Proficiency Tests
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Evaluation Report
proficiency test

DLA 43/2017

Food Supplement I:

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

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

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Vertraulichkeit *Confidentiality*	Die Teilnehmerergebnisse sind im EP-Bericht in anonymisierter Form mit Auswertenummern benannt. Daten einzelner Teilnehmer werden ausschließlich nach vorheriger Zustimmung des Teilnehmers an Dritte weitergegeben. Participant result are named anonymously with evaluation numbers in the PT report. Data of individual participants will be passed on to third parties only with prior consent of the participant.

Inhalt / Content

1. Introduction

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

2. Realisation

2.1 Test material

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

After homogenization the samples were portioned to approximately 50 g into metallised PET film bags. The portions were numbered chronologically.

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 (carrier: maltodextrin), riboflavin, thiamine mononitrate, pyridoxine hydrochloride, folic acid and cyanocobalamin (carrier: mannitol) and biotin. 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	1160 mg
Vitamin B2	1430 mg
Vitamin B6	353 mg
Vitamin B12	2140 µg
Biotin	14300 µg
Vitamin C	21400 mg
Folic acid	214000 µg
Niacin	14300 mg
Pantothenic acid	6580 mg

2.1.1 Homogeneity

The **mixture homogeneity before bottling** was examined 10-fold by determination of the paramaters Folic acid, Niacinamide, Pantothenic acid, Vitamin B1, B2, and B6 by HPLC-DAD. The repeatability standard deviations were with 0,53 - 4,01% in the range of repeatability standard deviations of the standardized methods (e.g. ASU-Methods, s. 3.6.2) (see Table 4) [16-23]. 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 < 10% (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) [16-23].
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	8,24 %
Vitamin B2	2,39 %
Vitamin B6	5,66 %
Vitamin B12	7,54 %
Biotin	14,1 %
Vitamin C	3,47 %
Folic acid	6,92 %
Niacin	3,86 %
Pantothenic acid	4,12 %

Furthermore, the homogeneity was characterized by the **trend line function of participants' results for chronological bottled single samples**. The maximum deviations from the mean value of the trend line were 2,0 %, 1,3 % and 0,30 % for Vitamin B6, Vitamin C and Folic acid respectively corresponding to 28 %, 51 % and 1,9 % of the target standard deviation and thus they can be regarded low to acceptable. The slightly increased value for Vitamin C regarding the target standard deviation is due to the relatively low value of the target standard deviation of 2,5% of the mean. Considering the corresponding HorRat-Value from 1,6, this value is acceptable.

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

2.1.2 Stability

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

Furthermore, **participants' results** were compared with the corresponding **date of analysis** to characterize the stability of the PT-material during the whole time of analysis of the present PT by using the trendline-functions.
The maximum deviations from the mean value of the trend line for Vitamin B6, Vitamin C and Folic acid were at 2,0 %, 1,5 % and 5,4 % respectively corresponding to 28 %, 58 % and 33 % of the target standard deviation and thus they can be regarded as low to acceptable. The slightly increased value for Vitamin C regarding the target standard deviation is due to its relatively low value of 2,6% of the mean. Considering the corresponding HorRat-Value from 1,6, this value is acceptable.

2.2 Sample shipment and information to the test

Two portions of test material were sent to every participating laboratory in the 20th week of 2017. The testing method was optional. The tests should be finished at 30st June 2017 the latest.

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

The two portions contain identical samples of a food supplement with above mentioned parameters in the matrix of capsule powder. 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 5.5 Information on the PT)

2.3 Submission of results

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

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

Queried and documented were single results, recovery and the used testing

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

25 out of 27 participants submitted results in time. Two participants submitted no results.

3. Evaluation

3.1 Consensus value from participants (assigned value)

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

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

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

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

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

3.2 Robust standard deviation

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

3.3 Repeatability standard deviation

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

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

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

3.4 Reproducibility standard deviation

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

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

The relative reproducibility standard deviation 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, and results for a another proficiency test item can be removed from the data set [2]. Even if a result clearly deviates from the robust mean (e.g. factor >10) and has an influence on the robust statistics, a result can be excluded from statistical evaluation [3].

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

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

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

3.6 Target standard deviation (for proficiency assessment)

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

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

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

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

For valuation of all following paramaters in the present PT the target standard deviation according to the general model of Horwitz was applied (see 3.6.1): Vitamin B12, Biotin, Vitamin C und Pantothenic Acid.

The target standard deviation of the evaluation by precision experiment (s. 3.6.2) was considered for the following parameters: (ASU §64 / EN-Norms) [16, 17, 19, 20, 23]: Vitamin B1, B2, B6, Folic Acid and Niacin.

Additionally for Vitamin B12 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]:

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

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/or to provide additional information for the statistical data.

Table 4: Relative repeatability standard deviations (RSD_r) and relative reproducibility standard deviations (RSD_R) according to selected evaluations of tests for precision and the resulting target standard deviation σ_{pt} [16-23]

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 [22] EN 15607
	Infant-Milk powder	18,0 µg/100 g	11,6%	29,8%	27,5%	HPLC [22] EN 15607
	Animal feed	15-58 µg/100g	7,2-9,4%	9,4-22,4%*	-	HPLC-MS/MS [24]
Vitamin C	Breakfast cereals	102,6 mg/100g	9,9%	19,3%	18,0%	HPLC [21] EN 14130
	Milk powder	100,3 mg/100 g	6,3%	11,4%	10,5%[1]	HPLC [21] EN 14130
Niacin	Breakfast cereals (Choco)	21,03 mg/100g	1,1%	4,3%	4,23%	HPLC [23] EN 15652
	Milk powder	16,66 mg/100 g	2,8%	4,3%	3,82%[1]	HPLC [23] EN 15652
	Wheat flour	0,72 mg/100 g	3,9%	29,2%	29,1%	HPLC [23] EN 15652
Vitamin B1	Food supplement	486 mg/100g	8,0 %	15,4%	14,3%[1]	HPLC [16] ASU L00.00-83
	Chocolate powder	1,55 mg/100g	8,0%	18,1%	17,2%	HPLC [16] ASU L00.00-83
Vitamin B2	Food supplement	87,1 mg/100g	3,9%	6,8%	6,2%[1]	HPLC [17] ASU L00.00-84
	Chocolate powder	1,26 mg/100g	3,7%	10,3%	9,7%	HPLC [17] ASU L00.00-84
Vitamin B6	Baby food	0,106 mg/100g	3,8%	6,6%	6,3%[1]	HPLC [19] ASU L00.00-130
	Baby food	0,101 mg/100g	4,0%	5,9%	5,2%	HPLC [19] ASU L00.00-130
Folic acid	Milk powder	-	-	-	15,9	microbiological [20] 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 partly using the z'-scores.

Table 5 shows selected statistic data of participants results of present PT compared to PT results of previous years.

3.7 z-Score

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

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

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

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

The valid z-Score for each parameter is indicated as z-Score (σ_{pt}). The value indicated as z-Score (Info) only obtains a informative character. The both z-Scores were calculated with the different target standard deviations in accordance with 3.6.

3.7.1 Warning and action signals

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

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

Table 5: Characteristics of the present PT (on grey) in comparison to previous PTs (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-capsule powder	215 mg/100g	31,1 mg/100g	14,5%	1,1	DLA 26/2013
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 B2	Multivitamin-capsule powder	201 mg/100g	30,1 mg/100g	15,0%	2,2	DLA 26/2013
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 B6	Multivitamin-capsule powder	188 mg/100g	27,4 mg/100g	14,6%	1,9	DLA 26/2013
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 B12	Multivitamin-capsule powder	185 µg/100g	80,0 µg/100g	43,2%	3,0	DLA 26/2013
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
Biotin	Multivitamin-capsule powder	23100 µg/100g	5500 µg/100g	23,8%	1,6	DLA 33/2015
Biotin	Multivitamin-capsule 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

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-capsule powder	13900 µg/100g	3790 µg/100g	27,3%	1,7	DLA 26/2013
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
Niacin	Multivitam-in-capsule powder	3100 mg/100g	115 mg/100g	3,71%	1,1	DLA 33/2015
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
Pantothenic acid	Multivitam-in-capsule powder	1060 mg/100g	99 mg/100g	9,34%	1,5	DLA 33/2015
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
Vitamin C	Multivitam-in-capsule powder	11200 mg/100g	951 mg/100g	8,49%	1,9	DLA 48/2016
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

3.8 z'-Score

The z'-score can be used for the valuation of the results of the participants, in cases the standard uncertainty has to be considered (s. 3.8). The z'-score represents the relation of the deviation of the result (x) of the participant from the respective consensus value (X) to the square root of quadrat sum of the target standard deviation (σ_{pt}) and the standard uncertainty (Ux_{pt}) [3].

The calculation is performed by:

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

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

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

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

For warning and action signals see 3.7.1.

3.9 Reproducibility coefficient of variation (CV)

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

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

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

3.10 Quotient S*/σ_{pt}

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

3.11 Standard uncertainty of the assigned value

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

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

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

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

4. Results

Comments to the distribution of the results:

The kernel density plots showed for all parameters nearly a normal distribution of results (figures see documentation 5.4). 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 Vitamin B12 and Pantothenic acid the distribution of results showed an increased variability with quotients above 2,0. These parameters were evaluated considering the standard uncertainty by z´-scores. For all other parameters the distribution showed a normal variability of results.

The quotients S^*/σ_{pt} or S^*/σ_{pt}' referring to the applied kind of target standard deviation were for all parameters in the range of 1,1 to 2,0 except for pantothenic acid with a quotient S^*/σ_{pt}' of 2,9 (s. Tab. 5).

The robust standard deviation 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.

The quotient $U(X_{pt})/\sigma_{pt}$ is increased with > 0,3 for all parameters (0,31 to 0,81). Five of these quotients were slightly increased in the range up to < 0,5 and three quotients at approx. 0,6 and one quotient above 0,6.

60% 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 91% to 112% of the vitamin contents according to the manufacturer specifications.

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 $U(X_{pt})$
Quotient $U(X_{pt})/\sigma_{pt}$ or $U(X_{pt})/\sigma_{pt}$'
Number of results in the target range
Percent in the target range

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

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

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

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

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

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	18*
Number of outliers	0
Mean	1290
Median	1300
Robust Mean (X)	**1290**
Robust standard deviation (S*)	**205**
Number with 2 replicates	18
Repeatability SD (S_r)	107
Repeatability (CV_r)	8,24%
Reproducibility SD (S_R)	210
Reproducibility (CV_R)	16,3%
Target range:	
Target standard deviation σ_{pt}	**185**
Target standard deviation (for Info	49,8
lower limit of target range	**923**
upper limit of target range	**1670**
Quotient S/σ_{pt}*	*1,1*
Standard uncertainty $U_{(Xpt)}$	*60*
Quotient $U_{(Xpt)}/\sigma_{pt}$	*0,33*
Results in the target range	*17*
Percent in the target range	*94%*

** Result no. 9 excluded*

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	1330	34,9	0,19	0,70	
2	1450	157	0,85	3,2	
3					
4	1150	-144	-0,78	-2,9	
5	1500	206	1,1	4,1	
6	1500	204	1,1	4,1	
7	1140	-153	-0,82	-3,1	
8	1320	21,4	0,12	0,43	
9	3,21				Ergebnis ausgeschlossen / Result excluded
10	1420	126	0,68	2,5	
11	1400	110	0,59	2,2	
12	1480	183	1,0	3,7	
13					
14	1220	-77,8	-0,42	-1,6	
15	1010	-283	-1,5	-5,7	
16	1070	-226	-1,2	-4,5	
17	1250	-48,1	-0,26	-1,0	
18					
19					
20					
21	1200	-98,6	-0,53	-2,0	
22	1280	-12,6	-0,07	-0,25	
23					
24	899	-395	-2,1	-7,9	
25	1660	369	2,0	7,4	

