Proficiency Tests

DLA

food
cosmetics
consumer goods
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Evaluation Report
proficiency test

DLA 44/2017

Food Supplement II:

B, Ca, Cr, Cu, Fe, K, Mg, Mn, Mo, P, Se, Zn

in Tablet and Capsule Powder

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

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Unteraufträge *Subcontractors*	Die Prüfung der Gehalte, Homogenität und Stabilität von EP-Parametern wird von DLA im Unterauftrag vergeben. The analysis of the content, homogeneity and stability of PT-parameters are 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.

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1. Introduction

The participation in proficiency testing schemes is an essential element of the quality-management-system of every laboratory testing food and feed, cosmetics and food contact materials. The implementation of proficiency tests enables the participating laboratories to prove their own analytical competence under realistic conditions. At the same time they receive valuable data regarding the verification and/or validation of the particular testing method [1, 5].
The purpose of DLA is to offer proficiency tests for selected parameters in concentrations with practical relevance.
Realisation and evaluation of the present proficiency test follows the technical requirements of DIN EN ISO/IEC 17043 (2010) and DIN ISO 13528:2009 / ISO 13528:2015 [2, 3].

2. Realisation

2.1 Test material

The test material is a mixture of three common in commerce food supplements, two multi-vitamin and multi-mineral products and one product containing boron compounds, and maltodextrin as carrier / bulking agent.

The raw materials were crushed and the capsule shells removed, respectively, then sieved by means of a centrifugal mill (mesh < 500 µm), mixed and afterwards homogenized.

Afterwards the samples were portioned to approximately 10 g into metal-lised PET film bags and chronologically numbered.

The composition (list of ingredients) of the samples is given in table 1. The contents of analytes given in table 2 were calculated according to the manufacturers specifications.

Table 1: Composition of DLA-Samples

Multi-Mineral-Powder

Ingredients (1. Food Supplement, Tablets):
Minerals, vitamins, buling agents: dicalcium phosphate, microcrystalline cellulose, hydroxypropylmethylcellulose, release agent: fatty acids, ginseng, stabilizers: sodium salts of fatty acids, silica, coating agents: magnesium salts of fatty acids, hydroxymethylcellulose, glycerol, talc, Carnaubawax

Ingredients (2. Food Supplement, Tablets):
Calcium hypophosphate, magnesium oxide, calcium carbonate, starch, bulking agent: hydroxypropylmethylcellulose, vitamin C, vitamin E, release agents: fatty acids and magnesium salts of fatty acids, niacinamide, iron fumarate, pantothenic acid, lutein, vitamin A, copper sulfate, zinc oxide, manganese sulfate, vitamin D3, vitamin B6, vitamin K1, vitamin B2, vitamin B1, coenzyme Q10, vitamin B12, folic acid, chromium chloride, potassium iodide, sodium molybdate, sodium selenite, biotin, dyes: titanium dioxide, iron oxide, coating agent: polyethylene glycol, polyvinylpyrrolidone, release agent: talc

Ingredients (3. Food Supplement, Capsule powder without capsule shells):
Boron citrate, boron aspartate, boron-gycinate, microcrystalline cellulose, riboflavin

Further Ingredient:
Potassium chloride, maltodextrin

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

Table 2: Calculated amounts of PT parameters according to the manufacturers specifications

Parameter	**Content per 100 g**
Boron	100 mg
Calcium	6900 mg *
Chromium	1800 µg
Iron	280 mg
Potassium	5400 mg
Copper	44 mg
Magnesium	4800 mg
Manganese	68 mg
Molybdenum	1000 µg
Phosphorus	4300 mg *
Selenium	1900 µg
Zinc	290 mg

** additional amounts for bulking agents possible*

2.1.1 Homogeneity

The **mixture homogeneity after bottling** was examined 8-fold by determination of copper by ICP-MS. The repeatability standard deviation was 1,44% and thus within the range of repeatability standard deviations of comparable methods (e.g. ASU §64 L 00.00-144, s. 3.6.2). 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 they are in the range of < 4,5% (from 1,35% to 4,12%). Thus they were similar to the repeatability standard deviations of the corresponding official methods (e.g. ASU methods, s. 3.6.2) (see Tab. 3) [16-26]. The repeatability standard deviations of the participants' results are given in the documentation in the statistic data (see 4.1 and 4.12).

Table 3: Repeatability standard deviation S_r of double determinations of the participants (coefficient of variation CV_r in %)

Parameter	**CV_r**
Boron	2,05 %
Calcium	1,58 %
Chromium	2,83 %
Iron	2,60 %
Potassium	2,47 %
Copper	1,89 %
Magnesium	1,77 %
Manganese	2,28 %
Molybdenum	4,12 %
Phosphorus	1,35 %
Selenium	3,52 %
Zinc	2,28 %

Furthermore, the homogeneity was characterized by the **trend line function of participants' results for chronological bottled single samples**. The maximum deviations from the mean value of the trend line were 5,4% of the target standard deviation σ_{pt} or σ_{pt}' (s. 5.2 Homogeneity) for copper and 36,5% for cadmium and thus they can be regarded as low to acceptable.

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

2.1.2 Stability

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

Furthermore, **participants' results** were compared with the corresponding **date of analysis** to characterize the stability of the PT-material during the whole time of analysis of the present PT (44-57 days) by using the trendline-functions.
The maximum deviations from the mean value of the trend line for copper and calcium were at 29% and 43% of the target standard deviation σ_{pt} or σ_{pt}' (see documentation section 5.3 Stability) and thus they can be regarded as low to acceptable.

2.2 Sample shipment and information to the test

Two portions of test material were sent to every participating laboratory in the 37th week of 2017. The testing method was optional. The tests should be finished at 4st August 2017 the latest.

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

The two portions contain identical samples of a food supplement with above mentioned parameters in the matrix of tablets and capsule powder, respectively. The analysis method is optional.

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

Please note the attached information on the proficiency test.
(see documentation, section 5.5 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 18 participants submitted their results in time.

3. Evaluation

3.1 Consensus value from participants (assigned value)

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

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

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

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

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 VK_R in percent of the mean is given as variation coefficient in the statistical data of participant for each parameter. The significance of VK_R is further explained in section 3.9.

3.5 Exclusion of results and outliers

Before statistical evaluation obvious blunders, such as those with incorrect units, decimal point errors, and results for a another proficiency test item can be removed from the data set [2]. Even if a result clearly deviates from the robust mean (e.g. factor >10) and has an influence on the robust statistics, a result can be excluded from statistical evaluation [3].

All results should be given at least with 2 significant digits. Specifying 3 significant digits is usually sufficient.

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

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

3.6 Target standard deviation (for proficiency assessment)

The target standard deviation of the assigned value 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.

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

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

For valuation of all following parameters in the present PT the target standard deviation according to the general model of Horwitz was applied (see 3.6.1): Copper, Potassium, Manganese, Molybdenum, Phosphorus and Zinc.

Additionally for Boron, Calcium, Chromium, Iron, Magnesium and Selenium the standard uncertainty was considered by evaluating with z'-scores (see 3.6.8).

The target standard deviation of the evaluation by precision experiment (s. 3.6.2) was not considered, because the precision data were not obtained with a comparable matrix and were not in a comparable range of concentration with respect to the present PT samples.

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 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.

Table 4: 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} [16-26]

Parameter	**Matrix**	**Mean [mg/kg]**	**RSD_r**	**RSD_R**	**σ_{pt}**	**Method / Literature**
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]
Cr	Infant food	0,17	7,3%	19%	18,3%[1]	GF-AAS [20]
	Rice powder	0,11	19,2%	35%	32,3%	GF-AAS [20]
Cu	Lobster	16,40	5,72%	6,82%	5,49%	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]
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	Infant food	0,50	6,6%	21%	20,5%[1]	GF-AAS [20]
	Rice powder	0,56	8,7%	20%	19,0%	GF-AAS [20]
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]
Se	Katfish	1,797	9,85%	10,1%	7,31%[1]	AAS [21]
	Rice	0,374	2,41%	11,8%	11,7%	AAS [21]
Zn	Lobster	13,9	4,63%	7,90%	7,19%	ICP-OES [22]
	Children´s food soy	43,5	2,60%	6,89%	6,64%[1]	ICP-OES [22]

[1] *used in evaluation (s. chapter 4) for information*

3.6.3 Value by perception

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

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

Table 5 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}). The value indicated as z-Score (Info) only obtains a informative character. The both z-Scores were calculated with the different target standard deviations in accordance with 3.6.

3.7.1 Warning and action signals

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

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

Table 5: Characteristics of the present PT (on grey) in comparison to previous PTs since 2016 (SD = standard deviation, CV = coefficient of variation)

Para-meter	**Matrix (Powder)**	**robust Mean [mg/kg]**	**rob. SD (S*) [mg/kg]**	**rel. SD (CV_{S*}) [%]**	**Quotient S*/σpt**	**DLA-report**
B	Potatoes	3,88	0,689	17,8%	1,4	DLA 46/2017
B	Tablets/Capsules	1170	183	15,6%	2,0[1]	DLA 44/2017
Ca	Potatoes	238	12,0	5,04%	0,72	DLA 46/2017
Ca	Tablets/Capsules	81630	5240	6,42%	1,8[1]	DLA 44/2017
Cr	Mussels-Fish	1,23	0,266	21,6%	1,4	DLA 58/2016
Cr	Potatoes	**	-	-	-	DLA 46/2017
Cr	Tablets/Capsules	21,0	4,74	22,6%	1,9	DLA 44/2017
Cu	Mussels-Fish	5,75	0,439	7,63%	0,62	DLA 58/2016
Cu	Potatoes	1,98	0,117	5,90%	0,41	DLA 46/2017
Cu	Tablets/Capsules	432	33,1	7,66%	1,2	DLA 44/2017
Fe	Mussels-Fish	305	22,1	7,24%	1,1	DLA 58/2016
Fe	Potatoes	15,0	1,22	8,10%	0,76	DLA 46/2017
Fe	Tablets/Capsules	3200	357	11,2%	2,0[1]	DLA 44/2017
K	Potatoes	13162	604	4,59%	1,2	DLA 46/2017
K	Tablets/Capsules	53370	3160	5,92%	1,9%	DLA 44/2017
Mg	Potatoes	736	27,1	3,68%	0,62	DLA 46/2017
Mg	Tablets/Capsules	48490	3660	7,55%	1,9[1]	DLA 44/2017
Mn	Mussels-Fish	8,79	0,696	7,93%	0,69	DLA 58/2016
Mn	Potatoes	3,66	0,327	8,9%	0,68	DLA 46/2017
Mn	Tablets/Capsules	678	73,9	10,1%	1,8	DLA 44/2017
Mo	Mussels-Fish	0,536	0,0400	7,45%	0,42	DLA 58/2016
Mo	Potatoes	0,197	0,0161	8,2%	0,40	DLA 46/2017
Mo	Tablets/Capsules	12,1	2,48	20,5%	1,9	DLA 44/2017
P	Potatoes	1451	49,1	3,38%	0,63	DLA 46/2017
P	Tablets/Capsules	53160	2720	5,11%	1,6	DLA 44/2017
Se	Tablets/Capsules	20,9	4,34	20,8%	1,8[1]	DLA 44/2017
Zn	Mussels-Fish	51,0	5,17	10,2%	1,1	DLA 58/2016
Zn	Potatoes	7,83	0,726	9,30%	0,79	DLA 46/2017
Zn	Tablets/Capsules	2960	143	4,85%	1,0	DLA 44/2017

[1] with target standard deviation σpt'
*** no statistical evaluation (< 7 or < 5 results)*

3.8 z'-Score

The z'-score can be used for the valuation of the results of the participants, in cases the standard uncertainty has to be considered (s. 3.8). The z'-score represents the relation of the deviation of the result (x) of the participant from the respective consensus value (X) to the square root of quadrat sum of the target standard deviation (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 coefficient of variation (CV)

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

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

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

3.10 Quotient S*/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 Quotient $U_{(Xpt)}/\sigma_{pt}$ is reported in the characteristics of the test.

4. Results

Comments to the distribution of the results:

The kernel density plots showed for all parameters nearly a normal distribution of results (figures see documentation 5.3). Partly slight shoulders and separate smaller peaks can be seen, which are due to individual values and outliers.
In the case of boron and chromium a distribution of results with two peaks can be seen. However, the information provided by the participants on the methods gave no obvious indications of such an array of results.
When using the robust standard deviation as an estimator h, the distributions are converted into single peak distributions, so that a statistical evaluation has been carried out.

Comments to the statistic data:

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

For Boron, Calcium, Chromium, Iron, Magnesium and Selenium the distribution of results showed an increased variability. The quotient S^*/σ_{pt} was clearly higher than 2,0. Thus the parameters were evaluated considering the standard uncertainty by z'-scores. The quotients S^*/σ_{pt}' were then in the range of ≤ 2,0 (s. Tab. 5).

For the other parameters the distribution of results showed a normal variability. The quotients S^*/σ_{pt} were all in the range of 1,0 to 2,0 (s. Tab. 4).

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

The comparability of results is given.

The quotient $U(X_{pt})/\sigma_{pt}$ and $U(X_{pt})/\sigma_{pt}'$ is for copper and zinc with 0,35 and 0,29 in the normal range. For all other parameters the quotients of 0,51 to 0,60 and for boron of 0,71 were increased). The standard uncertainty $U(X_{pt})$ was therefore considered for the evaluation of 6 parameters (s. above).

67% to 89% of results were in the respective target range.

The robust means of the participant results were for all parameters in the range of 98% to 124% of the contents according to the manufacturer specifications (s. Tab. 2): 98-102% for Cu, K, Mg, Mn and Zn, 110-118% for B, Ca, Cr, Fe and Se, for Mo 121% and P 124%.

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 ($X_{pt} + 2\sigma_{pt}$´) *
Variation coefficient V_K in %
Quotient S^*/σ_{pt} *or* S^*/σ_{pt}'
Standard uncertainty $U(X_{pt})$
Quotient $U(X_{pt})/\sigma_{pt}$ *or* $U(X_{pt})/\sigma_{pt}$'
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** σ_{pt}	**z-Score** (Info)	**Hinweis** / **Remark**

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

4.1 Bor / Boron in mg/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	12
Number of outliers	0
Mean	118
Median	116
Robust Mean (X)	**117**
Robust standard deviation (S*)	**18,3**
Number with 2 replicates	11
Repeatability SD (S_r)	2,44
Repeatability (CV_r)	2,05%
Reproducibility SD (S_R)	19,1
Reproducibility (CV_R)	16,0%
Target range:	
Target standard deviation σ_{pt}	**9,26**
lower limit of target range	**99,0**
upper limit of target range	**136**
Quotient S^*/σ_{pt}	*1,98*
Standard uncertainty $U_{(Xpt)}$	*6,61*
Quotient $U_{(Xpt)}/\sigma_{pt}$	*0,71*
Results in the target range	*9*
Percent in the target range	*75%*

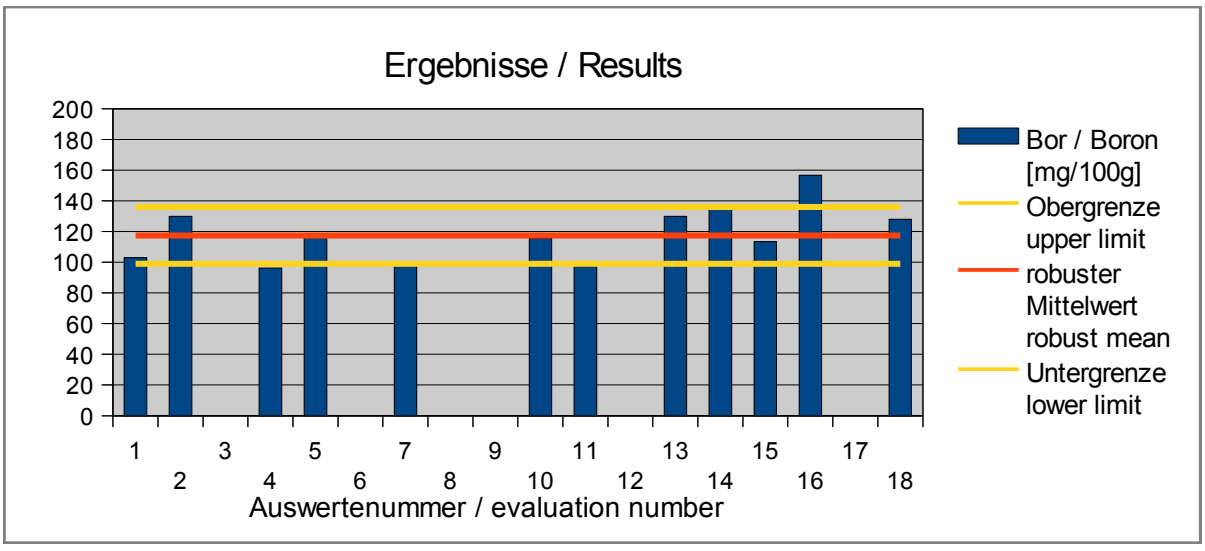


Abb. / Fig. 1: Ergebnisse Bor / Results Boron

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Bor / Boron [mg/100g]	Abweichung [mg/100g] Deviation [mg/100g]	z´-Score (σ_{pt})	Hinweis Remark
1	103	-14,5	-1,6	
2	130	12,5	1,4	
3				
4	96,2	-21,3	-2,3	
5	118	0,00517	0,0	
6				
7	99,8	-17,7	-1,9	
8				
9				
10	115	-2,09	-0,23	
11	97,3	-20,2	-2,2	
12				
13	130	12,5	1,4	
14	134 *	16,9	1,8	
15	113	-4,09	-0,44	
16	157	39,3	4,2	
17				
18	128	10,5	1,1	

* Mean calculated by DLA

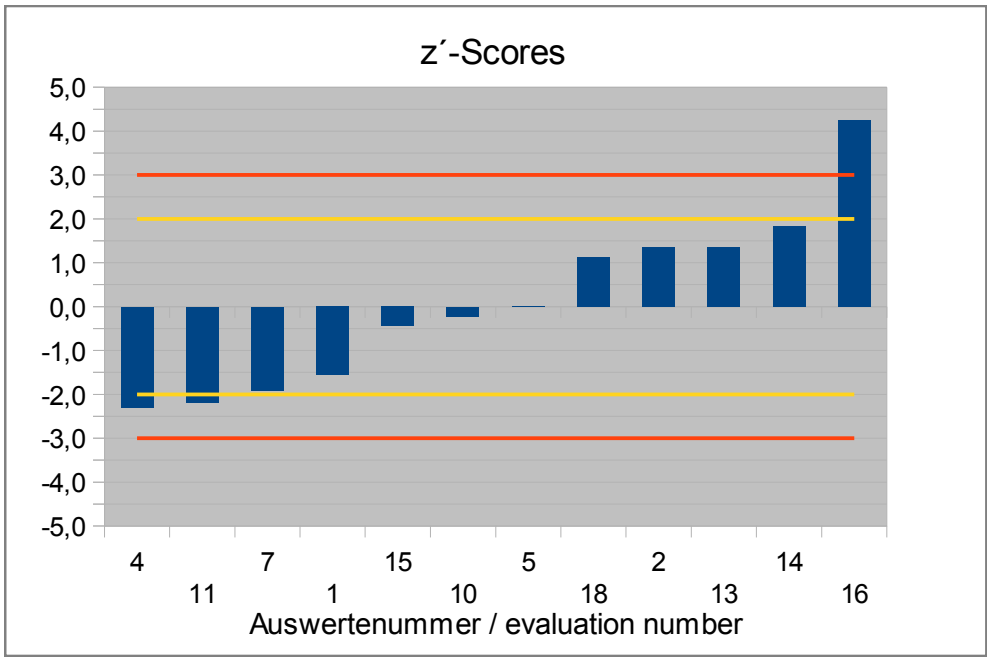


Abb. / Fig. 2: Z´-Scores Bor / Boron

4.2 Calcium in mg/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	17
Number of outliers	2
Mean	8130
Median	8250
Robust Mean (X)	**8160**
Robust standard deviation (S*)	**524**
Number with 2 replicates	14
Repeatability SD (S_r)	128
Repeatability (CV_r)	1,58%
Reproducibility SD (S_R)	403
Reproducibility (CV_R)	4,95%
Target range:	
Target standard deviation σ_{pt}´	**286**
Target standard deviation (for Information)	620
lower limit of target range	**7590**
upper limit of target range	**8730**
Quotient S/σ_{pt}´*	*1,8*
Standard uncertainty $U_{(Xpt)}$	*159*
Quotient $U_{(Xpt)}/\sigma_{pt}$´	*0,55*
Results in the target range	*13*
Percent in the target range	*76%*

