



Evaluation Report

proficiency test

DLA ptSU01 (2021)

Food Supplements I:

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

in Multivitamin-Powder

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<i>Vertraulichkeit Confidentiality</i>	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 metalised 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
<p><u>Ingredients:</u> 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).</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 vitamins according to the manufacturers specification

Vitamin	Content per 100 g	
Vitamin B1	500	mg
Vitamin B2	560	mg
Vitamin B6	530	mg
Vitamin B12	730	µg
Biotin	50000	µg
Vitamin C	15700	mg
Folic acid	66000	µg
Niacin	6100	mg
Pantothenic acid	1850	mg

2.1.1 Homogeneity

The **mixture homogeneity before bottling** was examined 10-fold by determination of the parameters Niacinamide, Pantothenic acid, Vitamin B1, B2, and B6 by HPLC-DAD. The repeatability standard deviations were with 0,97 - 2,26% 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 < 6,6% (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) [16-26].

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	4,10 %
Vitamin B2	2,72 %
Vitamin B6	2,89 %
Vitamin B12	4,02 %
Biotin	4,64 %
Vitamin C	1,76 %
Folic acid	2,99 %
Niacin	1,43 %
Pantothenic acid	6,55 %

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. 0,17 (18,4°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 17th week of 2021. The testing method was optional. The tests should be finished at 25th June 2021 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.*

Note: Please store samples at 2 - 10°C on arrival!

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 24 participants submitted at least one result.

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 $> 25 \text{ mg/kg}$ or $< 2,5 \text{ mg/kg}$) 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 an precision experiment is derived from collaborative studies with specified analytical methods.

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

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

For valuation of all following parameters in the present PT the target standard deviation according to the general model of Horwitz was applied (see 3.6.1): Folic Acid and Pantothenic Acid.

The target standard deviation of the evaluation by precision experiment (s. 3.6.2) was considered for the following parameters: (ASU S64 / EN-Norms / AOAC) [18, 19, 21, 22, 25, 32]: Biotin, Niacin, Vitamin B1, B2, B6, B12 and Vitamin C.

Additionally for Vitamin B2, B6, Folic Acid, Niacin and 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]:

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

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, 32]

Parameter	Matrix	Mean	RSD _r	RSD _R	σ _{pt}	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
Vitamin B12	Food supplement (Adult nutritional RTF)	1,54 µg/100g	6,50%	23,4%	22,9% ¹	HPLC [32] AOAC 2014.02
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 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}). 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*/opt	DLA-report
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 B1	Multivitamin-capsule powder	529 mg/100g	103 mg/100g	19,50%	1,4	DLA ptsU01 (2021)
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 B2	Multivitamin-capsule powder	600 mg/100g	78,0 mg/100g	13,00%	1,9*	DLA ptsU01 (2021)
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 B6	Multivitamin-capsule powder	560 mg/100g	68,9 mg/100g	12,30%	1,8*	DLA ptsU01 (2021)
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
Vitamin B12	Multivitamin-capsule powder	746 µg/100g	217 µg/100g	29,10%	1,3	DLA ptsU01 (2021)
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
Biotin	Multivitamin-capsule powder	49562 µg/100g	6902 µg/100g	13,9%	0,81	DLA ptsU01 (2021)

Continuation next page

Continuation Table 5:

Parameter	Matrix (Powder)	robust Mean	rob. SD (S*)	rel. SD (CV_{S*}) [%]	Quotient S*/σ_{pt}	DLA-report
Folic acid	Multivitamin-in-capsule powder	226000 µg/100g	39900 µg/100g	17,6%	1,1	DLA 43/2017
Folic acid	Multivitamin-in-capsule powder	88412 µg/100g	9691 µg/100g	11,0%	1,9	DLA 46/2019
Folic acid	Multivitamin-in-capsule powder	61059 µg/100g	8488 µg/100g	13,9%	1,9*	DLA ptsU01 (2021)
Niacin	Multivitamin-in-capsule powder	14400 mg/100g	1150 mg/100g	7,98&	1,9	DLA 43/2017
Niacin	Multivitamin-in-capsule powder	8062 mg/100g	324 mg/100g	4,02%	1,4	DLA 46/2019
Niacin	Multivitamin-in-capsule powder	6051 mg/100g	649 mg/100g	10,7%	2,2*	DLA ptsU01 (2021)
Pantothenic acid	Multivitamin-in-capsule powder	7100 mg/100g	1040 mg/100g	14,6%	2,9*	DLA 43/2017
Pantothenic acid	Multivitamin-in-capsule powder	2667 mg/100g	196 mg/100g	7,35%	1,8*	DLA 46/2019
Pantothenic acid	Multivitamin-in-capsule powder	1947 mg/100g	158 mg/100g	8,12%	1,8*	DLA ptsU01 (2021)
Vitamin C	Multivitamin-in-capsule powder	21200 mg/100g	839 mg/100g	3,96%	1,6	DLA 43/2017
Vitamin C	Multivitamin-in-capsule powder	34257 mg/100g	2644 mg/100g	7,72%	0,74	DLA 46/2019
Vitamin C	Multivitamin-in-capsule powder	15051 mg/100g	1282 mg/100g	8,52%	0,81	DLA ptsU01 (2021)

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

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

If $U(x_{pt}) \leq 0,3 \sigma_{opt}$ 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 / AOAC). The evaluation according to the general model of Horwitz was preferred as long as the quotient S^*/σ_{opt} was in the range of $\leq 2,0$. For all other parameters the target standard deviation from data by precision experiments was used, if available.

For vitamin B2, B6, folic acid, niacin and pantothenic acid the distribution of results showed increased variabilities with quotients above 2,0. Therefore these parameters were evaluated by z'-scores considering the standard uncertainty. Then the quotients S^*/σ_{opt}' were with the exception of niacin below 2,0 (s. Tab. 4).

For all other parameters the distribution showed a normal variability of results. The quotients S^*/σ_{opt} were in the range of 0,81 to 1,4 (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.

70% to 90% of results were in the respective target range.

The robust means of the participant results were for all parameters in the range of 93% to 107% 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	Parameter [Einheit / Unit]	Abweichung	z-Score σ_{pt}	z-Score (Info)	Hinweis
		Deviation			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	24
Number of outliers	-
Mean	571
Median	509
Robust Mean (\bar{X})	529
Robust standard deviation (S^*)	103
Number with 2 replicates	21
Repeatability SD (S_r)	21,6
Repeatability (CV_r)	4,10%
Reproducibility SD (S_R)	104
Reproducibility (CV_R)	19,7%
<i>Target range:</i>	
Target standard deviation σ_{opt}	75,8
Target standard deviation (for Information)	23,3
lower limit of target range	378
upper limit of target range	681
Quotient S^*/σ_{opt}	1,4
Standard uncertainty $U(X_{opt})$	26,3
Results in the target range	21
Percent in the target range	88%

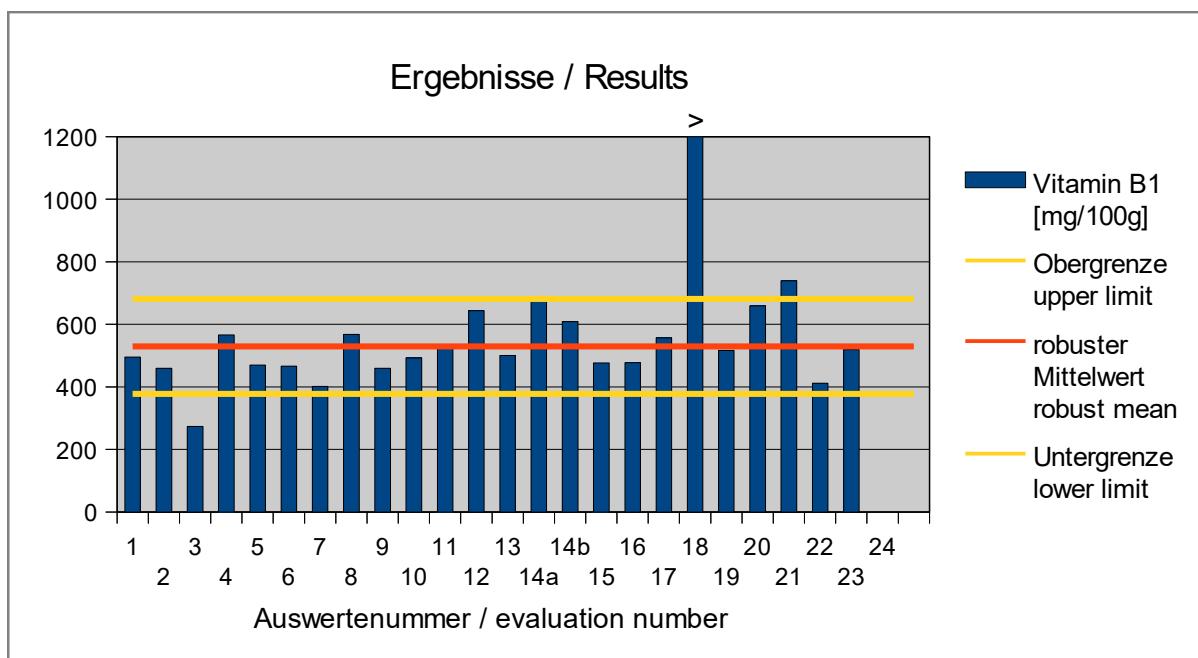


Abb. / Fig. 1: Ergebnisse Vitamin B1 / Results Vitamin B1

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte- nummer Evaluation number	Vitamin B1 [mg/100g]	Abweichung [mg/100g]	z-Score (σpt)	z-Score (Info)	Hinweis
		Deviation [mg/100g]			Remark
1	495	-34	-0,45	-1,5	
2	460	-69	-0,91	-3,0	
3	274	-256	-3,4	-11	
4	566	37	0,48	1,6	
5	470	-59	-0,78	-2,5	
6	466	-63	-0,83	-2,7	
7	402	-128	-1,7	-5,5	
8	568	39	0,51	1,7	
9	460	-69	-0,91	-3,0	
10	493	-36	-0,48	-1,6	
11	528 *	-1	-0,02	-0,06	
12	644	115	1,5	4,9	
13	501	-29	-0,38	-1,2	
14a	680	150	2,0	6,5	
14b	609	80	1,1	3,4	
15	477	-52	-0,69	-2,2	
16	478	-51	-0,68	-2,2	
17	557	28	0,37	1,2	
18	1720	1191	16	51	
19	517	-12	-0,16	-0,53	
20	659	130	1,7	5,6	
21	739	210	2,8	9,0	
22	412	-117	-1,5	-5,0	
23	519	-10	-0,14	-0,44	
24					

* Mean calculated by DLA

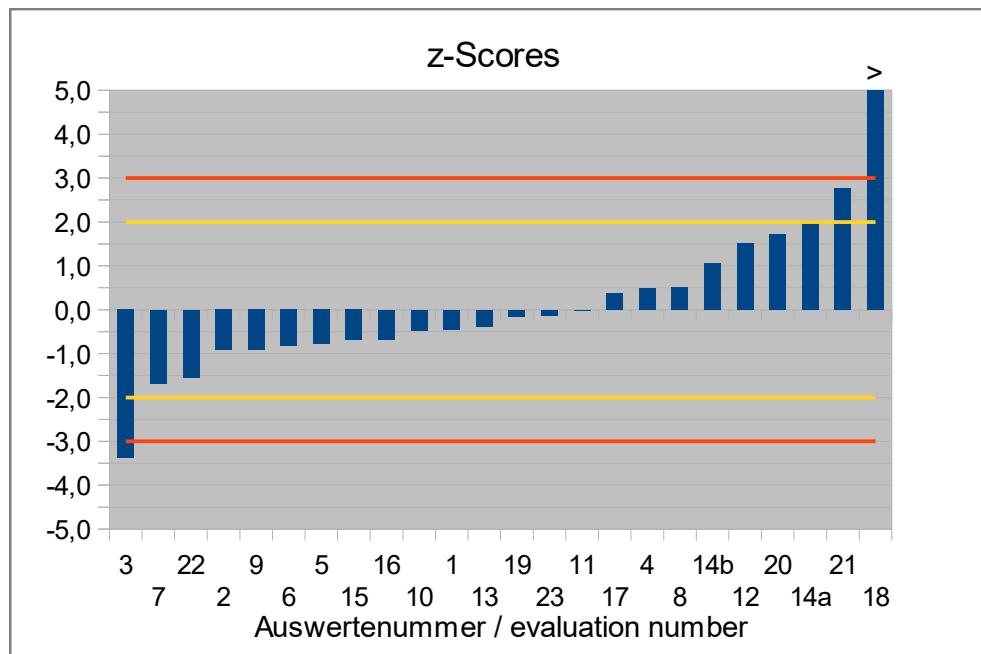


Abb. / Fig. 2: z-Scores Vitamin B1

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

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	25
Number of outliers	-
Mean	617
Median	590
Robust Mean (\bar{X})	600
Robust standard deviation (S^*)	78,0
Number with 2 replicates	22
Repeatability SD (S_r)	16,4
Repeatability (CV_r)	2,72%
Reproducibility SD (S_R)	70,2
Reproducibility (CV_R)	11,6%
Target range:	
Target standard deviation $\sigma_{opt'}$	42,1
Target standard deviation (for Information)	25,9
lower limit of target range	515
upper limit of target range	684
Quotient $S^*/\sigma_{opt'}$	1,9
Standard uncertainty $U(X_{opt})$	19,5
Results in the target range	20
Percent in the target range	80%

