Evaluation Report
proficiency test

DLA 49/2019

Heavy Metals and Trace Elements:

in Plant Food

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Allgemeine Informationen zur Eignungsprüfung (EP)
General Information on the proficiency test (PT)

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Unteraufträge *Subcontractors*	Falls im Rahmen der Eignungsprüfung eine Prüfung der Gehalte, Homogenität und Stabilität von EP-Parametern durchgeführt wurde, hat DLA diese im Unterauftrag vergeben. In case the analysis of the content, homogeneity and stability of PT-parameters was part of the proficiency test, the determinations were subcontracted by DLA.
Vertraulichkeit *Confidentiality*	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.

Inhalt / Content

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 moringa leaf powder from India. With the exception of the elements uranium and tin, which were added to the material, the contents of the parameters are of natural origin.

After homogenization of the raw material moringa leaf powder the spiking was done by adding standard solutions of the two above mentioned elements to an aliquot of the basic mixture followed by homogenization. Subsequently, moringa leaf powder was added again in 3 further steps and homogenized until the total amount was reached. Before the last homogenization the whole material was sieved (mesh <600µm).

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**
Organic Moringa Powder (Origin: India) Ingredients: Moringa leafs, dried	100 g/100 g
Sn - Tin Standard solution: Tin(IV)-Chloride in HCl	1,5 mg/kg
U - Uranium Standard solution: Uranium(VI)-Nitrate in HNO_3	0,30 mg/kg

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 and uranium by LC/MS (DIN EN ISO 17294-2). The repeatability standard deviation was 0,97% and 3,8% 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 0,8% – 2,9%. 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) [18-28]. 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 graphically characterized for information by the **trend line function of participants' results for chronological bottled single samples** (s. 5.2.1).

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

A water activity (a_W) of < 0,5 is an important factor to ensure the stability of dry or dried products during storage. Optimum conditions for storage is the a_W value range of 0,15 - 0,3. In this range the lowest possible degradation rate is to be expected [16].

The experience with various DLA test 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 a_W value of the EP samples was approx. 0,23 (21,9°C). The stability of the sample material was thus ensured during the investigation period under the specified storage conditions.

2.2 Sample shipment and information to the test

Two portions of test material were sent to every participating laboratory in the 15th week of 2019. The testing method was optional. The tests should be finished at 24th May 2019 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 moringa leaf powder with a mostly natural content of 20 elements to be determined quantitatively.

Note: Please indicate the applied digestion 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 digestion 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]. If there are < 12 quantitative results and an increased difference between robust mean and median, the median may be used as the assigned value (criterion: Δ median - rob. mean > 0,3 σ_{pt}) [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 justified cases, an evaluation may also be carried out from 5 results onwards.

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

3.2 Robust standard deviation

For comparison to the target standard deviation σ_{pt} (standard deviation for proficiency assessment) a robust standard deviation (S^x) 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 CV_R in percent of the mean is given as variation coefficient in the statistical data of participant for each parameter. The significance of CV_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, too few significant digits (valid digits) or results for another proficiency test item can be removed from the data set [2]. Even if a result e.g. with a factor >10 deviates significantly from the mean and has an influence on the robust statistics, a result of the statistical evaluation can be excluded [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 tested for outliers by the use of robust statistics (algorithm A): If a value deviates from the robust mean by more than 3 times the robust standard deviation, it can be classified as an outlier (see above) [3]. Due to the use of robust statistics outliers are not excluded, provided that no other reasons are present [3]. Detected outliers are only mentioned in the results section, if they have been excluded from the statistical evaluation.

3.6 Target standard deviation (for proficiency assessment)

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

If an acceptable quotient S^*/σ_{pt} is present, the target standard deviation of the general model by Horwitz is preferably used for the proficiency assessment. It is usually suitable for evaluation of interlaboratory studies, where different 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.

For valuation of all elements except Ca and P in the present PT the target standard deviation according to the general model of Horwitz was applied (see 3.6.1). For Ca and P the target standard deviation was calculated using data from a precision experiment (s. 3.6.2, ASU §64 method L 00.00-144).

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

Due to the number of < 5 the results for I adn Rb 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 σ_R [6]. Later the model was modified by Thompson for certain concentration ranges [10]. The reproducibility standard deviation σ_R can be applied as the relative target standard deviation σ_{pt} in % of the assigned values and calculated according to the following equations [3]. For this the assigned value X_{pt} is used for the concentration c.

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

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

3.6.2 Value by precision experiment

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

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

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 σ_{pt}, which were identified there, were used to evaluate the results and to provide additional information for the statistical data.

Tabelle 2: 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 σ_{pt} [21, 25-28]

Para-meter	**Matrix**	**Mean** [mg/kg]	**RSD_r**	**RSD_R**	**σ_{pt}**	**Method / Literature**
Al	Cocoa powder	205	3,25%	5,83%	5,36%[1]	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%[1]	ICP-OES [22]
Cd	Fish homogenate	0,87	7,3%	11%	9,71%[1]	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%[1]	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%[1]	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%[1]	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%[1]	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%[1]	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%[1]	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%[1]	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%[1]	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%[1]	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)

[1] *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.

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 (σ_{pt}) the result (x_i) of the participant is deviating from the assigned value (X_{pt}) [3].
Participants' z-scores are derived from:

$$z_i = \frac{\left(x_i - x_{pt}\right)}{\sigma_{pt}}$$

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 (σ_{pt}).

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.

An error or cause analysis can be carried out by checking the analysis process including understanding and implementation of the measurement by the staff, details of the measurement procedure, calibration of equipment and composition of reagents, transmission error or an error in the calculation, in the trueness and precision and use of reference material. If necessary, the problems must be addressed through appropriate corrective action [3].

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

Table 3: Characteristics of the present PT (on grey) in comparison to previous PTs since 2017 (SD = standard deviation, CV = coefficient of variation)

Para-meter	**Matrix (Powder)**	**robust Mean** [mg/kg]	**rob. SD (S*)** [mg/kg]	**rel. SD (VK_{S*}) [%]**	**Quotient S^*/σ_{pt}**	**DLA-report**
Al	Potatoes	0,527	0,283	53,7%	1,5[1]	DLA 46/2017
Al	Food Supplement	5,86	2,08	35,5%	1,7[1]	DLA 49/2018
Al	Moringa leafs	44,3	5,33	12,0%	1,3	DLA 49/2019
B	Potatoes	3,88	0,689	17,8%	1,4	DLA 46/2017
B	Moringa leafs	31,5	2,89	9,19%	0,97	DLA 49/2019
Ba	Potatoes	0,220	-	-	-	DLA 46/2017
Ba	Food Supplement	0,708	0,0791	11,2%	0,66	DLA 49/2018
Ba	Moringa leafs	71,3	2,00	2,80%	0,33	DLA 49/2019
Ca	Potatoes	238	12,0	5,04%	0,72	DLA 46/2017
Ca	Food Supplement	6540	259	3,97%	0,93	DLA 49/2018
Ca	Moringa leafs	19588	1727	8,81%	1,2	DLA 49/2019
Cu	Potatoes	1,98	0,117	5,90%	0,41	DLA 46/2017
Cu	Food Supplement	4,28	0,611	14,3%	1,1	DLA 49/2018
Cu	Moringa leafs	4,86	0,291	5,98%	0,47	DLA 49/2019
Fe	Potatoes	15,0	1,22	8,10%	0,76	DLA 46/2017
Fe	Food Supplement	60,3	4,41	7,31%	0,85	DLA 49/2018
Fe	Moringa leafs	108	6,65	6,14%	0,78	DLA 49/2019
K	Potatoes	13162	604	4,59%	1,2	DLA 46/2017
K	Food Supplement	3931	347	8,83%	1,9	DLA 49/2018
K	Moringa leafs	12420	1223	9,85%	1,7[1]	DLA 49/2019
Mg	Potatoes	736	27,1	3,68%	0,62	DLA 46/2017
Mg	Food Supplement	1149	36,3	3,16%	0,57	DLA 49/2018
Mg	Moringa leafs	4704	261	5,56%	1,2	DLA 49/2019
Mn	Potatoes	3,66	0,327	8,9%	0,68	DLA 46/2017
Mn	Food Supplement	3,58	0,326	9,10%	0,69	DLA 49/2018
Mn	Moringa leafs	61,9	4,93	7,96%	0,93	DLA 49/2019
Mo	Potatoes	0,197	0,0161	8,2%	0,40	DLA 46/2017
Mo	Food Supplement	0,830	0,130	15,7%	0,95	DLA 49/2018
Mo	Moringa leafs	0,449	0,0385	8,59%	0,48	DLA 49/2019

Continuation next page

Continuation Tab. 3:

Para-meter	**Matrix (Powder)**	**robust Mean** [mg/kg]	**rob. SD (S*)** [mg/kg]	**rel. SD (VK_{S*}) [%]**	**Quotient S^*/σ_{pt}**	**DLA-report**
Na	Potatoes	195	13,7	7,03%	1,0	DLA 46/2017
Na	Food Supplement	2944	154	5,23%	1,1	DLA 49/2018
Na	Moringa leafs	2478	92,0	4,08%	0,75	DLA 49/2019
Ni	Potatoes	0,0398	0,00645	16,2%	0,62	DLA 46/2017
Ni	Food Supplement	0,418	0,0590	14,1%	0,77	DLA 49/2018
Ni	Moringa leafs	0,709	0,122	17,2%	1,0	DLA 49/2019
P	Potatoes	1451	49,1	3,38%	0,63	DLA 46/2017
P	Food Supplement	4870	386	7,92%	1,8	DLA 49/2018
P	Moringa leafs	2310	167	7,24%	1,0	DLA 49/2019
Pb	Moringa leafs	0,258	0,0288	11,2%	0,57	DLA 49/2019
Se	Food Supplement	0,219	0,0461	21,1%	1,0	DLA 49/2018
Se	Moringa leafs	0,578	0,0961	16,6%	0,96	DLA 49/2019
Sn	Moringa leafs	1,52	0,0731	4,80%	0,32	DLA 49/2019
U	Moringa leafs	0,300	0,0310	10,3%	0,54	DLA 49/2019
Zn	Potatoes	7,83	0,726	9,3%	0,79	DLA 46/2017
Zn	Food Supplement	109	15,2	14,0%	1,8	DLA 49/2018
Zn	Moringa leafs	14,5	2,19	15,2%	1,4	DLA 49/2019

[1] *with target standard deviation σ_{pt}'*

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. 3.11). 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 (σ_{pt}) and the standard uncertainty (Ux_{pt}) [3].

The calculation is performed by:

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

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

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

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

For warning and action signals see 3.7.1.

3.9 Reproducibility cofficient of variation (CV_R)

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*/σ_{pt}

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

3.11 Standard uncertainty 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 ($U_{(Xpt)}$) for this PT is calculated as follows [3]:

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

If $U_{(Xpt)} \leq 0{,}3\ \sigma_{pt}$ 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 traceability of the assigned value is ensured on the basis of the consensus value as a robust mean of the participant results.

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

For P, Sn and U there were < 7 results, thus the significance of the statistical evaluation could be limited due to the low number of results. However, the statistical characteristics allow an evaluation of the results.

The target standard deviation was calculated with the exception of Ca and P for all other parameters according to the model of Horwitz. For Ca and P the target standard deviation calculated from statistical data obtained from precision experiments (ASU §64 method) was used.

For information the target standard deviation using statistical data obtained from precision experiments (ASU §64 method) was additionally given, when available. For Ca and P the standard deviation according to the model of Horwitz was given for information.

For K the distribution of results showed an increased variability. The quotient S^*/σ_{pt} was clearly > 2,0. Thus the parameter was evaluated considering the standard uncertainty by z'-scores. The quotient S^*/σ_{pt}' was then < 2,0 (s. Tab. 3).

For the other parameters the distribution of results showed a low to normal variability. The quotients S^*/σ_{pt} were all in the range of 0,3 to 1,4 (s. Tab. 3).

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.

78% 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:

Statistic Data
Number of results
Number of outliers
Mean
Median
Robust mean(X_{pt})
Robust standard deviation (S^*)
Number with m replicate measurements
Repeatability standard deviation (S_r)
Coefficient of Variation (CV_r)in %
Reproducibility standard deviation (S_R)
Coefficient of Variation (CV_R)in %
Target range:
Target standard deviation σ_{pt} or σ_{pt}'
Target standard deviation for information
lower limit of target range ($X_{pt} - 2\sigma_{pt}$) or ($X_{pt} - 2\sigma_{pt}$') *
upper limit of target range ($X_{pt} + 2\sigma_{pt}$) or ($X_{pt} + 2\sigma_{pt}$´) *
Quotient S/σ_{pt} or S*/σ_{pt}'*
Standard uncertainty U(X_{pt})
Number of results in the target range
Percent in the target range

* 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**:

Auswerte-nummer / **Evaluation number**	**Parameter [Einheit / Unit]**	**Abweichung** / **Deviation**	**z-Score σpt**	**z-Score** (Info)	**Hinweis** / **Remark**

*** 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

Statistic Data	
Number of results	9
Number of outliers	-
Mean	43,4
Median	46,0
Robust Mean (X)	**44,3**
Robust standard deviation (S*)	**5,33**
Number with 2 replicates	8
Repeatability SD (S_r)	1,32
Repeatability (CV_r)	2,91%
Reproducibility SD (S_R)	3,90
Reproducibility (CV_R)	8,63%
Target range:	
Target standard deviation σ_{pt}	**4,01**
Target standard deviation (for Information)	2,37
lower limit of target range	**36,3**
upper limit of target range	**52,3**
Quotient S/σ_{pt}*	*1,3*
Standard uncertainty $U_{(Xpt)}$	*2,22*
Results in the target range	*8*
Percent in the target range	*89%*

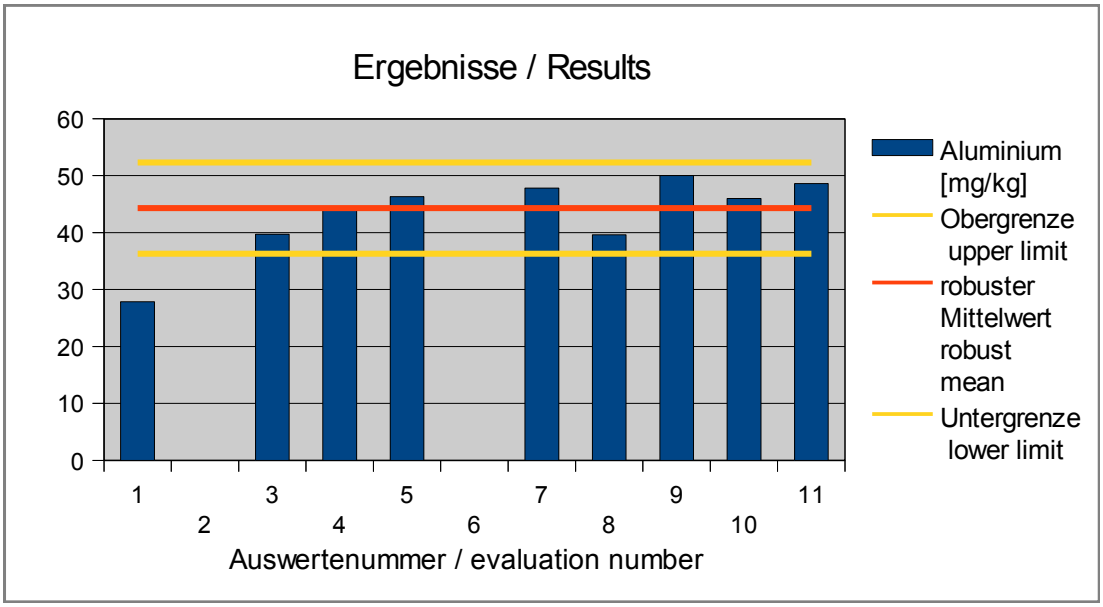


Abb. / Fig. 1: Ergebnisse Aluminium / Results Aluminium

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Aluminium [mg/kg]	Abweichung [mg/kg] Deviation [mg/kg]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	27,8	-16,47	-4,1	-6,9	
2					
3	39,7	-4,60	-1,1	-1,9	
4	44,5	0,17	0,04	0,07	
5	46,3	1,99	0,50	0,84	
6					
7	47,8	3,49	0,87	1,5	
8	39,6	-4,68	-1,2	-2,0	
9	50,0	5,69	1,4	2,4	
10	46,0	1,67	0,42	0,70	
11	48,6	4,29	1,1	1,8	

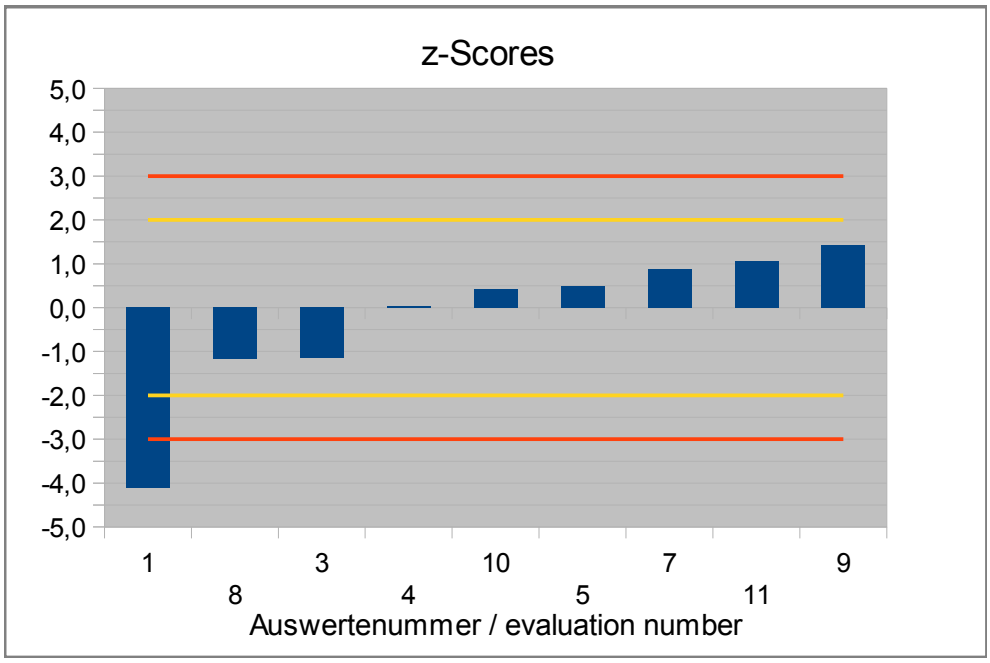


Abb. / Fig. 2: z-Scores Aluminium

4.2 B - Boron in mg/kg

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	7
Number of outliers	0
Mean	31,4
Median	31,0
Robust Mean (X)	**31,5**
Robust standard deviation (S*)	**2,89**
Number with 2 replicates	7
Repeatability SD (S_r)	0,426
Repeatability (CV_r)	1,36%
Reproducibility SD (S_R)	2,69
Reproducibility (CV_R)	8,54%
Target range:	
Target standard deviation σ_{pt}	**3,00**
lower limit of target range	**25,5**
upper limit of target range	**37,5**
Quotient S/σ_{pt}*	*0,97*
Standard uncertainty $U_{(Xpt)}$	*1,37*
Results in the target range	7
Percent in the target range	*100%*

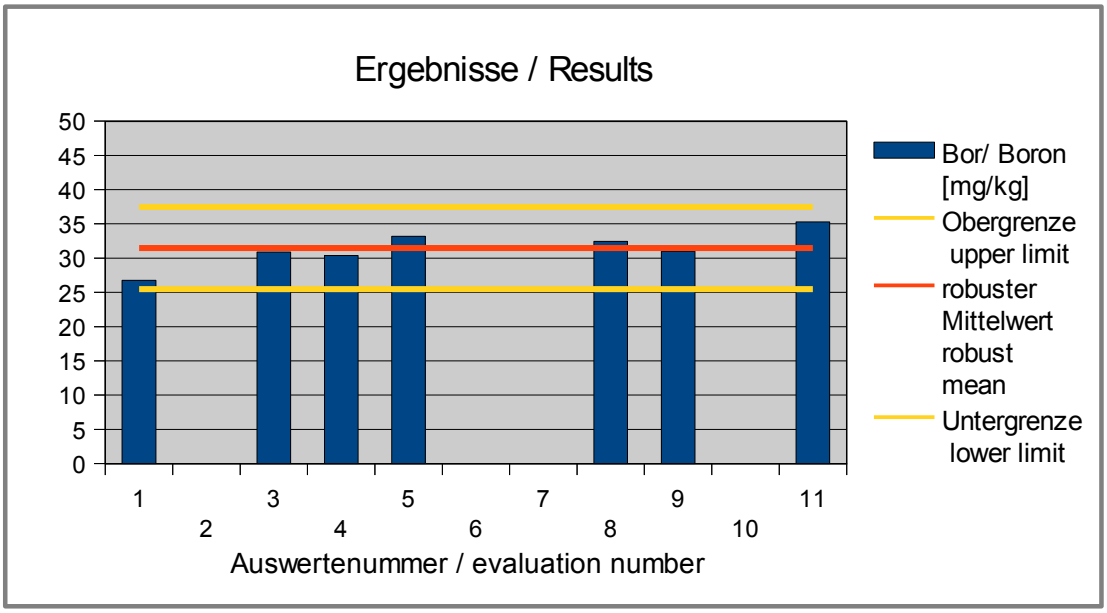


Abb. / Fig. 3: Ergebnisse Bor / Results Boron

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Bor/ Boron [mg/kg]	Abweichung [mg/kg] Deviation [mg/kg]	z-Score (σ_{pt})	Hinweis Remark
1	26,8	-4,70	-1,6	
2				
3	30,9	-0,57	-0,19	
4	30,4	-1,10	-0,37	
5	33,2	1,72	0,57	
6				
7				
8	32,5	0,97	0,32	
9	31,0	-0,48	-0,16	
10				
11	35,3	3,82	1,3	

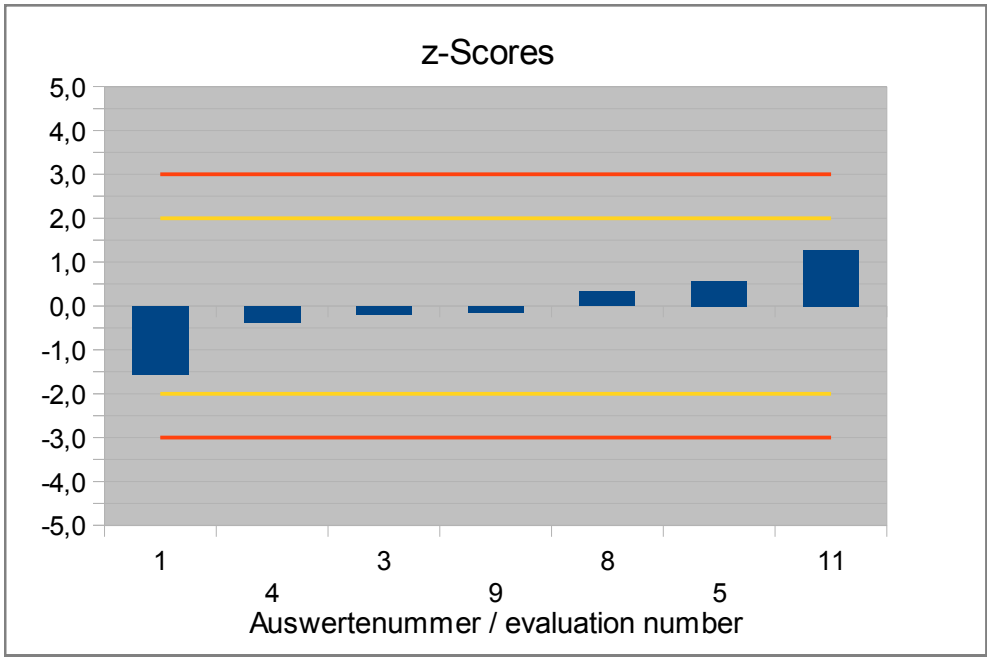


Abb. / Fig. 4: z-Scores Bor / Boron

4.3 Ba - Barium in mg/kg

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	8
Number of outliers	0
Mean	71,2
Median	71,9
Robust Mean (X)	**71,3**
Robust standard deviation (S*)	**2,00**
Number with 2 replicates	8
Repeatability SD (S_r)	0,564
Repeatability (CV_r)	0,793%
Reproducibility SD (S_R)	2,16
Reproducibility (CV_R)	3,03%
Target range:	
Target standard deviation σ_{pt}	**6,00**
lower limit of target range	**59,3**
upper limit of target range	**83,3**
Quotient S/σ_{pt}*	*0,33*
Standard uncertainty $U_{(Xpt)}$	*0,884*
Results in the target range	*8*
Percent in the target range	*100%*