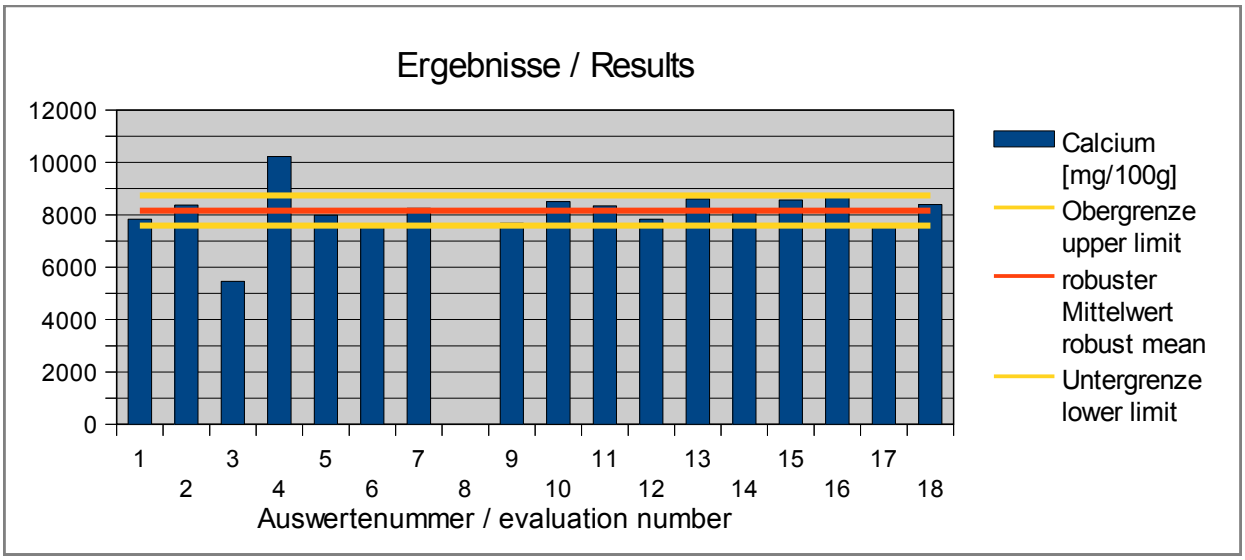


Abb. / Fig. 3: Ergebnisse Calcium / Results Calcium

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Calcium [mg/100g]	Abweichung [mg/100g] Deviation [mg/100g]	z´-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	7829	-334	-1,2	-0,54	
2	8370	207	0,72	0,33	
3	5460	-2703	-9,4	-4,4	Ausreisser / Outlier
4	10232	2069	7,2	3,3	Ausreisser / Outlier
5	7990	-173	-0,60	-0,28	
6	7624	-539	-1,9	-0,87	
7	8249	86,5	0,30	0,14	
8					
9	7680	-483	-1,7	-0,78	
10	8501	338	1,2	0,55	
11	8346	183	0,64	0,30	
12	7836	-327	-1,1	-0,53	
13	8607	444	1,6	0,72	
14	8105 *	-58,0	-0,20	-0,09	
15	8569	406	1,4	0,66	
16	8802	639	2,2	1,0	
17	7543	-620	-2,2	-1,0	
18	8389	226	0,79	0,37	

* Mean calculated by DLA

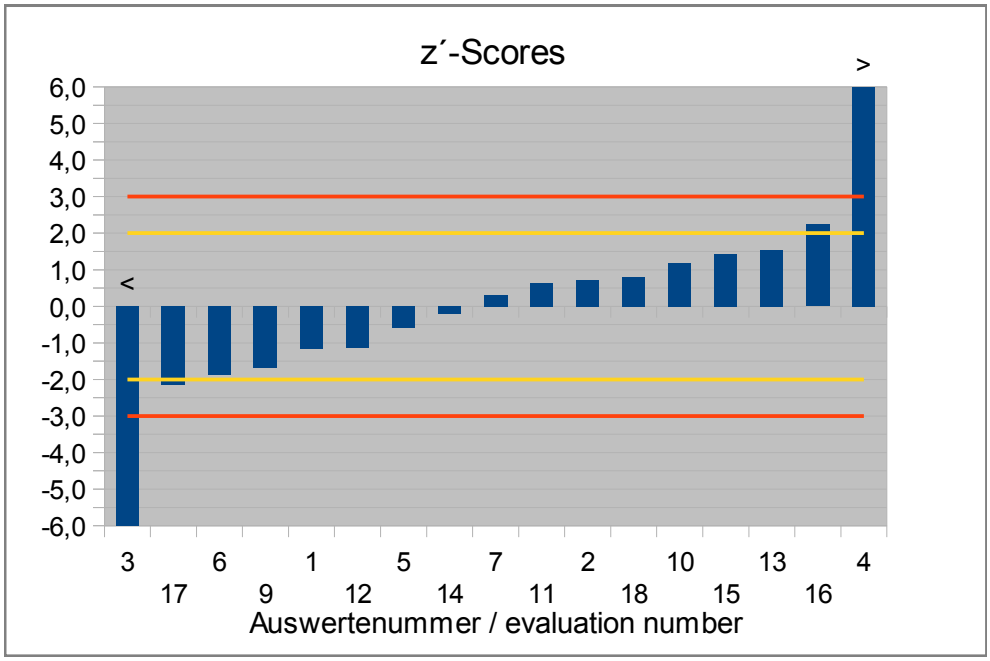


Abb. / Fig. 4: Z´-Scores Calcium

4.3 Chrom / Chromium in µg/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	19
Number of outliers	0
Mean	2110
Median	2230
Robust Mean (X)	**2100**
Robust standard deviation (S*)	**474**
Number with 2 replicates	18
Repeatability SD (S_r)	59,4
Repeatability (CV_r)	2,83%
Reproducibility SD (S_R)	516
Reproducibility (CV_R)	24,5%
Target range:	
Target standard deviation σ_{pt}	**252**
Target standard deviation (for Information)	384
lower limit of target range	**1600**
upper limit of target range	**2600**
Quotient S/σ_{pt}*	*1,9*
Standard uncertainty $U_{(Xpt)}$	*136*
Quotient $U_{(Xpt)}/\sigma_{pt}$	*0,54*
Results in the target range	*15*
Percent in the target range	*79%*

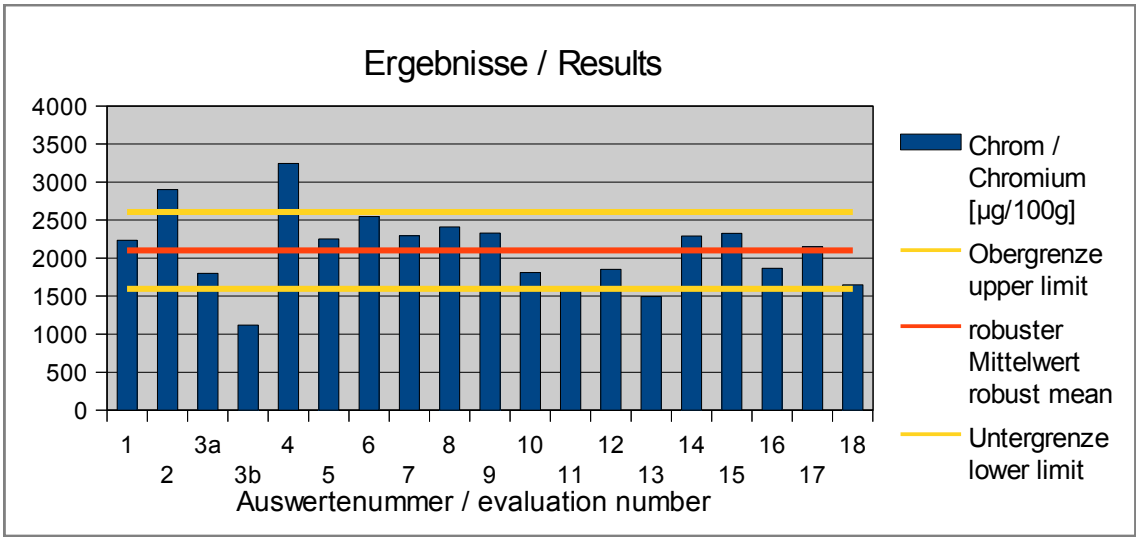


Abb. / Fig. 5: Ergebnisse Chrom / Results Chromium

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Chrom / Chromium [µg/100g]	Abweichung [mg/100g] Deviation [mg/100g]	z´-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	2233	133	0,53	0,35	
2	2900	800	3,2	2,1	
3a	1800	-300	-1,2	-0,78	
3b	1120	-980	-3,9	-2,6	
4	3243	1143	4,5	3,0	
5	2250	150	0,59	0,39	
6	2545	445	1,8	1,2	
7	2294	194	0,77	0,50	
8	2409	309	1,2	0,80	
9	2330	230	0,91	0,60	
10	1811	-289	-1,1	-0,75	
11	1598	-502	-2,0	-1,3	
12	1851	-249	-1,0	-0,65	
13	1495	-605	-2,4	-1,6	
14	2292 *	191	0,76	0,50	
15	2324	224	0,89	0,58	
16	1865	-235	-0,93	-0,61	
17	2149	48,6	0,19	0,13	
18	1650	-450,44	-1,8	-1,2	

* Mean calculated by DLA

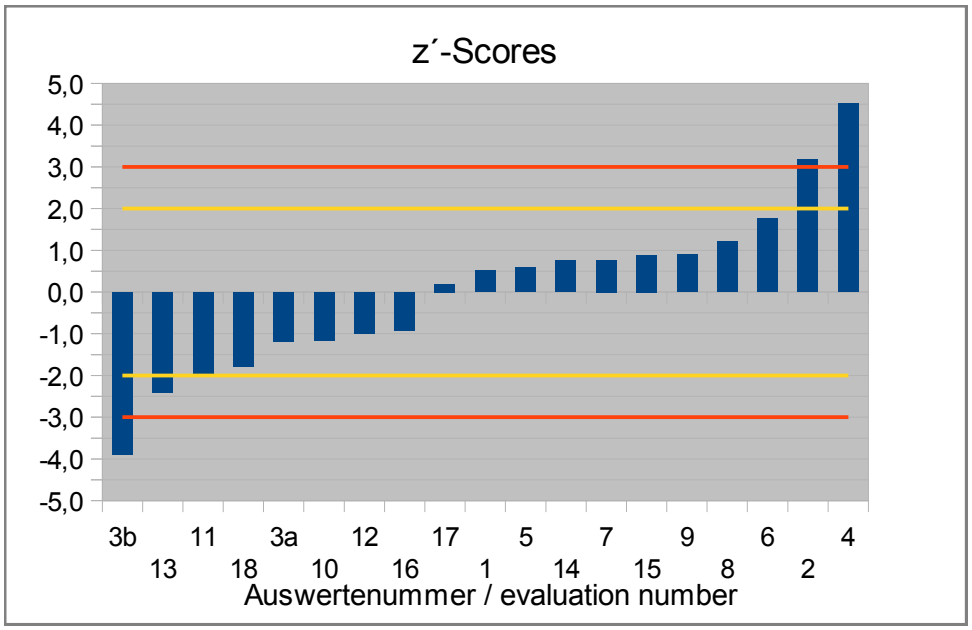


Abb. / Fig. 6: Z´-Scores Chrom / Chromium

4.4 Eisen /Iron in mg/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	19
Number of outliers	1
Mean	313
Median	324
Robust Mean (X)	**320**
Robust standard deviation (S*)	**35,7**
Number with 2 replicates	17
Repeatability SD (S_r)	8,41
Repeatability (CV_r)	2,60%
Reproducibility SD (S_R)	37,4
Reproducibility (CV_R)	11,6%
Target range:	
Target standard deviation σ_{pt}´	**18,3**
Target standard deviation (for Information)	21,5
lower limit of target range	**283**
upper limit of target range	**357**
Quotient S/σ_{pt}´*	*1,95*
Standard uncertainty $U_{(Xpt)}$	*10,2*
Quotient $U_{(Xpt)}/\sigma_{pt}$´	*0,56*
Results in the target range	*13*
Percent in the target range	*68%*

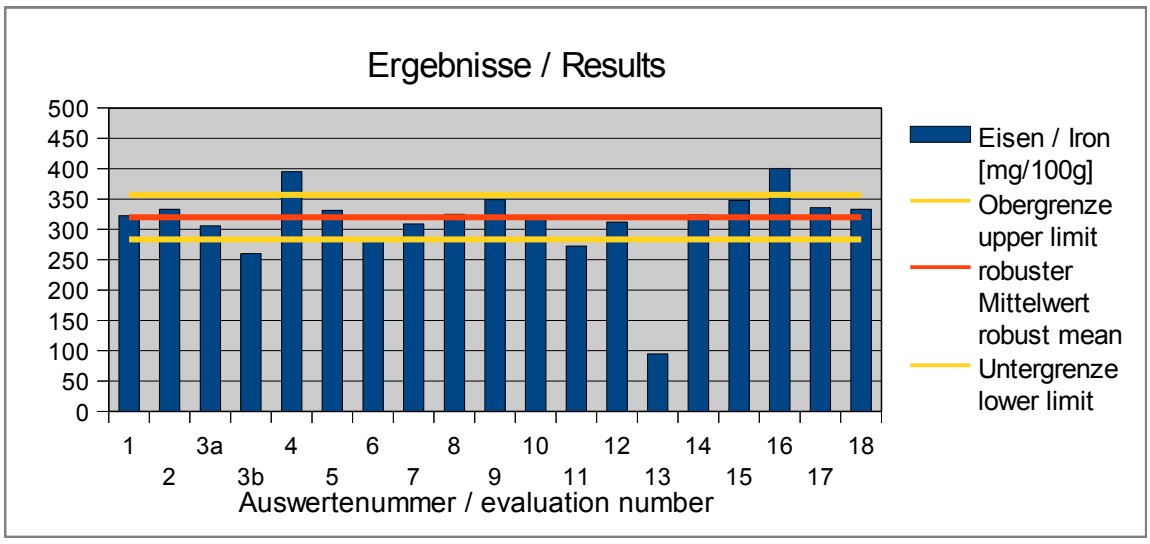


Abb. / Fig. 7: Ergebnisse Eisen / Results Iron

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Eisen / Iron [mg/100g]	Abweichung [mg/100g] Deviation [mg/100g]	z´-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	323	2,48	0,14	0,12	
2	333	13,0	0,71	0,60	
3a	306	-14,0	-0,77	-0,65	
3b	260	-60,0	-3,3	-2,8	
4	395	75,1	4,1	3,5	
5	332	11,5	0,63	0,53	
6	281	-38,8	-2,1	-1,8	
7	309	-11,0	-0,60	-0,51	
8	325	4,48	0,24	0,21	
9	349	29,0	1,6	1,4	
10	319	-1,22	-0,07	-0,06	
11	272	-47,6	-2,6	-2,2	
12	312	-8,02	-0,44	-0,37	
13	95,0	-225	-12	-10	Ausreisser / Outlier
14	324 *	3,98	0,22	0,19	
15	348	27,5	1,5	1,3	
16	400	80,2	4,4	3,7	
17	336	15,9	0,87	0,74	
18	333	13,0	0,71	0,60	

* Mean calculated by DLA

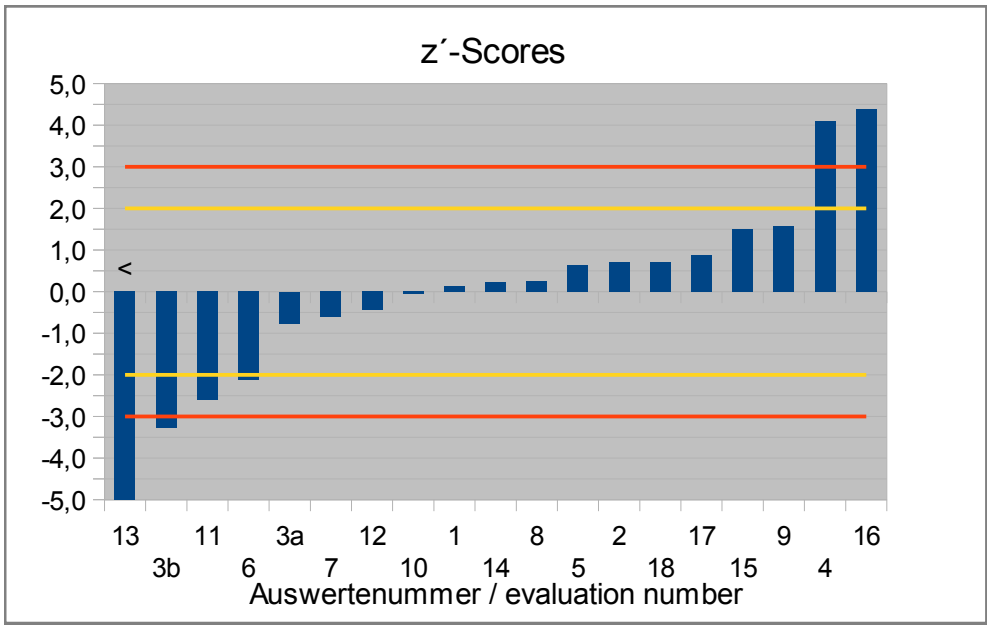


Abb. / Fig. 8: Z´-Scores Eisen / Iron

4.5 Kalium / Potassium in mg/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	16
Number of outliers	1
Mean	5270
Median	5260
Robust Mean (X)	**5340**
Robust standard deviation (S*)	**316**
Number with 2 replicates	14
Repeatability SD (S_r)	133
Repeatability (CV_r)	2,47%
Reproducibility SD (S_R)	317
Reproducibility (CV_R)	5,87%
Target range:	
Target standard deviation σ_{pt}	**166**
Target standard deviation (for Information)	249
lower limit of target range	**5000**
upper limit of target range	**5670**
Quotient S/σ_{pt}*	*1,9*
Standard uncertainty $U_{(Xpt)}$	*98,8*
Quotient $U_{(Xpt)}/\sigma_{pt}$	*0,60*
Results in the target range	*12*
Percent in the target range	*75%*

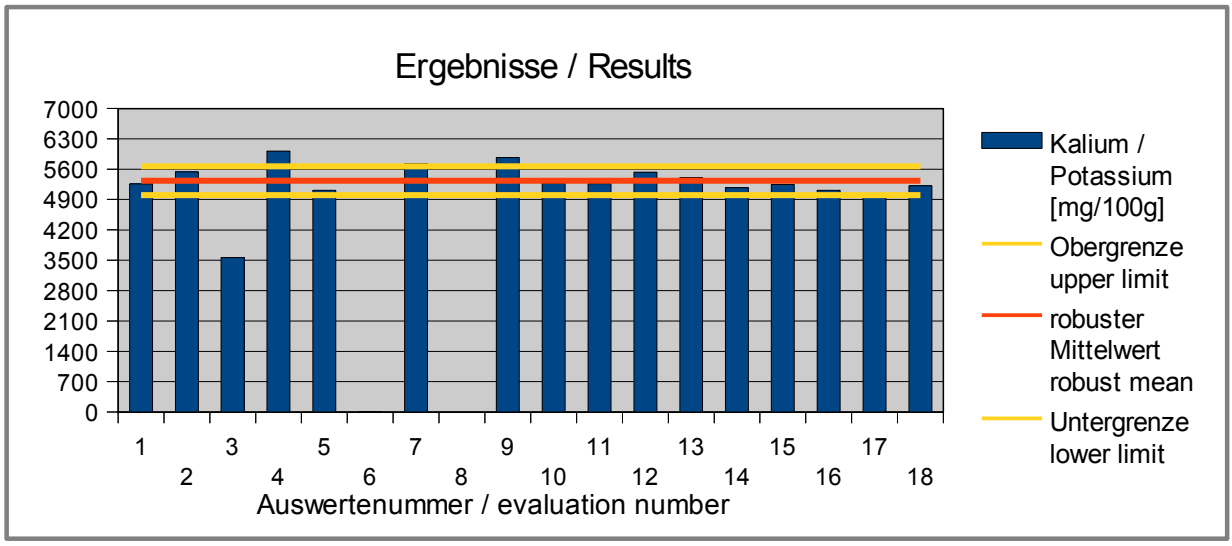


Abb. / Fig. 9: Ergebnisse Kalium / Results Potassium

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Kalium / Potassium [mg/100g]	Abweichung [mg/100g] Deviation [mg/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	5260	-77,3	-0,47	-0,31	
2	5540	203	1,2	0,82	
3	3560	-1777	-11	-7,1	Ausreisser / Outlier
4	6020	683	4,1	2,7	
5	5113	-224	-1,4	-0,90	
6	5,26				Ergebnis ausgeschlossen / Result excluded
7	5731	394	2,4	1,6	
8					
9	5870	533	3,2	2,1	
10	5275	-61,8	-0,37	-0,25	
11	5267	-69,4	-0,42	-0,28	
12	5529	192	1,2	0,77	
13	5409	72,2	0,44	0,29	
14	5173 *	-164	-1,0	-0,66	
15	5248	-88,5	-0,53	-0,35	
16	5111	-226	-1,4	-0,91	
17	5035	-302	-1,8	-1,2	
18	5216	-121	-0,73	-0,48	