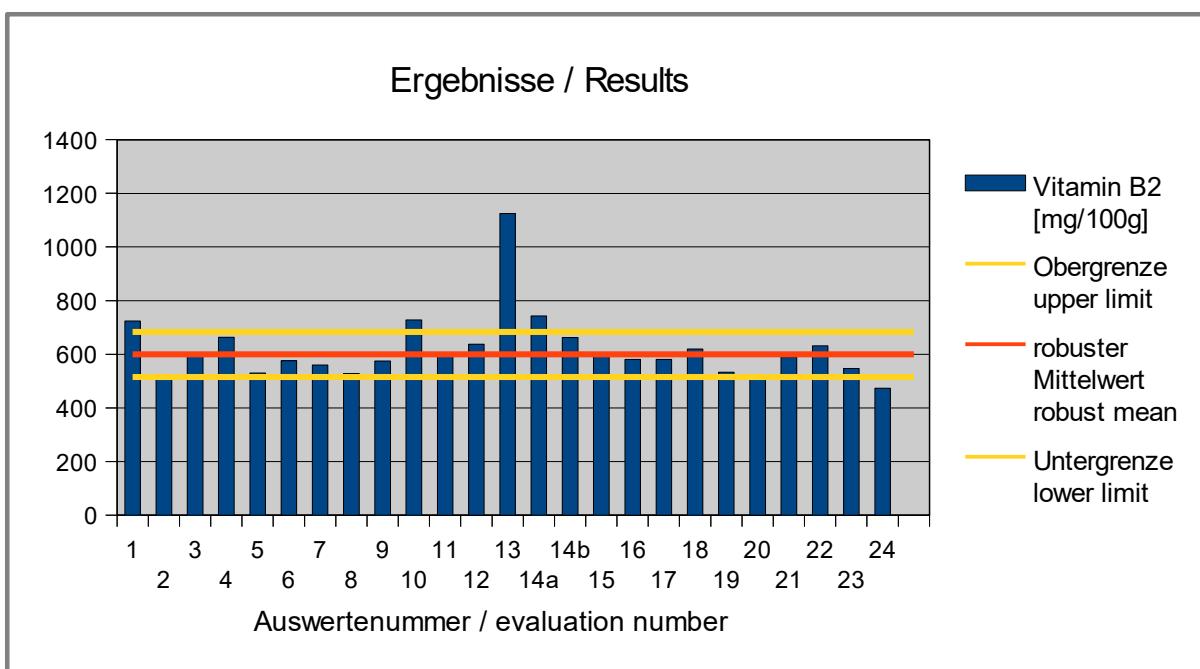


Abb. / Fig. 3: Ergebnisse Vitamin B2 / Results Vitamin B2

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte- nummer Evaluation number	Vitamin B2 [mg/100g]	Abweichung [mg/100g]	z'-Score	z-Score	Hinweis
		Deviation [mg/100g]	(σpt)	(Info)	Remark
1	724	124,5	3,0	4,8	
2	515	-84,5	-2,0	-3,3	
3	594	-6,0	-0,14	-0,23	
4	664	64,5	1,5	2,5	
5	530	-69,5	-1,7	-2,7	
6	576	-23,5	-0,56	-0,91	
7	560	-39,7	-0,94	-1,5	
8	528	-71,4	-1,7	-2,8	
9	575	*	-24,5	-0,58	-0,95
10	728	128,5	3,1	5,0	
11	595	-4,5	-0,11	-0,17	
12	638	38,5	0,91	1,5	
13	1125	525,2	12	20	
14a	743	143,6	3,4	5,5	
14b	662	62,5	1,5	2,4	
15	598	-1,5	-0,04	-0,06	
16	581	-18,5	-0,44	-0,72	
17	581	-18,5	-0,44	-0,72	
18	620	20,5	0,49	0,79	
19	533	-66,5	-1,6	-2,6	
20	520	-79,5	-1,9	-3,1	
21	590	*	-9,1	-0,22	-0,35
22	632	32,5	0,77	1,3	
23	548	-52,0	-1,2	-2,0	
24	473	-126,5	-3,0	-4,9	

* Mean calculated by DLA

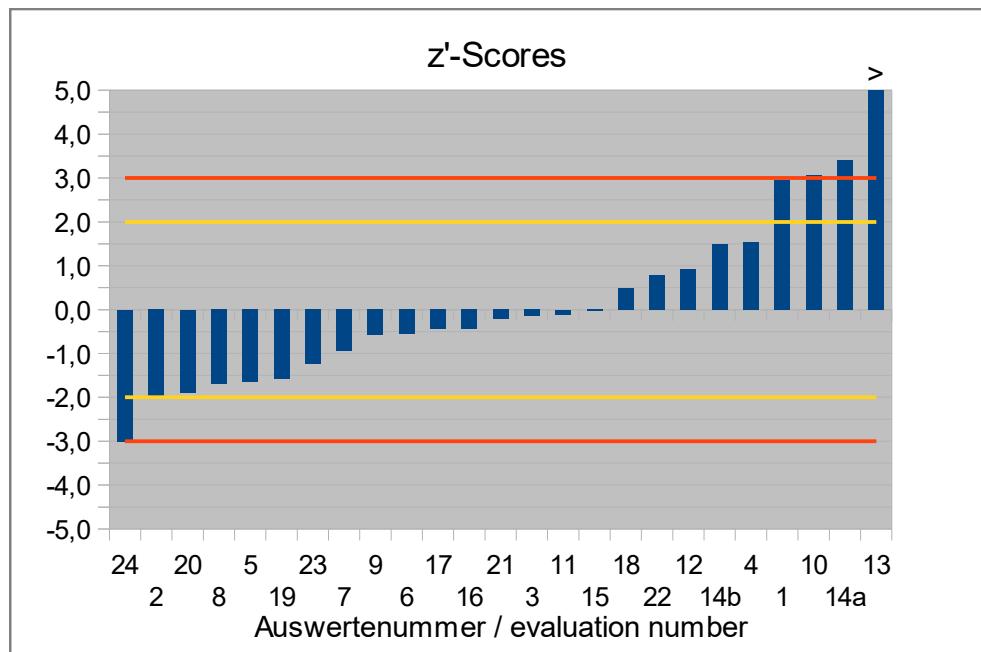


Abb. / Fig. 4: z'-Scores Vitamin B2

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

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
<i>Number of results</i>	25
<i>Number of outliers</i>	-
Mean	643
Median	553
Robust Mean (X_{pt})	560
Robust standard deviation (S^*)	68,9
<i>Number with 2 replicates</i>	20
Repeatability SD (S_r)	16,3
Repeatability (CV _r)	2,89%
Reproducibility SD (S_R)	64,9
Reproducibility (CV _R)	11,5%
<i>Target range:</i>	
Target standard deviation σ_{pt}'	37,9
Target standard deviation (for Information)	24,4
lower limit of target range	484
upper limit of target range	636
<i>Quotient S^*/σ_{pt}'</i>	1,8
<i>Standard uncertainty $U(X_{pt})$</i>	17,2
<i>Results in the target range</i>	18
<i>Percent in the target range</i>	72%

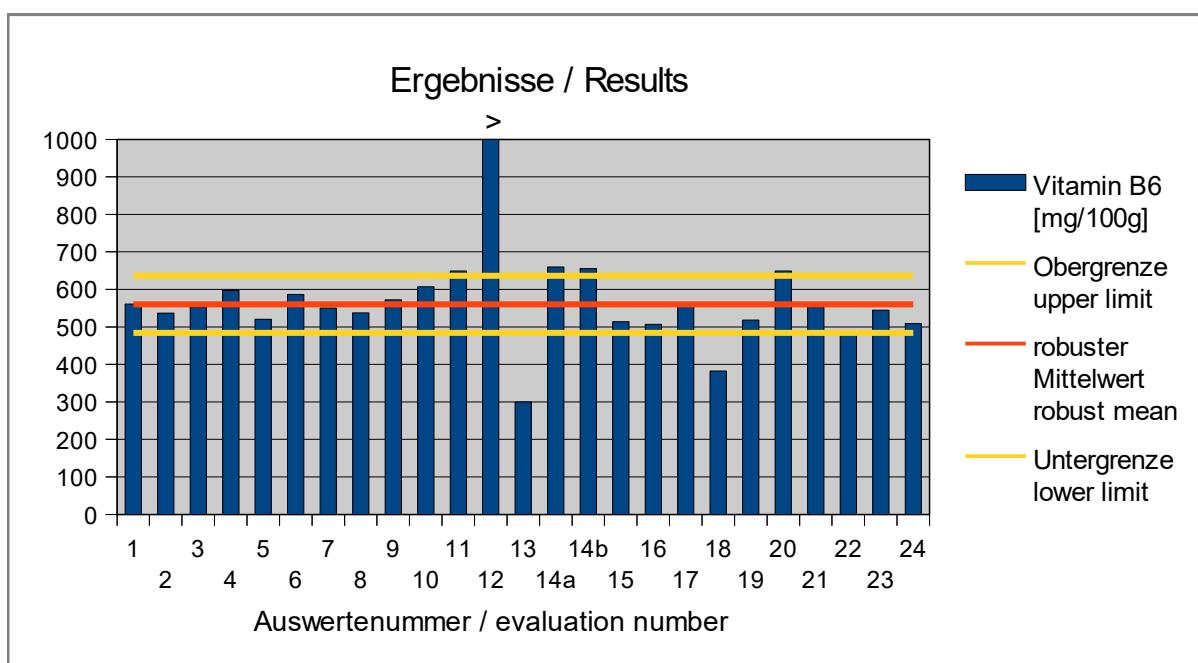


Abb. / Fig. 5: Ergebnisse Vitamin B6 / Results Vitamin B6

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte- nummer Evaluation number	Vitamin B6 [mg/100g]	Abweichung [mg/100g]	z'-Score (σpt)	z-Score (Info)	Hinweis
		Deviation [mg/100g]			Remark
1	561	0,8	0,02	0,03	
2	537	-23,2	-0,61	-0,95	
3	553	-7,2	-0,19	-0,29	
4	597	36,8	0,97	1,5	
5	520	-40,2	-1,1	-1,6	
6	586	26,2	0,69	1,1	
7	550	-10,1	-0,27	-0,41	
8	537	-23,0	-0,61	-0,94	
9	573 *	12,3	0,32	0,50	
10	607	46,8	1,2	1,9	
11	649	88,8	2,3	3,6	
12	2970	2409,8	64	99	
13	300	-260,3	-6,9	-11	
14a	659	99,3	2,6	4,1	
14b	656	95,4	2,5	3,9	
15	514	-46,2	-1,2	-1,9	
16	507	-53,2	-1,4	-2,2	
17	557	-3,2	-0,08	-0,13	
18	382	-178,2	-4,7	-7,3	
19	518	-42,2	-1,1	-1,7	
20	649	88,8	2,3	3,6	
21	554 *	-6,5	-0,17	-0,27	
22	490	-70,2	-1,9	-2,9	
23	545	-15,7	-0,41	-0,64	
24	509	-51,2	-1,4	-2,1	

* Mean calculated by DLA

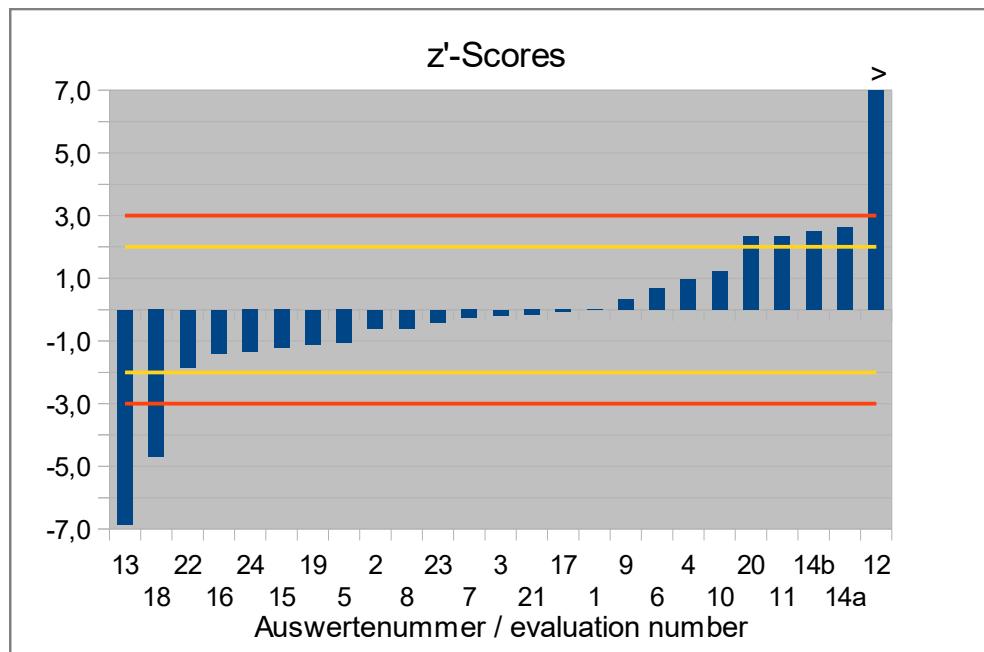


Abb. / Fig. 6: z'-Scores Vitamin B6

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

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results °	16
Number of outliers	1
Mean	770
Median	758
Robust Mean (x_{pt})	746
Robust standard deviation (S*)	217
Number with 2 replicates	15
Repeatability SD (S_r)	28,6
Repeatability (CV_r)	4,02%
Reproducibility SD (S_R)	205
Reproducibility (CV_R)	28,8%
Target range:	
Target standard deviation opt	171
lower limit of target range	404
upper limit of target range	1088
Quotient S^*/σ_{opt}	1,3
Standard uncertainty $U(x_{pt})$	67,9
Results in the target range	14
Percent in the target range	88%

° without outliers (result no. 13)

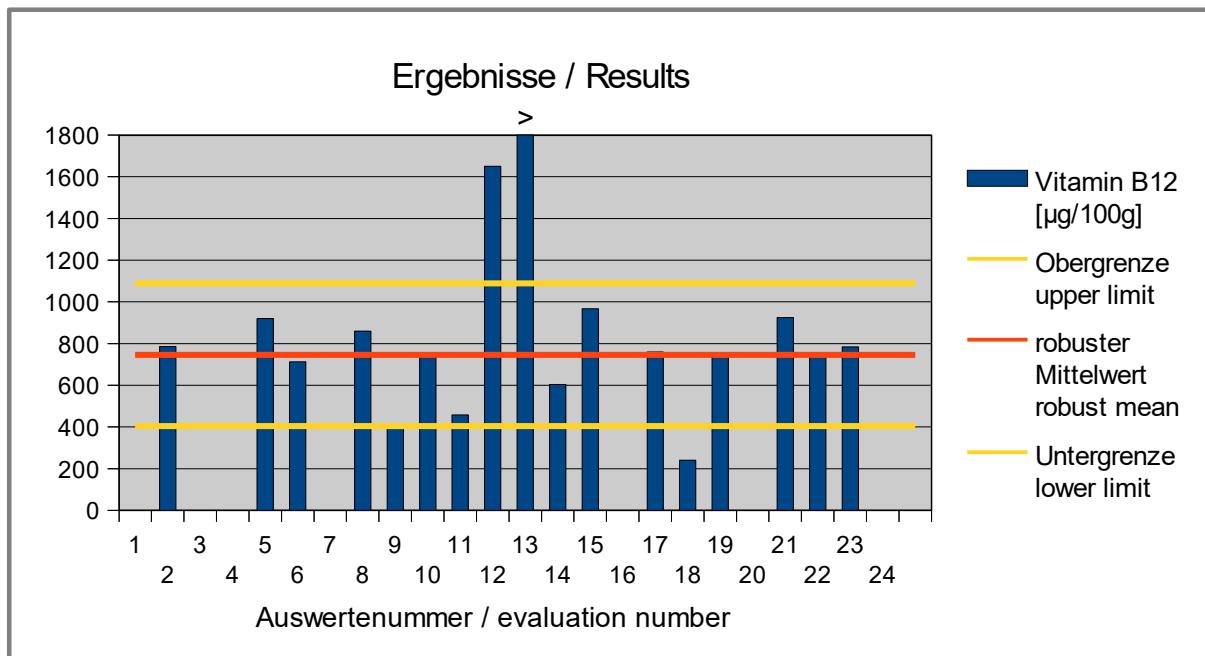


Abb. / Fig. 7: Ergebnisse Vitamin B12 / Results Vitamin B12

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-number Evaluation number	Vitamin B12 [µg/100g]	Abweichung [µg/100g] Deviation [µg/100g]	z-Score (σpt)	Hinweis Remark
1				
2	786	40	0,23	
3				
4				
5	920	174	1,0	
6	713	-33	-0,20	
7				
8	860	114	0,66	
9	413	-334	-1,9	
10	747	1	0,01	
11	458	-288	-1,7	
12	1650	904	5,3	
13	141431			Ausreißer ausgeschlossen / Outlier excluded
14	604	-142	-0,83	
15	968	222	1,3	
16				
17	760	14	0,08	
18	240	-506	-3,0	
19	744	-2	-0,01	
20				
21	925	179	1,0	
22	756	10	0,06	
23	784	38	0,22	
24				

