Evaluation Report
proficiency test

DLA 46/2019

Food Supplements I:

Vitamins B1, B2, B6, B12, Biotin, Vitamin C, Folic Acid, Niacin and Pantothenic Acid

in Multivitamin-Powder

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

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

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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 multivitamin-powder with added maltodextrin for manufacture of common in commerce food supplements from a European supplier.
The raw materials were sieved, mixed and homogenized.

Afterwards the samples were portioned to approximately 50 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 specification.

Table 1: Composition of DLA-Samples

Multivitamin-Powder
Ingredients: Ascorbic acid, nicotinamide, calcium D-pantothenate, riboflavin, pyridoxine hydrochloride, thiamine mononitrate, folic acid, biotin (carrier: mannitol) and cyanocobalamin (carrier: mannitol) (as well as retinyl acetate and cholecalciferol). Further Ingredient: 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 vitamins according to the manufacturers specification

Vitamin	Content per 100 g	
Vitamin B1	590	mg
Vitamin B2	780	mg
Vitamin B6	910	mg
Vitamin B12	870	µg
Biotin	62000	µg
Vitamin C	33000	mg
Folic acid	110000	µg
Niacin	8500	mg
Pantothenic acid	2500	mg

2.1.1 Homogeneity

The **mixture homogeneity before bottling** was examined 10-fold by determination of the paramaters Niacinamide, Pantothenic acid, Vitamin B1, B2, and B6 by HPLC-DAD. The repeatability standard deviations were with 1,08 - 2,34% in the range of repeatability standard deviations of the standardized methods (e.g. ASU-Methods, s. 3.6.2) (see Table 4) [18-26]. 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 except biotin the repeatability standard deviation was < 6% (see Table 3). Thus they were similar to corresponding repeatability standard deviations of precision data of the standardized methods (e.g. ASU-Methods, s. 3.6.2) (see Table 4) [16-25].
The repeatability standard deviations of the participants' results are given in the documentation in the statistic data (see 4.1 to 4.9).

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

Parameter	**CV_r**
Vitamin B1	2,62 %
Vitamin B2	2,43 %
Vitamin B6	1,92 %
Vitamin B12	4,82 %
Biotin	5,39 %
Vitamin C	2,41 %
Folic acid	2,47 %
Niacin	3,05 %
Pantothenic acid	5,15 %

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

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

The experience with various DLA test materials showed good storage stability with respect to the durability of the sample (spoilage) and the content of the PT parameters for comparable food matrices and water activity (a_W value <0,5).
The a_W value of the EP samples was approx. 0,18 (20,0°C). The stability of the sample material was thus ensured during the investigation period under the specified storage conditions.

2.2 Sample shipment and information to the test

Two portions of test material were sent to every participating laboratory in the 19th week of 2019. The testing method was optional. The tests should be finished at 21st June 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 above mentioned parameters in the matrix of multi-vitamin capsule powder (without capsule shells). The analysis method is optional. The results of the vitamins should be given as the sum of the equivalents in the form of the vitamin compound indicated in the result submission file.

Please note the attached information on the proficiency test.
(see documentation, section 5.4 Information on the PT)

2.3 Submission of results

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

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

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

All 25 participants submitted their results in time.

3. Evaluation

3.1 Consensus value from participants (assigned value)

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

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

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

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

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

3.2 Robust standard deviation

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

3.3 Repeatability standard deviation

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

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

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

3.4 Reproducibility standard deviation

The reproducibility standard deviation S_R represents a inter-laboratory estimate of the standard deviation for the determination of each parameter on the bases of (outlier free) individual participant results. It takes into account both the repeatability standard deviation S_r and the within-laboratory standard deviation S_S. Reproducibility standard deviations of PT´s may differ from reproducibility standard deviations of ring trials, because the participating laboratories of a PT generally use different internal conditions and methods for determining the measured values.
In the present evaluation, the specification of the reproducibility standard deviation, therefore, does not refer to a specific method, but characterizes approximately the comparability of results between the laboratories, assumed the effect of homogeneity and stability of the sample are negligible.

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

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

3.5 Exclusion of results and outliers

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

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

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

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

3.6 Target standard deviation (for proficiency assessment)

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

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

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 paramaters in the present PT the target standard deviation according to the general model of Horwitz was applied (see 3.6.1): Folic Acid, Niacin, Pantothenic Acid and Vitamin B12.

The target standard deviation of the evaluation by precision experiment (s. 3.6.2) was considered for the following parameters: (ASU §64 / EN--Norms) [18, 19, 21, 22, 25]: Biotin, Vitamin B1, B2, B6 and Vitamin C.

Additionally for Pantothenic Acid the standard uncertainty was considered by evaluation using z'-scores (see 3.6.8).

3.6.1 General model (Horwitz)

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

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

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

3.6.2 Value by precision experiment

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

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

The relative repeatability standard deviations (RSD_r) and relative reproducibility standard deviation (RSD_R) given in Table 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} [18-26]

Parameter	**Matrix**	**Mean**	**RSD_r**	**RSD_R**	**σpt**	**Method / Literature**
Biotin	Cereals-Powder	197 µg/100 g	4,5%	17,4%	17,1%[1]	HPLC [24] EN 15607
	Infant-Milk powder	18,0 µg/100 g	11,6%	29,8%	27,5%	HPLC [24] EN 15607
	Animal feed	15-58 µg/100g	7,2-9,4%	9,4-22,4%*	-	HPLC-MS/MS [26]
Vitamin C	Breakfast cereals	102,6 mg/100g	9,9%	19,3%	18,0%	HPLC [23] EN 14130
	Milk powder	100,3 mg/100 g	6,3%	11,4%	10,5%[1]	HPLC [23] EN 14130
Niacin	Breakfast cereals (Choco)	21,03 mg/100g	1,1%	4,3%	4,23%	HPLC [25] EN 15652
	Milk powder	16,66 mg/100 g	2,8%	4,3%	3,82%[1]	HPLC [25] EN 15652
	Wheat flour	0,72 mg/100 g	3,9%	29,2%	29,1%	HPLC [25] EN 15652
Vitamin B1	Food supplement	486 mg/100g	8,0 %	15,4%	14,3%[1]	HPLC [18] ASU L00.00-83
	Chocolate powder	1,55 mg/100g	8,0%	18,1%	17,2%	HPLC [18] ASU L00.00-83
Vitamin B2	Food supplement	87,1 mg/100g	3,9%	6,8%	6,2%[1]	HPLC [19] ASU L00.00-84
	Chocolate powder	1,26 mg/100g	3,7%	10,3%	9,7%	HPLC [19] ASU L00.00-84
Vitamin B6	Baby food	0,106 mg/100g	3,8%	6,6%	6,3%[1]	HPLC [21] ASU L00.00-130
	Baby food	0,101 mg/100g	4,0%	5,9%	5,2%[1]	HPLC [21] ASU L00.00-130
Folic acid	Milk powder	-	-	-	15,9	microbiological [22] ASU L00.00-87

[1] *used for evaluation or given for information (s. chapter 4), for Vitamin B6 as a mean value*

3.6.3 Value by perception

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

For the present evaluation the target standard deviation according to 3.6.1 was regarded suitable.

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

3.7 z-Score

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

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

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

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

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

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 (SD = standard deviation, CV = coefficient of variation)

Parameter	Matrix (Powder)	robust Mean	rob. SD (S*)	rel. SD (CV_{S*}) [%]	Quotient S*/σpt	DLA-report
Vitamin B1	Multivitamin-drink powder	3,11 mg/100g	0,606 mg/100g	19,5%	1,7*	DLA 32/2015
Vitamin B1	Multivitamin-capsule powder	1290 mg/100g	205 mg/100g	15,8%	1,1	DLA 43/2017
Vitamin B1	Multivitamin-capsule powder	690 mg/100g	98,1 mg/100g	14,2%	1,0	DLA 46/2019
Vitamin B2	Multivitamin-drink powder	2,89 mg/100g	0,890 mg/100g	30,8%	2,0*	DLA 32/2015
Vitamin B2	Multivitamin-capsule powder	1320 mg/100g	111 mg/100g	8,41%	1,4	DLA 43/2017
Vitamin B2	Multivitamin-capsule powder	783 mg/100g	58,3 mg/100g	7,45%	1,2	DLA 46/2019
Vitamin B6	Multivitamin-drink powder	3,86 mg/100g	0,329 mg/100g	8,5%	0,93	DLA 32/2015
Vitamin B6	Multivitamin-capsule powder	377 mg/100g	36,9 mg/100g	9,78%	1,4	DLA 43/2017
Vitamin B6	Multivitamin-capsule powder	749 mg/100g	58,4 mg/100g	7,80%	1,3	DLA 46/2019
Vitamin B12	Multivitamin-drink powder	7,90 µg/100g	2,66 µg/100g	33,7%	1,4	DLA 32/2015
Vitamin B12	Multivitamin-capsule powder	2380 µg/100g	597 µg/100g	25,1%	2,0*	DLA 43/2017
Vitamin B12	Multivitamin-capsule powder	1018 µg/100g	102 µg/100g	10,0%	0,89	DLA 46/2019
Biotin	Multivitamin-drink powder	11200 µg/100g	1190 µg/100g	10,6%	1,4	DLA 48/2016
Biotin	Multivitamin-capsule powder	15000 µg/100g	1840 µg/100g	12,3%	1,6	DLA 43/2017
Biotin	Multivitamin-capsule powder	67368 µg/100g	9709 µg/100g	14,4%	0,84	DLA 46/2019

Continuation next page

Continuation Table 5:

Parameter	Matrix (Powder)	robust Mean	rob. SD (S*)	rel. SD (CV_{S*}) [%]	Quotient S*/σpt	DLA-report
Folic acid	Multivitam-in-drink powder	710 µg/100g	148 µg/100g	20,8%	1,8	DLA 32/2015
Folic acid	Multivitam-in-capsule powder	226000 µg/100g	39900 µg/100g	17,6%	1,1	DLA 43/2017
Folic acid	Multivitam-in-capsule powder	88412 µg/100g	9691 µg/100g	11,0%	1,9	DLA 46/2019
Niacin	Multivitam-in-capsule powder	1530 mg/100g	107 mg/100g	6,98%	1,9	DLA 48/2016
Niacin	Multivitam-in-capsule powder	14400 mg/100g	1150 mg/100g	7,98&	1,9	DLA 43/2017
Niacin	Multivitam-in-capsule powder	8062 mg/100g	324 mg/100g	4,02%	1,4	DLA 46/2019
Pantothenic acid	Multivitam-in-capsule powder	598 mg/100g	41,1 mg/100g	6,88%	1,6	DLA 48/2016
Pantothenic acid	Multivitam-in-capsule powder	7100 mg/100g	1040 mg/100g	14,6%	2,9*	DLA 43/2017
Pantothenic acid	Multivitam-in-capsule powder	2667 mg/100g	196 mg/100g	7,35%	1,8*	DLA 46/2019
Vitamin C	Multivitam-in-capsule powder	6133 mg/100g	365 mg/100g	5,96%	1,4	DLA 48/2016
Vitamin C	Multivitam-in-capsule powder	21200 mg/100g	839 mg/100g	3,96%	1,6	DLA 43/2017
Vitamin C	Multivitam-in-capsule powder	34257 mg/100g	2644 mg/100g	7,72%	0,74	DLA 46/2019

** with target standard deviation σ_{pt}'*

3.8 z'-Score

The z'-score can be used for the valuation of the results of the participants, in cases the standard uncertainty has to be considered (s. 3.11). The z'-score represents the relation of the deviation of the result (x) of the participant from the respective consensus value (X) to the square root of quadrat sum of the target standard deviation (σ_{pt}) and the standard uncertainty (Ux_{pt}) [3].

The calculation is performed by:

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

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

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

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

For warning and action signals see 3.7.1.

3.9 Reproducibility cofficient of variation (CV_R)

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

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

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

3.10 Quotient S*/σ_{pt}

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

3.11 Standard uncertainty of the assigned value

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

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

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

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

4. Results

Comments to the distribution of the results:

The kernel density plots showed for all parameters nearly a symmetrical distribution of results (figures see documentation 5.3). Partly slight shoulders and separated smaller peaks can be seen, which are due to individual results and outliers. On the basis of the kernel density plots single results were excluded before statistic evaluation.

Comments to the statistic data:

The target standard deviation was calculated according to the general model of Horwitz or by data from precision experiments (ASU §64 methods / EN-methods). The evaluation according to the general model of Horwitz was preferred as long as the quotient S^*/σ_{pt} was in the range of ≤ 2,0. For all other parameters the target standard deviation from data by precision experiments was used, if available.

For pantothenic acid the distribution of results showed an increased variability with a quotient above 2,0. The parameter was evaluated by z´-scores considering the standard uncertainty. Then the quotient S^*/σ_{pt}' was 1,8 (s. Tab. 4).

For all other parameters the distribution showed a normal variability of results. The quotients S^*/σ_{pt} were in the range of 0,74 to 1,9 (s. Tab. 4).

The robust standard deviations and the repeatability and reproducibility standard deviation were in the range of of estbalised values for the used determination methods (s. 3.6.2).

The comparability of results is given.

75% to 94% of results were in the respective target range.

The robust means of the participant results were for all parameters in the range of 82% to 116% of the vitamin contents according to the manufacturer specifications (s. Tab. 2).