* Mean calculated by DLA

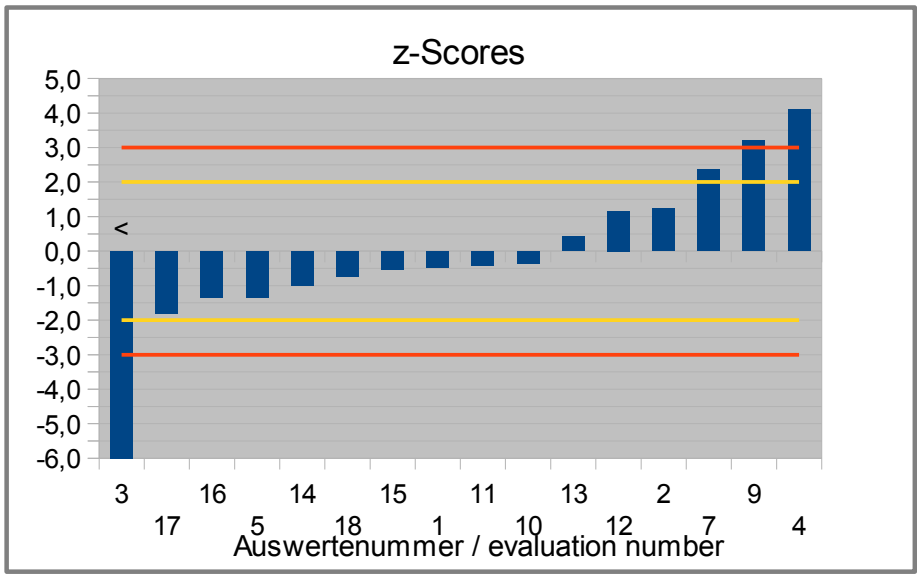


Abb. / Fig. 10: Z-Scores Kalium / Potassium

4.6 Kupfer / Copper in mg/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	18
Number of outliers	2
Mean	43,3
Median	43,8
Robust Mean (X)	**43,2**
Robust standard deviation (S*)	**3,31**
Number with 2 replicates	17
Repeatability SD (S_r)	1,01
Repeatability (CV_r)	1,89%
Reproducibility SD (S_R)	30,5
Reproducibility (CV_R)	57,3%
Target range:	
Target standard deviation σ_{pt}	**2,78**
Target standard deviation (for Information)	4,60
lower limit of target range	**37,7**
upper limit of target range	**48,8**
Quotient S/σ_{pt}*	*1,2*
Standard uncertainty $U_{(Xpt)}$	*0,976*
Quotient $U_{(Xpt)}/\sigma_{pt}$	*0,35*
Results in the target range	*16*
Percent in the target range	*89%*

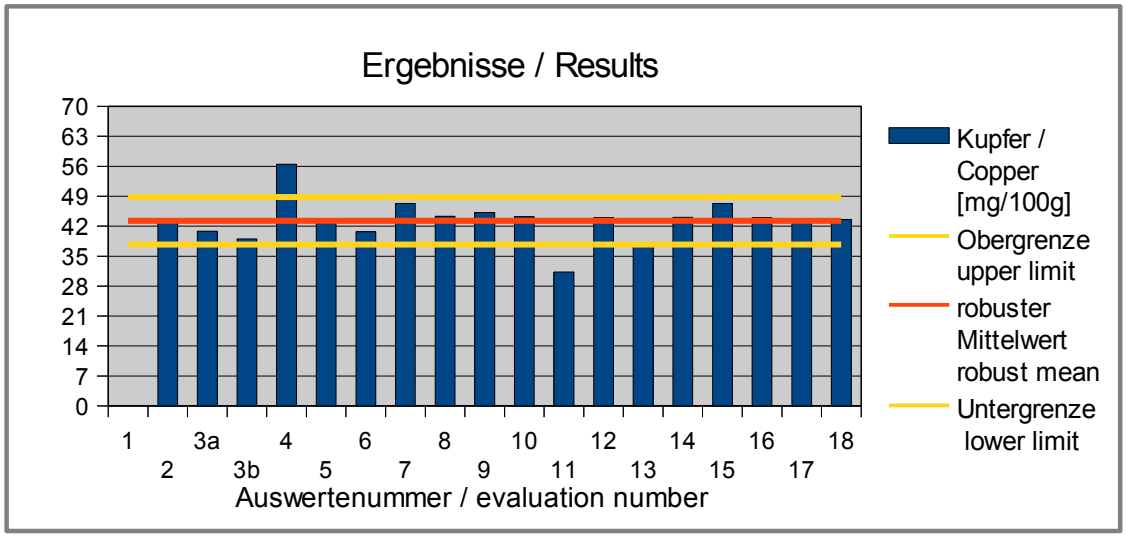


Abb. / Fig. 11: Ergebnisse Kupfer / Results Copper

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Kupfer / Copper [mg/100g]	Abweichung [mg/100g] Deviation [mg/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1					
2	43,0	-0,240	-0,09	-0,05	
3a	40,8	-2,44	-0,88	-0,53	
3b	39,0	-4,24	-1,5	-0,92	
4	56,5	13,3	4,8	2,9	Ausreisser / Outlier
5	42,8	-0,490	-0,18	-0,11	
6	40,7	-2,54	-0,92	-0,55	
7	47,3	4,06	1,5	0,88	
8	44,3	1,06	0,38	0,23	
9	45,2	1,96	0,71	0,43	
10	44,2	0,960	0,35	0,21	
11	31,3	-11,9	-4,3	-2,6	Ausreisser / Outlier
12	44,0	0,760	0,27	0,17	
13	37,8	-5,44	-2,0	-1,2	
14	44,1 *	0,810	0,29	0,18	
15	47,3	4,06	1,5	0,88	
16	44,0	0,760	0,27	0,17	
17	43,4	0,110	0,040	0,024	
18	43,6	0,360	0,13	0,08	

* Mean calculated by DLA

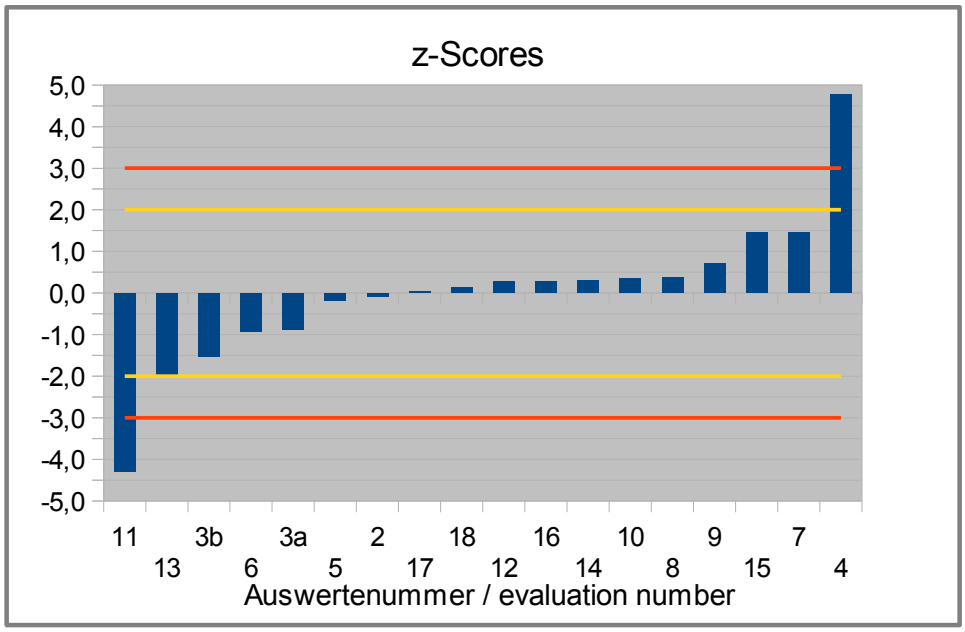


Abb. / Fig. 12: Z-Scores Kupfer / Copper

4.7 Magnesium in mg/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	16
Number of outliers	1
Mean	4930
Median	4810
Robust Mean (X)	**4850**
Robust standard deviation (S*)	**366**
Number with 2 replicates	14
Repeatability SD (S_r)	85,2
Repeatability (CV_r)	1,77%
Reproducibility SD (S_R)	336
Reproducibility (CV_R)	6,98%
Target range:	
Target standard deviation σ_{pt}´	**191**
Target standard deviation (for Information)	340
lower limit of target range	**4470**
upper limit of target range	**5230**
Quotient S/σ_{pt}´*	*1,9*
Standard uncertainty $U_{(Xpt)}$	*114*
Quotient $U_{(Xpt)}/\sigma_{pt}$´	*0,60*
Results in the target range	*11*
Percent in the target range	*69%*

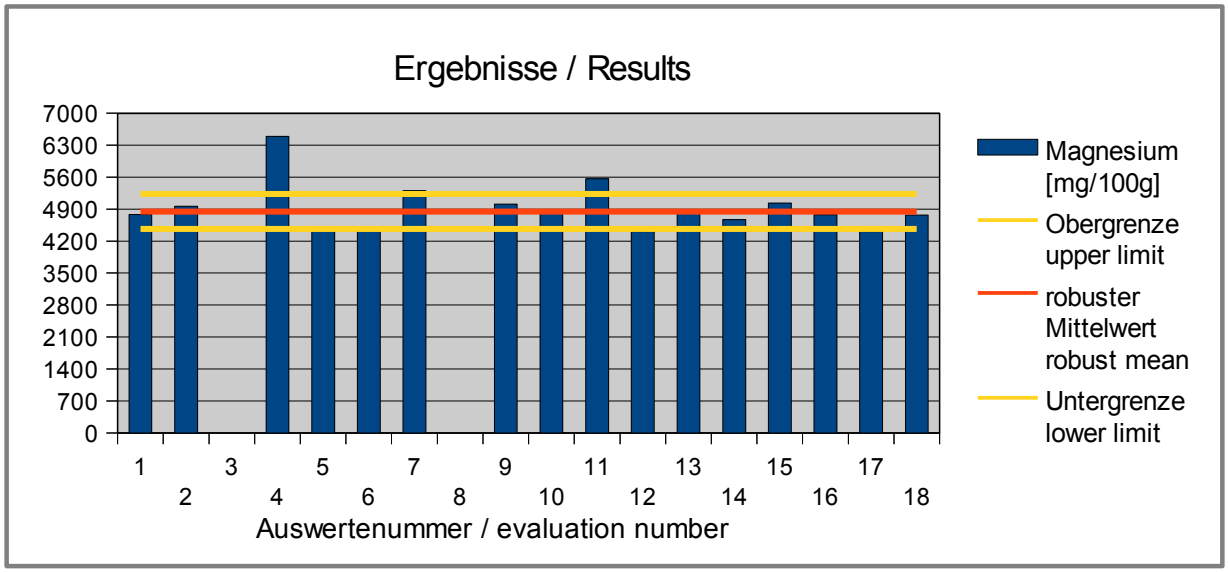


Abb. / Fig. 13: Ergebnisse Magnesium / Results Magnesium

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Magnesium [mg/100g]	Abweichung [mg/100g] Deviation [mg/100g]	z´-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	4783	-66,1	-0,35	-0,19	
2	4960	111	0,58	0,33	
3					
4	6495	1645	8,6	4,8	Ausreisser / Outlier
5	4507	-342	-1,8	-1,0	
6	4488	-361	-1,9	-1,1	
7	5300	451	2,4	1,3	
8					
9	5010	161	0,84	0,47	
10	4834	-15,1	-0,08	-0,044	
11	5565	716	3,7	2,1	
12	4406	-443	-2,3	-1,3	
13	4844	-5,06	-0,026	-0,015	
14	4670 *	-180	-0,94	-0,53	
15	5033	184	1,0	0,54	
16	4777	-72,0	-0,38	-0,21	
17	4410	-439	-2,3	-1,3	
18	4768	-81,1	-0,42	-0,24	

* Mean calculated by DLA

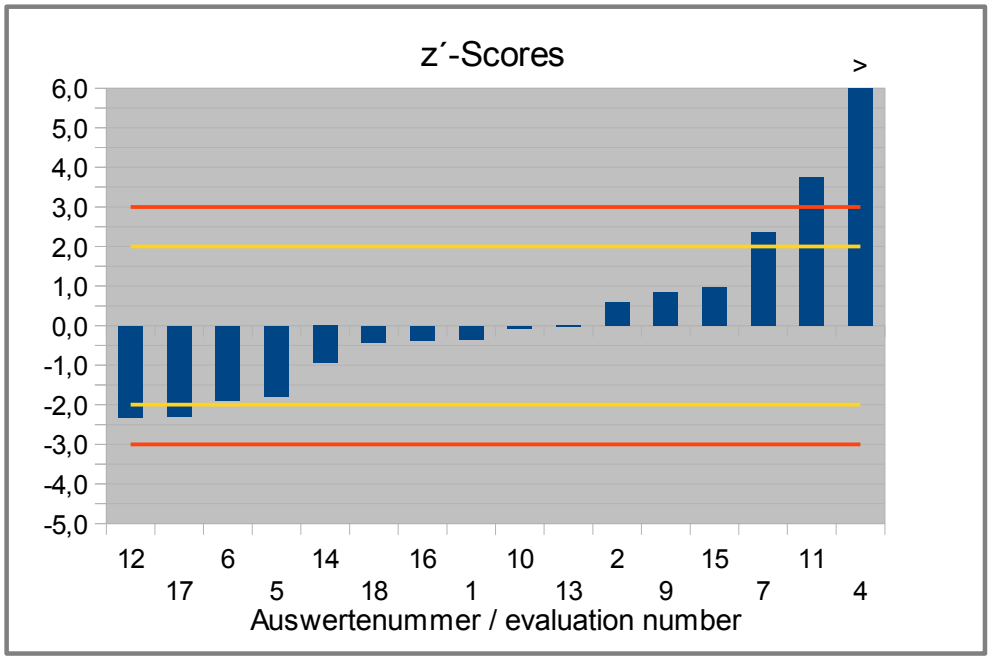


Abb. / Fig. 14: Z´-Scores Magnesium

4.8 Mangan / Manganese in mg/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	17
Number of outliers	1
Mean	67,5
Median	68,2
Robust Mean (X)	**67,8**
Robust standard deviation (S*)	**7,39**
Number with 2 replicates	15
Repeatability SD (S_r)	1,49
Repeatability (CV_r)	2,28%
Reproducibility SD (S_R)	8,40
Reproducibility (CV_R)	12,8%
Target range:	
Target standard deviation σ_{pt}	**4,07**
Target standard deviation (for Information)	9,02
lower limit of target range	**59,7**
upper limit of target range	**76,0**
Quotient S/σ_{pt}*	*1,8*
Standard uncertainty $U_{(Xpt)}$	*2,24*
Quotient $U_{(Xpt)}/\sigma_{pt}$	*0,55*
Results in the target range	*13*
Percent in the target range	*76%*

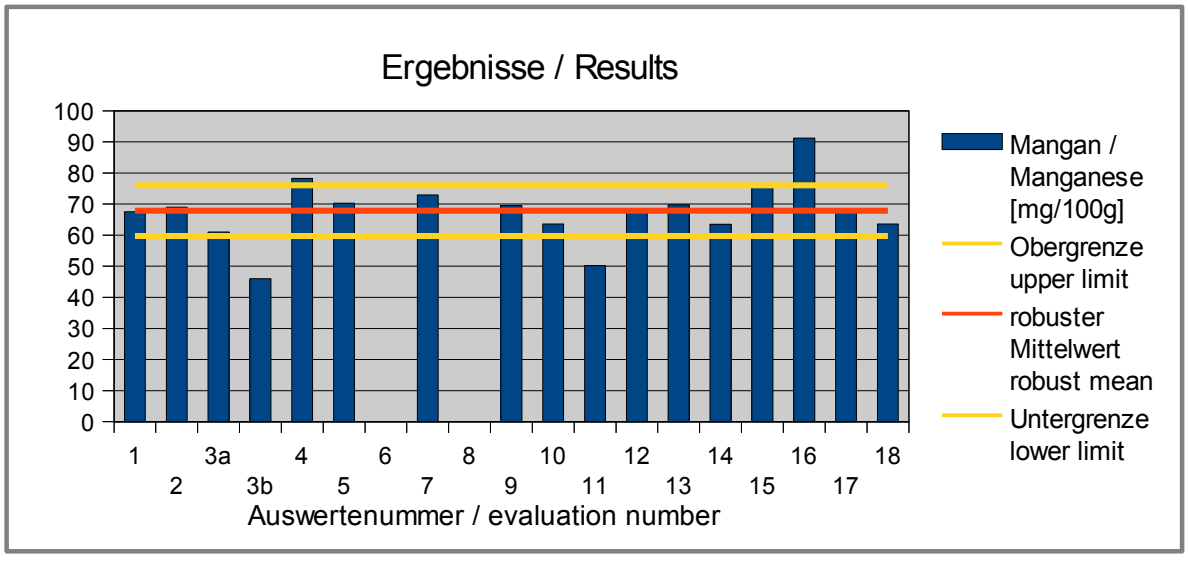


Abb. / Fig. 15: Ergebnisse Mangan / Results Manganese

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Mangan / Manganese [mg/100g]	Abweichung [mg/100g] Deviation [mg/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	67,6	-0,286	-0,070	-0,032	
2	69,0	1,16	0,29	0,13	
3a	61,0	-6,84	-1,7	-0,76	
3b	46,0	-21,8	-5,4	-2,4	
4	78,2	10,4	2,6	1,2	
5	70,3	2,41	0,59	0,27	
6					
7	72,9	5,06	1,2	0,56	
8					
9	69,5	1,66	0,41	0,18	
10	63,6	-4,28	-1,1	-0,47	
11	50,2	-17,6	-4,3	-2,0	
12	68,0	0,164	0,040	0,018	
13	69,8	1,91	0,47	0,21	
14	63,5 *	-4,34	-1,1	-0,48	
15	75,7	7,86	1,9	0,87	
16	91,3	23,4	5,8	2,6	Ausreisser / Outlier
17	68,2	0,404	0,10	0,045	
18	63,6	-4,24	-1,0	-0,47	

* Mean calculated by DLA

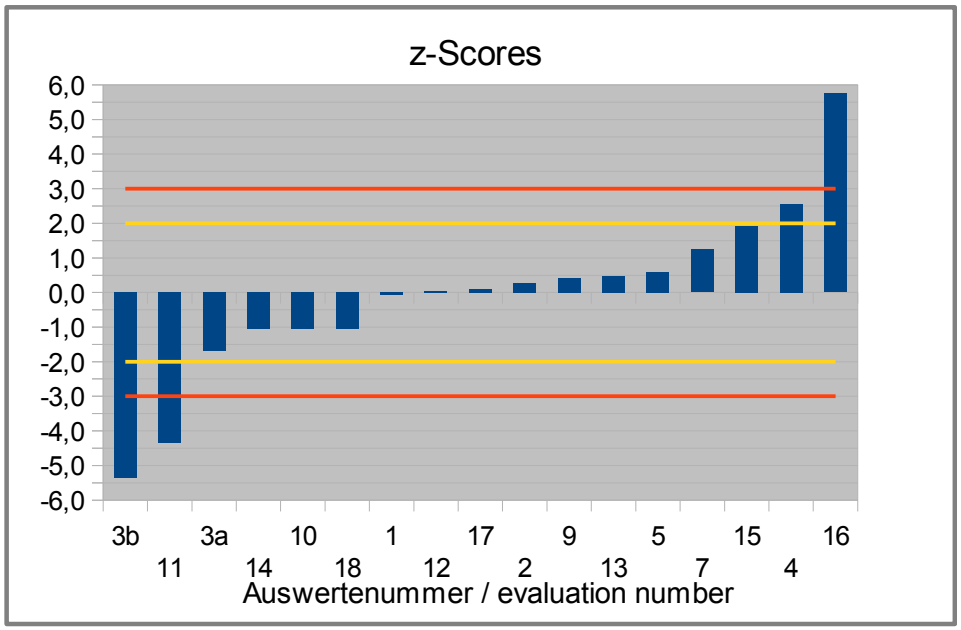


Abb. / Fig. 16: Z-Scores Mangan / Manganese

4.9 Molybdän / Molybdenum in µg/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	15
Number of outliers	1
Mean	1270
Median	1200
Robust Mean (X)	**1210**
Robust standard deviation (S*)	**248**
Number with 2 replicates	13
Repeatability SD (S_r)	49,1
Repeatability (CV_r)	4,12%
Reproducibility SD (S_R)	296
Reproducibility (CV_R)	24,9%
Target range:	
Target standard deviation σ_{pt}	**133**
Target standard deviation (for Information)	248
lower limit of target range	**946**
upper limit of target range	**1480**
Quotient S/σ_{pt}*	*1,9*
Standard uncertainty $U_{(Xpt)}$	*80,1*
Quotient $U_{(Xpt)}/\sigma_{pt}$	*0,60*
Results in the target range	*10*
Percent in the target range	*67%*