* Mean calculated by DLA

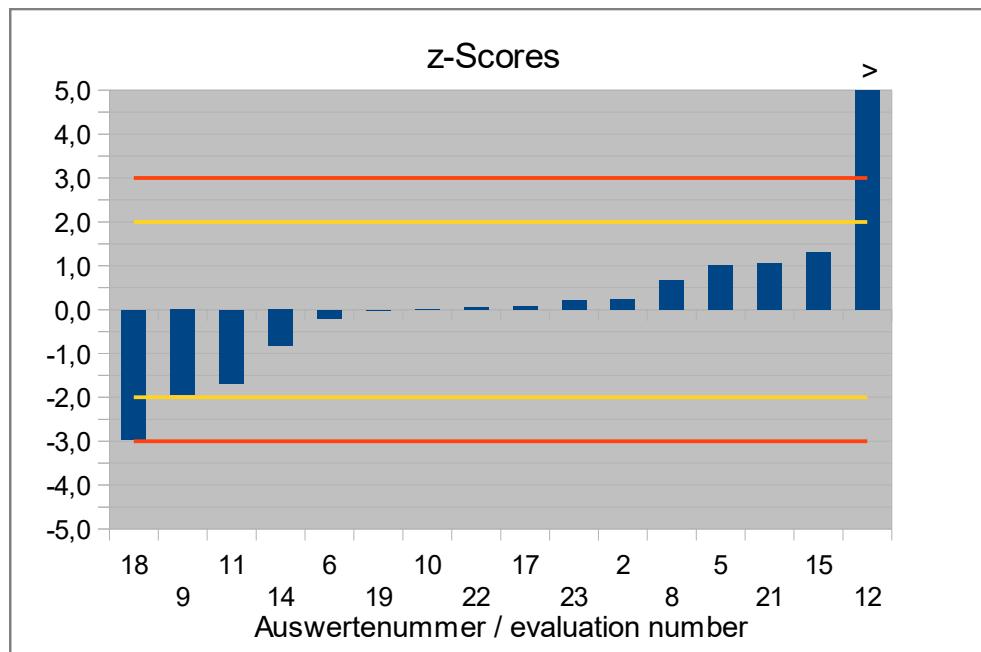


Abb. / Fig. 8: z-Scores Vitamin B12

4.5 Biotin (in µg/100g)

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
<i>Number of results</i>	17
<i>Number of outliers</i>	0
Mean	49636
Median	48950
Robust Mean (x_{pt})	49562
Robust standard deviation (S^*)	6902
<i>Number with 2 replicates</i>	16
Repeatability SD (S_r)	2309
Repeatability (CV_r)	4,64%
Reproducibility SD (S_R)	8651
Reproducibility (CV_R)	17,4%
<i>Target range:</i>	
Target standard deviation σ_{opt}	8478
Target standard deviation (for Information)	3116
lower limit of target range	32606
upper limit of target range	66519
Quotient S^*/σ_{opt}	0,81
Standard uncertainty $U(x_{pt})$	2093
<i>Results in the target range</i>	15
<i>Percent in the target range</i>	88%

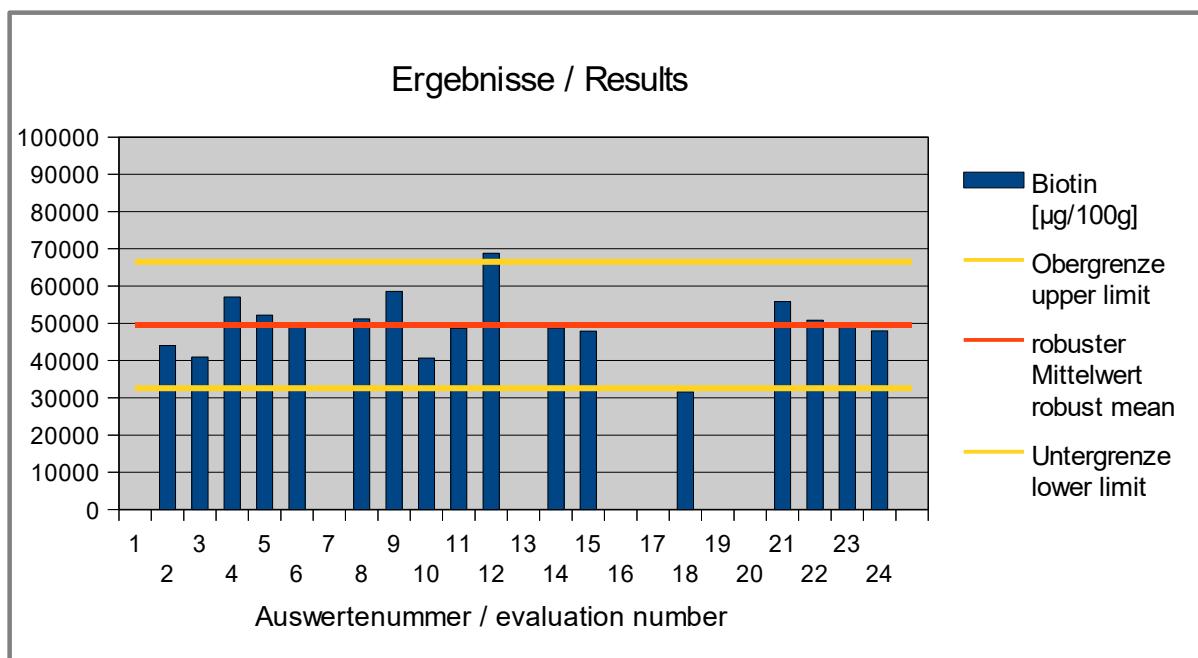


Abb. / Fig. 9: Ergebnisse Biotin / Results Biotin

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte- nummer Evaluation number	Biotin [µg/100g]	Abweichung [mg/100g]	z-Score (σpt)	z-Score (Info)	Hinweis
		Deviation [mg/100g]			Remark
1					
2	44000	-5562	-0,66	-1,8	
3	40950	-8612	-1,0	-2,8	
4	57050	7488	0,88	2,4	
5	52200	2638	0,31	0,85	
6	48950	-612	-0,07	-0,20	
7					
8	51185	1623	0,19	0,52	
9	58551 *	8989	1,1	2,9	
10	40700	-8862	-1,0	-2,8	
11	48675	-887	-0,10	-0,28	
12	68800	19238	2,3	6,2	
13					
14	48750	-812	-0,10	-0,26	
15	47932	-1630	-0,19	-0,52	
16					
17					
18	31583	-17979	-2,1	-5,8	
19					
20					
21	55863 *	6301	0,74	2,0	
22	50817	1255	0,15	0,40	
23	49813	251	0,03	0,08	
24	48000	-1562	-0,18	-0,50	

* Mean calculated by DLA

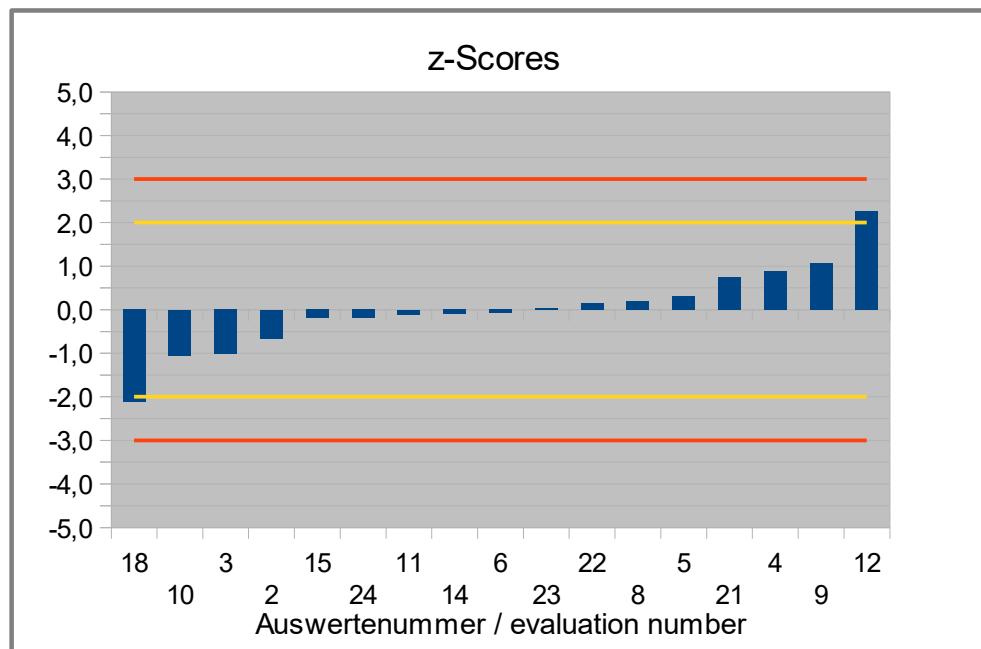


Abb. / Fig. 10: z-Scores Biotin

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

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
<i>Number of results</i>	20
<i>Number of outliers</i>	-
Mean	16316
Median	15205
Robust Mean (x_{pt})	15051
Robust standard deviation (S^*)	1282
<i>Number with 2 replicates</i>	16
Repeatability SD (S_r)	263
Repeatability (CV_r)	1,76%
Reproducibility SD (S_R)	1071
Reproducibility (CV_R)	7,17%
<i>Target range:</i>	
Target standard deviation σ_{pt}	1579
Target standard deviation (for Information)	400
lower limit of target range	11892
upper limit of target range	18210
Quotient S^*/σ_{pt}	0,81
<i>Standard uncertainty $U(x_{pt})$</i>	358
<i>Results in the target range</i>	18
<i>Percent in the target range</i>	90%

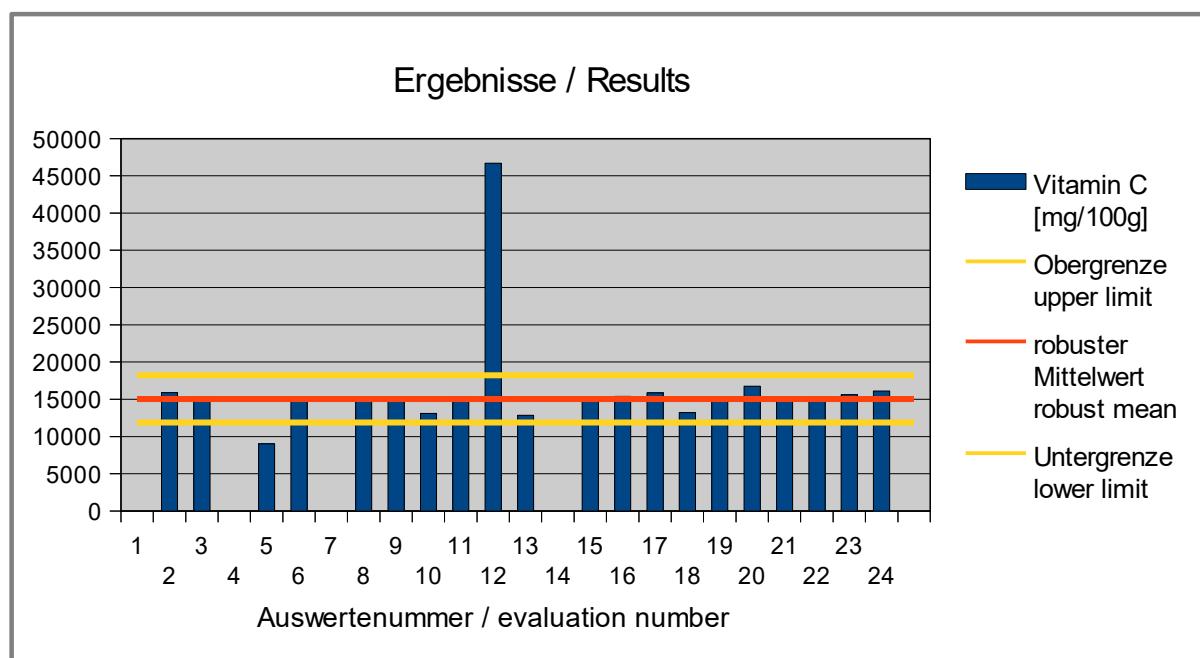


Abb. / Fig. 11: Ergebnisse Vitamin C / Results Vitamin C

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte- nummer Evaluation number	Vitamin C [mg/100g]	Abweichung [mg/100g]	z-Score (σpt)	z-Score (Info)	Hinweis
		Deviation [mg/100g]			Remark
1					
2	15900	849	0,54	2,1	
3	15300	249	0,16	0,62	
4					
5	9000	-6051	-3,8	-15,1	
6	15141	90	0,06	0,22	
7					
8	14845	-206	-0,13	-0,51	
9	15008 *	-43	-0,03	-0,11	
10	13100	-1951	-1,2	-4,9	
11	15347	296	0,19	0,74	
12	46700	31649	20	79	
13	12863	-2188	-1,4	-5,5	
14					
15	15012	-39	-0,02	-0,10	
16	15400	349	0,22	0,87	
17	15850	799	0,51	2,0	
18	13193	-1858	-1,2	-4,6	
19	15120	69	0,04	0,17	
20	16741	1690	1,1	4,2	
21	14835 *	-216	-0,14	-0,54	
22	15269	218	0,14	0,54	
23	15593	542	0,34	1,35	
24	16107	1056	0,67	2,6	

* Mean calculated by DLA

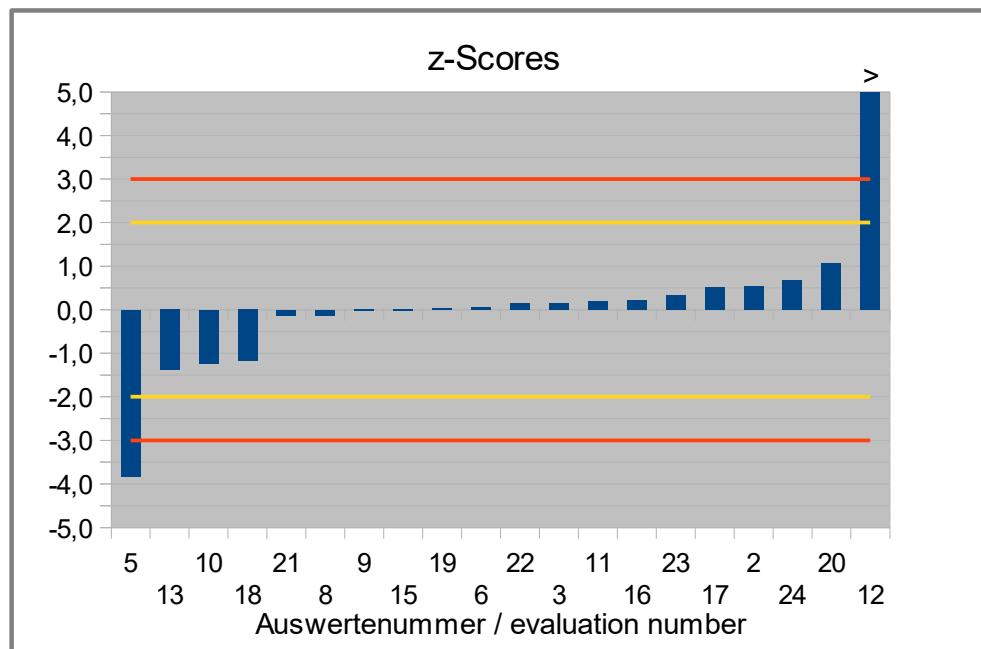


Abb. / Fig. 12: z-Scores Vitamin C

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

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	17
Number of outliers	3
Mean	62610
Median	60500
Robust Mean (X_{pt})	61059
Robust standard deviation (S^*)	8488
Number with 2 replicates	15
Repeatability SD (S_r)	1865
Repeatability (CV_r)	2,99%
Reproducibility SD (S_R)	17362
Reproducibility (CV_R)	27,8%
<i>Target range:</i>	
Target standard deviation $\sigma_{opt'}$	4524
Target standard deviation (for Information)	3720
lower limit of target range	52012
upper limit of target range	70106
Quotient $S^*/\sigma_{opt'}$	1,9
Standard uncertainty $U(X_{pt})$	2573
Results in the target range	13
Percent in the target range	87%