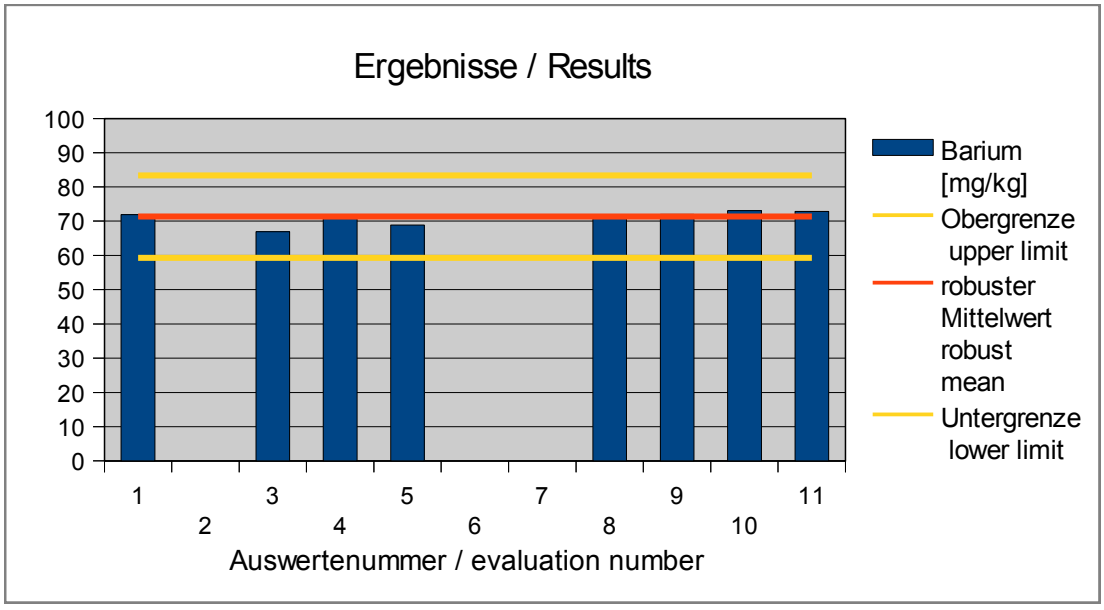


Abb. / Fig. 5: Ergebnisse Barium / Results Barium

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Barium [mg/kg]	Abweichung [mg/kg] Deviation [mg/kg]	z-Score (σ_{pt})	Hinweis Remark
1	72,0	0,61	0,10	
2				
3	66,9	-4,40	-0,73	
4	71,9	0,59	0,10	
5	68,9	-2,49	-0,41	
6				
7				
8	71,7	0,38	0,06	
9	72,0	0,66	0,11	
10	73,1	1,77	0,30	
11	72,8	1,46	0,24	

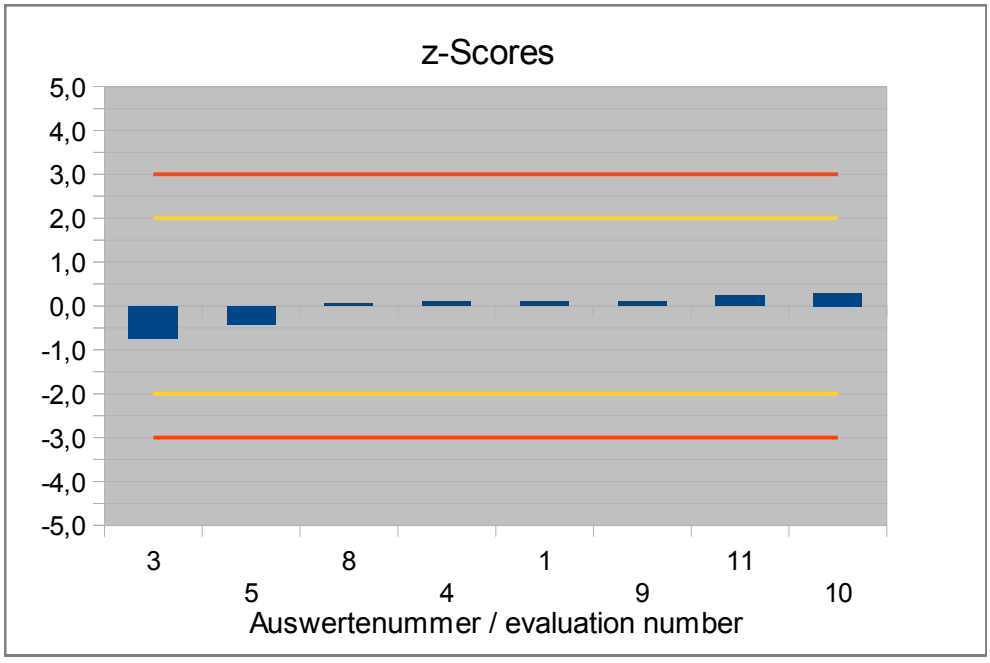


Abb. / Fig. 6: z-Scores Barium

4.4 Ca – Calcium in mg/kg

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	9
Number of outliers	-
Mean	20413
Median	19448
Robust Mean (X)	**19588**
Robust standard deviation (S*)	**1727**
Number with 2 replicates	8
Repeatability SD (S_r)	212
Repeatability (CV_r)	1,10%
Reproducibility SD (S_R)	1263
Reproducibility (CV_R)	6,56%
Target range:	
Target standard deviation σ_{pt}	**1488**
Target standard deviation (for Information)	708
lower limit of target range	**16612**
upper limit of target range	**22564**
Quotient S/σ_{pt}*	*1,2*
Standard uncertainty $U_{(Xpt)}$	*719*
Results in the target range	*8*
Percent in the target range	*89%*

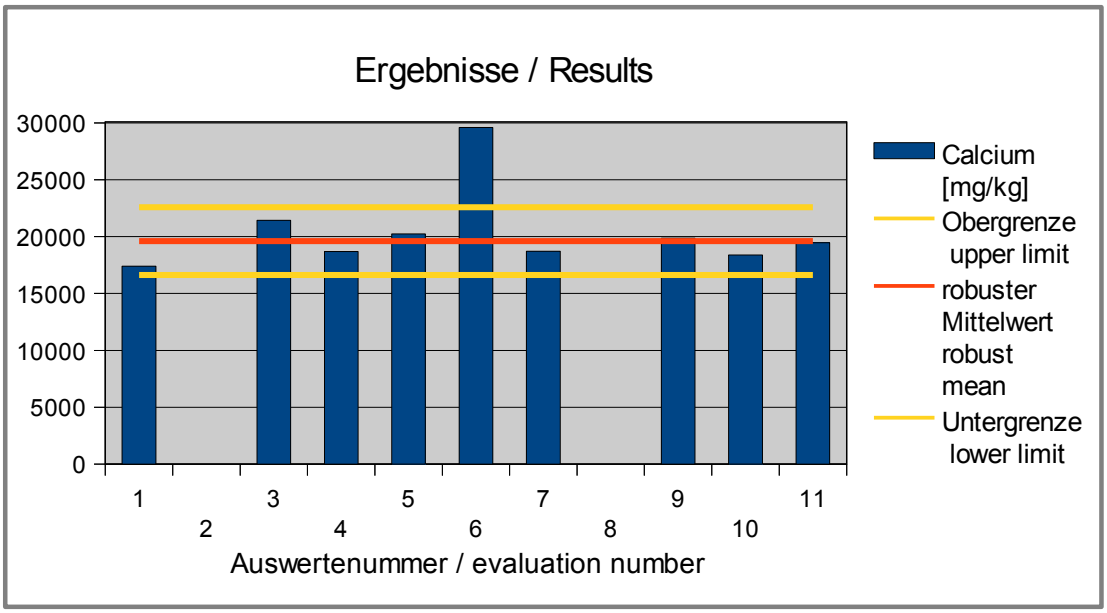


Abb. / Fig. 7: Ergebnisse Calcium / Results Calcium

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Calcium [mg/kg]	Abweichung [mg/kg] Deviation [mg/kg]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	17394	-2194	-1,5	-3,1	
2					
3	21440	1852	1,2	2,6	
4	18679	-909	-0,61	-1,3	
5	20227	639	0,43	0,90	
6	29600	10012	6,7	14,1	
7	18698	-890	-0,60	-1,3	
8					
9	19856	268	0,18	0,38	
10	18372	-1216	-0,82	-1,7	
11	19448	-140	-0,09	-0,20	

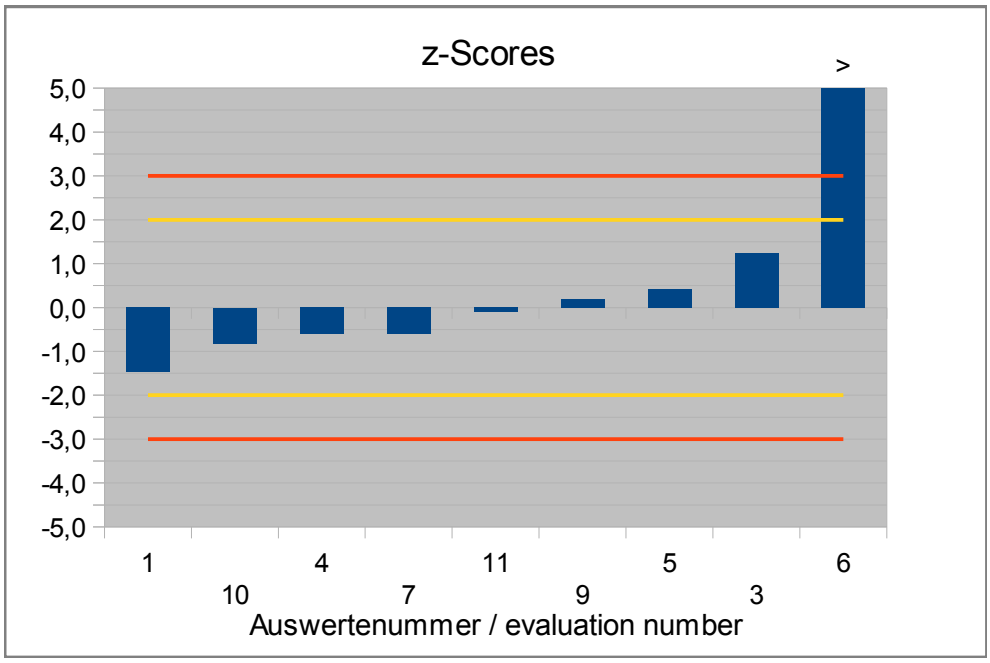


Abb. / Fig. 8: z-Scores Parameter Calcium

4.5 Cu – Copper in mg/kg

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	9
Number of outliers	-
Mean	4,90
Median	4,82
Robust Mean (X)	**4,86**
Robust standard deviation (S*)	**0,291**
Number with 2 replicates	8
Repeatability SD (S_r)	0,0591
Repeatability (CV_r)	1,23%
Reproducibility SD (S_R)	0,265
Reproducibility (CV_R)	5,54%
Target range:	
Target standard deviation σ_{pt}	**0,613**
Target standard deviation (for Information)	0,517
lower limit of target range	**3,64**
upper limit of target range	**6,09**
Quotient S/σ_{pt}*	*0,47*
Standard uncertainty $U_{(Xpt)}$	*0,121*
Results in the target range	*9*
Percent in the target range	*100%*

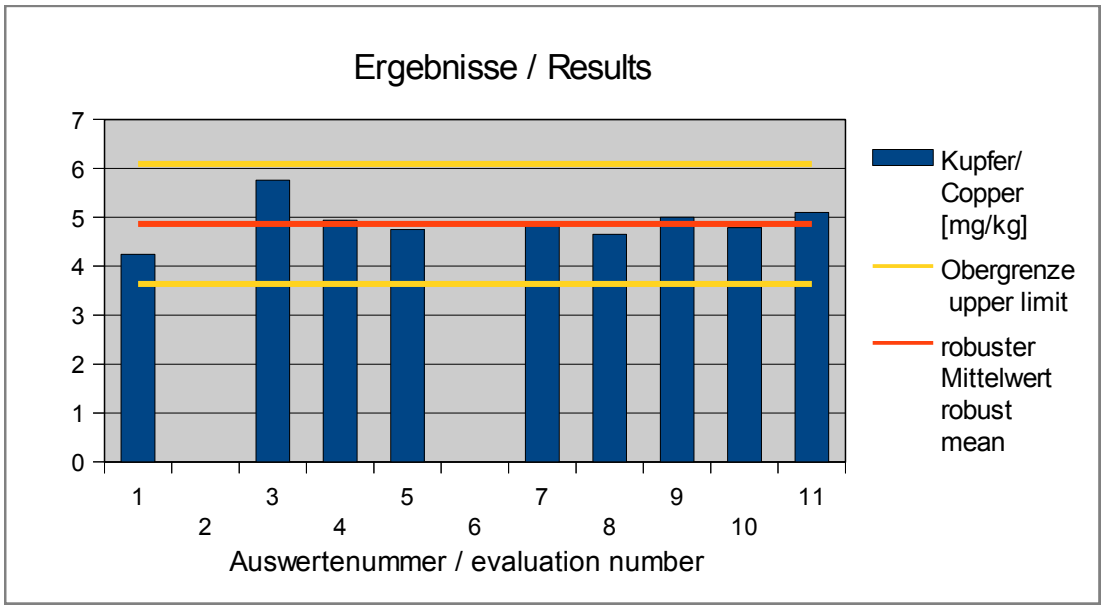


Abb. / Fig. 9: Ergebnisse Kupfer / Results Copper

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Kupfer/ Copper [mg/kg]	Abweichung [mg/kg] Deviation [mg/kg]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	4,25	-0,619	-1,0	-1,2	
2					
3	5,76	0,896	1,5	1,7	
4	4,94	0,075	0,12	0,14	
5	4,75	-0,114	-0,19	-0,22	
6					
7	4,82	-0,044	-0,07	-0,09	
8	4,65	-0,213	-0,35	-0,41	
9	5,00	0,136	0,22	0,26	
10	4,79	-0,074	-0,12	-0,14	
11	5,10	0,236	0,38	0,46	

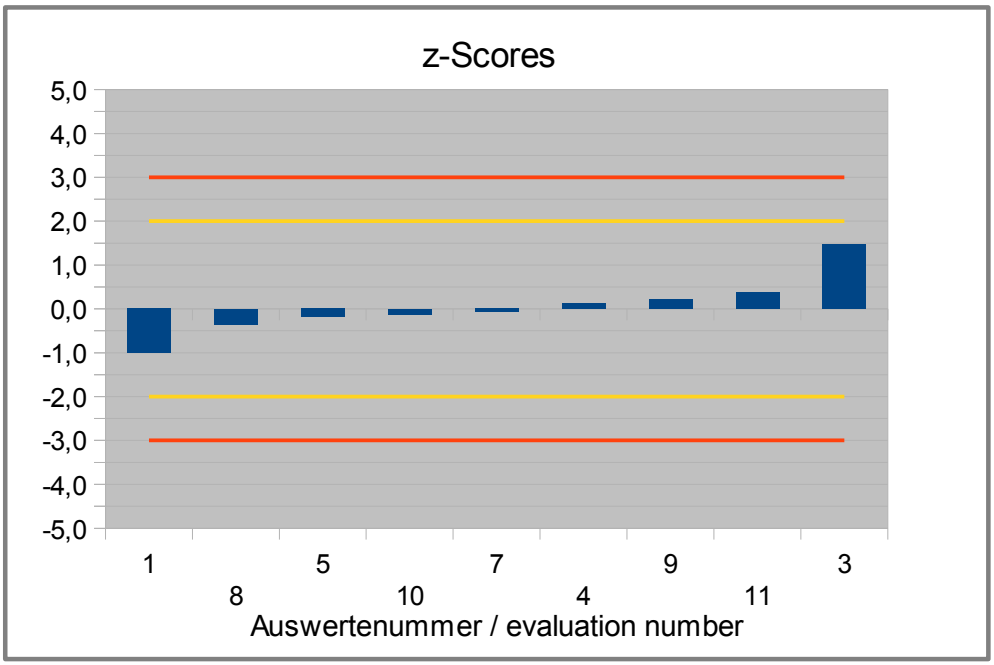


Abb. / Fig. 10: z-Scores Kupfer / Copper

4.6 Fe – Iron in mg/kg

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	10
Number of outliers	0
Mean	108
Median	109
Robust Mean (X)	**108**
Robust standard deviation (S*)	**6,65**
Number with 2 replicates	10
Repeatability SD (S_r)	2,01
Repeatability (CV_r)	1,86%
Reproducibility SD (S_R)	7,10
Reproducibility (CV_R)	6,59%
Target range:	
Target standard deviation σ_{pt}	**8,56**
Target standard deviation (for Information)	7,26
lower limit of target range	**91,2**
upper limit of target range	**125**
Quotient S/σ_{pt}*	*0,78*
Standard uncertainty $U_{(Xpt)}$	*2,63*
Results in the target range	*10*
Percent in the target range	*100%*

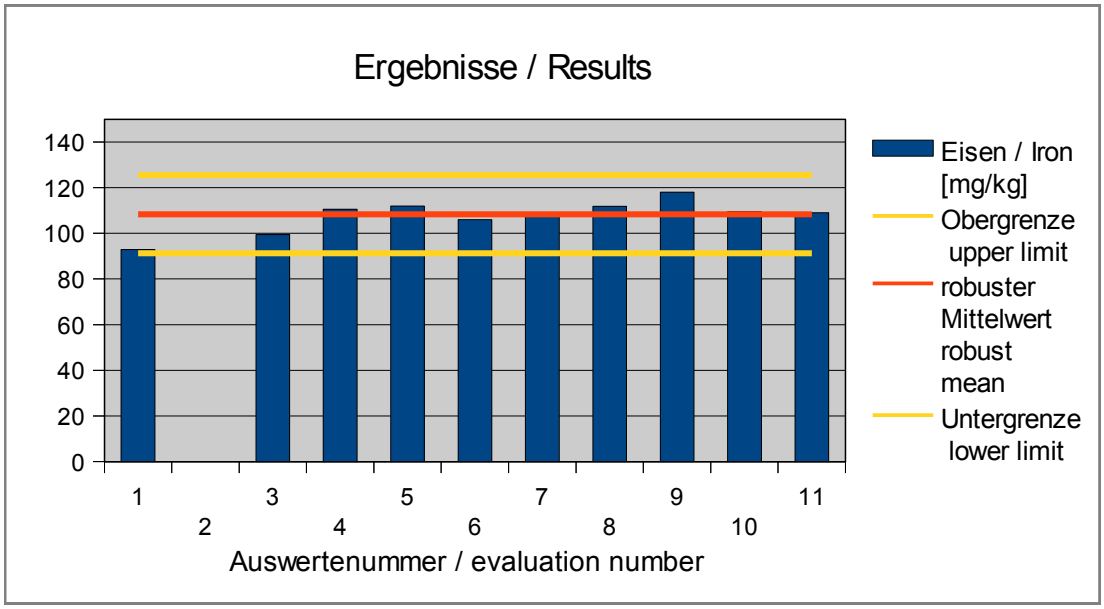


Abb. / Fig. 11: Ergebnisse Eisen / Results Iron

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Eisen / Iron [mg/kg]	Abweichung [mg/kg] Deviation [mg/kg]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	92,9	-15,42	-1,8	-2,1	
2					
3	99,4	-8,90	-1,0	-1,2	
4	111	2,20	0,26	0,30	
5	112	3,65	0,43	0,50	
6	106	-2,35	-0,27	-0,32	
7	109	0,45	0,05	0,06	
8	112	3,45	0,40	0,47	
9	118	9,65	1,1	1,3	
10	110	1,19	0,14	0,16	
11	109	0,65	0,08	0,09	

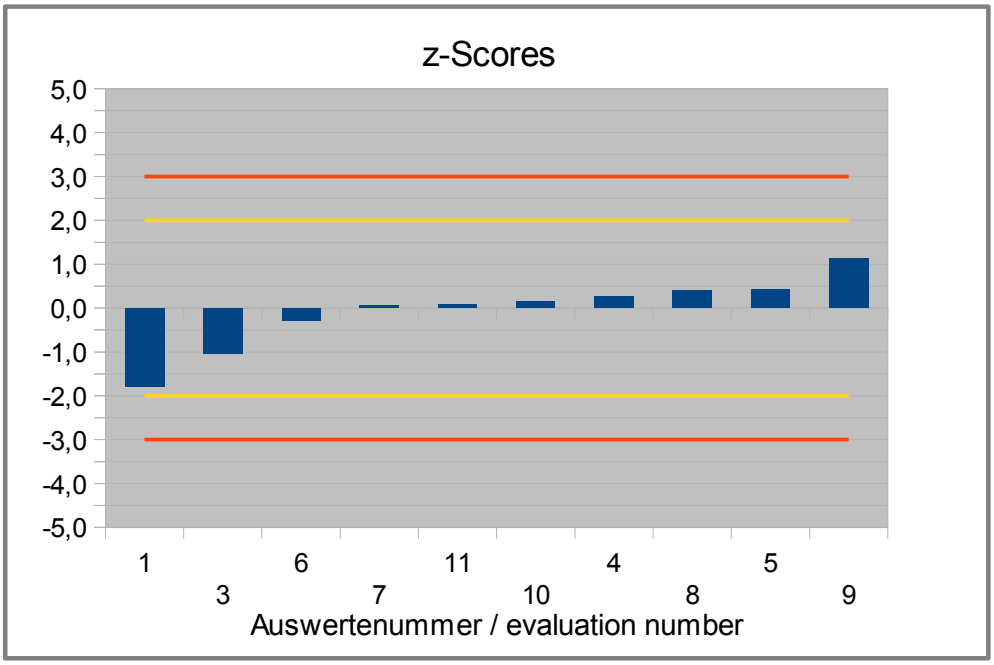


Abb. / Fig. 12: z-Scores Eisen / Iron

4.7 I - Iodine in mg/kg

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	4
Number of outliers	
Mean	0,421
Median	0,425
Robust Mean (X)	**0,421**
Robust standard deviation (S*)	**0,0360**
Number with 2 replicates	
Repeatability SD (S_r)	
Repeatability (CV_r)	
Reproducibility SD (S_R)	
Reproducibility (CV_R)	
Target range:	
Target standard deviation σ_{pt}	
Target standard deviation (for Information)	
lower limit of target range	
upper limit of target range	
Quotient S/σ_{pt}*	
Standard uncertainty $U_{(Xpt)}$	
Results in the target range	
Percent in the target range	

Aufgrund der geringen Anzahl der vorliegenden Ergebnisse (<5) wurde keine statistische Auswertung vorgenommen.