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

In the first table the characteristics are listed:

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

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

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

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

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

4.1 Vitamin B1 (as Thiamine-Cation in mg/100g)

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	21
Number of outliers	-
Mean	715
Median	667
Robust Mean (X)	**690**
Robust standard deviation (S*)	**98,1**
Number with 2 replicates	18
Repeatability SD (S_r)	17,8
Repeatability (CV_r)	2,62%
Reproducibility SD (S_R)	85,7
Reproducibility (CV_R)	12,6%
Target range:	
Target standard deviation σ_{pt}	**98,8**
Target standard deviation (for Information)	29,2
lower limit of target range	**492**
upper limit of target range	**888**
Quotient S/σ_{pt}*	*1,0*
Standard uncertainty $U_{(Xpt)}$	*26,8*
Results in the target range	*19*
Percent in the target range	*90%*

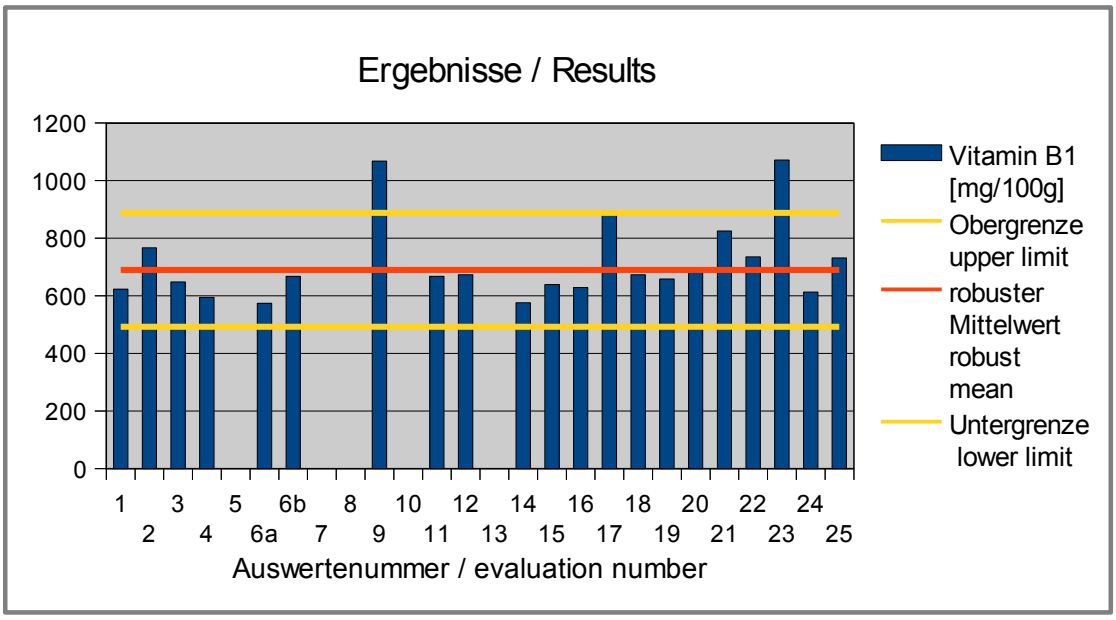


Abb. / Fig. 1: Ergebnisse Vitamin B1 / Results Vitamin B1

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Vitamin B1 [mg/100g]	Abweichung [mg/100g] Deviation [mg/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	623	-67,0	-0,68	-2,3	
2	767 *	76,5	0,77	2,6	
3	648	-42,0	-0,42	-1,4	
4	594	-96,0	-1,0	-3,3	
5					
6a	574	-116,0	-1,2	-4,0	
6b	667	-23,0	-0,23	-0,79	
7					
8					
9	1068 *	377,5	3,8	13	
10					
11	667	-22,7	-0,23	-0,78	
12	674	-16,5	-0,17	-0,56	
13					
14	576 *	-114,5	-1,16	-3,9	
15	639	-51,0	-0,52	-1,7	
16	628 *	-61,7	-0,62	-2,1	
17	892	201,5	2,0	6,9	
18	673	-17,0	-0,17	-0,58	
19	659	-31,5	-0,32	-1,1	
20	687	-3,0	-0,03	-0,10	
21	825	134,7	1,4	4,6	
22	735	45,0	0,46	1,5	
23	1071	381,0	3,9	13	
24	613	-77,5	-0,78	-2,7	
25	731	41,0	0,42	1,4	

* Mean calculated by DLA

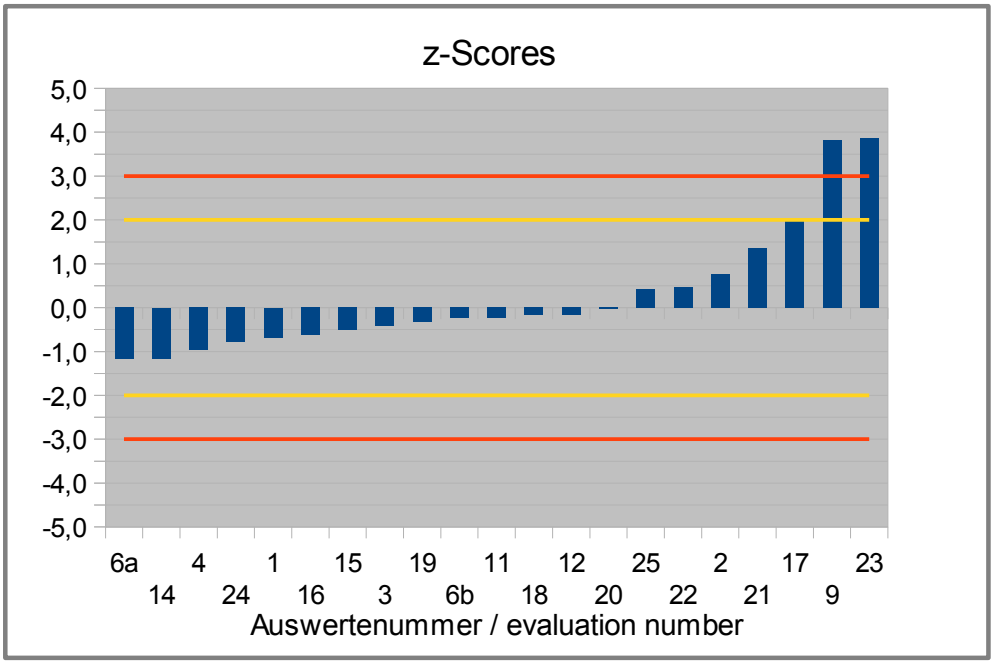


Abb. / Fig. 2: z-Scores Vitamin B1

4.2 Vitamin B2 (as Riboflavin in mg/100g)

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results°	20
Number of outliers	1
Mean	803
Median	780
Robust Mean (X)	**783**
Robust standard deviation (S*)	**58,3**
Number with 2 replicates	18
Repeatability SD (S_r)	18,8
Repeatability (CV_r)	2,43%
Reproducibility SD (S_R)	54,5
Reproducibility (CV_R)	7,04%
Target range:	
Target standard deviation σ_{pt}	**48,7**
Target standard deviation (for Information)	32,5
lower limit of target range	**686**
upper limit of target range	**880**
Quotient S/σ_{pt}*	*1,2*
Standard uncertainty $U_{(Xpt)}$	*16,3*
Results in the target range	*16*
Percent in the target range	*80%*

° *without outliers (result no. 15)*

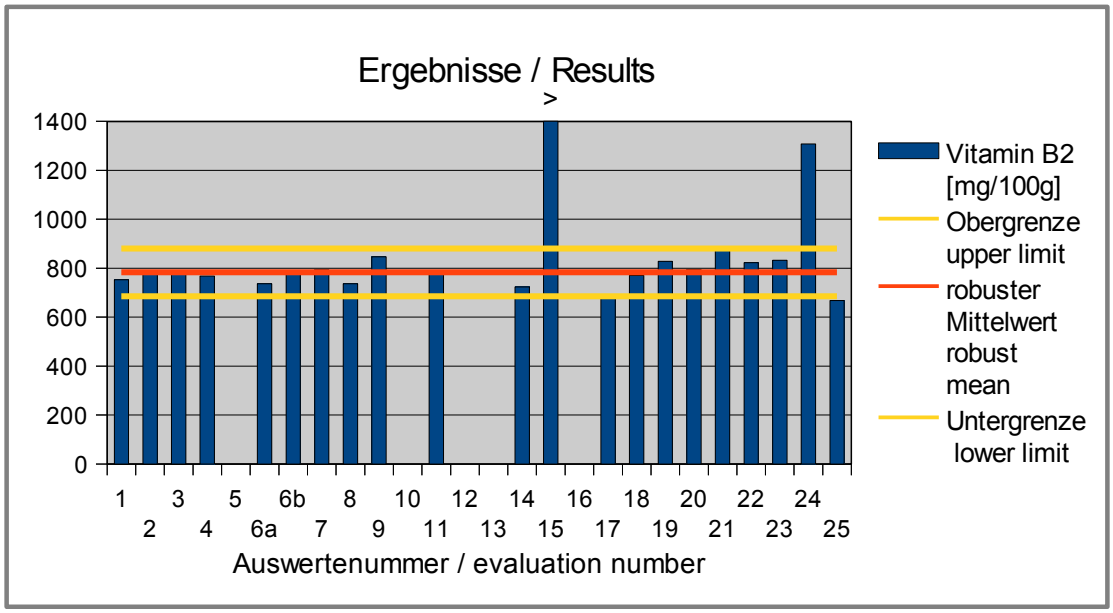


Abb. / Fig. 3: Ergebnisse Vitamin B2 / Results Vitamin B2

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Vitamin B2 [mg/100g]	Abweichung [mg/100g] Deviation [mg/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	753	-29,9	-0,62	-0,92	
2	777 *	-6,4	-0,13	-0,20	
3	775	-7,9	-0,16	-0,24	
4	768	-14,9	-0,31	-0,46	
5					
6a	736	-46,9	-1,0	-1,4	
6b	789	6,1	0,12	0,19	
7	793	10,3	0,21	0,32	
8	737 *	-45,9	-0,94	-1,4	
9	846	63,5	1,3	2,0	
10					
11	783	0,1	0,00	0,00	
12					
13					
14	723 *	-59,9	-1,2	-1,8	
15	7494				Ergebnis ausgeschlossen / Result excluded
16					
17	682	-101,4	-2,1	-3,1	
18	770	-12,9	-0,27	-0,40	
19	828	45,1	0,93	1,4	
20	794	11,1	0,23	0,34	
21	886	102,8	2,1	3,2	
22	822	39,1	0,80	1,2	
23	833	50,1	1,0	1,5	
24	1307	524,4	11	16	
25	668	-114,9	-2,4	-3,5	

* Mean calculated by DLA

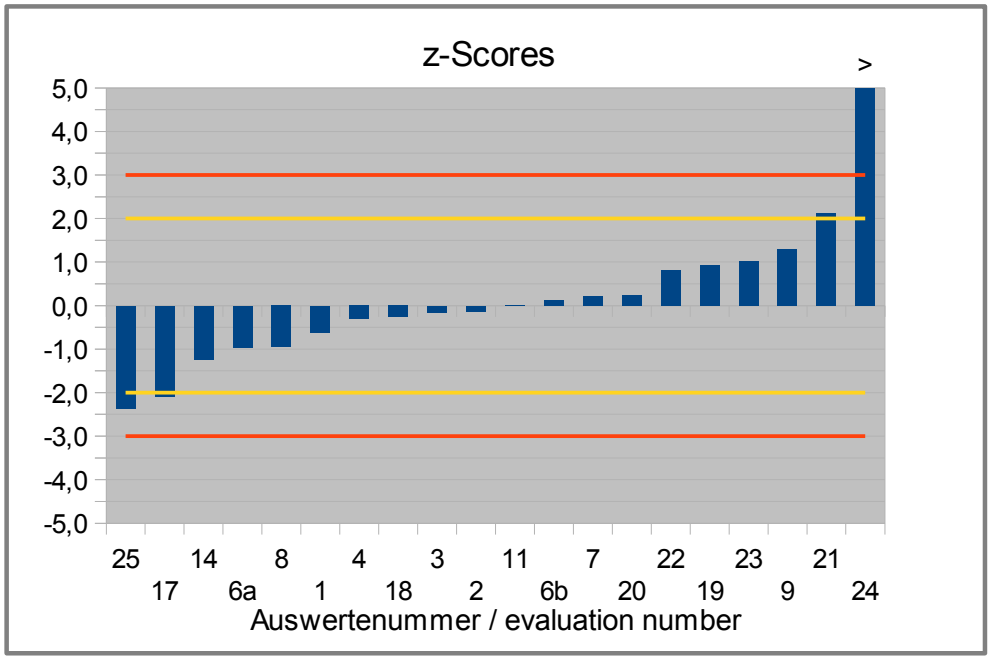


Abb. / Fig. 4: z-Scores Vitamin B2

4.3 Vitamin B6 (as Pyridoxine in mg/100g)

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results°	17
Number of outliers	2
Mean	749
Median	747
Robust Mean (x_{pt})	**749**
Robust standard deviation (S*)	**58,4**
Number with 2 replicates	17
Repeatability SD (S_r)	14,4
Repeatability (CV_r)	1,92%
Reproducibility SD (S_R)	62,1
Reproducibility (CV_R)	8,29%
Target range:	
Target standard deviation σ_{pt}	**45,2**
Target standard deviation (for Information)	31,3
lower limit of target range	**659**
upper limit of target range	**839**
Quotient S/σ_{pt}*	*1,3*
Standard uncertainty $U_{(xpt)}$	*17,7*
Results in the target range	*15*
Percent in the target range	*88%*

° *without outliers (results no. 8 and 15)*

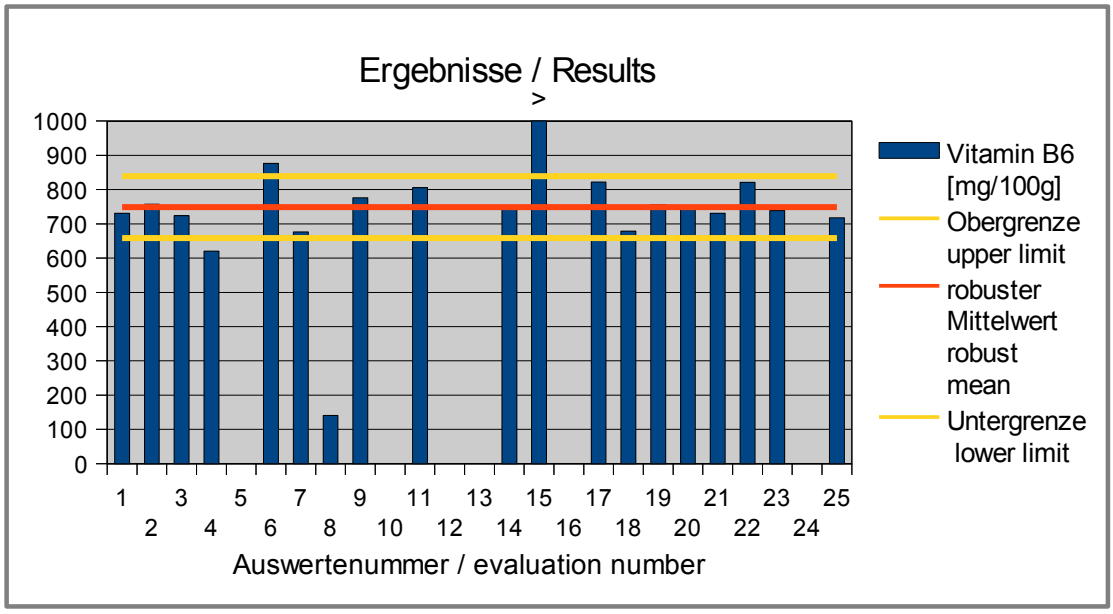


Abb. / Fig. 5: Ergebnisse Vitamin B6 / Results Vitamin B6

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Vitamin B6 [mg/100g]	Abweichung [mg/100g] Deviation [mg/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	731	-18,0	-0,40	-0,58	
2	758 *	8,5	0,19	0,27	
3	724	-25,0	-0,55	-0,80	
4	620	-129,0	-2,9	-4,1	
5					
6	877	127,5	2,8	4,1	
7	676	-72,7	-1,6	-2,3	
8	141				Ergebnis ausgeschlossen / Result excluded
9	776 *	26,8	0,59	0,86	
10					
11	805	56,4	1,2	1,8	
12					
13					
14	753	3,5	0,08	0,11	
15	7437 *				Ergebnis ausgeschlossen / Result excluded
16					
17	822 *	73,0	1,6	2,3	
18	679	-70,0	-1,6	-2,2	
19	756	7,0	0,15	0,22	
20	747	-2,0	-0,04	-0,06	
21	731	-18,4	-0,41	-0,59	
22	821	72,0	1,6	2,3	
23	739	-10,0	-0,22	-0,32	
24					
25	718	-31,0	-0,69	-0,99	