° without outliers (results no. 13, 18 and 22)

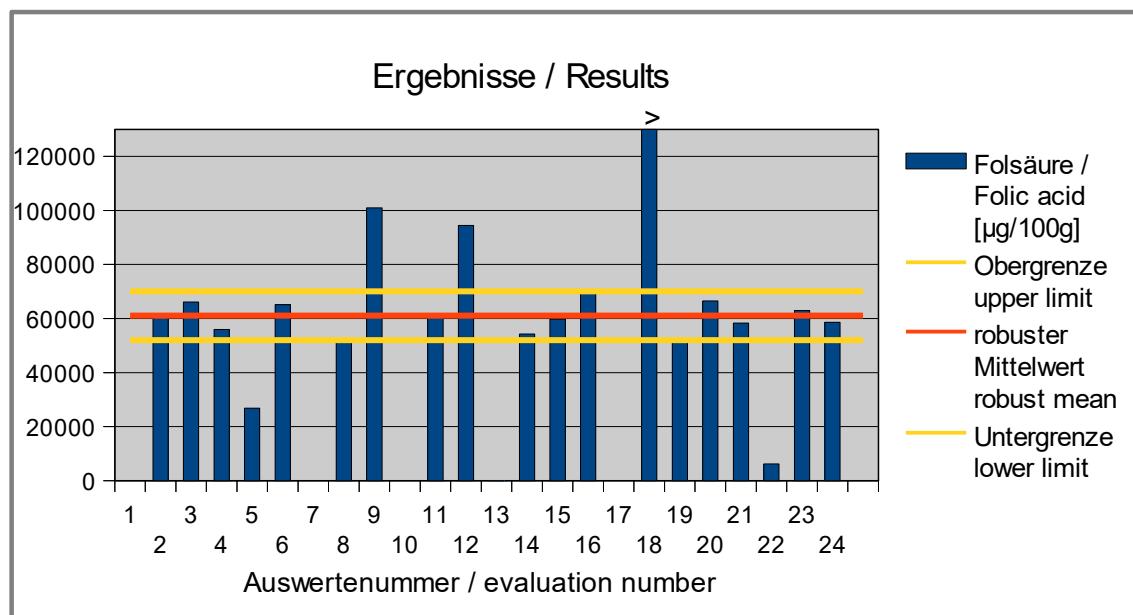


Abb. / Fig. 13: Ergebnisse Folsäure / Results Folic Acid

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-number Evaluation number	Folsäure / Folic acid [µg/100g]	Abweichung [µg/100g] Deviation [µg/100g]	z'-Score (σpt)	Hinweis Remark
1				
2	61200	141	0,03	
3	66100	5041	1,1	
4	55900	-5159	-1,1	
5	26800	-34259	-7,6	
6	65128	4069	0,90	
7				
8	51675	-9384	-2,1	
9	100980 *	39921	8,8	
10				
11	60500	-559	-0,12	
12	94500	33441	7,4	
13	2,4			Ausreißer ausgeschlossen / Outlier excluded
14	54250	-6809	-1,5	
15	59796	-1263	-0,28	
16	69400	8341	1,8	
17	<LOQ			
18	247900			Ausreißer ausgeschlossen / Outlier excluded
19	51644	-9415	-2,1	
20	66532	5473	1,2	
21	58323 *	-2736	-0,60	
22	6231			Ausreißer ausgeschlossen / Outlier excluded
23	62995	1936	0,43	
24	58650	-2409	-0,53	

* Mean calculated by DLA

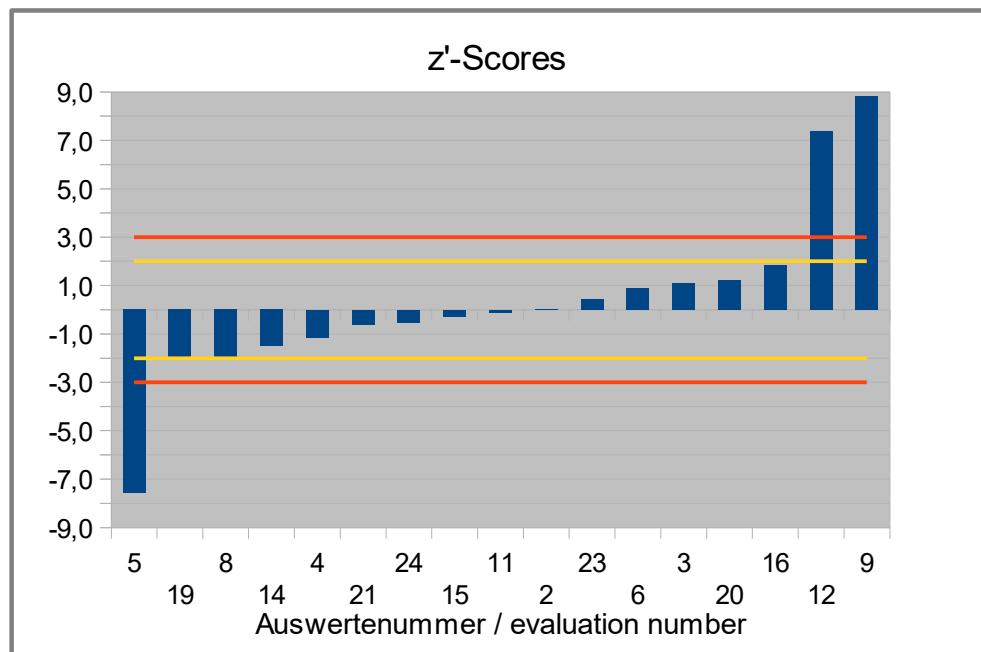


Abb. / Fig. 14: z'-Scores Folsäure / Folic Acid

4.8 Niacin (in mg/100g)

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results °	20
Number of outliers	2
Mean	6061
Median	5934
Robust Mean (X_{pt})	6051
Robust standard deviation (S^*)	649
Number with 2 replicates	18
Repeatability SD (S_r)	87
Repeatability (CV_r)	1,43%
Reproducibility SD (S_R)	823
Reproducibility (CV_R)	13,6%
<i>Target range:</i>	
Target standard deviation σ_{pt}'	294
Target standard deviation (for Information)	185
lower limit of target range	5464
upper limit of target range	6639
Quotient S^*/σ_{pt}'	2,2
Standard uncertainty $U(X_{pt})$	181
Results in the target range	14
Percent in the target range	70%

° without outliers (results no. 13 and 14a)

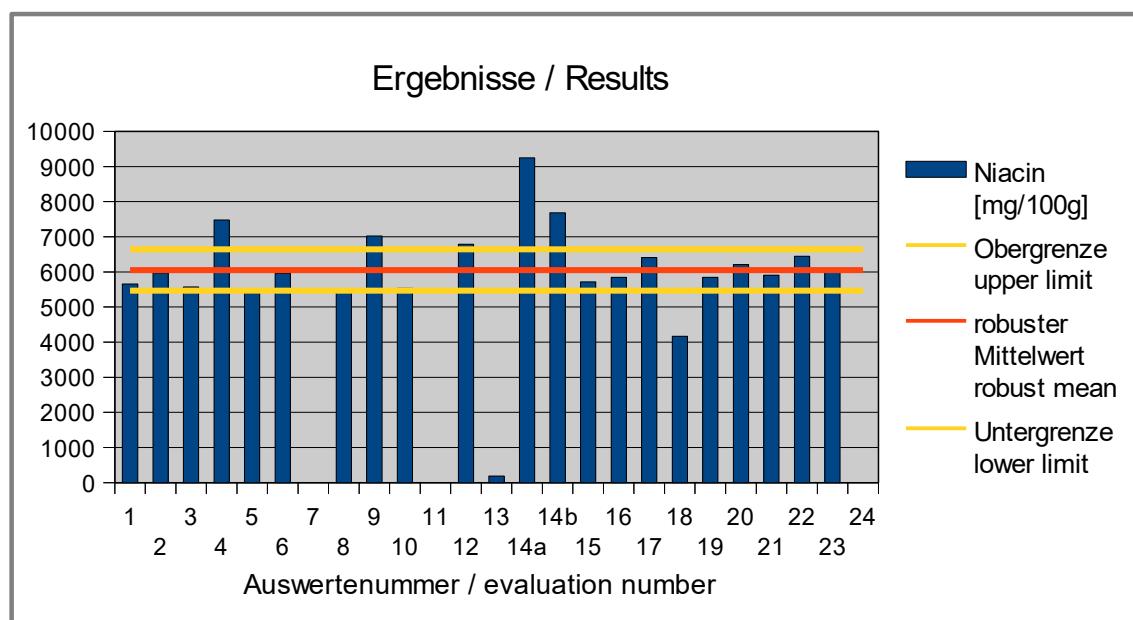


Abb. / Fig. 15: Ergebnisse Niacin / Results Niacin

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte- nummer Evaluation number	Niacin [mg/100g]	Abweichung [mg/100g]	z'-Score (σ_{pt})	z-Score (Info)	Hinweis
		Deviation [mg/100g]			Remark
1	5654	-397	-1,4	-2,2	
2	5970	-81	-0,28	-0,44	
3	5575	-476	-1,6	-2,6	
4	7480	1429	4,9	7,7	
5	5400	-651	-2,2	-3,5	
6	5959	-92	-0,31	-0,50	
7					
8	5529	-523	-1,8	-2,8	
9	7024 *	973	3,3	5,3	
10	5530	-521	-1,8	-2,8	
11					
12	6790	739	2,5	4,0	
13	186				Ausreißer ausgeschlossen / Outlier excluded
14a	9250				Ausreißer ausgeschlossen / Outlier excluded
14b	7683	1631	5,6	8,8	
15	5716	-335	-1,1	-1,8	
16	5850	-201	-0,69	-1,1	
17	6405	354	1,2	1,9	
18	4163	-1888	-6,4	-10	
19	5850	-201	-0,69	-1,1	
20	6207 *	156	0,53	0,84	
21	5908	-143	-0,49	-0,78	
22	6447	396	1,3	2,1	
23	6087	35	0,12	0,19	
24					

* Mean calculated by DLA

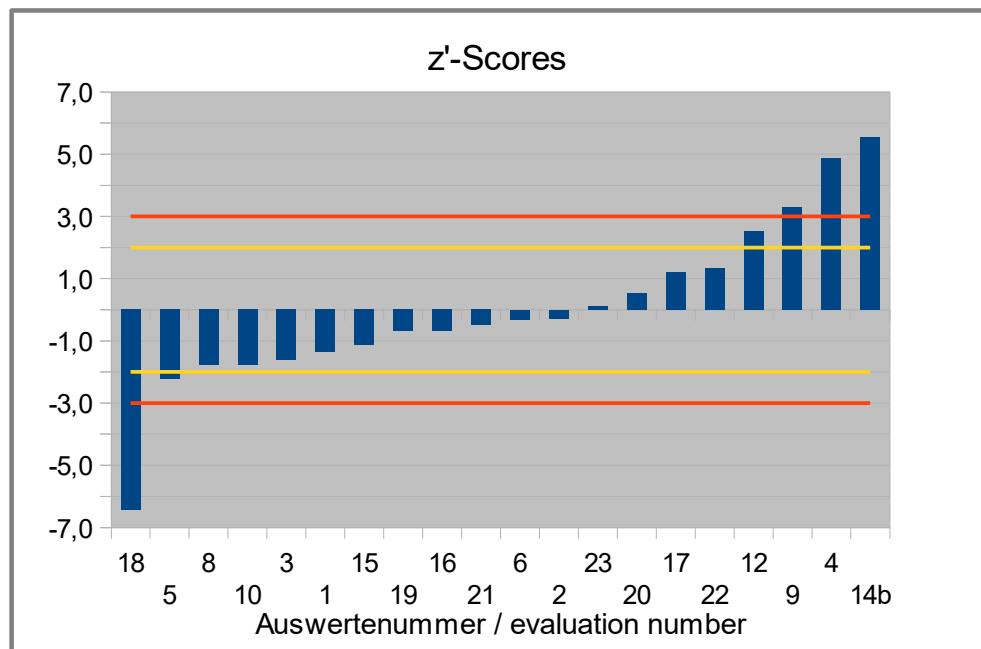


Abb. / Fig. 16: z'-Scores Niacin

4.9 Pantothenic acid (in mg/100g)

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results °	15
Number of outliers	4
Mean	1952
Median	1900
Robust Mean (x_{pt})	1947
Robust standard deviation (S*)	158
Number with 2 replicates	14
Repeatability SD (S_r)	132
Repeatability (CV _r)	6,55%
Reproducibility SD (S_R)	200
Reproducibility (CV _R)	9,99%
Target range:	
Target standard deviation σ_{pt}'	87,0
lower limit of target range	1773
upper limit of target range	2121
Quotient S^*/σ_{pt}'	1,8
Standard uncertainty $U(x_{pt})$	51,0
Results in the target range	12
Percent in the target range	80%

° without outliers (results no. 9, 11, 12 and 13)