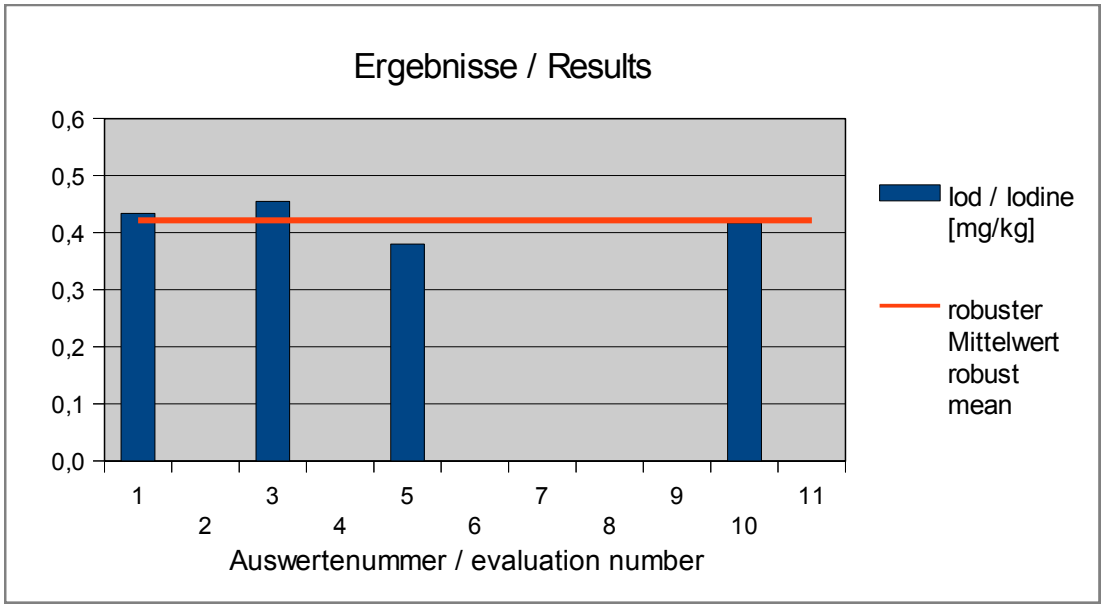


Abb. / Fig. 13: Ergebnisse Iod / Results Iodine

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Iod / Iodine [mg/kg]	Abweichung [mg/kg] Deviation [mg/kg]	z-Score (σ_{pt})	Hinweis Remark
1	0,434	0,0124		
2				
3	0,455	0,0335		
4				
5	0,380	-0,0415		
6				
7				
8				
9				
10	0,417	-0,0045		
11				

4.8 K – Potassium in mg/kg

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	9
Number of outliers	0
Mean	12496
Median	12500
Robust Mean (X)	**12420**
Robust standard deviation (S*)	**1223**
Number with 2 replicates	9
Repeatability SD (S_r)	164
Repeatability (CV_r)	1,32%
Reproducibility SD (S_R)	1590
Reproducibility (CV_R)	12,7%
Target range:	
Target standard deviation σ_{pt}'	**701**
Target standard deviation (for Information)	580
lower limit of target range	**11019**
upper limit of target range	**13822**
Quotient S/σ_{pt}'*	*1,7*
Standard uncertainty $U_{(Xpt)}$	*510*
Results in the target range	*7*
Percent in the target range	*78%*

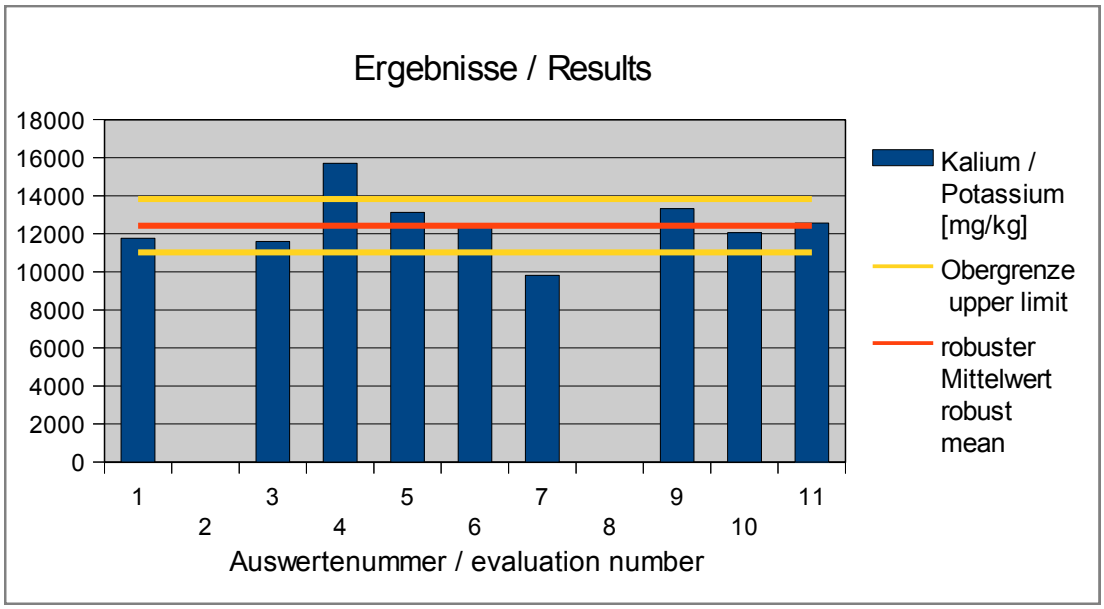


Abb. / Fig. 14: Ergebnisse Kalium / Results Potassium

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Kalium / Potassium [mg/kg]	Abweichung [mg/kg] Deviation [mg/kg]	z'-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	11765	-655	-0,93	-1,1	
2					
3	11600	-820	-1,2	-1,4	
4	15707	3286	4,7	5,7	
5	13130	710	1,0	1,2	
6	12500	80	0,11	0,14	
7	9813	-2607	-3,7	-4,5	
8					
9	13324	904	1,3	1,6	
10	12063	-357	-0,51	-0,62	
11	12560	140	0,20	0,24	

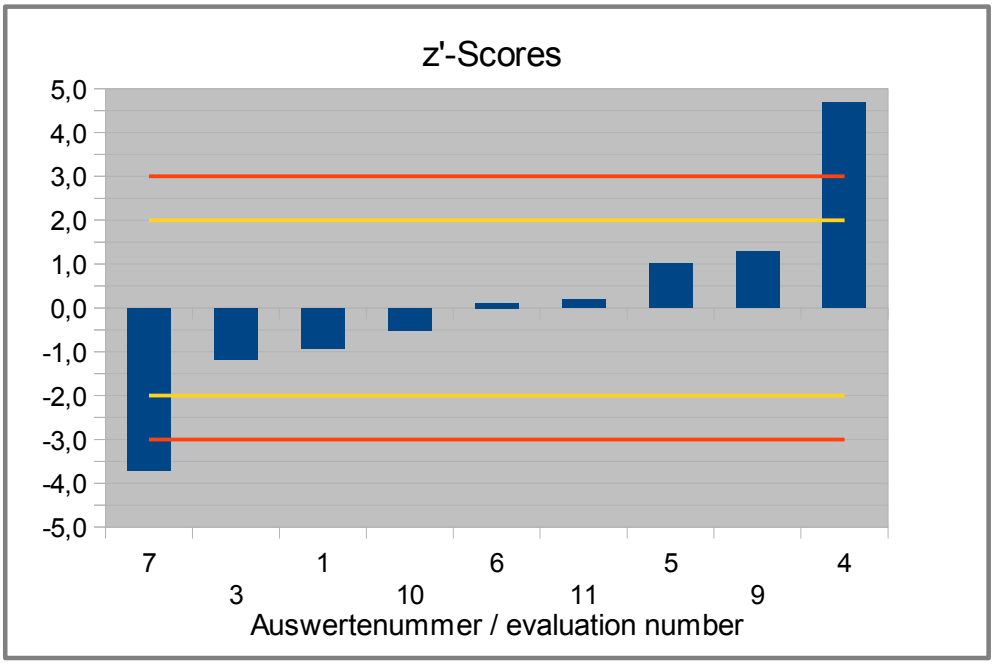


Abb. / Fig. 15: z'-Scores Kalium / Potassium

4.9 Mg – Magnesium in mg/kg

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	8
Number of outliers	0
Mean	4691
Median	4742
Robust Mean (X)	**4704**
Robust standard deviation (S*)	**261**
Number with 2 replicates	8
Repeatability SD (S_r)	99,0
Repeatability (CV_r)	2,11%
Reproducibility SD (S_R)	265
Reproducibility (CV_R)	5,65%
Target range:	
Target standard deviation σ_{pt}	**211**
Target standard deviation (for Information)	330
lower limit of target range	**4282**
upper limit of target range	**5125**
Quotient S/σ_{pt}*	*1,2*
Standard uncertainty $U_{(Xpt)}$	*116*
Results in the target range	*7*
Percent in the target range	*88%*

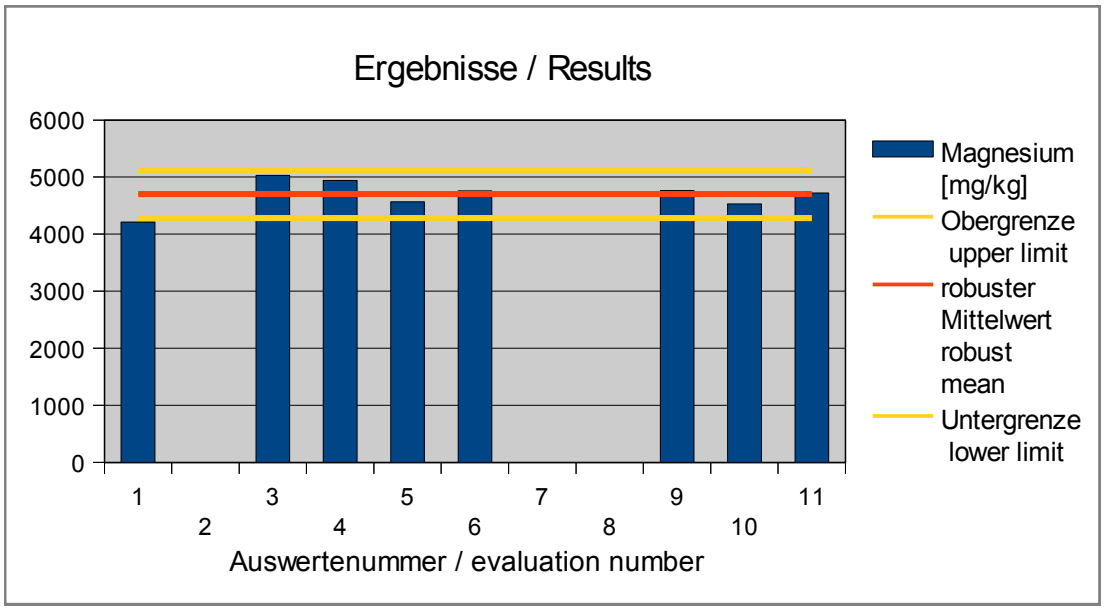


Abb. / Fig. 16: Ergebnisse Magnesium / Results Magnesium

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Magnesium [mg/kg]	Abweichung [mg/kg] Deviation [mg/kg]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	4214	-490	-2,3	-1,5	
2					
3	5028	324	1,5	1,0	
4	4943	239	1,1	0,73	
5	4569	-135	-0,64	-0,41	
6	4760	56	0,27	0,17	
7					
8					
9	4765	61	0,29	0,19	
10	4529	-175	-0,83	-0,53	
11	4724	20	0,10	0,06	

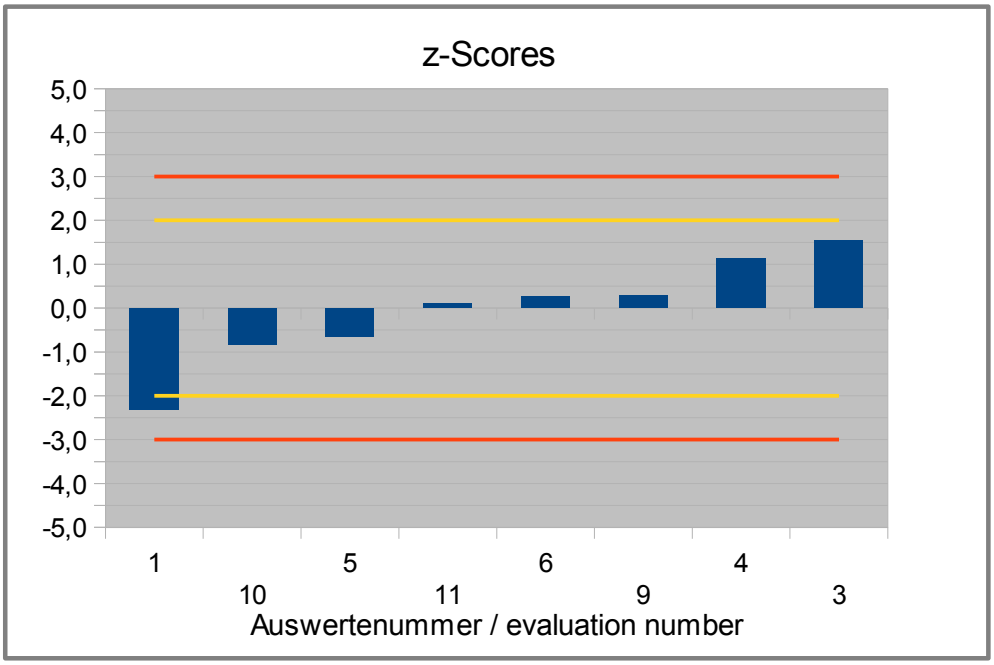


Abb. / Fig. 17: z-Scores Magnesium

4.10 Mn – Manganese in mg/kg

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	10
Number of outliers	0
Mean	61,9
Median	61,6
Robust Mean (X)	**61,9**
Robust standard deviation (S*)	**4,93**
Number with 2 replicates	10
Repeatability SD (S_r)	0,682
Repeatability (CV_r)	1,10%
Reproducibility SD (S_R)	4,37
Reproducibility (CV_R)	7,05%
Target range:	
Target standard deviation σ_{pt}	**5,32**
Target standard deviation (for Information)	8,23
lower limit of target range	**51,3**
upper limit of target range	**72,6**
Quotient S/σ_{pt}*	*0,93*
Standard uncertainty $U_{(Xpt)}$	*1,95*
Results in the target range	*10*
Percent in the target range	*100%*

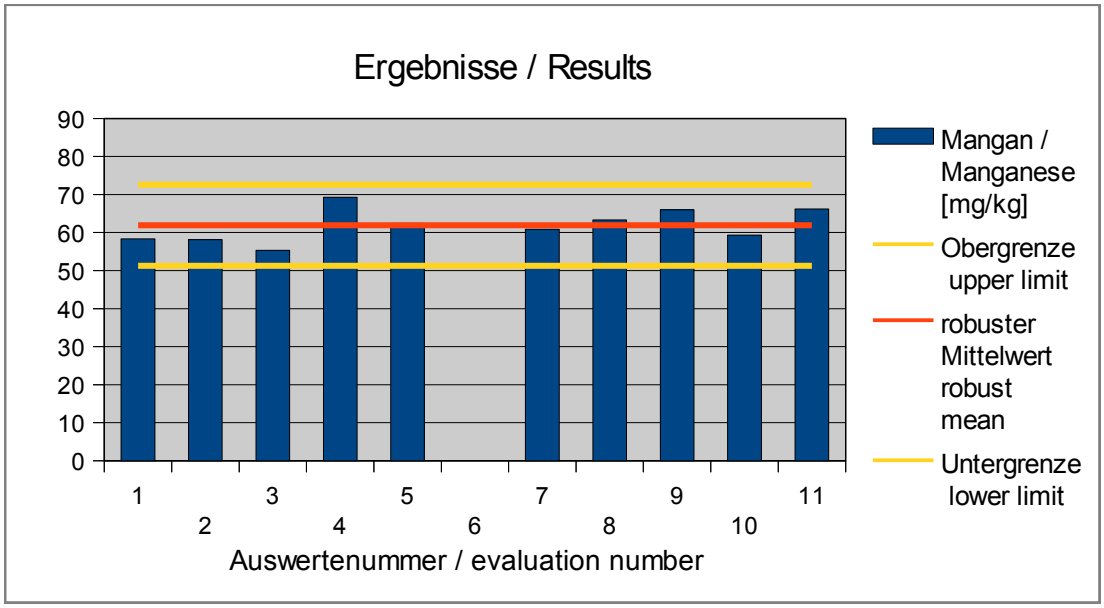


Abb. / Fig. 18: Ergebnisse Mangan / Results Manganese

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Mangan / Manganese [mg/kg]	Abweichung [mg/kg] Deviation [mg/kg]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	58,4	-3,56	-0,67	-0,43	
2	58,2	-3,76	-0,71	-0,46	
3	55,3	-6,63	-1,2	-0,81	
4	69,3	7,36	1,4	0,89	
5	62,4	0,48	0,09	0,06	
6					
7	60,9	-1,04	-0,20	-0,13	
8	63,3	1,40	0,26	0,17	
9	66,0	4,08	0,77	0,50	
10	59,3	-2,60	-0,49	-0,32	
11	66,2	4,28	0,80	0,52	

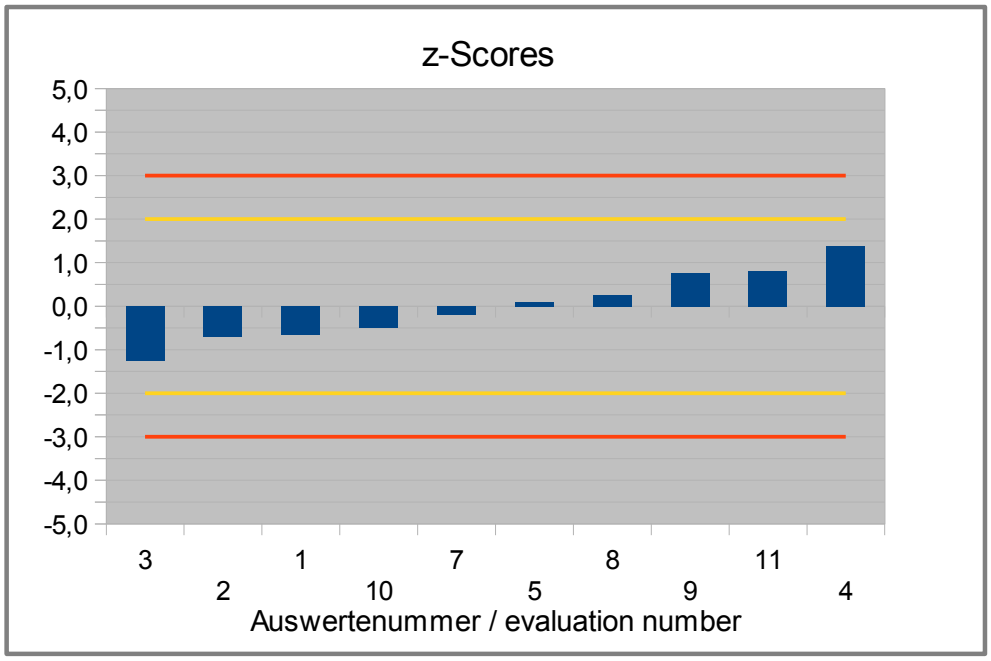


Abb. / Fig. 19: z-Scores Mangan / Manganese

4.11 Mo - Molybdenum in mg/kg

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	8
Number of outliers	0
Mean	0,447
Median	0,451
Robust Mean (X)	**0,449**
Robust standard deviation (S*)	**0,0385**
Number with 2 replicates	8
Repeatability SD (S_r)	0,00521
Repeatability (CV_r)	1,17%
Reproducibility SD (S_R)	0,0365
Reproducibility (CV_R)	8,18%
Target range:	
Target standard deviation σ_{pt}	**0,0810**
Target standard deviation (for Information)	0,0854
lower limit of target range	**0,287**
upper limit of target range	**0,610**
Quotient S/σ_{pt}*	*0,48*
Standard uncertainty $U_{(Xpt)}$	*0,0170*
Results in the target range	*8*
Percent in the target range	*100%*

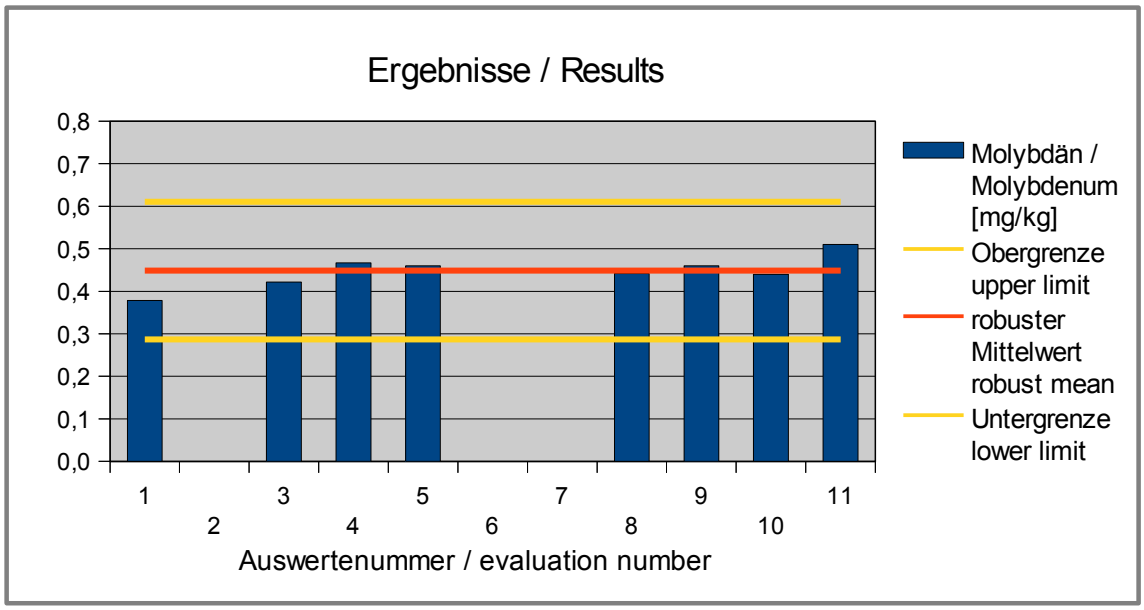


Abb. / Fig. 20: Ergebnisse Molybdän / Results Molybdenum

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Molybdän / Molybdenum [mg/kg]	Abweichung [mg/kg] Deviation [mg/kg]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	0,378	-0,0701	-0,87	-0,82	
2					
3	0,422	-0,0265	-0,33	-0,31	
4	0,467	0,0185	0,23	0,22	
5	0,460	0,0115	0,14	0,13	
6					
7					
8	0,442	-0,0065	-0,08	-0,08	
9	0,460	0,0115	0,14	0,13	
10	0,440	-0,0085	-0,10	-0,10	
11	0,510	0,0615	0,76	0,72	

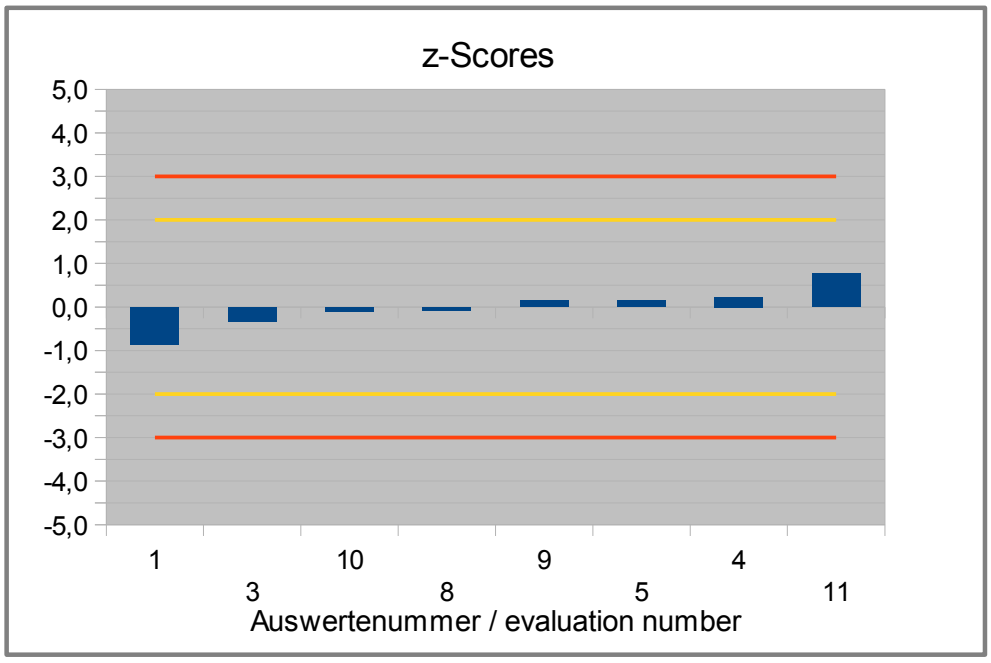


Abb. / Fig. 21: z-Scores Molybdän / Molybdenum

4.12 Na – Sodium in mg/kg

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	8
Number of outliers	0
Mean	2475
Median	2491
Robust Mean (X)	**2478**
Robust standard deviation (S*)	**92,0**
Number with 2 replicates	8
Repeatability SD (S_r)	67,2
Repeatability (CV_r)	2,72%
Reproducibility SD (S_R)	101
Reproducibility (CV_R)	4,08%
Target range:	
Target standard deviation σ_{pt}	**122**
Target standard deviation (for Information)	103
lower limit of target range	**2234**
upper limit of target range	**2723**
Quotient S/σ_{pt}*	*0,75*
Standard uncertainty $U_{(Xpt)}$	*40,7*
Results in the target range	*8*
Percent in the target range	*100%*