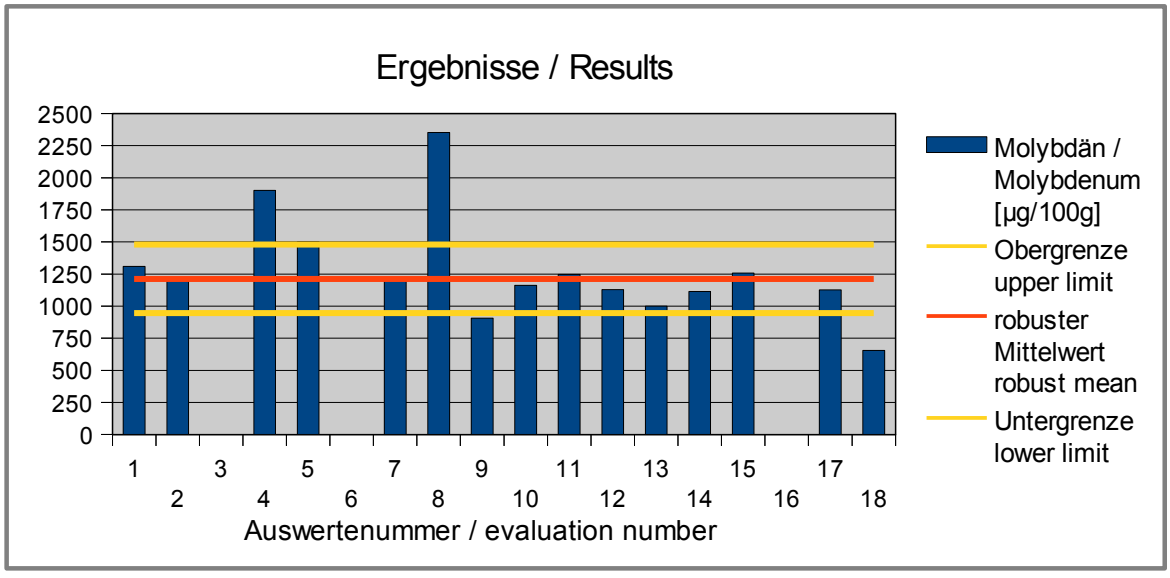


Abb. / Fig. 17: Ergebnisse Molybdän / Results Molybdenum

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Molybdän / Molybdenum [µg/100g]	Abweichung [µg/100g] Deviation [µg/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	1310	97,5	0,73	0,39	
2	1200	-12,5	-0,094	-0,050	
3					
4	1901	689	5,2	2,8	
5	1500	288	2,2	1,2	
6					
7	1230	17,5	0,13	0,071	
8	2353	1141	8,6	4,6	Ausreisser / Outlier
9	907	-305	-2,3	-1,2	
10	1162	-50,5	-0,38	-0,20	
11	1248	35,1	0,26	0,14	
12	1129	-83,5	-0,63	-0,34	
13	999	-213	-1,6	-0,86	
14	1115 *	-98,0	-0,74	-0,39	
15	1257	44,5	0,33	0,18	
16					
17	1126	-86,5	-0,65	-0,35	
18	655	-557,5	-4,2	-2,2	

* Mean calculated by DLA

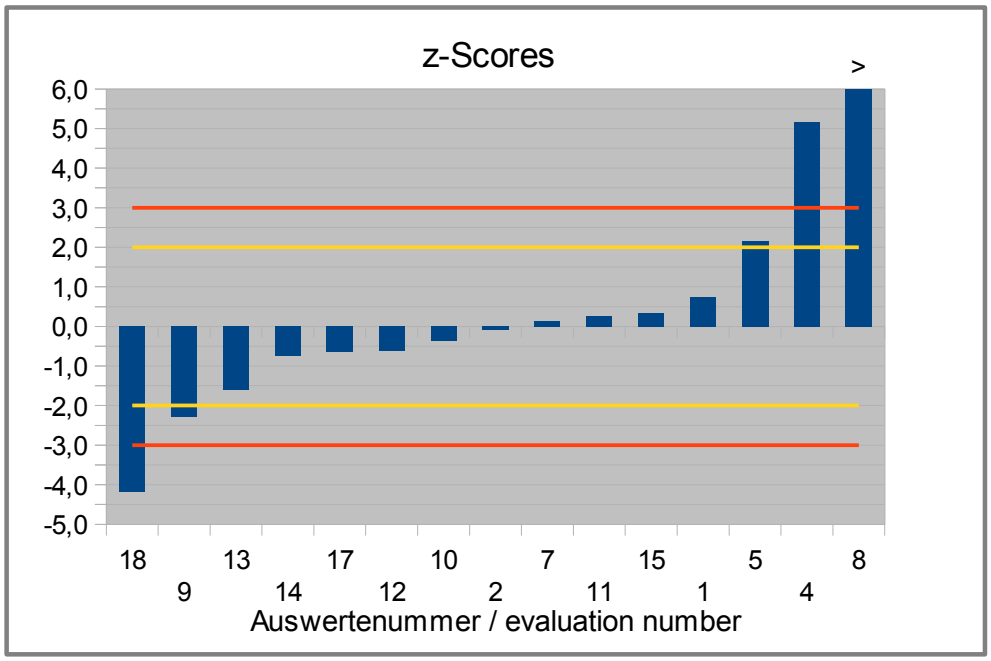


Abb. / Fig. 18: Z-Scores Molybdän / Molybdenum

4.10 Phosphor / Phosphorus in mg/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	13
Number of outliers	1
Mean	5380
Median	5230
Robust Mean (X)	**5320**
Robust standard deviation (S*)	**272**
Number with 2 replicates	11
Repeatability SD (S_r)	71,8
Repeatability (CV_r)	1,35%
Reproducibility SD (S_R)	286
Reproducibility (CV_R)	5,40%
Target range:	
Target standard deviation σ_{pt}	**165**
Target standard deviation (for Information)	398
lower limit of target range	**4990**
upper limit of target range	**5650**
Quotient S/σ_{pt}*	*1,6*
Standard uncertainty $U_{(Xpt)}$	*94,2*
Quotient $U_{(Xpt)}/\sigma_{pt}$	*0,57*
Results in the target range	*10*
Percent in the target range	*77%*

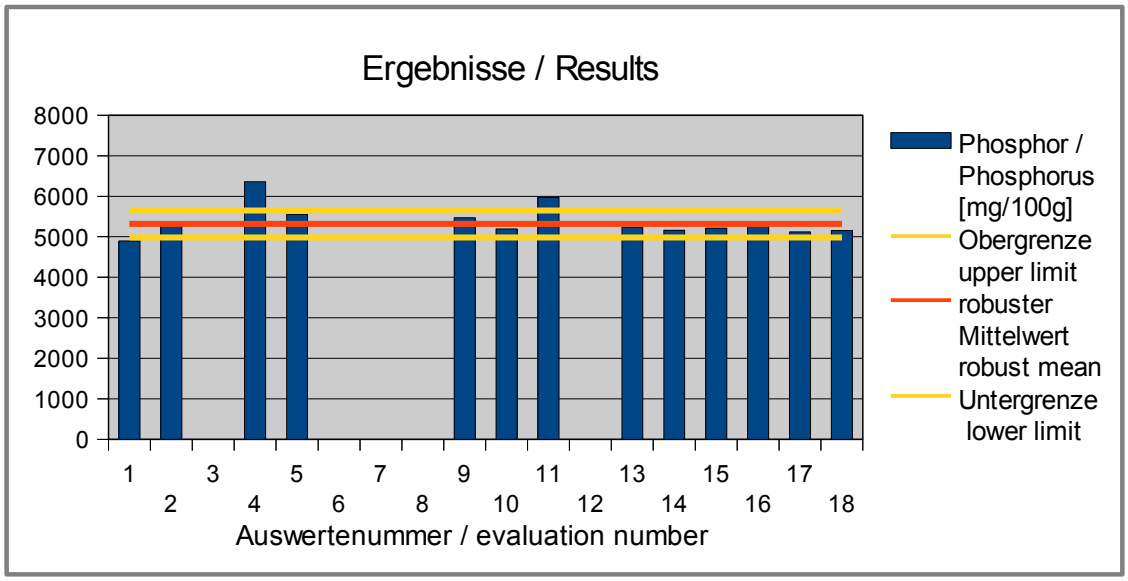


Abb. / Fig. 19: Ergebnisse Phosphor / Results Phosphorus

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Phosphor / Phosphorus [mg/100g]	Abweichung [mg/100g] Deviation [mg/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	4895	-421	-2,5	-1,1	
2	5370	54,0	0,33	0,14	
3					
4	6361	1045	6,3	2,6	Ausreisser / Outlier
5	5546	230	1,4	0,58	
6					
7					
8					
9	5470	154	0,93	0,39	
10	5194	-122	-0,74	-0,31	
11	5970	654	4,0	1,6	
12					
13	5233	-83,0	-0,50	-0,21	
14	5161 *	-155	-0,94	-0,39	
15	5213	-103	-0,62	-0,26	
16	5300	-15,9	-0,10	-0,040	
17	5120	-196	-1,2	-0,49	
18	5152	-164	-1,0	-0,41	

* Mean calculated by DLA

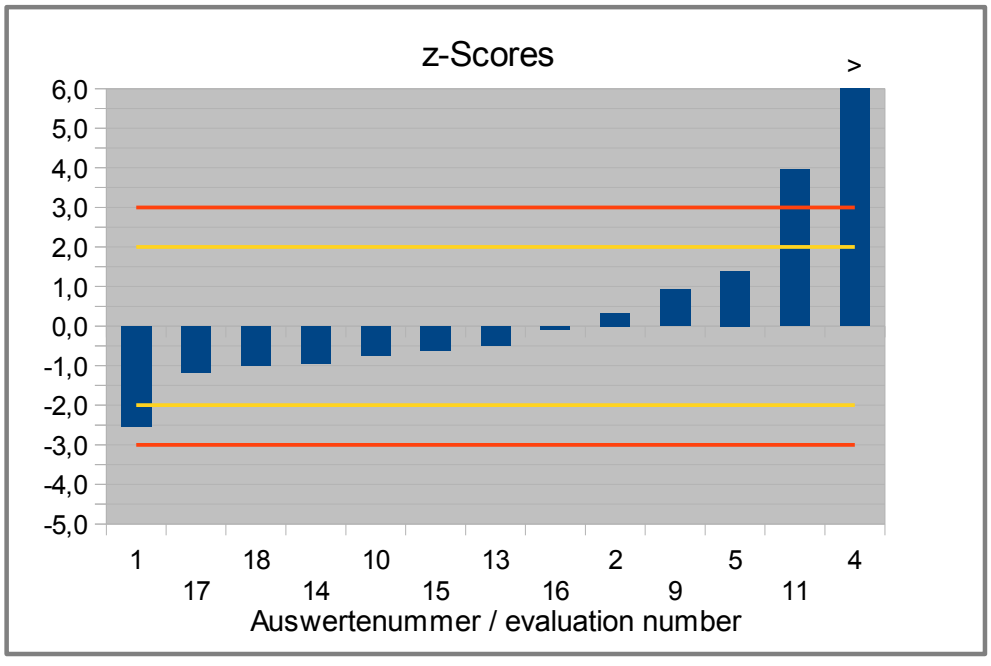


Abb. / Fig. 20: Z-Scores Phosphor / Phosphorus

4.11 Selen / Selenium in µg/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	19
Number of outliers	1
Mean	2180
Median	2050
Robust Mean (X)	**2090**
Robust standard deviation (S*)	**434**
Number with 2 replicates	17
Repeatability SD (S_r)	73,9
Repeatability (CV_r)	3,52%
Reproducibility SD (S_R)	481
Reproducibility (CV_R)	22,9%
Target range:	
Target standard deviation σ_{pt}´	**246**
Target standard deviation (for Information)	153
lower limit of target range	**1600**
upper limit of target range	**2580**
Quotient S/σ_{pt}´*	*1,8*
Standard uncertainty $U_{(Xpt)}$	*125*
Quotient $U_{(Xpt)}/\sigma_{pt}$´	*0,51*
Results in the target range	*14*
Percent in the target range	*74%*

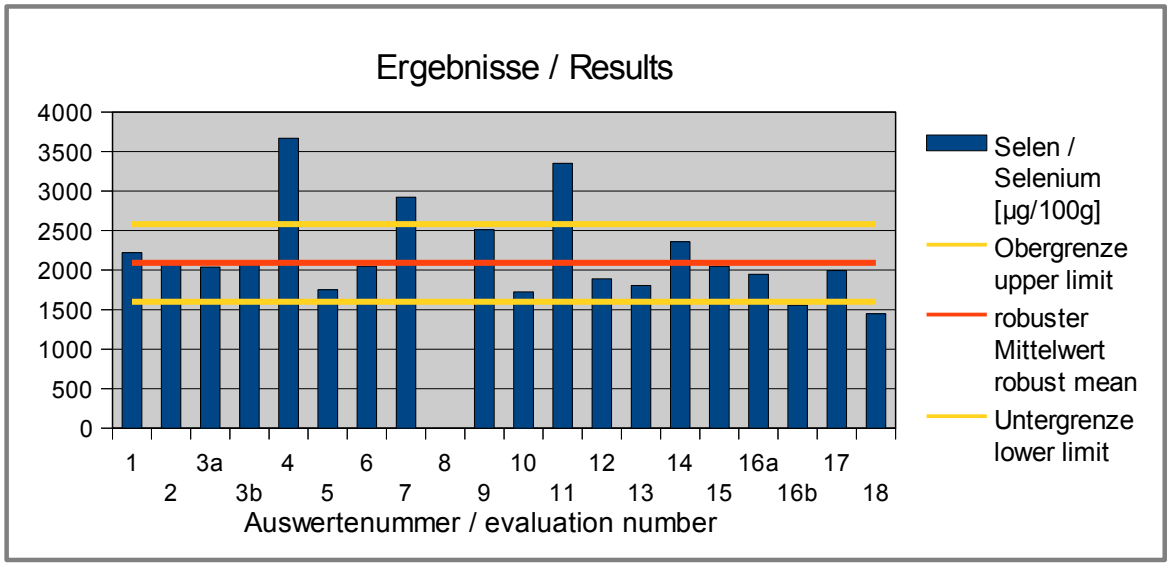


Abb. / Fig. 21: Ergebnisse Selen / Results Selenium

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Selen / Selenium [µg/100g]	Abweichung [mg/100g] Deviation [mg/100g]	z´-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	2220	129	0,53	0,84	
2	2100	9,11	0,037	0,060	
3a	2040	-50,9	-0,21	-0,33	
3b	2070	-20,9	-0,085	-0,14	
4	3669	1578	6,4	10,3	Ausreisser / Outlier
5	1750	-341	-1,4	-2,2	
6	2050	-40,9	-0,17	-0,27	
7	2923	832	3,4	5,4	
8					
9	2510	419	1,7	2,7	
10	1725	-366	-1,5	-2,4	
11	3353	1262	5,1	8,3	
12	1889	-202	-0,82	-1,3	
13	1805	-286	-1,2	-1,9	
14	2359 *	268	1,1	1,7	
15	2047	-43,9	-0,18	-0,29	
16a	1947	-144	-0,59	-0,94	
16b	1555	-535	-2,2	-3,5	
17	1993	-97,9	-0,40	-0,64	
18	1447	-644	-2,6	-4,2	

* Mean calculated by DLA

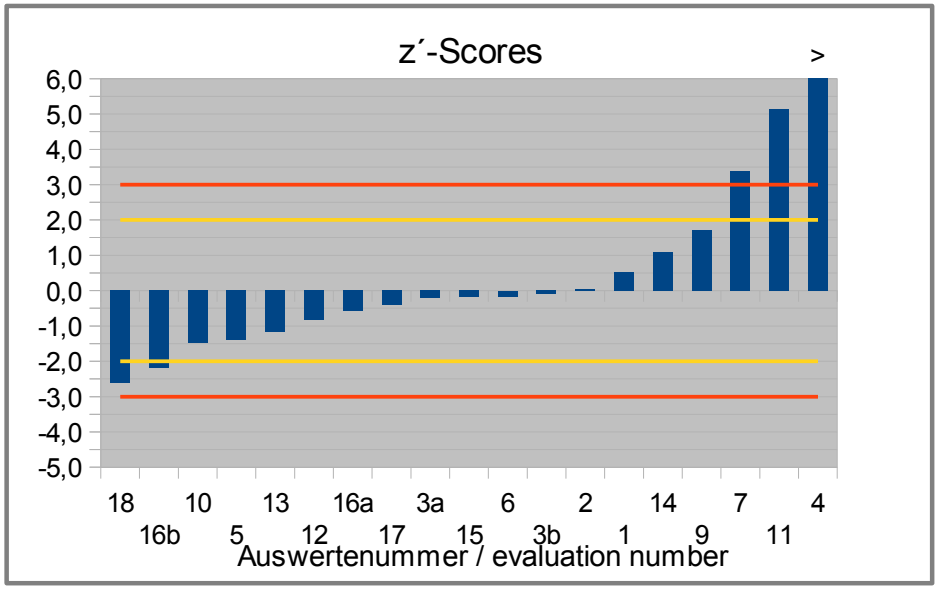


Abb. / Fig. 22: Z´-Scores Selen / Selenium

4.12 Zink / Zinc in mg/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	19
Number of outliers	2
Mean	295
Median	292
Robust Mean (X)	**296**
Robust standard deviation (S*)	**14,3**
Number with 2 replicates	16
Repeatability SD (S_r)	6,76
Repeatability (CV_r)	2,28%
Reproducibility SD (S_R)	15,2
Reproducibility (CV_R)	5,14%
Target range:	
Target standard deviation σ_{pt}	**14,2**
Target standard deviation (for Information)	19,6
lower limit of target range	**267**
upper limit of target range	**324**
Quotient S/σ_{pt}*	*1,0*
Standard uncertainty $U_{(Xpt)}$	*4,11*
Quotient $U_{(Xpt)}/\sigma_{pt}$	*0,29*
Results in the target range	*16*
Percent in the target range	*84%*

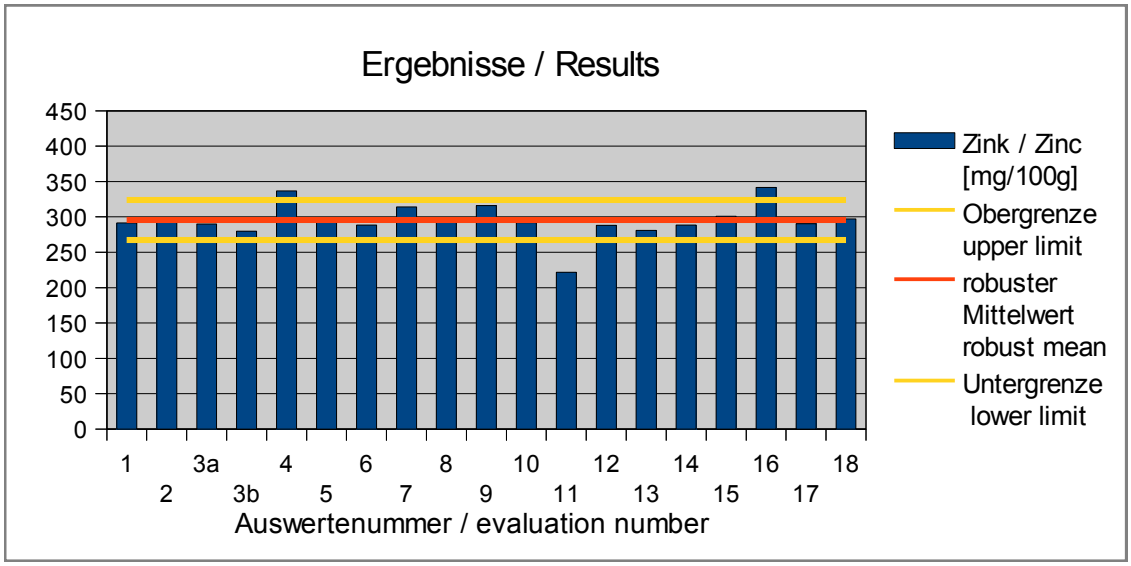


Abb. / Fig. 23: Ergebnisse Zink / Results Zinc

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Zink / Zinc [mg/100g]	Abweichung [mg/100g] Deviation [mg/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	292	-4,14	-0,29	-0,21	
2	299	3,36	0,24	0,17	
3a	290	-5,64	-0,40	-0,29	
3b	280	-15,6	-1,1	-0,80	
4	337	41,2	2,9	2,1	
5	295	-1,14	-0,08	-0,06	
6	289	-7,14	-0,50	-0,36	
7	314	18,4	1,3	0,94	
8	298	2,26	0,16	0,12	
9	316	20,4	1,4	1,0	
10	292	-3,74	-0,26	-0,19	
11	222	-74,0	-5,2	-3,8	Ausreisser / Outlier
12	288	-7,64	-0,54	-0,39	
13	281	-14,6	-1,0	-0,75	
14	289 *	-7,04	-0,50	-0,36	
15	301	5,16	0,36	0,26	
16	342	46,1	3,2	2,3	Ausreisser / Outlier
17	290	-5,44	-0,38	-0,28	
18	297	1,36	0,10	0,07	