* Mean calculated by DLA

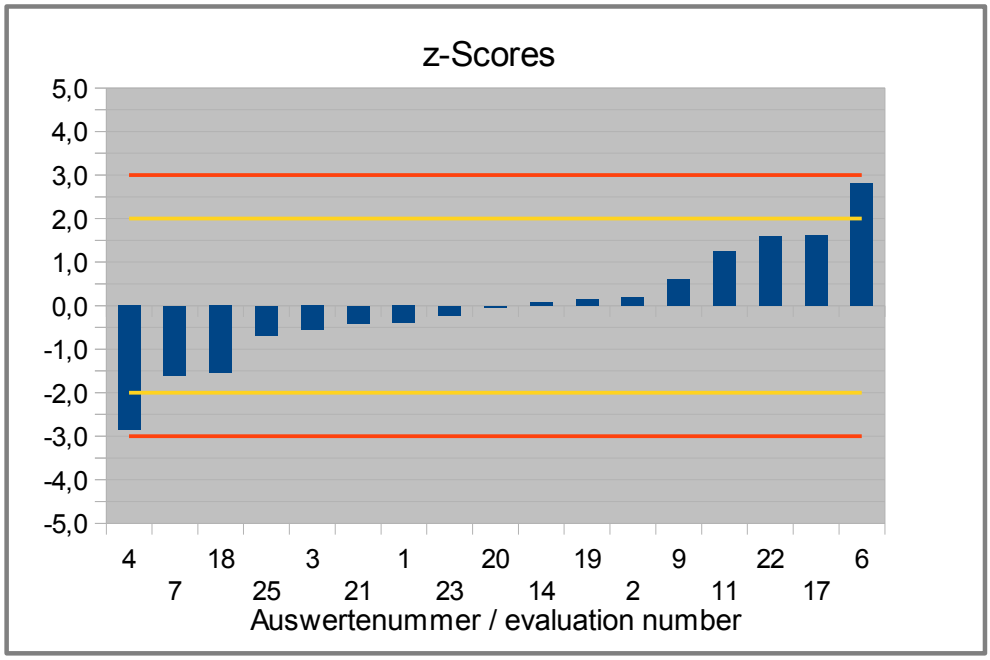


Abb. / Fig. 6: z-Scores Vitamin B6

4.4 Vitamin B12 (as Cyanocobalamine in µg/100g)

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	15
Number of outliers	-
Mean	1040
Median	1001
Robust Mean (x_{pt})	**1018**
Robust standard deviation (S*)	**102**
Number with 2 replicates	14
Repeatability SD (S_r)	49,0
Repeatability (CV_r)	4,82%
Reproducibility SD (S_R)	115
Reproducibility (CV_R)	11,3%
Target range:	
Target standard deviation σ_{pt}	**115**
lower limit of target range	**788**
upper limit of target range	**1247**
Quotient S/σ_{pt}*	*0,89*
Standard uncertainty $U_{(Xpt)}$	*32,9*
Results in the target range	*13*
Percent in the target range	*87%*

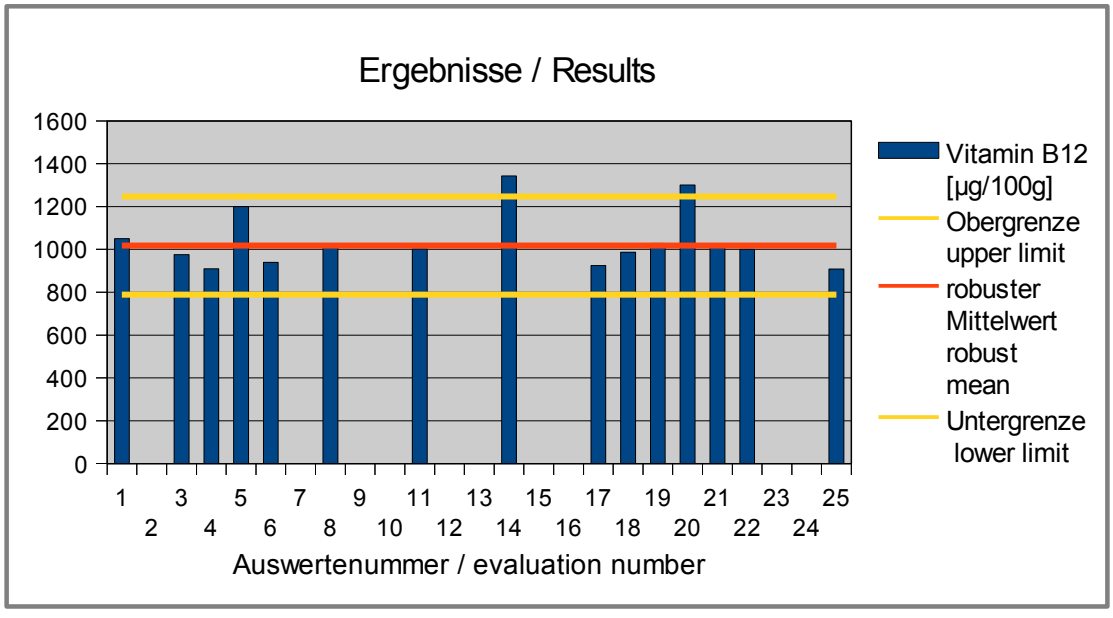


Abb. / Fig. 7: Ergebnisse Vitamin B12 / Results Vitamin B12

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Vitamin B12 [µg/100g]	Abweichung [µg/100g] Deviation [µg/100g]	z-Score (σ_{pt})	Hinweis Remark
1	1050	32	0,28	
2				
3	976	-42	-0,36	
4	910	-108	-0,94	
5	1200 *	182	1,6	
6	939	-79	-0,69	
7				
8	1020	2	0,02	
9				
10				
11	1001	-17	-0,15	
12				
13				
14	1344	326	2,8	
15				
16				
17	924 *	-94	-0,82	
18	986	-32	-0,28	
19	1028	10	0,09	
20	1300	282	2,5	
21	1013	-4	-0,04	
22	1000	-18	-0,16	
23				
24				
25	908	-110	-1,0	

* Mean calculated by DLA

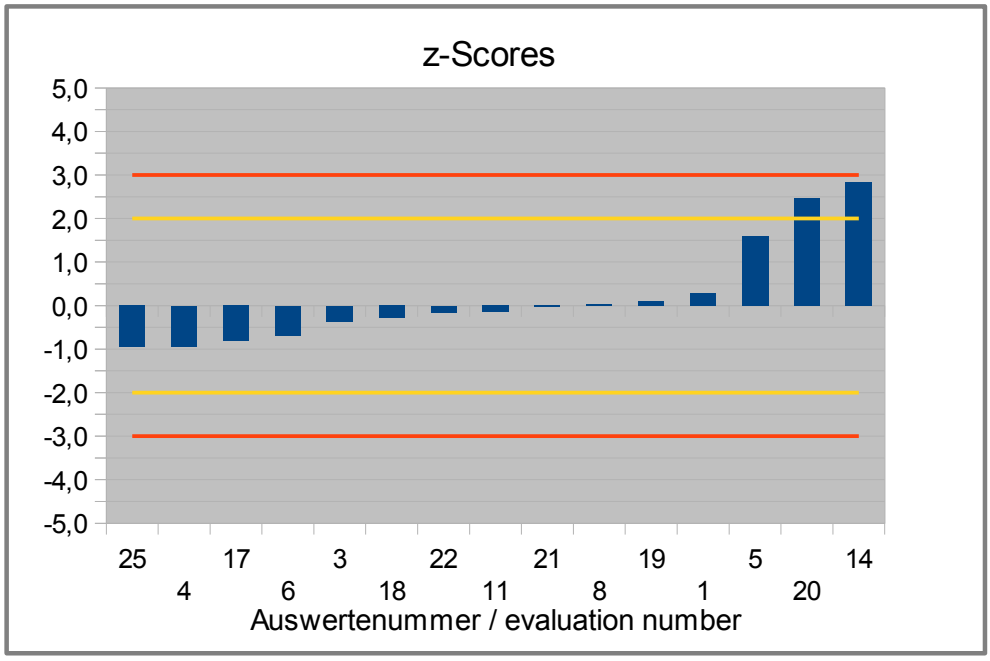


Abb. / Fig. 8: z-Scores Vitamin B12

4.5 Biotin (in µg/100g)

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results°	15
Number of outliers	2
Mean	66754
Median	66551
Robust Mean (X_{pt})	**67368**
Robust standard deviation (S*)	**9709**
Number with 2 replicates	14
Repeatability SD (S_r)	3709
Repeatability (CV_r)	5,39%
Reproducibility SD (S_R)	9214
Reproducibility (CV_R)	13,4%
Target range:	
Target standard deviation σ_{pt}	**11524**
Target standard deviation (for Information)	1430
lower limit of target range	**44319**
upper limit of target range	**90417**
Quotient S/σ_{pt}*	*0,84*
Standard uncertainty $U_{(Xpt)}$	*3134*
Results in the target range	*14*
Percent in the target range	*93%*

° *without outliers (results no. 7 and 18)*

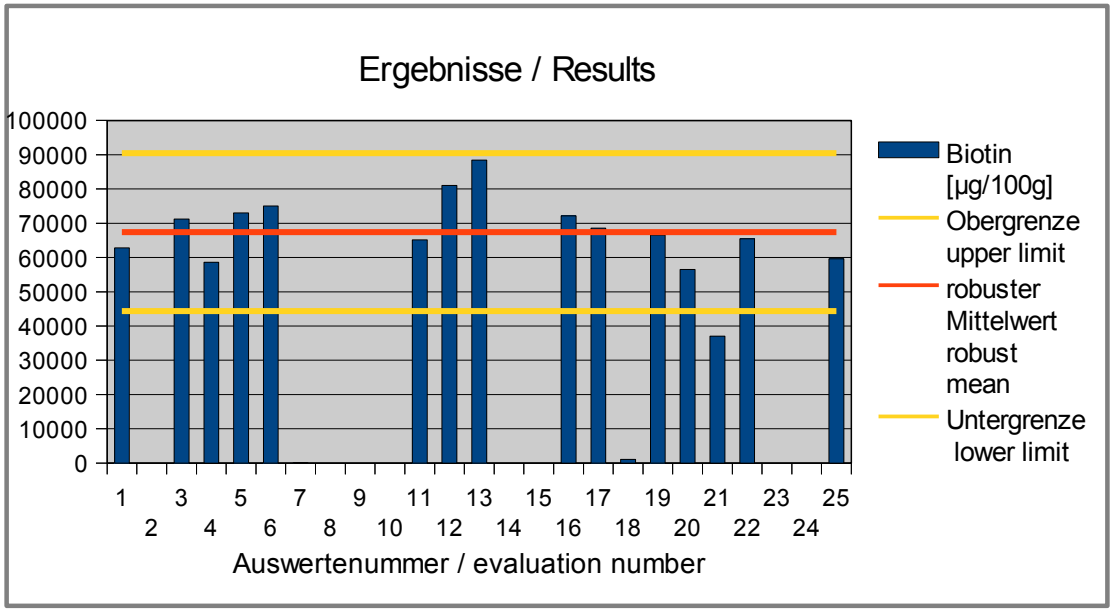


Abb. / Fig. 9: Ergebnisse Biotin / Results Biotin

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Biotin [µg/100g]	Abweichung [µg/100g] Deviation [µg/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	62800	-4568	-0,40	-3,2	
2					
3	71247	3879	0,34	2,7	
4	58600	-8768	-0,76	-6,1	
5	73000 *	5632	0,49	3,9	
6	75050	7682	0,67	5,4	
7	63,6				Ergebnis ausgeschlossen / Result excluded
8					
9					
10					
11	65162	-2206	-0,19	-1,5	
12	81015	13647	1,2	9,5	
13	88449	21081	1,8	15	
14					
15					
16	72184	4816	0,42	3,4	
17	68600 *	1232	0,11	0,86	
18	1058				Ergebnis ausgeschlossen / Result excluded
19	66551	-817	-0,07	-0,57	
20	56500	-10868	-0,94	-7,6	
21	37077	-30291	-2,6	-21	
22	65475	-1893	-0,16	-1,3	
23					
24					
25	59600	-7768	-0,67	-5,4	

* Mean calculated by DLA

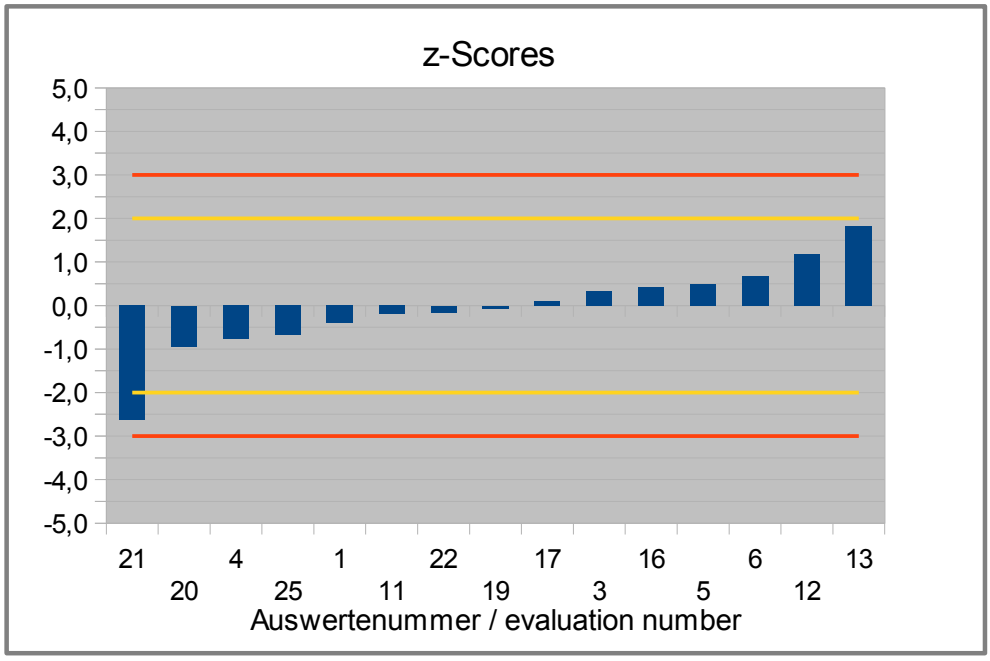


Abb. / Fig. 10: z-Scores Biotin

4.6 Vitamin C (as Ascorbic acid in mg/100g)

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	18
Number of outliers	-
Mean	36298
Median	33925
Robust Mean (X_{pt})	**34257**
Robust standard deviation (S*)	**2644**
Number with 2 replicates	17
Repeatability SD (S_r)	819
Repeatability (CV_r)	2,41%
Reproducibility SD (S_R)	2507
Reproducibility (CV_R)	7,38%
Target range:	
Target standard deviation σ_{pt}	**3595**
Target standard deviation (for Information)	805
lower limit of target range	**27067**
upper limit of target range	**41446**
Quotient S/σ_{pt}*	*0,74*
Standard uncertainty $U_{(Xpt)}$	*779*
Results in the target range	*17*
Percent in the target range	*94%*