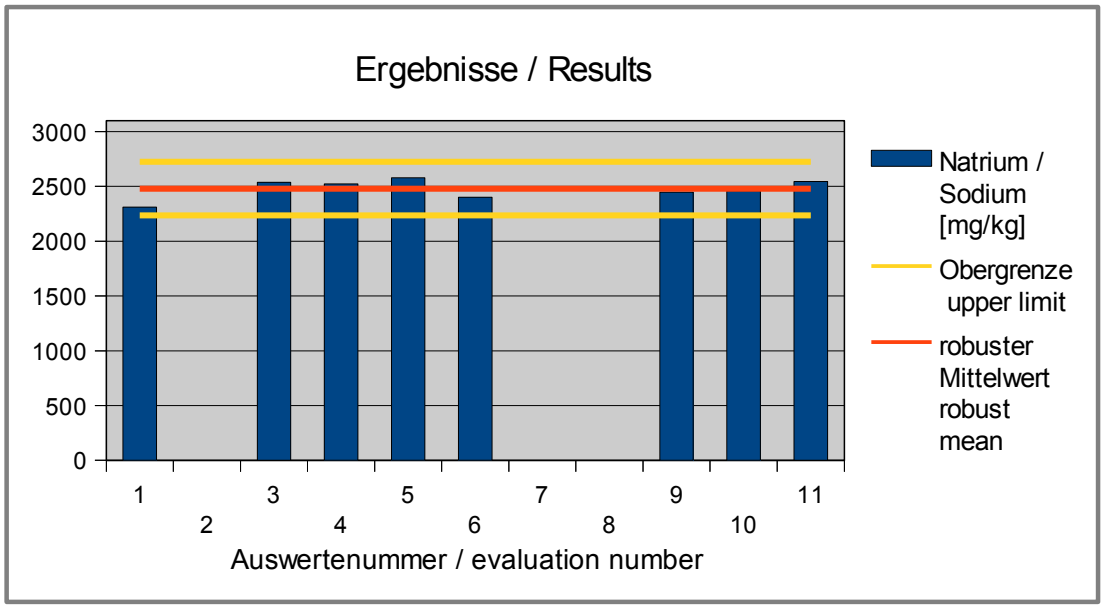


Abb. / Fig. 22: Ergebnisse Natrium / Results Sodium

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Natrium / Sodium [mg/kg]	Abweichung [mg/kg] Deviation [mg/kg]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	2309	-169,0	-1,4	-1,6	
2					
3	2537	58,6	0,48	0,57	
4	2523	44,6	0,36	0,43	
5	2578	99,6	0,81	1,0	
6	2400	-78,4	-0,64	-0,76	
7					
8					
9	2447	-31,4	-0,26	-0,31	
10	2459	-19,4	-0,16	-0,19	
11	2543	64,6	0,53	0,63	

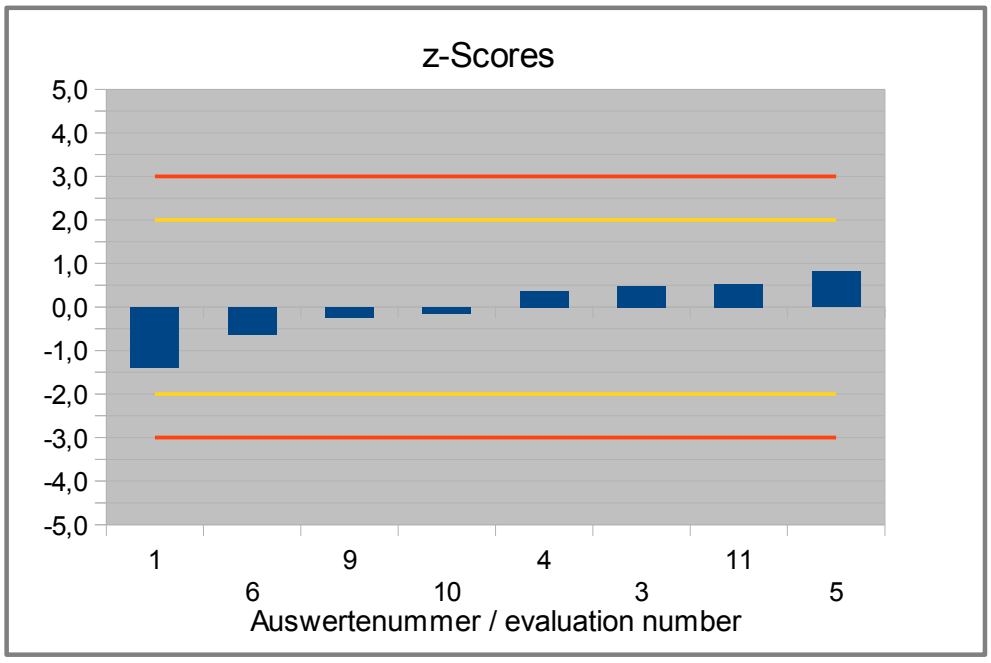


Abb. / Fig. 23: z-Scores Natrium / Sodium

4.13 Ni - Nickel in mg/kg

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	10
Number of outliers	-
Mean	0,811
Median	0,689
Robust Mean (X)	**0,709**
Robust standard deviation (S*)	**0,122**
Number with 2 replicates	9
Repeatability SD (S_r)	0,0173
Repeatability (CV_r)	2,49%
Reproducibility SD (S_R)	0,109
Reproducibility (CV_R)	15,7%
Target range:	
Target standard deviation σ_{pt}	**0,119**
lower limit of target range	**0,470**
upper limit of target range	**0,947**
Quotient S/σ_{pt}*	*1,0*
Standard uncertainty $U_{(Xpt)}$	*0,0481*
Results in the target range	*8*
Percent in the target range	*80%*

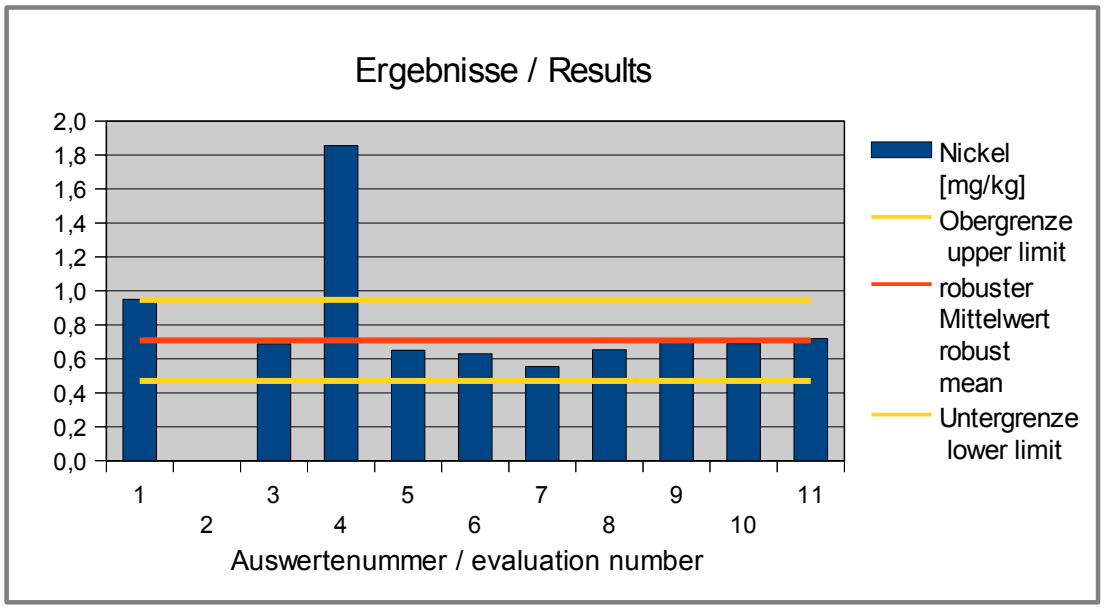


Abb. / Fig. 24: Ergebnisse Nickel / Results Nickel

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Nickel [mg/kg]	Abweichung [mg/kg] Deviation [mg/kg]	z-Score (σ_{pt})	Hinweis Remark
1	0,950	0,242	2,0	
2				
3	0,688	-0,021	-0,17	
4	1,86	1,146	9,6	
5	0,650	-0,059	-0,49	
6	0,630	-0,079	-0,66	
7	0,554	-0,155	-1,3	
8	0,654	-0,055	-0,46	
9	0,720	0,011	0,09	
10	0,690	-0,019	-0,16	
11	0,720	0,011	0,09	

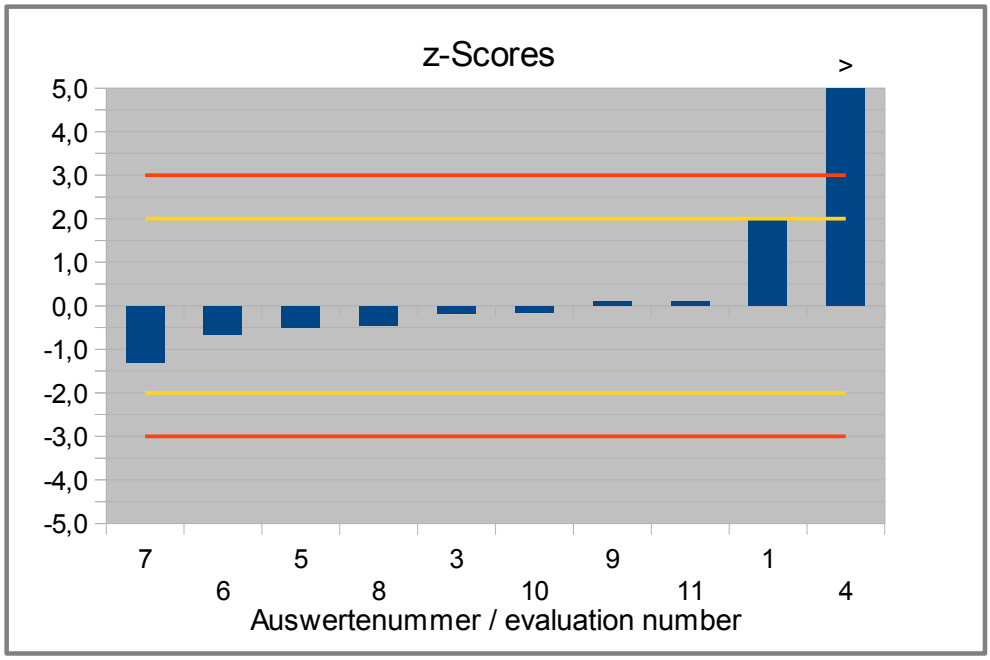


Abb. / Fig. 25: z-Scores Nickel

4.14 P – Phosphorus in mg/kg

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	6
Number of outliers	0
Mean	2292
Median	2310
Robust Mean (X)	**2310**
Robust standard deviation (S*)	**167**
Number with 2 replicates	6
Repeatability SD (S_r)	20,3
Repeatability (CV_r)	0,885%
Reproducibility SD (S_R)	187
Reproducibility (CV_R)	8,17%
Target range:	
Target standard deviation σ_{pt}	**173**
Target standard deviation (for Information)	115
lower limit of target range	**1965**
upper limit of target range	**2656**
Quotient S/σ_{pt}*	*1,0*
Standard uncertainty $U_{(Xpt)}$	*85,4*
Results in the target range	*5*
Percent in the target range	*83%*

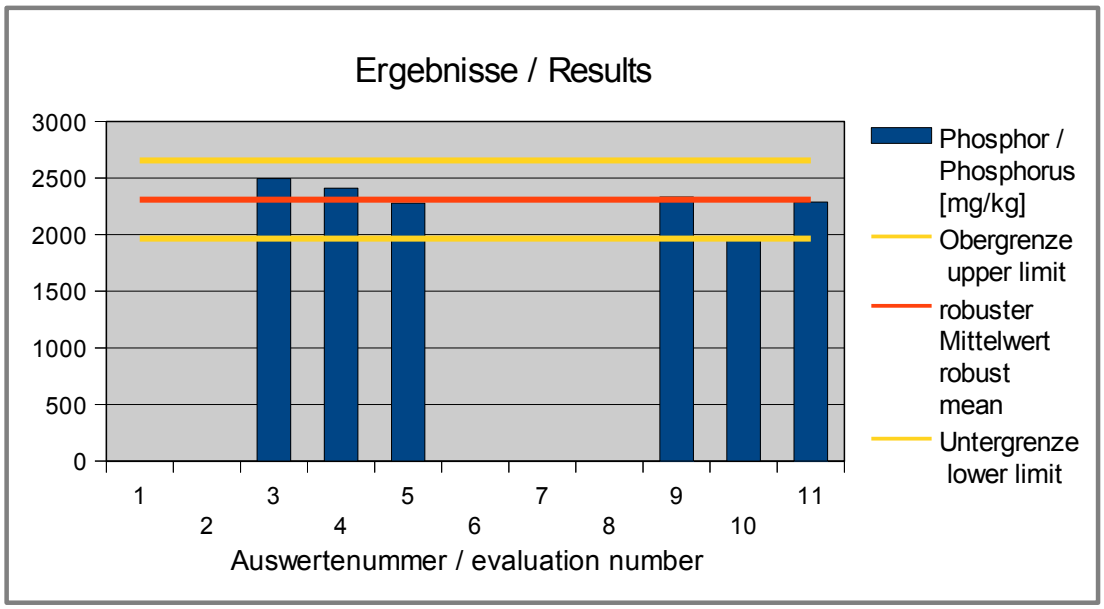


Abb. / Fig. 26: Ergebnisse Phosphor / Results Phosphorus

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Phosphor / Phosphorus [mg/kg]	Abweichung [mg/kg] Deviation [mg/kg]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1					
2					
3	2494	184	1,1	1,6	
4	2411	101	0,58	0,87	
5	2278	-32	-0,19	-0,28	
6					
7					
8					
9	2333	23	0,13	0,20	
10	1949	-361	-2,1	-3,1	
11	2287	-23	-0,14	-0,20	

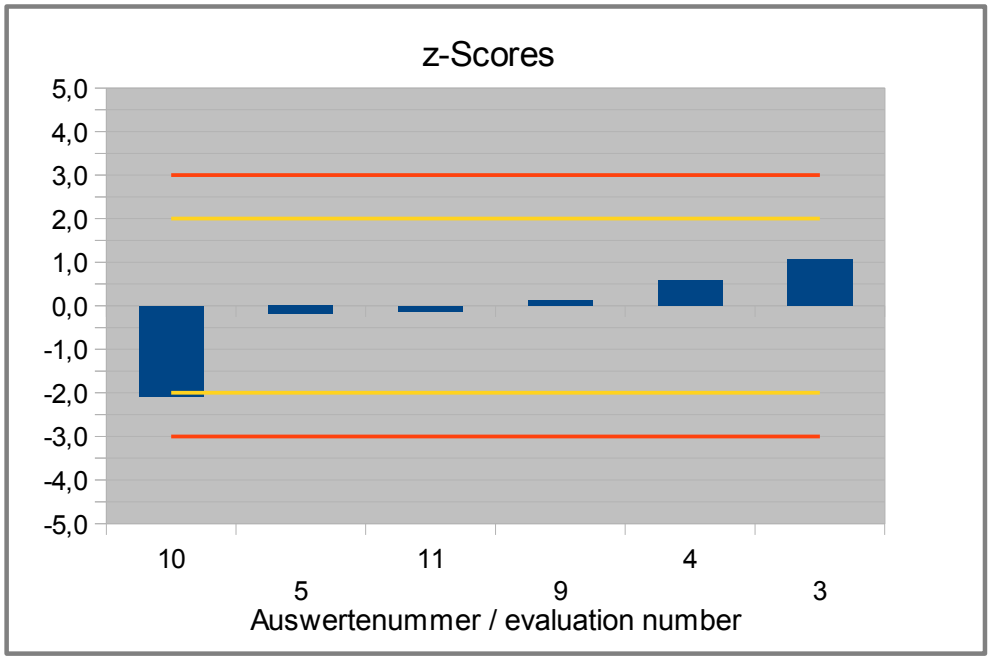


Abb. / Fig. 27: z-Scores Phosphor / Phosphorus

4.15 Pb - Lead in mg/kg

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	9
Number of outliers	-
Mean	0,247
Median	0,251
Robust Mean (X)	**0,258**
Robust standard deviation (S*)	**0,0288**
Number with 2 replicates	8
Repeatability SD (S_r)	0,00412
Repeatability (CV_r)	1,57%
Reproducibility SD (S_R)	0,0209
Reproducibility (CV_R)	7,95%
Target range:	
Target standard deviation σ_{pt}	**0,0506**
Target standard deviation (for Information)	0,0185
lower limit of target range	**0,157**
upper limit of target range	**0,359**
Quotient S/σ_{pt}*	*0,57*
Standard uncertainty $U_{(Xpt)}$	*0,0120*
Results in the target range	*8*
Percent in the target range	*89%*

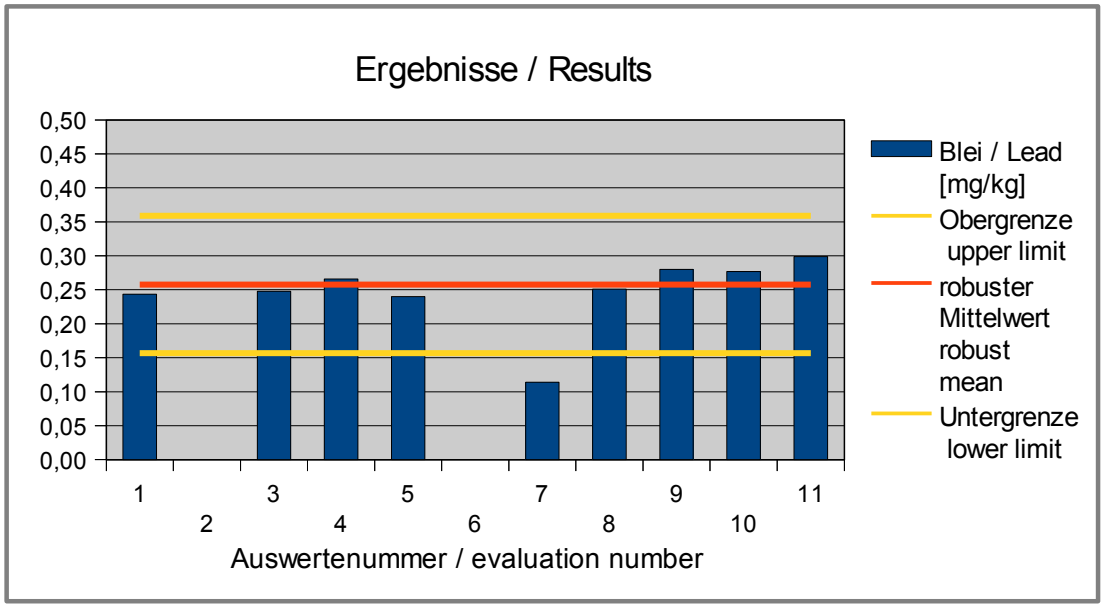


Abb. / Fig. 28: Ergebnisse Blei / Results Lead

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Blei / Lead [mg/kg]	Abweichung [mg/kg] Deviation [mg/kg]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	0,244	-0,0140	-0,28	-0,76	
2					
3	0,248	-0,0097	-0,19	-0,52	
4	0,266	0,0083	0,16	0,45	
5	0,240	-0,0177	-0,35	-1,0	
6	< 0,04				
7	0,114	-0,1437	-2,8	-7,8	
8	0,251	-0,0067	-0,13	-0,36	
9	0,280	0,0223	0,44	1,2	
10	0,277	0,0193	0,38	1,0	
11	0,299	0,0413	0,82	2,2	

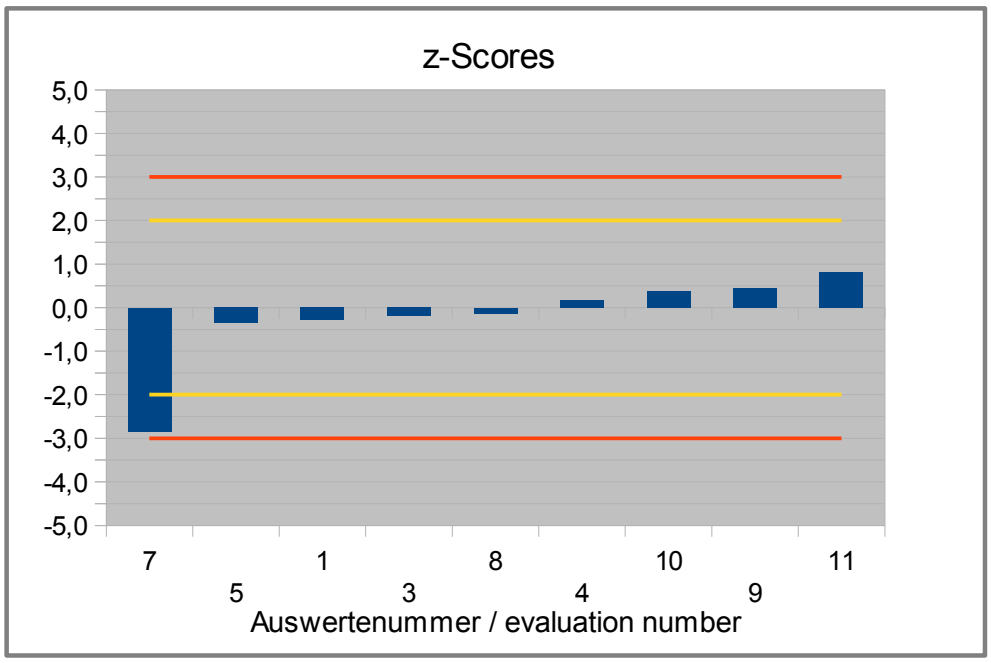


Abb. / Fig. 29: z-Scores Blei / Lead

4.16 Rb - Rubidium in mg/kg

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	4
Number of outliers	
Mean	18,1
Median	17,5
Robust Mean (X)	**18,1**
Robust standard deviation (S*)	**1,57**
Number with 2 replicates	
Repeatability SD (S_r)	
Repeatability (CV_r)	
Reproducibility SD (S_R)	
Reproducibility (CV_R)	
Target range:	
Target standard deviation σ_{pt}	
Target standard deviation (for Information)	
lower limit of target range	
upper limit of target range	
Quotient S/σ_{pt}*	
Standard uncertainty $U_{(Xpt)}$	
Results in the target range	
Percent in the target range	

Aufgrund der geringen Anzahl der vorliegenden Ergebnisse (<5) wurde keine statistische Auswertung vorgenommen.