* Mean calculated by DLA

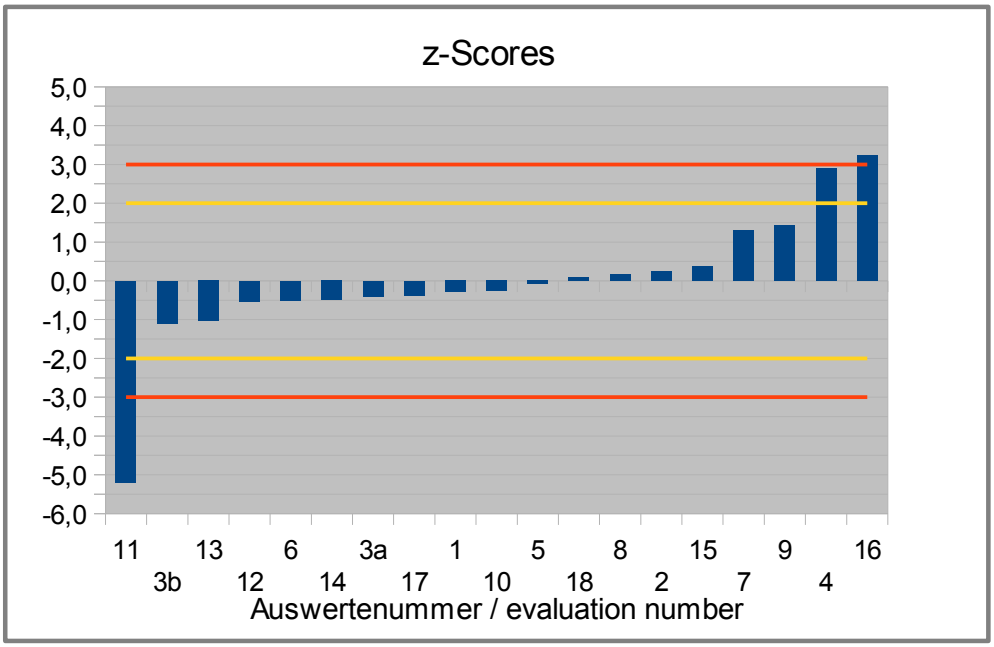


Abb. / Fig. 24: Z-Scores Zink / Zinc

5. Documentation

5.1 Details by the participants

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

5.1.1 Primary Data and analytical Methods

Parameter	Teilnehmer	Einheit	Proben-Nr. 1	Proben-Nr. 2	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis 1	Ergebnis 2	Bestim-mungsgren-ze	Inkl. WF	Wiederfin-dungsrate [%]
Analyte	**Participant**	**Unit**	**Sample No. 1**	**Sample No. 2**	**Date of analysis**	**Result (Mean)**	**Result 1**	**Result 2**	**LOQ**	**Incl. RR**	**Recovery rate [%]**
Bor / Boron	1	*mg/100g*	59	37	19.07.17	103	105	101	5ppb		
	2	*mg/100g*	19	77	15.06.17	130	130	130	0,02	no	
	3	*mg/100g*									
	4	*mg/100g*	12	84	02.08.17	96,2	97	95,4	0,1 mg/100g	no	97,7
	5	*mg/100g*	40	56	25.07.17	117,5	115	120	5		
	6	*mg/100g*									
	7	*mg/100g*	18	78	23.06.17	99,8	103	96,6		no	
	8	*mg/100g*									
	9	*mg/100g*									
	10	*mg/100g*	43	53	20.07.17	115,4	116,38	114,4	2,5	no	n.a.
	11	*mg/100g*	21	75	17.07.17	97,26	95,84	98,68			
	12	*mg/100g*									
	13	*mg/100g*	28	68	05.07.17	130	129	131	0,01	no	
	14	*mg/100g*	42	54	11.01.00	yes	133,5	135,3	0,05	no	-
	15	*mg/100g*	32	64		113,4				no	
	16	*mg/100g*	33	63	12.06.17	156,75	157,4	156,1	12,73 µg/L	yes	78,50%
	17	*mg/100g*				-	-	-			
	18	*mg/100g*	30	66	05.07.17	128	125	130	0,4	no	

Parameter	Teilnehmer	Einheit	Proben-Nr. 1	Proben-Nr. 2	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis 1	Ergebnis 2	Bestim-mungsgren-ze	Inkl. WF	Wiederfin-dungsrate [%]
Analyte	**Participant**	**Unit**	**Sample No. 1**	**Sample No. 2**	**Date of analysis**	**Result (Mean)**	**Result 1**	**Result 2**	**LOQ**	**Incl. RR**	**Recovery rate** [%]
Calcium	1	*mg/100g*	59	37	19.07.17	7829	7928	7730	5ppb		
	2	*mg/100g*	19	77	15.06.17	8370	8370	8370	5	no	
	3	*mg/100g*	34	62	17.07.17	5460	5450	5470		no	
	4	*mg/100g*	12	84	13.07.17	10232	10242	10222	8,0 mg/100g	yes	82,9
	5	*mg/100g*	40	56	25.07.17	7990	7930	8050	10		
	6	*mg/100g*	11	85	26.07.17	7623,5	7560,9	7686,2	1000	no	80-120
	7	*mg/100g*	18	78	23.06.17	8249	8157	8340		no	
	8	*mg/100g*									
	9	*mg/100g*	31	65	27.07.	7680	7493	7865	8	no	89,5
	10	*mg/100g*	43	53	27.07.17	8501	8612	8391	2,3	no	n.a.
	11	*mg/100g*	21	75	17.07.17	8345,66	8259,42	8431,89			
	12	*mg/100g*	23	73	30.06.17	7836	7822	7850		no	
	13	*mg/100g*	28	68	05.07.17	8607	8635	8579	0,01	no	
	14	*mg/100g*	42	54	11.01.00	yes	8135	8074	0,01	no	-
	15	*mg/100g*	32	64		8568,8				no	
	16	*mg/100g*	33	63	22.06.17	8801,75	8758,1	8845,4	0,172 mg/L	no	106%
	17	*mg/100g*	7	89	13.06.17	7543	7394	7691	0,1	yes	113
	18	*mg/100g*	30	66	05.07.17	8389	8293	8485	4	no	

Parameter	Teilnehmer	Einheit	Proben-Nr. 1	Proben-Nr. 2	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis 1	Ergebnis 2	Bestim-mungsgren-ze	Inkl. WF	Wiederfin-dungsrate [%]
Analyte	**Participant**	**Unit**	**Sample No. 1**	**Sample No. 2**	**Date of analysis**	**Result (Mean)**	**Result 1**	**Result 2**	**LOQ**	**Incl. RR**	**Recovery rate** [%]
Chrom / Chromium	1	*µg/100g*	59	37	19.07.17	2233	2276	2190	5ppb		
	2	*µg/100g*	19	77	04.08.17	2900	2900	2900	30	no	
	3a	*µg/100g*	34	62	17.07.17	1800	1730	1870		no	
	3b	*µg/100g*	34	62	02.08.17	1120	1170	1060		no	
	4	*µg/100g*	12	84	21.06.17	3243	3217	3269	0,1 mg/100g	no	103,2
	5	*µg/100g*	40	56	25.07.17	2250	2300	2200	100		
	6	*µg/100g*	11	85	20.07.17	2545	2530	2560	10	no	80-120
	7	*µg/100g*	18	78	23.06.17	2294	2387	2201		no	
	8	*µg/100g*	29	67	31.07.17	2409	2379	2438		no	
	9	*µg/100g*	31	65	27.07.	2330	2260	2393	100	no	115,1
	10	*µg/100g*	43	53	17.07.17	1811	1858	1765	25	no	n.a.
	11	*µg/100g*	21	75	17.07.17	1598,30	1587,30	1609,30			
	12	*µg/100g*	23	73	30.06.17	1851	1853	1849		no	
	13	*µg/100g*	28	68	05.07.17	1495	1470	1520	5	no	
	14	*µg/100g*	42	54	11.01.00	yes	2291	2292	100	no	-
	15	*µg/100g*	32	64		2324				no	
	16	*µg/100g*	33	63	09.06.17	1865,4	1852,45	1878,35	10,76 µg/L	no	102,20%
	17	*µg/100g*	7	89	13.06.17	2149	2117	2180	1	yes	99
	18	*µg/100g*	30	66	05.07.17	1650	1627	1672	0,4	no	

Parameter	Teilnehmer	Einheit	Proben-Nr. 1	Proben-Nr. 2	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis 1	Ergebnis 2	Bestim-mungsgren-ze	Inkl. WF	Wiederfin-dungsrate [%]
Analyte	**Participant**	**Unit**	**Sample No. 1**	**Sample No. 2**	**Date of analysis**	**Result (Mean)**	**Result 1**	**Result 2**	**LOQ**	**Incl. RR**	**Recovery rate [%]**
Kupfer / Copper	1	*mg/100g*	59	37	19.07.17				5ppb		
	2	*mg/100g*	19	77	04.08.17	43	44	43	0,2	no	
	3a	*mg/100g*	34	62	17.07.17	40,8	40,3	41,2		no	
	3b	*mg/100g*	34	62	02.08.17	39	39	39		no	
	4	*mg/100g*	21	84	23.06.17	56,5	57,26	55,74		yes	119,3
	5	*mg/100g*	40	56	25.07.17	42,75	42	43,5	0,55		
	6	*mg/100g*	11	85	12.07.17	40,7	41,3	40,1	0,01	no	80-120
	7	*mg/100g*	18	78	23.06.17	47,3	48,5	46		no	
	8	*mg/100g*	29	67	31.07.17	44,3	44,3	44,3		no	
	9	*mg/100g*	31	65	27.07.	45,2	44,5	45,8	0,1	no	103,4
	10	*mg/100g*	43	53	27.07.17	44,20	44,58	43,83	0,1	no	n.a.
	11	*mg/100g*	21	75	17.07.17	31,31	30,99	31,62			
	12	*mg/100g*	23	73	30.06.17	44	44	44		no	
	13	*mg/100g*	28	68	05.07.17	37,8	37,7	37,8	0,01	no	
	14	*mg/100g*	42	54	11.01.00	yes	43,6	44,5	0,05	no	-
	15	*mg/100g*	32	64		47,3				no	
	16	*mg/100g*	33	63	09.06.17	44	43,8	44,2	21,16 µg/L	no	107,80%
	17	*mg/100g*	7	89	13.06.17	43,35	42,69	44,01	0	yes	98
	18	*mg/100g*	30	66	05.07.17	43,6	43,6	43,5	0,4	no	

Parameter	Teilnehmer	Einheit	Proben-Nr. 1	Proben-Nr. 2	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis 1	Ergebnis 2	Bestim-mungsgren-ze	Inkl. WF	Wiederfin-dungsrate [%]
Analyte	**Participant**	**Unit**	**Sample No. 1**	**Sample No. 2**	**Date of analysis**	**Result (Mean)**	**Result 1**	**Result 2**	**LOQ**	**Incl. RR**	**Recovery rate [%]**
Eisen / Iron	1	*mg/100g*	59	37	19.07.17	322,5	325	320	5ppb		
	2	*mg/100g*	19	77	04.08.17	333	326	340	0,2	no	
	3a	*mg/100g*	34	62	17.07.17	306	301	310		no	
	3b	*mg/100g*	34	62	02.08.17	260	260	250		no	
	4	*mg/100g*	12	84	23.06.17	395,1	400,4	389,8	0,1 mg/100g	no	99,6
	5	*mg/100g*	40	56	25.07.17	331,5	325	338	5		
	6	*mg/100g*	11	85	05.07.17	281,2	280,7	281,6	0	no	80-120
	7	*mg/100g*	18	78	23.06.17	309	315	302		no	
	8	*mg/100g*	29	67	31.07.17	324,5	320,8	328,2		no	
	9	*mg/100g*	31	65	27.07.	349	347	351	0,05	no	86,1
	10	*mg/100g*	43	53	27.07.17	318,8	321,6	316,0	0,5	no	n.a.
	11	*mg/100g*	21	75	17.07.17	272,46	283,93	260,99			
	12	*mg/100g*	23	73	30.06.17	312	313	314		no	
	13	*mg/100g*	28	68	05.07.17	95	91,1	98,9	0,01	no	
	14	*mg/100g*	42	54	11.01.00	yes	322,5	325,5	0,03	no	-
	15	*mg/100g*	32	64		347,5				no	
	16	*mg/100g*	33	63	13.06.17	400,25	407,2	393,3	21,06 µg/L	no	107,90%
	17	*mg/100g*	7	89	13.06.17	335,9	329,9	341,9	0,01	yes	95
	18	*mg/100g*	30	66	05.07.17	333	321	345	4	no	

Parameter	Teilnehmer	Einheit	Proben-Nr. 1	Proben-Nr. 2	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis 1	Ergebnis 2	Bestim-mungsgren-ze	Inkl. WF	Wiederfin-dungsrate [%]
Analyte	**Participant**	**Unit**	**Sample No. 1**	**Sample No. 2**	**Date of analysis**	**Result (Mean)**	**Result 1**	**Result 2**	**LOQ**	**Incl. RR**	**Recovery rate** [%]
Kalium / Potassium	1	*mg/100g*	59	37	19.07.17	5259,5	5167	5352	5ppb		
	2	*mg/100g*	19	77	15.06.17	5540	5540	5540	5	no	
	3	*mg/100g*	34	62	17.07.17	3560	3500	3620		no	
	4	*mg/100g*	12	84	23.06.17	6019,5	6040	5999	6,9 mg/100g	no	97,9
	5	*mg/100g*	40	56	25.07.17	5112,5	5050	5175	20		
	6	*mg/100g*	11	85	27.07.17	5,26	5,17	5,34	0,01	no	80-120
	7	*mg/100g*	18	78	23.06.17	5731	5897	5565		no	
	8	*mg/100g*									
	9	*mg/100g*	31	65	27.07.	5870	5942	5798	5	no	98,6
	10	*mg/100g*	43	53	27.07.17	5275	5257	5292	3,6	no	n.a.
	11	*mg/100g*	21	75	17.07.17	5267,40	5396,61	5138,18			
	12	*mg/100g*	23	73	30.06.17	5529	5509	5549		no	
	13	*mg/100g*	28	68	05.07.17	5409	5436	5382	0,01	no	
	14	*mg/100g*	42	54	11.01.00	yes	5170	5175	1	no	-
	15	*mg/100g*	32	64		5248,3				no	
	16	*mg/100g*	33	63	06.07.17	5111	4929,1	5292,9	0,83mg/L	no	99,95
	17	*mg/100g*	7	89	13.06.17	5035	4903	5166	0,1	yes	106
	18	*mg/100g*	30	66	05.07.17	5216	5115	5316	40	no	

Parameter	Teilnehmer	Einheit	Proben-Nr. 1	Proben-Nr. 2	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis 1	Ergebnis 2	Bestim-mungsgren-ze	Inkl. WF	Wiederfin-dungsrate [%]
Analyte	**Participant**	**Unit**	**Sample No. 1**	**Sample No. 2**	**Date of analysis**	**Result (Mean)**	**Result 1**	**Result 2**	**Limit of de-termination**	**Incl. RR**	**Recovery rate [%]**
Magnesium	1	*mg/100g*	59	37	19.07.17	4783	4838	4728	5ppb		
	2	*mg/100g*	19	77	15.06.17	4960	4960	4960	5	no	
	3	*mg/100g*									
	4	*mg/100g*	12	84	21.06.17	6494,5	6613	6376	9,1 mg/100g	no	98,8
	5	*mg/100g*	40	56	25.07.17	4507	4550	4464	10		
	6	*mg/100g*	11	85	26.07.17	4488	4477,6	4498,4	1000	no	80-120
	7	*mg/100g*	18	78	23.06.17	5300	5466	5134		no	
	8	*mg/100g*									
	9	*mg/100g*	31	65	27.07.	5010	4973	5054	5	no	96,5
	10	*mg/100g*	43	53	27.07.17	4834	4895	4774	1,8	no	n.a.
	11	*mg/100g*	21	75	17.07.17	5564,73	5625,02	5504,43			
	12	*mg/100g*	23	73	30.06.17	4406	4439	4373		no	
	13	*mg/100g*	28	68	05.07.17	4844	4837	4850	0,01	no	
	14	*mg/100g*	42	54	11.01.00	yes	4683	4656	0,1	no	-
	15	*mg/100g*	32	64		5032,7				no	
	16	*mg/100g*	33	63	22.06.17	4777,1	4831,9	4722,3	0,01 mg/L	no	100,30%
	17	*mg/100g*	7	89	13.06.17	4410	4339	4480	0,01	yes	105
	18	*mg/100g*	30	66	05.07.17	4768	4761	4775	4	no	

Parameter	Teilnehmer	Einheit	Proben-Nr. 1	Proben-Nr. 2	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis 1	Ergebnis 2	Bestim-mungsgren-ze	Inkl. WF	Wiederfin-dungsrate [%]
Analyte	**Participant**	**Unit**	**Sample No. 1**	**Sample No. 2**	**Date of analysis**	**Result (Mean)**	**Result 1**	**Result 2**	**LOQ**	**Incl. RR**	**Recovery rate [%]**
Mangan / Manganese	1	*mg/100g*	59	37	19.07.17	67,55	68,6	66,5	5ppb		
	2	*mg/100g*	19	77	04.08.17	69	68	71	0,03	no	
	3a	*mg/100g*	34	62	17.07.17	61	61	61		no	
	3b	*mg/100g*	34	62	02.08.17	46	47	44		no	
	4	*mg/100g*	12	84	13.07.17	78,24	78,58	77,9	0,1 mg/100g	no	95,2
	5	*mg/100g*	40	56	25.07.17	70,25	70,9	69,6	1		
	6	*mg/100g*									
	7	*mg/100g*	18	78	23.06.17	72,9	75,5	70,2		no	
	8	*mg/100g*									
	9	*mg/100g*	31	65	27.07.	69,5	68,3	70,7	0,05	no	92,3
	10	*mg/100g*	43	53	17.07.17	63,56	63,56	63,55	0,03	no	n.a.
	11	*mg/100g*	21	75	17.07.17	50,19	50,61	49,77			
	12	*mg/100g*	23	73	30.06.17	68	68	68		no	
	13	*mg/100g*	28	68	05.07.17	69,75	69,8	69,7	0,01	no	
	14	*mg/100g*	42	54	11.01.00	yes	63,3	63,7	0,02	no	-
	15	*mg/100g*	32	64		75,7				no	
	16	*mg/100g*	33	63	13.06.17	91,25	91,9	90,6	7,06 µg/L	no	107,80%
	17	*mg/100g*	7	89	13.06.17	68,24	68,94	67,54	0	yes	99
	18	*mg/100g*	30	66	05.07.17	63,6	62,4	64,8	0,4	no	

Parameter	Teilnehmer	Einheit	Proben-Nr. 1	Proben-Nr. 2	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis 1	Ergebnis 2	Bestim-mungsgren-ze	Inkl. WF	Wiederfin-dungsrate [%]
Analyte	**Participant**	**Unit**	**Sample No. 1**	**Sample No. 2**	**Date of analysis**	**Result (Mean)**	**Result 1**	**Result 2**	**LOQ**	**Incl. RR**	**Recovery rate** [%]
Molybdän / Molybdenum	1	*µg/100g*	59	37	20.07.17	1310	1330	1290	5ppb		
	2	*µg/100g*	19	77	15.06.17	1200	1200	1200	20	no	
	3	*µg/100g*									
	4	*µg/100g*	12	84	13.07.17	1901	1893	1909	0,2 mg/100g	yes	72
	5	*µg/100g*	40	56	25.07.17	1500	1400	1600	100		
	6	*µg/100g*									
	7	*µg/100g*	18	78	23.06.17	1230	1290	1170		no	
	8	*µg/100g*	29	67	31.07.17	2353	2391	2314		no	
	9	*µg/100g*	31	65	27.07.	907	907	907	100	no	107,6
	10	*µg/100g*	43	53	17.07.17	1162	1194	1131	25	no	n.a.
	11	*µg/100g*	21	75	17.07.17	1247,55	1252,80	1242,30			
	12	*µg/100g*	23	73	30.06.17	1129	1110	1148		no	
	13	*µg/100g*	28	68	05.07.17	999	999	998	5	no	
	14	*µg/100g*	42	54	11.01.00	yes	1111	1118	100	no	-
	15	*µg/100g*	32	64		1257				no	
	16	*µg/100g*	33	63							
	17	*µg/100g*	7	89	13.06.17	1126	1115	1137	1	yes	100
	18	*µg/100g*	30	66	05.07.17	655	664	645	0,4	no	

Parameter	Teilnehmer	Einheit	Proben-Nr. 1	Proben-Nr. 2	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis 1	Ergebnis 2	Bestim-mungsgren-ze	Inkl. WF	Wiederfin-dungsrate [%]
Analyte	**Participant**	**Unit**	**Sample No. 1**	**Sample No. 2**	**Date of analysis**	**Result (Mean)**	**Result 1**	**Result 2**	**LOQ**	**Incl. RR**	**Recovery rate [%]**
Phosphor / Phosphorus	1	*mg/100g*	59	37	19.07.17	4894,5	4922	4867	5ppb		
	2	*mg/100g*	19	77	15.06.17	5370	5370	5370	2	no	
	3	*mg/100g*									
	4	*mg/100g*	12	84	13.07.17	6360,5	6366	6355	1,5 mg/100g	yes	88,8
	5	*mg/100g*	40	56	25.07.17	5546	5528	5564	20		
	6	*mg/100g*									
	7	*mg/100g*									
	8	*mg/100g*									
	9	*mg/100g*	31	65	27.07.	5470	5392	5544	0,1	no	108,2
	10	*mg/100g*	43	53	27.07.17	5194	5236	5152	3,6	no	n.a.
	11	*mg/100g*	21	75	17.07.17	5969,62	6018,62	5920,61			
	12	*mg/100g*									
	13	*mg/100g*	28	68	05.07.17	5233	5228	5237	0,01	no	
	14	*mg/100g*	42	54	11.01.00	yes	5166	5156	0,3	no	-
	15	*mg/100g*	32	64		5212,7				no	
	16	*mg/100g*	33	63	22.06.17	5300,1	5289,1	5311,1	0,67 mg/L	no	102,50%
	17	*mg/100g*	7	89	13.06.17	5120	5003	5236	0,1	yes	106
	18	*mg/100g*	30	66	05.07.17	5152	5092	5212	4	no	