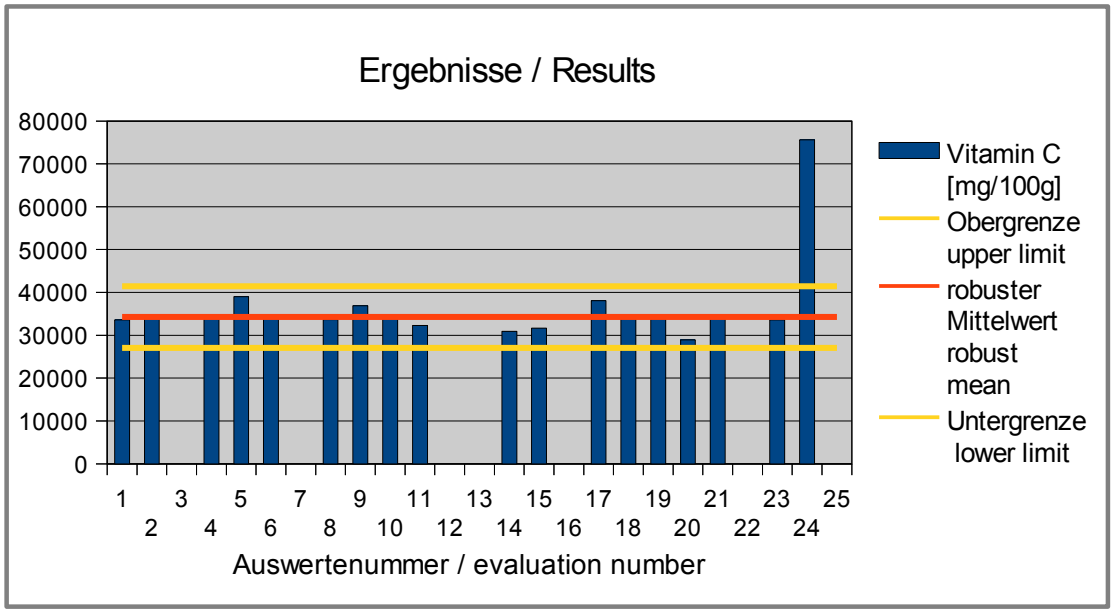


Abb. / Fig. 11: Ergebnisse Vitamin C / Results Vitamin C

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Vitamin C [mg/100g]	Abweichung [mg/100g] Deviation [mg/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	33610	-647	-0,18	-0,80	
2	34850 *	593	0,17	0,74	
3					
4	34547	290	0,08	0,36	
5	39000 *	4743	1,3	5,9	
6	34050	-207	-0,06	-0,26	
7					
8	33648	-609	-0,17	-0,76	
9	36898 *	2641	0,73	3,3	
10	33881	-376	-0,10	-0,47	
11	32264	-1993	-0,55	-2,5	
12					
13					
14	30926	-3331	-0,93	-4,1	
15	31668 *	-2589	-0,72	-3,2	
16					
17	38043 *	3786	1,1	4,7	
18	34107	-150	-0,04	-0,19	
19	33837	-420	-0,12	-0,52	
20	28900	-5357	-1,5	-6,7	
21	33969	-288	-0,08	-0,36	
22					
23	33589	-668	-0,19	-0,83	
24	75586	41330	11	51	
25					

* Mean calculated by DLA

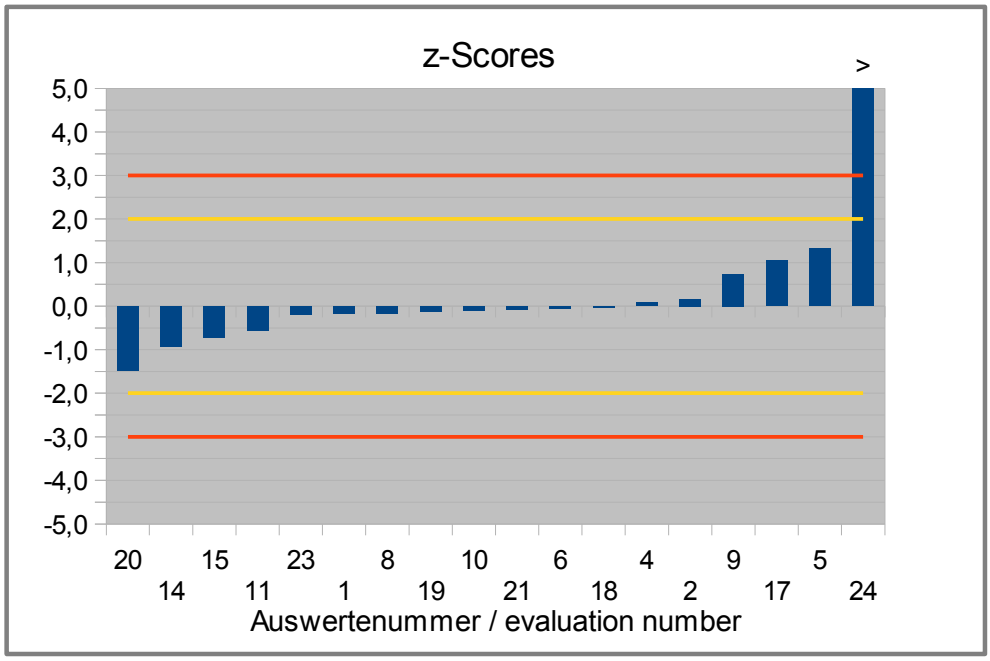


Abb. / Fig. 12: z-Scores Vitamin C

4.7 Folic acid (as Pteroylmonoglutamic acid in µg/100g)

Vergleichsuntersuchung / Proficiency Test

Kenndaten	
Anzahl der Messergebnisse°	15
Anzahl der Ausreißer	2
Mittelwert	88642
Median	87785
Robuster Mittelwert (X_{pt})	**88412**
Robuste Standardabweichung (S*)	**9691**
Anzahl mit 2 Wiederholmessungen	14
Wiederholstandardabweichung (S_r)	3691
Variationskoeffizient (VK_r)	4,27%
Vergleichsstandardabweichung (S_R)	10134
Variationskoeffizient (VK_R)	11,7%
Zielkenndaten:	
Zielstandardabweichung σ_{pt}	**5095**
Untere Grenze des Zielbereichs	**78223**
Obere Grenze des Zielbereichs	**98602**
Quotient S/σ_{pt}*	*1,9*
Standardunsicherheit $U_{(Xpt)}$	*3128*
Ergebnisse im Zielbereich	*12*
Prozent im Zielbereich	*80%*

° *Messergebnisse ohne Ausreißer Nr. 9 und 23*

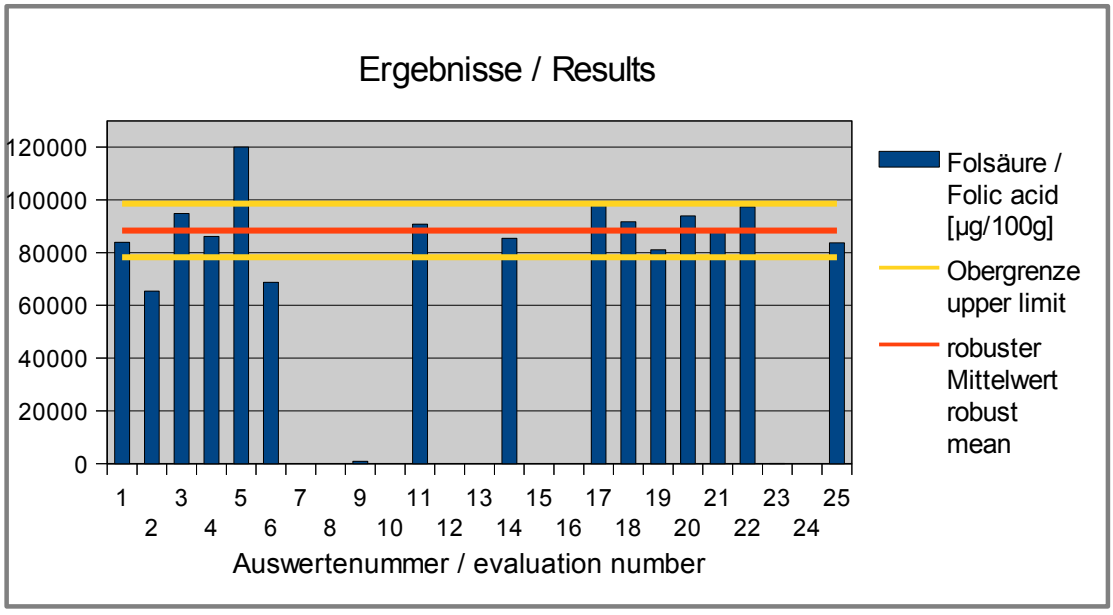


Abb. / Fig. 13: Ergebnisse Folsäure / Results Folic Acid

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Folsäure / Folic acid [µg/100g]	Abweichung [µg/100g] Deviation [µg/100g]	z-Score (σ_{pt})	Hinweis Remark
1	83950	-4462	-0,88	
2	65450 *	-22962	-4,5	
3	94865	6453	1,3	
4	86100	-2312	-0,45	
5	120000 *	31588	6,2	
6	68700	-19712	-3,9	
7				
8				
9	888 *			Ergebnis ausgeschlossen / Result excluded
10				
11	90827	2414	0,47	
12				
13				
14	85386	-3027	-0,59	
15				
16				
17	98825 *	10413	2,0	
18	91683	3271	0,64	
19	81065	-7347	-1,4	
20	93900	5488	1,1	
21	87785	-627	-0,12	
22	97400	8988	1,8	
23	92,7			Ergebnis ausgeschlossen / Result excluded
24				
25	83700	-4712	-0,92	

* Mean calculated by DLA

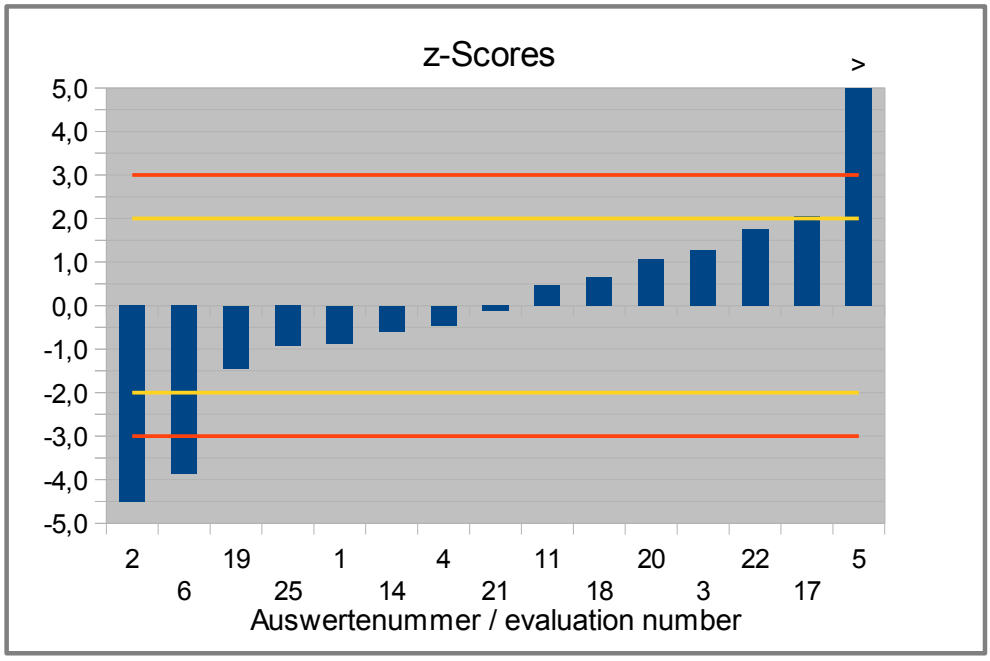


Abb. / Fig. 14: z-Scores Folsäure / Folic Acid

4.8 Niacin (in mg/100g)

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	18
Number of outliers	2
Mean	8043
Median	8035
Robust Mean (X_{pt})	**8062**
Robust standard deviation (S*)	**324**
Number with 2 replicates	18
Repeatability SD (S_r)	246
Repeatability (CV_r)	3,05%
Reproducibility SD (S_R)	470
Reproducibility (CV_R)	5,84%
Target range:	
Target standard deviation σ_{pt}	**236**
Target standard deviation (for Information)	308
lower limit of target range	**7591**
upper limit of target range	**8533**
Quotient S/σ_{pt}*	*1,4*
Standard uncertainty $U_{(Xpt)}$	*95*
Results in the target range	*15*
Percent in the target range	*83%*

° *without outliers (results no. 19 and 25)*

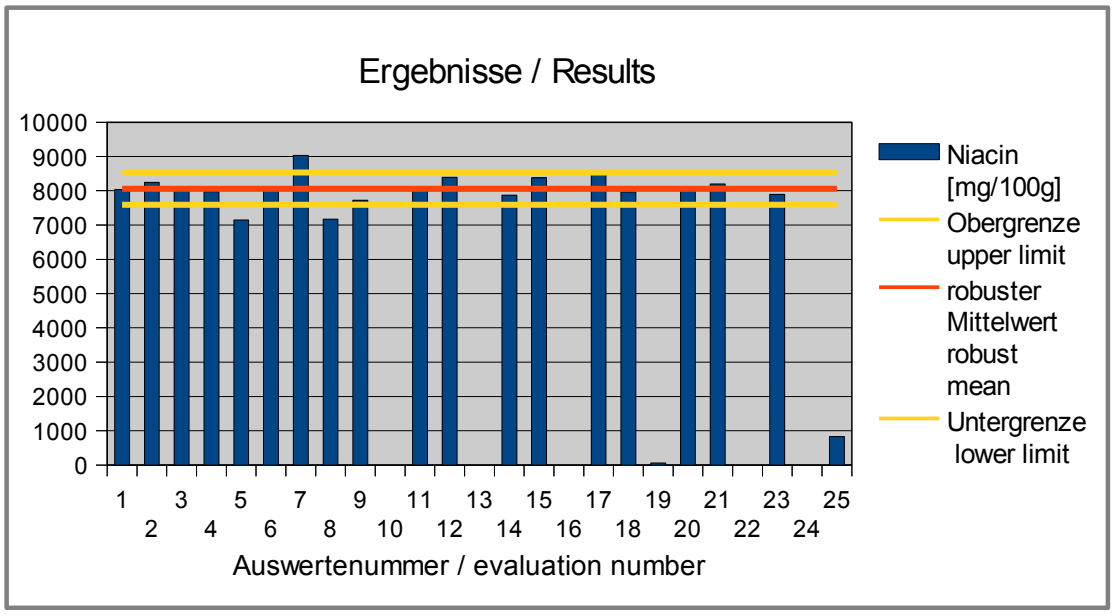


Abb. / Fig. 15: Ergebnisse Niacin / Results Niacin

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Niacin [mg/100g]	Abweichung [mg/100g] Deviation [mg/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	8030	-32	-0,14	-0,10	
2	8245 *	183	0,78	0,59	
3	8005	-57	-0,24	-0,19	
4	7969	-93	-0,40	-0,30	
5	7150 *	-912	-3,9	-3,0	
6	8115	53	0,22	0,17	
7	9031	968	4,1	3,1	
8	7170	-892	-3,8	-2,9	
9	7727 *	-336	-1,4	-1,09	
10					
11	8070	8	0,03	0,03	
12	8392	330	1,4	1,1	
13					
14	7871	-192	-0,81	-0,62	
15	8379 *	317	1,3	1,0	
16					
17	8525 *	463	2,0	1,5	
18	7966	-96	-0,41	-0,31	
19	61,5				Ergebnis ausgeschlossen / Result excluded
20	8040	-22	-0,09	-0,07	
21	8196	133	0,57	0,43	
22					
23	7891	-171	-0,73	-0,56	
24					
25	831				Ergebnis ausgeschlossen / Result excluded