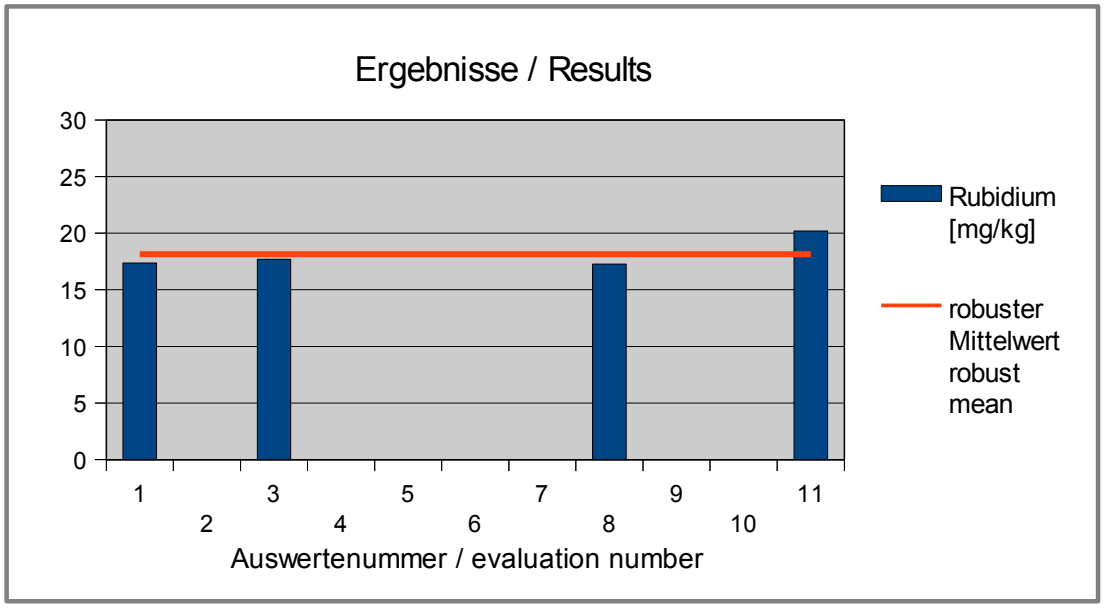


Abb. / Fig. 30: Ergebnisse Rubidium / Results Rubidium

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Rubidium [mg/kg]	Abweichung [mg/kg] Deviation [mg/kg]	z-Score (σ_{pt})	Hinweis Remark
1	17,4	-0,76		
2				
3	17,7	-0,44		
4				
5				
6				
7				
8	17,3	-0,87		
9				
10				
11	20,2	2,06		

4.17 Se - Selenium in mg/kg

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	8
Number of outliers	0
Mean	0,578
Median	0,560
Robust Mean (X)	**0,578**
Robust standard deviation (S*)	**0,0961**
Number with 2 replicates	8
Repeatability SD (S_r)	0,00954
Repeatability (CV_r)	1,65%
Reproducibility SD (S_R)	0,0849
Reproducibility (CV_R)	14,7%
Target range:	
Target standard deviation σ_{pt}	**0,100**
Target standard deviation (for Information)	0,0675
lower limit of target range	**0,377**
upper limit of target range	**0,779**
Quotient S/σ_{pt}*	*0,96*
Standard uncertainty $U_{(Xpt)}$	*0,0425*
Results in the target range	*8*
Percent in the target range	*100%*

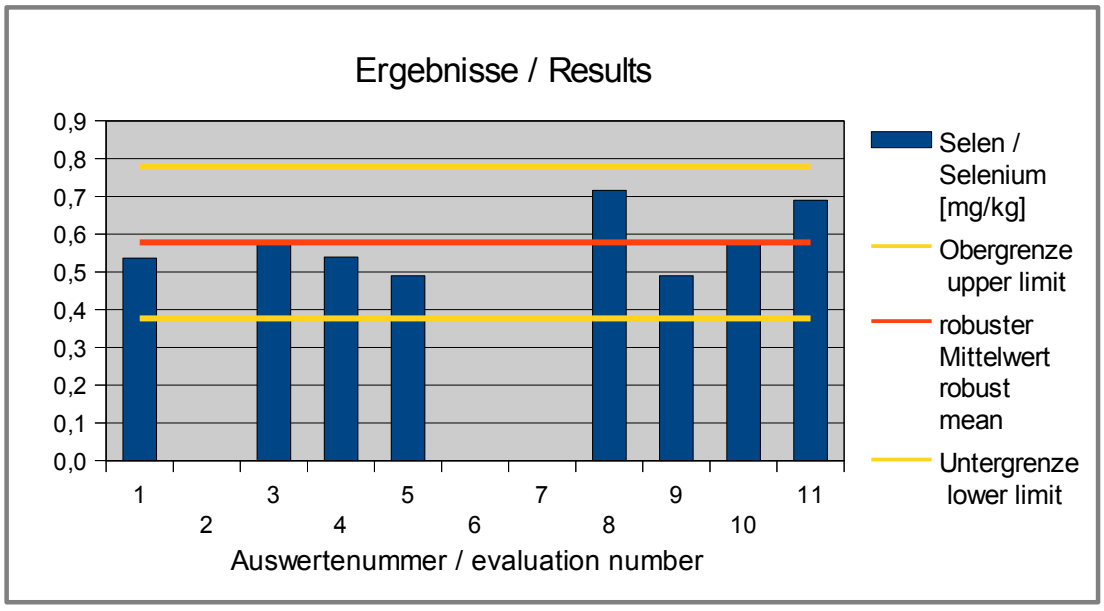


Abb. / Fig. 31: Ergebnisse Selen / Results Selenium

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Selen / Selenium [mg/kg]	Abweichung [mg/kg] Deviation [mg/kg]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	0,537	-0,0412	-0,41	-0,61	
2					
3	0,582	0,0040	0,04	0,06	
4	0,539	-0,0390	-0,39	-0,58	
5	0,490	-0,0880	-0,88	-1,3	
6					
7					
8	0,716	0,1380	1,4	2,0	
9	0,490	-0,0880	-0,88	-1,3	
10	0,580	0,0020	0,02	0,03	
11	0,690	0,1120	1,1	1,7	

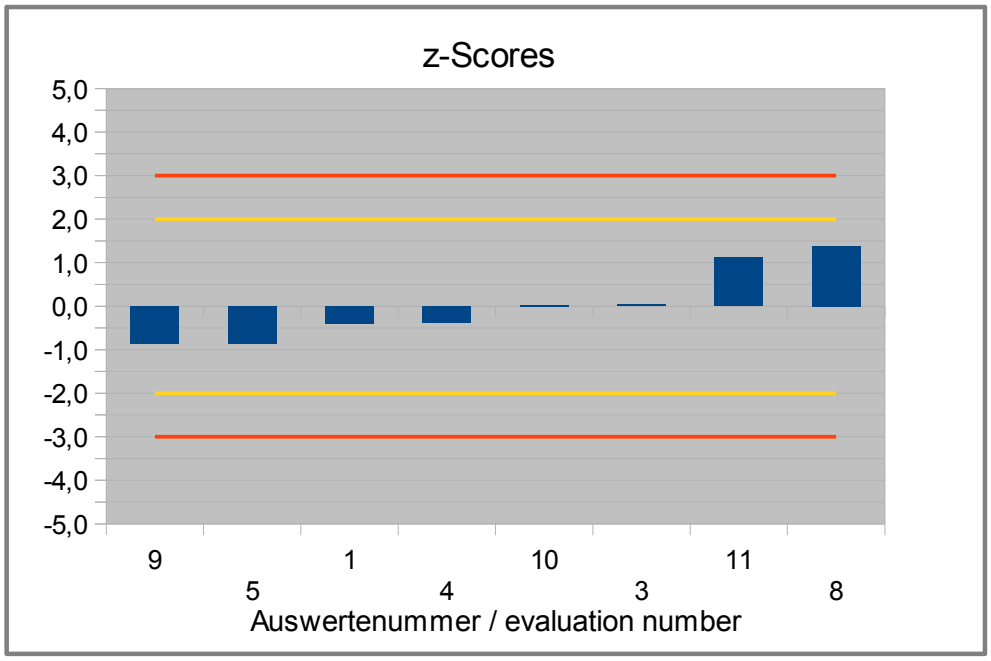


Abb. / Fig. 32: z-Scores Selen / Selenium

4.18 Sn - Tin in mg/kg

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	6
Number of outliers	0
Mean	1,52
Median	1,52
Robust Mean (X)	**1,52**
Robust standard deviation (S*)	**0,0731**
Number with 2 replicates	6
Repeatability SD (S_r)	0,0885
Repeatability (CV_r)	5,86%
Reproducibility SD (S_R)	0,0921
Reproducibility (CV_R)	6,09%
Target range:	
Target standard deviation σ_{pt}	**0,229**
lower limit of target range	**1,06**
upper limit of target range	**1,98**
Quotient S/σ_{pt}*	*0,32*
Standard uncertainty $U_{(Xpt)}$	*0,0373*
Results in the target range	*6*
Percent in the target range	*100%*

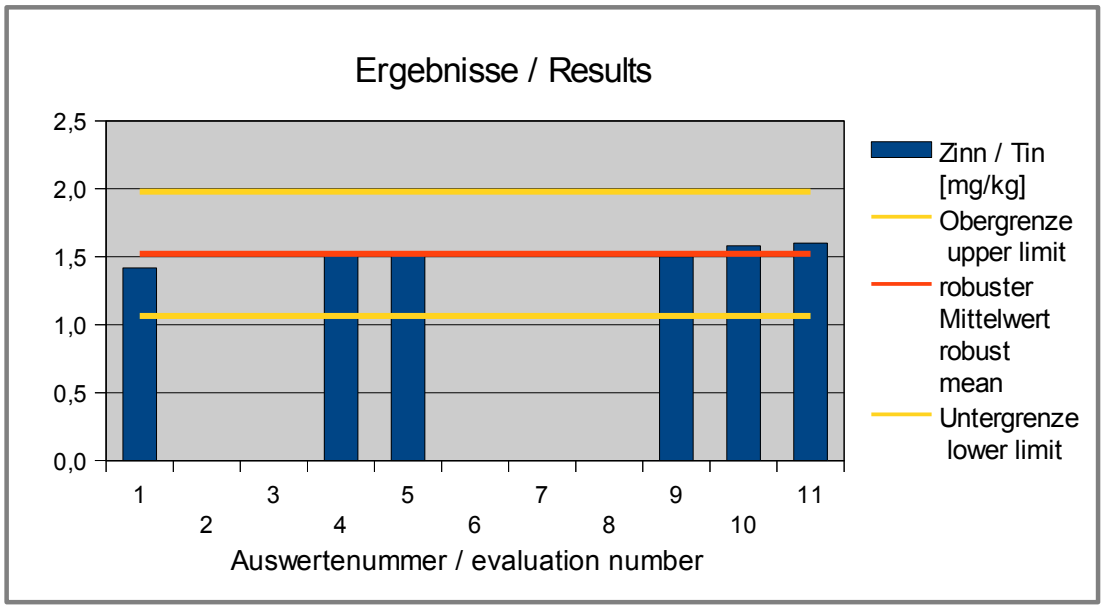


Abb. / Fig. 33: Ergebnisse Zinn / Tin

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Zinn / Tin [mg/kg]	Abweichung [mg/kg] Deviation [mg/kg]	z-Score (σ_{pt})	Hinweis Remark
1	1,42	-0,1041	-0,46	
2				
3				
4	1,52	-0,0072	-0,03	
5	1,52	-0,0022	-0,01	
6				
7				
8				
9	1,50	-0,0222	-0,10	
10	1,58	0,0578	0,25	
11	1,60	0,0778	0,34	

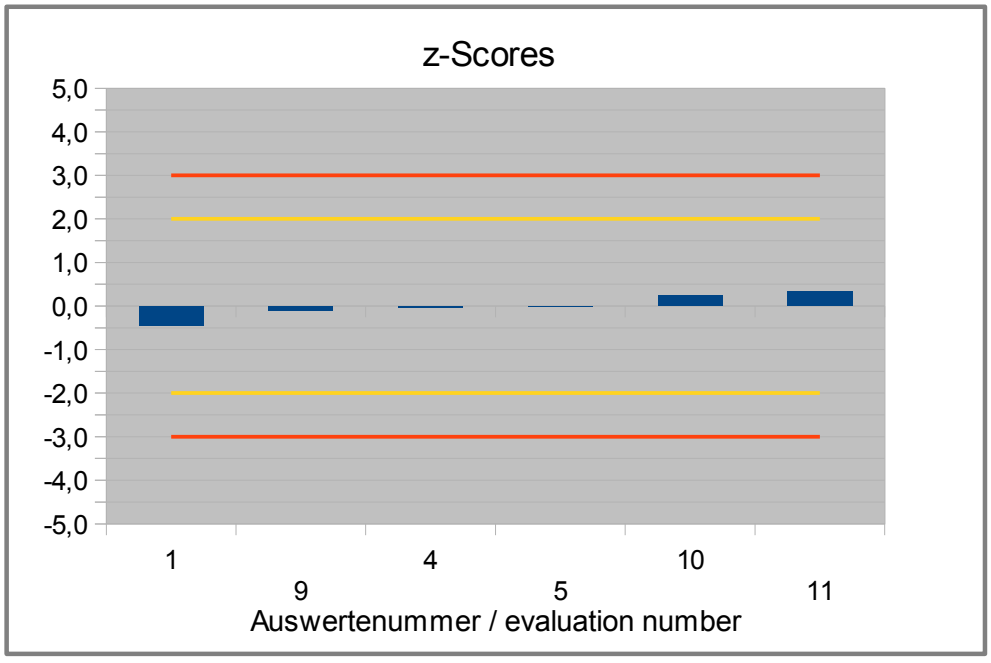


Abb. / Fig. 34: z-Scores Zinn / Tin

4.19 U - Uranium in mg/kg

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	6
Number of outliers	0
Mean	0,300
Median	0,291
Robust Mean (X)	**0,300**
Robust standard deviation (S*)	**0,0310**
Number with 2 replicates	6
Repeatability SD (S_r)	0,00549
Repeatability (CV_r)	1,83%
Reproducibility SD (S_R)	0,0263
Reproducibility (CV_R)	8,78%
Target range:	
Target standard deviation σ_{pt}	**0,0576**
lower limit of target range	**0,185**
upper limit of target range	**0,416**
Quotient S/σ_{pt}*	*0,54*
Standard uncertainty $U_{(Xpt)}$	*0,0158*
Results in the target range	*6*
Percent in the target range	*100%*

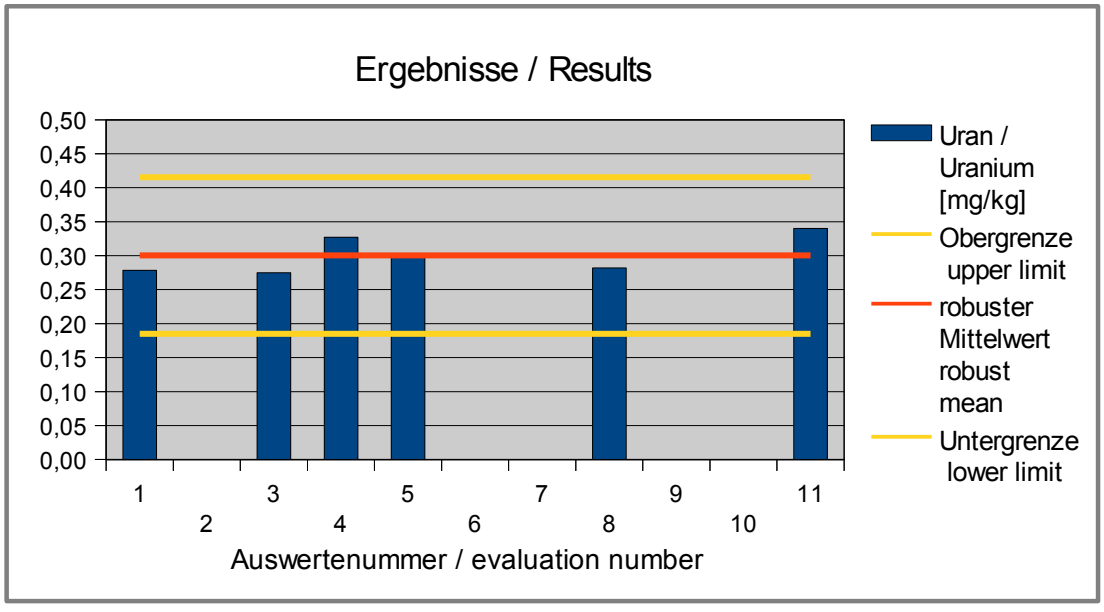


Abb. / Fig. 35: Ergebnisse Uran / Results Uranium

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Uran / Uranium [mg/kg]	Abweichung [mg/kg] Deviation [mg/kg]	z-Score (σ_{pt})	Hinweis Remark
1	0,278	-0,0219	-0,38	
2				
3	0,275	-0,0254	-0,44	
4	0,327	0,0266	0,46	
5	0,300	-0,0004	-0,01	
6				
7				
8	0,282	-0,0184	-0,32	
9				
10				
11	0,340	0,0396	0,69	

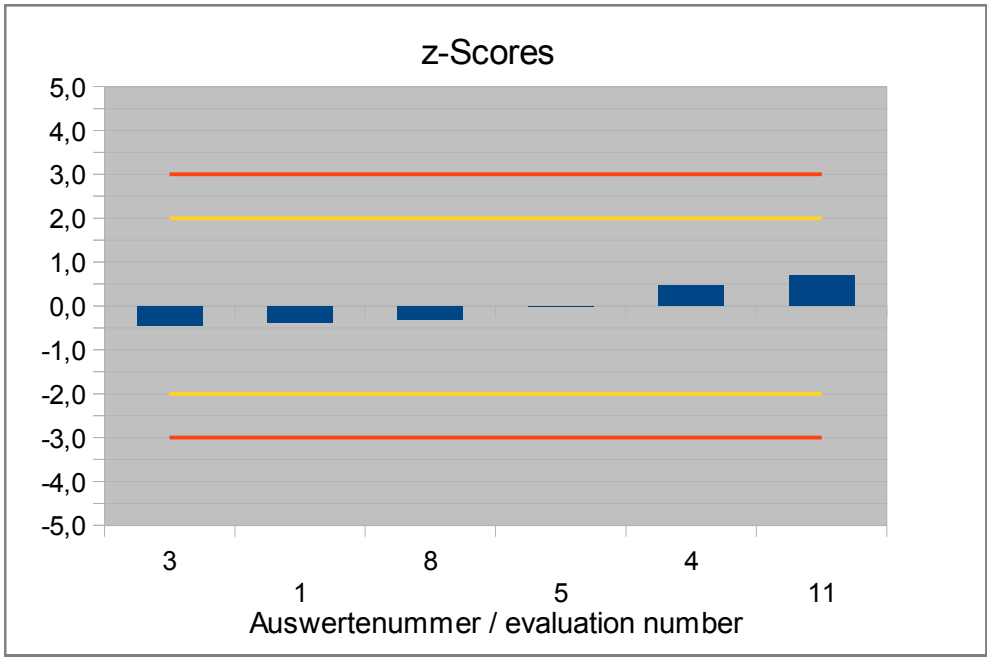


Abb. / Fig. 36: z-Scores Uran / Uranium

4.20 Zn – Zinc in mg/kg

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	9
Number of outliers	0
Mean	14,5
Median	14,2
Robust Mean (X)	**14,5**
Robust standard deviation (S*)	**2,19**
Number with 2 replicates	9
Repeatability SD (S_r)	0,280
Repeatability (CV_r)	1,94%
Reproducibility SD (S_R)	1,99
Reproducibility (CV_R)	13,7%
Target range:	
Target standard deviation σ_{pt}	**1,55**
Target standard deviation (for Information)	0,961
lower limit of target range	**11,4**
upper limit of target range	**17,6**
Quotient S/σ_{pt}*	*1,4*
Standard uncertainty $U_{(Xpt)}$	*0,914*
Results in the target range	*8*
Percent in the target range	*89%*

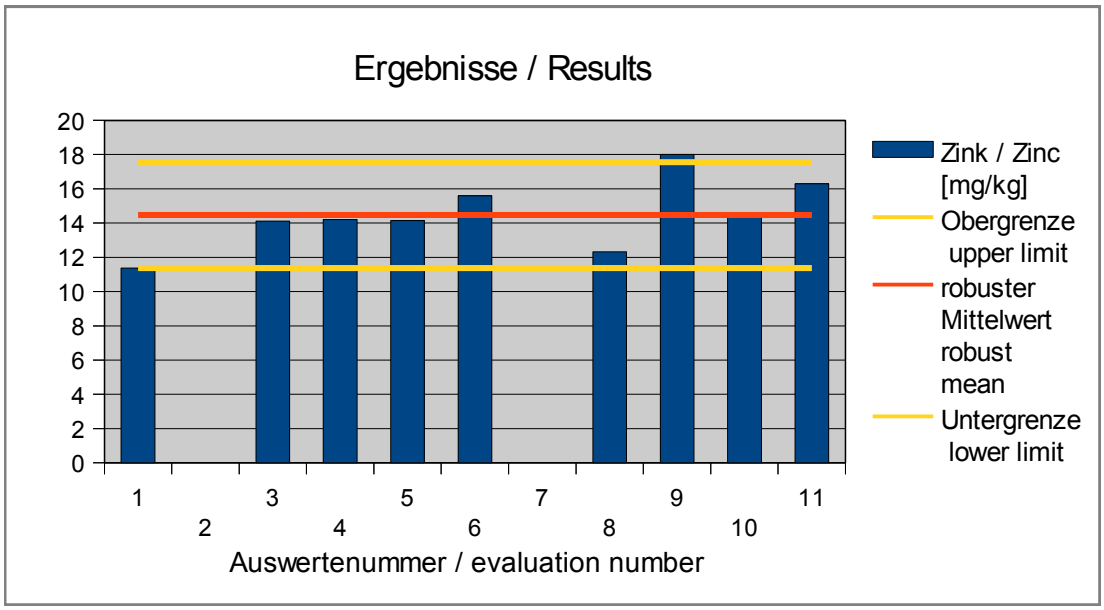


Abb. / Fig. 37: Ergebnisse Zink / Results Zinc

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Zink / Zinc [mg/kg]	Abweichung [mg/kg] Deviation [mg/kg]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	11,4	-3,09	-2,0	-3,2	
2					
3	14,1	-0,36	-0,23	-0,37	
4	14,2	-0,26	-0,17	-0,27	
5	14,1	-0,33	-0,21	-0,34	
6	15,6	1,13	0,73	1,2	
7					
8	12,3	-2,15	-1,4	-2,2	
9	18,0	3,53	2,3	3,7	
10	14,4	-0,07	-0,04	-0,07	
11	16,3	1,83	1,2	1,9	

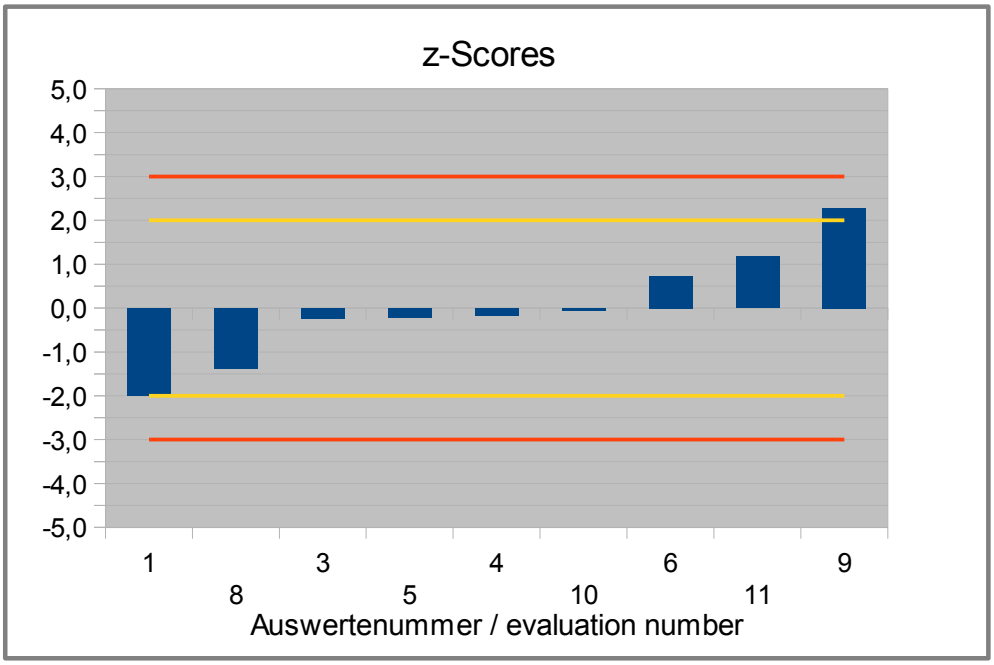


Abb. / Fig. 38: z-Scores Zink / Zinc

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

Analyte	Partici-pant	Unit	Sample I DLA No.	Sample II DLA No.	Date of analysis	Result (Mean)	Result I	Result II	Limit of determination	Incl. RR	Recovery rate
					Day/Month					yes / no	in %
Al-Aluminium	1	mg/kg	18	54	10. Apr	27,8487727062	27,930833	27,766713	0,05	no	99
	2	mg/kg	29	43							
	3	mg/kg	23	49	15.05.19	39,71	38,71	40,71	0,5	no	
	4	mg/kg	13	59	09.05.19	44,483	44,717	44,249	keine	yes	80-120
	5	mg/kg	33	39		46,3	46,46	46,13	<5,00	no	
	6	mg/kg	22	50							
	7	mg/kg	40	47	06.05.19	47,8	46,4	49,2	0,3	no	
	8	mg/kg	34	38	18.4.	39,63	40,84	38,41	1,3 µg/L	no	not done
	9	mg/kg	17	55	14.05.19	50	48	51	5	no	-
	10	mg/kg	12	60	03.05.19	45,98	45,65	46,31	3	no	n.a.
	11	mg/kg	11	61	8,05	48,6	48,9	48,4	1	no	-

