Kernel density plots of participants' results (with <math>h = 0,75 \times \sigma_{pt}</math> of <math>X_{pt}</math>)</p>	<p>Barium</p> <p>&lt; 8 Ergebnisse / &lt; 8 Results</p>
<p>Aluminium</p> <p>&lt; 8 Ergebnisse / &lt; 8 Results</p>	<p>Calcium</p> 
<p>Bor / Boron</p> <p>&lt; 8 Ergebnisse / &lt; 8 Results</p>	<p>Cadmium</p> 

<p><b>Abbildungen:</b>                  Kerndichte-Schätzungen                  der Teilnehmerergebnisse                  (mit <math>h = 0,75 \times \sigma_{pt}</math> von <math>X_{pt}</math>)</p> <p><b>Figures:</b>                  Kernel density plots                  of participants' results                  (with <math>h = 0,75 \times \sigma_{pt}</math> of <math>X_{pt}</math>)</p>	<p style="text-align: center;">Kupfer / Copper</p> 
<p style="text-align: center;">Cobalt</p> <p>&lt; 8 Ergebnisse /                  &lt; 8 Results</p>	<p style="text-align: center;">Eisen / Iron</p> 
<p style="text-align: center;">Chrom / Chromium</p> <p>&lt; 8 Ergebnisse /                  &lt; 8 Results</p>	<p style="text-align: center;">Kalium / Potassium</p> 

