



Evaluation Report

proficiency test

DLA 47/2019

Food Supplements II:

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

in Tablet / Capsule Powder

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

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<i>Unteraufträge</i> <i>Subcontractors</i>	<p>Falls im Rahmen der Eignungsprüfung eine Prüfung der Gehalte, Homogenität und Stabilität von EP-Parametern durchgeführt wurde, hat DLA diese im Unterauftrag vergeben.</p> <p>In case the analysis of the content, homogeneity and stability of PT-parameters was part of the proficiency test, the determinations were subcontracted by DLA.</p>
<i>Vertraulichkeit</i> <i>Confidentiality</i>	<p>Die Teilnehmerergebnisse sind im EP-Bericht in anonymisierter Form mit Auswertenummern benannt. Daten einzelner Teilnehmer werden ausschließlich nach vorheriger Zustimmung des Teilnehmers an Dritte weitergegeben.</p> <p>Participant result are named anonymously with evaluation numbers in the PT report. Data of individual participants will be passed on to third parties only with prior consent of the participant.</p>

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1. Introduction

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

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

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

2. Realisation

2.1 Test material

The test material is a 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 from European suppliers.

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 metalised PET film bags and chronologically numbered.

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

Table 1: Composition of DLA-Samples

Multi-Mineral-Powder
<p><u>Ingredients</u> (1. Food Supplement, Tablets): Calcium carbonate, microcrystalline cellulose, ascorbic acid, magnesium oxide, calcium phosphate, potassium chloride, croscarmellose sodium, nicotinamide, iron fumarate, d-alpha tocopherol acetate, release agents: magnesium salts of fatty acids, silicon dioxide, zinc oxide, calcium pantothenate, coating: hypromellose and polyvinyl alcohol, manganese sulfate, riboflavin, pyridoxine HCl, thiamine mononitrate, copper sulfate, vitamin A acetate, folic acid, potassium iodide, sodium tetraborate, sodium selenite, biotin, vitamin K1, sodium molybdate, chromium chloride, cholecalciferol, cyanocobalamin.</p> <p><u>Ingredients</u> (2. Food Supplement, Capsule powder without capsule shells): Dicalcium phosphate, magnesium oxide, vitamin C, potassium chloride, niacin, release agent magnesium stearate, vitamin E acetate, calcium D-pantothenate, iron sulfate, zinc oxide, vitamin B6 hydrochloride, copper sulfate, vitamin B2, vitamin B1 mononitrate, vitamin A acetate, folic acid, biotin, potassium iodide, chromium-III-chloride, sodium molybdate, sodium selenite, vitamin K1, vitamin D3, vitamin B12.</p> <p><u>Ingredients</u> (3. Food Supplement, Capsule powder without capsule shells): Boron citrate, boron aspartate, boron-glycinate, microcrystalline cellulose, riboflavin.</p> <p><u>Further Ingredient:</u> Maltodextrin</p>

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 100g
B - Bor	64 mg
Ca - Calcium	4897 mg
Cr - Chrom	1125 µg
Cu - Kupfer	49 mg
Fe - Eisen	341 mg
K - Kalium	1947 mg
Mg - Magnesium	3650 mg
Mn - Mangan	37 mg
Mo - Molybdän	1217 µg
P - Phosphor	1694 mg
Se - Selen	1764 µg
Zn - Zink	335 mg

2.1.1 Homogeneity

The **mixture homogeneity before bottling** was examined 8-fold by determination of copper by ICP-MS. The repeatability standard deviation was 2,45% and is less than the range of repeatability standard deviations of comparable methods (e.g. ASU §64 L 00.00-144, s. 3.6.1). The results of homogeneity analysis are given in the documentation.

The calculation of the **repeatability standard deviations S_r of the participants** was also used as an indicator of homogeneity. For all parameters they are <4,5% (1,3% - 4,5%). 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) [18-28]. 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
B - Bor	1,29 %
Ca - Calcium	2,12 %
Cr - Chrom	4,38 %
Cu - Kupfer	2,63 %
Fe - Eisen	2,45 %
K - Kalium	3,22 %
Mg - Magnesium	2,26 %
Mn - Mangan	3,33 %
Mo - Molybdän	4,50 %
P - Phosphor	2,43 %
Se - Selen	3,00 %
Zn - Zink	2,62 %

Furthermore, the homogeneity was graphically characterized for information by the **trend line function of participants' results for chronological bottled single samples** (s. 5.2.2).

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 test materials showed good storage stability with respect to the durability of the sample (spoilage) and the content of the PT parameters for comparable food matrices and water activity (a_w value <0,5). The stability of the sample material was thus ensured during the investigation period under the specified storage conditions.

2.2 Sample shipment and information to the test

Two portions of test material were sent to every participating laboratory in the 30th week of 2019. The testing method was optional. The tests should be finished at 20th September 2019 the latest.

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

The two portions contain identical samples of a food supplement with the above mentioned parameters in the matrix of tablet and capsule powder (without capsule shell) with maltodextrin as base. 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 5.4 Information on the PT)*

2.3 Submission of results

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

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

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

Of 14 participants, 13 participants submitted their results on time. One participant did not submit any results.

3. Evaluation

3.1 Consensus value from participants (assigned value)

The robust mean of the submitted results was used as assigned value (X_{pt}) („consensus value from participants“) providing a normal distribution. The calculation was done according to algorithm A as described in annex C of ISO 13528 [3]. If there are < 12 quantitative results and an increased difference between robust mean and median, the median may be used as the assigned value (criterion: Δ median - rob. mean > $0,3 \sigma_{pt}$) [3].

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

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

The statistical evaluation is carried out for all the parameters for a minimum of 7 values are present, in justified cases, an evaluation may also be carried out from 5 results onwards.

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

3.2 Robust standard deviation

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

3.3 Repeatability standard deviation

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

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

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

3.4 Reproducibility standard deviation

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

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

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

The relative reproducibility standard deviation CV_R in percent of the mean is given as variation coefficient in the statistical data of participant for each parameter. The significance of CV_R is further explained in section 3.9.

3.5 Exclusion of results and outliers

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

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

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

Results are tested for outliers by the use of robust statistics (algorithm A): If a value deviates from the robust mean by more than 3 times the robust standard deviation, it can be classified as an outlier (see above) [3]. Due to the use of robust statistics outliers are not excluded, provided that no other reasons are present [3]. Detected outliers are only mentioned in the results section, if they have been excluded from the statistical evaluation.

3.6 Target standard deviation (for proficiency assessment)

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

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

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): Boron, chromium, copper, iron, potassium, manganese, molybdenum, phosphorus, selenium and zinc.

For calcium and magnesium the target standard deviation was calculated using data from a precision experiment (s. 3.6.2, ASU §64 method L 00.00-144).

3.6.1 General model (Horwitz)

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

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

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

3.6.2 Value by precision experiment

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

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

The relative repeatability standard deviations (RSD_r) and relative reproducibility standard deviation (RSD_R) given in Table 4 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} [21-24]

Parameter	Matrix	Mean [mg/kg]	RSD_r	RSD_R	σ_{pt}	Method / Literature
Ca	Lobster	183	4,90%	6,31%	5,27%	ICP-OES [24]
	Children's food soy	6191	3,41%	7,97%	7,60% ¹	ICP-OES [24]
Cr	Infant food	0,17	7,3%	19%	18,3% ¹	GF-AAS [22]
	Rice powder	0,11	19,2%	35%	32,3%	GF-AAS [22]
Cu	Lobster	16,40	5,72%	6,82%	5,49%	ICP-OES [24]
	Children's food soy	4,51	4,30%	11,06%	10,6% ¹	ICP-OES [24]
Fe	Lobster	12,1	6,45%	8,59%	7,28%	ICP-OES [24]
	Children's food soy	77	2,75%	6,98%	6,70% ¹	ICP-OES [24]
K	Lobster	871	3,63%	6,27%	5,71%	ICP-OES [24]
	Children's food soy	6733	4,08%	5,49%	4,67% ¹	ICP-OES [24]
Mn	Lobster	1,20	4,74%	7,95%	7,21%	ICP-OES [24]
	Children's food soy	2,19	4,67%	13,7%	13,3% ¹	ICP-OES [24]
Mg	Lobster	85	3,73%	8,63%	8,21%	ICP-OES [24]
	Children's food soy	599	4,30%	7,64%	7,01% ¹	ICP-OES [24]
Mo	Infant food	0,50	6,6%	21%	20,5% ¹	GF-AAS [22]
	Rice powder	0,56	8,7%	20%	19,0%	GF-AAS [22]
P	Lobster	973	3,16%	7,13%	6,78%	ICP-OES [24]
	Children's food soy	4129	3,45%	7,87%	7,48% ¹	ICP-OES [24]
Se	Katfish	1,797	9,85%	10,1%	7,31% ¹	AAS [23]
	Rice	0,374	2,41%	11,8%	11,7%	AAS [23]
Zn	Lobster	13,9	4,63%	7,90%	7,19%	ICP-OES [24]
	Children's food soy	43,5	2,60%	6,89%	6,64% ¹	ICP-OES [24]

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 or 3.6.2. was regarded suitable.

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:

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

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

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

The valid z-Score for each parameter is indicated as z-Score (σ_{pt}).

3.7.1 Warning and action signals

In accordance with the norm ISO 13528 it is recommended that a result that gives rise to a z-score above 3,0 or below -3,0, shall be considered to give an "action signal" [3]. Likewise, a z-score above 2,0 or below -2,0 shall be considered to give a "warning signal". A single "action signal", or "warning signal" in two successive PT-rounds, shall be taken as evidence that an anomaly has occurred which requires investigation.

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

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

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

Parameter	Matrix (Powder)	robust Mean [mg/kg]	rob. SD (S*) [mg/kg]	rel. SD (CV_{s*}) [%]	Quotient S*/opt	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
B	Tablets/Capsules	637	25,8	4,04%	0,67	DLA 47/2019
Ca	Potatoes	238	12,0	5,04%	0,72	DLA 46/2017
Ca	Tablets/Capsules	81600	5240	6,42%	1,8 ¹	DLA 44/2017
Ca	Tablets/Capsules	52400	3650	6,95%	0,92	DLA 47/2019
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
Cr	Tablets/Capsules	13,5	2,06	15,2%	1,4	DLA 47/2019
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
Cu	Tablets/Capsules	441	23,8	5,40%	0,84	DLA 47/2019
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
Fe	Tablets/Capsules	3410	133	3,90%	0,83	DLA 47/2019
K	Potatoes	13200	604	4,59%	1,2	DLA 46/2017
K	Tablets/Capsules	53400	3160	5,92%	1,9	DLA 44/2017
K	Tablets/Capsules	19400	721	3,71%	1,0	DLA 47/2019
Mg	Potatoes	736	27,1	3,68%	0,62	DLA 46/2017
Mg	Tablets/Capsules	48500	3660	7,55%	1,9 ¹	DLA 44/2017
Mg	Tablets/Capsules	34500	2480	7,19%	1,0	DLA 47/2019
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
Mn	Tablets/Capsules	390	26,1	6,68%	1,0	DLA 47/2019
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
Mo	Tablets/Capsules	12,3	1,97	16,1%	1,5	DLA 47/2019
P	Potatoes	1451	49,1	3,38%	0,63	DLA 46/2017
P	Tablets/Capsules	53200	2720	5,11%	1,6	DLA 44/2017
P	Tablets/Capsules	17400	732	4,21%	1,1	DLA 47/2019
Se	Tablets/Capsules	20,9	4,34	20,8%	1,8 ¹	DLA 44/2017
Se	Tablets/Capsules	19,6	0,992	5,06%	0,49	DLA 47/2019
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

Parameter	Matrix (Powder)	robust Mean [mg/kg]	rob. SD (S*) [mg/kg]	rel. SD (CV _{s*}) [%]	Quotient S*/opt	DLA-report
Zn	Tablets/Capsules	2960	143	4,85%	1,0	DLA 44/2017
Zn	Tablets/Capsules	3170	118	3,74%	0,79	DLA 47/2019

¹ with target standard deviation σ_{pt}'

** 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. 3.11). The z'-score represents the relation of the deviation of the result (x_i) 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 ($U_{(x_{pt})}$) [3].

The calculation is performed by:

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

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

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

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

For warning and action signals see 3.7.1.

3.9 Reproducibility coefficient of variation (CV_R)

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

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

In contrast to the standard deviation as a measure of the absolute variability the CV 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*/opt

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 ($U_{(x_{pt})}$) for this PT is calculated as follows [3]:

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

If $U_{(x_{pt})} \leq 0,3 \sigma_{pt}$ the standard uncertainty of the assigned value needs not to be included in the interpretation of the results of the PT [3]. Values exceeding 0,3 imply, that the target standard deviation could be too low with respect to the standard uncertainty of the assigned value.

The traceability of the assigned value is ensured on the basis of the consensus value as a robust mean of the participant results.

4. Results

Comments to the distribution of the results:

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

Comments to the statistic data:

The target standard deviation was calculated for all parameters according to the model of Horwitz or was calculated from statistical data obtained from precision experiments (ASU §64 method). The evaluation according to the model of Horwitz was preferred, as long as the quotients S^*/σ_{pt} were ≤ 2.0 . In all other cases, the target standard deviation calculated from statistical data obtained from precision experiments (ASU §64 method) was used.

For all parameters the distribution of results showed a low to normal variability. The quotients S^*/σ_{pt} were all in the range of 0,49 to 1,5 (s. Tab. 4).

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

The comparability of results is given.

80% to 100% of the results were within the respective target range.

The robust means of the participant results were for all parameters in the range of 90% to 120% of the contents according to the manufacturer specifications (s. Tab. 2): 99-103% for B, Fe, K, Mo and P, 90-95% for Cu, Mg, and Zn, and 106-120% for Ca, Cr, Mn and Se.

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

In the first table the characteristics are listed:

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

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

In the table below, the results of the participating laboratories are formatted in 3 valid digits**:

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

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

4.1 B - Boron in mg/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	9
Number of outliers	-
Mean	122
Median	63,8
Robust Mean (X_{pt})	63,7
Robust standard deviation (S^*)	2,58
Number with 2 replicates	8
Repeatability SD (S_r)	0,814
Repeatability (CV_r)	1,29%
Reproducibility SD (S_R)	1,95
Reproducibility (CV_R)	3,08%
Target range:	
Target standard deviation σ_{pt}	3,86
lower limit of target range	56,0
upper limit of target range	71,5
Quotient S^*/σ_{pt}	0,67
Standard uncertainty $U(X_{pt})$	1,07
Results in the target range	8
Percent in the target range	89%

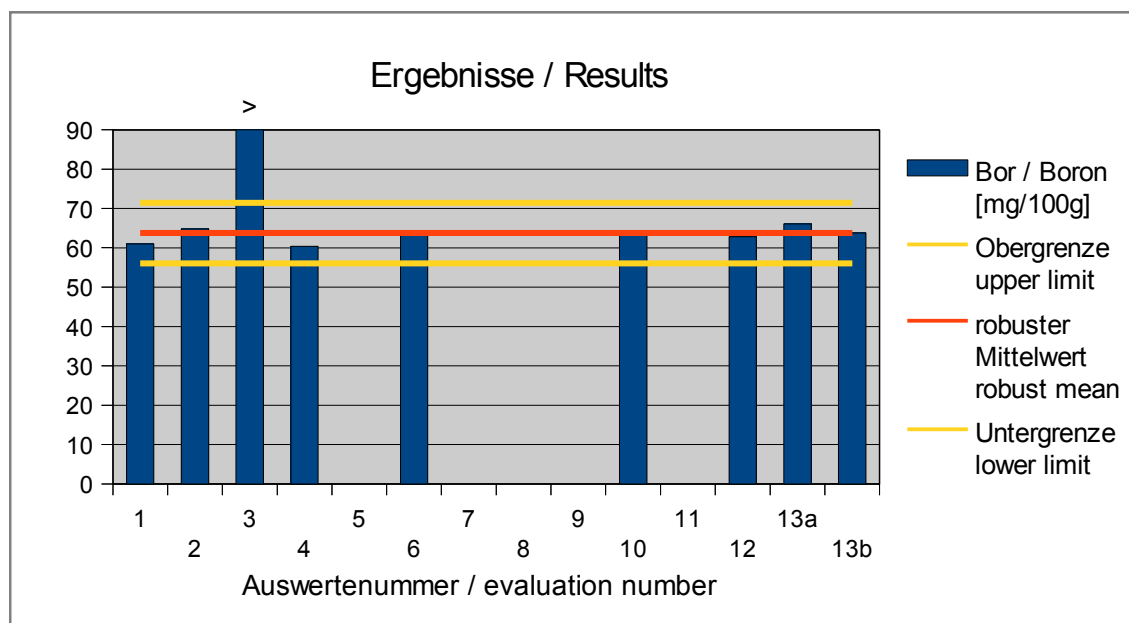


Abb. / Fig. 1: Ergebnisse Bor / Results Boron

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

Auswertenummer	Bor / Boron [mg/100g]	Abweichung [mg/100g]	z-Score	Hinweis
Evaluation number		Deviation [mg/100g]	(σ_{pt})	Remark
1	61,0	-2,74	-0,71	
2	64,8	1,06	0,28	
3	591	527	137	
4	60,4	-3,34	-0,86	
5				
6	64,0	0,26	0,07	
7				
8				
9				
10	63,2	-0,54	-0,14	
11				
12	62,8	-0,94	-0,24	
13a	66,1	2,31	0,60	
13b	63,8	0,04	0,01	



Abb. / Fig. 2: z-Scores Bor / Boron

4.2 Ca - Calcium in mg/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	12
Number of outliers	0
Mean	5240
Median	5300
Robust Mean (x_{pt})	5250
Robust standard deviation (S^*)	365
Number with 2 replicates	13
Repeatability SD (S_r)	110
Repeatability (CV_r)	2,12%
Reproducibility SD (S_R)	373
Reproducibility (CV_R)	7,18%
Target range:	
Target standard deviation σ_{pt}	398
Target standard deviation (for Information)	163
lower limit of target range	4450
upper limit of target range	6040
Quotient S^*/σ_{pt}	0,92
Standard uncertainty $U(x_{pt})$	132
Results in the target range	12
Percent in the target range	100%