Analyte	Partici-pant	Unit	Sample I DLA No.	Sample II DLA No.	Date of analysis	Result (Mean)	Result I	Result II	Limit of determination	Incl. RR	Recovery rate
					Day/Month					yes / no	in %
B-Boron	1	mg/kg	18	54	10. Apr	26,7786429365	26,657362	26,899924	0,05	no	95
	2	mg/kg	29	43							
	3	mg/kg	23	49	25.04.19	30,91	30,89	30,92	10	no	
	4	mg/kg	13	59	09.05.19	30,379	30,238	30,519	keine	yes	80-120
	5	mg/kg	33	39		33,2	33,03	33,37	<1,5	no	
	6	mg/kg	22	50							
	7	mg/kg	40	47							
	8	mg/kg	34	38	18.4.	32,45	33,20	31,69	9,1 µg/L	no	not done
	9	mg/kg	17	55	14.05.19	31	31	31	10	no	-
	10	mg/kg	12	60	not done	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	11	mg/kg	11	61	8,05	35,3	35,4	35,3	0,5	no	-

Analyte	Partici-pant	Unit	Sample I DLA No.	Sample II DLA No.	Date of analysis	Result (Mean)	Result I	Result II	Limit of determination	Incl. RR	Recovery rate
					Day/Month					yes / no	in %
Ba-Barium	1	mg/kg	18	54	10. Apr	71,9513309676	72,060362	71,8423	0,01	no	100
	2	mg/kg	29	43							
	3	mg/kg	23	49	25.04.19	66,94	67,006667	66,866667	10	no	
	4	mg/kg	13	59	09.05.19	71,932	71,911	71,952	keine	yes	80-120
	5	mg/kg	33	39		68,85	69,2	68,5	< 0,50	no	
	6	mg/kg	22	50							
	7	mg/kg	40	47							
	8	mg/kg	34	38	18.4.	71,72	71,05	72,39	0,091 µg/L	no	not done
	9	mg/kg	17	55	14.05.19	72	72	71	0,05	no	-
	10	mg/kg	12	60	08.05.19	73,11	72,5	73,72	0,09	no	n.a.
	11	mg/kg	11	61	8,05	72,8	73,1	72,6	0,2	no	-

Analyte	Partici-pant	Unit	Sample I DLA No.	Sample II DLA No.	Date of analysis	Result (Mean)	Result I	Result II	Limit of determination	Incl. RR	Recovery rate
					Day/Month					yes / no	in %
Ca-Calcium	1	mg/kg	18	54	10. Apr	17394,2895241329	17477,806	17310,773	5	no	100
	2	mg/kg	29	43							
	3	mg/kg	23	49	25.04.19	21440	21106,667	21780	50	no	
	4	mg/kg	13	59	09.05.19	18679,208	18764,352	18594,064	keine	yes	80-120
	5	mg/kg	33	39		20227	20240	20213	<50	no	
	6	mg/kg	22	50	24.04.	29600	29900	29300	30	no	
	7	mg/kg	40	47	06.05.19	18698	18594	18801	30	no	
	8	mg/kg	34	38							
	9	mg/kg	17	55	14.05.19	19856	20038	19674	10	no	-
	10	mg/kg	12	60	10.05.19	18372	18389	18354	18	no	n.a.
	11	mg/kg	11	61	8,05	19448	19539	19356	0,1	no	-

Analyte	Partici-pant	Unit	Sample I DLA No.	Sample II DLA No.	Date of analysis	Result (Mean)	Result I	Result II	Limit of determination	Incl. RR	Recovery rate
					Day/Month					yes / no	in %
Cu-Copper	1	mg/kg	18	54	10. Apr	4,2455725255	4,2588029	4,2323421	0,01	no	105
	2	mg/kg	29	43							
	3	mg/kg	23	49	25.04.19	5,76	5,805804	5,7122323	1	no	
	4	mg/kg	13	59	09.05.19	4,939	4,951	4,928	keine	yes	80-120
	5	mg/kg	33	39		4,75	4,78	4,73	<0,50	no	
	6	mg/kg	22	50							
	7	mg/kg	40	47	06.05.19	4,82	4,72	4,92	0,06	no	
	8	mg/kg	34	38	18.4.	4,651	4,682	4,619	0,038 µg/L	no	not done
	9	mg/kg	17	55	14.05.19	5	5	5	0,5	no	-
	10	mg/kg	12	60	06.05.19	4,79	4,83	4,74	0,06	no	n.a.
	11	mg/kg	11	61	8,05	5,1	5,1	5,1	0,3	no	-

Analyte	Partici-pant	Unit	Sample I DLA No.	Sample II DLA No.	Date of analysis	Result (Mean)	Result I	Result II	Limit of determination	Incl. RR	Recovery rate
					Day/Month					yes / no	in %
Fe-Iron	1	mg/kg	18	54	10. Apr	92,9308391649	94,338237	91,523441	0,2	no	99
	2	mg/kg	29	43							
	3	mg/kg	23	49	25.04.19	99,4483333333	99,406667	99,49	5	no	
	4	mg/kg	13	59	09.05.19	110,555	109,622	111,487	keine	yes	80-120
	5	mg/kg	33	39		112	112	112	<0,50	no	
	6	mg/kg	22	50	23.05.	106	110	102	20	no	
	7	mg/kg	40	47	06.05.19	108,8	108,6	109	0,6	no	
	8	mg/kg	34	38	18.4.	111,8	112,4	111,1	0,11 µg/L	no	not done
	9	mg/kg	17	55	14.05.19	118	118	117	0,5	no	-
	10	mg/kg	12	60	06.05.19	109,54	110,17	108,92	3	no	n.a.
	11	mg/kg	11	61	8,05	109	110	109	0,3	no	-

Analyte	Partici-pant	Unit	Sample I DLA No.	Sample II DLA No.	Date of analysis	Result (Mean)	Result I	Result II	Limit of determination	Incl. RR	Recovery rate
					Day/Month					yes / no	in %
I-Iodine	1	mg/kg	18	54	10. Apr	0,4338730434	0,4098235	0,4579226	0,05	no	100
	2	mg/kg	29	43							
	3	mg/kg	23	49	23.05.19	0,455	0,4694	0,44145	0,2	no	
	4	mg/kg	13	59							
	5	mg/kg	33	39		0,38	0,38	0,38	<0,10	no	
	6	mg/kg	22	50							
	7	mg/kg	40	47							
	8	mg/kg	34	38							
	9	mg/kg	17	55	-	-	-	-	-	-	-
	10	mg/kg	12	60	06.05.19	0,417	0,415	0,419	0,1	no	n.a.
	11	mg/kg	11	61	8,05	< 5.0	< 5.0	< 5.0	5	no	-

Analyte	Partici-pant	Unit	Sample I DLA No.	Sample II DLA No.	Date of analysis	Result (Mean)	Result I	Result II	Limit of determination	Incl. RR	Recovery rate
					Day/Month					yes / no	in %
K-Potassium	1	mg/kg	18	54	10. Apr	11765,2377703359	11816,187	11714,288	20	no	95
	2	mg/kg	29	43							
	3	mg/kg	23	49	25.04.19	11600	11626,667	11576,667	20	no	
	4	mg/kg	13	59	09.05.19	15706,748	15523,118	15890,379	none	yes	80-120
	5	mg/kg	33	39		13130	13120	13140	<50	no	
	6	mg/kg	22	50	24.04.	12500	12700	12300	30	no	
	7	mg/kg	40	47	29.04.19	9813	9669	9957	300	no	
	8	mg/kg	34	38							
	9	mg/kg	17	55	14.05.19	13324	13467	13181	20	no	-
	10	mg/kg	12	60	10.05.19	12063	12005	12122	18	no	n.a.
	11	mg/kg	11	61	8,05	12560	12554	12566	10	no	-

Analyte	Partici-pant	Unit	Sample I DLA No.	Sample II DLA No.	Date of analysis	Result (Mean)	Result I	Result II	Limit of determination	Incl. RR	Recovery rate
					Day/Month					yes / no	in %
Mg-Magnesium	1	mg/kg	18	54	10. Apr	4214,0717905739	4240,409	4187,7346	0,1	no	95
	2	mg/kg	29	43							
	3	mg/kg	23	49	25.04.19	5028	4922,6667	5132,3333	20	no	
	4	mg/kg	13	59	09.05.19	4942,714	4829,875	5055,552	none	yes	80-120
	5	mg/kg	33	39		4569	4556	4582	<50	no	
	6	mg/kg	22	50		4760	4880	4650	10	no	
	7	mg/kg	40	47							
	8	mg/kg	34	38							
	9	mg/kg	17	55	14.05.19	4765	4798	4732	10	no	-
	10	mg/kg	12	60	10.05.19	4529	4522	4537	6	no	n.a.
	11	mg/kg	11	61	8,05	4724	4738	4709	1	no	-

Analyte	Partici-pant	Unit	Sample I DLA No.	Sample II DLA No.	Date of analysis	Result (Mean)	Result I	Result II	Limit of determination	Incl. RR	Recovery rate
					Day/Month					yes / no	in %
Mn-Manganese	1	mg/kg	18	54	10. Apr	58,3652427502	58,62875	58,101736	0,01	no	105
	2	mg/kg	29	43		58,16	58,13	58,19			
	3	mg/kg	23	49	25.04.19	55,29	55,26	55,323333	1	no	
	4	mg/kg	13	59	09.05.19	69,279	68,712	69,847	keine	yes	80-120
	5	mg/kg	33	39		62,4	62,49	62,31	<0,50	no	
	6	mg/kg	22	50							
	7	mg/kg	40	47	06.05.19	60,88	60,69	61,06	0,06	no	
	8	mg/kg	34	38	18.4.	63,32	64,09	62,54	0,010 µg/L	no	not done
	9	mg/kg	17	55	14.05.19	66	66	66	0,05	no	-
	10	mg/kg	12	60	03.05.19	59,32	58,19	60,44	0,03	no	n.a.
	11	mg/kg	11	61	8,05	66,2	66,3	66	0,1	no	-

Analyte	Partici-pant	Unit	Sample I DLA No.	Sample II DLA No.	Date of analysis	Result (Mean)	Result I	Result II	Limit of determination	Incl. RR	Recovery rate
					Day/Month					yes / no	in %
Mo-Molybdenum	1	mg/kg	18	54	10. Apr	0,3783975194	0,3795023	0,3772927	0,01	no	100
	2	mg/kg	29	43							
	3	mg/kg	23	49	25.04.19	0,422	0,4247215	0,4201829	0,2	no	
	4	mg/kg	13	59	09.05.19	0,467	0,467	0,467	keine	yes	80-120
	5	mg/kg	33	39		0,46	0,45	0,46	<0,01	no	
	6	mg/kg	22	50							
	7	mg/kg	40	47							
	8	mg/kg	34	38	18.4.	0,442	0,440	0,443	0,013 µg/L	no	not done
	9	mg/kg	17	55	14.05.19	0,46	0,46	0,45	0,05	no	-
	10	mg/kg	12	60	08.05.19	0,44	0,44	0,45	0,03	no	n.a.
	11	mg/kg	11	61	8,05	0,51	0,51	0,5	0,1	no	-

Analyte	Partici-pant	Unit	Sample I DLA No.	Sample II DLA No.	Date of analysis	Result (Mean)	Result I	Result II	Limit of determination	Incl. RR	Recovery rate
					Day/Month					yes / no	in %
Na-Sodium	1	mg/kg	18	54	10. Apr	2309,4435899598	2315,1086	2303,7786	2	no	100
	2	mg/kg	29	43							
	3	mg/kg	23	49	25.04.19	2537	2440,3333	2634,3333	50	no	
	4	mg/kg	13	59	09.05.19	2523,04	2504,59	2541,49	keine	yes	80-120
	5	mg/kg	33	39		2578	2586	2570	<50	no	
	6	mg/kg	22	50	18.04.	2400	2490	2310	10	no	
	7	mg/kg	40	47							
	8	mg/kg	34	38							
	9	mg/kg	17	55	14.05.19	2447	2445	2448	10	no	-
	10	mg/kg	12	60	10.05.19	2459	2449	2469	9	no	n.a.
	11	mg/kg	11	61	8,05	2543	2546	2539	5	no	-

Analyte	Partici-pant	Unit	Sample I DLA No.	Sample II DLA No.	Date of analysis	Result (Mean)	Result I	Result II	Limit of determination	Incl. RR	Recovery rate
					Day/Month					yes / no	in %
Ni-Nickel	1	mg/kg	18	54	10. Apr	0,9503119615	0,9485692	0,9520547	0,01	no	100
	2	mg/kg	29	43							
	3	mg/kg	23	49	25.04.19	0,688	0,7069649	0,6690072	0,2	no	
	4	mg/kg	13	59	09.05.19	1,855	1,838	1,871	keine	yes	80-120
	5	mg/kg	33	39		0,65	0,63	0,67	<0,10	no	
	6	mg/kg	22	50	25.04.	0,63	0,65	0,61	0,05	no	
	7	mg/kg	40	47	06.05.19	0,554	0,545	0,562	0,06	no	
	8	mg/kg	34	38	18.4.	0,654	0,659	0,648	0,041 µg/L	no	not done
	9	mg/kg	17	55	14.05.19	0,72	0,72	0,71	0,5	no	-
	10	mg/kg	12	60	06.05.19	0,69	0,7	0,69	0,9	no	n.a.
	11	mg/kg	11	61	8,05	0,72	0,71	0,72	0,2	no	-

Analyte	Partici-pant	Unit	Sample I DLA No.	Sample II DLA No.	Date of analysis	Result (Mean)	Result I	Result II	Limit of determination	Incl. RR	Recovery rate
					Day/Month					yes / no	in %
P-Phosphorus	1	mg/kg	18	54	10. Apr						
	2	mg/kg	29	43							
	3	mg/kg	23	49	25.04.19	2494	2475,6667	2511,6667	50	no	
	4	mg/kg	13	59	09.05.19	2411,101	2423,207	2398,995	keine	yes	80-120
	5	mg/kg	33	39		2278	2273	2283	<50	no	
	6	mg/kg	22	50							
	7	mg/kg	40	47							
	8	mg/kg	34	38							
	9	mg/kg	17	55	14.05.19	2333	2330	2335	20	no	-
	10	mg/kg	12	60	03.05.19	1949	1975	1923	9	no	n.a.
	11	mg/kg	11	61	8,05	2287	2294	2279	3	no	-

Analyte	Partici-pant	Unit	Sample I DLA No.	Sample II DLA No.	Date of analysis	Result (Mean)	Result I	Result II	Limit of determination	Incl. RR	Recovery rate
					Day/Month					yes / no	in %
Pb-Lead	1	mg/kg	18	54	10. Apr	0,2436279852	0,2449355	0,2423205	0,01	no	95
	2	mg/kg	29	43							
	3	mg/kg	23	49	25.04.19	0,248	0,253844	0,2431308	0,02	no	
	4	mg/kg	13	59	09.05.19	0,266	0,269	0,264	keine	yes	80-120
	5	mg/kg	33	39		0,24	0,24	0,24	<0,01	no	
	6	mg/kg	22	50	26.04.	< 0,04	< 0,04	< 0,04	0,04	no	
	7	mg/kg	40	47	06.05.19	0,114	0,12	0,108	0,06	no	
	8	mg/kg	34	38	18.4.	0,251	0,252	0,249	0,007 µg/L	no	not done
	9	mg/kg	17	55	14. Mai	0,28	0,28	0,27	0,005	no	-
	10	mg/kg	12	60	06. Mai	0,277	0,279	0,275	0,03	no	n.a.
	11	mg/kg	11	61	8,05	0,299	0,3	0,3	0,1	no	-

Analyte	Partici-pant	Unit	Sample I DLA No.	Sample II DLA No.	Date of analysis	Result (Mean)	Result I	Result II	Limit of determination	Incl. RR	Recovery rate
					Day/Month					yes / no	in %
Rb-Rubidium	1	mg/kg	18	54	10. Apr	17,3744863128	17,426946	17,322027	0,01	no	95
	2	mg/kg	29	43							
	3	mg/kg	23	49	25. Apr	17,7	17,835838	17,559955	1	no	
	4	mg/kg	13	59							
	5	mg/kg	33	39							
	6	mg/kg	22	50							
	7	mg/kg	40	47							
	8	mg/kg	34	38	18.4.	17,27	17,30	17,23	0,038 µg/L	no	not done
	9	mg/kg	17	55	-	-	-	-	-	-	-
	10	mg/kg	12	60	not analyzed	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	11	mg/kg	11	61	8,05	20,2	20,1	20,2	0,3	no	-

Analyte	Partici-pant	Unit	Sample I DLA No.	Sample II DLA No.	Date of analysis	Result (Mean)	Result I	Result II	Limit of determination	Incl. RR	Recovery rate
					Day/Month					yes / no	in %
Se-Selen	1	mg/kg	18	54	10. Apr	0,5367621766	0,5367211	0,5368032	0,01	no	98
	2	mg/kg	29	43							
	3	mg/kg	23	49	25.04.19	0,582	0,5834701	0,5805439	0,06	no	
	4	mg/kg	13	59	09.05.19	0,539	0,543	0,535	keine	yes	80-120
	5	mg/kg	33	39		0,49	0,48	0,49	<0,10	no	
	6	mg/kg	22	50							
	7	mg/kg	40	47							
	8	mg/kg	34	38	18.4.	0,716	0,702	0,73	0,095 µg/L	no	not done
	9	mg/kg	17	55	14.05.19	0,49	0,49	0,49	0,05	no	-
	10	mg/kg	12	60	08.05.19	0,58	0,59	0,57	0,03	no	n.a.
	11	mg/kg	11	61	8,05	0,69	0,68	0,69	0,3	no	-

Analyte	Partici-pant	Unit	Sample I DLA No.	Sample II DLA No.	Date of analysis	Result (Mean)	Result I	Result II	Limit of determination	Incl. RR	Recovery rate
					Day/Month					yes / no	in %
Sn-Tin	1	mg/kg	18	54	10. Apr	1,4180859078	1,4366587	1,3995131	0,01	no	100
	2	mg/kg	29	43							
	3	mg/kg	23	49							
	4	mg/kg	13	59	09.05.19	1,515	1,438	1,592	keine	yes	80-120
	5	mg/kg	33	39		1,52	1,51	1,53	<0,10	no	
	6	mg/kg	22	50							
	7	mg/kg	40	47							
	8	mg/kg	34	38							
	9	mg/kg	17	55	14.05.19	1,5	1,5	1,4	0,05	no	-
	10	mg/kg	12	60	08.05.19	1,58	1,59	1,56	0,06	no	n.a.
	11	mg/kg	11	61	8,05	1,6	1,47	1,71	0,05	no	-

Analyte	Partici-pant	Unit	Sample I DLA No.	Sample II DLA No.	Date of analysis	Result (Mean)	Result I	Result II	Limit of determination	Incl. RR	Recovery rate
					Day/Month					yes / no	in %
U-Uranium	1	mg/kg	18	54	10. Apr	0,2784967045	0,2714933	0,2855001	0,01	no	100
	2	mg/kg	29	43							
	3	mg/kg	23	49	25.04.19	0,275	0,2773437	0,2718659	0,02	no	
	4	mg/kg	13	59	09.05.19	0,327	0,327	0,327	keine	yes	80-120
	5	mg/kg	33	39		0,3	0,3	0,3	<0,01	no	
	6	mg/kg	22	50							
	7	mg/kg	40	47							
	8	mg/kg	34	38	18.4.	0,282	0,285	0,279	0,7 ng/L	no	not done
	9	mg/kg	17	55	-	-			-	-	-
	10	mg/kg	12	60	not analyzed	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	11	mg/kg	11	61	8,05	0,34	0,33	0,34	0,05	no	-

Analyte	Partici-pant	Unit	Sample I DLA No.	Sample II DLA No.	Date of analysis	Result (Mean)	Result I	Result II	Limit of determination	Incl. RR	Recovery rate
					Day/Month					yes / no	in %
Zn-Zinc	1	mg/kg	18	54	10. Apr	11,3755517493	11,510069	11,241035	0,2	no	100
	2	mg/kg	29	43							
	3	mg/kg	23	49	25.04.19	14,11	14,250483	13,968466	1	no	
	4	mg/kg	13	59	09.05.19	14,208	14,076	14,34	keine	yes	80-120
	5	mg/kg	33	39		14,14	14,31	13,97	<0,50	no	
	6	mg/kg	22	50	23.05.	15,6	15,9	15,2	10	no	
	7	mg/kg	40	47							
	8	mg/kg	34	38	18.4.	12,32	12,20	12,44	0,23 µg/L	no	not done
	9	mg/kg	17	55	14.05.19	18	18	18	0,5	no	-
	10	mg/kg	12	60	06.05.19	14,4	14,5	14,3	0,9	no	n.a.
	11	mg/kg	11	61	8,05	16,3	15,9	16,6	0,5	no	-

5.1.2 Analytical Methods

Analyte	Particip ant	Method description as in test re-port / norm / literature	Sample preparation	Measuring method	Calibration / Reference material	Recovery rate with same matrix	Method accredited ISO/IEC 17025	Further Remarks
			Digestion: Method / Solution			yes / no	yes / no	
Al-Aluminium	1	§64 L00.00-19//1	Nitric acid digestion	ICP-MS	External calibration with Agilent Multi-element standard	no	yes	
	2							
	3	ICP-OES	pressure digestion (microwave); HNO3		external calibration		yes	
	4	Quantitative Analysis ICP-MS	microwave pressure digestion	Quantitative Analysis ICP-MS	external calibration and reference water	no	no	
	5	DIN EN ISO 11885 (E 22) (2009-09)	VDLUFA VII, 2.1.3 (2011)		yes		yes	
	6							
	7	DIN ISO 11885 (E22)	microwave pressure digestion	ICP-OES			yes	
	8	in-house method	microwave acid digestion withHNO3 uad H2O2 (4:1, v:v). sample weight 100 mg	Determination of sample 1 and 2 each 5 fold		no	no	Homogenization by stirring after filling into sample container
	9	ICP/MS - internal method PNTA0193			external calib. curve and internal RM	no	yes	
	10	Al with ICP-MS after microwave digestion	mixing	ICP-MS	ext. with IS In	n.a.	yes	n.a.
	11	ICP-ORG	microwave digestion / acid	ICP-OES	Element standard solution	-	yes	

Analyte	Participant	Method description as in test report / norm / literature	Sample preparation	Measuring method	Calibration / Reference material	Recovery rate with same matrix	Method accredited ISO/IEC 17025	Further Remarks
			Digestion: Method / Solution			yes / no	yes / no	
B-Boron	1	§64 L00.00-19//1	Nitric acid digestion	ICP-MS	External calibration with Agilent Multi-element standard	no	yes	
	2							
	3	ICP-OES	pressure digestion (microwave); HNO3		external calibration		yes	
	4	Quantitative Analysis ICP-MS	microwave pressure digestion	Quantitative Analysis ICP-MS	external calibration and reference water	no	no	
	5	DIN EN ISO 11885 (E 22) (2009-09)	VDLUFA VII, 2.1.3 (2011)		yes		yes	
	6							
	7							
	8	in-house method	microwave acid digestion withHNO3 uad H2O2 (4:1, v:v). sample weight 100 mg	Determination of sample 1 and 2 each 5 fold		no	no	Homogenization by stirring after filling into sample container
	9	ICP/MS - internal method PNTA0193			external calib. curve and internal RM	no	yes	
	10	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	11	ICP-ORG	microwave digestion / acid	ICP-OES	Element standard solution	-	yes	

Analyte	Participant	Method description as in test report / norm / literature	Sample preparation	Measuring method	Calibration / Reference material	Recovery rate with same matrix	Method accredited ISO/IEC 17025	Further Remarks
			Digestion: Method / Solution			yes / no	yes / no	
Ba-Barium	1	§64 L00.00-19//1	Nitric acid digestion	ICP-MS	External calibration with Agilent Multi-element standard	no	yes	
	2							
	3	ICP-OES	pressure digestion (microwave); HNO3		external calibration		yes	
	4	Quantitative Analysis ICP-MS	microwave pressure digestion	Quantitative Analysis ICP-MS	external calibration and reference water	no	no	
	5	DIN EN ISO 11885 (E 22) (2009-09)	VDLUFA VII, 2.1.3 (2011)		yes		yes	
	6							
	7							
	8	in-house method	microwave acid digestion withHNO3 uad H2O2 (4:1, v:v). sample weight 100 mg	Determination of sample 1 and 2 each 5 fold		no	no	Homogenization by stirring after filling into sample container
	9	ICP/MS - internal method PNTA0193			external calib. curve and internal RM	no	yes	
	10	Ba with ICP-MS after microwave digestion	mixing	ICP-MS	ext. with IS In	n.a.	yes	n.a.
	11	ICP-ORG	microwave digestion / acid	ICP-OES	Element standard solution	-	yes	