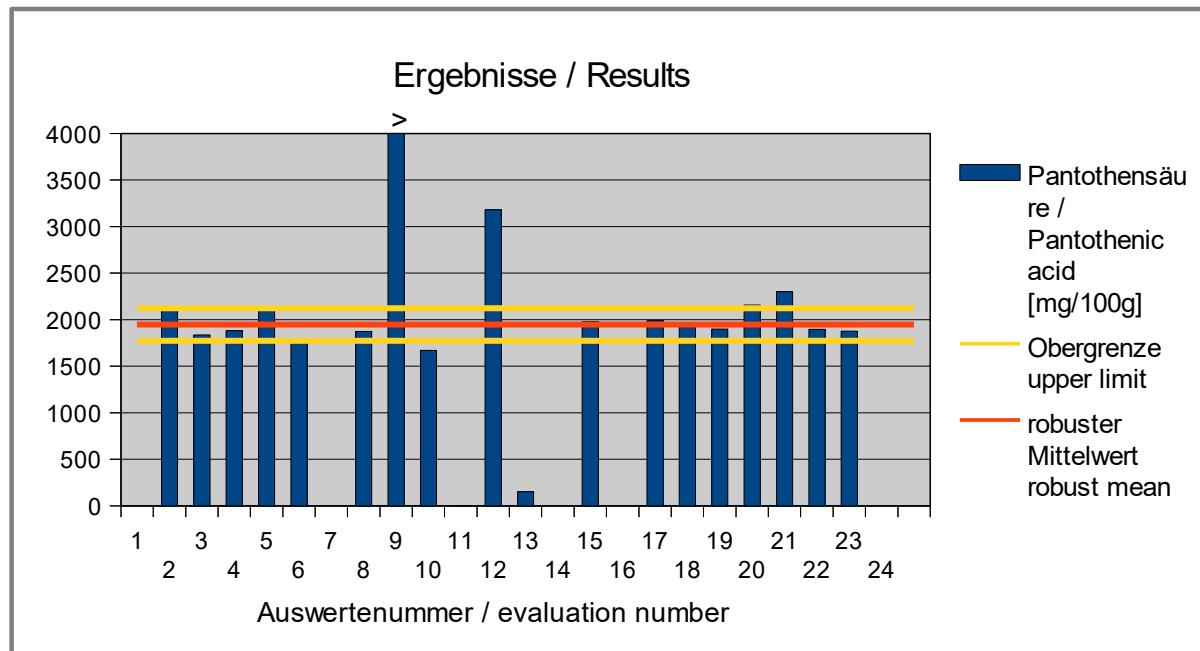


Abb. / Fig. 17: Ergebnisse Pantothenensäure / Results Pantothenic Acid

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-number Evaluation number	Pantothen-säure / Pantothenic acid [mg/100g]	Abweichung [mg/100g] Deviation [mg/100g]	z'-Score (σ_{pt})	Hinweis Remark
1				
2	2110	163	1,9	
3	1835	-112	-1,3	
4	1880	-67	-0,77	
5	2100	153	1,8	
6	1772	-176	-2,0	
7				
8	1873	-75	-0,86	
9	4736 *			Ausreißer ausgeschlossen / Outlier excluded
10	1670	-277	-3,2	
11	2,10			Ausreißer ausgeschlossen / Outlier excluded
12	3180			Ausreißer ausgeschlossen / Outlier excluded
13	151			Ausreißer ausgeschlossen / Outlier excluded
14				
15	1980	33	0,38	
16				
17	1987	40	0,46	
18	1953	6	0,06	
19	1900	-47	-0,54	
20	2156	209	2,4	
21	2300 *	353	4,1	
22	1896	-51	-0,59	
23	1875	-73	-0,84	
24				

* Mean calculated by DLA

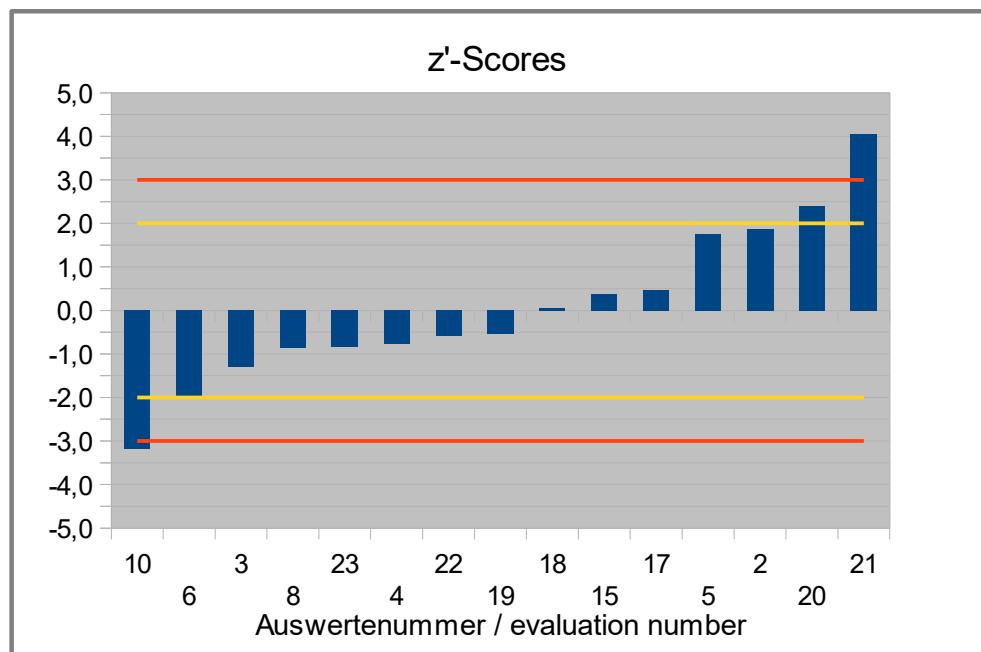


Abb. / Fig. 18: z'-Scores Pantothensäure / Pantothenic Acid

4.10 Participant z-Scores: overview table

Evaluation number	Vit. B1	Vit. B2°	Vit. B6°	Vit. B12	Biotin	Vit. C	Folic Acid°	Niacin°	Pantothenic Acid°
1	-0,45	3,0	0,02					-1,4	
2	-0,91	-2,0	-0,61	0,23	-0,66	0,54	0,03	-0,28	1,9
3	-3,4	-0,14	-0,19		-1,0	0,16	1,1	-1,6	-1,3
4	0,48	1,5	0,97		0,88		-1,1	4,9	-0,77
5	-0,78	-1,7	-1,1	1,0	0,31	-3,8	-7,6	-2,2	1,8
6	-0,83	-0,56	0,69	-0,20	-0,07	0,06	0,90	-0,31	-2,0
7	-1,7	-0,94	-0,27						
8	0,51	-1,7	-0,61	0,66	0,19	-0,13	-2,1	-1,8	-0,86
9	-0,91	-0,58	0,32	-1,9	1,1	-0,03	8,8	3,3	
10	-0,48	3,1	1,2	0,01	-1,0	-1,2		-1,8	-3,2
11	-0,02	-0,11	2,3	-1,7	-0,10	0,19	-0,12		
12	1,5	0,91	64	5,3	2,3	20	7,4	2,5	
13	-0,38	12	-6,9			-1,4			
14/14a	2,0	3,4	2,6	-0,83	-0,10		-1,5		
14b	1,1	1,5	2,5					5,6	
15	-0,69	-0,04	-1,2	1,3	-0,19	-0,02	-0,28	-1,1	0,38
16	-0,68	-0,44	-1,4			0,22	1,8	-0,69	
17	0,37	-0,44	-0,08	0,08		0,51		1,2	0,46
18	16	0,49	-4,7	-3,0	-2,1	-1,2		-6,4	0,06
19	-0,16	-1,6	-1,1	-0,01		0,04	-2,1	-0,69	-0,54
20	1,7	-1,9	2,3			1,1	1,2	0,53	2,4
21	2,8	-0,22	-0,17	1,0	0,74	-0,14	-0,60	-0,49	4,1
22	-1,5	0,77	-1,9	0,06	0,15	0,14		1,3	-0,59
23	-0,14	-1,2	-0,41	0,22	0,03	0,34	0,43	0,12	-0,84
24		3,0	-1,4		-0,18	0,67	-0,53		

° z'-Score

Bewertung des z-Scores / valuation of z-score (DIN ISO 13528:2009-01):

-2 ≤ z-score ≤ 2 erfolgreich / successful (in green)

-2 > z-score > 2 „Warnsignal“ / warning signal (in yellow)

-3 > z-score > 3 „Eingriffssignal“ / action signal (in red)

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 analysis	Result (Mean)	Result Sample I	Result Sample II	Limit of determination	Incl. RR	Recovery rate
					Day/Month					yes / no	in %
Vitamin B1 (als Thiamin-Kation) / Vitamin B1 (as Thiamine-cation)	1	mg/100g	58	14	27.05.2021	495	498	493	50	no	
	2	mg/100g	17	24	31.05.2021	460	446	474		no	
	3	mg/100g	29	43	11.05.2021	273,5	241	306	5	no	
	4	mg/100g	15	57	27.05.2021	566	551	581	3	no	94
	5	mg/100g	13	59	15.06.2021	470	460	490		no	
	6	mg/100g	2	70		466,39	467,61	465,16			
	7	mg/100g	18	54	02.06.2021	401,611	404,147	399,075	0,010 mg/100g	no	81,4
	8	mg/100g	68	4	11.05.2021	568,1	557,2	579	0,02		
	9	mg/100g	11	61	03.05.2021		464	456	2		
	10	mg/100g	1	71	12.05.2021	493	497	489	100	no	
	11	mg/100g	25	47	24.06.2021	528	527	529	0,2	no	
	12	mg/100g	33	39	24.06.2021	644	654	633	0,001	no	not determ.
	13	mg/100g	32	40	17.05.	500,5	490,5	510,6	0,1	no	
	14a	mg/100g	43	53	22.06.	679,8	707,9	651,7	0,01	no	-
	14b	mg/100g	43	53	23.06.	609,12	646,7	571,53		no	-
	15	mg/100g	10	62	05.05.2021	477	470	483	2	no	
	16	mg/100g	8	64	19.05.2021	478	478				
	17	mg/100g	27	45	18.5.	557	568	546		no	
	18	mg/100g	12	60	14.05.2021	1720	1749	1691	0.5mg/kg	no	
	19	mg/100g	9	63	10.06.2021	517	524	510	50	no	not determ.
	20	mg/100g	6	66	21.05.2021	659	661	657		no	
	21	mg/100g	5	67	18.06.2021	18.06.21	759,65	718,97			
	22	mg/100g	7	65	22/06	412	412		5 mg/100g	No	N/A
	23	mg/100g	35	37	14.06.2021	519	522	516	N/A	N/A	N/A
	24	mg/100g									

Analyte	Participant	Unit	Sample I DLA No.	Sample II DLA No.	Date of analysis	Result (Mean)	Result Sample I	Result Sample II	Limit of determination	Incl. RR	Recovery rate
					Day/Month					yes / no	in %
Vitamin B2 (als Riboflavin) / Vitamin B2 (as Riboflavin)	1	mg/100g	58	14	27.05.2021	724	735	713	50	no	
	2	mg/100g	17	24	31.05.2021	515	486	543		no	
	3	mg/100g	29	43	11.05.2021	593,5	570	617	3	no	
	4	mg/100g	15	57	27.05.2021	664	648	680	3	no	109
	5	mg/100g	13	59	15.06.2021	530	530	530		no	
	6	mg/100g	2	70		576,06	569,75	582,38			
	7	mg/100g	18	54	07.06.2021	559,875	555,002	564,748	0,010 mg/100g	no	102,3
	8	mg/100g	68	4	11.05.2021	528,15	521,6	534,7	0,02		
	9	mg/100g	11	61	03.05.2021		576	574	2		
	10	mg/100g	1	71	02.06.2021	728	729	726	100	no	
	11	mg/100g	25	47	24.06.2021	595	592	599	0,2	no	
	12	mg/100g	33	39	24.06.2021	638	633	642	0,005	no	not determ.
	13	mg/100g	32	40	17.05.	1124,7	1116	1133,5	0,1	no	
	14a	mg/100g	43	53	22.06.	743,1	721,99	764,2	0,01	no	-
	14b	mg/100g	43	53	23.06.	662,05	669,29	654,8		no	-
	15	mg/100g	10	62	06.05.2021	598	592	603	2	no	
	16	mg/100g	8	64	19.05.2021	581	581				
	17	mg/100g	27	45	18.5.	581	590	571		no	
	18	mg/100g	12	60	14.05.2021	620	623	617	0.5mg/kg	no	
	19	mg/100g	9	63	10.06.2021	533	537	528	25	no	not determ.
	20	mg/100g	6	66	11.06.2021	520	535	505		no	
	21	mg/100g	5	67	18.06.2021	18.06.21	586,05	594,76			
	22	mg/100g	7	65	22/06	632	632		1 mg/100g	no	N/A
	23	mg/100g	35	37	15.06.21	547,5	551	544	N/A	N/A	N/A
	24	mg/100g	473								

Analyte	Participant	Unit	Sample I DLA No.	Sample II DLA No.	Date of analysis	Result (Mean)	Result Sample I	Result Sample II	Limit of determination	Incl. RR	Recovery rate
					Day/Month					yes / no	in %
Vitamin B6 (als Pyridoxin) / Vitamin B6 (as Pyridoxine)	1	mg/100g	58	14	27.05.21	561	560	563	50	no	
	2	mg/100g	17	24	31.05.21	537	537	537		no	
	3	mg/100g	29	43	11.05.21	553	535	571	5	no	
	4	mg/100g	15	57	27.05.21	597	586	607	3	no	85
	5	mg/100g	13	59	15.06.21	520	510	530		no	
	6	mg/100g	2	70		586,42	588,07	584,78			
	7	mg/100g	18	54	08.06.21	550,1115	552,513	547,71	0,012 mg/100g	no	100,85
	8	mg/100g	68	4	11.05.21	537,2	540,8	533,6	0,04		
	9	mg/100g	11	61	03.05.21		578	567	2		
	10	mg/100g	1	71	12.05.21	607	575	639	100	no	
	11	mg/100g	25	47	24.06.21	649	663	636	0,2	no	
	12	mg/100g	33	39	24.06.21	2970	2820	3120	0,005	no	not determ.
	13	mg/100g	32	40	17.05.	299,9	313,7	286	0,1	no	
	14a	mg/100g	43	53	16.06.	659,47	672,18	646,76	0,01	no	-
	14b	mg/100g	43	53	23.06.	655,54	664,25	646,83		no	-
	15	mg/100g	10	62	05.05.21	514	523	504	1	no	
	16	mg/100g	8	64	10.05.21	507	507				
	17	mg/100g	27	45	18.5.	557	569	544		no	
	18	mg/100g	12	60	14.05.21	382	395	369	0.5mg/kg	no	
	19	mg/100g	9	63	09.06.21	518	525	511	20	no	not determ.
	20	mg/100g	6	66	19.05.21	649	636	661		no	
	21	mg/100g	5	67	18.06.2021	18.06.21	557,66	549,66			
	22	mg/100g	7	65	22/06	490	490		2.5 mg/100g	no	N/A
	23	mg/100g	35	37	16.06.21	544,5	545	544	N/A	N/A	N/A
	24	mg/100g	509								

Analyte	Participant	Unit	Sample I DLA No.	Sample II DLA No.	Date of analysis	Result (Mean)	Result Sample I	Result Sample II	Limit of determination	Incl. RR	Recovery rate
					Day/Month					yes / no	in %
Vitamin B12 (als Cyanocobalamin) / Vitamin B12 (as Cyanocobalamine)	1	µg/100g									
	2	µg/100g	17	24	28.05.2021	786	760	811		no	
	3	µg/100g									
	4	µg/100g	15	57							
	5	µg/100g	13	59	15.06.2021	920	920	910		no	
	6	µg/100g	2	70		712,58	715,17	709,98			
	7	µg/100g									
	8	µg/100g	68	4	13.05.2021	859,55	847,6	871,5	0,1		
	9	µg/100g	11	61	03.05.2021		381	444	0,3		
	10	µg/100g	1	71	25.05.2021	747	803	690	225	no	
	11	µg/100g	25	47	02.06.2021	458	445	470	0,03	no	
	12	µg/100g	33	39	24.06.2021	1650	1760	1530	100	no	not determ.
	13	µg/100g	32	40	17.05.	141431,4	144565,1	138297,6	0,5	no	
	14	µg/100g	16	56	20.05.	604	608	600	0,03	no	-
	15	µg/100g	10	62	05.05.2021	968	984	952	5	no	
	16	µg/100g	N/A								
	17	µg/100g	27	45	18.5.	760	751	768		no	
	18	µg/100g	12	60	14.05.2021	240	253	226	0.5mg/kg	no	
	19	µg/100g	9	63	09.06.2021	744	731	757	450	no	not determ.
	20	µg/100g	6	66	11.06.2021						
	21	µg/100g	5	67	14.06.2021	16.06.21	925	925			
	22	µg/100g	7	65	25/06	756	764	747	200 ug/100g	no	N/A
	23	µg/100g	35	37	17.06.2021	784	798	770	N/A	N/A	N/A
	24	µg/100g									