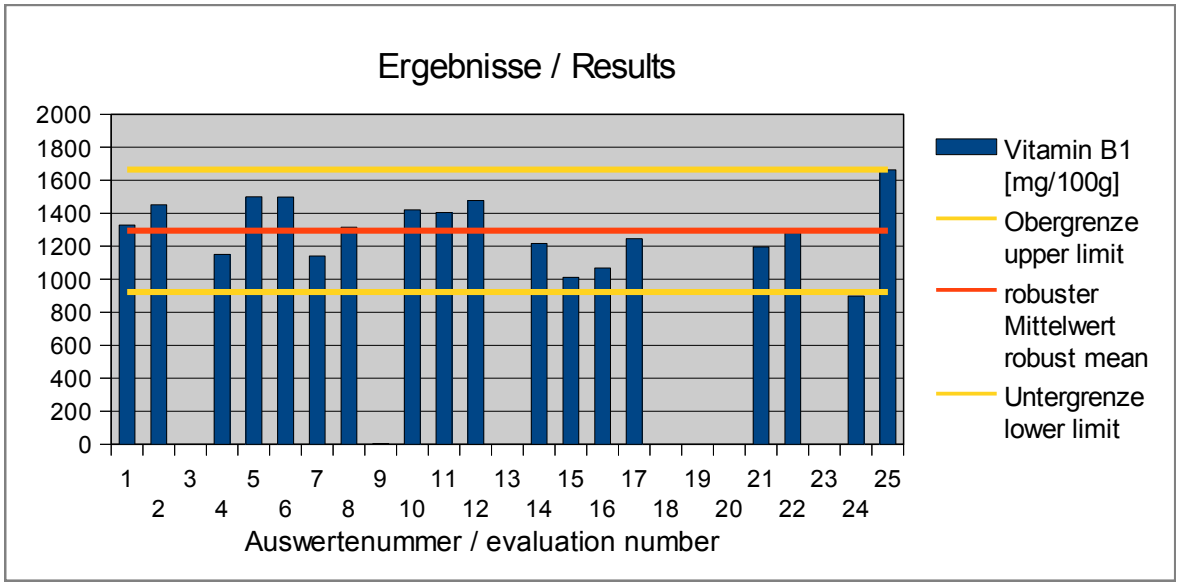


Abb. / Fig. 1: Ergebnisse Vitamin B1 / Results Vitamin B1

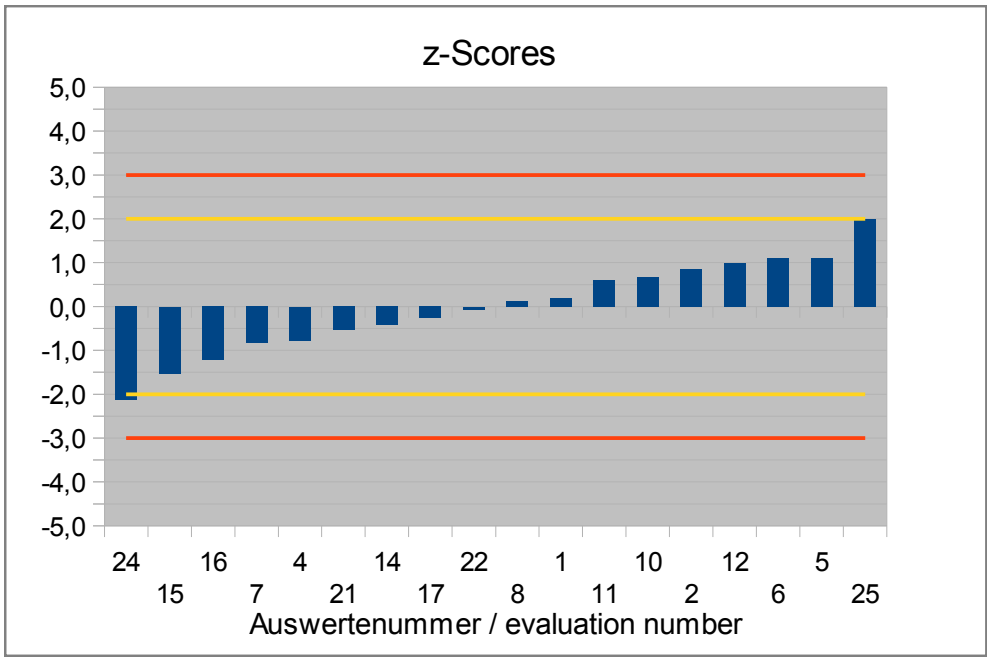


Abb. / Fig. 2: Z´-Scores Vitamin B1

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

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	15*
Number of outliers	2
Mean	1320
Median	1310
Robust Mean (X)	**1320**
Robust standard deviation (S*)	**111**
Number with 2 replicates	14
Repeatability SD (S_r)	30,7
Repeatability (CV_r)	2,39%
Reproducibility SD (S_R)	143
Reproducibility (CV_R)	11,2%
Target range:	
Target standard deviation σ_{pt}	**81,8**
Target standard deviation (for Information)	50,5
lower limit of target range	**1150**
upper limit of target range	**1480**
Quotient S/σ_{pt}*	*1,4*
Standard uncertainty $U_{(Xpt)}$	*35,9*
Quotient $U_{(Xpt)}/\sigma_{pt}$	*0,44*
Results in the target range	*12*
Percent in the target range	*80%*

** Results no. 1, 4, 9 and 25 excluded*

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	584				Ergebnis ausgeschlossen / Result excluded
2	1250	-69,8	-0,85	-1,4	
3	1280	-41,7	-0,51	-0,82	
4	607				Ergebnis ausgeschlossen / Result excluded
5	1270	-46,8	-0,57	-0,93	
6	1380	67,2	0,82	1,3	
7	1380	62,7	0,77	1,2	
8	1060	-253	-3,1	-5,0	
9	0,981				Ergebnis ausgeschlossen / Result excluded
10	1320	7,21	0,088	0,14	
11					
12	1440	118	1,4	2,3	
13					
14	1430	115	1,4	2,3	
15	1310	-4,79	-0,06	-0,09	
16	913	-404	-4,9	-8,0	Ausreisser / Outlier
17	1370	52,2	0,64	1,0	
18					
19					
20					
21	1270	-49,8	-0,61	-1,0	
22	1270	-42,8	-0,52	-0,85	
23					
24	1790	471	5,8	9,3	Ausreisser / Outlier
25	262				Ergebnis ausgeschlossen / Result excluded

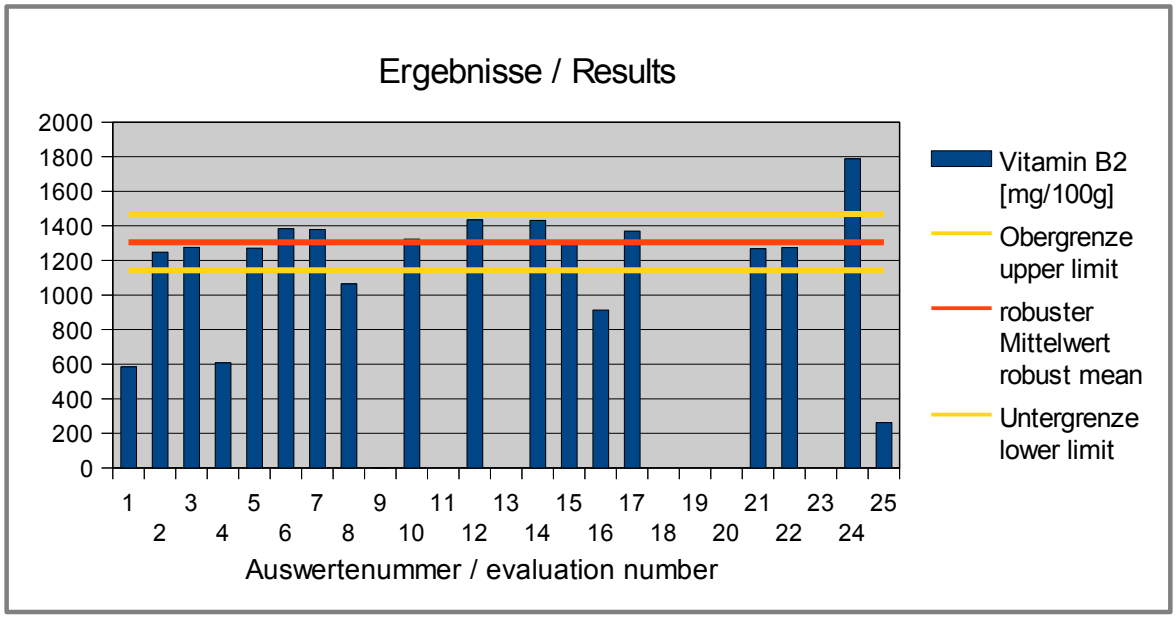


Abb. / Fig. 3: Ergebnisse Vitamin B2 / Results Vitamin B2

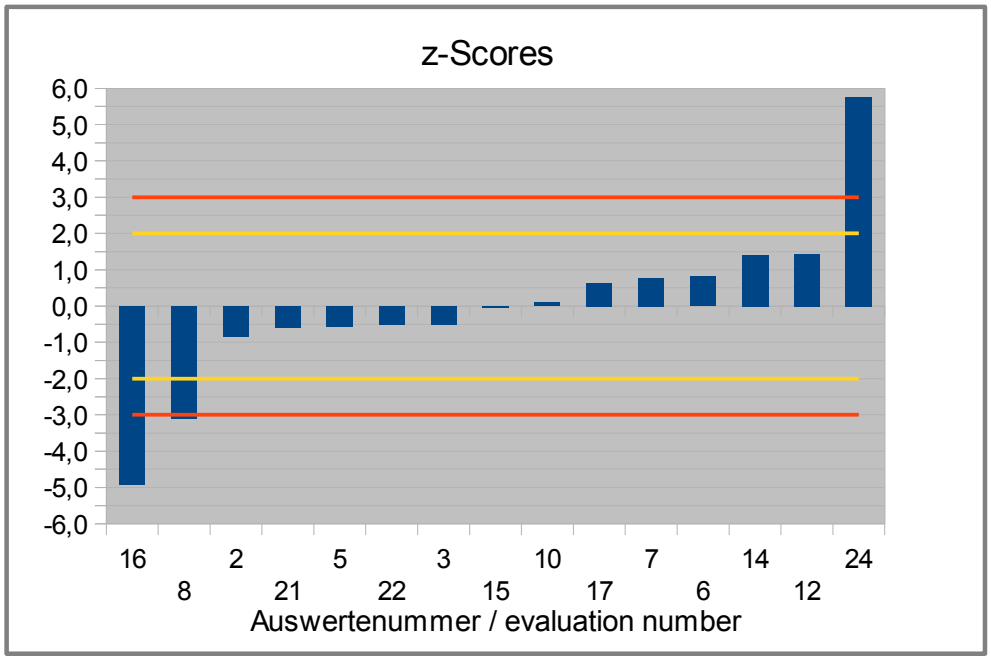


Abb. / Fig. 4: Z-Scores Vitamin B2

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

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	20
Number of outliers	1
Mean	381
Median	379
Robust Mean (X)	**377**
Robust standard deviation (S*)	**36,9**
Number with 2 replicates	19
Repeatability SD (S_r)	22,0
Repeatability (CV_r)	5,89%
Reproducibility SD (S_R)	36,0
Reproducibility (CV_R)	9,6%
Target range:	
Target standard deviation σ_{pt}	**26,1**
Target standard deviation (for Information)	17,5
lower limit of target range	**325**
upper limit of target range	**429**
Quotient S/σ_{pt}*	*1,4*
Standard uncertainty $U_{(Xpt)}$	*10,3*
Quotient $U_{(Xpt)}/\sigma_{pt}$	*0,396*
Results in the target range	*17*
Percent in the target range	*85%*