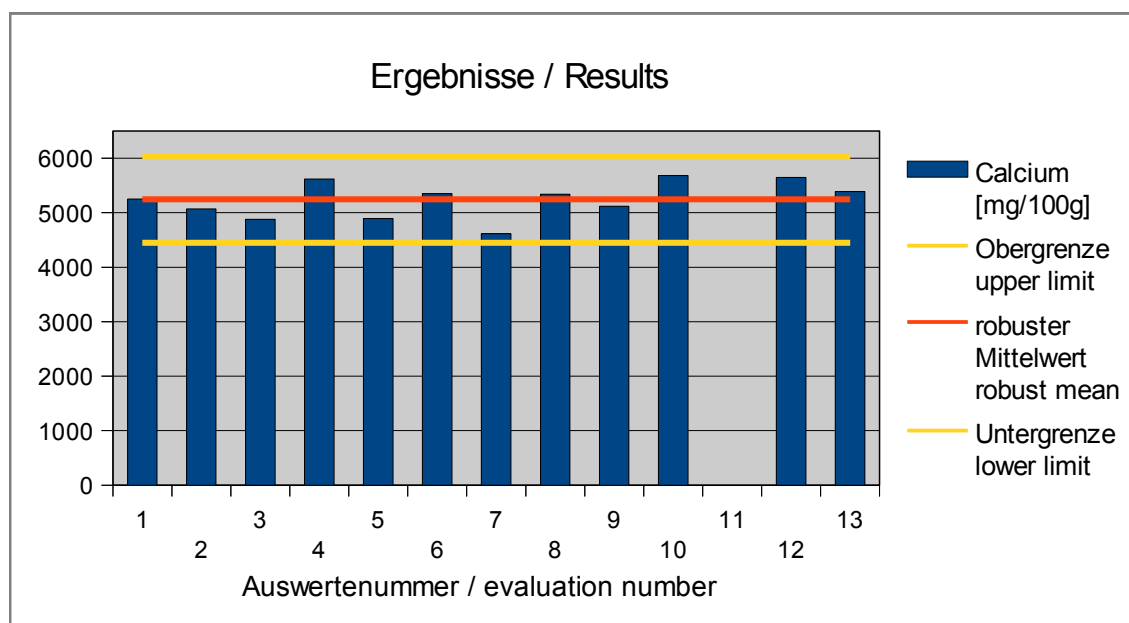


Abb. / Fig. 3: Ergebnisse Calcium / Results Calcium

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

Auswertenummer	Calcium [mg/100g]	Abweichung [mg/100g]	z-Score (σ _{pt})	z-Score (Info)	Hinweis
Evaluation number		Deviation [mg/100g]		(Info)	Remark
1	5250	5	0,01	0,03	
2	5070	-175	-0,44	-1,1	
3	4880	-365	-0,92	-2,2	
4	5620	375	0,94	2,3	
5	4892	-353	-0,89	-2,2	
6	5350	105	0,26	0,64	
7	4615	-630	-1,6	-3,9	
8	5342	97	0,24	0,59	
9	5119	-126	-0,32	-0,77	
10	5681	436	1,1	2,7	
11					
12	5649	404	1,0	2,5	
13	5386	141	0,35	0,86	

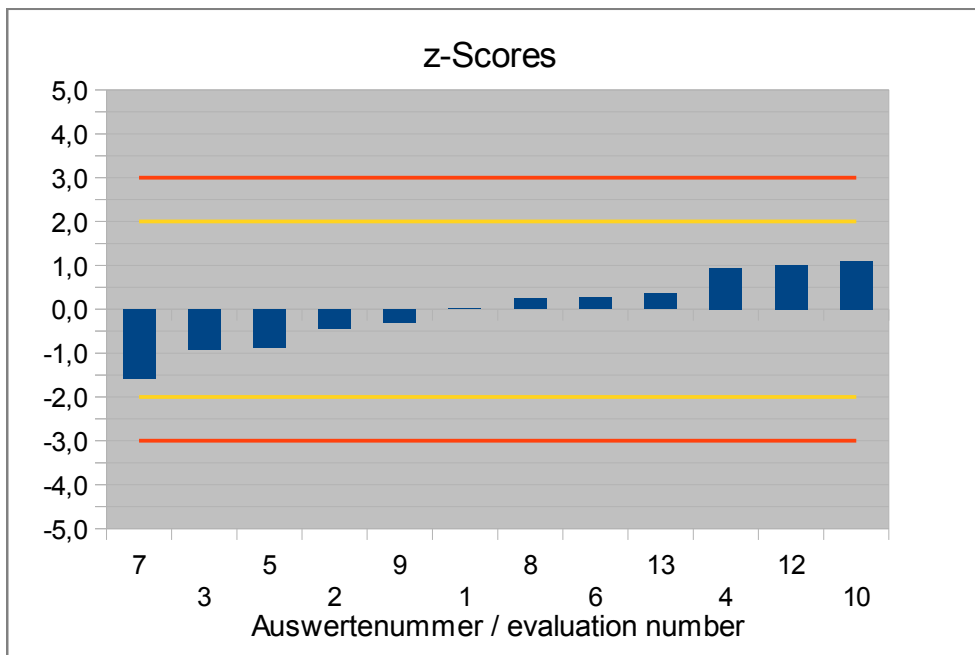


Abb. / Fig. 4: z-Scores Caclium

4.3 Cr - Chromium in $\mu\text{g}/100\text{g}$

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	10
Number of outliers	1
Mean	1300
Median	1380
Robust Mean (X_{pt})	1350
Robust standard deviation (S^*)	206
Number with 2 replicates	10
Repeatability SD (S_r)	63,1
Repeatability (CV_r)	4,38%
Reproducibility SD (S_R)	219
Reproducibility (CV_R)	15,2%
Target range:	
Target standard deviation σ_{pt}	146
Target standard deviation (for Information)	247
lower limit of target range	1060
upper limit of target range	1650
Quotient S^*/σ_{pt}	1,4
Standard uncertainty $U(X_{pt})$	81,3
Results in the target range	8
Percent in the target range	80%

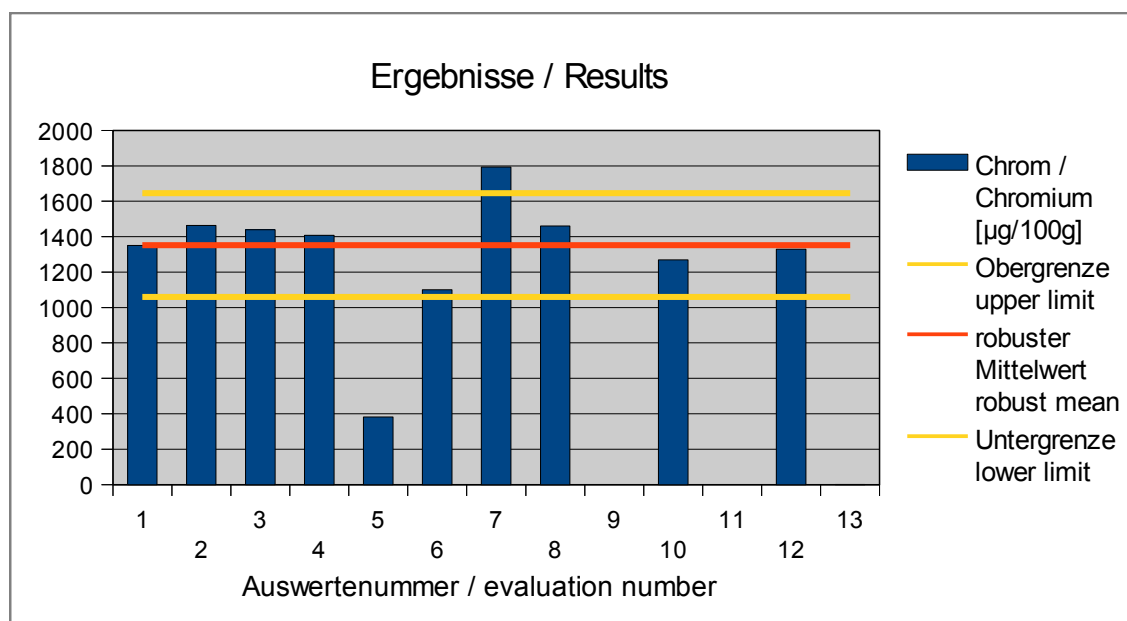


Abb. / Fig. 5: Ergebnisse Chrom / Results Chromium

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

Auswertenummer	Chrom / Chromium [µg/100g]	Abweichung [µg/100g]	z-Score (σ _{pt})	z-Score (Info)	Hinweis
Evaluation number		Deviation [µg/100g]		(Info)	Remark
1	1350	-2,8	-0,02	-0,01	
2	1464	111	0,76	0,45	
3	1440	87	0,60	0,35	
4	1408	55	0,38	0,22	
5	382	-971	-6,6	-3,9	
6	1100	-253	-1,7	-1,0	
7	1793	440	3,0	1,8	
8	1460	107	0,73	0,43	
9					
10	1270	-83	-0,57	-0,33	
11					
12	1330	-23	-0,16	-0,09	
13	0,00148				Ausreißer ausgeschlossen / Outlier excluded

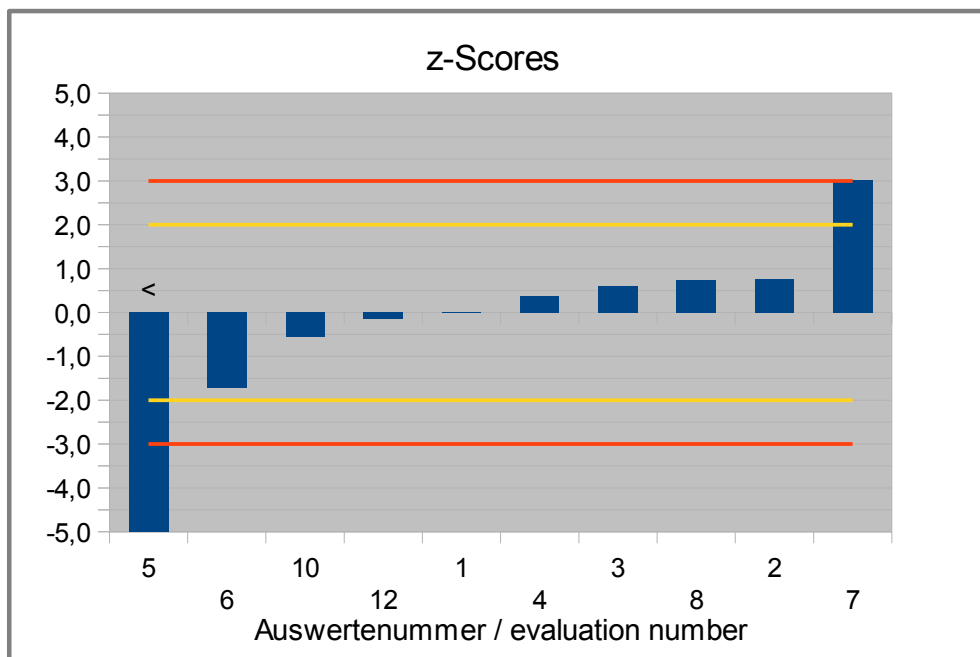


Abb. / Fig. 6: z-Scores Chrom / Chromium

4.4 Cu - Copper in mg/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	12
Number of outliers	0
Mean	44,1
Median	44,0
Robust Mean (X_{pt})	44,1
Robust standard deviation (S^*)	2,38
Number with 2 replicates	13
Repeatability SD (S_r)	1,16
Repeatability (CV_r)	2,63%
Reproducibility SD (S_R)	2,24
Reproducibility (CV_R)	5,06%
Target range:	
Target standard deviation σ_{pt}	2,82
Target standard deviation (for Information)	4,69
lower limit of target range	38,4
upper limit of target range	49,7
Quotient S^*/σ_{pt}	0,84
Standard uncertainty $U(X_{pt})$	0,858
Results in the target range	12
Percent in the target range	100%

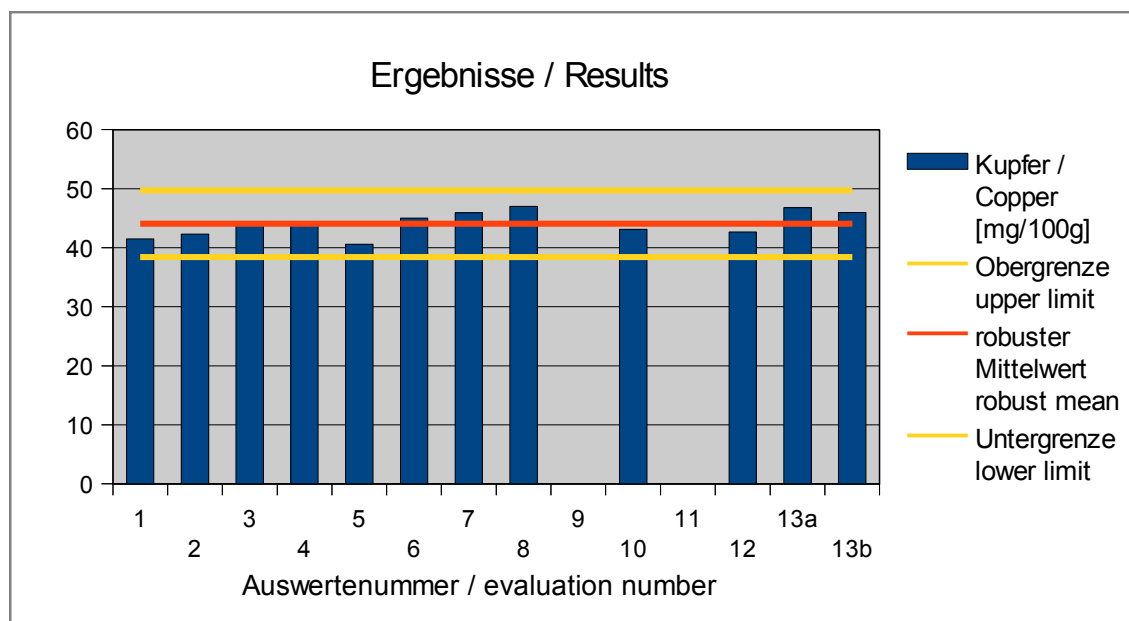


Abb. / Fig. 7: Ergebnisse Kupfer / Results Copper

Ergebnisse der Teilnehmer:
Results of Participants:

Auswertenummer	Kupfer / Copper [mg/100g]	Abweichung [mg/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis
Evaluation number		Deviation [mg/100g]			Remark
1	41,5	-2,57	-0,91	-0,55	
2	42,3	-1,77	-0,63	-0,38	
3	44,1	0,04	0,01	0,01	
4	43,9	-0,16	-0,06	-0,04	
5	40,6	-3,47	-1,2	-0,74	
6	45,0	0,94	0,33	0,20	
7	45,9	1,84	0,65	0,39	
8	47,0	2,94	1,0	0,6	
9					
10	43,1	-0,96	-0,34	-0,21	
11					
12	42,7	-1,42	-0,50	-0,30	
13a	46,8	2,70	1,0	0,58	
13b	46,0	1,91	0,68	0,41	

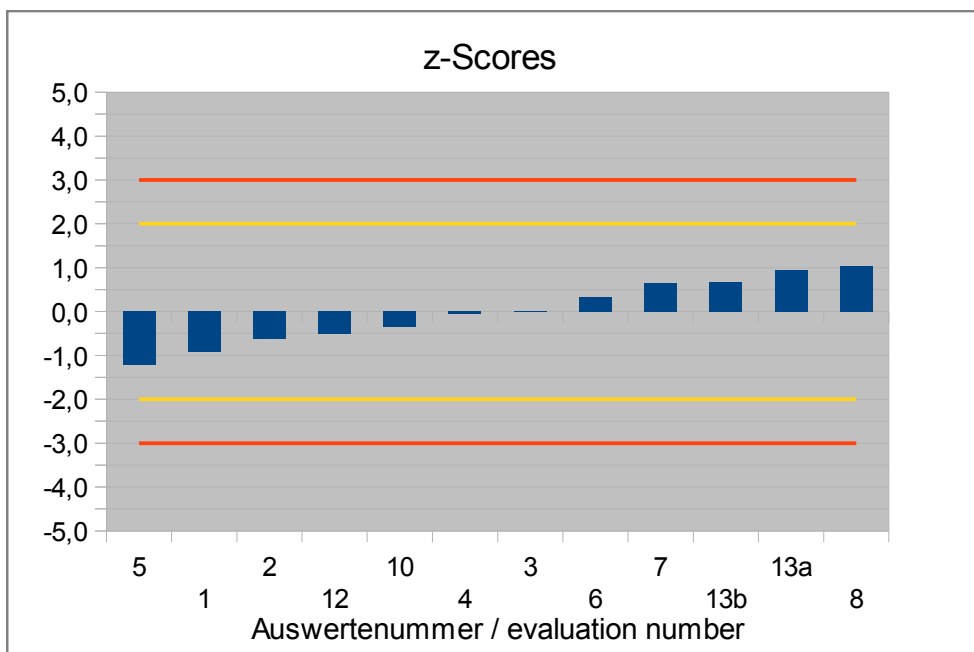


Abb. / Fig. 8: z-Scores Kupfer / Copper

4.5 Fe - Iron in mg/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	12
Number of outliers	0
Mean	340
Median	342
Robust Mean (X_{pt})	341
Robust standard deviation (S^*)	13,3
Number with 2 replicates	13
Repeatability SD (S_r)	8,35
Repeatability (CV_r)	2,45%
Reproducibility SD (S_R)	13,9
Reproducibility (CV_R)	4,08%
Target range:	
Target standard deviation σ_{pt}	16,0
Target standard deviation (for Information)	22,8
lower limit of target range	309
upper limit of target range	373
Quotient S^*/σ_{pt}	0,83
Standard uncertainty $U(X_{pt})$	4,79
Results in the target range	12
Percent in the target range	100%