Analyte	Participant	Unit	Sample I	Sample II	Date of	Result (Mean)	Result	Result	Limit of	Incl. RR	Recovery rate
			DLA No.	DLA No.	analysis		Sample I	Sample II	determination		
Biotin	1	µg/100g			Day/Month					yes / no	in %
	2	µg/100g	17	24	27.05.2021	44000	44500	43400		no	
	3	µg/100g	29	43	11.05.2021	40950	39400	42500	20000	no	
	4	µg/100g	15	57	17.06.2021	57050	53900	60200	0,08	no	
	5	µg/100g	13	59	15.06.2021	52200	51800	52500		no	
	6	µg/100g	2	70		48950,1	48258,07	49642,13			
	7	µg/100g									
	8	µg/100g	68	4	13.05.2021	51185	54285	48085	2		
	9	µg/100g	11	61	03.05.2021		56392	60709	1		
	10	µg/100g	1	71	12.05.2021	40700	41400	40000	4000	no	
	11	µg/100g	25	47	20.05.2021	48675	48900	48450	0,08	no	
	12	µg/100g	33	39	24.06.2021	68800	68600	69700	200	no	not determ.
	13	µg/100g									
	14	µg/100g	16	56	23.06.	48750	49300	48200	0,08	no	-
	15	µg/100g	10	62	06.05.2021	47932	47631	48232	2500	no	
	16	µg/100g	N/A								
	17	µg/100g	27	45	18.5.						
	18	µg/100g	12	60	14.05.2021	31583	28600	34567	0.5mg/kg	no	
	19	µg/100g	9	63						no	not determ.
	20	µg/100g	6	66							
	21	µg/100g	5	67	15.06.2021	17.06.21	56848,18	54878,05			
	22	µg/100g	7	65	24/06	50817	52641	48993	0.75 µg/100g	no	N/A
	23	µg/100g	35	37	15.06.2021	49813	50650	48975	N/A	N/A	N/A
	24	µg/100g	48000			48000					

Analyte	Participant	Unit	Sample I DLA No.	Sample II DLA No.	Date of analysis	Result (Mean)	Result Sample I	Result Sample II	Limit of determination	Incl. RR	Recovery rate
					Day/Month					yes / no	in %
Vitamin C (als Ascorbinsäure) / Vitamin C (as Ascorbic acid)	1	mg/100g									
	2	mg/100g	17	24	31.05.2021	15900	16340	15390		no	
	3	mg/100g	29	43	11.05.2021	15300	15400	15200	200	no	
	4	mg/100g									
	5	mg/100g	13	59	15.05.2021	9000	8800	9300		no	
	6	mg/100g	2	70		15140,75	15152,43	15129,07			
	7	mg/100g									
	8	mg/100g	68	4	12.05.2021	14845	14680	15010	2		
	9	mg/100g	11	61	03.05.2021		14893	15122	1		
	10	mg/100g	1	71	06.05.2021	13100	13150	13000	500	no	
	11	mg/100g	25	47	14.05.2021	15347	15253	15441	2,5	no	
	12	mg/100g	33	39	24.06.2021	46700	46100	47200	0,1	no	not determ.
	13	mg/100g	32	40	17.05.	12863	12985,3	12740,8	1	no	
	14	mg/100g									
	15	mg/100g	10	62	23.06.2021	15012	15370	14654	8	no	
	16	mg/100g	8	64	11.05.2021	15400		15400			
	17	mg/100g	27	45	18.5.	15850	15800	15900		no	
	18	mg/100g	12	60	14.05.2021	13193	13199	13186	0.5mg/kg	no	
	19	mg/100g	9	63	02.06.2021	15120	15126	15114	4000	no	not determ.
	20	mg/100g	6	66	19.05.2021	16741	16919	16562		no	
	21	mg/100g	5	67	18.06.2021	18.06.21	14923,4	14746,99			
	22	mg/100g	7	65	21/06	15269	15418	15119	1 mg/100g	no	N/A
	23	mg/100g	35	37	01.06.2021	15593	15823	15362	N/A	N/A	N/A
	24	mg/100g	16107								

Analyte	Participant	Unit	Sample I DLA No.	Sample II DLA No.	Date of analysis	Result (Mean)	Result Sample I	Result Sample II	Limit of determination	Incl. RR	Recovery rate
					Day/Month					yes / no	in %
Folsäure (als Pteroylmonoglutamin säure) / Folic acid (as Pteroylmonoglutamini c acid)	1	µg/100g									
	2	µg/100g	17	24	28.05.2021	61200	60600	61700		no	
	3	µg/100g	29	43	11.05.2021	66100	63900	68300	5000	no	
	4	µg/100g	15	57	17.05.2021	55900	56650	55150	0,16	no	
	5	µg/100g	13	59	15.06.2021	26800	26500	27000		no	
	6	µg/100g	2	70		65128,03	65371,67	64884,39			
	7	µg/100g									
	8	µg/100g	68	4	25.05.2021	51675	52943,5	50406,5	1		
	9	µg/100g	11	61	03.05.2021		103740	98211	1		
	10	µg/100g									
	11	µg/100g	25	47	20.05.2021	60500	61200	59800	0,2	no	
	12	µg/100g	33	39	24.06.2021	94500	91800	97200	100	no	not determ.
	13	µg/100g	32	40	17.05.	2,4	2,4	2,4	0,1	no	
	14	µg/100g	16	56	21.05.	54250	53200	55300	0,16	no	-
	15	µg/100g	10	62	07.05.2021	59796	60106	59485	1000	no	
	16	µg/100g	8	64	12.05.2021	69400	69400				
	17	µg/100g	27	45	18.5.	<BG	<BG	>BG	100000	no	
	18	µg/100g	12	60	14.05.2021	247900	280400	215400	0.5mg/kg	no	
	19	µg/100g	9	63	03.06.2021	51644	51906	51381	15610	no	not determ.
	20	µg/100g	6	66	19.05.2021	66532	67792	65272		no	
	21	µg/100g	5	67	15.06.2021	17.06.21	58069,31	58576,05			
	22	µg/100g	7	65	22/06	6231	6231		1000 µg/100g	No	N/A
	23	µg/100g	35	37	16.06.2021	62995	62444	63545	N/A	N/A	N/A
	24	µg/100g	58650								

Analyte	Participant	Unit	Sample I DLA No.	Sample II DLA No.	Date of analysis	Result (Mean)	Result Sample I	Result Sample II	Limit of determination	Incl. RR	Recovery rate
					Day/Month					yes / no	in %
Niacin	1	mg/100g	58	14	27.05.2021	5654	5710	5598	50	no	
	2	mg/100g	17	24	31.05.2021	5970	5900	6040		no	
	3	mg/100g	29	43	11.05.2021	5575	5630	5520	5	no	
	4	mg/100g	15	57	27.05.2021	7480	7522	7439	30	no	113
	5	mg/100g	13	59	15.06.2021	5400	5300	5500		no	
	6	mg/100g	2	70		5959,14	5929,86	5988,41			
	7	mg/100g									
	8	mg/100g	68	4	19.05.2021	5528,8	5545,5	5512,1	0,2		
	9	mg/100g	11	61	03.05.2021	DLA7024	7059	6989	0,08		
	10	mg/100g	1	71	12.05.2021	5530	5500	5560	320	no	
	11	mg/100g	25	47					0,2	no	
	12	mg/100g	33	39	24.06.2021	6790	6790	6790	0,005	no	not determ.
	13	mg/100g	32	40	17.05.	185,7	191,3	180,1	0,1	no	
	14a	mg/100g	16	56	09.06.	9250	9250	9250	0,016	no	-
	14b	mg/100g	43	53	23.06.	7682,92	7713,05	7652,79		no	-
	15	mg/100g	10	62	23.06.2021	5716	5739	5693	1,5	no	
	16	mg/100g	8	64	12.05.2021	5850	5850				
	17	mg/100g	27	45	18.5.	6405	6440	6370		no	
	18	mg/100g	12	60	14.05.2021	4163	4281	4045	0.5mg/kg	no	
	19	mg/100g	9	63	12.05.2021	5850	5700	6000	1800	no	not determ.
	20	mg/100g	6	66	07.06.2021	6207	6218	6195		no	
	21	mg/100g	5	67	14.06.2021	6.06.2021DLA5907,9	5870,65	5945,27			
	22	mg/100g	7	65	22/06	6447	6447		1 mg/100g	no	N/A
	23	mg/100g	35	37	14.06.2021	6086,5	6057	6116	N/A	N/A	N/A
	24	mg/100g									

Analyte	Participant	Unit	Sample I DLA No.	Sample II DLA No.	Date of analysis	Result (Mean)	Result Sample I	Result Sample II	Limit of determination	Incl. RR	Recovery rate
					Day/Month					yes / no	in %
Pantothenensäure / Pantothenic acid	1	mg/100g									
	2	mg/100g	17	24	31.05.21	2110	2030	2190		no	
	3	mg/100g	29	43	11.05.21	1835	1770	1900	10	no	
	4	mg/100g	15	57	20.05.21	1880	1947	1813	0,04	no	
	5	mg/100g	13	59	15.06.21	2100	2100	2100		no	
	6	mg/100g	2	70		1771,74	1811,96	1731,52			
	7	mg/100g									
	8	mg/100g	68	4	13.05.21	1872,5	1895	1850	0,05		
	9	mg/100g	11	61	03.05.21		2643	2093	0,05		
	10	mg/100g	1	71	12.05.21	1670	1660	1670	240	no	
	11	mg/100g	25	47	11.06.21	2,1	2,01	2,19	0,04	no	
	12	mg/100g	33	39	24.06.21	3180	3100	3260	0,005	no	not determ.
	13	mg/100g	32	40	17.05.	150,8	150,8	150,7	0,1	no	
	14	mg/100g									
	15	mg/100g	10	62	22.06.21	1980	2003	1956	2,5	no	
	16	mg/100g	N/A								
	17	mg/100g	27	45	18.5.	1987	1963	2010		no	
	18	mg/100g	12	60	14.05.21	1953	2107	1799	0.5mg/kg	no	
	19	mg/100g	9	63	11.05.21	1900	1900	1900	210	no	not determ.
	20	mg/100g	6	66	21.05.21	2156	2102	2209		no	
	21	mg/100g	5	67	15.06.2021	16.06.21	2300	2300			
	22	mg/100g	7	65	22/06	1896	1896		5 mg/100g	no	N/A
	23	mg/100g	35	37	15.06.21	1874,5	1897	1852	N/A	N/A	N/A
	24	mg/100g									

5.1.2 Analytical Methods

Parameter	Partici-pant	Method description as in test report / norm / literature	Sample preparation	Measuring method	Calibration / Reference ma-terial	Recovery rate with same matrix	Method ac-credited ISO/IEC 17025	Further Remarks
				HPLC-PDA		yes / no	yes / no	
	1						yes	
	2	SOP M843, HPLC-UV					yes	
	3			HPLC-DAD-FLD	external calibration		no	
	4	internal method HPLC-MS-MS (P4-02-01-12-2423)	enzymic digest, centrifugation, filtration, dilution; addition ISTD	HPLC-MS/MS	external calibration with ISTD	no	yes	recovery with NIST-SRM-3280 determined.
	5	UHPLC-MS/MS	liquefaction / dilution	LC-ESI-MS/MS	external standard row		no	
	6			HPLC			yes	
	7	ASU L00.00-83	Extraction	HPLC with post-column derivatization and fluorescence detection	Ext. calibration with reference material	yes	yes	
	8							
	9	VDLUFA III, 13.9.1 : 2006 (mod.)		HPLC			yes	
	10	LCMSMS	no	no			yes	
	11	internes Verfahren HPLC					yes	
Vitamin B1 (als Thiamin-Kation) / Vitamin B1 (as Thiamine-cation)	12	in-house Method	in Methanol/Water (50/50) dissolved	LC-MS/MS	external calibration	no	no	
	13	ASU L00.00-83:2015-06		HPLC			yes	
	14a	PV 0016-04 (ASU L 00.00-83 (2015-06))		HPLC-FLD	T21119QC (Powdered Baby Food)	no	yes	
	14b	ISO/DIN 21470		LC-MS/MS		no	no (not yet)	
	15	MI_558_2020_Rev.2	-	-	Thiamine hydrochloride	no	yes	
	16						Yes	
	17	HPLC-DAD / FLD					yes	
	18	The rapid analysis of water soluble vitamins, Waters	water dilution	UV 270 nm	calibration curve	no	no	
	19	Quantitative determination of water-soluble vitamins by HPLC in food supplements			PT Material		yes	
	20						yes	
	21	PB-257/LF - "Oznaczanie zawartości witamin metodą HPLC "	wg instrukcji	HPLC-UV		no	yes	
	22	HPLC-DAD; 232 nm	Extraction with EDTA-Na2, sodium thiosulfate, phosphoric acid, dilution with ammonium hydroxide		Six level external calibration	No	No	Reported as thiamine cation in mg/100g
	23	MQLTM-0153						
	24							

Parameter	Partici-pant	Method description as in test report / norm / literature	Sample preparation	Measuring method	Calibration / Reference ma-terial	Recovery rate with same matrix	Method ac-credited ISO/IEC 17025	Further Remarks
				HPLC-FL		yes / no	yes / no	
1				HPLC-DAD-FLD	externe Kalibrierung		yes	
2	SOP M843, HPLC-UV						yes	
3							no	
4	internal method HPLC-MS-MS (P4-02-01-12-2423)	enzymic digest, centrifugation, filtration, dilution; addition ISTD		HPLC-MS/MS	external calibration with ISTD	no	yes	recovery with NIST-SRM-3280 determined.
5	UHPLC-MS/MS	liquefaction / dilution		LC-ESI-MS/MS	external standard row		no	
6				HPLC			yes	
7	ASU L00.00-84	Extraction		HPLC fluorescence detection	Ext. calibration with reference material	yes	yes	
8								
9	VDLUFA III, 13.9.1 : 2006 (mod.)			HPLC			yes	
10	LCMSMS	no		no			yes	
11	internal method HPLC						yes	
12	in-house Method	in Methanol/Water (50/50) dissolved		LC-MS/MS	external calibration	no	no	
13	ASU L00.00-84:2015-06			HPLC			yes	
14a	PV 0017-04 (ASU L 00.00-84 (2015-06))			HPLC-FLD	T21119QC (Powdered Baby Food)	no	yes	
14b	ISO/DIN 21471			LC-MS/MS		no	no (not yet)	
15	MI_558_2020_Rev.2	-	-	Riboflavin		no	yes	
16							Yes	
17	HPLC-DAD / FLD						yes	
18	The rapid analysis of water soluble vitamins, Waters	water dilution	UV 270 nm	calibration curve	no	no		
19	Quantitative determination of water-soluble vitamins by HPLC in food supplements				PT Material		yes	
20							yes	
21	PB-257/LF - "Oznaczanie zawartości witamin metodą HPLC "	wg instrukcji	HPLC-UV			no	yes	
22	HPLC-DAD; 268 nm	Extraction with EDTA-Na2, sodium thiosulfate, phosphoric acid, dilution with ammonium hydroxide			Six level external calibration	No	No	Reported as riboflavin in mg/100g
23	MQLTM-0153							
24								