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	380	2,76	0,11	0,16	
2	335	-42,2	-1,6	-2,4	
3	521	144	5,5	8,2	Ausreisser / Outlier
4	381	3,86	0,15	0,22	
5	376	-1,24	-0,048	-0,071	
6	388	10,8	0,41	0,62	
7	382	4,26	0,16	0,24	
8					
9	418	40,8	1,6	2,3	
10	346	-31,2	-1,2	-1,8	
11	349	-28,0	-1,1	-1,6	
12	378	0,661	0,025	0,038	
13	336	-41,2	-1,6	-2,4	
14	386	8,58	0,33	0,49	
15	366	-11,2	-0,43	-0,64	
16	354	-23,2	-0,89	-1,3	
17	439	61,8	2,4	3,5	
18					
19					
20					
21	356	-21,5	-0,82	-1,2	
22	414	36,8	1,4	2,1	
23					
24	310	-66,8	-2,6	-3,8	
25	413	36,2	1,4	2,1	

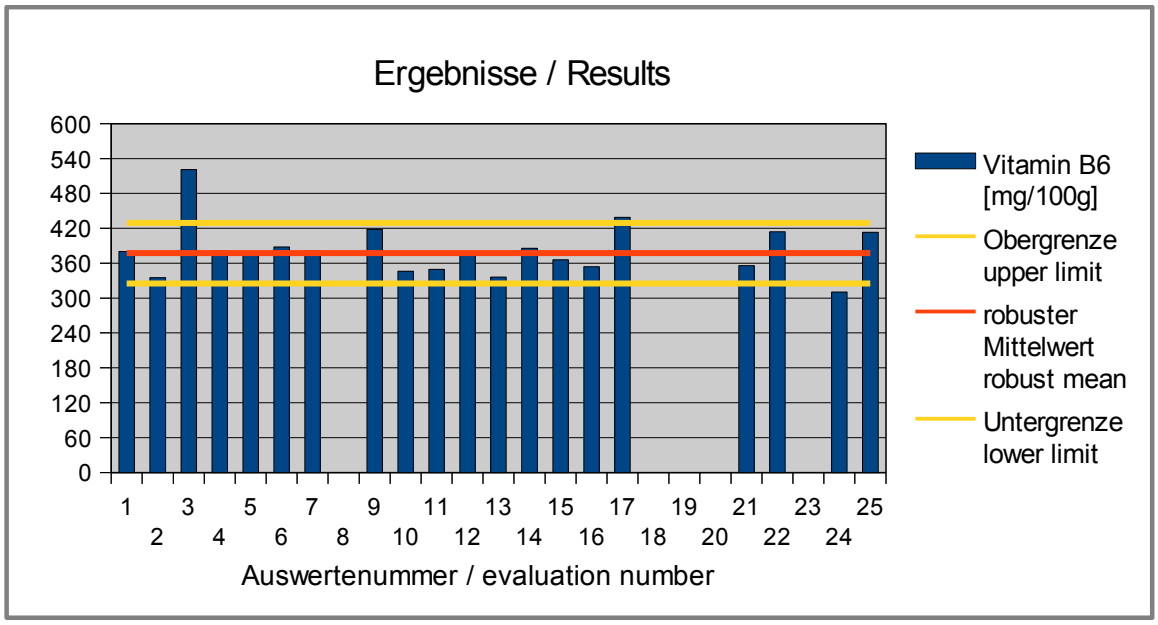


Abb. / Fig. 5: Ergebnisse Vitamin B6 / Results Vitamin B6

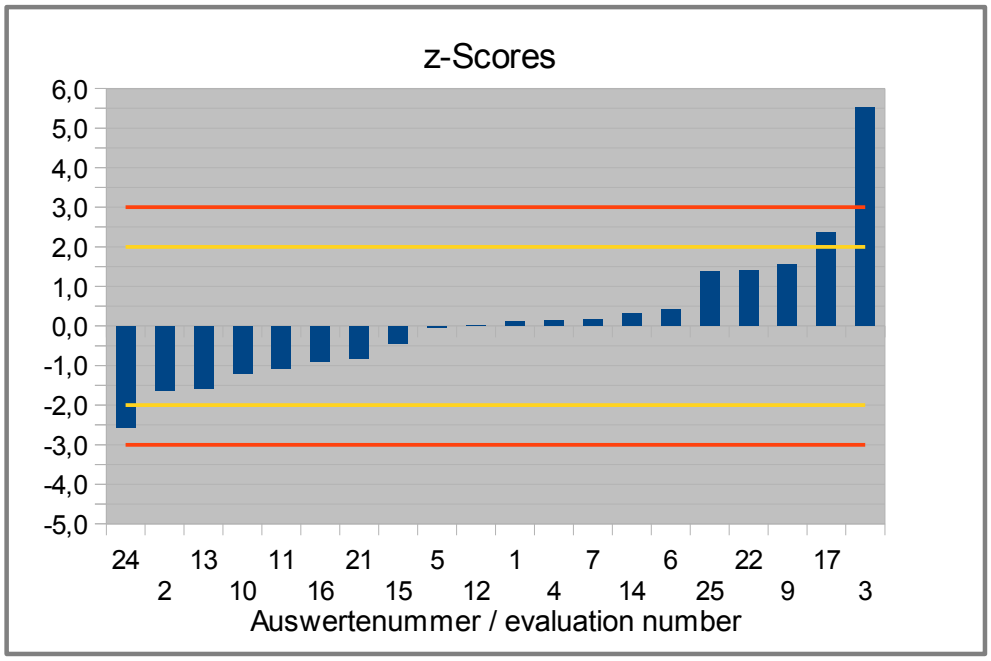


Abb. / Fig. 6: Z-Scores Vitamin B6

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

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	18
Number of outliers	1
Mean	2730
Median	2340
Robust Mean (X)	**2380**
Robust standard deviation (S*)	**597**
Number with 2 replicates	17
Repeatability SD (S_r)	174
Repeatability (CV_r)	7,54%
Reproducibility SD (S_R)	588
Reproducibility (CV_R)	25,5%
Target range:	
Target standard deviation σ_{pt}'	**294**
lower limit of target range	**1790**
upper limit of target range	**2960**
Quotient S/σ_{pt}'*	*2,0*
Standard uncertainty $U_{(Xpt)}$	*176*
Quotient $U_{(Xpt)}/\sigma_{pt}'$	*0,60*
Results in the target range	*13*
Percent in the target range	*72%*

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Vitamin B12 [µg/100g]	Abweichung [µg/100g] Deviation [µg/100g]	z´-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	1440	-934	-3,2	-4,0	
2					
3	387000				Ergebnis ausgeschlossen / Result excluded
4					
5	2350	-25,0	-0,08	-0,11	
6	3440	1070	3,6	4,5	
7	2380	0,0215	0,0	0,0	
8	3160	785	2,7	3,3	
9	1020	-1360	-4,6	-5,8	
10	2510	137	0,47	0,58	
11	2810	433	1,5	1,8	
12	1920	-453	-1,5	-1,9	
13					
14	1970	-401	-1,4	-1,7	
15	2470	97,0	0,33	0,41	
16	10000	7650	26	32	Ausreisser / Outlier
17	2650	275	0,93	1,2	
18					
19	2220	-155	-0,53	-0,66	
20					
21	1930	-450	-1,5	-1,9	
22	2270	-107	-0,37	-0,46	
23	2290	-81,9	-0,28	-0,35	
24	2320	-55,0	-0,19	-0,23	
25					

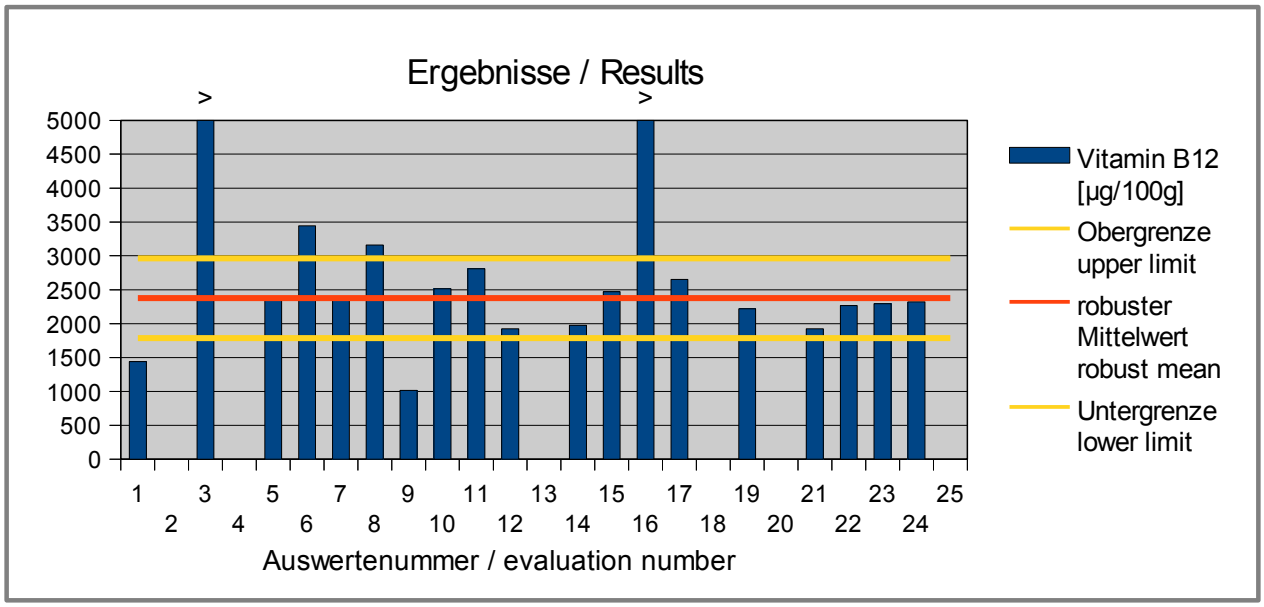


Abb. / Fig. 7: Ergebnisse Vitamin B12 **/** Results Vitamin B12

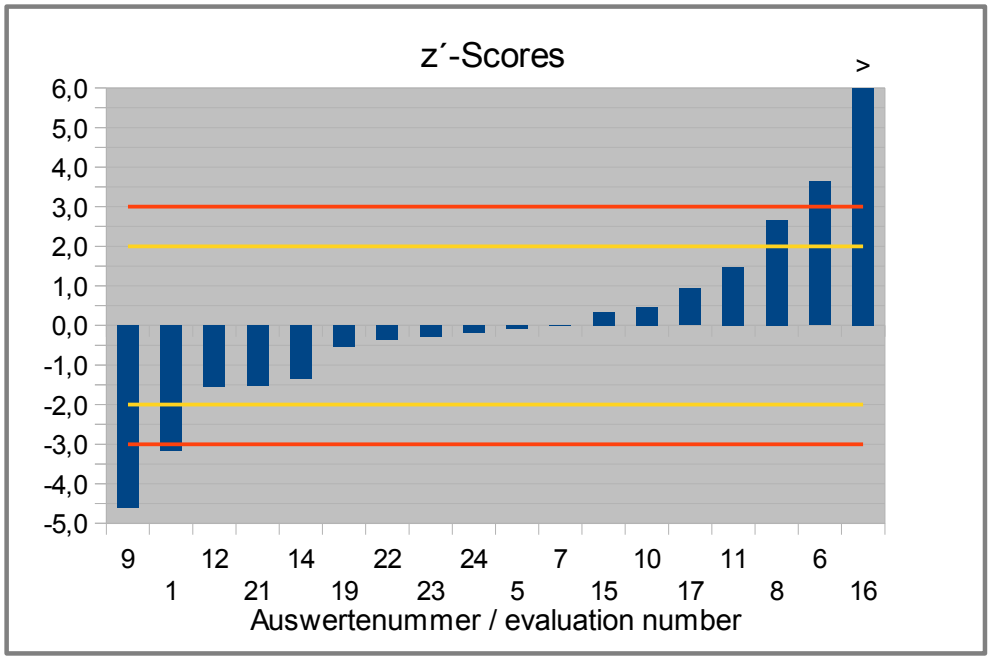


Abb. / Fig. 8: Z´-Scores Vitamin B12

4.5 Biotin (in µg/100g)

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	13
Number of outliers	2
Mean	21600
Median	15000
Robust Mean (X)	**15000**
Robust standard deviation (S*)	**1840**
Number with 2 replicates	11
Repeatability SD (S_r)	2040
Repeatability (CV_r)	14,1%
Reproducibility SD (S_R)	2730
Reproducibility (CV_R)	18,9%
Target range:	
Target standard deviation σ_{pt}	**1130**
Target standard deviation (for Information)	2570
lower limit of target range	**12700**
upper limit of target range	**17300**
Quotient S/σ_{pt}*	*1,6*
Standard uncertainty $U_{(Xpt)}$	*639*
Quotient $U_{(Xpt)}/\sigma_{pt}$	*0,57*
Results in the target range	*10*
Percent in the target range	*77%*