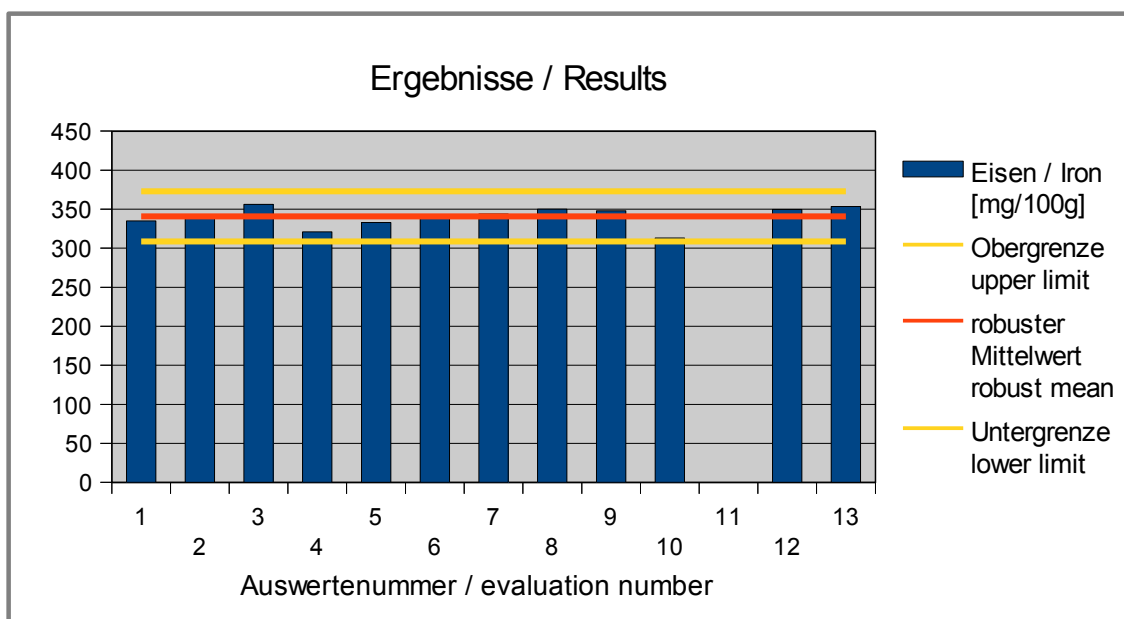


Abb. / Fig. 9: Ergebnisse Eisen / Results Iron

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

Auswertenummer	Eisen / Iron [mg/100g]	Abweichung [mg/100g]	z-Score (σ _{pt})	z-Score (Info)	Hinweis
Evaluation number		Deviation [mg/100g]		(Info)	Remark
1	335	-5,7	-0,36	-0,25	
2	338	-2,7	-0,17	-0,12	
3	356	15,3	1,0	0,67	
4	321	-19,7	-1,2	-0,86	
5	333	-7,7	-0,48	-0,34	
6	340	-0,7	-0,05	-0,03	
7	344	3,3	0,2	0,14	
8	350	9,4	0,58	0,41	
9	348	7,2	0,45	0,31	
10	313	-27,7	-1,7	-1,2	
11					
12	350	8,8	0,55	0,38	
13	353	12,7	0,79	0,55	

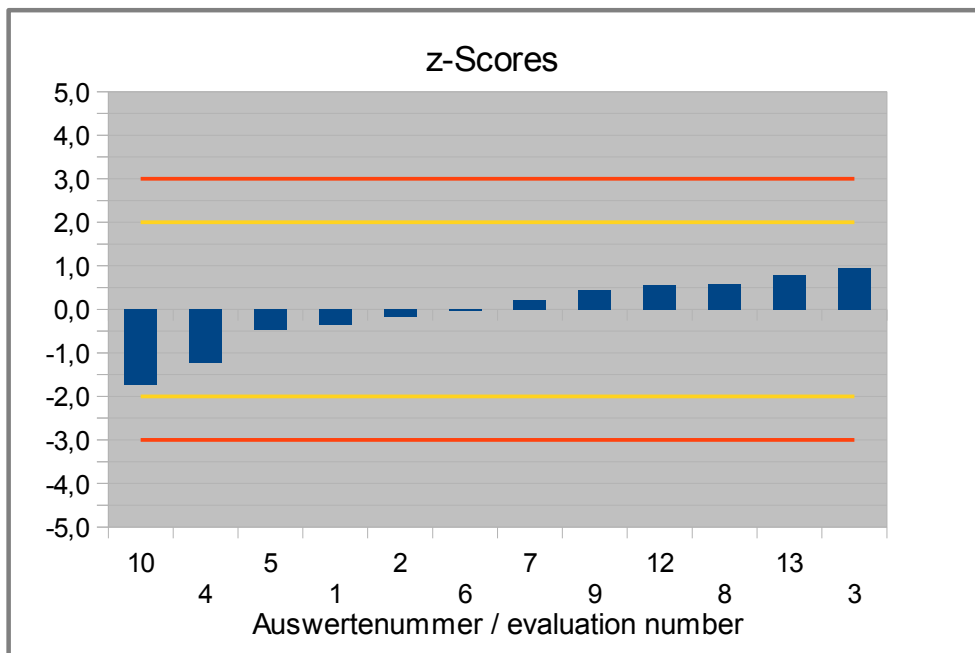


Abb. / Fig. 10: z-Scores Eisen / Iron

4.6 K - Potassium in mg/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	12
Number of outliers	0
Mean	1940
Median	1950
Robust Mean (X_{pt})	1940
Robust standard deviation (S^*)	72,1
Number with 2 replicates	13
Repeatability SD (S_r)	61,9
Repeatability (CV_r)	3,22%
Reproducibility SD (S_R)	100
Reproducibility (CV_R)	5,22%
Target range:	
Target standard deviation σ_{pt}	70,3
Target standard deviation (for Information)	90,7
lower limit of target range	1800
upper limit of target range	2080
Quotient S^*/σ_{pt}	1,0
Standard uncertainty $U(X_{pt})$	26,0
Results in the target range	11
Percent in the target range	92%

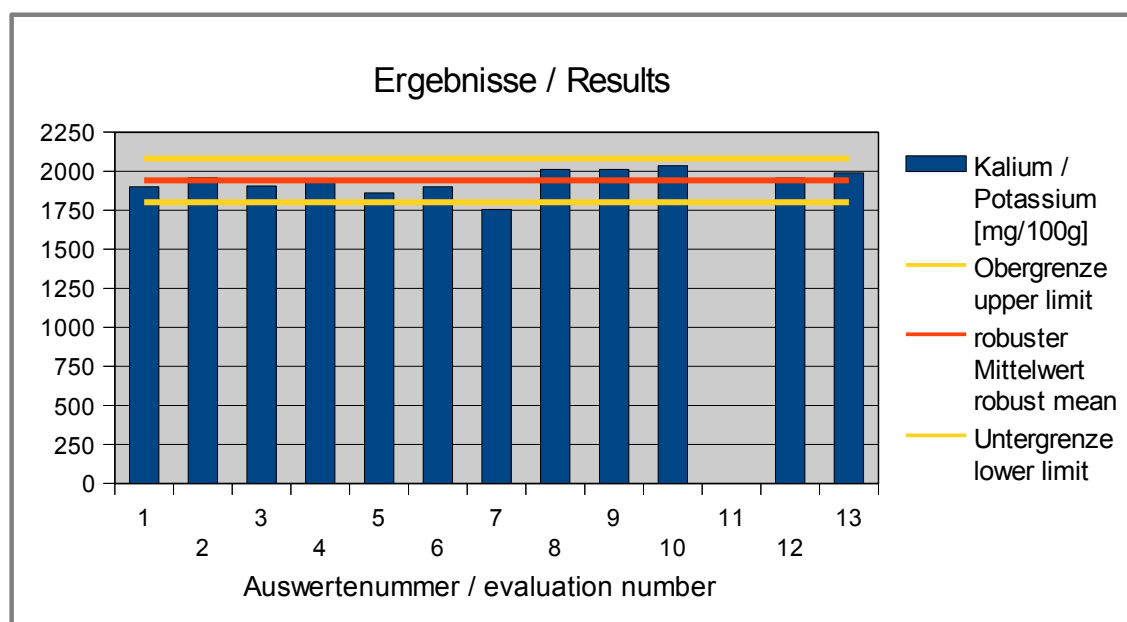


Abb. / Fig. 11: Ergebnisse Kalium / Results Potassium

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

Auswertenummer	Kalium / Potassium [mg/100g]	Abweichung [mg/100g]	z-Score (σ _{pt})	z-Score (Info)	Hinweis
Evaluation number		Deviation [mg/100g]			Remark
1	1900	-41,6	-0,59	-0,46	
2	1958	16,4	0,23	0,18	
3	1905	-36,6	-0,52	-0,40	
4	1939	-2,6	-0,04	-0,03	
5	1860	-81,6	-1,2	-0,90	
6	1900	-41,6	-0,59	-0,46	
7	1755	-186,6	-2,7	-2,1	
8	2012	70,2	1,0	0,77	
9	2012	70,5	1,0	0,78	
10	2035	93,4	1,3	1,0	
11					
12	1957	14,9	0,21	0,16	
13	1988	46,8	0,67	0,52	

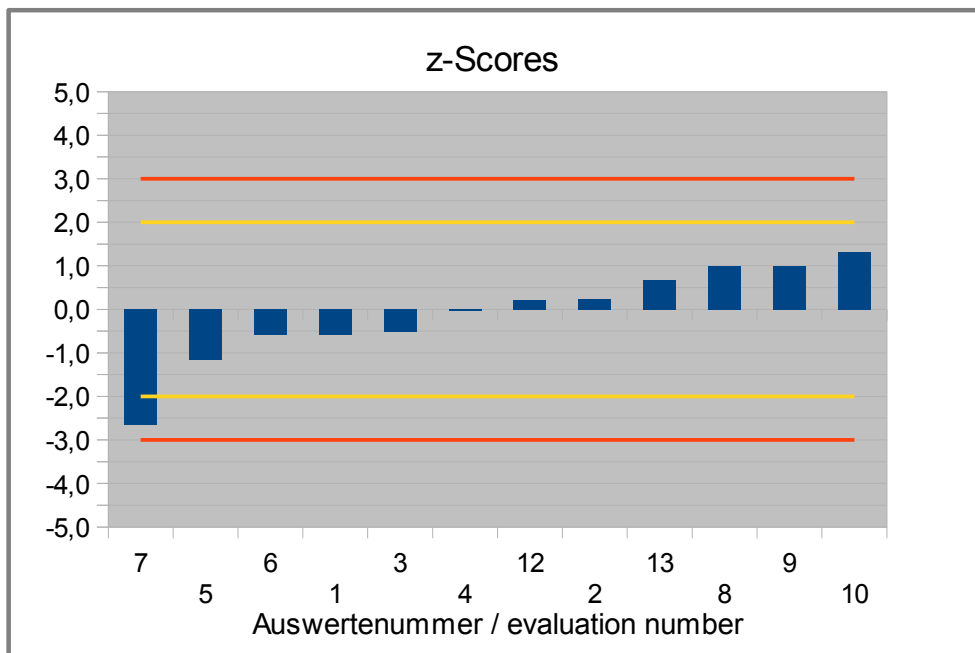


Abb. / Fig. 12: z-Scores Kalium / Potassium

4.7 Mg - Magnesium in mg/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	11
Number of outliers	0
Mean	3450
Median	3480
Robust Mean (X_{pt})	3450
Robust standard deviation (S^*)	248
Number with 2 replicates	11
Repeatability SD (S_r)	78,1
Repeatability (CV_r)	2,26%
Reproducibility SD (S_R)	226
Reproducibility (CV_R)	6,54%
Target range:	
Target standard deviation σ_{pt}	242
Target standard deviation (for Information)	115
lower limit of target range	2970
upper limit of target range	3930
Quotient S^*/σ_{pt}	1,0
Standard uncertainty $U(X_{pt})$	93,5
Results in the target range	11
Percent in the target range	100%

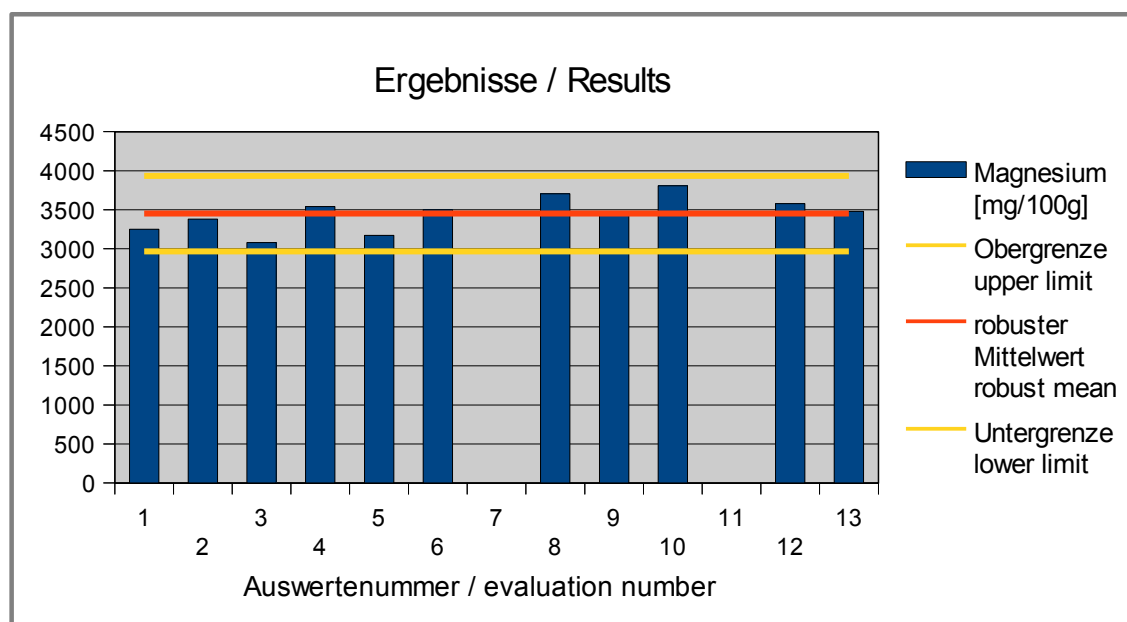


Abb. / Fig. 13: Ergebnisse Magnesium / Results Magnesium

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

Auswertenummer	Magnesium [mg/100g]	Abweichung [mg/100g]	z-Score (σ _{pt})	z-Score (Info)	Hinweis
Evaluation number		Deviation [mg/100g]		(Info)	Remark
1	3250	-200	-0,83	-1,7	
2	3380	-70	-0,29	-0,61	
3	3080	-370	-1,5	-3,2	
4	3540	90	0,37	0,78	
5	3172	-278	-1,2	-2,4	
6	3500	50	0,21	0,44	
7					
8	3705	255	1,1	2,2	
9	3463	12	0,05	0,11	
10	3806	356	1,5	3,1	
11					
12	3578	127	0,53	1,1	
13	3479	29	0,12	0,25	

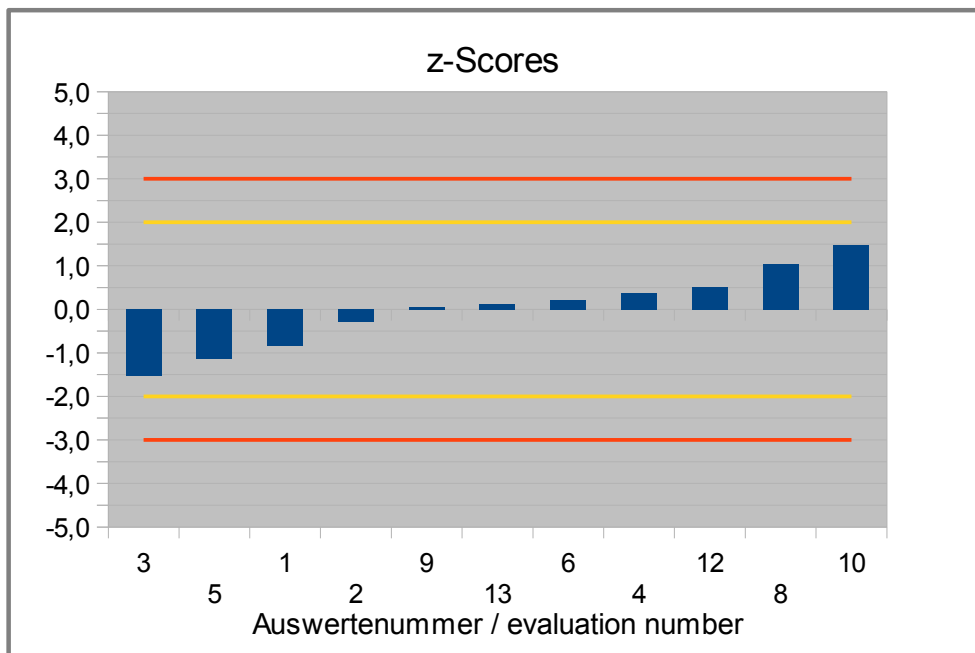


Abb. / Fig. 14: z-Scores Magnesium

4.8 Mn - Manganese in mg/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	11
Number of outliers	0
Mean	39,0
Median	39,9
Robust Mean (X_{pt})	39,0
Robust standard deviation (S^*)	2,61
Number with 2 replicates	12
Repeatability SD (S_x)	1,30
Repeatability (CV_x)	3,33%
Reproducibility SD (S_R)	2,42
Reproducibility (CV_R)	6,19%
Target range:	
Target standard deviation σ_{pt}	2,54
Target standard deviation (for Information)	5,18
lower limit of target range	33,9
upper limit of target range	44,1
Quotient S^*/σ_{pt}	1,0
Standard uncertainty $U(X_{pt})$	0,98
Results in the target range	11
Percent in the target range	100%

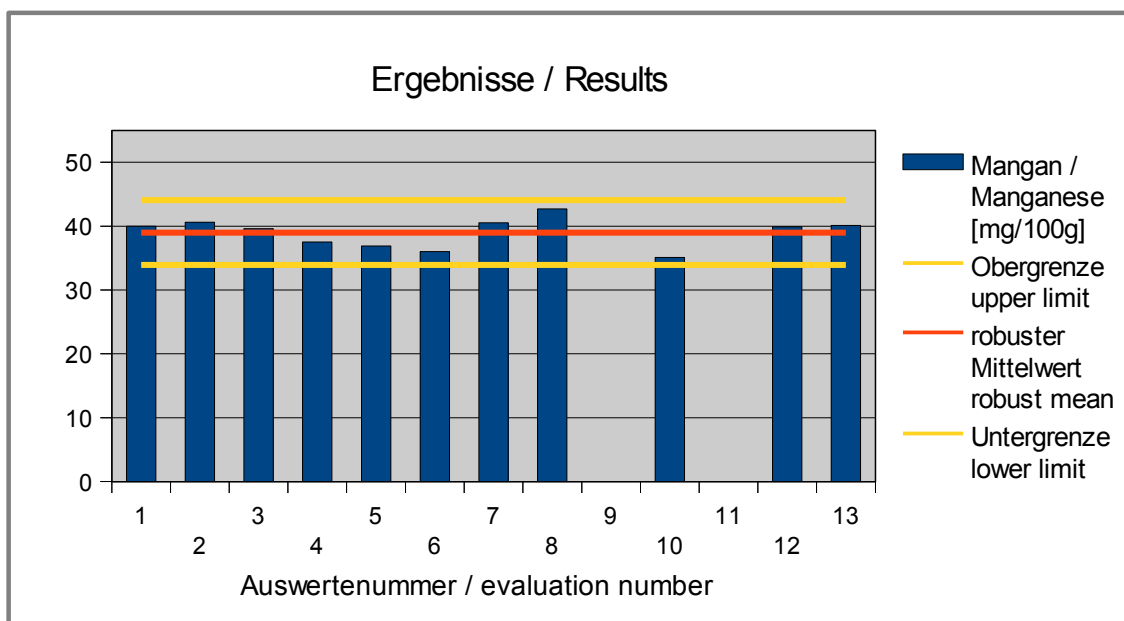


Abb. / Fig. 15: Ergebnisse Mangan / Results Manganese

Ergebnisse der Teilnehmer:
Results of Participants:

Auswertenummer	Mangan / Manganese [mg/100g]	Abweichung [mg/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis
Evaluation number		Deviation [mg/100g]			Remark
1	40,0	1,01	0,40	0,19	
2	40,6	1,61	0,63	0,31	
3	39,6	0,61	0,24	0,12	
4	37,5	-1,49	-0,59	-0,29	
5	36,9	-2,09	-0,82	-0,40	
6	36,0	-2,99	-1,2	-0,58	
7	40,5	1,51	0,59	0,29	
8	42,7	3,71	1,5	0,72	
9					
10	35,1	-3,89	-1,5	-0,75	
11					
12	39,9	0,91	0,36	0,18	
13	40,1	1,12	0,44	0,22	

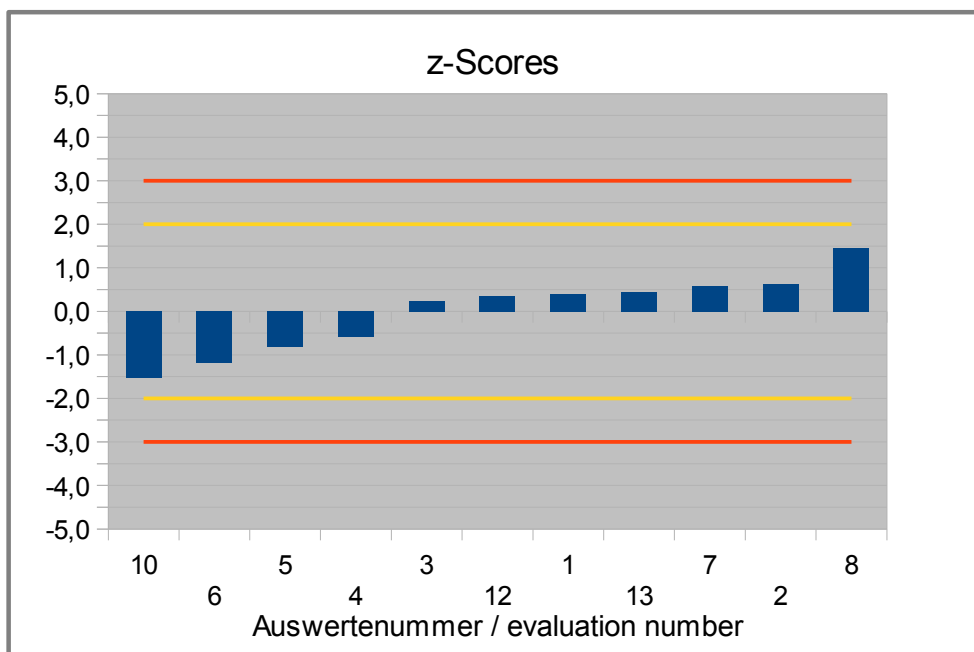


Abb. / Fig. 16: z-Scores Mangan / Manganese

4.9 Mo Molybdenum in $\mu\text{g}/100\text{g}$

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	10
Number of outliers	0
Mean	1230
Median	1230
Robust Mean (X_{pt})	1230
Robust standard deviation (S^*)	197
Number with 2 replicates	10
Repeatability SD (S_r)	55,3
Repeatability (CV_r)	4,50%
Reproducibility SD (S_R)	187
Reproducibility (CV_R)	15,2%
Target range:	
Target standard deviation σ_{pt}	134
Target standard deviation (for Information)	251
lower limit of target range	957
upper limit of target range	1500
Quotient S^*/σ_{pt}	1,5
Standard uncertainty $U(X_{pt})$	78,0
Results in the target range	9
Percent in the target range	90%

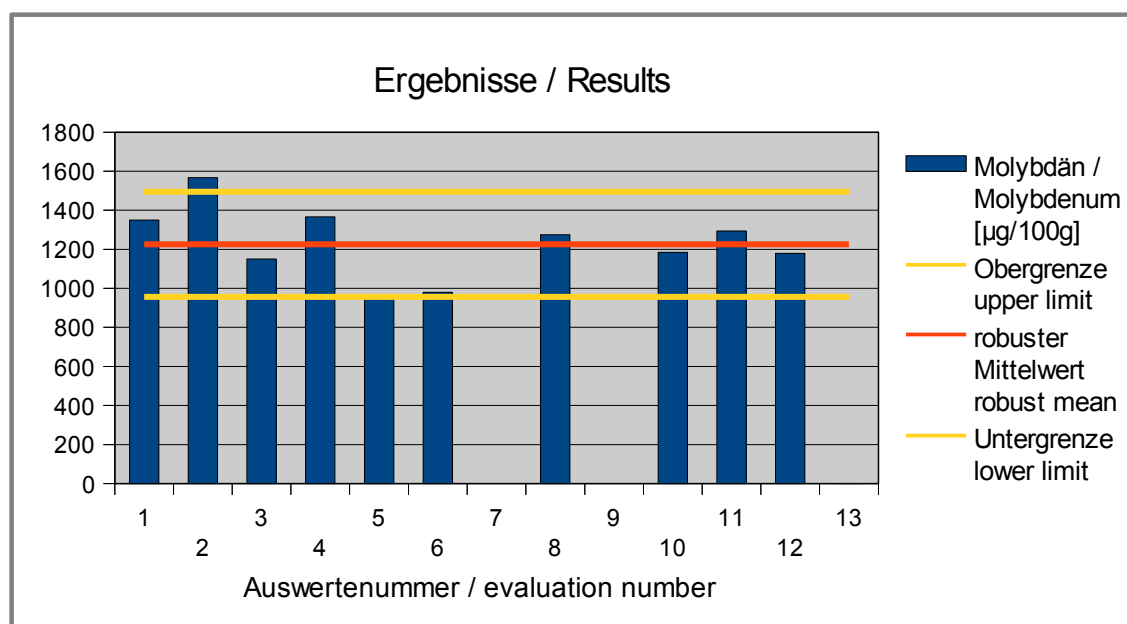


Abb. / Fig. 17: Ergebnisse Molybdän / Results Molybdenum

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

Auswertenummer Evaluation number	Molybdän / Molybdenum [µg/100g]	Abweichung [µg/100g] Deviation [µg/100g]	z-Score (σ _{pt})	z-Score (Info)	Hinweis Remark
1	1350	124	0,92	0,50	
2	1567	341	2,5	1,4	
3	1150	-76	-0,56	-0,30	
4	1366	140	1,0	0,56	
5	956	-270	-2,0	-1,1	
6	980	-246	-1,8	-0,98	
7					
8	1275	50	0,37	0,20	
9					
10	1185	-41	-0,30	-0,16	
11	1294	68	0,51	0,27	
12	1180	-46	-0,34	-0,18	
13	0,00145				Ausreißer ausgeschlossen / Outlier excluded

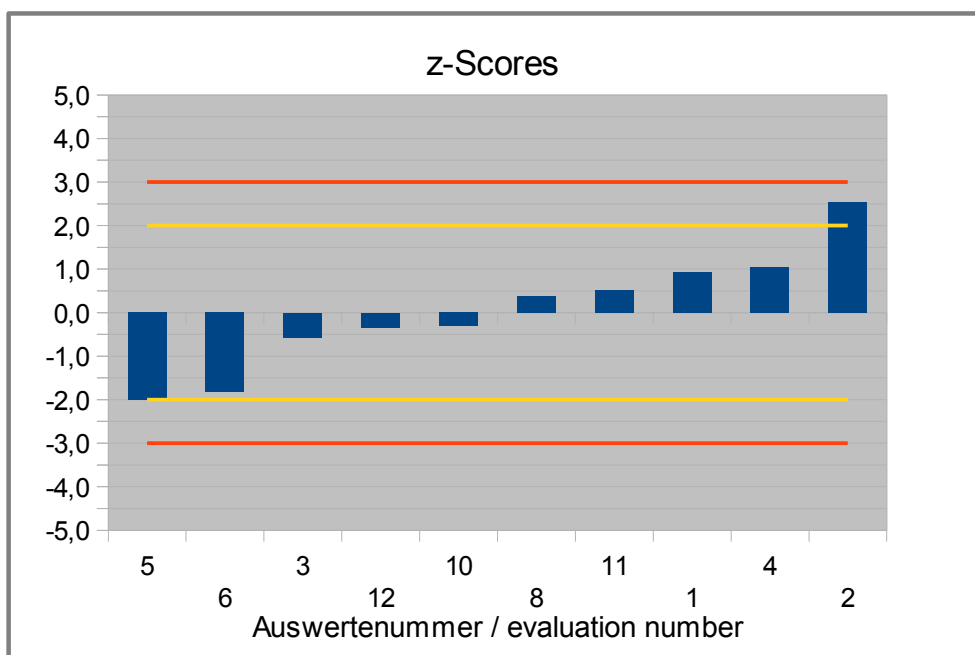


Abb. / Fig. 18: z-Scores Molybdän / Molybdenum

4.10 P - Phosphorus in mg/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	9
Number of outliers	0
Mean	1740
Median	1760
Robust Mean (X_{pt})	1740
Robust standard deviation (S^*)	73,2
Number with 2 replicates	9
Repeatability SD (S_r)	42,1
Repeatability (CV_r)	2,43%
Reproducibility SD (S_R)	76,9
Reproducibility (CV_R)	4,43%
Target range:	
Target standard deviation σ_{pt}	64,0
Target standard deviation (for Information)	130
lower limit of target range	1620
upper limit of target range	1870
Quotient S^*/σ_{pt}	1,1
Standard uncertainty $U(X_{pt})$	30,5
Results in the target range	9
Percent in the target range	100%

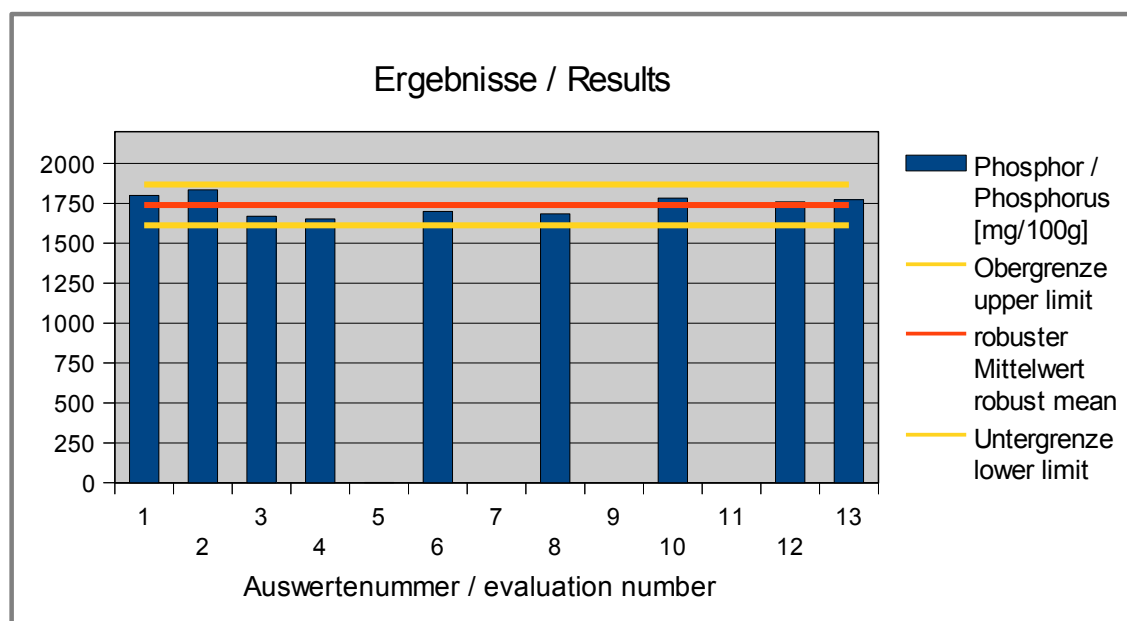


Abb. / Fig. 19: Ergebnisse Phosphor / Results Phosphorus

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

Auswertenummer	Phosphor / Phosphorus [mg/100g]	Abweichung [mg/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis
Evaluation number		Deviation [mg/100g]			Remark
1	1800	60,5	0,94	0,46	
2	1835	95,5	1,5	0,73	
3	1670	-69,5	-1,1	-0,53	
4	1652	-87,5	-1,4	-0,67	
5	1,71				Ausreißer ausgeschlossen / Outlier excluded
6	1700	-39,5	-0,62	-0,30	
7					
8	1684	-56,0	-0,87	-0,43	
9					
10	1783	43,5	0,68	0,33	
11					
12	1759	19,5	0,30	0,15	
13	1773	33,6	0,52	0,26	

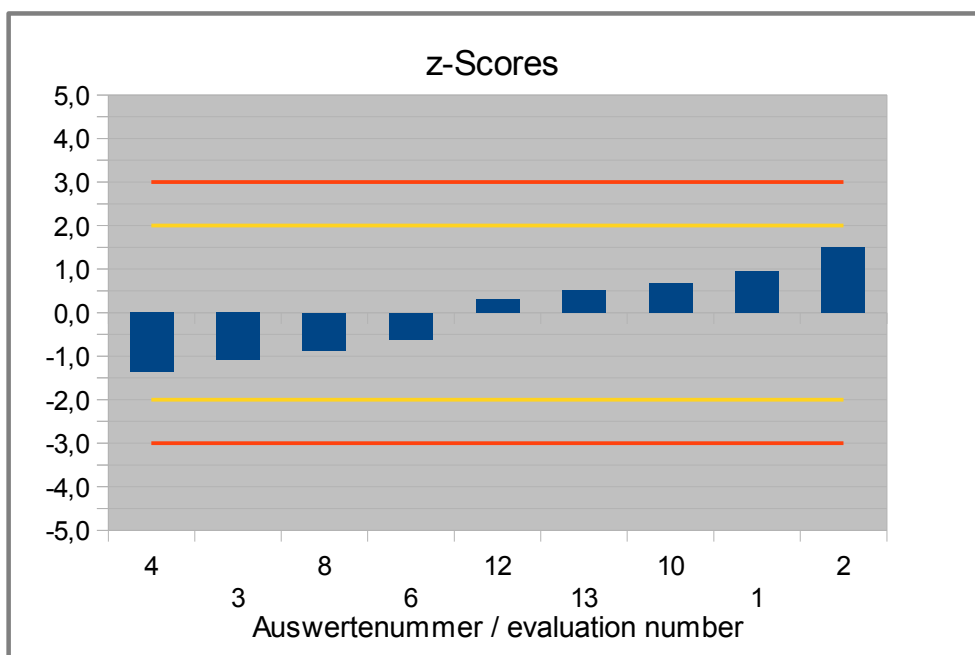


Abb. / Fig. 20: z-Scores Phosphor / Phosphorus

4.11 Se - Selenium in µg/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	11
Number of outliers	0
Mean	1970
Median	1980
Robust Mean (X_{pt})	1960
Robust standard deviation (S^*)	99,2
Number with 2 replicates	12
Repeatability SD (S_r)	59,0
Repeatability (CV_r)	3,00%
Reproducibility SD (S_R)	99,4
Reproducibility (CV_R)	5,07%
<i>Target range:</i>	
Target standard deviation σ_{pt}	201
Target standard deviation (for Information)	144
lower limit of target range	1560
upper limit of target range	2360
Quotient S^*/σ_{pt}	0,49
Standard uncertainty $U(X_{pt})$	37,4
Results in the target range	11
Percent in the target range	100%

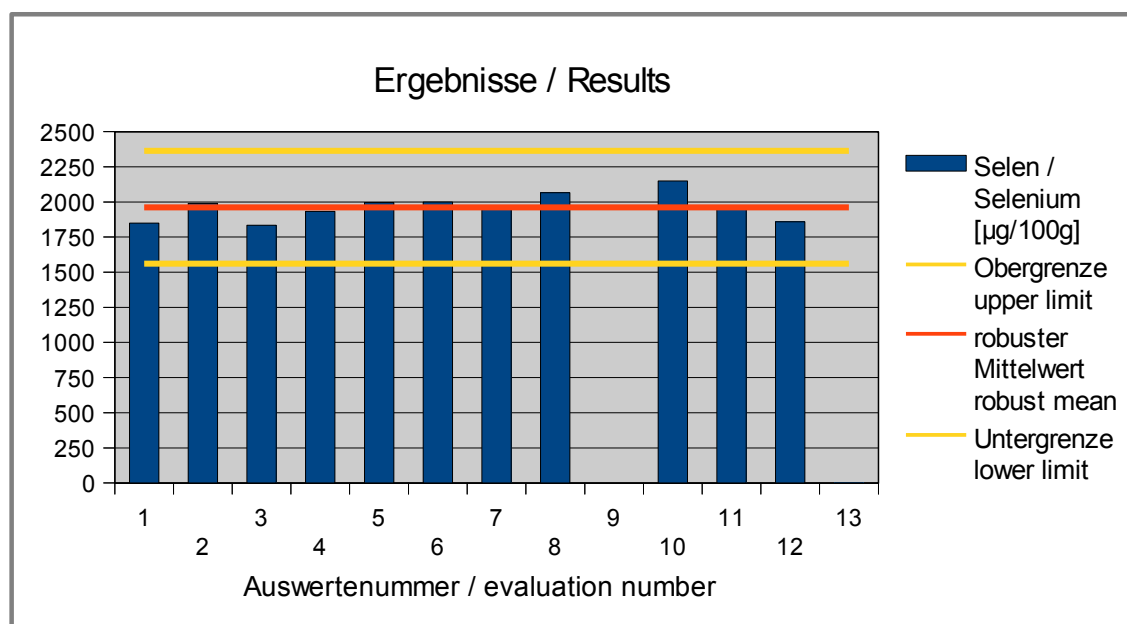


Abb. / Fig. 21: Ergebnisse Selen / Results Selenium

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

Auswertenummer	Selen / Selenium [µg/100g]	Abweichung [µg/100g]	z-Score (σ _{pt})	z-Score (Info)	Hinweis
Evaluation number		Deviation [µg/100g]		(Info)	Remark
1	1850	-112,1	-0,56	-0,78	
2	1990	27,9	0,14	0,19	
3	1835	-127,1	-0,63	-0,89	
4	1933	-29,1	-0,15	-0,20	
5	1996	33,9	0,17	0,24	
6	2000	37,9	0,19	0,26	
7	1975	12,9	0,06	0,09	
8	2066	103,6	0,52	0,72	
9					
10	2150	187,9	0,94	1,31	
11	1968	5,9	0,03	0,04	
12	1860	-102,6	-0,51	-0,71	
13	0,00164				Ausreißer ausgeschlossen / Outlier excluded