Parameter	Teilnehmer	Einheit	Proben-Nr. 1	Proben-Nr. 2	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis 1	Ergebnis 2	Bestim-mungsgren-ze	Inkl. WF	Wiederfin-dungsrate [%]
Analyte	**Participant**	**Unit**	**Sample No. 1**	**Sample No. 2**	**Date of analysis**	**Result (Mean)**	**Result 1**	**Result 2**	**LOQ**	**Incl. RR**	**Recovery rate** [%]
Selen / Selenium	1	*µg/100g*	59	37	20.07.17	2220	2210	2230	5ppb		
	2	*µg/100g*	19	77	04.08.17	2100	2100	2100	120	no	
	3a	*µg/100g*	34	62	17.07.17	2040	2020	2060		no	
	3b	*µg/100g*	34	62	02.08.17	2070	2040	2090		no	
	4	*µg/100g*	12	84	03.08.17	3668,5	3696	3641	1,0 µg/100g	yes	75
	5	*µg/100g*	40	56	25.07.17	1750	1700	1800	100		
	6	*µg/100g*	11	85	25./27.07. 2017	2050	2030	2070	20	no	80-120
	7	*µg/100g*	18	78	23.06.17	2923	3115	2731		no	
	8	*µg/100g*									
	9	*µg/100g*	31	65	16.06.	2510	2495	2516	0,4	no	113,4
	10	*µg/100g*	43	53	17.07.17	1725	1773	1676	25	no	n.a.
	11	*µg/100g*	21	75	17.07.17	3352,95	3342,40	3363,50			
	12	*µg/100g*	23	73	30.06.17	1889	1887	1891		no	
	13	*µg/100g*	28	68	05.07.17	1805	1800	1810	5	no	
	14	*µg/100g*	42	54	11.01.00	yes	2349	2368	1000	no	-
	15	*µg/100g*	32	64		2047				no	
	16a	*µg/100g*	33	63	13.06.17	1946,65	1907,9	1985,4	6,60 µg/L	no	103,26%
	16b	*µg/100g*	33	63	12.06.17	1555,4	1531,7	1579,1	2,46 µgL	yes	112,5
	17	*µg/100g*	7	89	13.06.17	1993	1973	2012	1	yes	101
	18	*µg/100g*	30	66	05.07.17	1447	1467	1427	0,8	no	

Parameter	Teilnehmer	Einheit	Proben-Nr. 1	Proben-Nr. 2	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis 1	Ergebnis 2	Bestim-mungsgren-ze	Inkl. WF	Wiederfin-dungsrate [%]
Analyte	**Participant**	**Unit**	**Sample No. 1**	**Sample No. 2**	**Date of analysis**	**Result (Mean)**	**Result 1**	**Result 2**	**LOQ**	**Incl. RR**	**Recovery rate [%]**
Zink / Zinc	1	*mg/100g*	59	37	19.07.17	291,5	294	289	5ppb		
	2	*mg/100g*	19	77	04.08.17	299	294	304	0,2	no	
	3a	*mg/100g*	34	62	17.07.17	290	290	290		no	
	3b	*mg/100g*	34	62	02.08.17	280	280	280		no	
	4	*mg/100g*	12	84	23.06.17	336,8	349,6	324	0,1 mg/100g	no	90,4
	5	*mg/100g*	40	56	25.07.17	294,5	296	293	1		
	6	*mg/100g*	11	85	17.07.17	288,5	284,4	292,6	0,01	no	80-120
	7	*mg/100g*	18	78	23.06.17	314	324	303		no	
	8	*mg/100g*	29	67	31.07.17	297,9	292,4	303,4		no	
	9	*mg/100g*	31	65	27.07.	316	316	316	0,1	no	97,5
	10	*mg/100g*	43	53	27.07.17	291,9	293,5	290,2	0,5	no	n.a.
	11	*mg/100g*	21	75	17.07.17	221,67	216,15	227,18			
	12	*mg/100g*	23	73	30.06.17	288	288	288		no	
	13	*mg/100g*	28	68	05.07.17	281	281	281	0,01	no	
	14	*mg/100g*	42	54	11.01.00	yes	289,7	287,5	0,05	no	-
	15	*mg/100g*	32	64		300,8				no	
	16	*mg/100g*	33	63	13.06.17	341,75	344,3	339,2	7,94 µg/L	no	104,40%
	17	*mg/100g*	7	89	13.06.17	290,2	287,8	292,6	0,01	yes	96
	18	*mg/100g*	30	66	05.07.17	297	296	298	4	no	

5.1.2 Analytical Methods

Parameter	Teilnehmer	Methodenbeschreibung	Homoge-nisierung	Einwaage	Aufschluss: Metho-de	Aufschluss: Lö-sung	Kalibrierverfahren/ Referenzmaterial	Methode ak-kreditiert	Sonstige Hinweise
Analyte	Participant	Method description	Homoge-nization	Sample weight	Hydrolization Me-thod	Hydrolization Solu-tion	Calibration and re-ference material	Method ac-credited	Further remarks
Bor / Boron	1	AOAC 993.14		0.25g	Microwave	5% H NO3	NIST	Yes	
	2	ICP-OES	NO	0,5 g	Digestion with microwave oven	8 ml HNO3 (65%)+ 1 ml H2O2 (30%)	Monoelemental standard (1000 mg/l)	no	
	3								
	4	In-house method		0,5 g	Microwave pressure digestion		AQS 1/17 A5	yes	
	5	EN 15763		0,4 g	Microwave pressure digestion	HNO3 / H2O2		yes	
	6								
	7	ICP-MS		0.4 g	Mikrowave	HNO3/H2O2	external Standard	no (request in process)	
	8								
	9								not determined
	10	ICP-OES			microwave destruction	HNO3 and H2O2	0-250-500-1000 µg/l	no	RM: BCR, in house RM including old DLA PT material
	11	AOAC 2015.01, AOAC 2015.06						yes	
	12								
	13	USP-35 NF 30	yes	0,4g	Turbowave Microwave	HNO3	6-Point-Calibration	yes	
	14	ICP-OES	no	200mg	Mikrowave	HNO3 / H2O2 / H2O	external Calibration	yes	
	15	DIN EN 15621		1g		Nitric acid		yes	
	16	SOP 504-0230	shaking	0,5g	Microwave pressure digestion (H2O / HNO3 / H2O2)	100 mL		no	ICP-OES
	17								
	18	ASU L 00.00-144	yes	0,5 g	ASU L 00.00-19/1	HNO3		yes	

Parameter	Teilnehmer	Methodenbeschreibung	Homogenisierung	Einwaage	Aufschluss: Methode	Aufschluss: Lösung	Kalibrierverfahren/ Referenzmaterial	Methode akkreditiert	Sonstige Hinweise
Analyte	Participant	Method description	Homogenization	Sample weight	Hydrolization Method	Hydrolization Solution	Calibration and reference material	Method accredited	Further remarks
Calcium	1	AOAC 993.14		0.25g	Microwave	5% H NO3	NIST	Yes	
	2	ICP-OES	NO	0,5 g	Digestion with microwave oven	8 ml HNO3 (65%)+ 1 ml H2O2 (30%)	Monoelemental standard (1000 mg/l)	no	
	3	Total x- ray fluoreszence analysis by in-house-method	manual mixing	100 mg	Wet milling process by ball mill	20%ige HNO_3	internal Standard / Gallium	no	
	4	ASU L00.00-144		1 g	Microwave pressure digestion		muva-NEM 1603	Yes	
	5	EN 15763		0,4 g	Microwave pressure digestion	HNO3 / H2O2		Yes	
	6	Incineration at 550°C, measurement via AAS using ASU L 31.00 - 10	stirring	1,0184 g (A), 1,1293 g (B)	Incineration at 550°C		external	Yes	
	7	ICP-MS		0.4 g	Microwave	HNO3/H2O2	external Standard	no (request in process)	
	8								
	9	ASU § 64 L 00.00-144	yes	0,5	Microwave pressure digestion	Nitric acid	Standardsolution/ SRM 1547	Yes	
	10	ICP-OES			microwave destruction	HNO3 and H2O2	0-1000-5000-10000 µg/l	yes	
	11	AOAC 2015.01, AOAC 2015.06						yes	
	12	AA53, ICP-MS	ball mill	1g	AA30	HNO3, H2O2	NIST SRM 3280	Yes	
	13	USP-35 NF 30	yes	0,4g	Turbowave Microwave	HNO3	6-Point-Calibration	Yes	
	14	ICP-OES	NO	200mg	Microwave	HNO3 / H2O2 / H2O	external Calibration	Yes	
	15	DIN EN 15621		1g		Nitric acid		Yes	
	16	SOP 504-0230	shaking	0,5g	Microwave pressure digestion (H2O / HNO3 / H2O2)	100 mL		no	ICP-OES
	17	EN 15763 mod.; DIN 17294-2 mod.	mixing	0,5-1g	Microwave pressure digestion	8ml HNO3 + 2 ml H2O2	external / Ultra Scientific	Yes	
	18	ASU L 00.00-144	yes	0,5 g	ASU L 00.00-19/1	HNO3		Yes	

Parameter	Teilnehmer	Methodenbeschreibung	Homoge-nisierung	Einwaage	Aufschluss: Methode	Aufschluss: Lö-sung	Kalibrierverfahren/Re ferenzmaterial	Methode ak-kreditiert	Sonstige Hinweise
Analyte	**Participant**	**Method description**	**Homoge-nization**	**Sample weight**	**Hydrolization Method**	**Hydrolization Solu-tion**	**Calibration and refe-rence material**	**Method ac-credited**	**Further remarks**
Chrom / Chromium	1	AOAC 993.14		0.25g	Microwave	5% H NO3	NIST	Yes	
	2	ICP-OES	NO	0,5 g	Digestion with microwave oven	8ml HNO3 (65%)+ 2 ml HCl (37%)	Monoelemental standard (1000 mg/l)	no	
	3a	Total x- ray fluoreszence analysis by in-house-method	manual mixing	100 mg	wet milling process with ball mill	20%ige HNO_3	internal Standard / Gallium	no	
	3b	Total x- ray fluoreszence analysis by in-house-method	manual mixing	100 mg	acidic diegstion	65%ige HNO_3	internal Standard / Gallium	no	
	4	In-house-method		1 g	Microwave pressure digestion		LGC7162	Yes	
	5	EN 15763		0,4 g	Microwave pressure digestion	HNO3 / H2O2		Yes	
	6	ASU L 00.00.-19/3	stirring	0,5055 g (A), 0,50627 g (B)	ASU L 00.00-19/1		external	Yes	
	7	ICP-MS		0.4 g	Microwave	HNO3/H2O2	external Standard	no (request in process)	
	8	UNI EN 14083	yes	300 mg	HNO3 + H2O2 conc. - 200 °C	HNO3 0,1 %	Fluka 02733 batch BCBN6619V	yes	
	9	ASU § 64 L 00.00-144	yes	0,5	Microwave pressure digestion	Nitric acid	Standardsolution/ TM 25.4	Yes	
	10	ICP-MS			microwave destruction	HNO3 and H2O2	0-10-20-50-100-200 µg/l	no	
	11	AOAC 2015.01, AOAC 2015.06						yes	
	12	AA53, ICP-MS	ball mill	1g	AA30	HNO3, H2O2	NIST SRM 3280	Yes	
	13	USP-35 NF 30	yes	0,4g	Turbowave Microwave	HNO3	6-Point-Calibration	Yes	
	14	ICP-OES	no	200mg	Microwave	HNO3 / H2O2 / H2O	external Calibration	Yes	
	15	DIN EN 15765		1g		Nitric acid		Yes	
	16	SOP 504-0230	shaking	0,5g	Microwave pressure digestion (H2O / HNO3 / H2O2)	100 mL		no	ICP-OES
	17	EN 15763 mod.; DIN 17294-2 mod.	mixing	0,5-1g	Microwave pressure digestion	8ml HNO3 + 2 ml H2O2	external / Ultra Scientific	Yes	
	18	ASU L 00.00-144	yes	0,5 g	ASU L 00.00-19/1	HNO3		Yes	

Parameter	Teilnehmer	Methodenbeschreibung	Homogenisierung	Einwaage	Aufschluss: Methode	Aufschluss: Lösung	Kalibrierverfahren/ Referenzmaterial	Methode akkreditiert	Sonstige Hinweise
Analyte	**Participant**	**Method description**	**Homogenization**	**Sample weight**	**Hydrolization Method**	**Hydrolization Solution**	**Calibration and reference material**	**Method accredited**	**Further remarks**
Kupfer / Copper	1	AOAC 993.14		0.25g	Microwave	5% H NO3	NIST		
	2	ICP-OES	no	0,5 g	Digestion with microwave oven	8ml HNO3 (65%)+ 2 ml HCl (37%)	Monoelemental standard (1000 mg/l)	no	
	3a	Total x- ray fluoreszence analysis by in-house-method	manual mixing	100 mg	wet milling process with ball mill	20%ige HNO_3	internal Standard / Gallium	no	
	3b	Total x- ray fluoreszence analysis by in-house-method	manual mixing	100 mg	acidic diegstion	65%ige HNO_3	internal Standard / Gallium	no	
	4	ASU L00.00-144		1 g	Microwave pressure digestion		muva-NEM 1603	yes	
	5	EN 15763		0,4 g	Microwave pressure digestion	HNO3 / H2O2		yes	
	6	Incineration at 400°C, Measurement via Flame-AAS by ASU L 00.00-19/2	stirring	1,0068 g (A), 1,0015 g (B)	Incineration at 400°C		external	yes	
	7	ICP-MS		0.4 g	Mikrowelle	HNO3/H2O2	external Standard	no (request in process)	
	8	UNI EN 14084	yes	300 mg	HNO3 + H2O2 conc. - 200 °C	HNO3 0,1 %	Sigma 38996 batch BCBS0415V	yes	
	9	ASU § 64 L 00.00-144	yes	0,5	Microwave pressure digestion	Nitric acid	Standardsolution/ SRM 1547	yes	
	10	ICP-OES			microwave destruction	HNO3 and H2O2	0-100-250-500 µg/l	yes	
	11	AOAC 2015.01, AOAC 2015.06						yes	
	12	AA53, ICP-MS	ball mill	1g	AA30	HNO3, H2O2	NIST SRM 3280	yes	
	13	USP-35 NF 30	ja	0,4g	Turbowave Microwave	HNO3	6-Point-Calibration	yes	
	14	ICP-OES	no	200mg	Microwave	HNO3 / H2O2 / H2O	external Calibration	yes	
	15	DIN EN 15621		1g		Nitric acid		yes	
	16	SOP 504-0230	shaking	0,5g	Microwave pressure digestion (H2O / HNO3 / H2O2)	100mL		no	ICP-OES
	17	EN 15763 mod.; DIN 17294-2 mod.	mixing	0,5-1g	Microwave pressure digestion	8ml HNO3 + 2 ml H2O2	external / Ultra Scientific	yes	
	18	ASU L 00.00-144	yes	0,5 g	ASU L 00.00-19/1	HNO3		yes	

Parameter	Teilnehmer	Methodenbeschreibung	Homogenisierung	Einwaage	Aufschluss: Methode	Aufschluss: Lösung	Kalibrierverfahren/ Referenzmaterial	Methode akkreditiert	Sonstige Hinweise
Analyte	**Participant**	**Method description**	**Homogenization**	**Sample weight**	**Hydrolization Method**	**Hydrolization Solution**	**Calibration and reference material**	**Method accredited**	**Further remarks**
Eisen / Iron	1	AOAC 993.14		0.25g	Microwave	5% H NO3	NIST	Yes	
	2	ICP-OES	no	0,5 g	Digestion with microwave oven	8ml HNO3 (65%)+ 2 ml HCl (37%)	Monoelemental standard (1000 mg/l)	no	
	3a	Total x- ray fluoreszence analysis by in-house-method	manual mixing	100 mg	wet milling process with ball mill	20%ige HNO_3	internal Standard / Gallium	no	
	3b	Total x- ray fluoreszence analysis by in-house-method	manual mixing	100 mg	acidic diegstion	65%ige HNO_3	internal Standard / Gallium	no	
	4	ASU L00.00-144		1 g	Microwave pressure digestion		muva-NEM 1603	Yes	
	5	EN 15763		0,4 g	Microwave pressure digestion	HNO3 / H2O2		Yes	
	6	Incineration at 400°C, Measurement via Flame-AAS by ASU L 00.00-19/2	stirring	1,0068 g (A), 1,0015 g (B)	Incineration at 400°C		external	Yes	
	7	ICP-MS		0.4 g	Mikrowelle	HNO3/H2O2	external Standard	no (request in process)	
	8	UNI EN 14084	yes	300 mg	HNO3 + H2O2 conc. - 200 °C	HNO3 0,1 %	Fluka 16596 batch BCBS2569V	yes	
	9	ASU § 64 L 00.00-144	yes	0,5	Microwave pressure digestion	Nitric acid	Standardsolution/ SRM 1547	Yes	
	10	ICP-OES			microwave destruction	HNO3 and H2O2	0-100-250-500 µg/l	yes	
	11	AOAC 2015.01, AOAC 2015.06						yes	
	12	AA53, ICP-MS	ball mill	1g	AA30	HNO3, H2O2	NIST SRM 3280	Yes	
	13	USP-35 NF 30	yes	0,4g	Turbowave Microwave	HNO3	6-Point-Calibration	Yes	
	14	ICP-OES	no	200mg	Microwave	HNO3 / H2O2 / H2O	external Calibration	Yes	
	15	DIN EN 15621		1g		Nitric acid		Yes	
	16	SOP 504-0230	shaking	0,5g	Microwave pressure digestion (H2O / HNO3 / H2O2)	100 mL		Yes	ICP-OES
	17	EN 15763 mod.; DIN 17294-2 mod.	mixing	0,5-1g	Microwave pressure digestion	8ml HNO3 + 2 ml H2O2	external / Ultra Scientific	Yes	
	18	ASU L 00.00-144	yes	0,5 g	ASU L 00.00-19/1	HNO3 / HCl		Yes	

Parameter	Teilnehmer	Methodenbeschreibung	Homogenisierung	Einwaage	Aufschluss: Methode	Aufschluss: Lösung	Kalibrierverfahren/ Referenzmaterial	Methode akkreditiert	Sonstige Hinweise
Analyte	**Participant**	**Method description**	**Homogenization**	**Sample weight**	**Hydrolization Method**	**Hydrolization Solution**	**Calibration and reference material**	**Method accredited**	**Further remarks**
Kalium / Potassium	1	AOAC 993.14		0.25g	Microwave	5% H NO3	NIST	Yes	
	2	ICP-OES	no	0,5 g	Digestion with microwave oven	8 ml HNO3 (65%)+ 1 ml H2O2 (30%)	Monoelemental standard (1000 mg/l)	no	
	3	Total x- ray fluoreszence analysis by in-house-method	manual mixing	100 mg	wet milling process with ball mill	20%ige HNO_3	internal Standard / Gallium	no	
	4	ASU L00.00-144		1 g	Microwave pressure digestion		muva-NEM 1603	Yes	
	5	EN 15763		0,4 g	Microwave pressure digestion	HNO3 / H2O2		Yes	
	6	Incineration at 400°C, Measurement via Flame-AAS by ASU L 00.00-19/2	stirring	1,0068 g (A), 1,0015 g (B)	Incineration at 400°C		external	Yes	
	7	ICP-MS		0.4 g	Microwave	HNO3/H2O2	external Standard	no (request in process)	
	8								
	9	ASU § 64 L 00.00-144	yes	0,5	Microwave pressure digestion	Nitric acid	Standardsolution/ SRM 1547	Yes	
	10	ICP-OES			microwave destruction	HNO3 and H2O2	0-1000-5000-10000 µg/l	yes	
	11	AOAC 2015.01, AOAC 2015.06						yes	
	12	AA53, ICP-MS	ball mill	1g	AA30	HNO3, H2O2	NIST SRM 3280	Yes	
	13	USP-35 NF 30	yes	0,4g	Turbowave Microwave	HNO3	6-Point-Calibration	Yes	
	14	ICP-OES	no	200mg	Microwave	HNO3 / H2O2 / H2O	External Calibration	Yes	
	15	DIN EN 15621		1g		Nitric acid		Yes	
	16	SOP 504-0230	shaking	0,5g	Microwave pressure digestion (H2O / HNO3 / H2O2)	100 mL		no	ICP-OES
	17	EN 15763 mod.; DIN 17294-2 mod.	mixing	0,5-1g	Microwave pressure digestion	8ml HNO3 + 2 ml H2O2	external / Ultra Scientific	Yes	
	18	ASU L 00.00-144	yes	0,5 g	ASU L 00.00-19/1	HNO3		Yes	