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	15500	495	0,44	0,19	
2					
3	956000				Ergebnis ausgeschlossen / Result excluded
4					
5	16000	994	0,88	0,39	
6					
7					
8	22800	7760	6,9	3,0	Ausreisser / Outlier
9	15900	849	0,75	0,33	
10					
11					
12	14400	-588	-0,52	-0,23	
13	14200	-796	-0,70	-0,31	
14	15000	-49	-0,04	-0,02	
15	12200	-2760	-2,4	-1,1	
16	98900	83900	74	33	Ausreisser / Outlier
17					
18					
19					
20					
21	13100	-1900	-1,7	-0,7	
22	14000	-1030	-0,91	-0,40	
23	15400	394	0,35	0,15	
24	13900	-1100	-1,0	-0,43	
25					

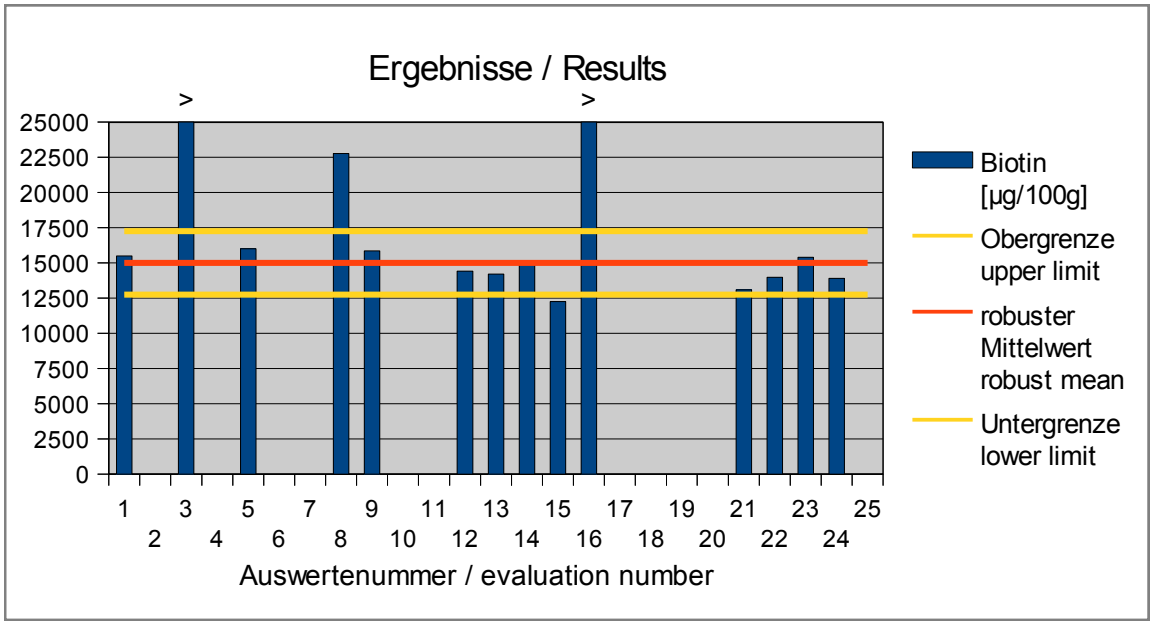


Abb. / Fig. 9: Ergebnisse Biotin / Results Biotin

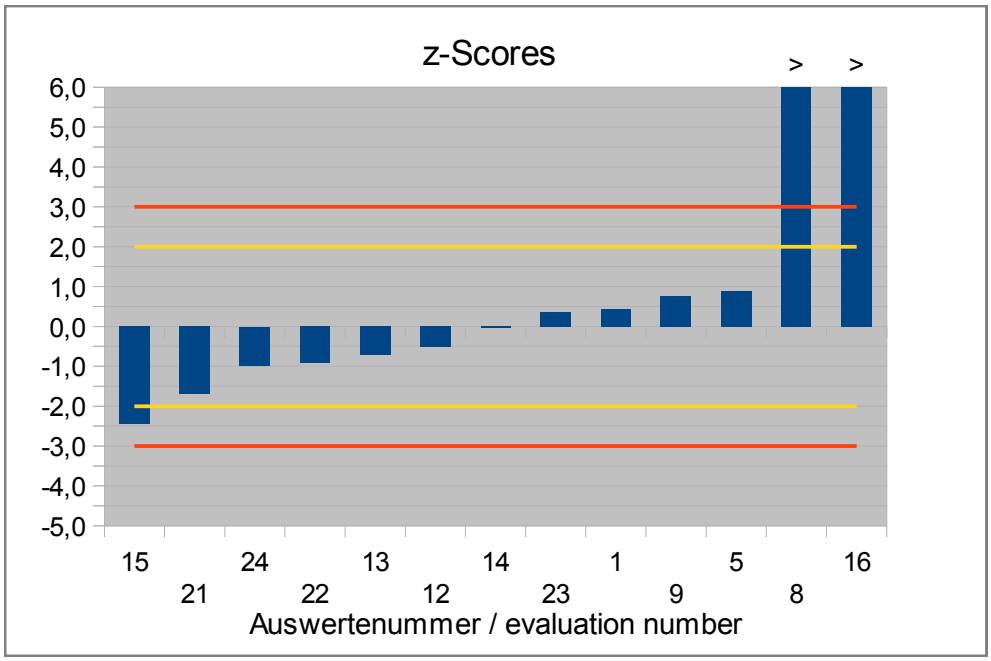


Abb. / Fig. 10: Z-Scores Biotin

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

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	23
Number of outliers	2
Mean	20200
Median	21200
Robust Mean (X)	**21200**
Robust standard deviation (S*)	**839**
Number with 2 replicates	21
Repeatability SD (S_r)	739
Repeatability (CV_r)	3,47%
Reproducibility SD (S_R)	921
Reproducibility (CV_R)	4,33%
Target range:	
Target standard deviation σ_{pt}	**535**
Target standard deviation (for Information)	1317
lower limit of target range	**20100**
upper limit of target range	**22300**
Quotient S/σ_{pt}*	*1,6*
Standard uncertainty $U_{(Xpt)}$	*219*
Quotient $U_{(Xpt)}/\sigma_{pt}$	*0,41*
Results in the target range	*19*
Percent in the target range	*83%*

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	21700	463	0,86	0,35	
2	20900	-339	-0,63	-0,26	
3	20700	-524	-1,0	-0,40	
4	22200	955	1,8	0,73	
5	22200	1000	1,9	0,76	
6	21500	310	0,58	0,23	
7	19100	-2080	-3,9	-1,6	
8	20200	-989	-1,8	-0,75	
9	206				Ergebnis ausgeschlossen / Result excluded
10	22100	1080	2,0	0,82	
11	21100	-97	-0,18	-0,07	
12	21300	97	0,18	0,07	
13	20800	-357	-0,67	-0,27	
14	22100	934	1,7	0,71	
15	21100	-97	-0,18	-0,07	
16	21900	705	1,3	0,53	
17	21000	-179	-0,34	-0,14	
18	20800	-435	-0,81	-0,33	
19					
20	21200	35	0,06	0,03	
21	11200	-10000	-19	-7,6	Ausreisser / Outlier
22	22000	789	1,5	0,6	
23	21200	16	0,03	0,01	
24	21600	410	0,76	0,31	
25	6140	-15100	-28	-11,4	Ausreisser / Outlier

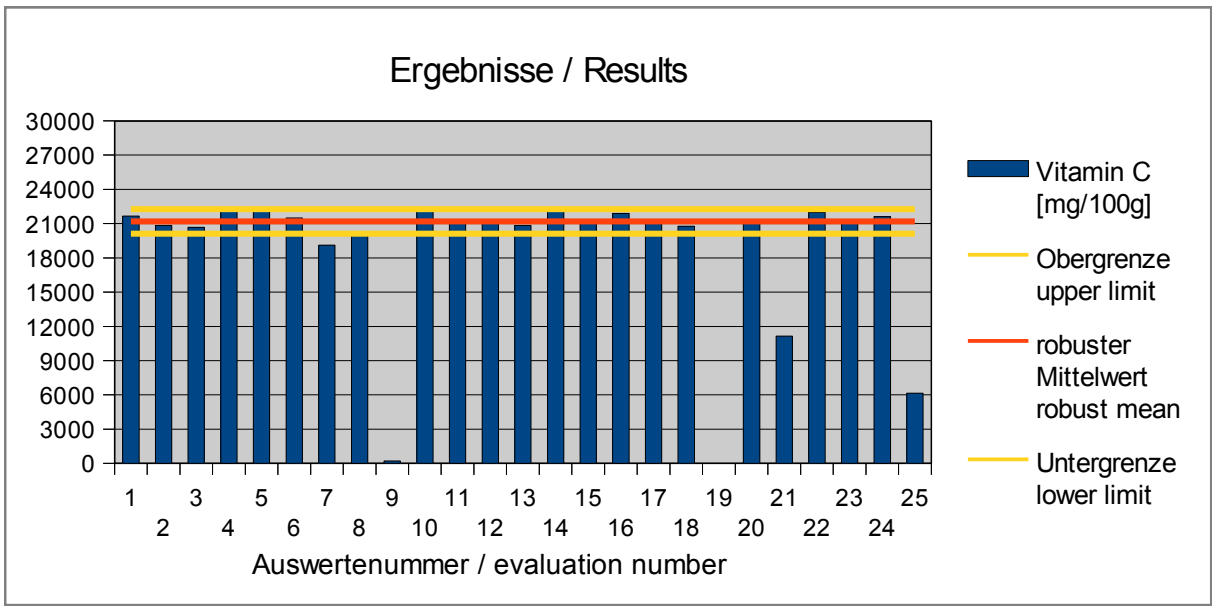


Abb. / Fig. 11: Ergebnisse Vitamin C / Results Vitamin C

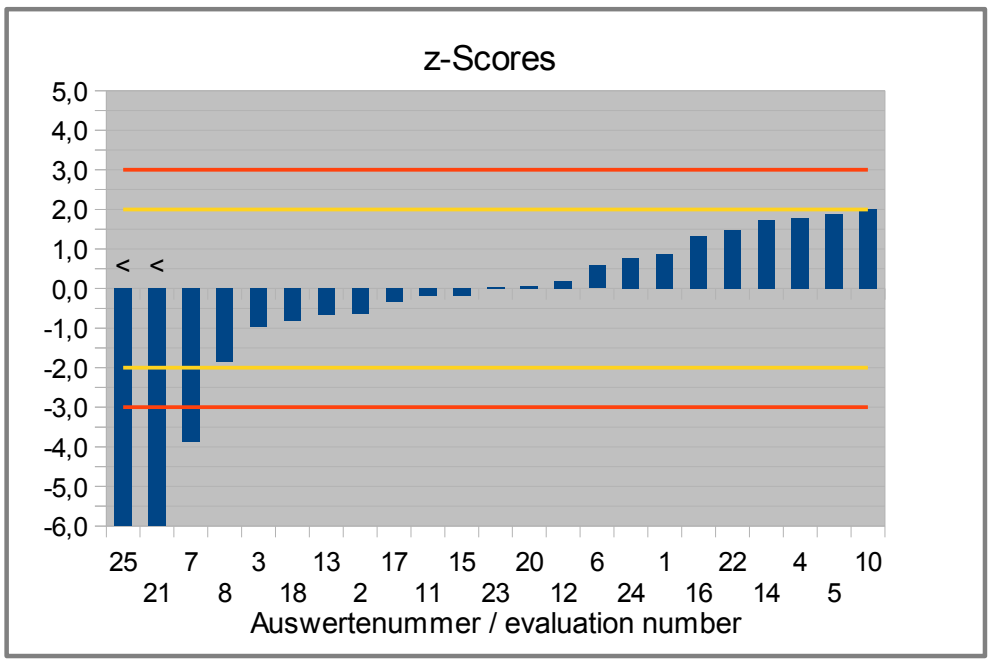


Abb. / Fig. 12: Z-Scores Vitamin C

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

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	16
Number of outliers	0
Mean	225000
Median	223000
Robust Mean (X)	**226000**
Robust standard deviation (S*)	**39900**
Number with 2 replicates	16
Repeatability SD (S_r)	15600
Repeatability (CV_r)	6,92%
Reproducibility SD (S_R)	45800
Reproducibility (CV_R)	20,4%
Target range:	
Target standard deviation σ_{pt}	**36700**
Target standard deviation (for Information)	11300
lower limit of target range	**153000**
upper limit of target range	**299000**
Quotient S/σ_{pt}*	*1,1*
Standard uncertainty $U_{(Xpt)}$	*12500*
Quotient $U_{(Xpt)}/\sigma_{pt}$	*0,34*
Results in the target range	*13*
Percent in the target range	*81%*