Abb. / Fig. 22: z-Scores Selen / Selenium

4.12 Zn - Zinc in mg/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	12
Number of outliers	0
Mean	317
Median	320
Robust Mean (X_{pt})	317
Robust standard deviation (S^*)	11,8
Number with 2 replicates	13
Repeatability SD (S_r)	8,31
Repeatability (CV_r)	2,62%
Reproducibility SD (S_R)	12,1
Reproducibility (CV_R)	3,81%
Target range:	
Target standard deviation σ_{pt}	15,1
Target standard deviation (for Information)	21,0
lower limit of target range	287
upper limit of target range	347
Quotient S^*/σ_{pt}	0,79
Standard uncertainty $U(X_{pt})$	4,27
Results in the target range	12
Percent in the target range	100%

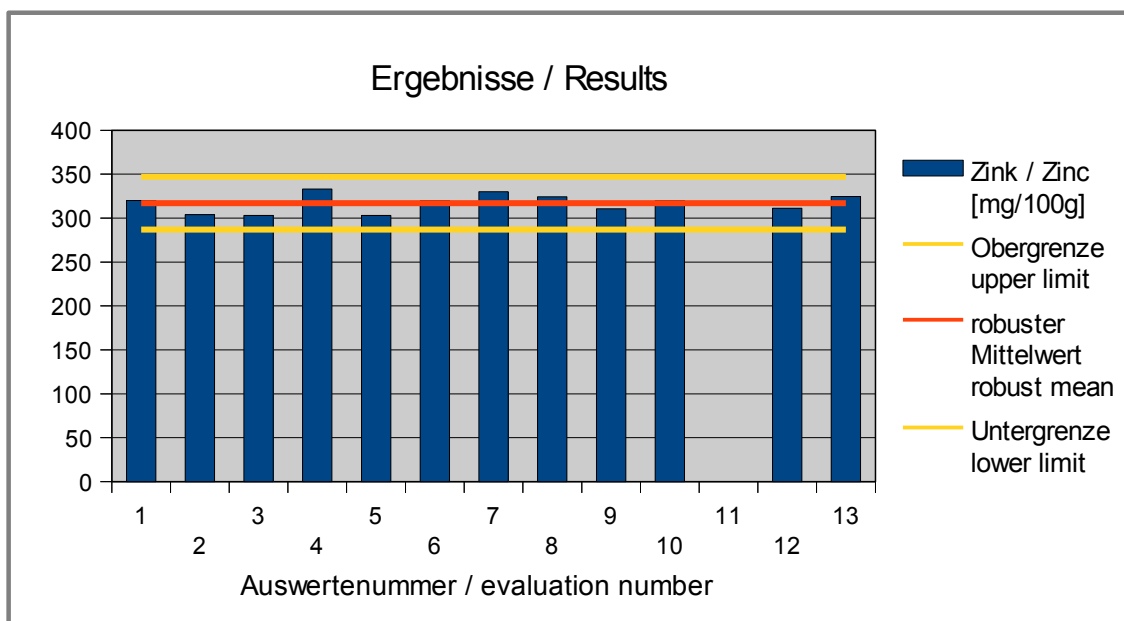


Abb. / Fig. 23: Ergebnisse Zink / Results Zinc

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

Auswertenummer	Zink / Zinc [mg/100g]	Abweichung [mg/100g]	z-Score (σ _{pt})	z-Score (Info)	Hinweis
Evaluation number		Deviation [mg/100g]		(Info)	Remark
1	320	3,1	0,20	0,15	
2	304	-12,9	-0,86	-0,61	
3	303	-13,9	-0,92	-0,66	
4	333	16,1	1,1	0,76	
5	303	-13,9	-0,92	-0,66	
6	320	3,1	0,20	0,15	
7	330	13,1	0,87	0,62	
8	324	7,3	0,48	0,35	
9	310	-6,5	-0,43	-0,31	
10	320	3,1	0,20	0,15	
11					
12	311	-5,9	-0,39	-0,28	
13	325	7,6	0,50	0,36	

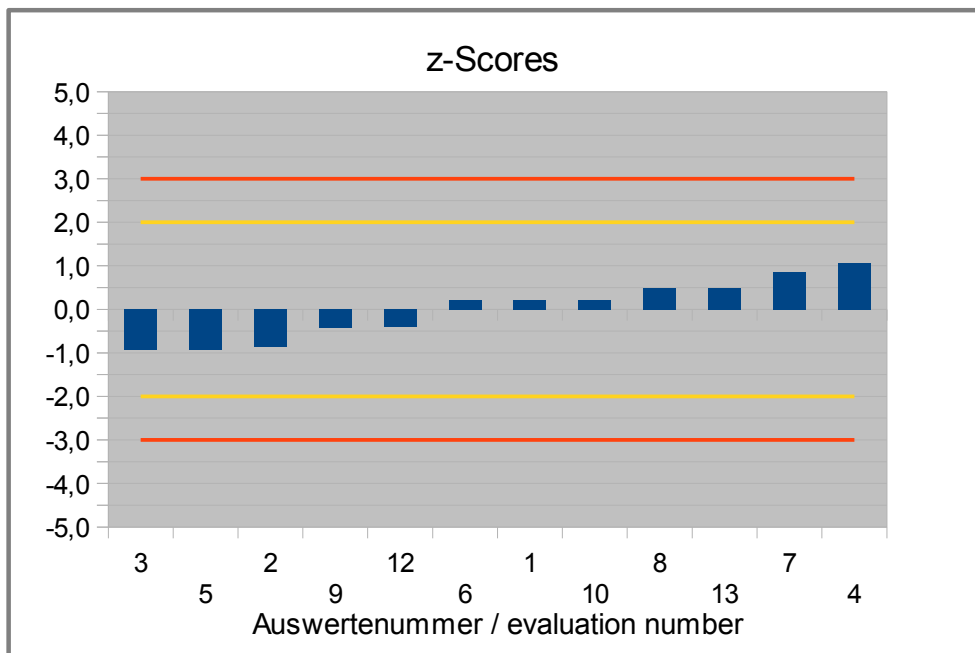


Abb. / Fig. 24: z-Scores Zink / Zinc

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

Analyte	Participant	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana- lysis	Result (Mean)	Result I	Result II	Limit of determination	Incl. RR	Recovery rate
					Day/Month					yes / no	in %
B – Bor / Boron	1	mg/100g	21	63	20.09.19	61	62	60	0,2	no	
	2	mg/100g	39	45	10.08.19	64,8	64,8	64,7	0.01	no	
	3	mg/100g	11	73	21.08.19	591	592	590	0,01	no	-
	4	mg/100g	35	49		60,4	60	60,8	< 0,02	no	
	5	mg/100g	23	61							
	6	mg/100g	28	56	01.08.19	64	64	64	2,5	no	
	7	mg/100g	2 / 66	18 / 82							
	8	mg/100g	8	76							
	9	mg/100g	32	52							
	10	mg/100g	36	48	07.08.19	63,2	62,1	64,3	0,5	no	-
	11	mg/100g	22	62							
	12	mg/100g	12	72	08.08.19	62,8	62,3	63,3	0,2	no	
	13a	mg/100g	37	47	26.08.19	66,05	66,03	66,07	5	no	NA
13b	mg/100g	37	47	26.08.19	63,78	63,94	63,62	1	no	NA	

Analyte	Participant	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana- lysis	Result (Mean)	Result I	Result II	Limit of determination	Incl. RR	Recovery rate
					Day/Month					yes / no	in %
Ca - Calcium	1	mg/100g	21	63	20.09.19	5250	5300	5200	1	no	
	2	mg/100g	39	45	10.08.19	5070	4980	5160	0.2	no	
	3	mg/100g	11	73	21.08.19	4880	4930	4830	5	no	-
	4	mg/100g	35	49		5620	5643	5597	< 5	no	
	5	mg/100g	23	61	18.09.19	4892	4863	4921	0,2		
	6	mg/100g	28	56	01.08.19	5350	5200	5500	60	no	
	7a	mg/100g	2	18	16.Sep.	4615	4670	4600		no	
	7b	mg/100g	66	82	16.Sep.	4615	4610	4580		no	
	8	mg/100g	8	76	19.09.19	5341,7	5493	5190,4		no	
	9	mg/100g	32	52	15.08.19	5118,8	5070,4	5167,3		no	96,5
	10	mg/100g	36	48	07.08.19	5681	5663	5700	5	no	-
	11	mg/100g	22	62							
	12	mg/100g	12	72	08.08.19	5648,5	5539	5758	0,2	no	
13	mg/100g	37	47	28.08.19	5385,6	5435,8	5335,5	2,5	no	NA	

Analyte	Participant	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana- lysis	Result (Mean)	Result I	Result II	Limit of determination	Incl. RR	Recovery rate
					Day/Month					yes / no	in %
Cr – Chrom / Chromium	1	mg/100g	21	63	20.09.19	1350	1400	1300	20	no	
	2	mg/100g	39	45	10.08.19	1464	1470	1460	3	no	
	3	mg/100g	11	73	21.08.19	1440	1420	1460	5	no	-
	4	mg/100g	35	49		1408	1401	1415	< 10	no	
	5	mg/100g	23	61	19.08.19	382	396	368	1		
	6	mg/100g	28	56	01.08.19	1100	1000	1200	125	no	
	7a	mg/100g	2	18	16.Sep.	1793	1810	1790		no	
	7b	mg/100g	66	82	16.Sep.	1793	1780	1790		no	
	8	mg/100g	8	76	19.09.19	1460,2	1540,7	1379,6		no	
	9	mg/100g	32	52							
	10	mg/100g	36	48	07.08.19	1270	1260	1280	0,5	no	-
	11	mg/100g	22	62							
	12	mg/100g	12	72	08.08.19	1330	1315	1345	40	no	
13	mg/100g	37	47	27.08.19	0,001484	0,001495	0,001472	0,00025	no	NA	

Analyte	Participant	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana- lysis	Result (Mean)	Result I	Result II	Limit of determination	Incl. RR	Recovery rate
					Day/Month						
Cu – Kupfer / Copper	1	mg/100g	21	63	20.09.19	41,5	43	40	0,01	no	
	2	mg/100g	39	45	10.08.19	42,3	41,9	42,7	0.01	no	
	3	mg/100g	11	73	21.08.19	44,1	43,9	44,2	0,01	no	-
	4	mg/100g	35	49		43,9	43,1	44,7	< 0,05	no	
	5	mg/100g	23	61	19.08.19	40,6	40,9	40,2	0,02		
	6	mg/100g	28	56	01.08.19	45	45	45	1	no	
	7a	mg/100g	2	18	16.Sep.	45,9	46,7	45,4		no	
	7b	mg/100g	66	82	16.Sep.	45,9	45,7	45,8		no	
	8	mg/100g	8	76	19.09.19	47	48,8	45,2		no	
	9	mg/100g	32	52							
	10	mg/100g	36	48	07.08.19	43,1	42,2	44	0,5	no	-
	11	mg/100g	22	62							
	12	mg/100g	12	72	08.08.19	42,65	41,7	43,6	0,1	no	
	13a	mg/100g	37	47	28.08.19	46,76	46,98	46,54	0,5	no	NA
13b	mg/100g	37	47	26.08.19	45,97	46,34	45,61	1	no	NA	

Analyte	Participant	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana- lysis	Result (Mean)	Result I	Result II	Limit of determination	Incl. RR	Recovery rate
					Day/Month						
Fe – Eisen / Iron	1	mg/100g	21	63	20.09.19	335	340	330	0,1	no	
	2	mg/100g	39	45	10.08.19	338	336	339	0.05	no	
	3	mg/100g	11	73	21.08.19	356	356	355	0,1	no	-
	4	mg/100g	35	49		321	319	322	< 0,05	no	
	5	mg/100g	23	61	17.09.19	333	328	339	1		
	6	mg/100g	28	56	01.08.19	340	350	330	1	no	
	7a	mg/100g	2	18	16.Sep.	344	349	344		no	
	7b	mg/100g	66	82	16.Sep.	344	344	338		no	
	8	mg/100g	8	76	19.09.19	350,1	358	342,2		no	
	9	mg/100g	32	52	08.08.19	347,91	348,34	347,47		no	100,4
	10	mg/100g	36	48	07.08.19	313	314	312	0,5	no	-
	11	mg/100g	22	62							
	12	mg/100g	12	72	08.08.19	349,5	335	364	0,2	no	
	13	mg/100g	37	47	28.08.19	353,4	355,3	351,4	5	no	NA

Analyte	Participant	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana- lysis	Result (Mean)	Result I	Result II	Limit of determination	Incl. RR	Recovery rate
					Day/Month					yes / no	in %
K – Kalium / Potassium	1	mg/100g	21	63	20.09.19	1900	1900	1900	1	no	
	2	mg/100g	39	45	10.08.19	1958	1950	1970	0.5	no	
	3	mg/100g	11	73	21.08.19	1905	1850	1960	1	no	-
	4	mg/100g	35	49		1939	1936	1941	< 5	no	
	5	mg/100g	23	61	18.09.19	1860	1869	1850	1		
	6	mg/100g	28	56	01.08.19	1900	1800	2000	85	no	
	7a	mg/100g	2	18	16.Sep.	1755	1790	1750		no	
	7b	mg/100g	66	82	16.Sep.	1755	1750	1730		no	
	8	mg/100g	8	76	19.09.19	2011,8	2091,2	1932,4		no	
	9	mg/100g	32	52	16.08.19	2012,1	1963,3	2060,9		no	97,3
	10	mg/100g	36	48	07.08.19	2035	2030	2040	5	no	-
	11	mg/100g	22	62							
	12	mg/100g	12	72	08.08.19	1956,5	1912	2001	4	no	
13	mg/100g	37	47	28.08.19	1988,4	1966,5	2010,3	2,5	no	NA	

Analyte	Participant	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana- lysis	Result (Mean)	Result I	Result II	Limit of determination	Incl. RR	Recovery rate
					Day/Month					yes / no	in %
Mg - Magnesium	1	mg/100g	21	63	20.09.19	3250	3300	3200	0,3	no	
	2	mg/100g	39	45	10.08.19	3380	3370	3390	0.05	no	
	3	mg/100g	11	73	21.08.19	3080	3130	3030	1	no	-
	4	mg/100g	35	49		3540	3552	3527	< 5	no	
	5	mg/100g	23	61	18.09.19	3172	3146	3197	0,2		
	6	mg/100g	28	56	01.08.19	3500	3400	3600	75	no	
	7	mg/100g	2 / 66	18 / 82							
	8	mg/100g	8	76	19.09.19	3704,9	3797,8	3611,9		no	
	9	mg/100g	32	52	15.08.19	3462,6	3485,5	3439,6		no	96,5
	10	mg/100g	36	48	07.08.19	3806	3735	3878	5	no	-
	11	mg/100g	22	62							
	12	mg/100g	12	72	08.08.19	3577,5	3527	3628	0,2	no	
	13	mg/100g	37	47	28.08.19	3478,8	3507	3450,5	2,5	no	NA

Analyte	Participant	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana- lysis	Result (Mean)	Result I	Result II	Limit of determination	Incl. RR	Recovery rate
					Day/Month					yes / no	in %
Mn -Mangan / Manganese	1	mg/100g	21	63	20.09.19	40	41	39	0,01	no	
	2	mg/100g	39	45	10.08.19	40,6	40,1	41	0.0005	no	
	3	mg/100g	11	73	21.08.19	39,6	39,2	39,9	0,005	no	-
	4	mg/100g	35	49		37,5	37,4	37,6	< 0,05	no	
	5	mg/100g	23	61	19.08.19	36,9	36,1	37,8	0,01		
	6	mg/100g	28	56	01.08.19	36	35	37	1	no	
	7a	mg/100g	2	18	16.Sep.	40,5	41	40		no	
	7b	mg/100g	66	82	16.Sep.	40,5	42	39		no	
	8	mg/100g	8	76	19.09.19	42,7	44,5	41		no	
	9	mg/100g	32	52							
	10	mg/100g	36	48	07.08.19	35,1	34,4	35,7	0,5	no	-
	11	mg/100g	22	62							
	12	mg/100g	12	72	08.08.19	39,9	38,9	40,9	0,1	no	
13	mg/100g	37	47	26.08.19	40,11	39,77	40,45	0,25	no	NA	

Analyte	Participant	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana- lysis	Result (Mean)	Result I	Result II	Limit of determination	Incl. RR	Recovery rate
					Day/Month					yes / no	in %
Mo – Molybdän / Molybdenum	1	µg/100g	21	63	20.09.19	1350	1300	1400	10	no	
	2	µg/100g	39	45	10.08.19	1567	1510	1620	0.5	no	
	3	µg/100g	11	73	21.08.19	1150	1130	1170	5	no	-
	4	µg/100g	35	49		1366	1376	1356	< 10	no	
	5	µg/100g	23	61	19.08.19	956	969	944	50		
	6	µg/100g	28	56	01.08.19	980	950	1000	125	no	
	7	µg/100g	2 / 66	18 / 82							
	8	µg/100g	8	76	19.09.19	1275,3	1351,8	1198,8		no	
	9	µg/100g	32	52							
	10	µg/100g	36	48	07.08.19	1185	1180	1190	0,5	no	-
	11	µg/100g	22	62	05.09.19	1294	1282	1306	0.025 ppm	N/A	N/A
	12	µg/100g	12	72	28.08.19	1179,5	1229	1130	100	no	
	13	µg/100g	37	47	26.08.19	0,001447	0,00145	0,001444	0,00025	no	NA