* Mean calculated by DLA

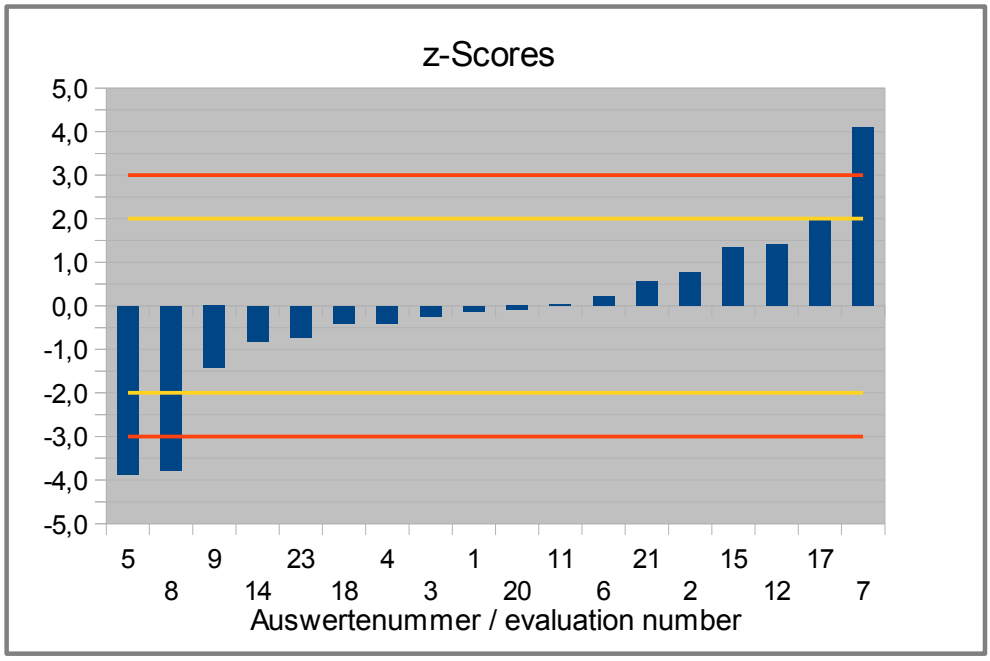


Abb. / Fig. 16: z-Scores Niacin

4.9 Pantothenic acid (in mg/100g)

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	16
Number of outliers	-
Mean	2664
Median	2641
Robust Mean (x_{pt})	**2667**
Robust standard deviation (S*)	**196**
Number with 2 replicates	15
Repeatability SD (S_r)	140
Repeatability (CV_r)	5,15%
Reproducibility SD (S_R)	233
Reproducibility (CV_R)	8,60%
Target range:	
Target standard deviation σ_{pt}'	**111**
Target standard deviation (for Information)	92,0
lower limit of target range	**2446**
upper limit of target range	**2888**
Quotient S/σ_{pt}'*	*1,8*
Standard uncertainty $U_{(xpt)}$	*61,3*
Results in the target range	*12*
Percent in the target range	*75%*

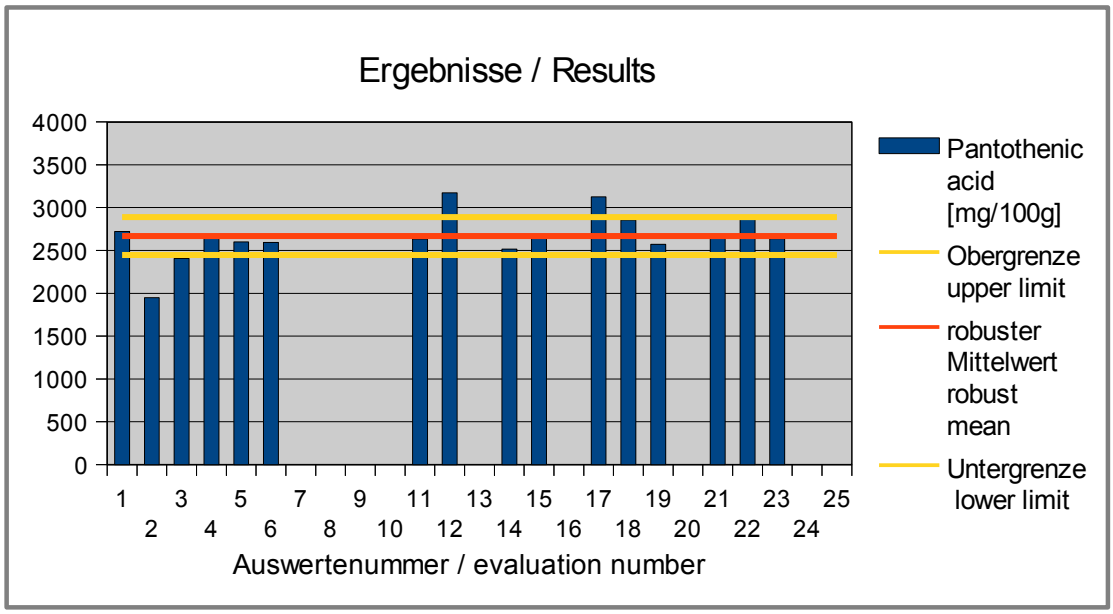


Abb. / Fig. 17: Ergebnisse Pantothensäure / Results Pantothenic Acid

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Pantothenic Acid [mg/100g]	Abweichung [mg/100g] Deviation [mg/100g]	z'-Score (σ_{pt})	Hinweis Remark
1	2720	53	0,48	
2	1950 *	-717	-6,5	
3	2407	-260	-2,3	
4	2645	-22	-0,20	
5	2600 *	-67	-0,60	
6	2595	-72	-0,65	
7				
8				
9				
10				
11	2633	-33	-0,30	
12	3172	505	4,6	
13				
14	2516	-151	-1,4	
15	2685 *	18	0,17	
16				
17	3125 *	458	4,1	
18	2855	188	1,7	
19	2573	-94	-0,85	
20				
21	2643	-23	-0,21	
22	2863	196	1,8	
23	2638	-29	-0,26	
24				
25				

* Mean calculated by DLA

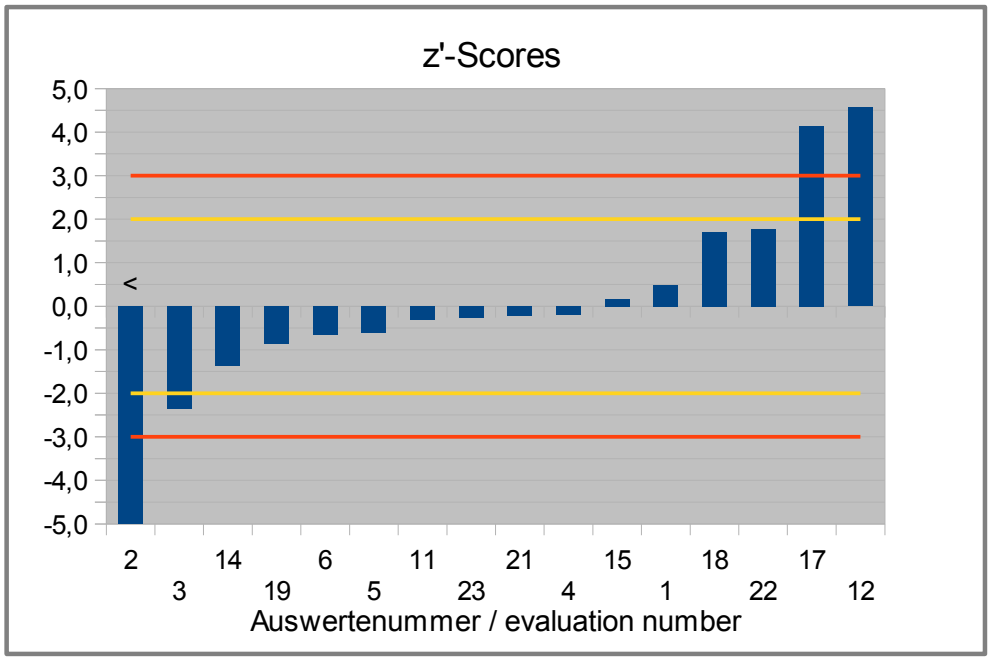


Abb. / Fig. 18: z'-Scores Pantothensäure / Pantothenic Acid

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

Parameter	Participant	Unit	Sample I DLA No.	Sample II DLA No.	Date of analysis	Result (Mean)	Result I	Result II	Limit of quan-tification	Incl. RR	Recovery rate
					Day/Month					yes / no	in %
Vitamin B1 (als Thiamin-Kation) / Vitamin B1 (as Thiamine-cation)	1	mg/100g	25	71	14.05.2019	623	635	610			
	2	mg/100g	38	58			757	776		no	
	3	mg/100g	3	93	20.06.2019	648	624	672	0,179	no	not determined
	4	mg/100g	19	77	24.05.2019	594	591	598	0,2	no	
	5	mg/100g									
	6	mg/100g	20	76	28.05.2019	574	540	608	0,1	no	77
	7	mg/100g									
	8	mg/100g									
	9	mg/100g	18	78	11.06.2019		1089	1046	22	no	
	10	mg/100g									
	11	mg/100g	40	55		667,21	668,29	666,12			
	12	mg/100g	46	50	22.05.2019	673,5	671,1	676		no	
	13	mg/100g									
	14	mg/100g	42	54	23.05.2019	575,5	571	580	48	no	100,78
	15	mg/100g			17.06.		641	637	2,5	no	
	16	mg/100g	29	68	28.05.2019	628,22	623,74	632,69		no	
	17	mg/100g	30	66	24.05.2019		885	898		no	
	18	mg/100g	80	16	23.05.2019	673	675,64	670,54	0,1	no	
	19	mg/100g	5	91	11.06.2019	658,5	651	666	n/a	no	n/a
	20	mg/100g	8	88		687	689	684			
	21	mg/100g	13	84	27.05.2019	824,7	842,4	807		no	
	22	mg/100g	1	95	30.05.2019	735	716	753	3	no	120
	23	mg/100g	24	72	05.06.2019	1071	1070	1072		no	
	24	mg/100g	36	60	21.06.	612,5	611,1	613,9	0,1	yes	87
	25	mg/100g	31	65	20.06.	731	736	726	0,1	no	90

Parameter	Participant	Unit	Sample I DLA No.	Sample II DLA No.	Date of analysis	Result (Mean)	Result I	Result II	Limit of quan-tification	Incl. RR	Recovery rate
					Day/Month					yes / no	in %
Vitamin B2 (als Riboflavin) / Vitamin B2 (as Riboflavin)	1	mg/100g	25	71	14.05.2019	753	769	736			
	2	mg/100g	38	58			755	798		no	
	3	mg/100g	3	93	20.06.2019	775	768	782	0,05	no	not determined
	4	mg/100g	19	77	24.05.2019	768	768	767	0,2	no	
	5	mg/100g									
	6	mg/100g	20	76	28.05.2019	736	712	760	0,1	no	93
	7	mg/100g	11	87	07.06.2019	793,2	778,5	807,9			
	8	mg/100g	21	73	05.06.2019	737	731	742	0,05		
	9	mg/100g	18	78	11.06.2019		841,6	851,2	7	no	
	10	mg/100g									
	11	mg/100g	40	55		783,01	787,13	778,88			
	12	mg/100g									
	13	mg/100g									
	14	mg/100g	42	54	22.05.2019	723	718	728	35	no	93,63
	15	mg/100g			17.06.		7516	7471	2,5	no	
	16	mg/100g									
	17	mg/100g	30	66	24.05.2019		693	670		no	
	18	mg/100g	80	16	23.05.2019	770	772,66	767,03	0,01	no	
	19	mg/100g	5	91	11.06.2019	828	826	830	n/a	no	n/a
	20	mg/100g	8	88		794	808	779			
	21	mg/100g	13	84	27.05.2019	885,7	848,4	867		no	
	22	mg/100g	1	95	30.05.2019	822	851	792	3	no	106
	23	mg/100g	24	72	20.05.2019	833	838	827		no	
	24	mg/100g	36	60	21.06.	1307,3	1125,2	1489,3	0,1	yes	91
	25	mg/100g	31	65	20.06.	668	681	654	0,1	no	106

Parameter	Participant	Unit	Sample I DLA No.	Sample II DLA No.	Date of analysis	Result (Mean)	Result I	Result II	Limit of quantification	Incl. RR	Recovery rate
					Day/Month					yes / no	in %
Vitamin B6 (als Pyridoxin) / Vitamin B6 (as Pyridoxine)	1	mg/100g	25	71	14.05.2019	731	745	717			
	2	mg/100g	38	58			746	769		no	
	3	mg/100g	3	93	20.06.2019	724	730	718	0,11	no	not determined
	4	mg/100g	19	77	24.05.2019	620	616	624	0,2	no	
	5	mg/100g									
	6	mg/100g	20	76	28.05.2019	876,5	902	851	0,1	no	97
	7	mg/100g	11	87	13.06.2019	676,3	672,9	679,7			
	8	mg/100g	21	73	17.06.2019	141	143	139	0,05		
	9	mg/100g	18	78	11.06.2019		768,8	782,9	9	no	
	10	mg/100g									
	11	mg/100g	40	55		805,36	796,47	814,26			
	12	mg/100g									
	13	mg/100g									
	14	mg/100g	42	54	21.05.2019	752,5	745	760	14	no	99,7
	15	mg/100g			17.06.		7502	7371	5	no	
	16	mg/100g									
	17	mg/100g	30	66	27.05.2019		812	832		no	
	18	mg/100g	80	16	23.05.2019	679	689,67	667,83	4	no	
	19	mg/100g	5	91	11.06.2019	756	752	760	n/a	no	n/a
	20	mg/100g	8	88		747	746	748			
	21	mg/100g	13	84	27.05.2019	730,6	722,4	738,8		no	
	22	mg/100g	1	95	30.05.2019	821	817	824	3	no	115
	23	mg/100g	24	72	20.05.2019	739	750	727		no	
	24	mg/100g									
	25	mg/100g	31	65	28.06.	718	709	727	0,1	no	105

Parameter	Participant	Unit	Sample I DLA No.	Sample II DLA No.	Date of analysis	Result (Mean)	Result I	Result II	Limit of quantification	Incl. RR	Recovery rate
					Day/Month					yes / no	in %
Vitamin B12 (als Cyanocobalamin) / Vitamin B12 (as Cyanocobalamine)	1	µg/100g	25	71	16.05.2019	1050	1010	1010			
	2	µg/100g	38	58							
	3	µg/100g	3	93	06.06.2019	976	887	1065	0,03	no	not determined
	4	µg/100g	19	77	24.05.2019	910	970	850	0,03	no	
	5	µg/100g	52	44	29.05.2019	31.05.2019	1200	1200			
	6	µg/100g	20	76	28.05.2019	939	901	977	0,2	no	95
	7	µg/100g									
	8	µg/100g	21	73	14.06.2019	1020	1060	985	0,5		
	9	µg/100g									
	10	µg/100g									
	11	µg/100g	40	55		1.001,00	997,27	1.004,74			
	12	µg/100g									
	13	µg/100g									
	14	µg/100g	42	54	21.05.2019	1343,5	1354	1333	600	no	-
	15	µg/100g			17.06.						
	16	µg/100g									
	17	µg/100g	30	66	18.06.2019		944	904		no	
	18	µg/100g	80	16	24.05.2019	986	999,22	973	25	no	
	19	µg/100g	5	91	10.06.2019	1027,5	1011	1044	n/a	no	n/a
	20	µg/100g	8	88		1300	1300	1300			
	21	µg/100g	13	84	03.06.2019	1013,3	1001,4	1025,3		yes	89
	22	µg/100g	1	95	28.05.2019	999,8	1029,8	969,8	0,5	no	
	23	µg/100g									
	24	µg/100g									
	25	µg/100g	31	65	31.05.	908	885	930	0,03	no	106