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})	z-Score (Info)	Hinweis Remark
1					
2	7730				Ergebnis ausgeschlossen / Result excluded
3	245000	18400	0,50	1,6	
4	260000	33700	0,92	3,0	
5	190000	-36100	-1,0	-3,2	
6	206000	-20500	-0,56	-1,8	
7	317000	90900	2,5	8,0	
8					
9	6490				Ergebnis ausgeschlossen / Result excluded
10	219000	-7490	-0,2	-0,66	
11	145000	-81386	-2,2	-7,2	
12	228000	2050	0,06	0,18	
13	201				Ergebnis ausgeschlossen / Result excluded
14	232000	6240	0,17	0,55	
15	211000	-14700	-0,40	-1,3	
16	145000	-81000	-2,2	-7,2	
17					
18					
19	239000	13300	0,36	1,2	
20					
21	217000	-9350	-0,25	-0,83	
22	212000	-13900	-0,38	-1,2	
23	270000	44300	1,2	3,9	
24	270000	43800	1,2	3,9	
25	3850				Ergebnis ausgeschlossen / Result excluded

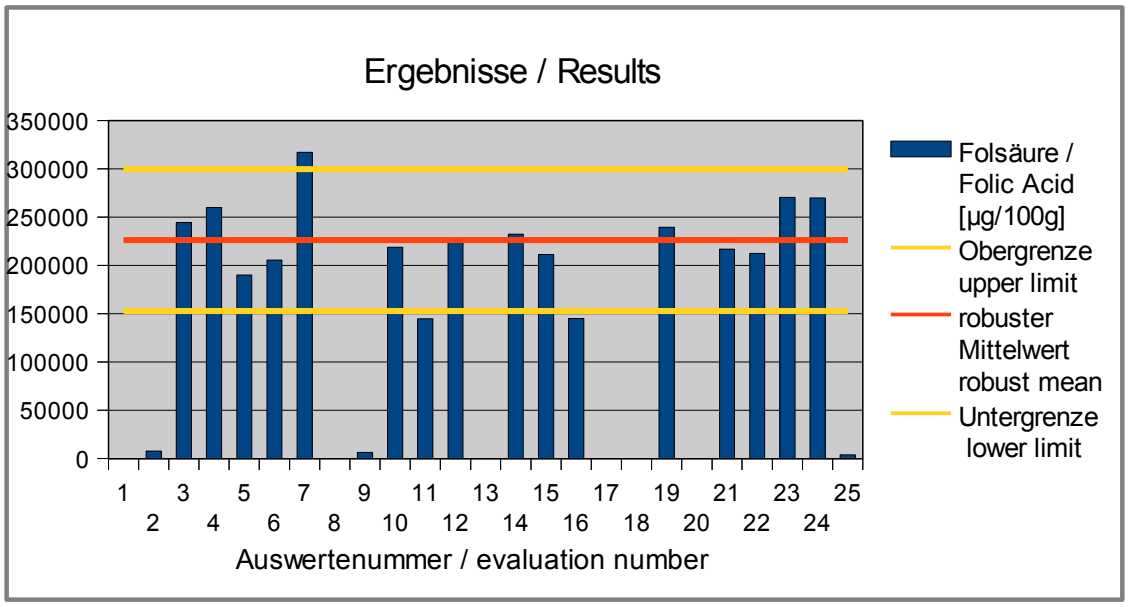


Abb. / Fig. 13: Ergebnisse Folsäure / Results Folic Acid

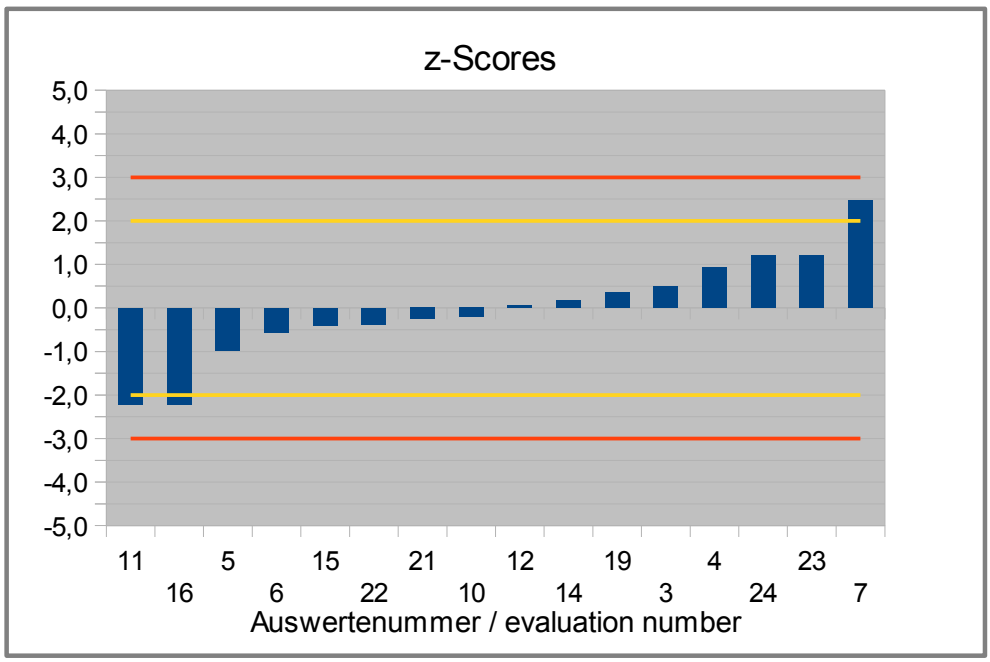


Abb. / Fig. 14: Z-Scores Folsäure / Folic Acid

4.8 Niacin (in mg/100g)

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	15
Number of outliers	0
Mean	14400
Median	14600
Robust Mean (X)	**14400**
Robust standard deviation (S*)	**1150**
Number with 2 replicates	15
Repeatability SD (S_r)	555
Repeatability (CV_r)	3,86%
Reproducibility SD (S_R)	1120
Reproducibility (CV_R)	7,79%
Target range:	
Target standard deviation σ_{pt}	**607**
Target standard deviation (for Information)	384
lower limit of target range	**13100**
upper limit of target range	**15600**
Quotient S/σ_{pt}*	*1,9*
Standard uncertainty $U_{(Xpt)}$	*371*
Quotient $U_{(Xpt)}/\sigma_{pt}$	*0,61*
Results in the target range	*12*
Percent in the target range	*80%*

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	27700				Ergebnis ausgeschlossen / Result excluded
2					
3	15400	1070	1,8	2,8	
4	13300	-1090	-1,8	-2,8	
5	13700	-653	-1,1	-1,7	
6	14600	289	0,48	0,75	
7	21,7				Ergebnis ausgeschlossen / Result excluded
8	15200	799	1,3	2,1	
9	90,6				Ergebnis ausgeschlossen / Result excluded
10	12600	-1730	-2,9	-4,5	
11	12600	-1740	-2,9	-4,5	
12	14900	500	0,82	1,3	
13	15000	652	1,1	1,7	
14	13900	-503	-0,83	-1,3	
15	13800	-598	-1,0	-1,6	
16	14800	397	0,65	1,0	
17	16300	1980	3,3	5,2	
18					
19					
20					
21	15100	730	1,2	1,9	
22	14500	128	0,21	0,33	
23					
24	122				Ergebnis ausgeschlossen / Result excluded
25					