Analyte	Participant	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana- lysis	Result (Mean)	Result I	Result II	Limit of determination	Incl. RR	Recovery rate
					Day/Month					yes / no	in %
P – Phosphor / Phosphorus	1	mg/100g	21	63	20.09.19	1800	1800	1800	0,2	no	
	2	mg/100g	39	45	10.08.19	1835	1820	1850	0.05	no	
	3	mg/100g	11	73	21.08.19	1670	1630	1700	2	no	-
	4	mg/100g	35	49		1652	1642	1662	< 5	no	
	5	mg/100g	23	61	30.08.19	1,71	1,7	1,72	1,5		
	6	mg/100g	28	56	01.08.19	1700	1600	1700	25	no	
	7	mg/100g	2 / 66	18 / 82							
	8	mg/100g	8	76	19.09.19	1683,5	1737,7	1629,3		no	
	9	mg/100g	32	52							
	10	mg/100g	36	48	07.08.19	1783	1773	1793	5	no	-
	11	mg/100g	22	62							
	12	mg/100g	12	72	08.08.19	1759	1730	1788	0,2	no	
	13	mg/100g	37	47	28.08.19	1773,1	1778,8	1767,5	2,5	no	NA

Analyte	Participant	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana- lysis	Result (Mean)	Result I	Result II	Limit of determination	Incl. RR	Recovery rate
					Day/Month					yes / no	in %
Se – Selen / Selenium	1	mg/100g	21	63	20.09.19	1850	1900	1800	20	no	
	2	mg/100g	39	45	10.08.19	1990	1920	2070	10	no	
	3	mg/100g	11	73	21.08.19	1835	1800	1870	1	no	-
	4	mg/100g	35	49		1933	1918	1948	< 10	no	
	5	mg/100g	23	61	19.08.19	1996	1989	2003	5		
	6	mg/100g	28	56	01.08.19	2000	2000	1900	125	no	
	7a	mg/100g	2	18	16.Sep.	1975	2040	1950		no	
	7b	mg/100g	66	82	16.Sep.	1975	1970	1940		no	
	8	mg/100g	8	76	19.09.19	2065,7	2140,8	1990,6		no	
	9	mg/100g	32	52							
	10	mg/100g	36	48	07.08.19	2150	2130	2170	0,5	no	-
	11	mg/100g	22	62	05.09.19	1968	1950	1985	0.05 ppm	N/A	N/A
	12	mg/100g	12	72	28.08.19	1859,5	1848	1871	200	no	
13	mg/100g	37	47	27.08.19	0,00164	0,00167	0,001609	0,00025	no	NA	

Analyte	Participant	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana- lysis	Result (Mean)	Result I	Result II	Limit of determination	Incl. RR	Recovery rate
					Day/Month					yes / no	in %
Zn – Zink / Zinc	1	mg/100g	21	63	20.09.19	320	320	320	0,02	no	
	2	mg/100g	39	45	10.08.19	304	304	305	0.01	no	
	3	mg/100g	11	73	21.08.19	303	301	305	0,01	no	-
	4	mg/100g	35	49		333	331	334	< 0,05	no	
	5	mg/100g	23	61	18.09.19	303	299	307	0,1		
	6	mg/100g	28	56	01.08.19	320	330	300	1,5	no	
	7a	mg/100g	2	18	16.Sep.	330	335	327		no	
	7b	mg/100g	66	82	16.Sep.	330	333	326		no	
	8	mg/100g	8	76	19.09.19	324,2	332,3	316,1		no	
	9	mg/100g	32	52	08.08.19	310,47	310,81	310,12		no	100,8
	10	mg/100g	36	48	07.08.19	320	318	322	0,5	no	-
	11	mg/100g	22	62							
	12	mg/100g	12	72	08.08.19	311	301	321	0,2	no	
13	mg/100g	37	47	28.08.19	324,5	326,4	322,6	1	no	NA	

5.1.2 Analytical Methods

Analyte	Participant	Method description as in test report / norm / literature	Homogenization	Sample weight	Hydrolyzation Method	Hydrolyzation Solution	Calibration and reference material	Method accredited ISO/IEC 17025 yes / no	Further remarks
B – Bor / Boron	1	acid digestion	no	1g			CRM	yes	
	2	ICP-MS (EPA 6020)	full amount of the sample was homogenized before analysis	0.25 g	acid digestion, MSZ EN 13805:2015	5 ml nitric acid + 2 ml hydrogen peroxide	calibration solutions: CPAchem 7105L-0-B9-62, Sigma-Aldrich 49596-100ML; check solutions: Ultra Scientific IMS-102, High-Purity Standards ICP-AM-15-5M, Merck 1.19898.0500	yes	
	3	DIN EN 15763 : 2010 mod.	hand mortar	800 mg	DIN EN 13805 : 2014	HNO ₃ + H ₂ O ₂	external cali./ Celery powder	yes	
	4	DIN EN ISO 11885 (E 22) (2009-09)		approx. 0.5 g / 25 ml weighed exactly	Microwave pressure digestion	HNO ₃ , H ₂ O ₂ , H ₂ O		yes	
	5								
	6	ICP-OES; DIN EN ISO 11885-E22	shaking	0,4g	L155; Microwave pressure digestion	HNO ₃ /H ₂ O	7-point calibration	yes	
	7								
	8								
	9								
	10	DIN EN ISO 11885	mixing	ca. 0,5 g	microwave	HNO ₃	external	yes	
	11								
	12	ASU L 00.00-144	yes	0,5	ASU L 00.00-19/1	HNO ₃	Plant and animal RM	yes	
	13a	ICP-OES (MET-209)	mixing	< 1 g	pressure digestion, microwave	HNO ₃ -H ₂ O ₂	CRM	no	
13b	ICP-MS (MET-206)	mixing	< 1 g	pressure digestion, microwave	HNO ₃ -H ₂ O ₂	CRM	no		

Analyte	Participant	Method description as in test report / norm / literature	Homogenization	Sample weight	Hydrolyzation Method	Hydrolyzation Solution	Calibration and reference material	Method accredited ISO/IEC 17025 yes / no	Further remarks
Ca - Calcium	1	acid digestion	no	1g			CRM	yes	
	2	ICP-MS (EPA 6020)	full amount of the sample was homogenized before analysis	0.25 g	acid digestion, MSZ EN 13805:2015	5 ml nitric acid + 2 ml hydrogen peroxide	calibration solutions: CPAchem 7105L-0-B9-62, Sigma-Aldrich 49596-100ML; check solutions: Ultra Scientific IMS-102, High-Purity Standards ICP-AM-15-5M, Merck 1.19898.0500	yes	
	3	ASU L 00.00-144 : 2019	hand mortar	800 mg	DIN EN 13805 : 2014	HNO ₃ + H ₂ O ₂	external cali./ Milk powder	yes	
	4	DIN EN ISO 11885 (E 22) (2009-09)		approx. 0.5 g / 25 ml weighed exactly	Microwave pressure digestion	HNO ₃ , H ₂ O ₂ , H ₂ O		yes	
	5	EN 15763	no	0,2g	pressure digestion	HNO ₃	external calibration	yes	
	6	ICP-OES; DIN EN ISO 11885-E22	shaking	0,4g	L155; microwave pressure digestion	HNO ₃ /H ₂ O	3-point calibration	yes	
	7a	total X-ray fluorescence analysis by house method	manual mixing	100 mg	Wet grinding process with ball mill	20% HNO ₃	internal standard / Gallium	no	
	7b	total X-ray fluorescence analysis by house method	manual mixing	100 mg	Wet grinding process with ball mill	20% HNO ₃	internal standard / Gallium	no	
	8	AA53, ICP-MS	ball mill	0,2g	AA30	HNO ₃ , H ₂ O ₂	NIST SRM 3280	yes	
	9	§ 64 LFGB - L 31.00-10, modifiziert	mortar	0,19697	microwave	HNO ₃	Merck 1-19778	yes	
	10	DIN EN ISO 11885	mixing	ca. 0,5 g	microwave	HNO ₃	external	yes	
	11								
	12	ASU L 00.00-144	yes	0,5	ASU L 00.00-19/1	HNO ₃	plant and animal RM	yes	
13	ICP-OES (MET-208)	mixing	< 1 g	pressure digestion, microwave	HNO ₃ -H ₂ O ₂	CRM	yes		

Analyte	Participant	Method description as in test report / norm / literature	Homogenization	Sample weight	Hydrolization Method	Hydrolization Solution	Calibration and reference material	Method accredited ISO/IEC 17025 yes / no	Further remarks
Cr – Chrom / Chromium	1	acid digestion	no	1g			CRM	yes	
	2	ICP-MS (EPA 6020)	full amount of the sample was homogenized before analysis	0.25 g	acid digestion, MSZ EN 13805:2015	5 ml nitric acid + 2 ml hydrogen peroxide	calibration solutions: CPAchem 7105L-0-B9-62, Sigma-Aldrich 49596-100ML; check solutions: Ultra Scientific IMS-102, High-Purity Standards ICP-AM-15-5M, Merck 1.19898.0500	yes	
	3	DIN EN 15763 : 2010 mod.	hand mortar	800 mg	DIN EN 13805 : 2014	HNO ₃ + H ₂ O ₂	external cali./ Cocoa	yes	
	4	DIN EN ISO 17294-2 (E 29) (2005-02)		approx. 0.5 g / 25 ml weighed exactly	Microwave pressure digestion	HNO ₃ , H ₂ O ₂ , H ₂ O		yes	
	5	EN 15763	no	0,2g	pressure digestion	HNO ₃	external calibration	yes	
	6	ICP-MS, DIN EN ISO 17294-2	shaking	0,4g	L155; microwave pressure digestion	HNO ₃ /H ₂ O	7-point calibration	yes	
	7a	total X-ray fluorescence analysis by house method	manual mixing	100 mg	Wet grinding process with ball mill	20% HNO ₃	internal standard / Gallium	no	
	7b	total X-ray fluorescence analysis by house method	manual mixing	100 mg	Wet grinding process with ball mill	20% HNO ₃	internal standard / Gallium	no	
	8	AA53, ICP-MS	ball mill	0,2g	AA30	HNO ₃ , H ₂ O ₂	NIST SRM 3280	yes	
	9								
	10	DIN EN ISO 11885	mixing	ca. 0,5 g	microwave	HNO ₃	external	yes	
	11								
	12	ASU L 00.00-144	yes	0,5	ASU L 00.00-19/1	HNO ₃	plant and animal RM	yes	
13	ICP-MS (MET-206)	mixing	< 1 g	pressure digestion, microwave	HNO ₃ -H ₂ O ₂	CRM	yes		

Analyte	Participant	Method description as in test report / norm / literature	Homogenization	Sample weight	Hydrolyzation Method	Hydrolyzation Solution	Calibration and reference material	Method accredited ISO/IEC 17025 yes / no	Further remarks
Cu – Kupfer / Copper	1	acid digestion	no	1g			CRM	yes	
	2	ICP-MS (EPA 6020)	full amount of the sample was homogenized before analysis	0.25 g	acid digestion, MSZ EN 13805:2015	5 ml nitric acid + 2 ml hydrogen peroxide	calibration solutions: CPAchem 7105L-0-B9-62, Sigma-Aldrich 49596-100ML; check solutions: Ultra Scientific IMS-102, High-Purity Standards ICP-AM-15-5M, Merck 1.19898.0500	yes	
	3	DIN EN 15763 : 2010 mod.	hand mortar	800 mg	DIN EN 13805 : 2014	HNO ₃ + H ₂ O ₂	external cali./ milk powder	yes	
	4	DIN EN ISO 11885 (E 22) (2009-09)		approx. 0.5 g / 25 ml weighed exactly	Microwave pressure digestion	HNO ₃ , H ₂ O ₂ , H ₂ O		yes	
	5	EN 15763	no	0,2g	pressure digestion	HNO ₃	external calibration	yes	
	6	ICP-MS, DIN EN ISO 17294-2	shaking	0,4g	L155; microwave pressure digestion	HNO ₃ /H ₂ O	7-point calibration	yes	
	7a	total X-ray fluorescence analysis by house method	manual mixing	100 mg	Wet grinding process with ball mill	20% HNO ₃	internal standard / Gallium	no	
	7b	total X-ray fluorescence analysis by house method	manual mixing	100 mg	Wet grinding process with ball mill	20% HNO ₃	internal standard / Gallium	no	
	8	AA53, ICP-MS	ball mill	0,2g	AA30	HNO ₃ , H ₂ O ₂	NIST SRM 3280	yes	
	9								
	10	DIN EN ISO 11885	mixing	ca. 0,5 g	microwave	HNO ₃	external	yes	
	11								
	12	ASU L 00.00-144	yes	0,5	ASU L 00.00-19/1	HNO ₃	plant and animal RM	yes	
	13a	ICP-OES (MET-209)	mixing	< 1 g	pressure digestion, microwave	HNO ₃ -H ₂ O ₂	CRM	yes	
13b	ICP-MS (MET-206)	mixing	< 1 g	pressure digestion, microwave	HNO ₃ -H ₂ O ₂	CRM	yes		

Analyte	Participant	Method description as in test report / norm / literature	Homogenization	Sample weight	Hydrolyzation Method	Hydrolyzation Solution	Calibration and reference material	Method accredited ISO/IEC 17025 yes / no	Further remarks
Fe – Eisen / Iron	1	acid digestion	no	1g			CRM	yes	
	2	ICP-MS (EPA 6020)	full amount of the sample was homogenized before analysis	0.25 g	acid digestion, MSZ EN 13805:2015	5 ml nitric acid + 2 ml hydrogen peroxide	calibration solutions: CPAchem 7105L-0-B9-62, Sigma-Aldrich 49596-100ML; check solutions: Ultra Scientific IMS-102, High-Purity Standards ICP-AM-15-5M, Merck 1.19898.0500	yes	
	3	DIN EN 15763 : 2010 mod.	hand mortar	800 mg	DIN EN 13805 : 2014	HNO ₃ + H ₂ O ₂	external cali./ milk powder	yes	
	4	DIN EN ISO 11885 (E 22) (2009-09)		approx. 0.5 g / 25 ml weighed exactly	Microwave pressure digestion	HNO ₃ , H ₂ O ₂ , H ₂ O		yes	
	5	EN 15763	no	0,2g	pressure digestion	HNO ₃ +HCl	external calibration	yes	
	6	ICP-OES; DIN EN ISO 11885-E22	shaking	0,4g	L155; microwave pressure digestion	HNO ₃ /H ₂ O	3-point calibration	yes	
	7a	total X-ray fluorescence analysis by house method	manual mixing	100 mg	Wet grinding process with ball mill	20% HNO ₃	internal standard / Gallium	no	
	7b	total X-ray fluorescence analysis by house method	manual mixing	100 mg	Wet grinding process with ball mill	20% HNO ₃	internal standard / Gallium	no	
	8	AA53, ICP-MS	ball mill	0,2g	AA30	HNO ₃ , H ₂ O ₂	NIST SRM 3280	yes	
	9	§ 64 LFGB - ASU L 00.00-19/2, modifiziert	mortar	0,19697	microwave	HNO ₃	Merck 1-70326	yes	
	10	DIN EN ISO 11885	mixing	ca. 0,5 g	microwave	HNO ₃	external	yes	
	11								
	12	ASU L 00.00-144	yes	0,5	ASU L 00.00-19/1	HNO ₃	plant and animal RM	yes	
13	ICP-OES (MET-209)	mixing	< 1 g	pressure digestion, microwave	HNO ₃ -H ₂ O ₂	CRM	yes		

Analyte	Participant	Method description as in test report / norm / literature	Homogenization	Sample weight	Hydrolyzation Method	Hydrolyzation Solution	Calibration and reference material	Method accredited ISO/IEC 17025 yes / no	Further remarks
K – Kalium / Potassium	1	acid digestion	no	1g			CRM	yes	
	2	ICP-MS (EPA 6020)	full amount of the sample was homogenized before analysis	0.25 g	acid digestion, MSZ EN 13805:2015	5 ml nitric acid + 2 ml hydrogen peroxide	calibration solutions: CPAchem 7105L-0-B9-62, Sigma-Aldrich 49596-100ML; check solutions: Ultra Scientific IMS-102, High-Purity Standards ICP-AM-15-5M, Merck 1.19898.0500	yes	
	3	ASU L 00.00-144 : 2019	hand mortar	800 mg	DIN EN 13805 : 2014	HNO ₃ + H ₂ O ₂	external cali./ milk powder	yes	
	4	DIN EN ISO 11885 (E 22) (2009-09)		approx. 0.5 g / 25 ml weighed exactly	Microwave pressure digestion	HNO ₃ , H ₂ O ₂ , H ₂ O		yes	
	5	EN 15763	no	0,2g	pressure digestion	HNO ₃	external calibration	yes	
	6	ICP-OES; DIN EN ISO 11885-E22	shaking	0,4g	L155; microwave pressure digestion	HNO ₃ /H ₂ O	3-point calibration	yes	
	7a	total X-ray fluorescence analysis by house method	manual mixing	100 mg	Wet grinding process with ball mill	20% HNO ₃	internal standard / Gallium	no	
	7b	total X-ray fluorescence analysis by house method	manual mixing	100 mg	Wet grinding process with ball mill	20% HNO ₃	internal standard / Gallium	no	
	8	AA53, ICP-MS	ball mill	0,2g	AA30	HNO ₃ , H ₂ O ₂	NIST SRM 3280	yes	
	9	§ 64 LFGB - L 31.00-10, modifiziert	mortar	0,19697	microwave	HNO ₃	Merck 1-70230	yes	
	10	DIN EN ISO 11885	mixing	ca. 0,5 g	microwave	HNO ₃	external	yes	
	11								
	12	ASU L 00.00-144	yes	0,5	ASU L 00.00-19/1	HNO ₃	plant and animal RM	yes	
13	ICP-OES (MET-208)	mixing	< 1 g	pressure digestion, microwave	HNO ₃ -H ₂ O ₂	CRM	yes		