Parameter	Teilnehmer	Methodenbeschreibung	Homogenisierung	Einwaage	Aufschluss: Methode	Aufschluss: Lösung	Kalibrierverfahren/ Referenzmaterial	Methode akkreditiert	Sonstige Hinweise
Analyte	**Participant**	**Method description**	**Homogenization**	**Sample weight**	**Hydrolization Method**	**Hydrolization Solution**	**Calibration and reference material**	**Method accredited**	**Further remarks**
Magnesium	1	AOAC 993.14		0.25g	Microwave	5% H NO3	NIST	Yes	
	2	ICP-OES	no	0,5 g	Digestion with microwave oven	8 ml HNO3 (65%)+ 1 ml H2O2 (30%)	Monoelemental standard (1000 mg/l)	no	
	3								
	4	ASU L00.00-144		1 g	Microwave pressure digestion		muva-NEM 1603	Yes	
	5	EN 15763		0,4 g	Microwave pressure digestion	HNO3 / H2O2		Yes	
	6	Incineration at 550°C, Measurement by AAS using ASU L 31.00 – 10	stirring	1,0184 g (A), 1,1293 g (B)	Incineration at 550°C		external	Yes	
	7	ICP-MS		0.4 g	Microwave	HNO3/H2O2	external Standard	no (request in process)	
	8								
	9	ASU § 64 L 00.00-144	yes	0,5	Microwave pressure digestion	Nitric acid	Standardsolution/ SRM 1547	Yes	
	10	ICP-OES			microwave destruction	HNO3 and H2O2	0-1000-5000-10000 µg/l	yes	
	11	AOAC 2015.01, AOAC 2015.06						yes	
	12	AA53, ICP-MS	ball mill	1g	AA30	HNO3, H2O2	NIST SRM 3280	Yes	
	13	USP-35 NF 30	yes	0,4g	Turbowave Microwave	HNO3	6-Point-Calibration	Yes	
	14	ICP-OES	no	200mg	Microwave	HNO3 / H2O2 / H2O	external Calibration	Yes	
	15	DIN EN 15621		1g		Nitric acid		Yes	
	16	SOP 504-0230	shaking	0,5g	Microwave pressure digestion (H2O / HNO3 / H2O2)	100 mL		no	ICP-OES
	17	EN 15763 mod.; DIN 17294-2 mod.	mixing	0,5-1g	Microwave pressure digestion	8ml HNO3 + 2 ml H2O2	external / Ultra Scientific	Yes	
	18	ASU L 00.00-144	yes	0,5 g	ASU L 00.00-19/1	HNO3		Yes	

Parameter	Teilnehmer	Methodenbeschreibung	Homogenisierung	Einwaage	Aufschluss: Methode	Aufschluss: Lösung	Kalibrierverfahren/ Referenzmaterial	Methode akkreditiert	Sonstige Hinweise
Analyte	**Participant**	**Method description**	**Homogenization**	**Sample weight**	**Hydrolization Method**	**Hydrolization Solution**	**Calibration and reference material**	**Method accredited**	**Further remarks**
Mangan / Manganese	1	AOAC 993.14		0.25g	Microwave	5% H NO3	NIST	Yes	
	2	ICP-OES	no	0,5 g	Digestion with microwave oven	8ml HNO3 (65%)+ 2 ml HCl (37%)	Monoelemental standard (1000 mg/l)	no	
	3a	Total x- ray fluoreszence analysis by in-house-method	manual mixing	100 mg	wet milling process with ball mill	20%ige HNO_3	internal Standard / Gallium	no	
	3b	Total x- ray fluoreszence analysis by in-house-method	manual mixing	100 mg	acidic diegstion	65%ige HNO_3	internal Standard / Gallium	no	
	4	ASU L00.00-144		1 g	Microwave pressure digestion		muva-NEM 1603	Yes	
	5	EN 15763		0,4 g	Microwave pressure digestion	HNO3 / H2O2		Yes	
	6								
	7	ICP-MS		0.4 g	Microwave	HNO3/H2O2	external Standard	no (request in process)	
	8								
	9	ASU § 64 L 00.00-144	yes	0,5	Microwave pressure digestion	Nitric acid	Standardsolution/ SRM 1547	Yes	
	10	ICP-MS			microwave destruction	HNO3 and H2O2	0-100-250-500 µg/l	yes	
	11	AOAC 2015.01, AOAC 2015.06						yes	
	12	AA53, ICP-MS	ball mill	1g	AA30	HNO3, H2O2	NIST SRM 3280	Yes	
	13	USP-35 NF 30	yes	0,4g	Turbowave Microwave	HNO3	6-Point-Calibration	Yes	
	14	ICP-OES	no	200mg	Microwave	HNO3 / H2O2 / H2O	external Calibration	Yes	
	15	DIN EN 15621		1g		Nitric acid		Yes	
	16	SOP 504-0230	shaking	0,5g	Microwave pressure digestion (H2O / HNO3 / H2O2)	100 mL		no	ICP-OES
	17	EN 15763 mod.; DIN 17294-2 mod.	mixing	0,5-1g	Microwave pressure digestion	8ml HNO3 + 2 ml H2O2	external / Ultra Scientific	Yes	
	18	ASU L 00.00-144	yes	0,5 g	ASU L 00.00-19/1	HNO3		Yes	

Parameter	Teilnehmer	Methodenbeschreibung	Homogenisierung	Einwaage	Aufschluss: Methode	Aufschluss: Lösung	Kalibrierverfahren/ Referenzmaterial	Methode akkreditiert	Sonstige Hinweise
Analyte	**Participant**	**Method description**	**Homogenization**	**Sample weight**	**Hydrolization Method**	**Hydrolization Solution**	**Calibration and reference material**	**Method accredited**	**Further remarks**
Molybdän / Molybdenum	1	AOAC 993.14		0.25g	Microwave	5% H NO3	NIST	Yes	
	2	ICP-OES	no	0,5 g	Digestion with microwave oven	8 ml HNO3 (65%)+ 1 ml H2O2 (30%)	Monoelemental standard (1000 mg/l)	no	
	3								
	4	In-house-method		1 g	Microwave pressure digestion		LGC7162	Yes	
	5	EN 15763		0,4 g	Microwave pressure digestion	HNO3 / H2O2		Yes	
	6								
	7	ICP-MS		0.4 g	Microwave	HNO3/H2O2	external Standard	no (request in process)	
	8	UNI EN 14083	yes	300 mg	HNO3 + H2O2 conc. - 200 °C	HNO3 0,1 %	Fluka 67210 batch BCBN4955V	yes	
	9	ASU § 64 L 00.00-144	yes	0,5	Microwave pressure digestion	Nitric acid	Standardsolution/ SRM 1547	Yes	
	10	ICP-MS			microwave destruction	HNO3 and H2O2	0-10-20-50-100-200 µg/l	no	
	11	AOAC 2015.01, AOAC 2015.06						yes	
	12	AA53, ICP-MS	ball mill	1g	AA30	HNO3, H2O2	NIST SRM 3280	Yes	
	13	USP-35 NF 30	yes	0,4g	Turbowave Microwave	HNO3	6-Point-Calibration	Yes	
	14	ICP-OES	no	200mg	Microwave	HNO3 / H2O2 / H2O	external Calibration	Yes	
	15	DIN EN 15765		1g		Nitric acid		Yes	
	16								
	17	EN 15763 mod.; DIN 17294-2 mod.	mixing	0,5-1g	Microwave pressure digestion	8ml HNO3 + 2 ml H2O2	external / Ultra Scientific	Yes	
	18	ASU L 00.00-144	yes	0,5 g	ASU L 00.00-19/1	HNO3		Yes	

Parameter	Teilnehmer	Methodenbeschreibung	Homogenisierung	Einwaage	Aufschluss: Methode	Aufschluss: Lösung	Kalibrierverfahren/ Referenzmaterial	Methode akkreditiert	Sonstige Hinweise
Analyte	**Participant**	**Method description**	**Homogenization**	**Sample weight**	**Hydrolization Method**	**Hydrolization Solution**	**Calibration and reference material**	**Method accredited**	**Further remarks**
Phosphor / Phosphorus	1	AOAC 993.14		0.25g	Microwave	5% H NO3	NIST	Yes	
	2	ICP-OES	no	0,5 g	Digestion with microwave oven	8 ml HNO3 (65%)+ 1 ml H2O2 (30%)	Monoelemental standard (1000 mg/l)	no	
	3								
	4	ASU L00.00-144		1 g	Microwave pressure digestion		muva-NEM 1603	Yes	
	5	EN 15763		0,4 g	Microwave pressure digestion	HNO3 / H2O2		Yes	
	6								
	7								not determined
	8								
	9	ASU § 64 L 00.00-144	yes	0,5	Microwave pressure digestion	Nitric acid	Standardsolution/ TM 25.4	Yes	
	10	ICP-OES			microwave destruction	HNO3 and H2O2	0-1000-5000-10000 µg/l	yes	
	11	AOAC 2015.01, AOAC 2015.06						yes	
	12								
	13	USP-35 NF 30	yes	0,4g	Turbowave Microwave	HNO3	6-Point-Calibration	Yes	
	14	ICP-OES	no	200mg	Microwave	HNO3 / H2O2 / H2O	external Calibration	Yes	
	15	DIN EN 15621		1g		Nitric acid		Yes	
	16	SOP 504-0230	shaking	0,5g	Microwave pressure digestion (H2O / HNO3 / H2O2)	100 mL		no	ICP-OES
	17	EN 15763 mod.; DIN 17294-2 mod.	mixing	0,5-1g	Microwave pressure digestion	8ml HNO3 + 2 ml H2O2	external / Ultra Scientific	Yes	
	18	ASU L 00.00-144	yes	0,5 g	ASU L 00.00-19/1	HNO3		Yes	

Parameter	Teilnehmer	Methodenbeschreibung	Homogenisierung	Einwaage	Aufschluss: Methode	Aufschluss: Lösung	Kalibrierverfahren/Referenzmaterial	Methode akkreditiert	Sonstige Hinweise
Analyte	**Participant**	**Method description**	**Homogenization**	**Sample weight**	**Hydrolization Method**	**Hydrolization Solution**	**Calibration and reference material**	**Method accredited**	**Further remarks**
Selen / Selenium	1	AOAC 993.14		0.25g	Microwave	5% H NO3	NIST	Yes	
	2	ICP-OES	no	0,5 g	Digestion with microwave oven	8ml HNO3 (65%)+ 2 ml HCl (37%)	Monoelemental standard (1000 mg/l)	no	
	3a	Total x- ray fluoreszence analysis by in-house-method	manual mixing	100 mg	wet milling process with ball mill	20%ige HNO_3	internaklStandard / Gallium	no	
	3b	Total x- ray fluoreszence analysis by in-house-method	manual mixing	100 mg	acidic diegstion	65%ige HNO_3	internal Standard / Gallium	no	
	4	In-house-method		0,5 g	Microwave pressure digestion		ERM-CE287k	Yes	
	5	EN 15763		0,4 g	Microwave pressure digestion	HNO3 / H2O2		Yes	
	6	ASU L 00.00.-19/3 modified for Selen	stirring	0,51398 (A) g, 0,50501 g (B)	ASU L 00.00-19/1		Addition	Yes	
	7	ICP-MS		0.4 g	Microwave	HNO3/H2O2	external Standard	no (request in process)	
	8								
	9	ASU § 64 L 00.00-135	yes	0,5	Microwave pressure digestion	Nitric acid	Standardsolution/ SRM 3280	Yes	
	10	ICP-MS			microwave destruction	HNO3 and H2O2	0-10-20-50-100-200 µg/l	no	
	11	AOAC 2015.01, AOAC 2015.06						yes	
	12	AA53, ICP-MS	ball mill	1g	AA30	HNO3, H2O2	NIST SRM 3280	Yes	
	13	USP-35 NF 30	yes	0,4g	Turbowave Microwave	HNO3	6-Point-Calibration	Yes	
	14	ICP-OES	no	200mg	Microwave	HNO3 / H2O2 / H2O	external Calibration	Yes	
	15	DIN EN 15765		1g		Nitric acid		Yes	
	16a	SOP 504-0163	shaking	0,5g	Microwave pressure digestion (H2O / HNO3 / H2O2)	100 mL		Yes	Graphite furnace - AAS
	16b	SOP 504-0230	shaking	0,5g	Microwave pressure digestion (H2O / HNO3 / H2O2)	100 mL		no	ICP-OES
	17	EN 15763 mod.; DIN 17294-2 mod.	mixing	0,5-1g	Microwave pressure digestion	8ml HNO3 + 2 ml H2O2	external / Ultra Scientific	Yes	
	18	ASU L 00.00-144	ja	0,5 g	ASU L 00.00-19/1	HNO3		Yes	

Parameter	Teilnehmer	Methodenbeschreibung	Homogenisierung	Einwaage	Aufschluss: Methode	Aufschluss: Lösung	Kalibrierverfahren/ Referenzmaterial	Methode akkreditiert	Sonstige Hinweise
Analyte	**Participant**	**Method description**	**Homogenization**	**Sample weight**	**Hydrolization Method**	**Hydrolization Solution**	**Calibration and reference material**	**Method accredited**	**Further remarks**
Zink / Zinc	1	AOAC 993.14		0.25g	Microwave	5% H NO3	NIST	Yes	
	2	ICP-OES	no	0,5 g	Digestion with microwave oven	8ml HNO3 (65%)+ 2 ml HCl (37%)	Monoelemental standard (1000 mg/l)	no	
	3a	Total x- ray fluoreszence analysis by in-house-method	manual mixing	100 mg	wet milling process with ball mill	20%ige HNO_3	internal Standard / Gallium	no	
	3b	Total x- ray fluoreszence analysis by in-house-method	manual mixing	100 mg	acidic diegstion	65%ige HNO_3	internal Standard / Gallium	Nein	
	4	ASU L00.00-144		1 g	Microwave pressure digestion		muva-NEM 1603	Yes	
	5	EN 15763		0,4 g	Microwave pressure digestion	HNO3 / H2O2		Yes	
	6	Incineration at 400°C, Measurement by Flame Flammen-AAS using ASU L 00.00-19/2	stirring	1,0068 g (A), 1,0015 g (B)	Incineration at 400°C		external	Yes	
	7	ICP-MS		0.4 g	Microwave	HNO3/H2O2	external Standard	no (request in process)	
	8	UNI EN 14084	yes	300 mg	HNO3 + H2O2 conc. - 200 °C	HNO3 0,1 %	Sigma 18827 batch BCBT6209	yes	
	9	ASU § 64 L 00.00-144	yes	0,5	Microwave pressure digestion	Nitric acid	Standardsolution/ SRM 1547	Yes	
	10	ICP-OES			microwave destruction	HNO3 and H2O2	0-100-250-500 µg/l	yes	
	11	AOAC 2015.01, AOAC 2015.06						yes	
	12	AA53, ICP-MS	ball mill	1g	AA30	HNO3, H2O2	NIST SRM 3280	Yes	
	13	USP-35 NF 30	yes	0,4g	Turbowave Microwave	HNO3	6-Point-Calibration	Yes	
	14	ICP-OES	no	200mg	Microwave	HNO3 / H2O2 / H2O	external Calibration	Yes	
	15	DIN EN 15621		1g		Nitric acid		Yes	
	16	SOP 504-0230	skahing	0,5g	Microwave pressure digestion (H2O / HNO3 / H2O2)	100 mL		no	ICP-OES
	17	EN 15763 mod.; DIN 17294-2 mod.	shaking	0,5-1g	Microwave pressure digestion	8ml HNO3 + 2 ml H2O2	external / Ultra Scientific	Yes	
	18	ASU L 00.00-144	yes	0,5 g	ASU L 00.00-19/1	HNO3		Yes	

5.2 Homogeneity

5.2.1 Homogeneity of bottled PT-samples

Homogeneity test of copper by ICP-MS:

Copper

Independant Samples	mg/kg
1	425
2	421
3	422
4	425
5	428
6	439
7	422
8	420

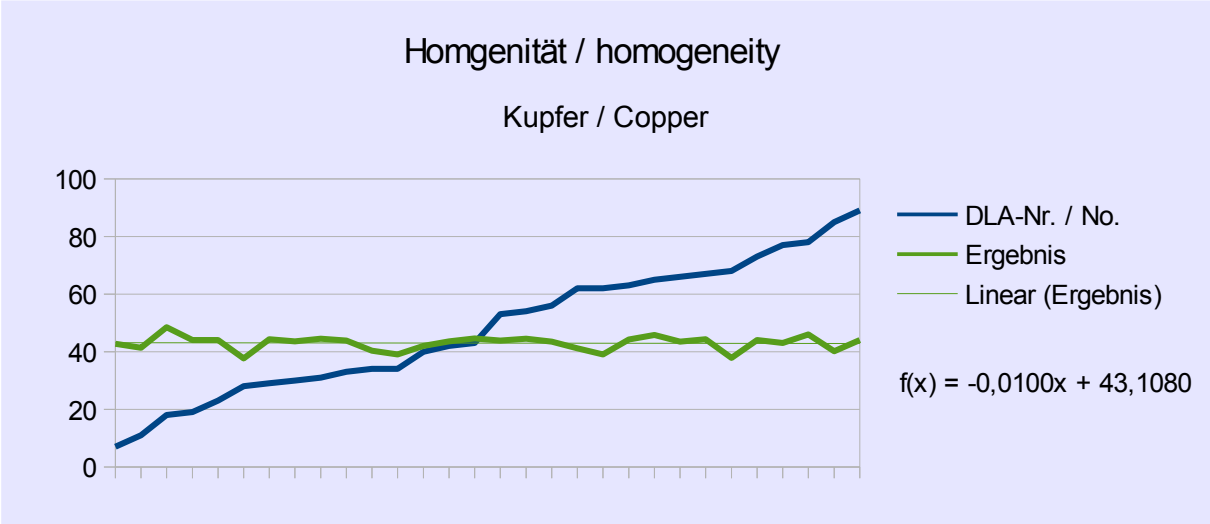
General Mean	425	
Repeatability standard deviation	6,14	1,44%

5.2.2 Comparison of sample numbers / test results and trend line

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

Copper				
Target standard deviation σ_{pt}	2,78			mg/100g
Sample numbers	7 – 89			
Total numbers of samples	30			
Slope	-0,0100			
Trend line range	43,1	-	43,4	mg/100g
Deviation trend line	43,3	±	0,150	mg/100g
Percent of σpt	5,40	%		

Calcium				
Target standard deviation σ_{pt}	286			mg/100g
Sample numbers	7 – 89			
Total numbers of samples	28			
Slope	7,45			
Trend line range	8025	-	8234	mg/100g
Deviation trend line	8130	±	104	mg/100g
Percent of σpt'	36,5	%		



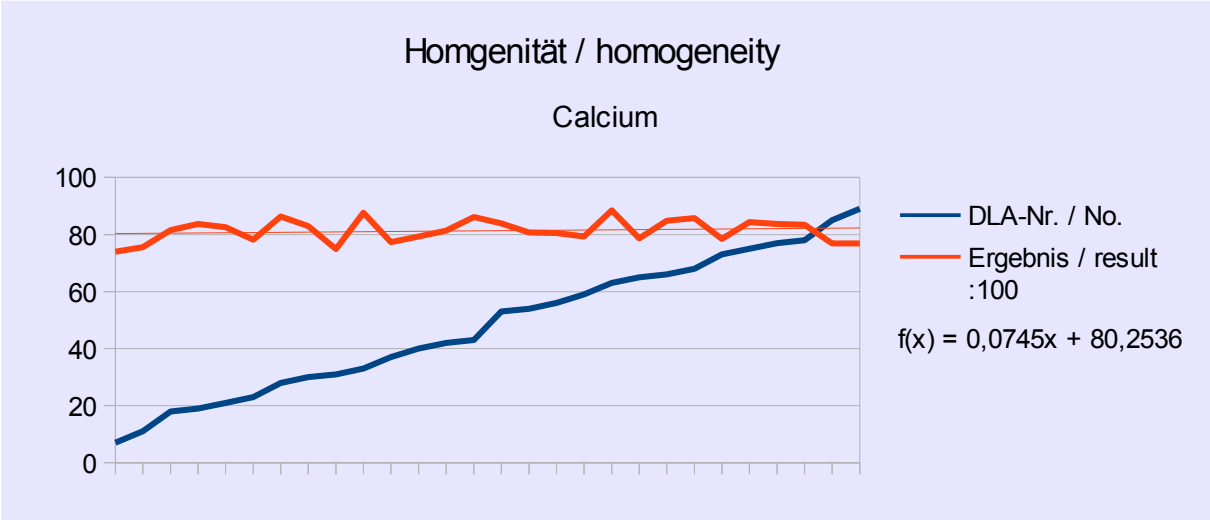


Abb./Fig. 25:
Trendfunktion Probennummern vs. Ergebnisse: Kupfer und Calcium (Calcium 1/100 dargestellt)
trend line function sample number vs. results: copper and calcium (calcium shown 1/100)