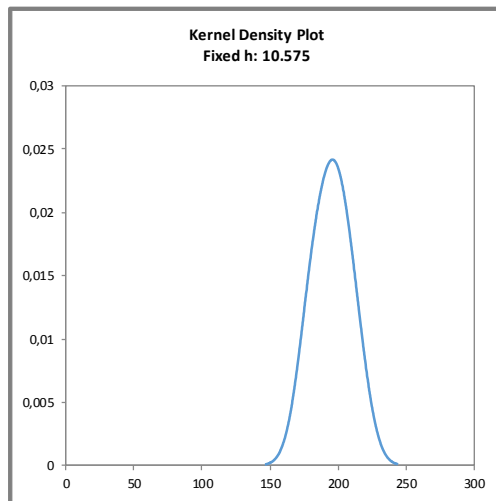
**Abbildungen:**

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

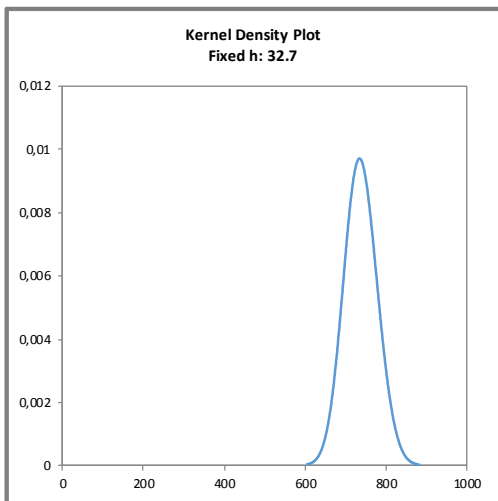
**Figures:**

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

Natrium / Sodium



Magnesium



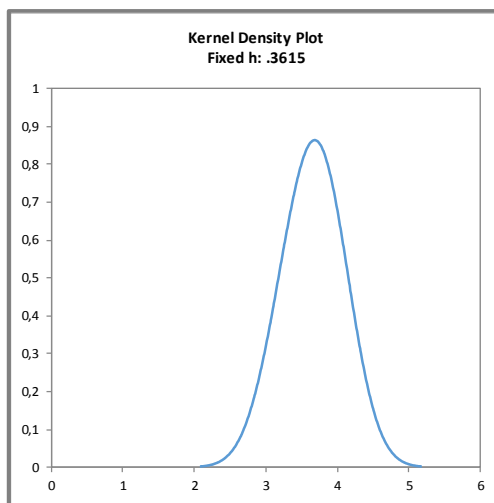
Nickel

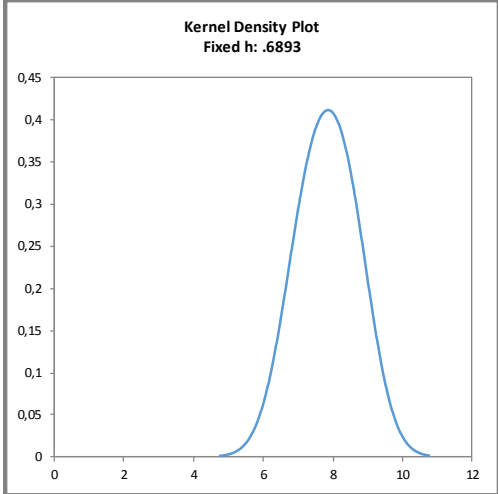
< 8 Ergebnisse /  
< 8 Results

Molybdän / Molybdenum

< 8 Ergebnisse /  
< 8 Results

Mangan / Manganese



<p><b>Abbildungen:</b> Kerndichte-Schätzungen der Teilnehmerergebnisse (mit <math>h = 0,75 \times \sigma_{pt}</math> von <math>X_{pt}</math>)</p> <p><b>Figures:</b> Kernel density plots of participants' results (with <math>h = 0,75 \times \sigma_{pt}</math> of <math>X_{pt}</math>)</p>	<p>Schwefel / Sulfur</p> <p>&lt; 8 Ergebnisse / &lt; 8 Results</p>
<p>Phosphor / Phosphorus</p> <p>&lt; 8 Ergebnisse / &lt; 8 Results</p>	<p>Strontium</p> <p>&lt; 8 Ergebnisse / &lt; 8 Results</p>
<p>Rubidium</p> <p>&lt; 8 Ergebnisse / &lt; 8 Results</p>	<p>Zink / Zinc</p> 



**5.4 Information on the Proficiency Test (PT)**

Before the PT the participants received the following information in the sample cover letter:

<i>PT number</i>	<b>DLA 46-2017</b>
<i>PT name</i>	<b>Heavy Metals and Trace Elements in plant food, approx. 20 Elements</b>
<i>Sample matrix*</i>	<b>Samples A + B: potato powder, ingredients: potatoes, mono- and diglycerides of fatty acids, fatty acid esters of ascorbic acid, sodium sulfite, dye: curcumin</b>
<i>Number of samples and sample amount</i>	2 identical samples a + B, 8 g each.
<i>Storage</i>	Samples A + B: room temperature
<i>Intentional use</i>	Laboratory use only (quality control samples)
<i>Parameter</i>	quantitative: <b>Al, B, Ba, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Mo, Na, Ni, P, Rb, S, Sr and Zn</b>
<i>Methods of analysis</i>	Analytical methods are optional
<i>Notes to analysis</i>	The analysis of PT samples should be performed like a routine laboratory analysis. In general we recommend to homogenize a representative sample amount before analysis according to good laboratory practice, especially in case of low sample weights.
<i>Result sheet</i>	The results for sample A and B as well as the final results calculated as mean of the double determination (samples A and B) should be filled in the result submission file. The recovery rates, if carried out, has to be included in the calculation.
<i>Units</i>	mg/kg
<i>Number of significant digits</i>	at least 2
<i>Further information</i>	For information please specify: <ul style="list-style-type: none"> <li>- Date of analysis</li> <li>- DLA-sample-numbers (for sample A and B)</li> <li>- Limit of detection</li> <li>- Assignment incl. Recovery</li> <li>- Recovery with the same matrix</li> <li>- Information on determination and hydrolization methods</li> <li>- Method is accredited</li> </ul>
<i>Result submission</i>	The result submission file should be sent by e-mail to: <b>pt@dla-lvu.de</b>
<i>Deadline</i>	<b>the latest May 26<sup>th</sup> 2017</b>
<i>Evaluation report</i>	The evaluation report is expected to be completed 6 weeks after deadline of result submission and sent as PDF file by e-mail.
<i>Coordinator and contact person of PT</i>	Dr. Matthias Besler

\* Control of mixture homogeneity and qualitative testings are carried out by DLA. Testing of the content, homogeneity and stability of PT parameters is subcontracted by DLA.

**6. Index of participant laboratories in alphabetical order**

Teilnehmer / Participant	Ort / Town	Land / Country
		Germany
		ITALY
		Germany
		USA
		Germany
		Germany
		Germany
		Germany
		Germany
		Germany
		Germany

*[Die Adressdaten der Teilnehmer wurden für die allgemeine Veröffentlichung des Auswertebereichs nicht angegeben.]*