Analyte	Participant	Method description as in test report / norm / literature	Homogenization	Sample weight	Hydrolyzation Method	Hydrolyzation Solution	Calibration and reference material	Method accredited ISO/IEC 17025 yes / no	Further remarks
Mg - Magnesium	1	acid digestion	no	1g			CRM	yes	
	2	ICP-MS (EPA 6020)	full amount of the sample was homogenized before analysis	0.25 g	acid digestion, MSZ EN 13805:2015	5 ml nitric acid + 2 ml hydrogen peroxide	calibration solutions: CPAchem 7105L-0-B9-62, Sigma-Aldrich 49596-100ML; check solutions: Ultra Scientific IMS-102, High-Purity Standards ICP-AM-15-5M, Merck 1.19898.0500	yes	
	3	ASU L 00.00-144 : 2019	hand mortar	800 mg	DIN EN 13805 : 2014	HNO ₃ + H ₂ O ₂	external cali./ milk powder	yes	
	4	DIN EN ISO 11885 (E 22) (2009-09)		approx. 0.5 g / 25 ml weighed exactly	Microwave pressure digestion	HNO ₃ , H ₂ O ₂ , H ₂ O		yes	
	5	EN 15763	no	0,2g	pressure digestion	HNO ₃	external calibration	yes	
	6	ICP-OES; DIN EN ISO 11885-E22	shaking	0,4g	L155; microwave pressure digestion	HNO ₃ /H ₂ O	3-point calibration	yes	
	7								
	8	AA53, ICP-MS	ball mill	0,2g	AA30	HNO ₃ , H ₂ O ₂	NIST SRM 3280	yes	
	9	§ 64 LFGB - L 31.00-10, modifiziert	mortar	0,19697	microwave	HNO ₃	Merck 1-19788	yes	
	10	DIN EN ISO 11885	mixing	ca. 0,5 g	microwave	HNO ₃	external	yes	
	11								
	12	ASU L 00.00-144	yes	0,5	ASU L 00.00-19/1	HNO ₃	plant and animal RM	yes	
	13	ICP-OES (MET-208)	mixing	< 1 g	pressure digestion, microwave	HNO ₃ -H ₂ O ₂	CRM	yes	

Analyte	Participant	Method description as in test report / norm / literature	Homogenization	Sample weight	Hydrolization Method	Hydrolization Solution	Calibration and reference material	Method accredited ISO/IEC 17025 yes / no	Further remarks
Mn – Mangan / Manganese	1	acid digestion	no	1g			CRM	yes	
	2	ICP-MS (EPA 6020)	full amount of the sample was homogenized before analysis	0.25 g	acid digestion, MSZ EN 13805:2015	5 ml nitric acid + 2 ml hydrogen peroxide	calibration solutions: CPAchem 7105L-0-B9-62, Sigma-Aldrich 49596-100ML; check solutions: Ultra Scientific IMS-102, High-Purity Standards ICP-AM-15-5M, Merck 1.19898.0500	yes	
	3	DIN EN 15763 : 2010 mod.	hand mortar	800 mg	DIN EN 13805 : 2014	HNO ₃ + H ₂ O ₂	external cali./ Cocoa	yes	
	4	DIN EN ISO 11885 (E 22) (2009-09)		approx. 0.5 g / 25 ml weighed exactly	Microwave pressure digestion	HNO ₃ , H ₂ O ₂ , H ₂ O		yes	
	5	EN 15763	no	0,2g	pressure digestion	HNO ₃	external calibration	yes	
	6	ICP-MS, DIN EN ISO 17294-2	shaking	0,4g	L155; microwave pressure digestion	HNO ₃ /H ₂ O	7-point calibration	yes	
	7a	total X-ray fluorescence analysis by house method	manual mixing	100 mg	Wet grinding process with ball mill	20% HNO ₃	internal standard / Gallium	no	
	7b	total X-ray fluorescence analysis by house method	manual mixing	100 mg	Wet grinding process with ball mill	20% HNO ₃	internal standard / Gallium	no	
	8	AA53, ICP-MS	ball mill	0,2g	AA30	HNO ₃ , H ₂ O ₂	NIST SRM 3280	yes	
	9								
	10	DIN EN ISO 11885	mixing	ca. 0,5 g	microwave	HNO ₃	external	yes	
	11								
	12	ASU L 00.00-144	yes	0,5	ASU L 00.00-19/1	HNO ₃	plant and animal RM	yes	
13	ICP-MS (MET-206)	mixing	< 1 g	pressure digestion, microwave	HNO ₃ -H ₂ O ₂	CRM	yes		

Analyte	Participant	Method description as in test report / norm / literature	Homogenization	Sample weight	Hydrolyzation Method	Hydrolyzation Solution	Calibration and reference material	Method accredited ISO/IEC 17025 yes / no	Further remarks
Mo – Molybdän / Molybdenu m	1	acid digestion	no	1g			CRM	yes	
	2	ICP-MS (EPA 6020)	full amount of the sample was homogenized before analysis	0.25 g	acid digestion, MSZ EN 13805:2015	5 ml nitric acid + 2 ml hydrogen peroxide	calibration solutions: CPAchem 7105L-0-B9-62, Sigma-Aldrich 49596-100ML; check solutions: Ultra Scientific IMS-102, High-Purity Standards ICP-AM-15-5M, Merck 1.19898.0500	yes	
	3	DIN EN 15763 : 2010 mod.	hand mortar	800 mg	DIN EN 13805 : 2014	HNO ₃ + H ₂ O ₂	external cali./ cocoa	yes	
	4	DIN EN ISO 17294-2 (E 29) (2005-02)		approx. 0.5 g / 25 ml weighed exactly	Microwave pressure digestion	HNO ₃ , H ₂ O ₂ , H ₂ O		yes	
	5	EN 15763	no	0,2g	pressure digestion	HNO ₃	external calibration	yes	
	6	ICP-MS, DIN EN ISO 17294-2	shaking	0,4g	L155; microwave pressure digestion	HNO ₃ /H ₂ O	7-point calibration	yes	
	7								
	8	AA53, ICP-MS	ball mill	0,2g	AA30	HNO ₃ , H ₂ O ₂	NIST SRM 3280	yes	
	9								
	10	DIN EN ISO 11885	mixing	ca. 0,5 g	microwave	HNO ₃	external	yes	
	11	QCL628	Mixing	0.8g	N/A	N/A	Spex Certiprep	Yes, but not this element yet	Only digestion performed
	12	ASU L 00.00-144	yes	0,5	ASU L 00.00-19/1	HNO ₃	plant and animal RM	yes	
	13	ICP-MS (MET-206)	mixing	< 1 g	pressure digestion, microwave	HNO ₃ -H ₂ O ₂	CRM	yes	

Analyte	Participant	Method description as in test report / norm / literature	Homogenization	Sample weight	Hydrolyzation Method	Hydrolyzation Solution	Calibration and reference material	Method accredited ISO/IEC 17025 yes / no	Further remarks
P – Phosphor / Phosphorus	1	acid digestion	no	1g			CRM	yes	
	2	ICP-MS (EPA 6020)	full amount of the sample was homogenized before analysis	0.25 g	acid digestion, MSZ EN 13805:2015	5 ml nitric acid + 2 ml hydrogen peroxide	calibration solutions: CPAchem 7105L-0-B9-62, Sigma-Aldrich 49596-100ML; check solutions: Ultra Scientific IMS-102, High-Purity Standards ICP-AM-15-5M, Merck 1.19898.0500	yes	
	3	ASU L 00.00-144 : 2019	hand mortar	800 mg	DIN EN 13805 : 2014	HNO ₃ + H ₂ O ₂	external cali./ milk powder	yes	
	4	DIN EN ISO 11885 (E 22) (2009-09)		approx. 0.5 g / 25 ml weighed exactly	Microwave pressure digestion	HNO ₃ , H ₂ O ₂ , H ₂ O		yes	
	5	EN 15763	no	0,2g	pressure digestion	HNO ₃	external calibration	yes	
	6	ICP-OES; DIN EN ISO 11885-E22	shaking	0,4g	L155; microwave pressure digestion	HNO ₃ /H ₂ O	3-point calibration	yes	
	7								
	8	AA53, ICP-MS	ball mill	0,2g	AA30	HNO ₃ , H ₂ O ₂	NIST SRM 3280	no	
	9								
	10	DIN EN ISO 11885	mixing	ca. 0,5 g	microwave	HNO ₃	external	yes	
	11								
	12	ASU L 00.00-144	yes	0,5	ASU L 00.00-19/1	HNO ₃	plant and animal RM	yes	
	13	ICP-OES (MET-208)	mixing	< 1 g	pressure digestion, microwave	HNO ₃ -H ₂ O ₂	CRM	yes	

Analyte	Participant	Method description as in test report / norm / literature	Homogenization	Sample weight	Hydrolyzation Method	Hydrolyzation Solution	Calibration and reference material	Method accredited ISO/IEC 17025 yes / no	Further remarks
Se – Selen / Selenium	1	acid digestion	no	1g			CRM	yes	
	2	ICP-MS (EPA 6020)	full amount of the sample was homogenized before analysis	0.25 g	acid digestion, MSZ EN 13805:2015	5 ml nitric acid + 2 ml hydrogen peroxide	calibration solutions: CPAchem 7105L-0-B9-62, Sigma-Aldrich 49596-100ML; check solutions: Ultra Scientific IMS-102, High-Purity Standards ICP-AM-15-5M, Merck 1.19898.0500	yes	
	3	DIN EN 15763 : 2010 mod.	hand mortar	800 mg	DIN EN 13805 : 2014	HNO ₃ + H ₂ O ₂	external cali./ milk powder	yes	
	4	DIN 38405-D 23 (1994-10)		approx. 0.5 g / 25 ml weighed exactly	Microwave pressure digestion	HNO ₃ , H ₂ O ₂ , H ₂ O		yes	
	5	EN 15763	no	0,2g	pressure digestion	HNO ₃	external calibration	yes	
	6	ICP-MS, DIN EN ISO 17294-2	shaking	0,4g	L155; microwave pressure digestion	HNO ₃ /H ₂ O	7-point calibration	yes	
	7a	total X-ray fluorescence analysis by house method	manual mixing	100 mg	Wet grinding process with ball mill	20% HNO ₃	internal standard / Gallium	no	
	7b	total X-ray fluorescence analysis by house method	manual mixing	100 mg	Wet grinding process with ball mill	20% HNO ₃	internal standard / Gallium	no	
	8	AA53, ICP-MS	ball mill	0,2g	AA30	HNO ₃ , H ₂ O ₂	NIST SRM 3280	yes	
	9								
	10	DIN EN ISO 11885	mixing	ca. 0,5 g	microwave	HNO ₃	external	yes	
	11	QCL628	Mixing	0.8g	N/A	N/A	Spex Certiprep	Yes, but not this element yet	Only digestion performed
	12	ASU L 00.00-144	yes	0,5	ASU L 00.00-19/1	HNO ₃	plant and animal RM	yes	
13	ICP-MS (MET-206)	mixing	< 1 g	pressure digestion, microwave	HNO ₃ -H ₂ O ₂	CRM	yes		

Analyte	Participant	Method description as in test report / norm / literature	Homogenization	Sample weight	Hydrolyzation Method	Hydrolyzation Solution	Calibration and reference material	Method accredited ISO/IEC 17025 yes / no	Further remarks
Zn – Zink / Zinc	1	acid digestion	no	1g			CRM	yes	
	2	ICP-MS (EPA 6020)	full amount of the sample was homogenized before analysis	0.25 g	acid digestion, MSZ EN 13805:2015	5 ml nitric acid + 2 ml hydrogen peroxide	calibration solutions: CPAchem 7105L-0-B9-62, Sigma-Aldrich 49596-100ML; check solutions: Ultra Scientific IMS-102, High-Purity Standards ICP-AM-15-5M, Merck 1.19898.0500	yes	
	3	DIN EN 15763 : 2010 mod.	hand mortar	800 mg	DIN EN 13805 : 2014	HNO ₃ + H ₂ O ₂	external cali./ milk powder	yes	
	4	DIN EN ISO 11885 (E 22) (2009-09)		approx. 0.5 g / 25 ml weighed exactly	Microwave pressure digestion	HNO ₃ , H ₂ O ₂ , H ₂ O		yes	
	5	EN 15763	no	0,2g	pressure digestion	HNO ₃	external calibration	yes	
	6	ICP-OES; DIN EN ISO 11885-E22	shaking	0,4g	L155; microwave pressure digestion	HNO ₃ /H ₂ O	3-point calibration	yes	
	7a	total X-ray fluorescence analysis by house method	manual mixing	100 mg	Wet grinding process with ball mill	20% HNO ₃	internal standard / Gallium	no	
	7b	total X-ray fluorescence analysis by house method	manual mixing	100 mg	Wet grinding process with ball mill	20% HNO ₃	internal standard / Gallium	no	
	8	AA53, ICP-MS	ball mill	0,2g	AA30	HNO ₃ , H ₂ O ₂	NIST SRM 3280	yes	
	9	§ 64 LFGB - ASU L 00.00-19/2, modifiziert	mortar	0,19697	microwave	HNO ₃	Merck 1-70369	yes	
	10	DIN EN ISO 11885	mixing	ca. 0,5 g	microwave	HNO ₃	external	yes	
	11								
	12	ASU L 00.00-144	yes	0,5	ASU L 00.00-19/1	HNO ₃	plant and animal RM	yes	
13	ICP-OES (MET-209)	mixing	< 1 g	pressure digestion, microwave	HNO ₃ -H ₂ O ₂	CRM	yes		

5.2 Homogeneity

5.2.1 Homogeneity of bottled PT-samples

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

Copper

Independant Samples	mg/kg
1	450
2	470
3	480
4	460
5	450
6	460
7	450
8	450

General Mean 459
 Repeatability standard deviation 11,3 2,45%

5.2.2 Trend line function of the participants results

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

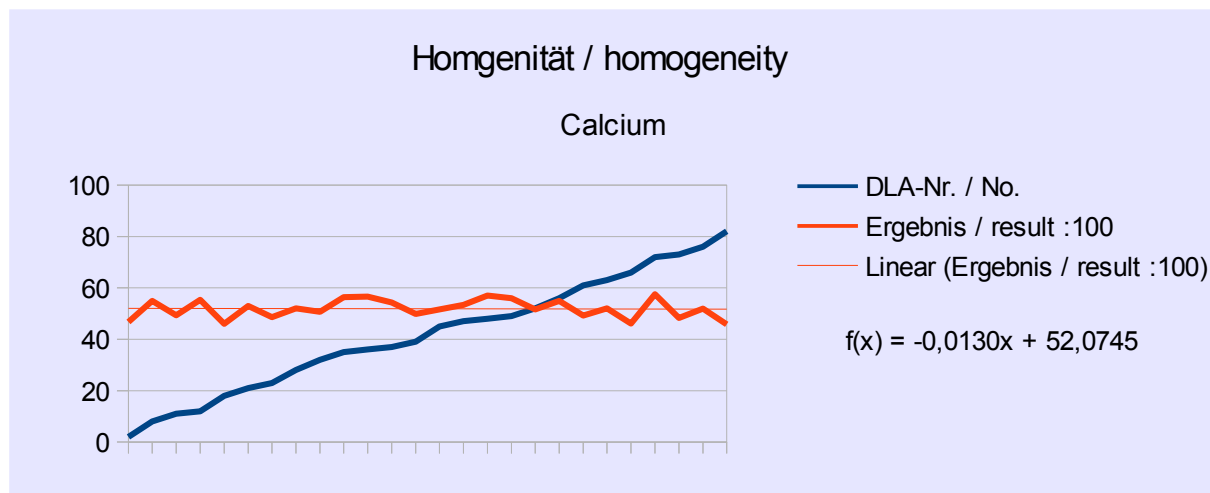


Abb./Fig. 25:

Trendfunktion Probennummern vs. Ergebnisse: Calcium (1/100 dargestellt)
 trend line function sample number vs. results: calcium (1/100 shown)

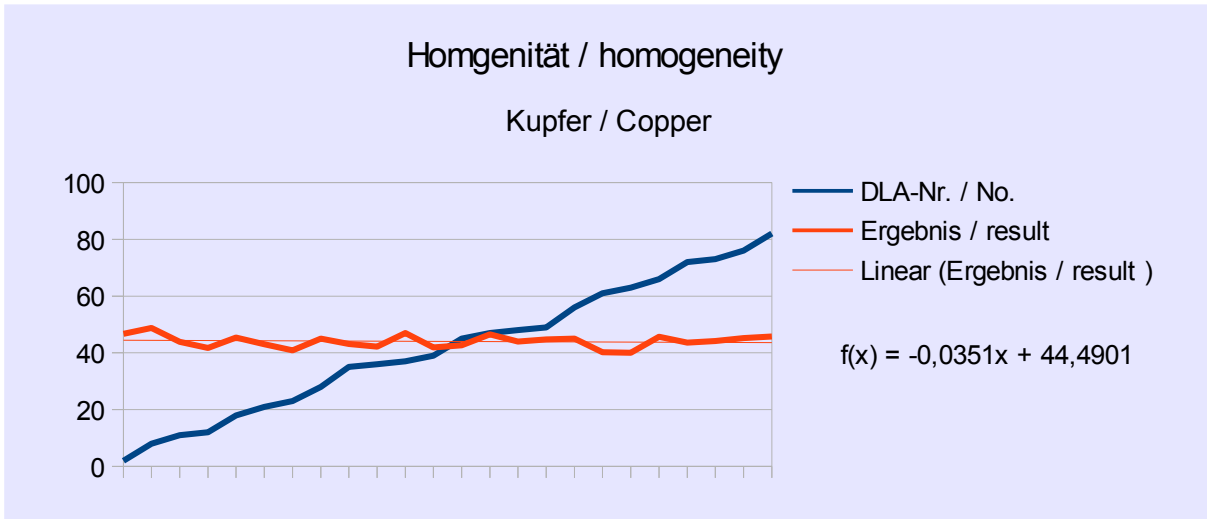


Abb./Fig. 26:
Trendfunktion Probennummern vs. Ergebnisse: Kupfer
trend line function sample number vs. results: copper

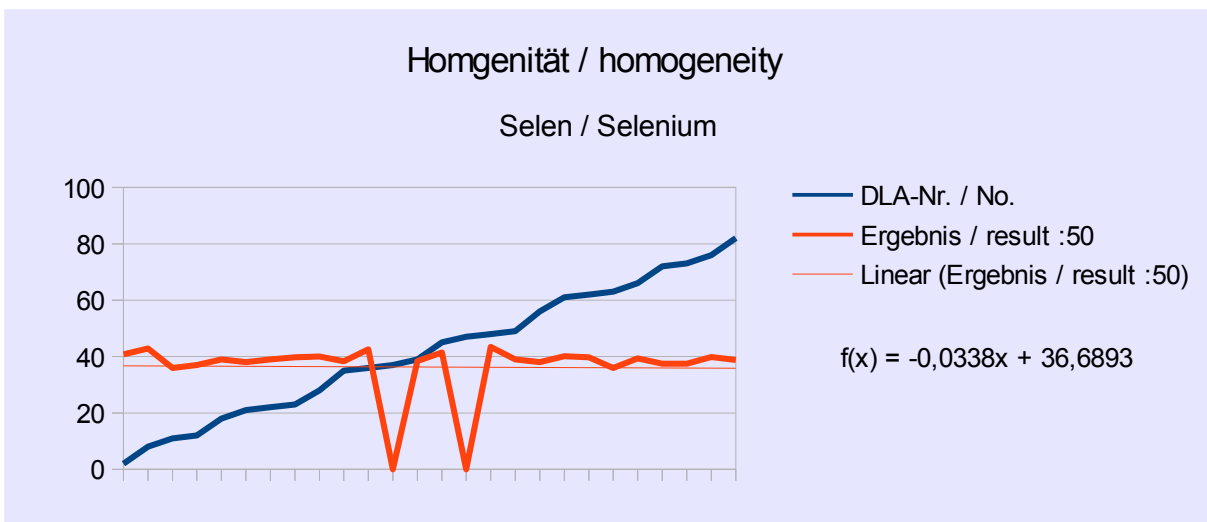


Abb./Fig. 27:
Trendfunktion Probennummern vs. Ergebnisse: Selen (1/50 dargestellt)
trend line function sample number vs. results: selenium (1/50 shown)