Parameter	Partici-pant	Method description as in test report / norm / literature	Sample preparation	Measuring method	Calibration / Reference ma-terial	Recovery rate with same matrix	Method ac-credited ISO/IEC 17025	Further Remarks
				HPLC-FL		yes / no	yes / no	
1							yes	
2	SOP M843, HPLC-UV						yes	
3				HPLC-DAD-FLD	external calibration		no	
4	internal method HPLC-MS-MS (P4-02-01-12-2423)	enzymic digest, centrifugation, filtration, dilution; addition ISTD		HPLC-MS/MS	external calibration with ISTD	no	yes	recovery with NIST-SRM-3280 determined.
5	UHPLC-MS/MS	liquefaction / dilution		LC-ESI-MS/MS	external standard row		no	
6				HPLC			yes	
7	ASU L00.00-97	Extraction		HPLC fluorescence detection	Ext. calibration with reference material	yes	yes	
8								
9	VDLUFA III, 13.9.1 : 2006 (mod.)			HPLC			yes	
10	LCMSMS	no		no			yes	
11	internal method HPLC						yes	
12	in-house Method	in Methanol/Water (50/50) dissolved		LC-MS/MS	external calibration	no	no	
13	Swiss Food Handbook Chapt 12.1:2000			HPLC			no	
14a	PV 0018-02 (ASU L 00.00-97 (2006-12))			HPLC-FLD	DLA ptSU06 RM2020-SU06 (Drink powder)	no	yes	
14b	ISO/DIN 21472			LC-MS/MS		no	no (not yet)	
15	MI_558_2020_Rev.2	-	-		Pyridoxine hydrochloride	no	yes	
16							Yes	
17	HPLC-DAD / FLD						yes	
18	The rapid analysis of water soluble vitamins, Waters	water dilution	UV 270 nm	calibration curve	no	no		
19	Quantitative determination of water-soluble vitamins by HPLC in food supple-ments				PT Material		yes	
20							yes	
21	PB-257/LF - "Oznaczanie zawartości vitamin metodą HPLC "	wg instrukcji	HPLC-UV		no	yes		
22	HPLC-DAD; 220 nm	Extraction with EDTA-Na2, sodium thiosulfate, phosphoric acid, dilution with ammonium hydroxide			Six level external calibration	No	No	Reported as pyridoxine in mg/100g
23	MQLTM-0153							
24								

Parameter	Partici-pant	Method description as in test report / norm / literature	Sample preparation	Measuring method	Calibration / Reference ma-terial	Recovery rate with same matrix	Method ac-credited ISO/IEC 17025	Further Remarks
						yes / no	yes / no	
Vitamin B12 (als Cyanocobalamin) / Vitamin B12 (as Cyanocobalamine)	1							
	2	SOP M844, HPLC-UV					yes	
	3							
	4						yes	
	5	UHPLC-MS/MS	liquefaction / dilution	LC-ESI-MS/MS	external standard row		no	
	6			HPLC			yes	
	7							
	8							
	9	USP 39, method 171 : 2016 (mod.)		Bioassay			yes	
	10	LCMSMS	no	no			yes	
	11	Vita Fast B12 (RBiopharm)					yes	
	12	in-house Method	in Methanol/Water (50/50) dissolved	LC-MS/MS	external calibration	no	no	
	13	Fast ELISA B12 r-Biopharm		ELISA			no	
	14	Mikrobiologisch (VitaFast Vitamin B12 - Cyanocobalamin / r-biopharm Art. No. P1002)		Microbiological	SRM 3280	no	yes	
	15	MI 540 2019 Rev.2	-	-	Cyanocobalamin	no	yes	
	16							
	17	HPLC / VIS,MS					yes	
	18	The rapid analysis of water soluble vitamins, Waters	water dilution	UV 270 nm	calibration curve	no	no	
	19	R-Biopharm Vitafast - K12 44203 2022-1			PT Material		yes	
	20						no	
	21	Instrukcyes testu VitaFast Vitamin B12 (Cyanocobalamin) firmy R-Biopharm AG	wg instrukcji	Metoda mikrobiologiczna - mikroplytkowa z odczytem spektrofotometrycznym		no	yes	
	22	UHPLC-DAD; 361 nm	Extraction with pH adjusted buffer; immunoaffinity column purification		Six level external calibration	No	No	Reported as cyanocobalamin
	23	MQLTM-0153						
	24							

Parameter	Partici-pant	Method description as in test report / norm / literature	Sample preparation	Measuring method	Calibration / Reference ma-terial	Recovery rate with same matrix	Method ac-credited ISO/IEC 17025	Further Remarks
						yes / no	yes / no	
Biotin	1							
	2	SOP M3532, LC-MS/MS					yes	
	3			HPLC-DAD-FLD	external calibration		no	
	4	Test-Kit r-biopharm Vitafast® Vitamin B7 (Biotin) Art.No.P1003	as per kit instructions		as per kit instructions		yes	
	5	UHPLC-MS/MS	liquefaction / dilution	LC-ESI-MS/MS	external standard row		no	
	6			HPLC			yes	
	7							
	8							B8
	9	USP 21,3. suppl, method 88 : 1986		Bioassay			yes	
	10	LCMSMS	no	no			yes	
	11	Vita Fast Biotin (RBiopharm)					yes	
	12	in-house Method	in Methanol/Water (50/50) dissolved	LC-MS/MS	external calibration	no	no	
	13							
	14	Microbiological (VitaFast Vitamin B7 - Biotin / r-biopharm Art. No. P1003)		Microbiological	SRM 3280	no	yes	
	15	MI 558 2020 Rev.2	-	-	Biotin	no	yes	
	16							
	17							
	18	The rapid analysis of water soluble vitamins, Waters	water dilution	UV 205 nm	calibration curve	no	no	
	19	R-Biopharm Vitafast - KB44 265 2022-03			PT Material		yes	Analytical problems
	20						no	
	21	Instrukcjes testu VitaFast Biotin firmy R-Biopharm AG	wg instrukcji	Metoda mikrobiologiczna - mikroplytkowa z odczytem spektrofotometrycznym		no	yes	
	22	LC/MS/MS; Precursor 245 m/z; Product ions 227, 97 and 123 m/z	Extraction with dilute sodium hydroxide		Six level calibration; Biotin-d4 internal standard	No	No	Reported as biotin in ug/100g
	23	MQLTM-0155						
	24							

Parameter	Partici-pant	Method description as in test report / norm / literature	Sample preparation	Measuring method	Calibration / Reference ma-terial	Recovery rate with same matrix	Method ac-credited ISO/IEC 17025	Further Remarks
						yes / no	yes / no	
Vitamin C (als Ascorbinsäure) / Vitamin C (as Ascorbic acid)	1							
	2	SOP M547, HPLC-FI					yes	
	3			HPLC-DAD	external calibration		yes	
	4							
	5	UHPLC-MS/MS	liquefaction / reduction / dilution	LC-ESI-MS/MS	external standard row		no	
	6			HPLC			yes	
	7							
	8							
	9	Hausmethode		HPLC			yes	
	10	HPLC-UV	no	no			yes	
	11	internal method HPLC					yes	
	12	in-house Method	in Methanol/Water (50/50) dissolved	LC-MS/MS	external calibration	no	no	
	13	enzymatic determination by Boehringer Kit r-Biopharm		Photometry			no	
	14							
	15	MI_089_2012_Rev.5	-	-	Ascorbic acid	no	yes	
	16						Yes	
	17	HPLC-DAD					yes	
	18	The rapid analysis of water soluble vitamins, Waters	water dilution	UV 270 nm	calibration curve	no	no	
	19	Vitamin C in foods, HPLC-FLD			PT Material		yes	
	20						yes	
	21	PB-257/LF - "Oznaczanie zawartości witamin metodą HPLC"	wg instrukcji	HPLC-UV		no	yes	
	22	HPLC-DAD; 247 nm	Extraction with EDTA-Na2, sodium thiosulfate, phosphoric acid, dilution with ammonium hydroxide; TCEP reduction		Six level external calibration	No	No	Reported as ascorbic acid in mg/100g
	23	MQLTM-0149						
	24							

Parameter	Participant	Method description as in test report / norm / literature	Sample preparation	Measuring method	Calibration / Reference material	Recovery rate with same matrix	Method accredited ISO/IEC 17025	Further Remarks
						yes / no	yes / no	
Folsäure (als Pteroylmonoglutaminsäure) / Folic acid (as Pteroylmonoglutaminic acid)	1							
	2	SOP M3816, LC-MS/MS					yes	
	3			HPLC-DAD-FLD	external calibration		no	
	4	Test-Kit r-biopharm Vitafast® Folsäure Art.no.P1001	as per kit instructions		as per kit instructions		yes	
	5	UHPLC-MS/MS	liquefaction / dilution	LC-ESI-MS/MS	external standard row		no	
	6			HPLC			yes	
	7							
	8							B9
	9	DIN EN 14131 : 2003-09 (mod.)		Bioassay			yes	
	10							
	11	Vita Fast Folic Acid (RBiopharm)					yes	
	12	in-house Method	in Methanol/Water (50/50) dissolved	LC-MS/MS	external calibration	no	no	
	13	Swiss Food Handbook Chapt. 12.1:2000		HPLC			no	
	14	Microbiological (VitaFast Vitamin B9 - Folic Acid / r-biopharm Art. no. P1001)		Microbiological	SRM 3280	no	yes	
	15	MI_558_2020_Rev.2	-	-	Folic acid	no	yes	
	16						Yes	
	17	HPLC-DAD / FLD					yes	not detectable
	18	The rapid analysis of water soluble vitamins, Waters	water dilution	UV 270 nm	calibration curve	no	no	
	19	R-Biopharm VitaFast - Folic Acid KF44054 2021-08			LVU Material		yes	
	20						yes	
	21	Instrukcja testu VitaFast Folic Acid firmy R-Biopharm AG	wg instrukcji	Metoda mikrobiologiczna - mikrofotokwowa z odczytem spektrofotometrycznym		no	yes	
	22	HPLC-DAD; 280 nm	Extraction with EDTA-Na2, sodium thiosulfate, phosphoric acid, dilution with ammonium hydroxide		Six level external calibration	No	No	Reported as folic acid in ug/100g
	23	MQLTM-0150						
	24							

Parameter	Partici-pant	Method description as in test report / norm / literature	Sample preparation	Measuring method	Calibration / Reference ma-terial	Recovery rate with same matrix	Method ac-credited ISO/IEC 17025	Further Remarks
				HPLC-PDA		yes / no	yes / no	
	1						yes	
	2	SOP M843, HPLC-UV					yes	
	3			HPLC-DAD-FLD	external calibration		yes	
	4	internal method HPLC-MS-MS (P4-02-01-12-2423)	enzymic digest, centrifugation, filtration, dilution; addition ISTD	HPLC-MS/MS	external calibration with ISTD	no	no	recovery with NIST-SRM-3280 determined.
	5	UHPLC-MS/MS	liquefaction / dilution	LC-ESI-MS/MS	external standard row		no	
	6			HPLC			yes	
	7							
	8							B3
	9	USP 34, method 441 : 2011 (mod.)		Bioassay			yes	
	10	LCMSMS	no	no			yes	
	11	internes Verfahren HPLC					yes	
	12	in-house Method	in Methanol/Water (50/50) dissolved	LC-MS/MS	external calibration	no	no	
	13	Swiss Food Handbook Chapt12.1:2000		HPLC			no	
Niacin	14a	Microbiological (VitaFast Vitamin B3 - Niacin / r-biopharm Art. No. P1004)		Microbiological	SRM 3280	no	yes	
	14b	ISO/DIN 21472		LC-MS/MS		no	no (not yet)	
	15	MI_558_2020_Rev.2	-	-	Nicotinamide	no	yes	
	16						Yes	
	17	HPLC-DAD / FLD					yes	
	18	The rapid analysis of water soluble vitamins, Waters	water dilution	UV 270 nm	calibration curve	no	no	
	19	Quantitative determination of water-soluble vitamins by HPLC in food supplements			PT Material		yes	
	20						yes	
	21	Instrukcja testu VitaFast Vitamin B3 (Niacin) firmy R-Biopharm AG	wg instrukcji	Metoda mikrobiologiczna - mikropłytkowa z odczytem spektrofotometrycznym		no	yes	
	22	HPLC-DAD; 214 nm	Extraction with EDTA-Na2, sodium thiosulfate, phosphoric acid, dilution with ammonium hydroxide		Six level external calibration	No	No	Reported as the sum of niacin (28 mg/100g) and niacinamide (6419 mg/100g)
	23	MQLTM-0153						
	24							

Parameter	Partici-pant	Method description as in test report / norm / literature	Sample preparation	Measuring method	Calibration / Reference ma-terial	Recovery rate with same matrix	Method ac-credited ISO/IEC 17025	Further Remarks
						yes / no	yes / no	
Pantothenic acid / Pantothenic acid	1							
	2	SOP M856, HPLC-UV					yes	
	3			HPLC-DAD-FLD	external calibration		no	
	4	Test-Kit r-biopharm Vitafast® Pantothenic Acid Art.No.P1005	as per kit instructions		as per kit instructions		yes	
	5	UHPLC-MS/MS	liquefaction / dilution	LC-ESI-MS/MS	external standard row		no	
	6			HPLC			yes	
	7							
	8							B5
	9	USP 39, method 91 : 2016		Bioassay			yes	
	10	LCMSMS	no	no			yes	
	11	Vita Fast Pantothenic acid (RBiopharm)					yes	
	12	in-house Methode	in Methanol/Wasser (50/50) gelöst	LC-MS/MS	external calibration	no	no	
	13	Vita Fast ELISA Pantothenic acid r-Biopharm		ELISA			no	
	14							
	15	MI_558_2020_Rev.2	-	-	Calcium pantothenate	no	yes	
	16							
	17	LC / MS					yes	2 measuring days
	18	The rapid analysis of water soluble vitamins, Waters	water dilution	UV 205 nm	calibration curve	no	no	
	19	Quantitative determination of water-soluble vitamins by HPLC in food supplements			PT Material		yes	
	20						yes	
	21	Instrukcje testu VitaFast Pantothenic Acid firmy R-Biopharm AG	wg instrukcji	Metoda mikrobiologiczna - mikropłytkowa z odczytem spektrofotometrycznym		no	yes	
	22	HPLC-DAD; 214 nm	Extraction with EDTA-Na2, sodium thiosulfate, phosphoric acid, dilution with ammonium hydroxide		Six level external calibration	No	No	Reported as pantothenic acid in mg/100g
	23	MQLTM-0153						
	24							

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	5,181
2	5,117
3	5,170
4	4,945
5	4,936
6	4,903
7	4,903
8	4,966
9	4,919
10	4,926

General Mean 5,00
Repeatability standard deviation 0,113 2,26%

Vitamin B2

Independant samples	g/kg
1	5,601
2	5,445
3	5,443
4	5,503
5	5,568
6	5,526
7	5,553
8	5,499
9	5,492
10	5,597

General Mean 5,52
Repeatability standard deviation 0,057 1,03%

Vitamin B6

Independant samples	g/kg
1	5,297
2	5,278
3	5,252
4	5,304
5	5,374
6	5,326
7	5,276
8	5,128
9	5,274
10	5,374

General Mean 5,29
Repeatability standard deviation 0,070 1,32%

Niacinamide

Independant samples	g/kg
1	61,52
2	60,91
3	60,76
4	60,36
5	61,52
6	62,31
7	61,29
8	61,21
9	60,56