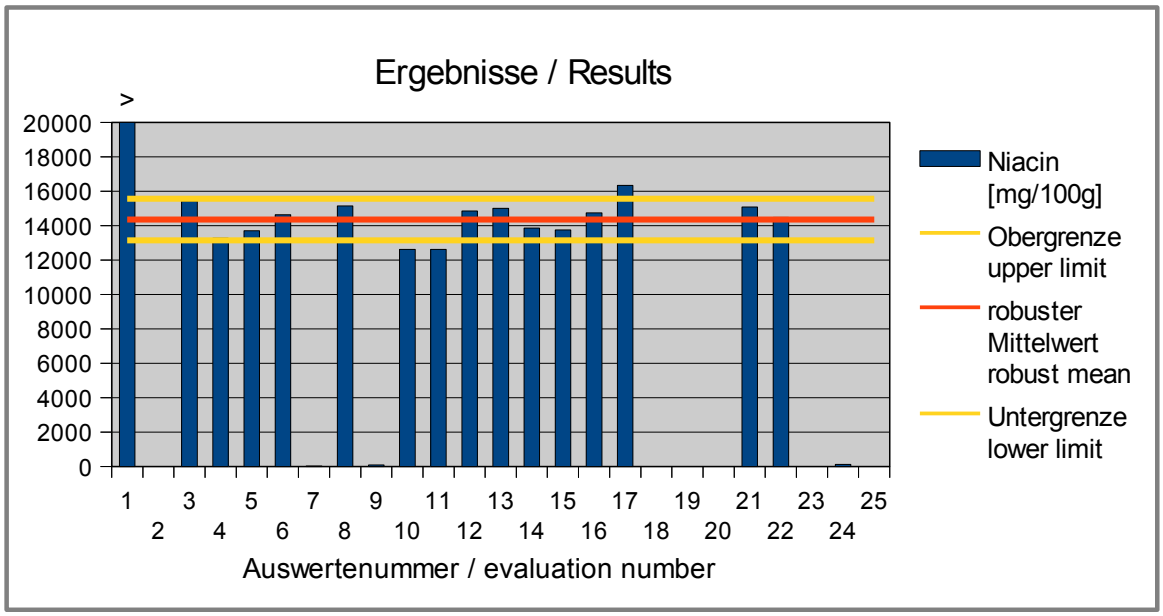


Abb. / Fig. 15: Ergebnisse Niacin / Results Niacin

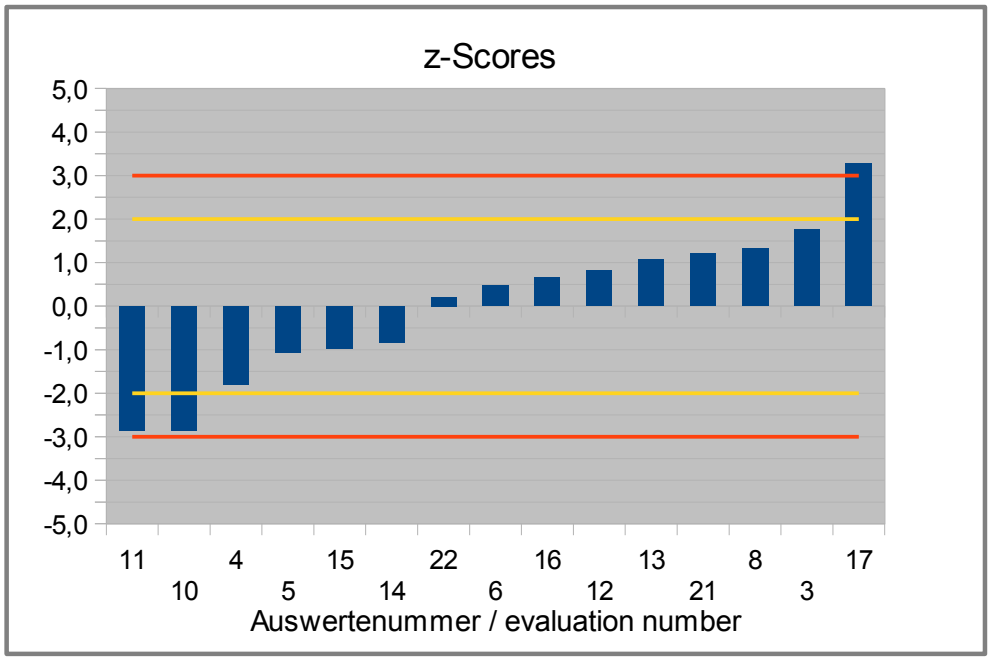


Abb. / Fig. 16: Z-Scores Niacin

4.9 Pantothenic acid (in mg/100g)

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	20
Number of outliers	2
Mean	6990
Median	7040
Robust Mean (X)	**7110**
Robust standard deviation (S*)	**1040**
Number with 2 replicates	18
Repeatability SD (S_r)	293
Repeatability (CV_r)	4,12%
Reproducibility SD (S_R)	850
Reproducibility (CV_R)	11,9%
Target range:	
Target standard deviation $\sigma_{pt'}$	**360**
lower limit of target range	**6390**
upper limit of target range	**7830**
Quotient S/$\sigma_{pt'}$*	*2,9*
Standard uncertainty $U_{(Xpt)}$	*291*
Quotient $U_{(Xpt)}/\sigma_{pt'}$	*0,81*
Results in the target range	*12*
Percent in the target range	*60%*

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Pantothensäure / Pantothenic Acid [mg/100g]	Abweichung [mg/100g] Deviation [mg/100g]	z´-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	7100	-8,69	-0,02	-0,04	
2					
3	8470	1360	3,8	6,4	
4	7150	37,3	0,10	0,18	
5					
6	6420	-690	-1,9	-3,3	
7	7290	185	0,51	0,87	
8	7160	49,8	0,14	0,24	
9	1010	-6100	-17	-28,8	Ausreisser / Outlier
10	7320	214	0,60	1,0	
11	6260	-849	-2,4	-4,0	
12	6980	-128	-0,35	-0,60	
13	6760	-347	-1,0	-1,6	
14	6790	-321	-0,89	-1,5	
15	6420	-684	-1,9	-3,2	
16	7800	692	1,9	3,3	
17	8790	1690	4,7	8,0	
18					
19					
20					
21	6660	-445	-1,2	-2,1	
22	6150	-955	-2,7	-4,5	
23	8540	1430	4,0	6,8	
24	6000	-1110	-3,1	-5,2	
25	10700	3580	9,9	16,9	Ausreisser / Outlier

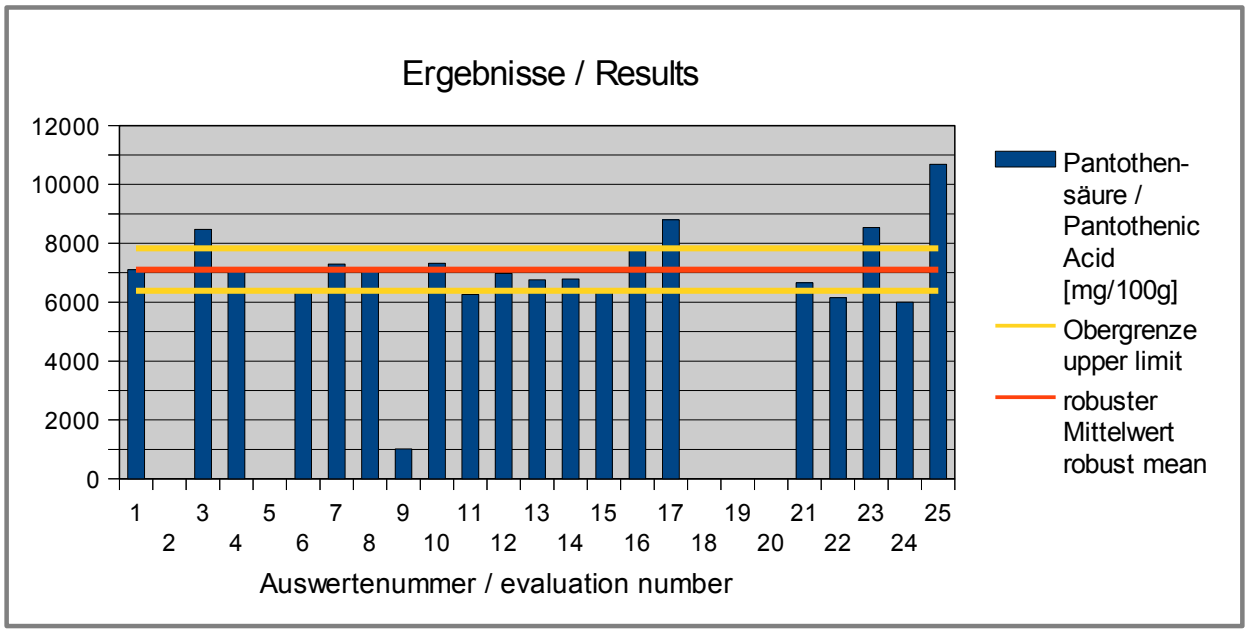


Abb. / Fig. 17: Ergebnisse Pantothensäure / Results Pantothenic Acid

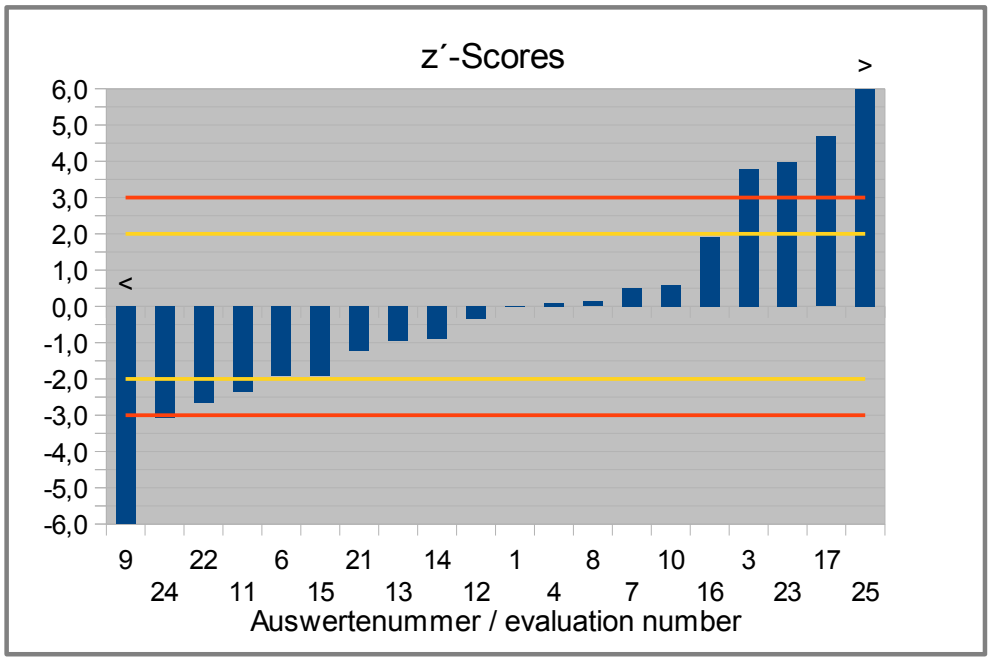


Abb. / Fig. 18: Z´-Scores Pantothensäure / Pantothenic Acid

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

Analyte	Participant	Unit	Sample No. 1	Sample No. 2	Date of analysis	Result (Mean)	Result 1	Result 2	Limit of determination	Incl. RR	Recovery rate [%]
Vitamin B1	1	mg/100g	82	37	20.06.17	1329	1302	1356	0.02ug/g	no	n.a.
	2	mg/100g	52	68	01.06.17	1451	1474	1427	0,03	no	-
	3	mg/100g	50	71							
	4	mg/100g	46	74	22.06.17	1150,1	1108,2	1192	70	no	98
	5	mg/100g	24	96	12.06.17	1500	1490	1500	0,05	no	N/A
	6	mg/100g	12	108	29.06.	1498	1503	1493	0,6	no	107,2
	7	mg/100g	20	100	22.06.17	1141,5	1174	1109	250	no	95,75
	8	mg/100g	51	69	23.05.17	1315,5	1296	1335	100	no	-
	9	mg/100g	6	114	06.06.17	3,21	3,21	3,22		yes	80
	10	mg/100g	13	107	June 2017	1420	1417	1422	0,02	no	101/100
	11	mg/100g	84	36	19.06.17	1404	1655	1154	0,1	yes	90
	12	mg/100g	28	92	15.06.17	1477,55	1446,39	1508,72	5mg/100g	no	
	13	mg/100g	31	89	22.06.17	6761	6861	6660		no	
	14	mg/100g	48	72		1216	1245	1187		no	
	15	mg/100g	61	59	30.05.17	1011	1026	994,9	50	no	
	16	mg/100g	16	104	20.06.17	1068,00	1073,00	1063,00	0,04	no	
	17	mg/100g	56	64	May	1246	1253	1240	Standard range: 0,012-0,060 mg /100 g (ml)		
	18	mg/100g	47	73							
	19	mg/100g	11	109	15.06.	239400	239400	239400,0	0,2	no	
	20	mg/100g	4	116							
	21	mg/100g	43	77	12.06.17	1195,48	1206,39	1184,6	80,0	no	no
	22	mg/100g	45	75	01.06.17	1281,5	1284	1279,0	n/a	no	n/a
	23	mg/100g	35	85	06.06.17	8537,5	8600,0	8475,0	0,0	no	
	24	mg/100g	21	99	28.06.2017	899	863	935	10	no	
	25	mg/100g	22	98	10.07.	1663	1486	1840	0,1	no	100

Analyte	Participant	Unit	Sample No. 1	Sample No. 2	Date of analysis	Result (Mean)	Result 1	Result 2	Limit of de-termination	Incl. RR	Recovery rate [%]
Vitamin B2	1	mg/100g	82	37	20.06.17	584	561	607	0.02ug/g	no	n.a.
	2	mg/100g	52	68	01.06.17	1247	1253	1241	0	no	-
	3	mg/100g	50	71	29.05.17	1275,12	1271,04	1279,21	0,01 µg/ml	yes	105,93
	4	mg/100g	46	74	22.06.17	607,1	603	611,2	70	no	98
	5	mg/100g	24	96	12.06.17	1270	1300	1240	0,05	no	N/A
	6	mg/100g	12	108	26.-28.06.	1384	1381	1386	0,03	no	107,2
	7	mg/100g	20	100	22.06.17	1379,5	1383	1376	500	no	100,12
	8	mg/100g	51	69	23.05.17	1063,5	1091	1036	100	no	-
	9	mg/100g	6	114	06.06.17	0,98	0,97	0,99		yes	80
	10	mg/100g	13	107	June 2017	1324	1336	1311	0,03	no	100/102
	11	mg/100g	84	36	19.06.17	1404	1655	1154	0,1	yes	90
	12	mg/100g	28	92	15.06.17	1434,51	1385,89	1470,98	5mg/100g	no	
	13	mg/100g	31	89	22.06.17	6761	6861	6660		no	
	14	mg/100g	48	72		1432	1441	1423		no	
	15	mg/100g	61	59	30.05.17	1312	1332	1291	10	no	
	16	mg/100g	16	104	20.06.17	913,00	896,00	931,00	0,10	no	
	17	mg/100g	56	64	May	1369	1372	1367	0,04 mg / 100 g (ml)		
	18	mg/100g	47	73							
	19	mg/100g	11	109	15.06.	239400	239400	239400,0	0,2	no	
	20	mg/100g	4	116							
	21	mg/100g	43	77	12.06.17	1267,03	1288,89	1245,2	80,0	no	no
	22	mg/100g	45	75	01.06.17	1274	1234	1314,0	n/a	no	n/a
	23	mg/100g	35	85	06.06.17	8537,5	8600,0	8475,0	0,0	no	
	24	mg/100g	21	99	28.06.2017	1788	1693	1883	10	no	
	25	mg/100g	22	98	10.07.	262,4	231,2	293,7	0,1	no	96