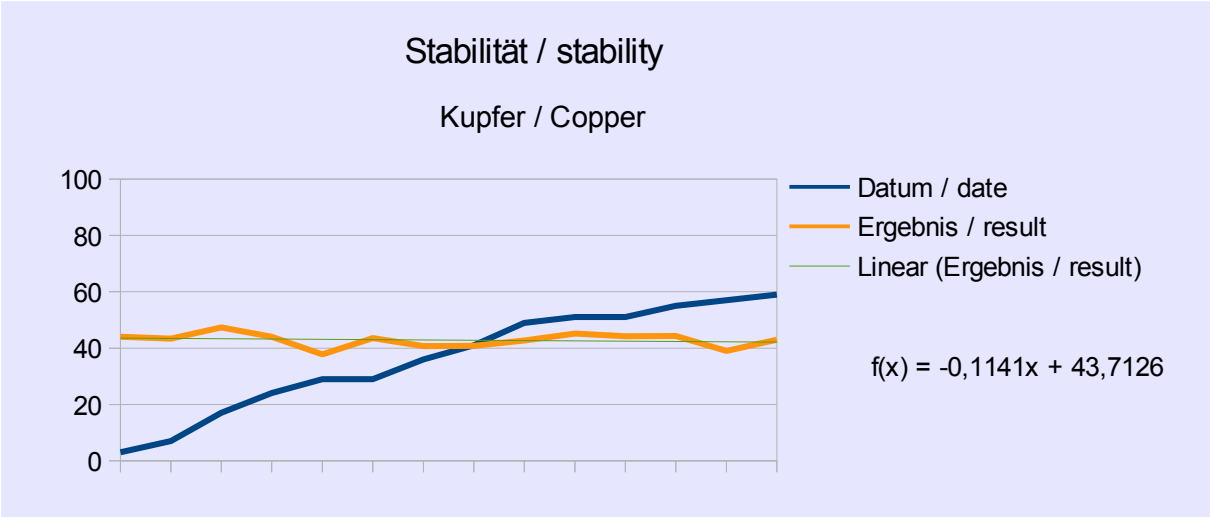
5.3 Stability

5.3.1 Trend line function of participant results

By comparison of the participant results with the corresponding date of analysis the stability of the PT-material can be characterized for the range of analysis time of the present PT by the trend line functions:

Copper				
Target standard deviation σ_{pt}	2,78			mg/100g
Time of analysis	57			Days
Number of results	14			
Slope	-0,114			
Trend line range	43,7	-	45,3	mg/100g
Deviation trend line	44,5	±	0,799	mg/100g
Percent of σ_{pt}	28,7	%		

Calcium				
Target standard deviation σ_{pt}	286			mg/100g
Time of analysis	44			Days
Number of results	13			
Slope	-19,1			
Trend line range	8269	-	8517	mg/100g
Deviation trend line	8393	±	124	mg/100g
Percent of σ_{pt}'	43,3	%		



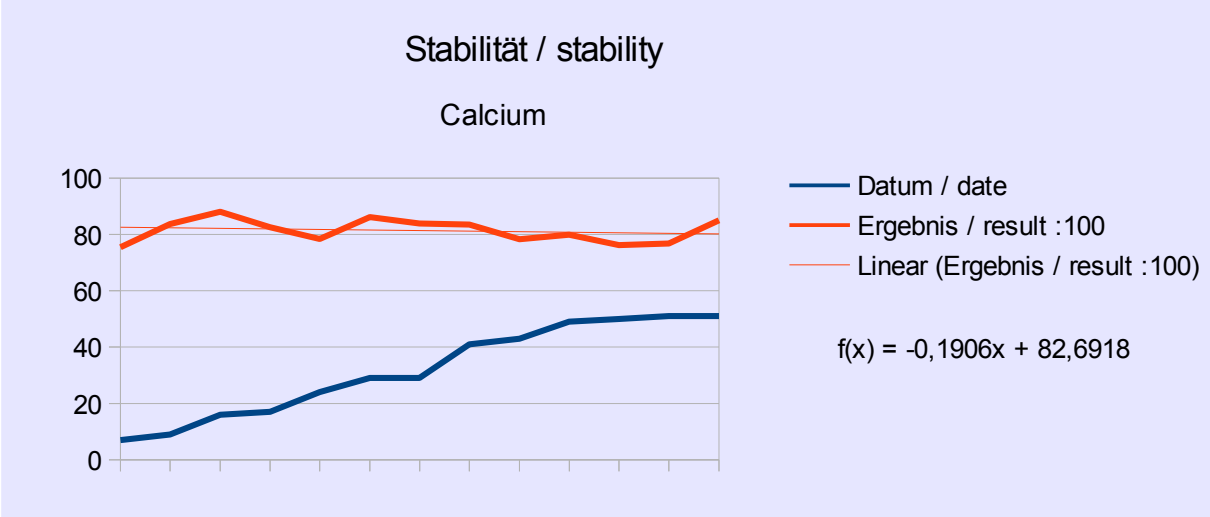


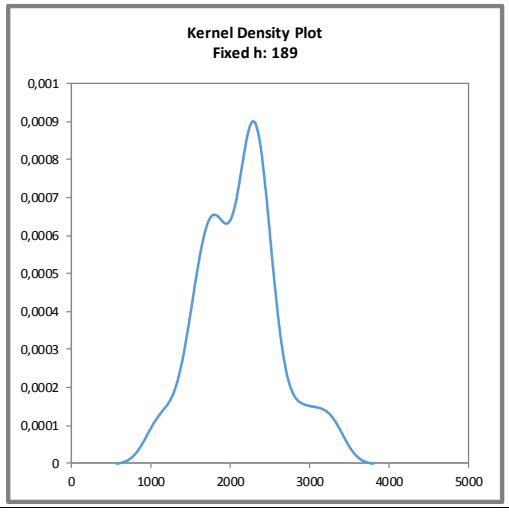
Abb./Fig. 26:
Trendfunktion Analysendatum vs. Ergebnisse: Kupfer und Calcium (Calcium 1/100 dargestellt)
trend line function date of analysis vs. results: copper and calcium (calcium shown 1/100)

5.4 Kernel Density Plots of Results

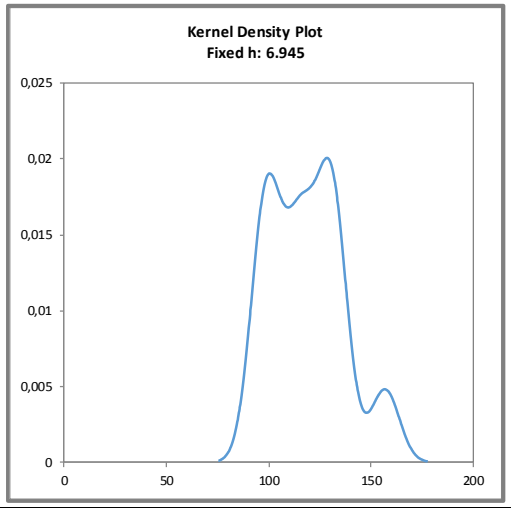
Abbildungen:
Kerndichte-Schätzungen
der Teilnehmerergebnisse
(mit h = 0,75 x σ_{pt} von Xpt)

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

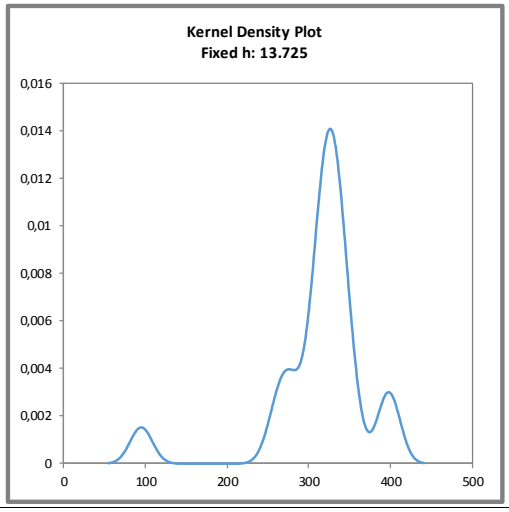
Chrom / Chromium



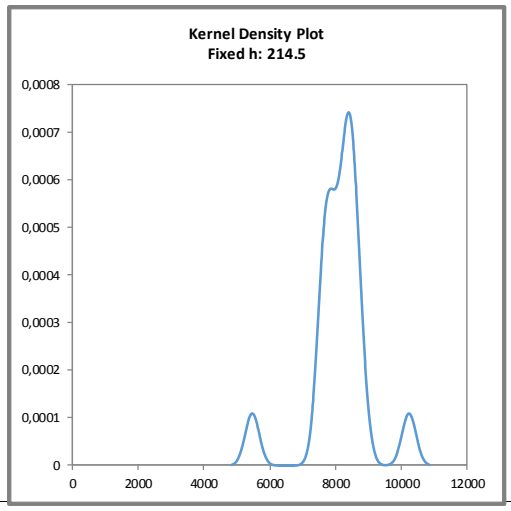
Bor / Boron



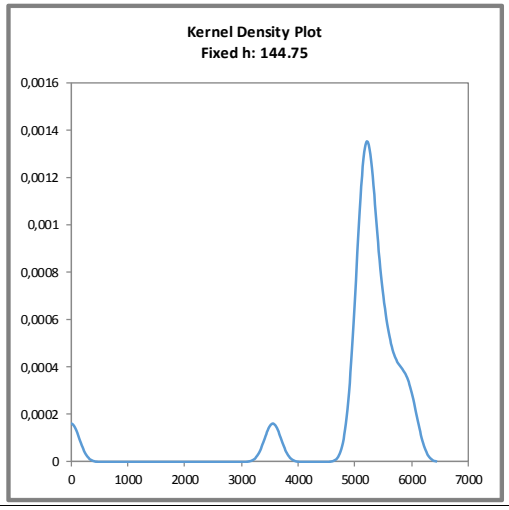
Eisen /Iron



Calcium



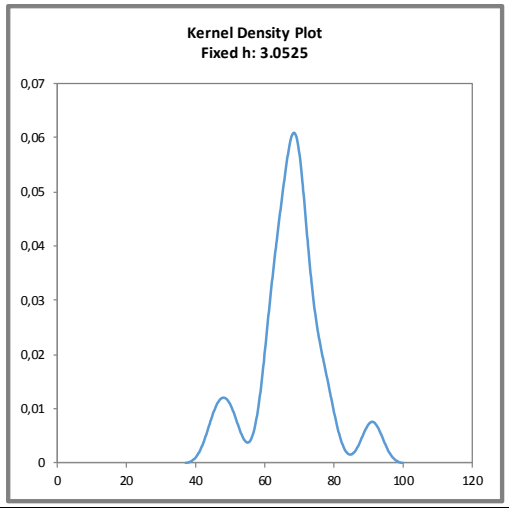
Kalium / Potassium



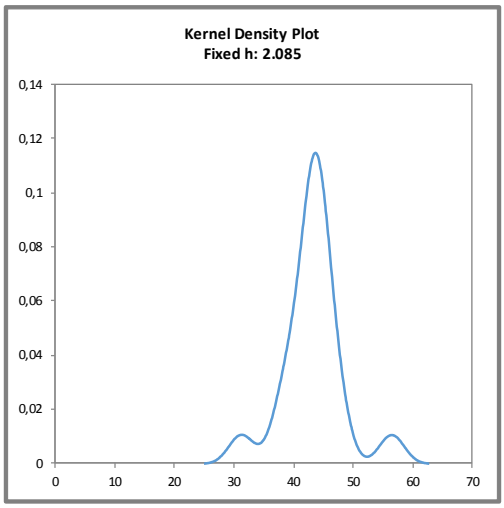
Abbildungen:
Kerndichte-Schätzungen
der Teilnehmerergebnisse
(mit h = 0,75 x σ_{pt} von Xpt)

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

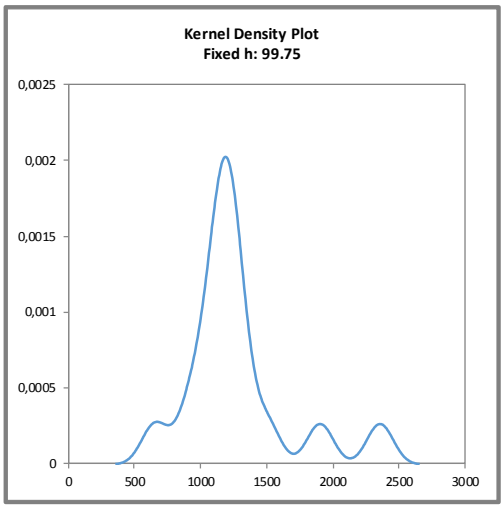
Mangan / Manganese



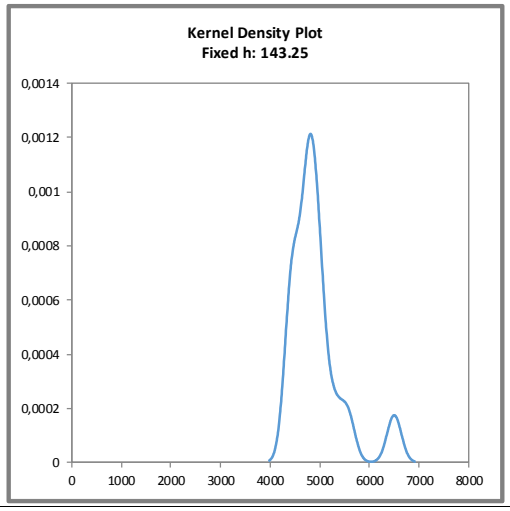
Kupfer / Copper



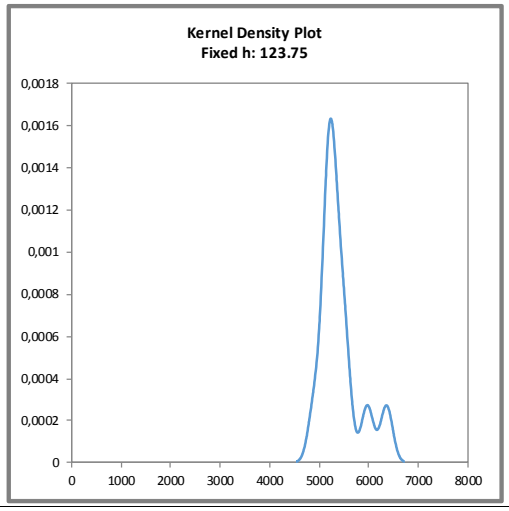
Molybdän / Molybdenum



Magnesium



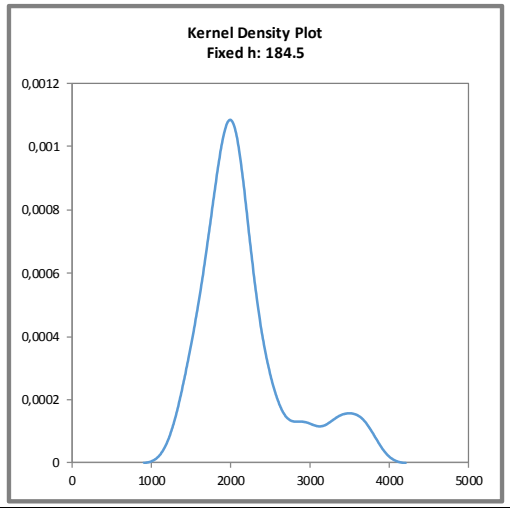
Phosphor / Phosphorus



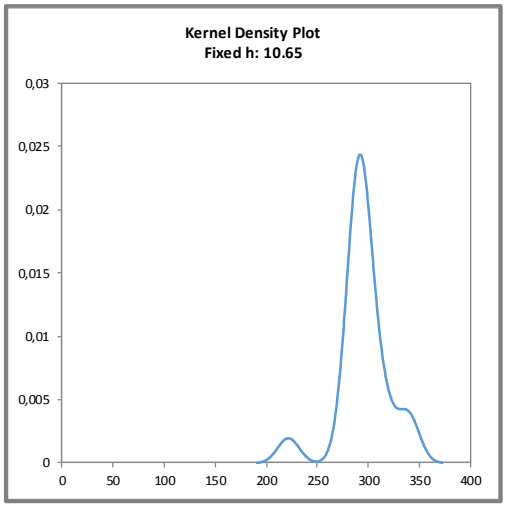
Abbildungen:
Kerndichte-Schätzungen
der Teilnehmerergebnisse
(mit h = 0,75 x σ_{pt} von Xpt)

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

Selen / Selenium



Zink / Zinc



5.5 Information on the Proficiency Test (PT)

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

PT number	***DLA 44-2017***
PT name	***Food Supplement II:*** ***B, Ca, Cr, Cu, Fe, K, Mg, Mn, Mo, P, Se and Zn***
*Sample matrix**	*Samples A + B: Multi mineral and vitamin tablets and capsule powder (without capsule shell) / ingredients: maltodextrin, mineral and vitamin compounds as well as technological food additives*
Number of samples and sample amount	*2 identical samples A + B, 10 g each.*
Storage	*Samples A + B: room temperature*
Intentional use	*Laboratory use only (quality control samples)*
Parameter	*quantitative:* ***B, Ca, Cr, Cu, Fe, K, Mg, Mn, Mo, P, Se and Zn***
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 A and B as well as the final results calculated as mean of the double determination (samples A and B) should be filled in the result submission file. The recovery rates, if carried out, has to be included in the calculation.*
Units	*mg/100g and µg/100g*
Number of significant digits	*at least 2*
Further information	*For information please specify:* – *Date of analysis* – *DLA-sample-numbers (for sample A and B)* – *Limit of detection* – *Assignment incl. Recovery* – *Recovery with the same matrix* – *Information on determination and hydrolization methods* – *Method is accredited*
Result submission	*The result submission file should be sent by e-mail to:* ***pt@dla-lvu.de***
Deadline	***the latest 04th August 2017***
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	*Dr. Matthias Besler*

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

6. Index of participant laboratories in alphabetical order

Teilnehmer / Participant	Ort / Town	Land / Country
		Germany
		Germany
		Germany
		ITALY
		Germany
		BELGIUM
		USA
		Germany
		Germany
		Germany
		SWITZERLAND
		SPAIN
		Germany
		Germany
		Germany
		Germany
		SWITZERLAND
		INDIA

[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)
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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)
11. The International Harmonised Protocol for the Proficiency Testing of Analytical Chemistry Laboratories; Pure Appl Chem, 78, 145 – 196 (2006)
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13. EURACHEM/CITAC Leitfaden, Ermittlung der Messunsicherheit bei analytischen Messungen (2003); Quantifying Uncertainty in Analytical Measurement (1999)
14. GMP+ Feed Certification scheme, Module: Feed Safety Assurance, chapter 5.7 Checking procedure for the process accuracy of compound feed with micro tracers in GMP+ BA2 Control of residues, Version: 1st of January 2015 GMP+ International B.V.
15. MTSE SOP No. 010.01 (2014): Quantitative measurement of mixing uniformity and carry-over in powder mixtures with the rotary detector technique, MTSE Micro Tracers Services Europe GmbH
16. ASU §64 L 00.00-157 (2016-2): Bestimmung von Aluminium in Lebensmitteln mit der Massenspektrometrie mit induktiv gekoppeltem Plasma (ICP-MS) [Determination of aluminium in foods by inductively coupled plasma mass spectrometry (ICPMS) after pressure digestion]
17. 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]
18. ASU §64 L 00.00-135 (2011-01) / DIN EN 15763:2010: Bestimmung von Arsen, Cadmium, Quecksilber und Blei in Lebensmitteln mit ICP-MS nach Druckaufschluss / Foodstuffs. Determination of trace elements. Determination of arsenic, cadmium, mercury and lead in foodstuffs by inductively coupled plasma mass spectrometry (ICP-MS) after pressure digestion
19. ASU §64 L 00.00-19/2: Bestimmung von Eisen, Kupfer, Mangan und Zink mit der Atomabsorptionsspektrometrie (AAS) in der Flamme [Determination of

iron, copper, manganese and zinc by atomic absorption spectrometry (AAS) in the flame]
20. ASU §64 L 00.00-19/3 / DIN EN 14083: Bestimmung von Blei, Cadmium, Chrom und Molybdän mit Graphitofen-Atomabsorptionsspektrometrie (GFAAS) nach Druckaufschluss / Foodstuffs. Determination of trace elements. Determination of lead, cadmium, chromium and molybdenum by graphite furnace atomic absorption spectrometry (GFAAS) after pressure digestion
21. 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]
22. ASU §64 L 00.00-144 : Bestimmung der Mineralstoffe Ca, K, Mg, Na, P und S sowie der Spurenelemente Fe, Cu, Mn und Zn in Lebensmitteln mit ICP-OES [Determination of minerals Ca, K, Mg, Na, P and S and trace elements Fe, Cu, Mn and Zn in foods by ICP-OES]
23. ASU §64 L 00.00-93 / DIN EN 15111: Bestimmung von Iod in Lebensmitteln - ICP-MS-Verfahren / Foodstuffs. Determination of trace elements. Determination of iodine by ICP-MS (inductively coupled plasma mass spectrometry)
24. ASU §64 L 00.00-127 / EN 15764: Bestimmung von Zinn in Lebensmitteln mit der Flammen- und Graphitrohr-Atomabsorptionsspektrometrie (GFAAS) nach Druckaufschluss / Foodstuffs. Determination of trace elements. Determination of tin by flame and graphite furnace atomic absorption spectrometry (FAAS and GFAAS) after pressure digestion
25. ASU §64 L 00.00-128 / DIN EN 15765: Bestimmung Zinn in Lebensmitteln mit der 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
26. ASU §64 L 31.00-10: Bestimmung der Gehalte an Natrium, Kalium, Calcium und Magnesium in Frucht- und Gemüsesäften – Atomabsorptionsspektrometrisches Verfahren (AAS) [Determination of sodium, potassium, calcium and magnesium in fruit and vegetable juices - atomic absorption spectrometry (AAS)]