General Mean 61,2
Repeatability standard deviation 0,594 0,97%

Pantothenic acid

Independant samples	g/kg
1	19,17
2	18,67
3	18,67
4	18,82
5	18,78
6	18,31
7	18,25
8	18,29
9	18,11
10	18,31

General Mean 18,5
Repeatability standard deviation 0,334 1,80%

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:

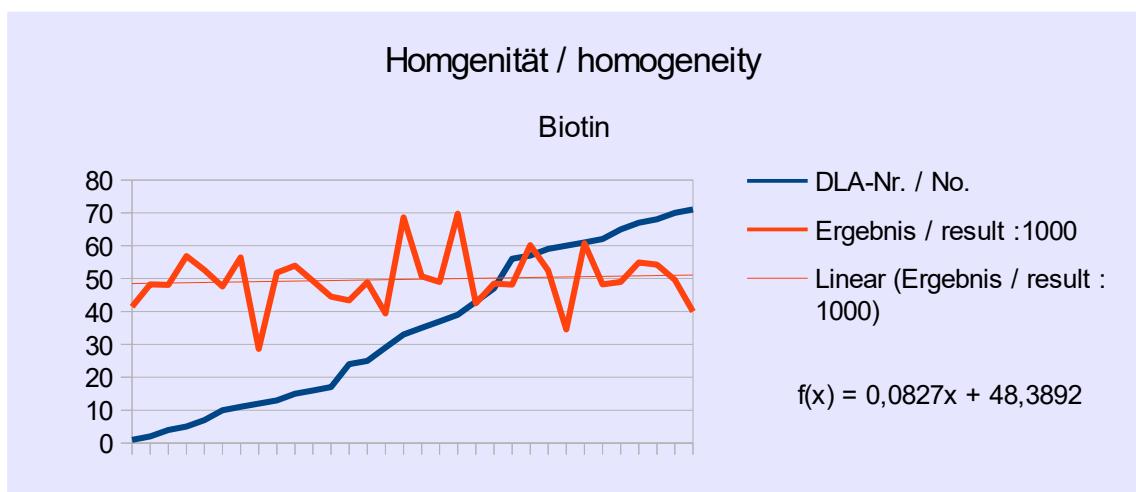
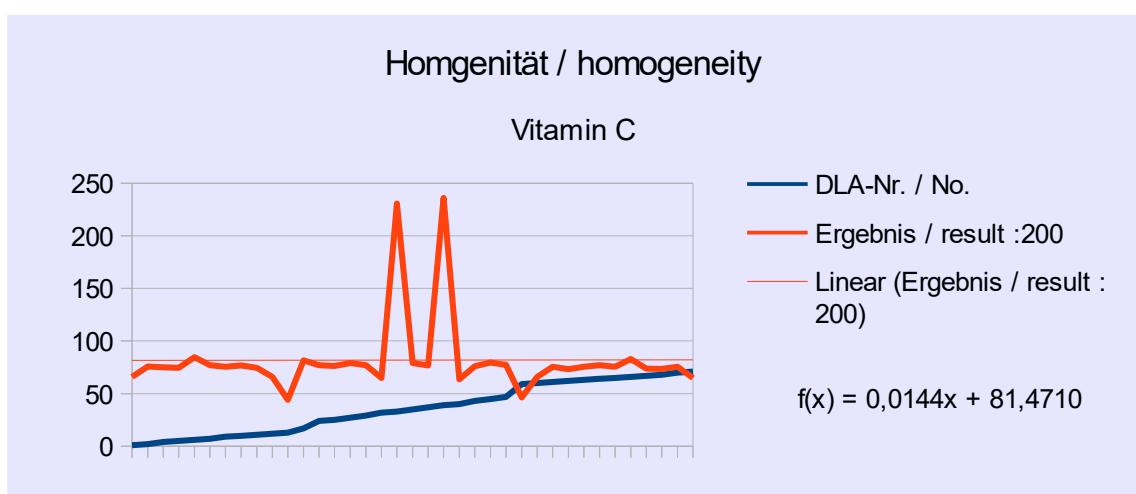
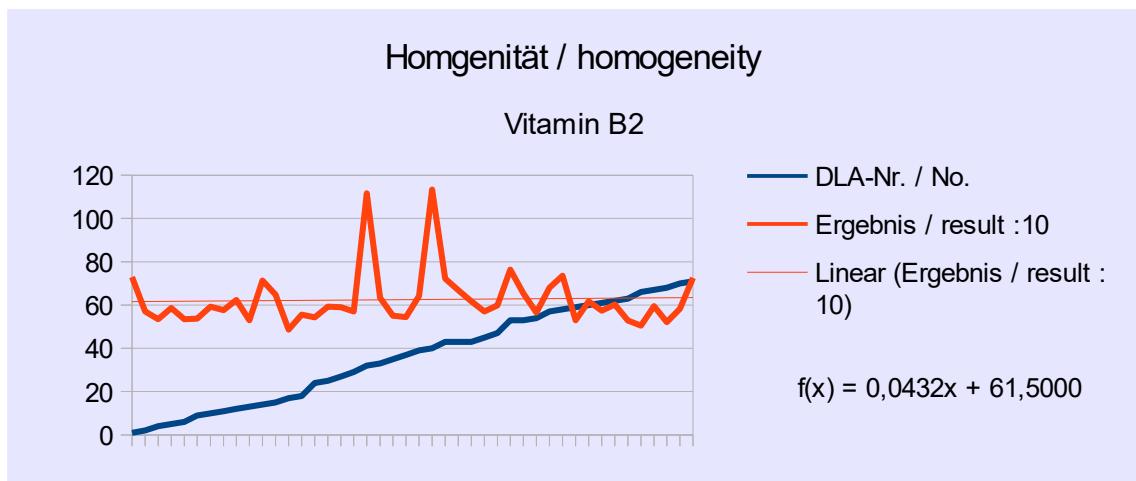


Abb./Fig. 19: Trendfunktion Probennummern vs. Ergebnisse: Vitamin B2, Vitamin C und Biotin (1/10, 1/200 und 1/1000 dargestellt)
trend line function sample number vs. results: vitamin B2, vitamin C and biotin (1/10, 1/200 and 1/1000 shown)

5.3 Kernel Density Plots of Results

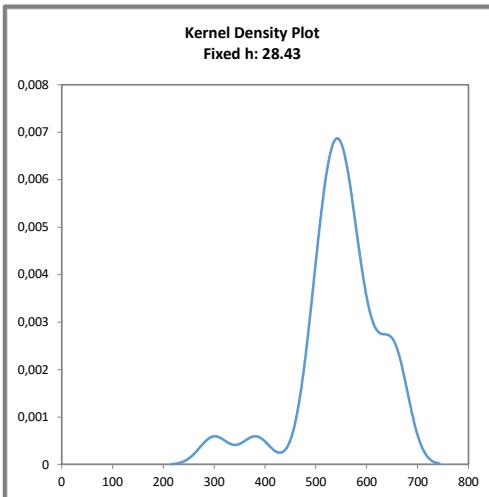
Abbildungen:

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

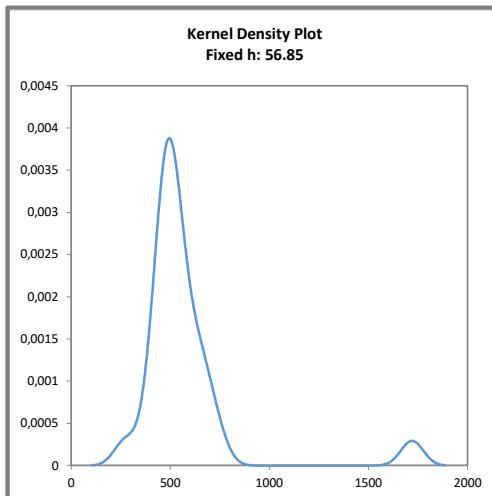
Figures:

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

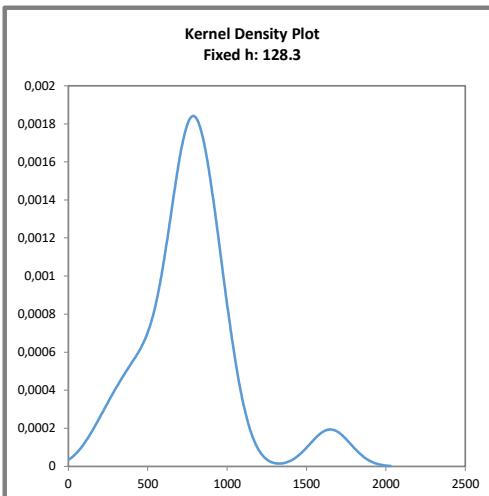
Vitamin B6



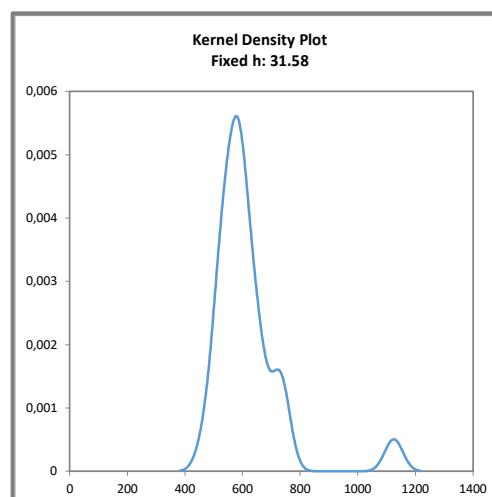
Vitamin B1



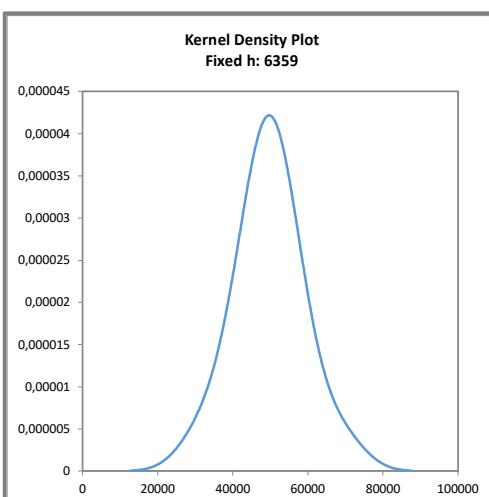
Vitamin B12



Vitamin B2



Biotin



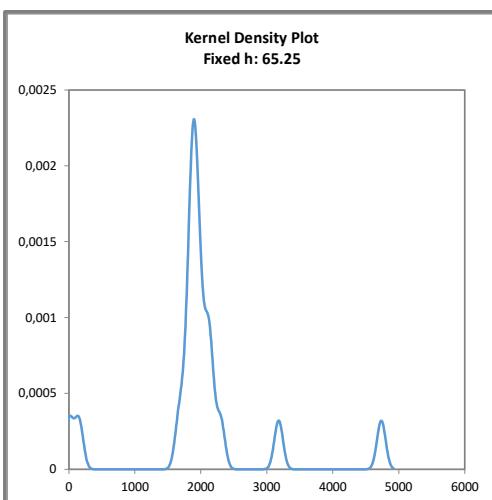
Abbildungen:

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

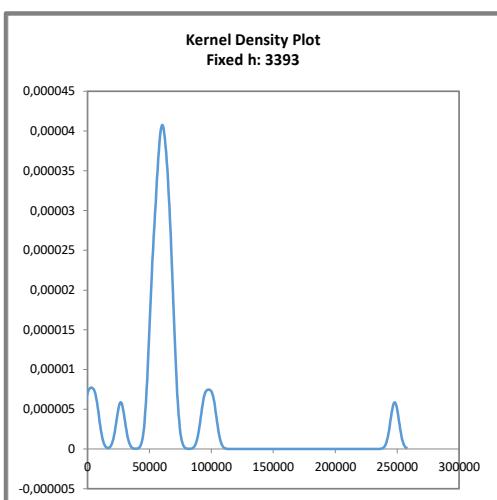
Figures:

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

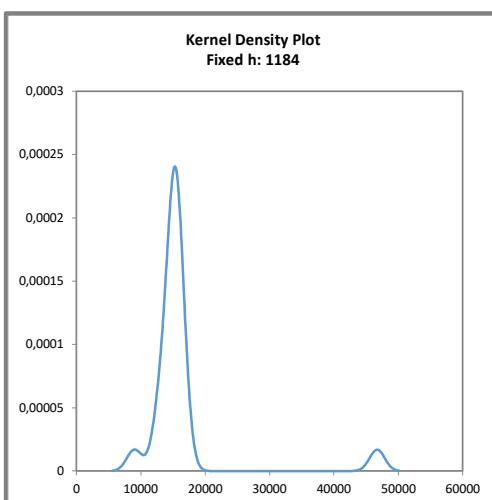
Pantothenensäure / Pantothenic Acid



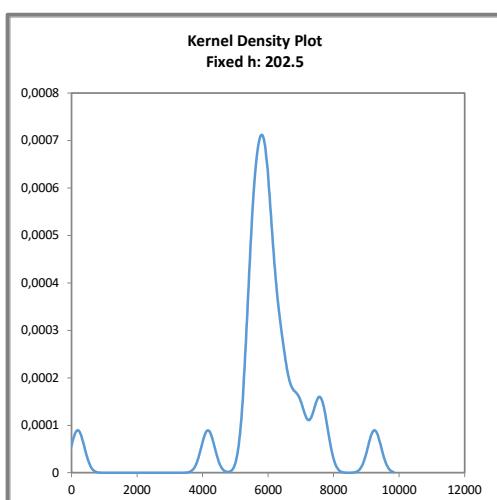
Folsäure / Folic Acid



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 ptsU01-2021
PT name	Food Supplement I: Vitamins B1, B2, B6, B12, Biotin, Vitamin C, Folic Acid, Niacin and Pantothenic Acid
Sample matrix*	<i>Samples I + II: Multi-vitamin capsule powder (without capsule shell) / ingredients: maltodextrin, vitamins and carrier: mannitol</i>
Number of samples and sample amount	2 identical samples I + II, 50 g each.
Storage	<i>Samples I + II: cooled 2 - 10°C</i>
Intentional use	<i>Laboratory use only (quality control samples)</i>
Parameter	<i>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 – 2 g)</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/100 g and µg/100 g, respectively (see results file)</i>
Number of significant digits	<i>at least 2</i>
Further information	<i>For information please specify:</i> <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
Result submission	<i>The result submission file should be sent by e-mail to: pt@dla-lvu.de</i>
Last Deadline	<i>the latest 25th June 2021</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</i>

* 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
		Germany
		GREAT BRITAIN
		Germany
		Germany
		Germany
		ITALY
		CYPRUS
		Germany
		Germany
		POLAND
		BELGIUM
		Germany
		Germany
		Germany
		USA
		Germany
		USA
		Germany
		SPANIEN
		Germany
		Germany
		Germany

[Die Adressdaten der Teilnehmer wurden für die allgemeine Veröffentlichung des Auswertungsberichts 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 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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17. AOAC Official Methods of Analysis: Guidelines for Standard Method Performance Requirements, Appendix F, p. 2, AOAC Int (2016)
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 (einschließlich 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

- 23.EN 14130:2003; Untersuchung von Lebensmitteln: Bestimmung von Vitamin C mit HPLC (zurückgezogen) / Foodstuffs. Determination of vitamin C by HPLC (withdrawn)
- 24.EN 15607:2009; Untersuchung von Lebensmitteln: Bestimmung von D-Biotin mit HPLC / Foodstuffs. Determination of d-biotin by HPLC
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