Parameter	Participant	Unit	Sample I DLA No.	Sample II DLA No.	Date of analysis	Result (Mean)	Result I	Result II	Limit of quan-tification	Incl. RR	Recovery rate
					Day/Month					yes / no	in %
	1	µg/100g	25	71	16.05.2019	62800	62700	62900			
	2	µg/100g	38	58							
	3	µg/100g	3	93	20.06.2019	71247	69209	73284	0,08	no	not determined
	4	µg/100g	19	77	29.05.2019	58600	58300	58900	0,08	no	
	5	µg/100g	52	44	28.05.2019	30.05.2019	75000	71000			
	6	µg/100g	20	76	28.05.2019	75050	71200	78900	4	no	110
	7	µg/100g	11	87	31.05.2019	63,6	62,7	64,5			
	8	µg/100g									
	9	µg/100g									
	10	µg/100g									
	11	µg/100g	40	55		65.161,60	64.721,16	65.602,04			
	12	µg/100g	46	50	17.05.2019	81015	77100	84900		no	
Biotin	13	µg/100g	47	49	18.06.2019	88449,46	84496,97	92401,95	0,5	yes	98
	14	µg/100g	-	-	-	-	-	-	-	-	-
	15	µg/100g			17.06.						
	16	µg/100g	29	68	17.06.2019	72184	72416	71952		no	
	17	µg/100g	30	66	18.06.2019		63000	74200		no	
	18	µg/100g	80	16	29.05.2019	1058	1078,76	1038,05	0,1	no	
	19	µg/100g	5	91	17.06.2019	66551	68136	64966	n/a	no	n/a
	20	µg/100g	8	88		56500	56000	57000			
	21	µg/100g	13	84	17.05.2019	37076,5	39283	34870		yes	91,6
	22	µg/100g	1	95	13.05.2019	65475	68300	62650	0,08	no	
	23	µg/100g									
	24	µg/100g									
	25	µg/100g	31	65	29.05.	59600	59400	59800	0,08	no	96

Parameter	Participant	Unit	Sample I DLA No.	Sample II DLA No.	Date of analysis	Result (Mean)	Result I	Result II	Limit of quantification	Incl. RR	Recovery rate
					Day/Month					yes / no	in %
Vitamin C (als Ascorbinsäure) / Vitamin C (as Ascorbic acid)	1	mg/100g	25	71	15.05.2019	33610	33830	33390			
	2	mg/100g	38	58			35200	34500		no	
	3	mg/100g			not determined	not determined	not determined	not determined	not determined	not determined	not determined
	4	mg/100g	19	77	May	34547	35230	33864		no	
	5	mg/100g	52	44	14.06.2019	14.06.2019	39000	39000			
	6	mg/100g	20	76	03.06.2019	34050	32800	35300	1	no	98
	7	mg/100g									
	8	mg/100g	21	73	17.06.2019	33648	33369	33927	5		
	9	mg/100g	18	78	04.06.2019		37731	36064	5	no	
	10	mg/100g	15	79	27.05.2019	33881	33965	33797	10	yes	99,5
	11	mg/100g	40	55		32.263,52	32.591,63	31.935,40			
	12	mg/100g									
	13	mg/100g									
	14	mg/100g	42	54	03.+04.+05. 06.2019	30925,5	30815	31036	2814	no	89,30%
	15	mg/100g			17.06.		31797	31539		no	
	16	mg/100g									
	17	mg/100g	30	66	24.05.2019		38929	37157		no	
	18	mg/100g	80	16	23.05.2019	34107	34567,7	33645,31	3	no	
	19	mg/100g	5	91	07.06.2019	33837	33856	33818	n/a	no	n/a
	20	mg/100g	8	88		28900	30100	27700			
	21	mg/100g	13	84	15.05.2019	33969	34314	33624		no	
	22	mg/100g									
	23	mg/100g	24	72	13.05.2019	33589	33770	33409		no	
	24	mg/100g	36	60	21.06.	75586,3	75673,9	75498,6	0,1	yes	90
	25	mg/100g									

Parameter	Participant	Unit	Sample I DLA No.	Sample II DLA No.	Date of analysis	Result (Mean)	Result I	Result II	Limit of quan-tification	Incl. RR	Recovery rate
					Day/Month					yes / no	in %
Folsäure (als Pteroylmonoglutaminsäure) / Folic acid (as Pteroylmonoglutaminic acid)	1	µg/100g	25	71	15.05.2019	83950	85400	82500			
	2	µg/100g	38	58			63100	67800		no	
	3	µg/100g	3	93	20.06.2019	94865	96300	93430	0,181	no	not determined
	4	µg/100g	19	77	13.06.2019	86100	91400	80800	0,2	no	
	5	µg/100g	52	44	21.05.2019	23.05.2019	130000	110000			
	6	µg/100g	20	76	28.05.2019	68700	66900	70500	16	no	76
	7	µg/100g									
	8	µg/100g									
	9	µg/100g	18	78	11.06.2019		885	890	7000	no	
	10	µg/100g									
	11	µg/100g	40	55		90.826,85	89.193,83	92.459,86			
	12	µg/100g									
	13	µg/100g									
	14	µg/100g	42	54	14.05.2019	85385,5	83524	87247	40000	no	-
	15	µg/100g			17.06.						
	16	µg/100g									
	17	µg/100g	30	66	24.05.2019		97150	100500		no	
	18	µg/100g	80	16	23.05.2019	91683	93125,03	90239,92	1	no	
	19	µg/100g	5	91	06.06.2019	81065	80967	81163	n/a	no	n/a
	20	µg/100g	8	88		93900	97000	90800			
	21	µg/100g	13	84	15.05.2019	87785	89412	86158		yes	95,3
	22	µg/100g	1	95	21.05.2019	97400	102867	91817	0,16	no	
	23	µg/100g	24	72	20.05.2019	92,7	93,1	92,2		no	
	24	µg/100g									
	25	µg/100g	31	65	31.05.	83700	82950	84450	0,16	no	104

Parameter	Participant	Unit	Sample I DLA No.	Sample II DLA No.	Date of analysis	Result (Mean)	Result I	Result II	Limit of quan-tification	Incl. RR	Recovery rate
					Day/Month					yes / no	in %
Niacin	1	mg/100g	25	71	14.05.2019	8030	8150	7910			
	2	mg/100g	38	58			8140	8350		no	
	3	mg/100g	3	93	20.06.2019	8005	7963	8046	0,128	no	not determined
	4	mg/100g	19	77	24.05.2019	7969	8077	7861	0,2	no	
	5	mg/100g	52	44	20.05.2019	22.05.2019	7300	7000			
	6	mg/100g	20	76	28.05.2019	8115	8450	7780	0,3	no	98
	7	mg/100g	11	87	05.06.2019	9030,5	9184	8877			
	8	mg/100g	21	73	21.06.2019	7170	7180	7160	0,5		
	9	mg/100g	18	78	11.06.2019		7915	7538	7	no	
	10	mg/100g									
	11	mg/100g	40	55		8.070,03	8.146,72	7.993,35			
	12	mg/100g	46	50	20.05.2019	8392,1	8351,3	8432,9		no	
	13	mg/100g									
	14	mg/100g	42	54	12.06.2019	7870,5	7740	8001	1	no	108,5 % and 109,9%
	15	mg/100g			17.06.		8362	8396	2,5	no	
	16	mg/100g									
	17	mg/100g	30	66	18.06.2019		9040	8010		no	
	18	mg/100g	80	16	23.05.2019	7966	7952,97	7978,4	0,5	no	
	19	mg/100g	5	91	11.06.2019	61,5	63	60	n/a	no	n/a
	20	mg/100g	8	88		8040	8010	8070			
	21	mg/100g	13	84	27.05.2019	8195,7	8059,2	8332,3		no	
	22	mg/100g									
	23	mg/100g	24	72	20.05.2019	7891	7899	7883		no	
	24	mg/100g									
	25	mg/100g	31	65	20.06.	831	818	845	0,016	no	103

Parameter	Participant	Unit	Sample I DLA No.	Sample II DLA No.	Date of analysis	Result (Mean)	Result I	Result II	Limit of quan-tification	Incl. RR	Recovery rate
					Day/Month					yes / no	in %
Pantothensäure / Pantothenic acid	1	mg/100g	25	71	14.05.2019	2720	2530	2900			
	2	mg/100g	38	58			1900	2000		no	
	3	mg/100g	3	93	20.06.2019	2407	2342	2472	0,195	no	not determined
	4	mg/100g	19	77	21.06.2019	2645	2576	2714	0,04	no	
	5	mg/100g	52	44	24.05.2019	25.05.2019	2700	2500			
	6	mg/100g	20	76	28.05.2019	2595	2450	2740	0,2	no	100
	7	mg/100g									
	8	mg/100g									
	9	mg/100g									
	10	mg/100g									
	11	mg/100g	40	55		2.633,32	2.616,77	2.649,88			
	12	mg/100g	46	50	13.06.2019	3171,9	3368,6	2975,2		no	
	13	mg/100g									
	14	mg/100g	42	54	22.05.2019	2515,5	2373	2658	2000	no	-
	15	mg/100g			17.06.		2740	2630	0,05	no	
	16	mg/100g									
	17	mg/100g	30	66	18.06.2019		3080	3170		no	
	18	mg/100g	80	16	23.05.2019	2855	2895,95	2813,95	0,02	no	
	19	mg/100g	5	91	11.06.2019	2572,5	2548	2597	n/a	no	n/a
	20	mg/100g	-	-	-	-	-	-	-	-	-
	21	mg/100g	13	84	27.05.2019	2643,2	2647,7	2638,8		no	
	22	mg/100g	1	95	15.05.2019	2863	2795	2930	0,04	no	
	23	mg/100g	24	72	20.05.2019	2638	2664	2613		no	
	24	mg/100g									
	25	mg/100g									

5.1.2 Analytical Methods

Parameter	Partici-pant	Method description as in test report / norm / literature	Sample preparation	Measuring method	Calibration / Reference material	Recovery rate with same matrix	Method accredited ISO/IEC 17025	Further Remarks
						yes / no	yes / no	
Vitamin B1 (als Thiamin-Kation) / Vitamin B1 (as Thiamine-cation)	1	SOP M843, HPLC-UV		HPLC-UV	available		yes	
	2						yes	
	3	Quantitative determination of water-soluble vitamins by HPLC in food supplements			PT-Material		yes	
	4	internal method HPLC		HPLC based on VDLUFA methods III No. 13.9.1			yes	
	5							
	6	in-house method , LCMSMS				yes	no	old method : 667 mg/kg
	7							
	8							
	9						yes	
	10							
	11			HPLC			yes	
	12							
	13							
	14	2.019/006-05	without enzym. clean-up / sample preparation	HPLC	Thiamin-HCl	yes	yes	-
	15	in-house method, premix, N04_22ME					yes	
	16	ASU L00.00-83, June 2015					yes	
	17	DIN EN 14122:2006					yes	
	18	in house method	Extraction in 0,1% H3PO4	HPLC DAD C18 column	3-5-point calibration	no	yes	
	19	HPLC	n/a	MQLTM-0153	USP	no	yes	n/a
	20						yes	MW = 265.37
	21			HPLC/UV	external calibration	no	no	
	22	internal method HPLC-MS-MS (P4-02-01-12-2423)	enzymatic digestion, centrifugation, filtration, dilution, addition ISTD	HPLC-MS/MS	external calibration with ISTD	no	yes	recovery with NIST-SRM-3280
	23	LAV 21.0010-02; HPLC-FLD					yes	
	24	In house method	Extraction with water	UPLC-MS/MS		yes	no	
	25	HPLC			LVU-Lippold No.17-20	no	yes	

Parameter	Partici-pant	Method description as in test report / norm / literature	Sample preparation	Measuring method	Calibration / Re-ference material	Recovery rate with same matrix	Method ac-credited ISO/IEC 17025	Further Remarks
						yes / no	yes / no	
Vitamin B2 (als Riboflavin) / Vitamin B2 (as Riboflavin)	1	SOP M843, HPLC-UV		HPLC-UV	available		yes	
	2						yes	
	3	Quantitative determination of water-soluble vitamins by HPLC in food supplements			PT-Material		yes	
	4	internal method HPLC		HPLC based on VDLUFA methods III No. 13.9.1			yes	
	5							
	6	in-house method , LCMSMS				yes	no	old method : 789mg/kg
	7	HPLC fluo (in house method)			yes (10 calibration points + reference material)	yes	yes	
	8	HPLC with FL					yes	
	9						yes	
	10							
	11			HPLC			yes	
	12							
	13							
	14	2.019/006-05	without enzym. clean-up / sample preparation	HPLC	Riboflavin	yes	yes	-
	15	in-house method, premix, N04_22ME					yes	
	16							
	17	DIN EN 14152:2006					yes	
	18	in house method	Extraction in 0,1% H3PO4	HPLC DAD C18 column	3-5-point calibration	no	yes	
	19	HPLC	n/a	MQLTM-0153	USP	no	yes	n/a
	20						yes	
	21			HPLC/UV	external calibration	no	no	
	22	internal method HPLC-MS-MS (P4-02-01-12-2423)	enzymatic digestion, centrifugation, filtration, dilution, addition ISTD	HPLC-MS/MS	external calibration with ISTD	no	yes	recovery with NIST-SRM-3280
	23	LAV 21.0017-02; HPLC-FLD					yes	
	24	In house method	Extraction with water	UPLC-MS/MS		yes	no	
	25	HPLC			LVU-Lippold Nr. 17-20	no	yes	

Parameter	Partici-pant	Method description as in test report / norm / literature	Sample preparation	Measuring method	Calibration / Re-ference material	Recovery rate with same matrix	Method ac-credited ISO/IEC 17025	Further Remarks
						yes / no	yes / no	
Vitamin B6 (als Pyridoxin) / Vitamin B6 (as Pyridoxine)	1	SOP M843, HPLC-UV		HPLC-UV	available		yes	
	2						yes	
	3	Quantitative determination of water-soluble vitamins by HPLC in food supplements			PT-Material		yes	
	4	internal method HPLC		HPLC based on VDLUFA methods III No. 13.9.1			yes	
	5							
	6	in-house method , LCMSMS				yes	no	
	7	HPLC fluo (in house method)			yes (9 calibration points + reference material)	yes	no	
	8	HPLC with FL					yes	
	9						yes	
	10							
	11			HPLC			yes	
	12							
	13							
	14	2.019/008-06	without enzym. clean-up / sample preparation	HPLC	Pyridoxin-HCl	yes	yes	-
	15	in-house method, premix, N04_22ME					yes	
	16							
	17	DIN EN 14663:2006					yes	
	18	in house method	Extraction in 0,1% H3PO4	HPLC DAD C18 column	3-5-point calibration	no	yes	
	19	HPLC	n/a	MQLTM-0153	USP	no	yes	n/a
	20						yes	
	21			HPLC/UV	external calibration	no	no	
	22	internal method HPLC-MS-MS (P4-02-01-12-2423)	enzymatic digestion, centrifugation, filtration, dilution, addition ISTD	HPLC-MS/MS	external calibration with ISTD	no	yes	recovery with NIST-SRM-3280
	23	LAV 21.0017-02; HPLC-FLD					yes	
	24							
	25	HPLC			LVU-Lippold No.17-20	no	yes	