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

## 7. Index of references

1. DIN EN ISO/IEC 17025:2005; Allgemeine Anforderungen an die Kompetenz von Prüf- und Kalibrierlaboratorien / General requirements for the competence of testing and calibration laboratories
2. DIN EN ISO/IEC 17043:2010; Konformitätsbewertung - Allgemeine Anforderungen an Eignungsprüfungen / Conformity assessment - General requirements for proficiency testing
3. ISO 13528:2015 & DIN ISO 13528:2009; Statistische Verfahren für Eignungsprüfungen durch Ringversuche / Statistical methods for use in proficiency testing by interlaboratory comparisons
4. ASU §64 LFGB: Planung und statistische Auswertung von Ringversuchen zur Methodenvalidierung / DIN ISO 5725 series part 1, 2 and 6 Accuracy (trueness and precision) of measurement methods and results
5. Verordnung / Regulation 882/2004/EU; Verordnung über über amtliche Kontrollen zur Überprüfung der Einhaltung des Lebensmittel- und Futtermittelrechts sowie der Bestimmungen über Tiergesundheit und Tierschutz / Regulation on official controls performed to ensure the verification of compliance with feed and food law, animal health and animal welfare rules
6. Evaluation of analytical methods used for regulation of food and drugs; W. Horwitz; Analytical Chemistry, 54, 67-76 (1982)
7. The International Harmonised Protocol for the Proficiency Testing of Analytical Laboratories ; J.AOAC Int., 76(4), 926 - 940 (1993)
8. A Horwitz-like funktion describes precision in proficiency test; M. Thompson, P.J. Lowthian; Analyst, 120, 271-272 (1995)
9. Protocol for the design, conduct and interpretation of method performance studies; W. Horwitz; Pure & Applied Chemistry, 67, 331-343 (1995)
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12. AMC Kernel Density - Representing data distributions with kernel density estimates, amc technical brief, Editor M Thompson, Analytical Methods Committee, AMCTB No 4, Revised March 2006 and Excel Add-in Kernel.xla 1.0e by Royal Society of Chemistry
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14. GMP+ Feed Certification scheme, Module: Feed Safety Assurance, chapter 5.7 Checking procedure for the process accuracy of compound feed with micro tracers in GMP+ BA2 Control of residues, Version: 1st of January 2015 GMP+ International B.V.
15. MTSE SOP No. 010.01 (2014): Quantitative measurement of mixing uniformity and carry-over in powder mixtures with the rotary detector technique, MTSE Micro Tracers Services Europe GmbH
16. ASU §64 L 00.00-157 (2016-2): Bestimmung von Aluminium in Lebensmitteln mit der Massenspektrometrie mit induktiv gekoppeltem Plasma (ICP-MS)
17. ASU §64 L 00.00-158 (2016-2): Bestimmung von Aluminium in Lebensmitteln mit der optischen Emmissionsspektrometrie mit induktiv gekoppeltem Plasma (ICP-OES)
18. ASU §64 L 00.00-135 (2011-01) / DIN EN 15763:2010: Bestimmung von Arsen, Cadmium, Quecksilber und Blei in Lebensmitteln mit ICP-MS nach Druckaufschluss / Foodstuffs. Determination of trace elements. Determination of arsenic, cadmium, mercury and lead in foodstuffs by inductively coupled plasma mass spectrometry (ICPMS) after pressure digestion
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. Determina-

- tion of lead, cadmium, chromium and molybdenum by graphite furnace atomic absorption spectrometry (GFAAS) after pressure digestion
21. ASU §64 L 00.00-19/5: Bestimmung von Selen mit der Atomabsorptionsspektrometrie (AAS) -Hydridtechnik
  22. ASU §64 L 00.00-144 : Bestimmung der Mineralstoffe Ca, K, Mg, Na, P und S sowie der Spurenelemente Fe, Cu, Mn und Zn in Lebensmitteln mit ICP-OES
  23. ASU §64 L 00.00-93 / DIN EN 15111: Bestimmung von Iod in Lebensmitteln - ICP-MS-Verfahren / Foodstuffs. Determination of trace elements. Determination of iodine by ICP-MS (inductively coupled plasma mass spectrometry)
  24. ASU §64 L 00.00-127 / EN 15764: Bestimmung von Zinn in Lebensmitteln mit der Flammen- und Graphitrohr-Atomabsorptionsspektrometrie (GFAAS) nach Druckaufschluss / Foodstuffs. Determination of trace elements. Determination of tin by flame and graphite furnace atomic absorption spectrometry (FAAS and GFAAS) after pressure digestion
  25. ASU §64 L 00.00-128 / DIN EN 15765: Bestimmung Zinn in Lebensmitteln mit der Massenspektrometrie mit induktiv gekoppeltem Plasma (ICP-MS) nach Druckaufschluss / Foodstuffs. Determination of trace elements. Determination of tin by inductively coupled plasma mass spectrometry (ICPMS) after pressure digestion
  26. ASU §64 L 31.00-10: Bestimmung der Gehalte an Natrium, Kalium, Calcium und Magnesium in Frucht- und Gemüsesäften - Atomabsorptionsspektrometrisches Verfahren (AAS) [Determination of sodium, potassium, calcium and magnesium in fruit and vegetable juices - atomic absorption spectrometry (AAS)]