Analyte	Participant	Method description as in test report / norm / literature	Sample preparation	Measuring method	Calibration / Reference material	Recovery rate with same matrix	Method accredited ISO/IEC 17025	Further Remarks
			Digestion: Method / Solution			yes / no	yes / no	
Ca-Calcium	1	§64 L00.00-19//1	Nitric acid digestion	ICP-MS	External calibration with Agilent Multi-element standard	no	yes	
	2							
	3	ICP-OES	pressure digestion (microwave); HNO3		external calibration		yes	
	4	Quantitative Analysis ICP-MS	microwave pressure digestion	Quantitative Analysis ICP-MS	external calibration and reference water	no	no	
	5	DIN EN ISO 11885 (E 22) (2009-09)	VDLUFA VII, 2.1.3 (2011)		yes		yes	
	6	ASU L 31.00-10: 1997-01	microwave / conc. HNO3	Flame-AAS			yes	
	7	DIN ISO 11885 (E22)	microwave pressure digestion	ICP-OES			yes	
	8							
	9	ICP/MS - internal method PNTA0193			external calib. curve and internal RM	no	yes	
	10	Ca with ICP-MS after microwave digestion	mixing	ICP-MS	ext. with IS In	n.a.	yes	n.a.
	11	ICP-ORG	microwave digestion / acid	ICP-OES	Element standard solution	-	yes	

Analyte	Participant	Method description as in test report / norm / literature	Sample preparation	Measuring method	Calibration / Reference material	Recovery rate with same matrix	Method accredited ISO/IEC 17025	Further Remarks
			Digestion: Method / Solution			yes / no	yes / no	
Cu-Copper	1	§64 L00.00-19//1	Nitric acid digestion	ICP-MS	External calibration with Agilent Multi-element standard	no	yes	
	2							
	3	ICP-MS (Quadrupol)	pressure digestion (microwave); HNO3		external calibration		yes	
	4	Quantitative Analysis ICP-MS	microwave pressure digestion	Quantitative Analysis ICP-MS	external calibration and reference water	no	no	
	5	DIN EN ISO 11885 (E 22) (2009-09)	VDLUFA VII, 2.1.3 (2011)		yes		yes	
	6							
	7	DIN ISO 11885 (E22)	microwave pressure digestion	ICP-OES			yes	
	8	in-house method	microwave acid digestion withHNO3 uad H2O2 (4:1, v:v). sample weight 100 mg	Determination of sample 1 and 2 each 5 fold		no	no	Homogenization by stirring after filling into sample container
	9	ICP/MS - internal method PNTA0193			external calib. curve and internal RM	no	yes	
	10	Cu with ICP-MS after microwave digestion	mixing	ICP-MS	ext. with IS In	n.a.	yes	n.a.
	11	IMS-ORG	microwave digestion / acid	ICP-MS	Element standard solution	-	yes	

Analyte	Participant	Method description as in test report / norm / literature	Sample preparation	Measuring method	Calibration / Reference material	Recovery rate with same matrix	Method accredited ISO/IEC 17025	Further Remarks
			Digestion: Method / Solution			yes / no	yes / no	
Fe-Iron	1	§64 L00.00-19//1	Nitric acid digestion	ICP-MS	External calibration with Agilent Multi-element standard	no	yes	
	2							
	3	ICP-OES	pressure digestion (microwave); HNO3		external calibration		yes	
	4	Quantitative Analysis ICP-MS	microwave pressure digestion	Quantitative Analysis ICP-MS	external calibration and reference water	no	no	
	5	DIN EN ISO 11885 (E 22) (2009-09)	VDLUFA VII, 2.1.3 (2011)		yes		yes	
	6	ASU L 00.00-19/2: 1993-08	microwave / conc. HNO3	Flame-AAS			yes	
	7	DIN ISO 11885 (E22)	microwave pressure digestion	ICP-OES			yes	
	8	in-house method	microwave acid digestion withHNO3 uad H2O2 (4:1, v:v). sample weight 100 mg	Determination of sample 1 and 2 each 5 fold		no	no	Homogenization by stirring after filling into sample container
	9	ICP/MS - internal method PNTA0193			external calib. curve and internal RM	no	yes	
	10	Fe with ICP-MS after microwave digestion	mixing	ICP-MS	ext. with IS In	n.a.	yes	n.a.
	11	ICP-ORG	microwave digestion / acid	ICP-OES	Element standard solution	-	yes	

Analyte	Participant	Method description as in test report / norm / literature	Sample preparation	Measuring method	Calibration / Reference material	Recovery rate with same matrix	Method accredited ISO/IEC 17025	Further Remarks
			Digestion: Method / Solution			yes / no	yes / no	
I-Iodine	1	§64 L00.00-19//1	Nitric acid digestion	ICP-MS	External calibration with Agilent Multi-element standard	no	yes	
	2							
	3	ICP-MS (Quadrupol)	alkaline digestion (TMAH)		external calibration		yes	
	4							not measured
	5	DIN EN 15111 (2007-06)			yes		yes	
	6							
	7							
	8							
	9							
	10	Iodine ICP-MS, DIN/EN 15111, mod.	mixing	ICP-MS	ext. with IS In	n.a.	yes	n.a.
	11	ICP-ORG	microwave digestion / acid	ICP-OES	Element standard solution	-	yes	

Analyte	Participant	Method description as in test report / norm / literature	Sample preparation	Measuring method	Calibration / Reference material	Recovery rate with same matrix	Method accredited ISO/IEC 17025	Further Remarks
			Digestion: Method / Solution			yes / no	yes / no	
K-Potassium	1	§64 L00.00-19//1	Nitric acid digestion	ICP-MS	External calibration with Agilent Multi-element standard	no	yes	
	2							
	3	ICP-OES	pressure digestion (microwave); HNO3		external calibration		yes	
	4	Quantitative Analysis ICP-MS	microwave pressure digestion	Quantitative Analysis ICP-MS	external calibration and reference water	no	no	
	5	DIN EN ISO 11885 (E 22) (2009-09)	VDLUFA VII, 2.1.3 (2011)		yes		yes	
	6	ASU L 31.00-10: 1997-01	microwave / conc. HNO3	Flame-AAS			yes	
	7	DIN ISO 11885 (E22)	microwave pressure digestion	ICP-OES			yes	
	8							
	9	ICP/MS - internal method PNTA0193			external calib. curve and internal RM	no	yes	
	10	K with ICP-MS after microwave digestion	mixing	ICP-MS	ext. with IS In	n.a.	yes	n.a.
	11	ICP-ORG	microwave digestion / acid	ICP-OES	Element standard solution	-	yes	

Analyte	Participant	Method description as in test report / norm / literature	Sample preparation	Measuring method	Calibration / Reference material	Recovery rate with same matrix	Method accredited ISO/IEC 17025	Further Remarks
			Digestion: Method / Solution			yes / no	yes / no	
Mg-Magnesium	1	§64 L00.00-19//1	Nitric acid digestion	ICP-MS	External calibration with Agilent Multi-element standard	no	yes	
	2							
	3	ICP-OES	pressure digestion (microwave); HNO3		external calibration		yes	
	4	Quantitative Analysis ICP-MS	microwave pressure digestion	Quantitative Analysis ICP-MS	external calibration and reference water	no	no	
	5	DIN EN ISO 11885 (E 22) (2009-09)	VDLUFA VII, 2.1.3 (2011)		yes		yes	
	6	ASU L 31.00-10: 1997-01	microwave / conc. HNO3	Flame-AAS			yes	
	7							
	8							
	9	ICP/MS - internal method PNTA0193			external calib. curve and internal RM	no	yes	
	10	Mg with ICP-MS after microwave digestion	mixing	ICP-MS	ext. with IS In	n.a.	yes	n.a.
	11	ICP-ORG	microwave digestion / acid	ICP-OES	Element standard solution	-	yes	

Analyte	Participant	Method description as in test report / norm / literature	Sample preparation	Measuring method	Calibration / Reference material	Recovery rate with same matrix	Method accredited ISO/IEC 17025	Further Remarks
			Digestion: Method / Solution			yes / no	yes / no	
Mn-Manganese	1	§64 L00.00-19//1	Nitric acid digestion	ICP-MS	External calibration with Agilent Multi-element standard	no	yes	
	2	ASU L 00.00-144					yes	
	3	ICP-OES	pressure digestion (microwave); HNO3		external calibration		yes	
	4	Quantitative Analysis ICP-MS	microwave pressure digestion	Quantitative Analysis ICP-MS	external calibration and reference water	no	no	
	5	DIN EN ISO 11885 (E 22) (2009-09)	VDLUFA VII, 2.1.3 (2011)		yes		yes	
	6							
	7	DIN ISO 11885 (E22)	microwave pressure digestion	ICP-OES			yes	
	8	in-house method	microwave acid digestion withHNO3 uad H2O2 (4:1, v:v). sample weight 100 mg	Determination of sample 1 and 2 each 5 fold		no	no	Homogenization by stirring after filling into sample container
	9	ICP/MS - internal method PNTA0193			external calib. curve and internal RM	no	yes	
	10	Mn with ICP-MS after microwave digestion	mixing	ICP-MS	ext. with IS In	n.a.	yes	n.a.
	11	ICP-ORG	microwave digestion / acid	ICP-OES	Element standard solution	-	yes	

Analyte	Participant	Method description as in test report / norm / literature	Sample preparation	Measuring method	Calibration / Reference material	Recovery rate with same matrix	Method accredited ISO/IEC 17025	Further Remarks
			Digestion: Method / Solution			yes / no	yes / no	
Mo-Molybdenum	1	§64 L00.00-19//1	Nitric acid digestion	ICP-MS	External calibration with Agilent Multi-element standard	no	yes	
	2							
	3	ICP-MS (Quadrupol)	pressure digestion (microwave); HNO3		external calibration		yes	
	4	Quantitative Analysis ICP-MS	microwave pressure digestion	Quantitative Analysis ICP-MS	external calibration and reference water	no	no	
	5	DIN EN ISO 11885 (E 22) (2009-09)	VDLUFA VII, 2.1.3 (2011)		yes		yes	
	6							
	7							
	8	in-house method	microwave acid digestion withHNO3 uad H2O2 (4:1, v:v). sample weight 100 mg	Determination of sample 1 and 2 each 5 fold		no	no	Homogenization by stirring after filling into sample container
	9	ICP/MS - internal method PNTA0193			external calib. curve and internal RM	no	yes	
	10	Mo with ICP-MS after microwave digestion	mixing	ICP-MS	ext. with IS In	n.a.	yes	n.a.
	11	IMS-ORG	microwave digestion / acid	ICP-MS	Element standard solution	-	yes	

Analyte	Participant	Method description as in test report / norm / literature	Sample preparation	Measuring method	Calibration / Reference material	Recovery rate with same matrix	Method accredited ISO/IEC 17025	Further Remarks
			Digestion: Method / Solution			yes / no	yes / no	
Na-Sodium	1	§64 L00.00-19//1	Nitric acid digestion	ICP-MS	External calibration with Agilent Multi-element standard	no	yes	
	2							
	3	ICP-OES	pressure digestion (microwave); HNO3		external calibration		yes	
	4	Quantitative Analysis ICP-MS	microwave pressure digestion	Quantitative Analysis ICP-MS	external calibration and reference water	no	no	
	5	DIN EN ISO 11885 (E 22) (2009-09)	VDLUFA VII, 2.1.3 (2011)		yes		yes	
	6	ASU L 31.00-10: 1997-01	microwave / conc. HNO3	GF-AAS			ja	
	7							
	8							
	9	ICP/MS - internal method PNTA0193			external calib. curve and internal RM	no	yes	
	10	Na with ICP-MS after microwave digestion	mixing	ICP-MS	ext. with IS In	n.a.	yes	n.a.
	11	ICP-ORG	microwave digestion / acid	ICP-OES	Element standard solution	-	yes	

Analyte	Participant	Method description as in test report / norm / literature	Sample preparation	Measuring method	Calibration / Reference material	Recovery rate with same matrix	Method accredited ISO/IEC 17025	Further Remarks
			Digestion: Method / Solution			yes / no	yes / no	
Ni-Nickel	1	§64 L00.00-19//1	Nitric acid digestion	ICP-MS	External calibration with Agilent Multi-element standard	no	yes	
	2							
	3	ICP-MS (Quadrupol)	pressure digestion (microwave); HNO3		external calibration		yes	
	4	Quantitative Analysis ICP-MS	microwave pressure digestion	Quantitative Analysis ICP-MS	external calibration and reference water	no	no	
	5	DIN EN ISO 17294-2 (E 29) (2005-02)	VDLUFA VII, 2.1.3 (2011)		yes		yes	
	6	ASU L 59.11-3: 2000-07 (on the basis)	Mikrowelle / konz. HNO3	GF-AAS			nein	
	7	DIN ISO 11885 (E22)	microwave pressure digestion	ICP-OES			yes	
	8	in-house method	microwave acid digestion withHNO3 uad H2O2 (4:1, v:v). sample weight 100 mg	Determination of sample 1 and 2 each 5 fold		no	no	Homogenization by stirring after filling into sample container
	9	ICP/MS - internal method PNTA0193			external calib. curve and internal RM	no	yes	
	10	Ni with ICP-MS after microwave digestion	mixing	ICP-MS	ext. with IS In	n.a.	yes	n.a.
	11	IMS-ORG	microwave digestion / acid	ICP-MS	Element standard solution	-	yes	

Analyte	Participant	Method description as in test report / norm / literature	Sample preparation	Measuring method	Calibration / Reference material	Recovery rate with same matrix	Method accredited ISO/IEC 17025	Further Remarks
			Digestion: Method / Solution			yes / no	yes / no	
P-Phosphorus	1							
	2							
	3	ICP-OES	pressure digestion (microwave); HNO3		external calibration		yes	
	4	Quantitative Analysis ICP-MS	microwave pressure digestion	Quantitative Analysis ICP-MS	external calibration and reference water	no	no	
	5	DIN EN ISO 11885 (E 22) (2009-09)	VDLUFA VII, 2.1.3 (2011)		yes		yes	
	6							
	7							
	8							
	9	ICP/MS - internal method PNTA0193			external calib. curve and internal RM	no	yes	
	10	P with ICP-MS after microwave digestion	mixing	ICP-MS	ext. with IS In	n.a.	yes	n.a.
	11	ICP-ORG	microwave digestion / acid	ICP-OES	Element standard solution	-	yes	

Analyte	Participant	Method description as in test report / norm / literature	Sample preparation	Measuring method	Calibration / Reference material	Recovery rate with same matrix	Method accredited ISO/IEC 17025	Further Remarks
			Digestion: Method / Solution			yes / no	yes / no	
Pb-Lead	1	§64 L00.00-19//1	Nitric acid digestion	ICP-MS	External calibration with Agilent Multi-element standard	no	yes	
	2							
	3	ICP-MS (Quadrupol)	pressure digestion (microwave); HNO3		external calibration		yes	
	4	Quantitative Analysis ICP-MS	microwave pressure digestion	Quantitative Analysis ICP-MS	external calibration and reference water	no	no	
	5	DIN EN ISO 11885 (E 22) (2009-09)	VDLUFA VII, 2.1.3 (2011)		yes		yes	
	6	ASU L 00.00-19/3: 2004-07	microwave / conc. HNO3	GF-AAS			yes	
	7	DIN ISO 11885 (E22)	microwave pressure digestion	ICP-OES			yes	
	8	in-house method	microwave acid digestion withHNO3 uad H2O2 (4:1, v:v). sample weight 100 mg	Determination of sample 1 and 2 each 5 fold		no	no	Homogenization by stirring after filling into sample container
	9	ICP/MS - internal method PNTA0193			external calib. curve and internal RM	no	yes	
	10	Pb with ICP-MS after microwave digestion	mixing	ICP-MS	ext. with IS In	n.a.	yes	n.a.
	11	IMS-ORG	microwave digestion / acid	ICP-MS	Element standard solution	-	yes	

Analyte	Participant	Method description as in test report / norm / literature	Sample preparation	Measuring method	Calibration / Reference material	Recovery rate with same matrix	Method accredited ISO/IEC 17025	Further Remarks
			Digestion: Method / Solution			yes / no	yes / no	
Rb-Rubidium	1	§64 L00.00-19//1	Nitric acid digestion	ICP-MS	External calibration with Agilent Multi-element standard	no	yes	
	2							
	3	ICP-MS (Quadrupol)	pressure digestion (microwave); HNO3		external calibration		yes	
	4							not measured
	5							
	6							
	7							
	8	in-house method	microwave acid digestion withHNO3 uad H2O2 (4:1, v:v). sample weight 100 mg	Determination of sample 1 and 2 each 5 fold		no	no	Homogenization by stirring after filling into sample container
	9	-			-	-	-	
	10	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	11	IMS-ORG	microwave digestion / acid	ICP-MS	Element standard solution	-	yes	

Analyte	Participant	Method description as in test report / norm / literature	Sample preparation	Measuring method	Calibration / Reference material	Recovery rate with same matrix	Method accredited ISO/IEC 17025	Further Remarks
			Digestion: Method / Solution			yes / no	yes / no	
Se-Selenium	1	§64 L00.00-19//1	Nitric acid digestion	ICP-MS	External calibration with Agilent Multi-element standard	no	yes	
	2							
	3	ICP-MS (Quadrupol)	pressure digestion (microwave); HNO3		external calibration		yes	
	4	Quantitative Analysis ICP-MS	microwave pressure digestion	Quantitative Analysis ICP-MS	external calibration and reference water	no	no	
	5	DIN 38405-D 23 (1994-10)	VDLUFA VII, 2.1.3 (2011)		yes		yes	
	6							
	7							
	8	in-house method	microwave acid digestion withHNO3 uad H2O2 (4:1, v:v). sample weight 100 mg	Determination of sample 1 and 2 each 5 fold		no	no	Homogenization by stirring after filling into sample container
	9	ICP/MS - internal method PNTA0193			external calib. curve and internal RM	no	yes	
	10	Se with ICP-MS after microwave digestion	mixing	ICP-MS	ext. with IS In	n.a.	yes	n.a.
	11	IMS-ORG	microwave digestion / acid	ICP-MS	Element standard solution	-	yes	

Analyte	Participant	Method description as in test report / norm / literature	Sample preparation	Measuring method	Calibration / Reference material	Recovery rate with same matrix	Method accredited ISO/IEC 17025	Further Remarks
			Digestion: Method / Solution			yes / no	yes / no	
Sn-Zinn	1	§64 L00.00-19//1	Nitric acid digestion	ICP-MS	External calibration with Agilent Multi-element standard	no	yes	
	2							
	3							
	4	Quantitative Analysis ICP-MS	microwave pressure digestion	Quantitative Analysis ICP-MS	external calibration and reference water	no	no	
	5	DIN EN ISO 17294-2 (E 29) (2005-02)	VDLUFA VII, 2.1.3 (2011)		yes		yes	
	6							
	7							
	8							
	9	ICP/MS - internal method PNTA0193			external calib. curve and internal RM	no	yes	
	10	Sn with ICP-MS after microwave digestion	mixing	ICP-MS	ext. with IS In	n.a.	yes	n.a.
	11	IMS-ORG	microwave digestion / acid	ICP-MS	Element standard solution	-	yes	

Analyte	Participant	Method description as in test report / norm / literature	Sample preparation	Measuring method	Calibration / Reference material	Recovery rate with same matrix	Method accredited ISO/IEC 17025	Further Remarks
			Digestion: Method / Solution			yes / no	yes / no	
U-Uranium	1	§64 L00.00-19//1	Nitric acid digestion	ICP-MS	External calibration with Agilent Multi-element standard	no	yes	
	2							
	3	ICP-MS (Quadrupol)	pressure digestion (microwave); HNO3		external calibration		yes	
	4	Quantitative Analysis ICP-MS	microwave pressure digestion	Quantitative Analysis ICP-MS	external calibration and reference water	no	no	
	5	DIN EN ISO 11885 (E 22) (2009-09)	VDLUFA VII, 2.1.3 (2011)		yes		yes	
	6							
	7							
	8	in-house method	microwave acid digestion withHNO3 uad H2O2 (4:1, v:v). sample weight 100 mg	Determination of sample 1 and 2 each 5 fold		no	no	Homogenization by stirring after filling into sample container
	9	-			-	-	-	
	10	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	11	IMS-ORG	microwave digestion / acid	ICP-MS	Element standard solution	-	yes	

Analyte	Participant	Method description as in test report / norm / literature	Sample preparation	Measuring method	Calibration / Reference material	Recovery rate with same matrix	Method accredited ISO/IEC 17025	Further Remarks
			Digestion: Method / Solution			yes / no	yes / no	
Zn-Zinc	1	§64 L00.00-19//1	Nitric acid digestion	ICP-MS	External calibration with Agilent Multi-element standard	no	yes	
	2							
	3	ICP-MS (Quadrupol)	pressure digestion (microwave); HNO3		external calibration		yes	
	4	Quantitative Analysis ICP-MS	microwave pressure digestion	Quantitative Analysis ICP-MS	external calibration and reference water	no	no	
	5	DIN EN ISO 11885 (E 22) (2009-09)	VDLUFA VII, 2.1.3 (2011)		yes		yes	
	6	ASU L 00.00-19/2: 1993-08	microwave / conc. HNO3	GF-AAS			yes	
	7							
	8	in-house method	microwave acid digestion withHNO3 uad H2O2 (4:1, v:v). sample weight 100 mg	Determination of sample 1 and 2 each 5 fold		no	no	Homogenization by stirring after filling into sample container
	9	ICP/MS - internal method PNTA0193			external calib. curve and internal RM	no	yes	
	10	Zn with ICP-MS after microwave digestion	mixing	ICP-MS	ext. with IS In	n.a.	yes	n.a.
	11	IMS-ORG	microwave digestion / acid	ICP-MS	Element standard solution	-	yes	

5.2 Homogeneity

5.2.1 Homogeneity of bottled PT-samples

Homogeneity test of copper and uranium by ICP-MS (EN ISO 17294-2):

Copper

Independant Samples	mg/kg
1	4,7
2	4,8
3	4,8
4	4,7
5	4,8
6	4,8
7	4,8
8	4,8

General Mean	4,78	
Repeatability standard deviation	0,0463	0,97%

Uranium

Independant Samples	mg/kg
1	0,31
2	0,31
3	0,32
4	0,34
5	0,31
6	0,31
7	0,30
8	0,31

General Mean	0,314	
Repeatability standard deviation	0,0119	3,8%

5.2.2 Trend line function of the participants results

By comparison of the increasing sample numbers and the measurement results of participants, the homogeneity of the chronological bottled PT items can be shown by the trend line for information:

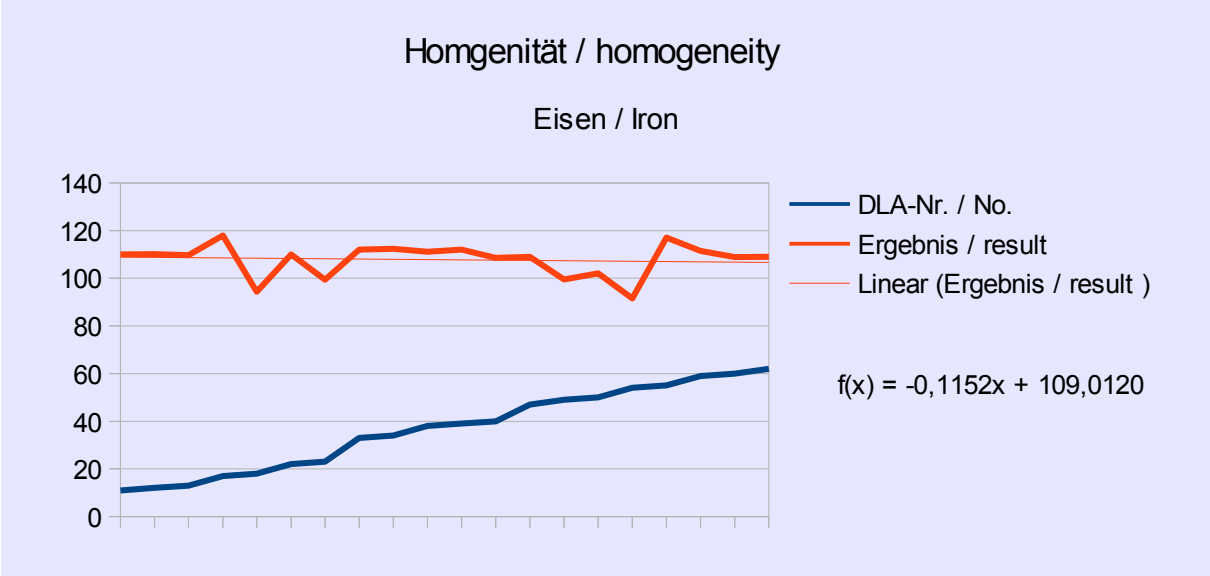


Abb./Fig. 39:
Trendfunktion Probennummern vs. Ergebnisse
trend line function sample number vs. results