Parameter	Partici-pant	Method description as in test report / norm / literature	Sample preparation	Measuring method	Calibration / Reference material	Recovery rate with same matrix	Method accredited ISO/IEC 17025	Further Remarks
						yes / no	yes / no	
Vitamin B12 (als Cyanocobalamin) / Vitamin B12 (as Cyanocobalamine)	1	SOP M844, HPLC-UV		HPLC-UV	available		yes	
	2						yes	
	3	R-Biopharm VitaFast Vitamin B12 P1002, 2016-10			PT-Material		yes	
	4	Vita Fast Vitamin B12 (RBiopharm)					yes	
	5	Instrukcja testu VitaFast Vitamin B12 (Cyanocobalamin) firmy R-Biopharm AG	wg instrukcji	Metoda mikrobiologiczna - mikropłytkowa z odczytem spektrofotometrycznym		no	yes	
	6	in-house method , LCMSMS				yes	no	
	7							
	8	HPLC with DAD					yes	
	9							
	10							
	11			HPLC			yes	
	12							
	13							
	14	VitaFast P1002	as per testkit	VitaFast	Cyanocobalamin	-	no	-
	15							
	16							
	17	USP 39, method 171:2016, mod.					yes	
	18	in house method	immunoaffinity column	HPLC DAD C18 column	3-5-point calibration	no	yes	
	19	HPLC	n/a	MQLTM-0107	USP	no	yes	n/a
	20						yes	
	21			HPLC/UV	external calibration	no	no	
	22	RIDASCREEN® FAST Vitamin B12 Elisa Kit (R2103)	as per testkit instructions		as per testkit instructions		no	Method not finally validated. PT participation as test.
	23							not known, because Vitamin B2 was not sufficiently separated from B12 despite IAS-clean-up
	24							
	25	Microbiological (VitaFast Vitamin B12 - Cyanocobalamin / r-biopharm Art. No. P1002)			SRM 3280	no	yes	

Parameter	Partici-pant	Method description as in test report / norm / literature	Sample preparation	Measuring method	Calibration / Re-ference material	Recovery rate with same matrix	Method ac-credited ISO/IEC 17025	Further Remarks
						yes / no	yes / no	
Biotin	1	SOP M3532, LC-MS/MS		LC-MS/MS	available		yes	
	2							
	3	R-Biopharm VitaFast Biotin P1003, 2016-10			PT-Material		yes	
	4	Vita Fast Biotin (RBiopharm)					yes	
	5	Instrukcja testu VitaFast Biotin firmy R-Biopharm AG	wg instrukcji	Metoda mikrobiologiczna - mikropłytkowa z odczytem spektrofotometrycznym		no	yes	
	6	in-house method , LCMSMS				yes	no	
	7	LC-MS/MS (in-house method)			yes (9 calibration points + reference material)	yes	yes	
	8							
	9							
	10							
	11			HPLC			yes	
	12							
	13	Elisa Demeditec					no	
	14	-	-	-	-	-	-	-
	15							
	16	VitaFast Testkit					yes	
	17	USP 21,3. suppl, method 88:1986					yes	
	18	in house method	immunoaffinity column	HPLC DAD C18 column	3-5-point calibration	no	yes	
	19	HPLC	n/a	MQLTM-0155	USP	no	yes	n/a
	20						yes	
	21			HPLC/UV	external calibration	no	no	
	22	internal method microbiology (P2-02-02-02-0393)	as per testkit instructions		as per testkit instructions		yes	Vita Fast®Vitamin B7 (P1003)
	23							
	24							
	25	Microbiological (VitaFast Vitamin B7 - Biotin / r-biopharm Art. No. P1003)			SRM 3280	no	yes	

Parameter	Partici-pant	Method description as in test report / norm / literature	Sample preparation	Measuring method	Calibration / Reference material	Recovery rate with same matrix	Method accredited ISO/IEC 17025	Further Remarks
						yes / no	yes / no	
Vitamin C (als Ascorbinsäure) / Vitamin C (as Ascorbic acid)	1	SOP M547, HPLC/FI		HPLC-FI	available		yes	
	2						yes	
	3	not determined	not determined	not determined	not determined		not determined	
	4	internes Verfahren HPLC		UV-Detector			yes	
	5	PN - EN 14130, PB-257/LF	wg metod	HPLC-UV			yes	
	6	in-house method , HPLC-DAD				no	yes	
	7							
	8	HPLC with DAD					no	
	9						yes	
	10	HPLC				yes	yes	
	11			HPLC			yes	
	12							
	13							
	14	Enzymatic 10409677035	as per kit instructions	Enzymatikc	L+Ascorbic acid	yes	yes	-
	15	AOAC 2012.22, HPLC					yes	
	16							
	17	DIN EN 14130:2003 mod.					yes	
	18	in house method	Extraction in Extraction Solution (Tween+ HCl in Water)	HPLC DAD C18 column	3-5-point calibration	no	yes	
	19	Titration	n/a	MQLTM-0149	n/a	no	yes	n/a
	20						yes	Tested upon opening
	21			HPLC/UV	external calibration	no	no	
	22							
	23	LAV 21.0052-01; HPLC-DAD					yes	
	24	In house method	Extraction with water	UPLC-MS/MS		yes	no	
	25							

Parameter	Partici-pant	Method description as in test report / norm / literature	Sample preparation	Measuring method	Calibration / Reference material	Recovery rate with same matrix	Method accredited ISO/IEC 17025	Further Remarks
						yes / no	yes / no	
Folsäure (als Pteroylmonoglutaminsäure) / Folic acid (as Pteroylmonoglutaminic acid)	1	SOP M851, HPLC-UV		HPLC-UV	available		yes	
	2						yes	
	3	Quantitative determination of water soluble vitamins by HPLC in food supplements			PT-Material		yes	
	4	Vita Fast Folsäure (RBiopharm)					yes	
	5	Instrukcja testu VitaFast Folic Acid firmy R-Biopharm AG	wg instrukcji	Metoda mikrobiologiczna - mikropłytkowa z odczytem spektrofotometrycznym		no	yes	
	6	in-house method , LCMSMS				yes	no	
	7							
	8							
	9						yes	
	10							
	11			HPLC			yes	
	12							
	13							
	14	VitaFast P1001	as per testkit	VitaFast	folic acid	-	no	-
	15							
	16							
	17	SLMB 62/11.2.2 mod.					yes	
	18	in-house method	Extraction in 0,1 M NaOH	HPLC DAD C18 column	3-5-point calibration	no	yes	
	19	HPLC	n/a	MQLTM-0150	USP	no	yes	n/a
	20						yes	Free folic acid only
	21			HPLC/UV	external calibration	no	no	
	22	internal method microbiology (P2-02-02-02-0390)	as per testkit instructions		as per testkit instructions		yes	Vita Fast®Folic acid (P1001)
	23	LAV 21.0017-02; HPLC-DAD					yes	
	24							
	25	Microbiological (VitaFast Vitamin B9 - Folic acid / r-biopharm Art. No. P1001)			SRM 3280	no	yes	

Parameter	Partici-pant	Method description as in test report / norm / literature	Sample preparation	Measuring method	Calibration / Re-ference material	Recovery rate with same matrix	Method ac-credited ISO/IEC 17025	Further Remarks
						yes / no	yes / no	
Niacin	1	SOP M843, HPLC-UV		HPLC-UV	available		yes	
	2						yes	
	3	Quantitative determination of water soluble vitamins by HPLC in food supplements			PT-Material		yes	
	4	internal method HPLC		HPLC based on VDLUFA method III No. 13.9.1			yes	Measured with Vita Fast Niacin (RBiopharm): mean 8250 mg/100g
	5	Instrukcja testu VitaFast Vitamin B3 (Niacin) firmy R-Biopharm AG	wg instrukcji	Metoda mikrobiologiczna - mikropłytkowa z odczytem spektrofotometrycznym		no	yes	
	6	in-house method , LCMSMS				yes	no	
	7	HPLC-Fluo (NF EN 15652)			yes (6 calibration points + reference material)	yes	yes	
	8	HPLC with DAD					yes	
	9						yes	
	10							
	11			HPLC			yes	
	12							
	13							
	14	2.019/016-03	Nicotinic acid + Nicotinamide	HPLC	Nicotinamide / Nicotinic acid	yes	no	-
	15	in-house method, premix, N04_22ME					yes	
	16							
	17	USP 34, method 441:2011, mod.					yes	
	18	in-house method	Extraction in 0,1 M NaOH	HPLC DAD C18 column	3-5-point calibration	no	yes	
	19	HPLC	n/a	MQLTM-0153	USP	no	yes	n/a
	20						yes	
	21			HPLC/UV	external calibration	no	no	
	22							
	23	LAV 21.0017-02; HPLC-DAD					yes	
	24							
	25	Microbiological (VitaFast Vitamin B3 - Niacin / r-biopharm Art. No. P1004)			DLA 43-2017	no	yes	

Parameter	Partici-pant	Method description as in test report / norm / literature	Sample preparation	Measuring method	Calibration / Reference material	Recovery rate with same matrix	Method accredited ISO/IEC 17025	Further Remarks
						yes / no	yes / no	
Pantothensäure / Pantothenic acid	1	SOP M856, HPLC-UV		HPLC-UV	available		yes	
	2						yes	
	3	Quantitative determination of water soluble vitamins by HPLC in food supplements			PT-Material		yes	
	4	Vita Fast Pantothenic acide (RBiopharm)					yes	
	5	Instrukcja testu VitaFast Pantothenic Acid firmy R-Biopharm AG	wg instrukcji	Metoda mikrobiologiczna - mikropłytkowa z odczytem spektrofotometrycznym		no	yes	
	6	in-house method , LCMSMS				yes	no	
	7							
	8							
	9							
	10							
	11			HPLC			yes	
	12							
	13							
	14	VitaFast P1003	as per testkit	VitaFast	Pantothenic acid	-	no	-
	15	ISO 20639:2015-11					yes	
	16							
	17	USP 39, method					yes	
	18	in-house method	Extraction in 0,1 M NaOH	HPLC DAD C18 column	3-5-point calibration	no	yes	
	19	HPLC	n/a	MQLTM-0153	USP	no	yes	n/a
	20	-	-	-	-	-	-	Not Performed
	21			HPLC/UV	external calibration	no	no	
	22	internal method Microbiology (P2-02-02-02-0391)	as per testkit instructions		as per testkit instructions		yes	VitaFast® Pantothenic acid (P1005)
	23	LAV 21.0017-02; HPLC-DAD					yes	
	24							
	25							

5.2 Homogeneity

5.2.1 Homogeneity of bottled PT-samples

Homogeneity test by determination of Niacinamide, Pantothenic acid, Vitamin B1, B2 and B6 by HPLC-DAD:

Vitamin B1

Independant samples	g/kg
1	6,33
2	6,24
3	6,21
4	6,25
5	6,19
6	6,12
7	6,16
8	6,37
9	6,35
10	6,05

General Mean	6,23	
Repeatability standard deviation	0,103	1,66%

Vitamin B2

Independant samples	g/kg
1	7,65
2	7,59
3	7,48
4	7,45
5	7,64
6	7,70
7	7,35
8	7,11
9	7,43
10	7,59

General Mean	7,50	
Repeatability standard deviation	0,176	2,34%

Vitamin B6

Independant samples	g/kg
1	7,25
2	7,32
3	7,33
4	7,26
5	7,52
6	7,27
7	7,04
8	7,14
9	7,24
10	7,19

General Mean	7,26	
Repeatability standard deviation	0,128	1,76%

Niacinamide

Independant samples	g/kg
1	81,5
2	81,3
3	82,0
4	83,4
5	81,7
6	81,7
7	83,2
8	82,4
9	81,3
10	83,6

General Mean	82,2	
Repeatability standard deviation	0,891	1,08%

Pantothenic acid

Independant samples	g/kg
1	25,5
2	25,1
3	25,0
4	25,4
5	25,7
6	25,9
7	25,3
8	25,2
9	25,2
10	25,8

General Mean	25,4	
Repeatability standard deviation	0,318	1,25%

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:

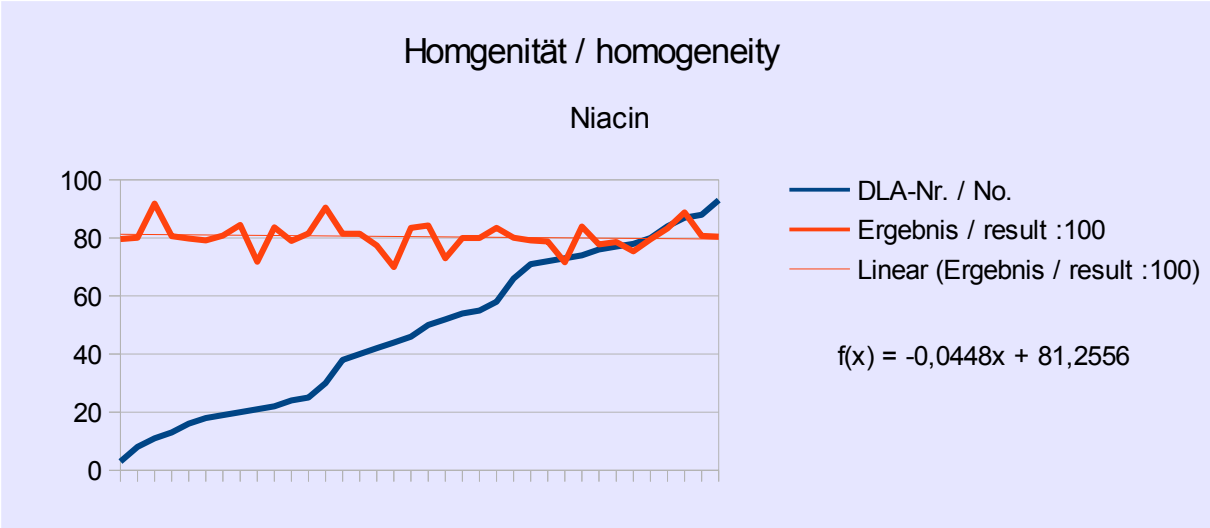


Abb./Fig. 19:
Trendfunktion Probennummern vs. Ergebnisse (1*100 dargestellt)
trend line function sample number vs. results (1*100 shown)
(ohne Ausreißer / without outliers)

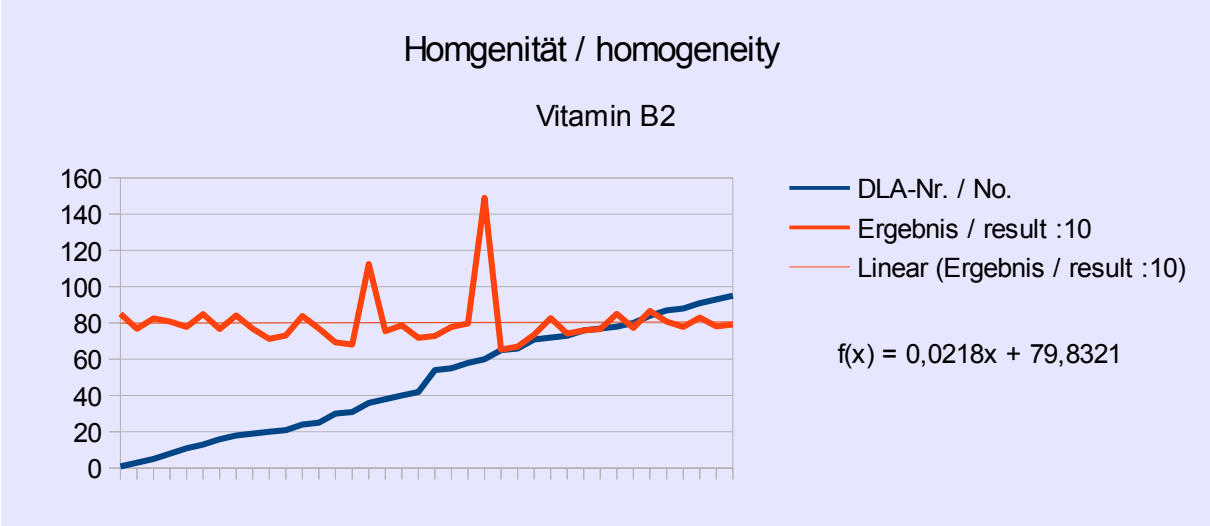


Abb./Fig. 20:
Trendfunktion Probennummern vs. Ergebnisse (1*10 dargestellt)
trend line function sample number vs. results (1*10 shown)
(ohne Ausreißer / without outliers)