5.3 Kernel Density Plots of Results

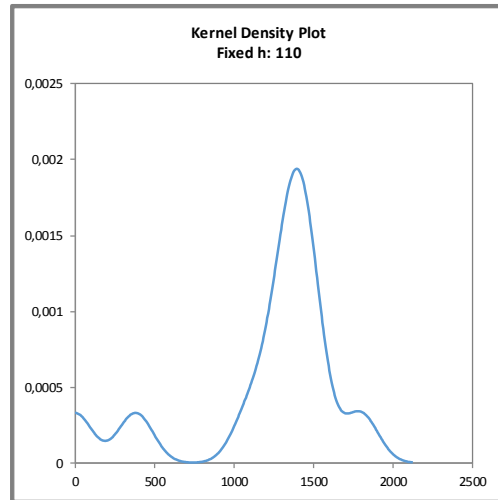
Abbildungen:

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

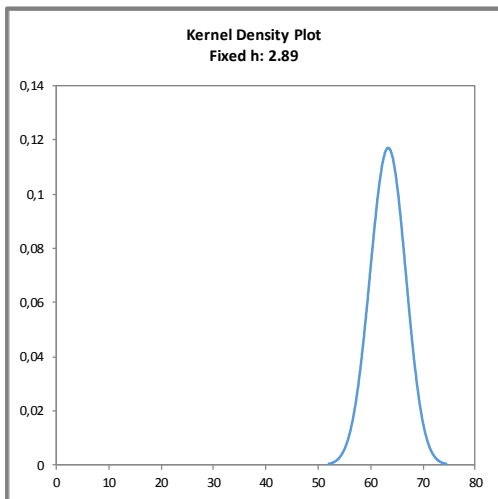
Figures:

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

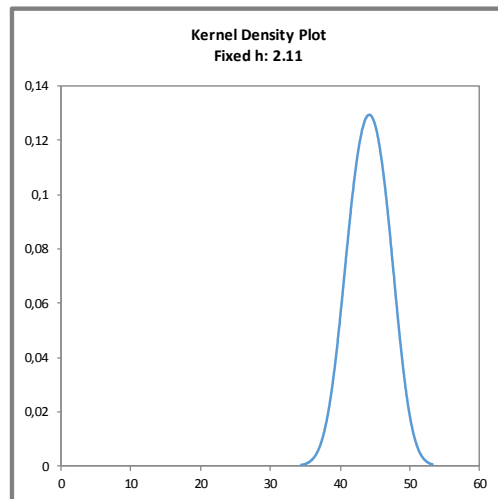
Cr - Chrom / Chromium



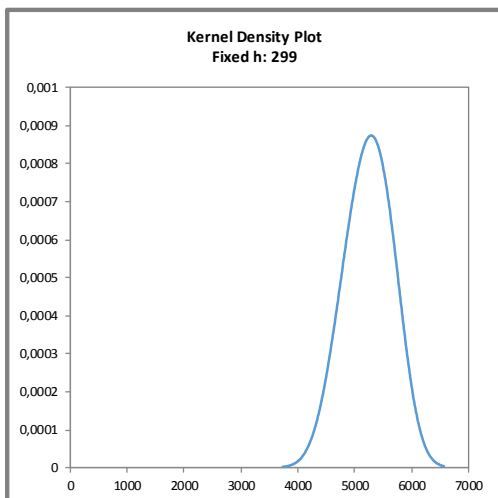
B - Bor / Boron
(Darstellung ohne Ausreißer bei 591 mg/100g)



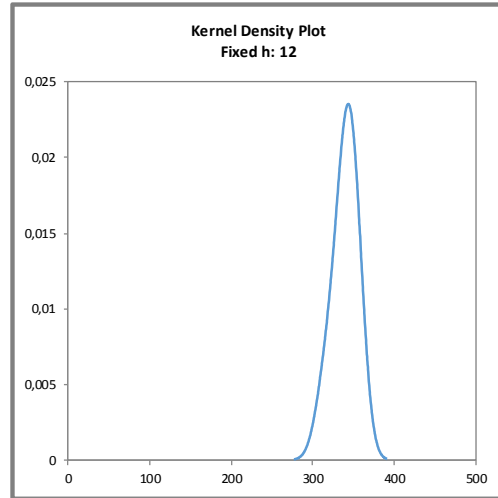
Cu - Kupfer / Copper



Ca - Calcium



Fe - Eisen / Iron



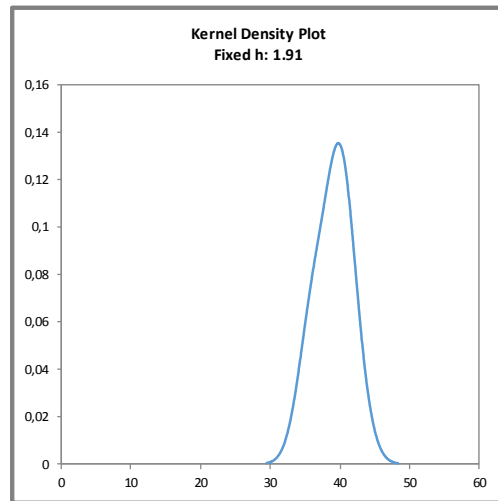
Abbildungen:

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

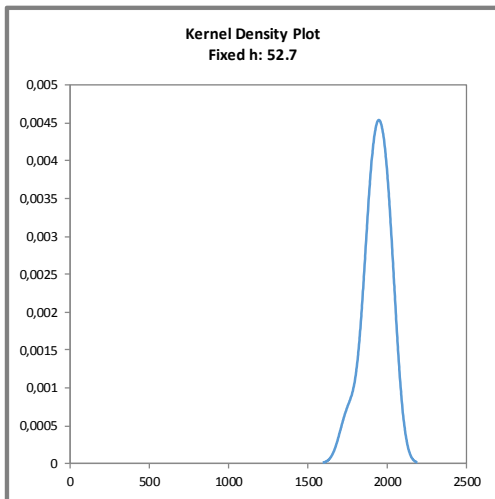
Figures:

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

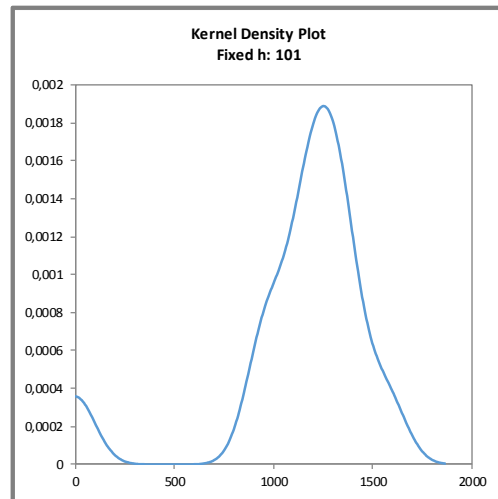
Mn – Mangan / Manganese



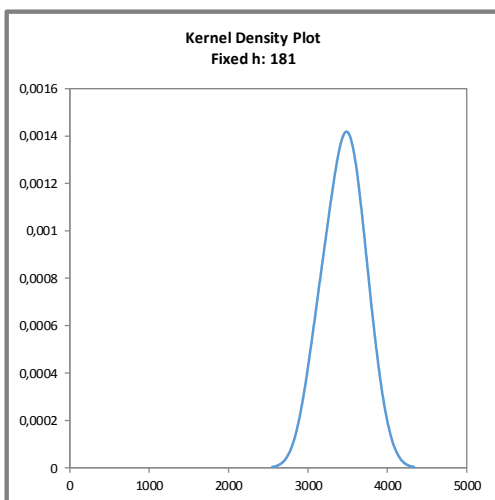
K Kalium / Potassium



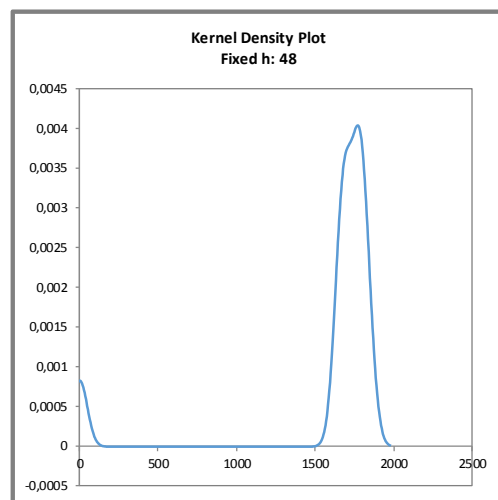
Mo – Molybdän / Molybdenum



Mg – Magnesium



P – Phosphor / Phosphorus



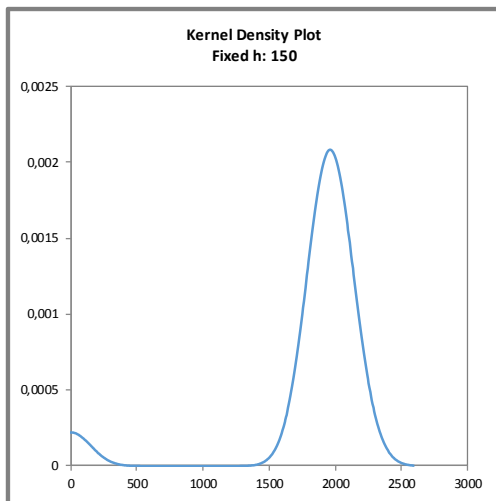
Abbildungen:

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

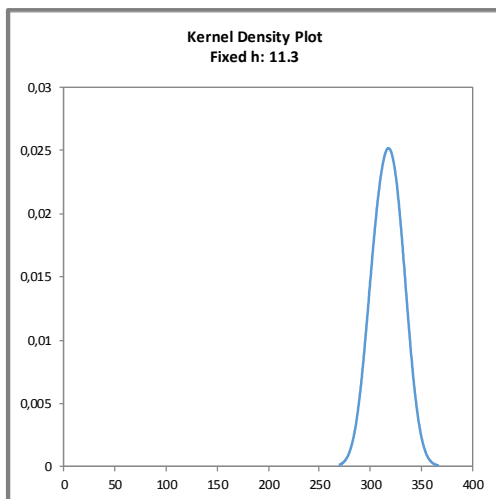
Figures:

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

Se - Selen / Selenium



Zn - Zink / Zinc



5.4 Information on the Proficiency Test (PT)

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

<i>PT number</i>	DLA 47-2019
<i>PT name</i>	Food Supplement II: B, Ca, Cr, Cu, Fe, K, Mg, Mn, Mo, P, Se, Zn
<i>Sample matrix*</i>	Samples I + II: Multi mineral and vitamin tablets and capsule powder (without capsule shell) / ingredients: maltodextrin, mineral and vitamin compounds as well as technological food additives
<i>Number of samples and sample amount</i>	2 identical samples I + II, 10 g each.
<i>Storage</i>	Samples I + II: room temperature
<i>Intentional use</i>	Laboratory use only (quality control samples)
<i>Parameter</i>	quantitative: B, Ca, Cr, Cu, Fe, K, Mg, Mn, Mo, P, Se and Zn
<i>Methods of analysis</i>	Analytical methods are optional
<i>Notes to analysis</i>	The analysis of PT samples should be performed like a routine laboratory analysis. In general we recommend to homogenize a representative sample amount before analysis according to good laboratory practice, especially in case of low sample weights.
<i>Result sheet</i>	The results for sample I and II as well as the final results calculated as mean of the double determination (samples I and II) should be filled in the result submission file. The recovery rates, if carried out, has to be included in the calculation.
<i>Units</i>	mg/100g and µg/100g
<i>Number of significant digits</i>	at least 2
<i>Further information</i>	For information please specify: <ul style="list-style-type: none"> - Date of analysis - DLA-sample-numbers (for sample I and II) - Limit of detection - Assignment incl. Recovery - Recovery with the same matrix - Method is accredited
<i>Result submission</i>	The result submission file should be sent by e-mail to: pt@dla-lvu.de
<i>Deadline</i>	the latest 20th September 2019
<i>Evaluation report</i>	The evaluation report is expected to be completed 6 weeks after deadline of result submission and sent as PDF file by e-mail.
<i>Coordinator and contact person of PT</i>	Matthias Besler-Scharf PhD

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

6. Index of participant laboratories in alphabetical order

Teilnehmer / Participant	Ort / Town	Land / Country
		CZECH REPUBLIC
		AUSTRIA
		Germany
		HUNGARY
		Germany
		Germany
		BELGIUM
		Germany
		USA
		Germany
		Germany
		Germany
		Germany
		Germany

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

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

7. Index of references

1. DIN EN ISO/IEC 17025:2005; Allgemeine Anforderungen an die Kompetenz von Prüf- und Kalibrierlaboratorien / General requirements for the competence of testing and calibration laboratories
2. DIN EN ISO/IEC 17043:2010; Konformitätsbewertung – Allgemeine Anforderungen an Eignungsprüfungen / Conformity assessment – General requirements for proficiency testing
3. ISO 13528:2015 & DIN ISO 13528:2009; Statistische Verfahren für Eignungsprüfungen durch Ringversuche / Statistical methods for use in proficiency testing by inter-laboratory 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 amtliche Kontrollen zur Überprüfung der Einhaltung des Lebensmittel- und Futtermittelrechts sowie der Bestimmungen über Tiergesundheit und Tierschutz / Regulation on official controls performed to ensure the verification of compliance with feed and food law, animal health and animal welfare rules
6. Evaluation of analytical methods used for regulation of food and drugs; W. Horwitz; Analytical Chemistry, 54, 67-76 (1982)
7. The International Harmonised Protocol for the Proficiency Testing of Analytical Laboratories ; J.AOAC Int., 76(4), 926 – 940 (1993)
8. A Horwitz-like funktion describes precision in proficiency test; M. Thompson, P.J. Lowthian; Analyst, 120, 271-272 (1995)
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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)
12. AMC Kernel Density – Representing data distributions with kernel density estimates, amc technical brief, Editor M Thompson, Analytical Methods Committee, AMCTB No 4, Revised March 2006 and Excel Add-in Kernel.xla 1.0e by Royal Society of Chemistry
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. Homogeneity and stability of reference materials; Linsinger et al.; Accred Qual Assur, 6, 20-25 (2001)
17. AOAC Official Methods of Analysis: Guidelines for Standard Method Performance Requirements, Appendix F, p. 2, AOAC Int (2016)
18. ASU §64 L 00.00-157 (2016-2): Bestimmung von Aluminium in Lebensmitteln mit der Massenspektrometrie mit induktiv gekoppeltem Plasma (ICP-MS) [Determination of aluminium in foods by inductively coupled plasma mass spectrometry (ICPMS) after pressure digestion]
19. ASU §64 L 00.00-158 (2016-2): Bestimmung von Aluminium in Lebensmitteln mit der optischen Emissionsspektrometrie mit induktiv gekoppeltem Plasma (ICP-OES) [Determination of aluminium in foods by inductively coupled plasma emission spectrometry (ICP-OES) after pressure digestion]
20. ASU §64 L 00.00-135 (2011-01) / DIN EN 15763:2010: Bestimmung von Arsen, Cadmium, Quecksilber und Blei in Lebensmitteln mit ICP-MS nach Druckaufschluss / Foodstuffs. Determination of trace elements. Determination of arsenic, cadmium, mercury and lead in foodstuffs by inductively coupled plasma mass spectrometry (ICP-MS) after pressure digestion
21. ASU §64 L 00.00-19/2: Bestimmung von Eisen, Kupfer, Mangan und Zink mit der Atomabsorptionsspektrometrie (AAS) in der Flamme [Determination of iron, copper, manganese and zinc by atomic absorption spectrometry (AAS) in the flame]

22. ASU §64 L 00.00-19/3 / DIN EN 14083: Bestimmung von Blei, Cadmium, Chrom und 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
23. ASU §64 L 00.00-19/5: Bestimmung von Selen mit der Atomabsorptionsspektrometrie (AAS) -Hydridtechnik [Determination of selenium by atomic absorption spectrometry (AAS) - hydride technique]
24. ASU §64 L 00.00-144 : Bestimmung der Mineralstoffe Ca, K, Mg, Na, P und S sowie der Spurenelemente Fe, Cu, Mn und Zn in Lebensmitteln mit ICP-OES [Determination of minerals Ca, K, Mg, Na, P and S and trace elements Fe, Cu, Mn and Zn in foods by ICP-OES]
25. ASU §64 L 00.00-93 / DIN EN 15111: Bestimmung von Iod in Lebensmitteln - ICP-MS-Verfahren / Foodstuffs. Determination of trace elements. Determination of iodine by ICP-MS (inductively coupled plasma mass spectrometry)
26. ASU §64 L 00.00-127 / EN 15764: Bestimmung von Zinn in Lebensmitteln mit der Flammen- und Graphitrohr-Atomabsorptionsspektrometrie (GFAAS) nach Druckaufschluss / Foodstuffs. Determination of trace elements. Determination of tin by flame and graphite furnace atomic absorption spectrometry (FAAS and GFAAS) after pressure digestion
27. ASU §64 L 00.00-128 / DIN EN 15765: Bestimmung Zinn in Lebensmitteln mit der Massenspektrometrie mit induktiv gekoppeltem Plasma (ICP-MS) nach Druckaufschluss / Foodstuffs. Determination of trace elements. Determination of tin by inductively coupled plasma mass spectrometry (ICPMS) after pressure digestion
28. ASU §64 L 31.00-10: Bestimmung der Gehalte an Natrium, Kalium, Calcium und Magnesium in Frucht- und Gemüsesäften - Atomabsorptionsspektrometrisches Verfahren (AAS) [Determination of sodium, potassium, calcium and magnesium in fruit and vegetable juices - atomic absorption spectrometry (AAS)]