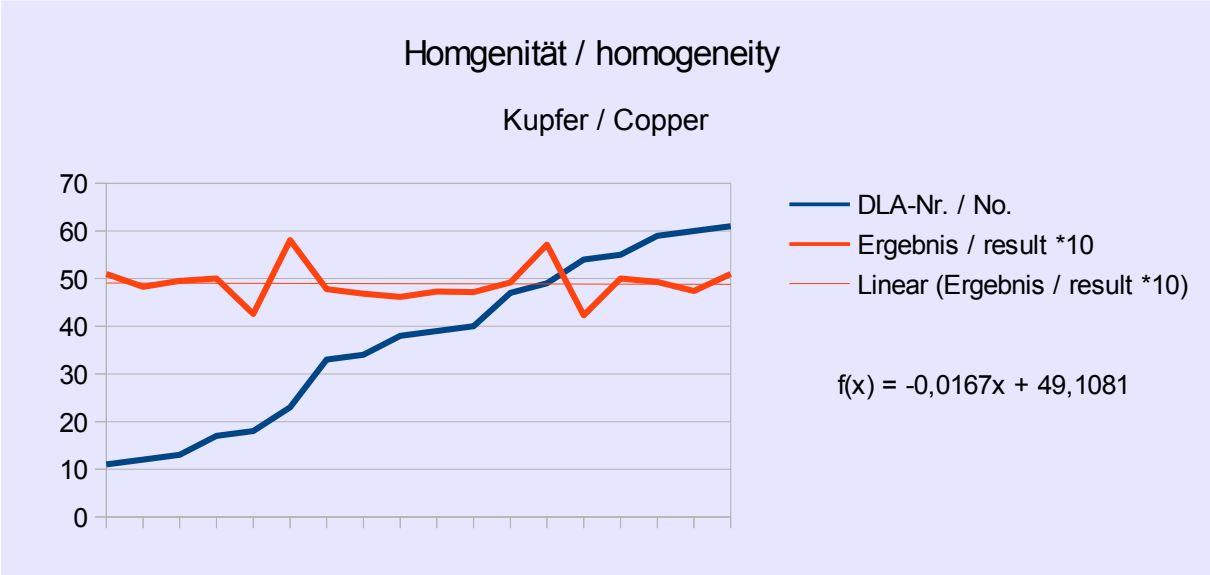


Abb./Fig. 40:
Trendfunktion Probennummern vs. Ergebnisse (1*10 dargestellt)
trend line function sample number vs. results (1*10 shown)

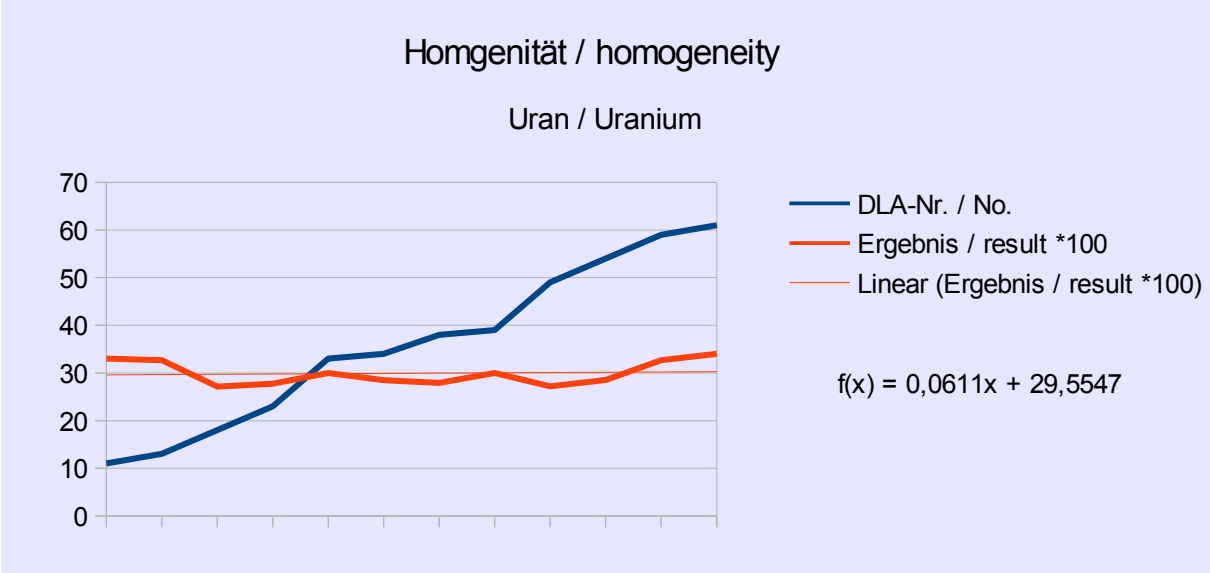


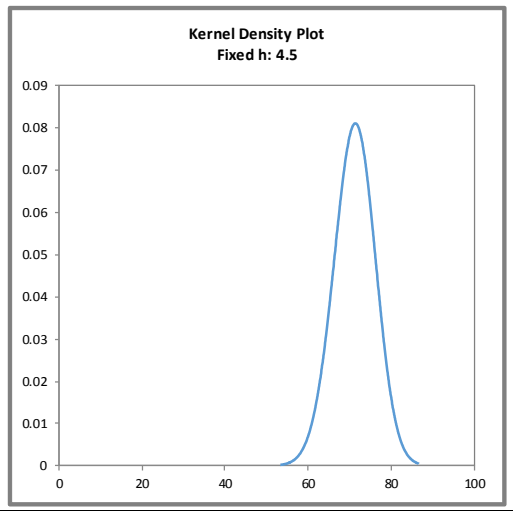
Abb./Fig. 41:
Trendfunktion Probennummern vs. Ergebnisse (1*100 dargestellt)
trend line function sample number vs. results (1*100 shown)

5.3 Kernel Density Plots of Results

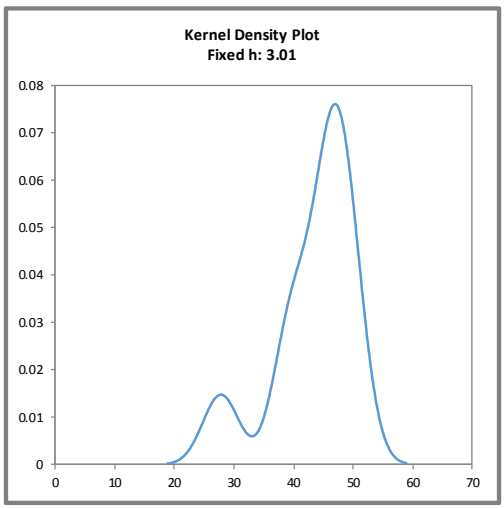
Abbildungen:
Kerndichte-Schätzungen
der Teilnehmerergebnisse
(mit h = 0,75 x σ_{pt} von Xpt)

Figures:
Kernel density plots
of participants' results
(with h = 0,75 x σ_{pt} of Xpt)

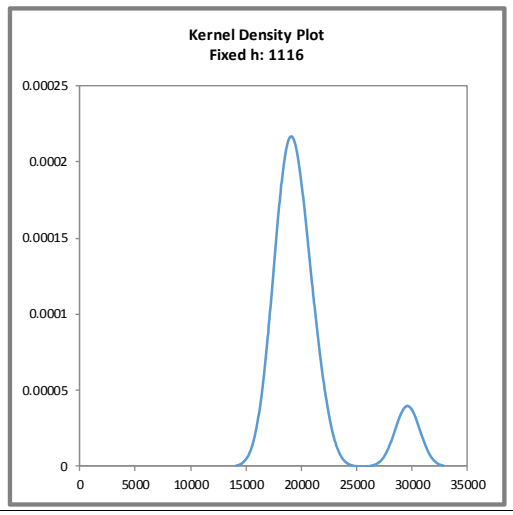
Barium



Aluminium



Calcium

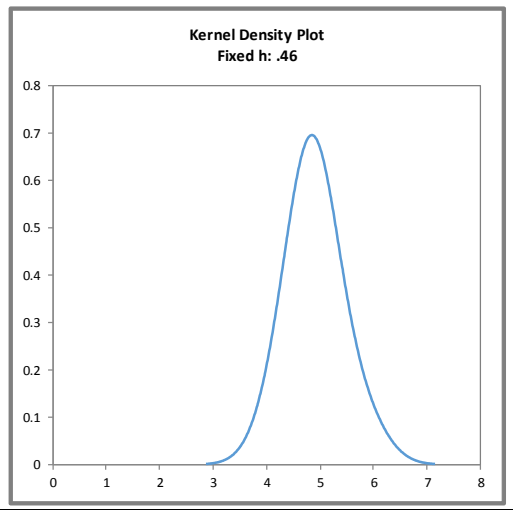


Bor / Boron

< 8 Ergebnisse

< 8 Results

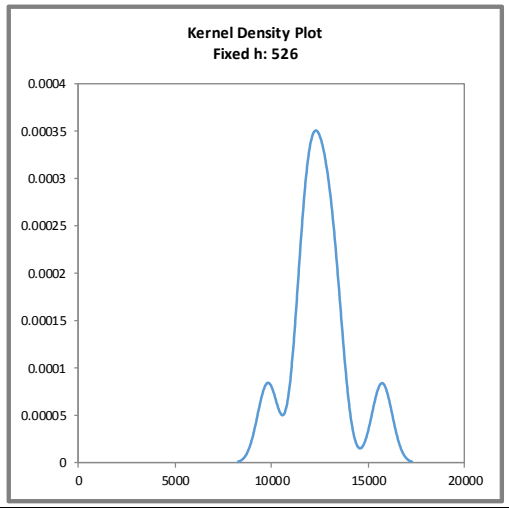
Kupfer / Copper



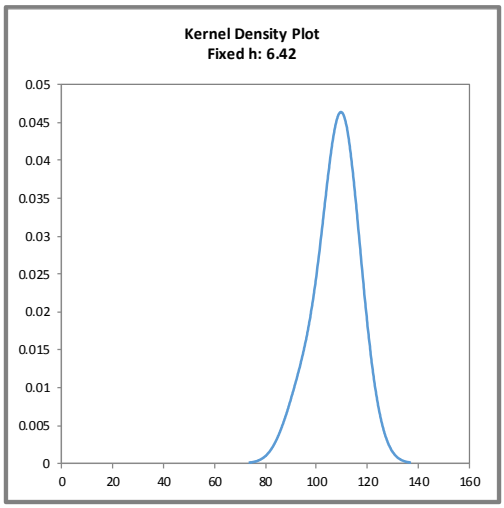
Abbildungen:
Kerndichte-Schätzungen
der Teilnehmerergebnisse
(mit h = 0,75 x σpt von Xpt)

Figures:
Kernel density plots
of participants' results
(with h = 0,75 x σpt of Xpt)

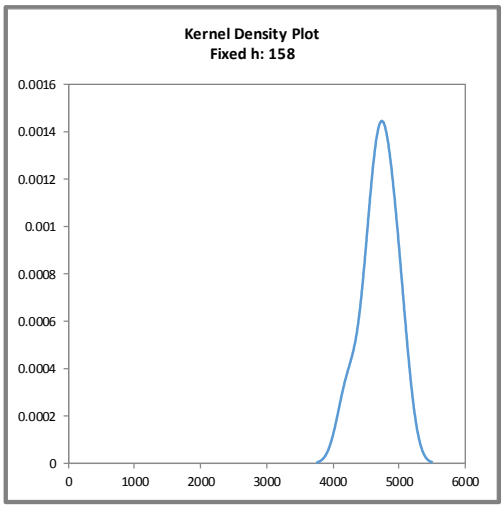
Kalium / Potassium



Eisen / Iron



Magnesium

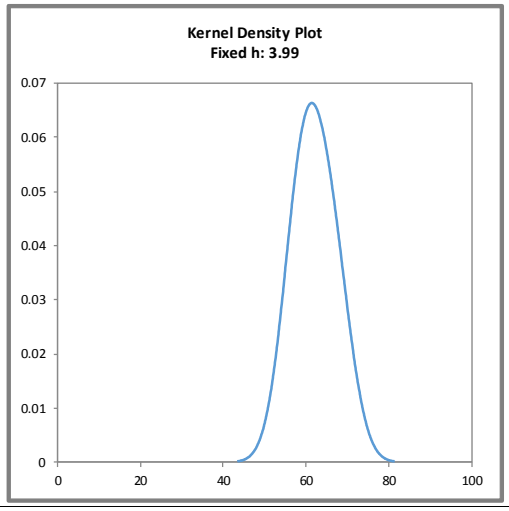


Iod / Iodine

< 8 Ergebnisse

< 8 Results

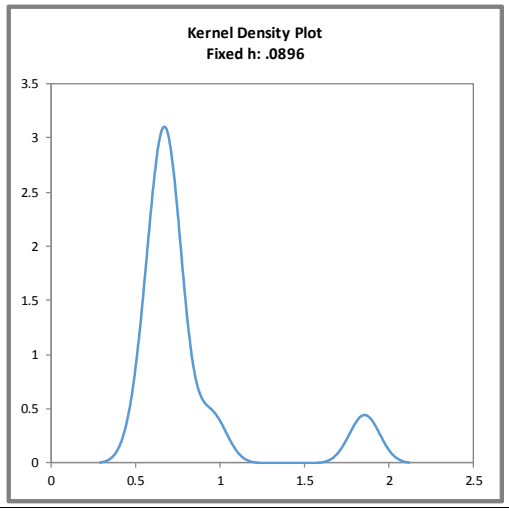
Mangan / Manganese



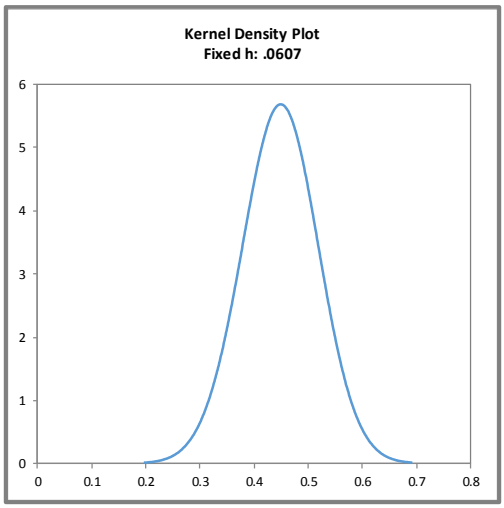
Abbildungen:
Kerndichte-Schätzungen
der Teilnehmerergebnisse
(mit h = 0,75 x σpt von Xpt)

Figures:
Kernel density plots
of participants' results
(with h = 0,75 x σpt of Xpt)

Nickel



Molybdän / Molybdenum

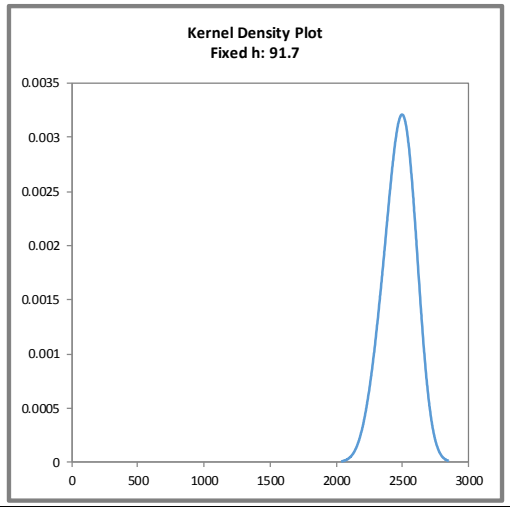


Phosphor / Phosphorus

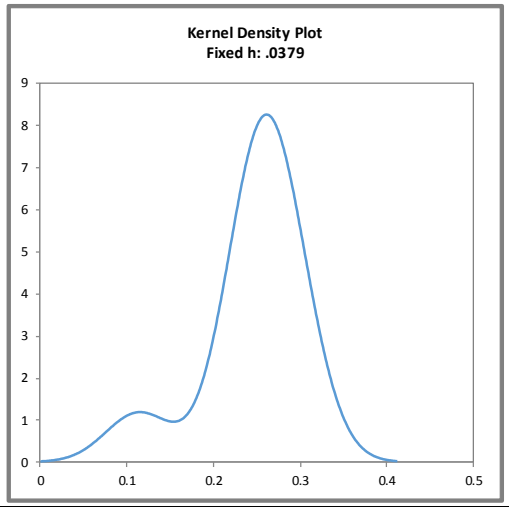
< 8 Ergebnisse

< 8 Results

Natrium / Sodium



Blei / Lead



Abbildungen:
Kerndichte-Schätzungen
der Teilnehmerergebnisse
(mit h = 0,75 x σ_{pt} von X_{pt})

Figures:
Kernel density plots
of participants' results
(with h = 0,75 x σ_{pt} of X_{pt})

Zinn / Tin

< 8 Ergebnisse

< 8 Results

Rubidium

< 8 Ergebnisse

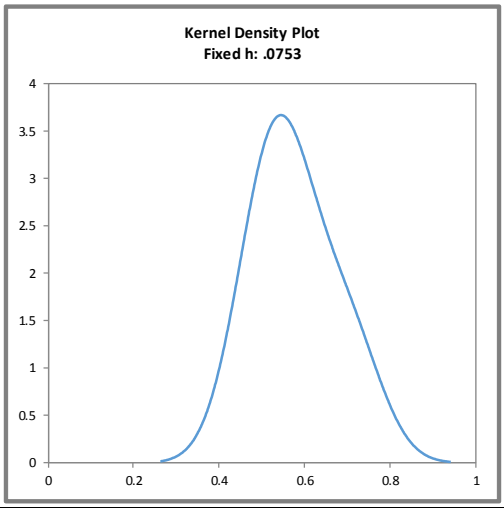
< 8 Results

Uran / Uranium

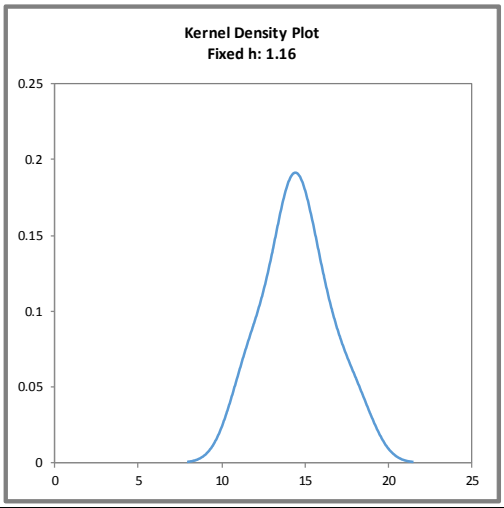
< 8 Ergebnisse

< 8 Results

Selen / Selenium



Zink / Zinc



5.4 Information on the Proficiency Test (PT)

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

PT number	***DLA 49 -2019***
PT name	***Heavy Metals and Trace Elements in plant food , approx. 20 Elements***
*Sample matrix**	*Samples I + II:* ***Moringa Leaf Powder***
Number of samples and sample amount	*2 identical samples I + II, 8 g each.*
Storage	*Samples I + II: room temperature*
Intentional use	*Laboratory use only (quality control samples)*
Parameter	*quantitative:* ***Al, B, Ba, Ca, Cu, Fe, I, K, Mg, Mn, Mo, Na, Ni, P, Pb, Rb, Se, Sn, U*** *and* ***Zn*** *(natural contents, exept Sn and U: spiked)*
Methods of analysis	*Analytical methods are optional*
Notes to analysis	*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.*
Result sheet	*The results for sample I and II as well as the final results calculated as mean of the double determination (samples I and II) should be filled in the result submission file. The recovery rates, if carried out, has to be included in the calculation.*
Units	*mg/kg*
Number of significant digits	*at least 2*
Further information	*For information please specify:* – *Date of analysis* – *DLA-sample-numbers (for sample I and II)* – *Limit of detection* – *Assignment incl. Recovery* – *Recovery with the same matrix* – *Method is accredited*
Result submission	*The result submission file should be sent by e-mail to:* ***pt@dla-lvu.de***
Deadline	***the latest 24th May 2019***
Evaluation report	*The evaluation report is expected to be completed 6 weeks after deadline of result submission and sent as PDF file by e-mail.*
Coordinator and contact person of PT	*Matthias Besler-Scharf PhD / Alexandra.Scharf MSc.*

* Control of mixture homogeneity and qualitative testings are carried out by DLA. Any 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
		Germany
		Germany
		Germany
		Germany
		Germany
		SWITZERLAND
		Germany
		Germany
		SPAIN
		SWITZERLAND

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

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

7. Index of references

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 Ananlytical Laboratories ; J.AOAC Int., 76(4), 926 – 940 (1993)
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10. Recent trends in inter-laboratory precision at ppb and sub-ppb concentrations in relation to fitness for purpose criteria in proficiency testing; M. Thompson; Analyst, 125, 385-386 (2000)
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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. Homogeneity and stability of reference materials; Linsinger et al.; Accred Qual Assur, 6, 20-25 (2001)
17. AOAC Official Methods of Analysis: Guidelines for Standard Method Performance Requirements, Appendix F, p. 2, AOAC Int (2016)
18. ASU §64 L 00.00-157 (2016-2): Bestimmung von Aluminium in Lebensmitteln mit der Massenspektrometrie mit induktiv gekoppeltem Plasma (ICP-MS) [Determination of aluminium in foods by inductively coupled plasma mass spectrometry (ICPMS) after pressure digestion]
19. ASU §64 L 00.00-158 (2016-2): Bestimmung von Aluminium in Lebensmitteln mit der optischen Emmissionsspektrometrie mit induktiv gekoppeltem Plasma (ICP-OES) [Determination of aluminium in foods by inductively coupled plasma emmission spectrometry (ICP-OES) after pressure digestion]
20. 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
21. ASU §64 L 00.00-19/2: Bestimmung von Eisen, Kupfer, Mangan und Zink mit der Atomabsorptionsspektrometrie (AAS) in der Flamme [Determination of iron, copper, manganese and zinc by atomic absorption spectrometry (AAS) in the flame]
22. ASU §64 L 00.00-19/3 / DIN EN 14083: Bestimmung von Blei, Cadmium, Chrom und Mo-

lybdän mit Graphitofen-Atomabsorptionsspektrometrie (GFAAS) nach Druckaufschluss / Foodstuffs. Determination of trace elements. Determination of lead, cadmium, chromium and molybdenum by graphite furnace atomic absorption spectrometry (GFAAS) after pressure digestion

23. ASU §64 L 00.00-19/5: Bestimmung von Selen mit der Atomabsorptionsspektrometrie (AAS) - Hydridtechnik [Determination of selenium by atomic absorption spectrometry (AAS) - hydride technique]
24. 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 [Determination of minerals Ca, K, Mg, Na, P and S and trace elements Fe, Cu, Mn and Zn in foods by ICP-OES]
25. ASU §64 L 00.00-93 / DIN EN 15111: Bestimmung von Iod in Lebensmitteln - ICP-M-S-Verfahren / Foodstuffs. Determination of trace elements. Determination of iodine by ICP-MS (inductively coupled plasma mass spectrometry)
26. 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
27. ASU §64 L 00.00-128 / DIN EN 15765: Bestimmung Zinn in Lebensmitteln mit der Massensprektrometrie 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
28. 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)]