Analyte	Participant	Unit	Sample No. 1	Sample No. 2	Date of analysis	Result (Mean)	Result 1	Result 2	Limit of de-termination	Incl. RR	Recovery rate [%]
Vitamin B6	1	mg/100g	82	37	20.06.17	380	374	386	0.03ug/g	no	n.a.
	2	mg/100g	52	68	29.06.17	335	360	310	0,11	no	-
	3	mg/100g	50	71	25.05.17	520,94	513,94	527,94	0.15 µg/ml	yes	92,18
	4	mg/100g	46	74	22.06.17	381,1	377,8	384,4	70	no	98
	5	mg/100g	24	96	13.06.17	376	377	374	0,02	no	N/A
	6	mg/100g	12	108	26.-28.06.	388	387	389	0,05	no	107,2
	7	mg/100g	20	100	22.06.17	381,5	373	390	160	no	97,05
	8	mg/100g	51	69	23.05.17	1063,5	1091	1036	100	no	-
	9	mg/100g	6	114	06.06.17	418	416	420		yes	80
	10	mg/100g	13	107	June 2017	346	342	349	0,03	no	97/99
	11	mg/100g	84	36	26.06.17	349,2	340,4	358	0,1	yes	100
	12	mg/100g	28	92	15.06.17	377,9	385,97	371,44	5mg/100g	no	
	13	mg/100g	31	89	15.06.17	336	360	312		no	
	14	mg/100g	48	72		386	385	387		no	
	15	mg/100g	61	59	30.05.17	366	370,6	361,3	10	no	
	16	mg/100g	16	104	20.06.17	354,00	340,00	368,00	0,10	no	
	17	mg/100g	56	64	May	439	432	445	Standard range: 0,002 - 0,012 mg / 100 g (ml)		
	18	mg/100g	47	73							
	19	mg/100g	11	109	15.06.	239400	239400	239400,0	0,2	no	
	20	mg/100g	4	116							
	21	mg/100g	43	77	12.06.17	355,78	353,08	358,5	80,0	no	no
	22	mg/100g	45	75	01.06.17	414	417	411,0	n/a	no	n/a
	23	mg/100g	35	85	06.06.17	8537,5	8600,0	8475,0	0,0	no	
	24	mg/100g	21	99	28.06.2017	310,4	287	334	10	no	
	25	mg/100g	22	98	10.07.	413,4	365,3	461,6	0,1	no	95

Analyte	Participant	Unit	Sample No. 1	Sample No. 2	Date of analysis	Result (Mean)	Result 1	Result 2	Limit of de-termination	Incl. RR	Recovery rate [%]
Vitamin B12	1	mg/100g	82	37	30.06.17	1440,5	1403	1478	100ng/g	no	n.a.
	2	mg/100g	52	68	29.06.17	335	360	310	0,11	no	-
	3	mg/100g	50	71	23.06.17	387005	406752	367258	0.4 µg/ml	yes	100,93
	4	mg/100g	46	74	22.06.17	381,1	377,8	384,4	70	no	98
	5	mg/100g	24	96	06.06.17	2350	1900	2800	0,2	no	N/A
	6	mg/100g	12	108	21.-22.06.	3444	3362	3526	300	no	107,2
	7	mg/100g	20	100	28.06.17	2375	2431	2319	600	no	98,1
	8	mg/100g	51	69	23.05.17	3160	3050	3270	100	no	-
	9	mg/100g	6	114	06.06.17	1016	1118	913		yes	80
	10	mg/100g	13	107	'June 2017	2512	2582	2441	0,03	no	114
	11	mg/100g	84	36	26.06.17	2808	2837	2779	0,1	yes	100
	12	mg/100g	28	92	29.06.17	1922,47	1918,49	1926,44	10µg/100g	no	
	13	mg/100g	31	89	15.06.17	336	360	312		no	
	14	mg/100g	48	72		1974	2009	1940		no	
	15	mg/100g	61	59	30.05.17	2472	2381	2563	500	no	
	16	mg/100g	16	104	20.06.17	10020,00	9973,00	10060,00	2,00	no	
	17	mg/100g	56	64	22.05.17	2650	2619	2680	LOD: 0,5 µg/L		
	18	mg/100g	47	73							
	19	mg/100g	11	109	08.06.	2220	2250	2190,0	0,0	no	
	20	mg/100g	4	116							
	21	mg/100g	43	77	14.06.17	1925,42	1919,2	1931,6	100,0	no	
	22	mg/100g	45	75	15.06.17	2267,5	2226	2309,0	n/a	no	n/a
	23	mg/100g	35	85	07.06.17	2293,1	2267,9	2318,2	0,5	no	
	24	mg/100g	21	99	28.06.2017	2320	2340	2300	1000	no	
	25	mg/100g	22	98	10.07.	413,4	365,3	461,6	0,1	no	95

Analyte	Participant	Unit	Sample No. 1	Sample No. 2	Date of analysis	Result (Mean)	Result 1	Result 2	Limit of determination	Incl. RR	Recovery rate [%]
Biotin	1	µg/100g	82	37	30.06.17	15501	16284	14718	250ng/g	no	n.a.
	2	µg/100g	52	68	01.09.16	1292	1304	1280	10	no	-
	3	µg/100g	50	71	29.06.17	956124,75	913906,56	953547,29	1 µg/ml	yes	96,12
	4	µg/100g	46	74	29.08.2016	1387,28	1385,66	1388,36	70	no	98
	5	µg/100g	24	96	01.06.17	16000	11000	20000	50	no	N/A
	6	µg/100g	12	108	24.08.16	1254,33	1238,71	1269,94	33	no	107,2
	7	µg/100g	20	100	---	---	---	---	---	---	---
	8	µg/100g	51	69	23.05.17	22766	22832	22701	100	no	-
	9	µg/100g	6	114	08.06.17	15855	16360	15350		yes	80
	10	µg/100g	13	107	not determined	not determined	not determined	not determined	cancelled	cancelled	cancelled
	11	µg/100g	84	36							
	12	µg/100g	28	92	29.06.17	14417,58	13845,1	14990,05	10µg/100g	no	
	13	µg/100g	31	89	16.06.17	14210	14516	13904		no	
	14	µg/100g	48	72		14956	15324	14588		no	
	15	µg/100g	61	59	30.05.17	12248	11857	12639	500	no	
	16	µg/100g	16	104	20.06.17	98900,00	99800,00	98000,00	2,00	no	
	17	µg/100g	56	64	-	not tested	not tested	not tested			
	18	µg/100g	47	73							
	19	µg/100g	11	109							
	20	µg/100g	4	116							
	21	µg/100g	43	77	13.06.17	13098,62	13492,6	12704,6	200,0	no	
	22	µg/100g	45	75	15.06.17	13975	14151	13799,0	n/a	no	n/a
	23	µg/100g	35	85	06.06.17	15400,0	14950,0	15850,0	0,1	no	
	24	µg/100g	21	99	28.06.2017	13900	13100	14600	10000	no	
	25	µg/100g	22	98							

Analyte	Participant	Unit	Sample No. 1	Sample No. 2	Date of analysis	Result (Mean)	Result 1	Result 2	Limit of determination	Incl. RR	Recovery rate [%]
Vitamin C	1	mg/100g	82	37	24.06.17	21658	21382	21934	2ug/g	no	n.a.
	2	mg/100g	52	68	28.06.17	20856	21165	20547	0,5	no	-
	3	mg/100g	50	71	16.06.17	20671,71	20407,97	20935,44	0.9 µg/ml	yes	96,52
	4	mg/100g	46	74	29.06.17	22150,8	21737,9	22563,6	70	no	98
	5	mg/100g	24	96	07.06.17	22200	22800	21500	0,5	no	N/A
	6	mg/100g	12	108	20.06.17	21505	21594	21416	10	no	107,2
	7	mg/100g	20	100	14.06.17	19115	19980	18250	300	no	91,5
	8	mg/100g	51	69	23.05.17	20206	21390	19022	100	no	-
	9	mg/100g	6	114	06.06.17	206	201	211		yes	80
	10	mg/100g	13	107	June 2017	22272	22841	21703	0,04	no	102/101
	11	mg/100g	84	36	07.06.17	21098,38	21211	20985,76	0,1	no	100,3
	12	mg/100g	28	92	22.06.17	21292,33	20759,46	22358,08	5mg/100g	no	
	13	mg/100g	31	89	08.06.17	20838	20917	20758		no	
	14	mg/100g	48	72		22129	22273	21985		no	
	15	mg/100g	61	59	30.05.17	21098,4	21418,5	20778,3	5	no	
	16	mg/100g	16	104	06.06.17	21900,00	22064,00	21760,00	1,00	no	
	17	mg/100g	56	64	May	21016	21149	20884	7,8 mg / 100 g (ml)		
	18	mg/100g	47	73	06.06.17	20760	20488	21032,0	200,0	no	100
	19	mg/100g	11	109	08.06.	2220	2250	2190,0	0,0	no	
	20	mg/100g	4	116	20.06.17	21230	21330	21130,0	100,0	no	
	21	mg/100g	43	77	07.06.17	11151,81	11253,97	11049,7	0,0	yes	99,43
	22	mg/100g	45	75	15.06.17	21984	21913	22055,0	n/a	no	n/a
	23	mg/100g	35	85	07.06.17	21211,5	21304,5	21118,5	2500,0	no	98,7%
	24	mg/100g	21	99	28.06.2017	21605	22830	20380	100	no	
	25	mg/100g	22	98	10.07.	6140	5750	6530	0,25	no	99

Analyte	Participant	Unit	Sample No. 1	Sample No. 2	Date of analysis	Result (Mean)	Result 1	Result 2	Limit of de-termination	Incl. RR	Recovery rate [%]
Folsäure / Folic Acid	1	µg/100g	82	37	30.06.17	15501	16284	14718	250ng/g	no	n.a.
	2	µg/100g	52	68	26.06.17	7730	6620	8840	0,3	no	-
	3	µg/100g	50	71	18.05.17	244513,25	243310	245716,5	0.07 µg/ml	yes	98,86
	4	µg/100g	46	74	29.06.17	259781,3	253262,5	266300	70	no	98
	5	µg/100g	24	96	08.06.17	190000	190000	180000	20	no	N/A
	6	µg/100g	12	108	26.-28.06.	205610	202280	208940	3500	no	107,2
	7	µg/100g	20	100	28.06.17	316978	320100	313856	1600	no	98,9
	8	µg/100g	51	69	23.05.17	22766	22832	22701	100	no	-
	9	µg/100g	6	114	08.06.17	6488	4300	8675		yes	80
	10	µg/100g	13	107	June 2017	218634	228713	208554	0,16	no	98,2
	11	µg/100g	84	36	19.06.17	144735	117733	171737	0,1	yes	75
	12	µg/100g	28	92	15.06.17	228167,43	228318,94	228659,17	200µg/100g	no	
	13	µg/100g	31	89	06.06.17	201	205	197		no	
	14	µg/100g	48	72		232362	233061	231662		no	
	15	µg/100g	61	59	30.05.17	211400	212220	210580	10000	no	
	16	µg/100g	16	104	20.06.17	145100,00	144200,00	146000,00	100,00	no	
	17	µg/100g	56	64	-	not tested	not tested	not tested			
	18	µg/100g	47	73							
	19	µg/100g	11	109	15.06.	239400	239400	239400,0	0,2	no	
	20	µg/100g	4	116							
	21	µg/100g	43	77	12.06.17	216769,79	215885,89	217654,1	20000,0	yes	100,86
	22	µg/100g	45	75	07.06.17	212264,55	213817,8	210711,3	n/a	no	n/a
	23	µg/100g	35	85	06.06.17	270375,0	264750,0	276000,0	0,2	no	
	24	µg/100g	21	99	28.06.2017	269950	238600	301300	10000	no	
	25	µg/100g	22	98	10.07.	3849	3530	4168	100	no	98