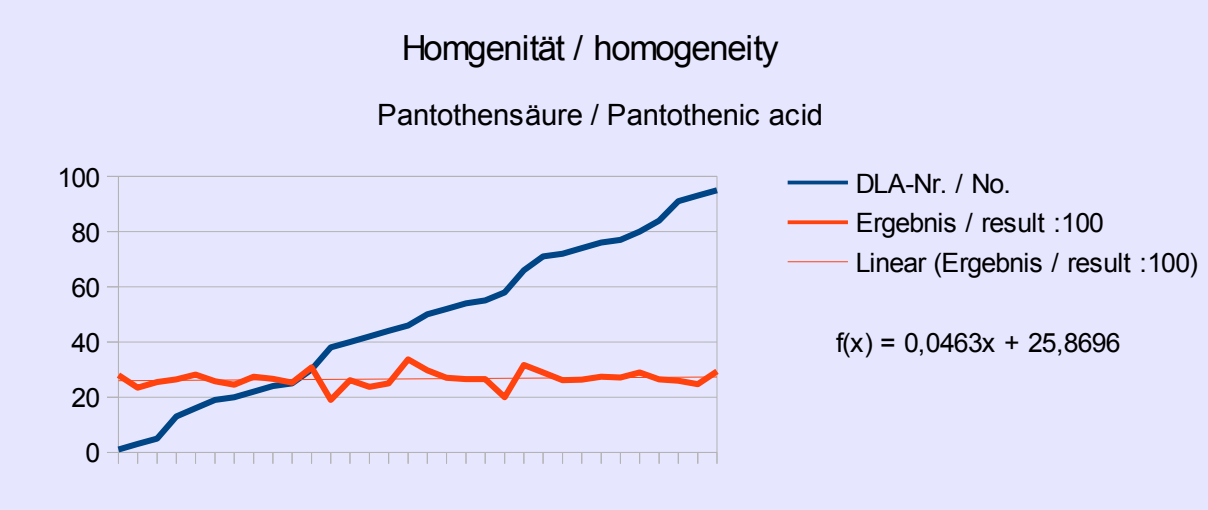


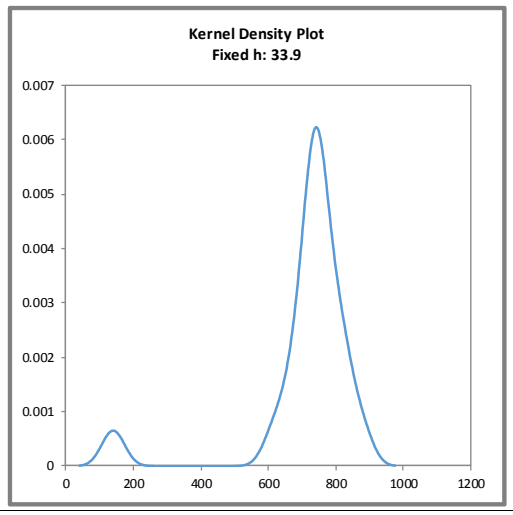
Abb./Fig. 21:
Trendfunktion Probennummern vs. Ergebnisse (1*100 dargestellt)
trend line function sample number vs. results (1*100 shown)

5.3 Kernel Density Plots of Results

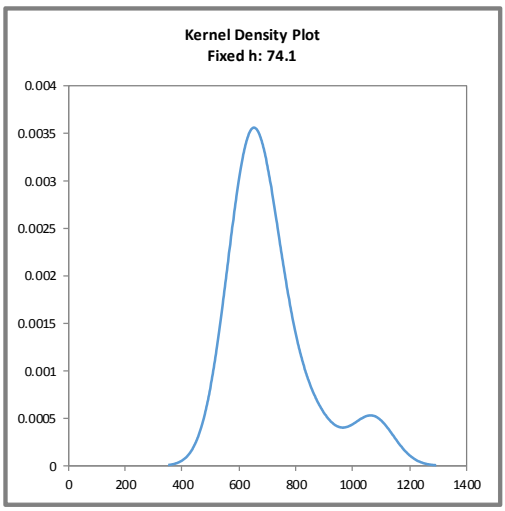
Abbildungen:
Kerndichte-Schätzungen
der Teilnehmerergebnisse
(mit h = 0,75 x σ_{pt} von Xpt)

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

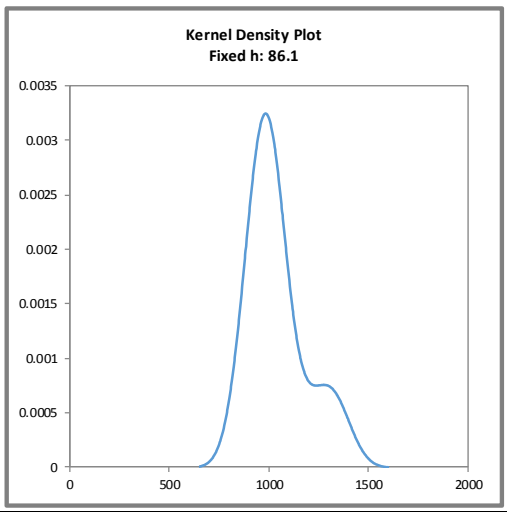
Vitamin B6



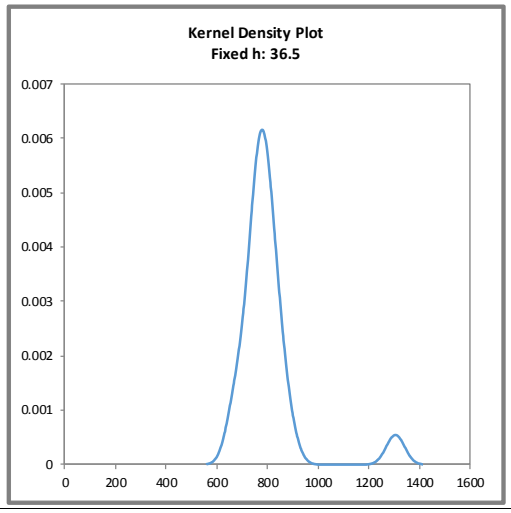
Vitamin B1



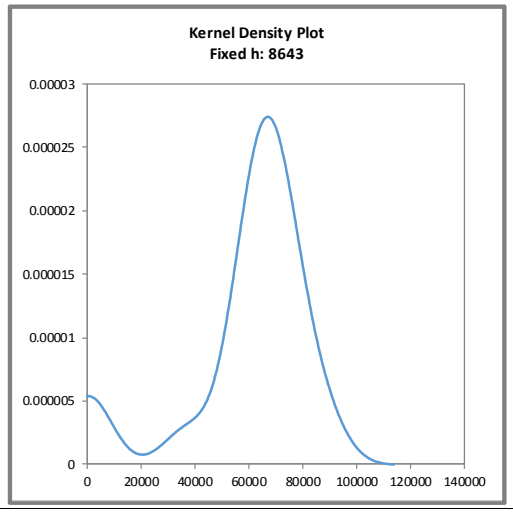
Vitamin B12



Vitamin B2



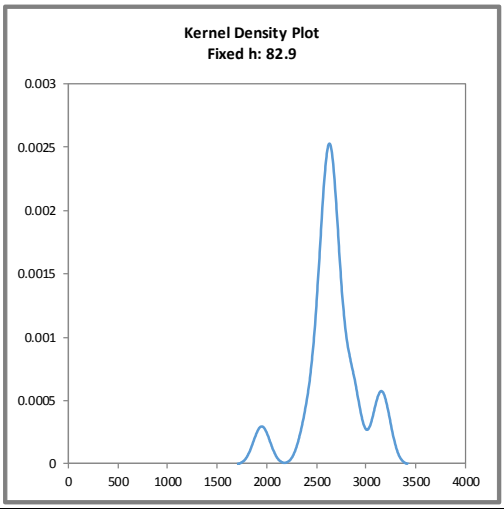
Biotin



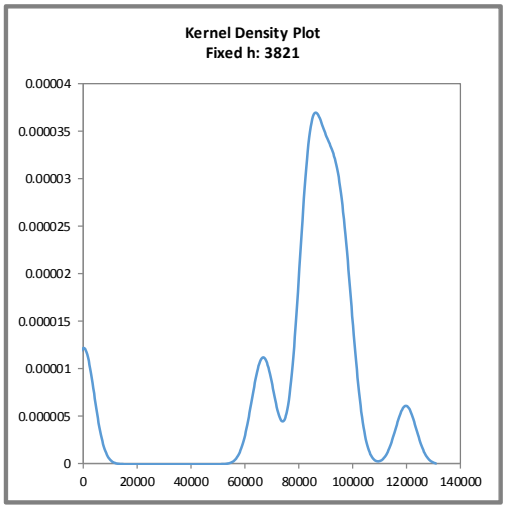
Abbildungen:
Kerndichte-Schätzungen
der Teilnehmerergebnisse
(mit h = 0,75 x σ_{pt} von X_{pt})

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

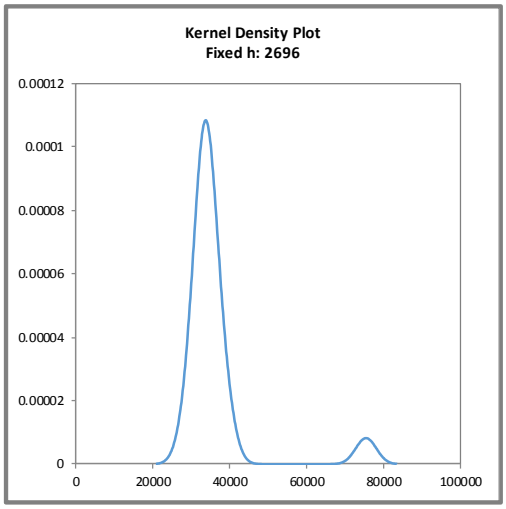
Pantothensäure / Pantothenic Acid



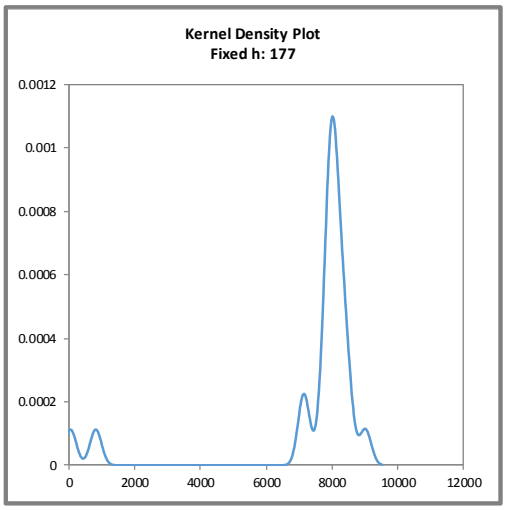
Folsäure / Folic Acid



Vitamin C



Niacin



5.4 Information on the Proficiency Test (PT)

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

PT number	***DLA 46 - 2019***
PT name	***Food Supplement I: Vitamins B1, B2, B6, B12, Biotin, Vitamin C, Folic Acid , Niacin and Pantothenic Acid***
*Sample matrix**	*Samples I + II: Multi-vitamin capsule powder (without capsule shell) / ingredients: maltodextrin, vitamins and carrier: mannitol*
Number of samples and sample amount	*2 identical samples I + II, 50 g each.*
Storage	*Samples I + II: cooled 2 - 10°C*
Intentional use	*Laboratory use only (quality control samples)*
Parameter	*quantitative: Vitamins B1, B2, B6, B12, Biotin, Vitamin C, Folic Acid, Niacin and Pantothenic Acid* *Contents: The contents are of the order of the nutrient reference values per recommended daily dose (1-3 capsules approx. 0.2 - 1 g)*
Methods of analysis	*Analytical methods are optional*
Notes to analysis	*The analysis of PT samples should be performed like a routine laboratory analysis.* *In general we recommend to homogenize a representative sample amount before analysis according to good laboratory practice, especially in case of low sample weights.*
Result sheet	*The results for sample I and II as well as the final results calculated as mean of the double determination (samples I and II) should be filled in the result submission file as the sum of vitamin equivalents. The recovery rates, if carried out, has to be included in the calculation.*
Units	*mg/100 g and µg/100 g , respectively (see results file)*
Number of significant digits	*at least 2*
Further information	*For information please specify:* – *Date of analysis* – *DLA-sample-numbers (for sample I and II)* – *Limit of detection* – *Assignment incl. Recovery* – *Recovery with the same matrix* – *Method is accredited*
Result submission	*The result submission file should be sent by e-mail to:* ***pt@dla-lvu.de***
Deadline	***the latest 21st June 2019***
Evaluation report	*The evaluation report is expected to be completed 6 weeks after deadline of result submission and sent as PDF file by e-mail.*
Coordinator and contact person of PT	*Matthias Besler-Scharf PhD*

* 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
		AUSTRIA
		FRANCE
		CZECH REPULIC
		GREAT BRITAIN
		Germany
		Germany
		Germany
		Germany
		Germany
		Germany
		Germany
		Germany
		BELGIUM
		Germany
		Germany
		POLAND
		BELGIUM
		Germany
		Germany
		Germany
		USA
		Germany
		Germany
		CROATIA
		Germany

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

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

7. Index of references

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
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18. ASU §64 LFGB: L 00.00-83 / EN 14122:2014 Bestimmung von Vitamin B1 in Lebensmitteln mit Hochleistungs-Flüssigchromatographie, Juni 2015 / Foodstuffs - Determination of vitamin B1 by high performance liquid chromatography
19. ASU §64 LFGB: L 00.00-84 / EN 14152:2014 Bestimmung von Vitamin B2 in Lebensmitteln mit Hochleistungs-Flüssigchromatographie, Juni 2015 / Foodstuffs - Determination of vitamin B2 by high performance liquid chromatography
20. ASU §64 LFGB: L 00.00-97 / EN 14663:2006 Bestimmung von Vitamin B6 (einschlieslich glucosidisch gebundener Verbindungen) in Lebensmitteln HPLC-Verfahren, Dezember 2006 / Foodstuffs - Determination of vitamin B6 (including its glycosylated forms) by HPLC
21. ASU §64 LFGB: L 00.00-130 / EN 14164:2014 Bestimmung von Vitamin B6 in Lebensmitteln mit Hochleistungs-Flüssigchromatographie, Juni 2015 / Foodstuffs - Determination of vitamin B6 by high performance liquid chromatography
22. ASU §64 LFGB: L 00.00-87 / EN 14131:2003 Mikrobiologische Bestimmung von Folat, Juli 2004 / Foodstuffs - Determination of folate by microbiological assay
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