Analyte	Participant	Unit	Sample No. 1	Sample No. 2	Date of analysis	Result (Mean)	Result 1	Result 2	Limit of determinati-on	Incl. RR	Recovery rate [%]
Niacin	1	mg/100g	82	37	22.06.17	27659	28343	26975	0.6ug/g	no	n.a.
	2	mg/100g	52	68	26.06.17	7730	6620	8840	0,3	no	-
	3	mg/100g	50	71	02.06.17	15422,57	15242,91	15602,24	1 µg/ml	yes	101,12
	4	mg/100g	46	74	22.06.17	13263,6	13402,2	13156,7	70	no	98
	5	mg/100g	24	96	09.06.17	13700	13600	13800	0,05	no	N/A
	6	mg/100g	12	108	26.-28.06.	14642	14708	14576	1	no	107,2
	7	mg/100g	20	100	29.06.17	21,75	22,53	20,96	5	no	98,2
	8	mg/100g	51	69	23.05.17	15152	14615	15689	100	no	-
	9	mg/100g	6	114	08.06.17	90,62	80,04	101,2		yes	80
	10	mg/100g	13	107	June 2017	12622	11334	13910	0,02	no	88,3/96,7
	11	mg/100g	84	36	26.06.17	12612	12677	12547	0,1	yes	98
	12	mg/100g	28	92	15.06.17	14852,56	14820,22	14884,9	5mg/100g	no	
	13	mg/100g	31	89	19.06.17	15005	15321	14688		no	
	14	mg/100g	48	72		13850	13890	13809		no	
	15	mg/100g	61	59	30.05.17	13755	13799	13710	10	no	
	16	mg/100g	16	104	20.06.17	14750,00	14704,00	14785,00	0,20	no	
	17	mg/100g	56	64	May	16337	16254	16420	Standard range: 0,016 - 0,160 mg / 100 g (ml)		
	18	mg/100g	47	73							
	19	mg/100g	11	109	15.06.	239400	239400	239400,0	0,2	no	
	20	mg/100g	4	116							
	21	mg/100g	43	77	12.06.17	15083,04	15158,68	15007,4	400,0	no	no
	22	mg/100g	45	75	01.06.17	14481	14903	14059,0	n/a	no	n/a
	23	mg/100g	35	85	06.06.17	270375,0	264750,0	276000,0	0,2	no	
	24	mg/100g	21	99	28.06.2017	121,5	115	128	10	no	
	25	mg/100g	22	98	10.07.	3849	3530	4168	100	no	98

Analyte	Participant	Unit	Sample No. 1	Sample No. 2	Date of analysis	Result (Mean)	Result 1	Result 2	Limit of de-termination	Incl. RR	Recovery rate [%]
Pantothen-säure / Pantothenic Acid	1	mg/100g	82	37	22.06.17	7099	7048	7150	1mg/g	no	n.a.
	2	mg/100g	52	68	26.06.17	7730	6620	8840	0,3	no	-
	3	mg/100g	50	71	24.05.17	8471,43	8447,36	8495,5	0,4 µg/ml	yes	100,29
	4	mg/100g	46	74	22.06.17	7145	7345,1	6944,9	70	no	98
	5	mg/100g	24	96	09.06.17	13700	13600	13800	0,05	no	N/A
	6	mg/100g	12	108	26.-29.06.	6418	6481	6355	1	no	107,2
	7	mg/100g	20	100	20.06.17	7292,5	7429	7156	40	no	100,5
	8	mg/100g	51	69	23.05.17	7157,5	7037	7278	100	no	-
	9	mg/100g	6	114	06.06.17	1010	982	1038		yes	80
	10	mg/100g	13	107	June 2017	7322	7386	7258	0,04	no	103
	11	mg/100g	84	36	26.06.17	6259	6976	5542	0,1	yes	115
	12	mg/100g	28	92	15.06.17	6979,98	7030,22	6929,74	5mg/100g	no	
	13	mg/100g	31	89	22.06.17	6761	6861	6660		no	
	14	mg/100g	48	72		6787	6859	6714		no	
	15	mg/100g	61	59	30.05.17	6424	6457	6390	100	no	
	16	mg/100g	16	104	20.06.17	7800,00	7765,00	7834,00	0,02	no	
	17	mg/100g	56	64	May	8794	8431	9156	0,04 mg / 100 g (ml)		
	18	mg/100g	47	73							
	19	mg/100g	11	109	15.06.	239400	239400	239400,0	0,2	no	
	20	mg/100g	4	116							
	21	mg/100g	43	77	16.06.17	6662,67	6734,97	6590,4	100,0	yes	98,76
	22	mg/100g	45	75	01.06.17	6153	6169	6137,0	n/a	no	n/a
	23	mg/100g	35	85	06.06.17	8537,5	8600,0	8475,0	0,0	no	
	24	mg/100g	21	99	28.06.2017	6000	5890	6110	10	no	
	25	mg/100g	22	98	10.07.	10687	9831	11543	0,1	no	101

5.1.2 Analytical Methods

Analyte	Participant	Method description	Sample preparation	Measuring method	Calibration / Reference material	Recovery with same matrix	Method accredited	Further remarks
Vitamin B1	1	In house developed method	Extraction through sonication	UPLC-DAD	External Standard	no	yes	
	2	DIN EN 14122:2006					yes	
	3	-	-	-	-	-	no	
	4	HPLC					yes	
	5		N/A	HPLC	N/A	no	yes	
	6	LAV 21.0010-02; HPLC-FLD					yes	
	7	2.019/006-04	Without enyzamtic clean up	---	Thiamine-HCl	no	yes	---
	8	HPLC-DAD				no	yes / no	
	9	ASU L 00.00-83			yes	yes	yes	
	10	ASU § 64 LFGB L 00.00-83 + L 00.00-84, 2006-12, modified, HPLC-FLD	none	none	PT-Material	yes	yes	none
	11		Sample diluted in H2O	LC-MS/MS		yes	yes	
	12	House method: L138	sauer gelöst	HPLC - DAD	Standard: Thiamine hydrochlorid	no	yes	
	13							
	14			HPLC		no	yes	
	15	House method		HPLC-DAD	external, Sigma Aldrich		yes	
	16	HPLC-FLD					yes	
	17		As per test instruction	VITA FAST P1006 Vitamin B 1			no	
	18							
	19							
	20							
	21	USP 36			no	no	yes	
	22	HPLC-UV	N/A	n/a	n/a	no	yes	
	23							
	24	In house method	Weighed about 2g of spl in 250ml added 5ml of 1M NaOH and diluent (750 water:240ml methanol:10ml gla acetic acid) sonicated and boiled 10 mts at 70°C, cool made up	AACC-89.90,FEFANA -via HPLC	Thiamine Nitrate	no	yes	as Thiamine Nitrate
	25	HPLC-UV	Extraction with Water			no	no	

Analyte	Participant	Method description	Sample preparation	Measuring method	Calibration / Reference material	Recovery with same matrix	Method accredited	Further remarks
Vitamin B2	1	In house developed method	Extraction through sonication	UPLC-DAD	External Standard	no	yes	
	2	DIN EN 14152:2006					yes	
	3	DIN EN 14152	-	HPLC	Calibration	yes	yes	
	4	HPLC					yes	
	5		N/A	HPLC	N/A	no	yes	
	6	LAV 21.0017-02; HPLC-FLD					yes	
	7	2.019/005-04	Without enzymatic clean up	---	Riboflavin	no	yes	---
	8	HPLC-DAD				no	yes / no	
	9	ASU L 00.00-84			yes	yes	yes	
	10	ASU § 64 LFGB L 00.00-83 + L 00.00-84, 2006-12, modified, HPLC-FLD	none	none	PT-Material	yes	yes	none
	11							
	12	House method: L138	acidic dissolved	HPLC - DAD	Standard: Riboflavin	no	yes	
	13							
	14			HPLC		no	yes	
	15	House method		HPLC-DAD	external, Sigma Aldrich		yes	
	16	HPLC-FLD					yes	
	17		As per test instruction	VITA FAST P1007 Vitamin B 2			no	
	18							
	19							
	20							
	21	USP 36	-	-	no	no	yes	
	22	HPLC-UV	N/A	n/a	n/a	no	yes	
	23							
	24		See Vit B1	AACC-89.90,FEFANA-by HPLC	Riboflavin	no	yes	as Riboflavin
	25	HPLC-UV	Extraction with Water			no	no	

Analyte	Participant	Method description	Sample preparation	Measuring method	Calibration / Reference material	Recovery with same matrix	Method accredited	Further remarks
Vitamin B6	1	In house developed method	Extraction through sonication	UPLC-DAD	External Standard	no	yes	no
	2	DIN EN 14663:2006					yes	
	3	DIN EN 14164	-	HPLC	Calibration	yes	yes	
	4	HPLC					yes	
	5		N/A	HPLC	N/A	No	Yes	
	6	LAV 21.0017-02; HPLC-FLD					yes	
	7	2.019/008-05	without enzymatic clean up	---	Pyridoxine-HCl	no	yes	---
	8	HPLC-DAD				no	yes / no	
	9	DIN EN 14164			yes	yes	yes	
	10	ASU § 64 LFGB L 00.00-97, 2006-12, HPLC-FLD	none	none	PT-Material	yes	yes	none
	11		Sample dissolved in H2O	LC-MS/MS		yes	yes	
	12	House method: L138	acidic dissolved	HPLC - DAD	Standard: Pyridoxine hydrochloride	no	yes	
	13	HPLC UV (House method)			calibration with 9 points	yes	no	
	14			HPLC		no	yes	
	15	House method		HPLC-DAD	external, Sigma Aldrich		yes	
	16	HPLC-FLD					yes	
	17		as per test instruction	VITA FAST P1008 Vitamin B 6			no	
	18							
	19							
	20							
	21	USP 36	-	-	no	no	yes	
	22	HPLC-UV	N/A	n/a	n/a	no	yes	
	23							
	24		see Vit B1	AACC-89.90,FEFANA -by HPLC	Pyridoxine HCl	no	yes	as Pyrioxine-HCl
	25	HPLC-UV	Extraction with water			no	no	

Analyte	Participant	Method description	Sample preparation	Measuring method	Calibration / Reference material	Recovery with same matrix	Method accredited	Further remarks
Vitamin B12	1	In house developed method	Extraction through sonication	LC-MS/MS	External Standard	no	no	no
	2							
	3	House method A.S. 06 2005-07	-	HPLC	Calibration	yes	yes	
	4							
	5		N/A	Biacore	N/A	no	yes	
	6	LAV 21.0057-01; HPLC-DAD					yes	
	7	Vitafast	---	---	Cyanocobalamine	no	no	---
	8	HPLC-DAD				no	yes / no	
	9	ELISA, Testkit r-biopharm			yes	yes	yes	
	10	R-Biopharm VitaFast Vitamin B12 P1002, 2011-06	none	none	PT-Material	yes	yes	none
	11		Sample soluted in H2O	LC-MS/MS		yes	yes	
	12	House method: L138	Immunuaffinitycolumns clean up Aufreinigung	HPLC - DAD	Standard: Cyanocobalamine	no	yes	
	13							
	14			HPLC		no	yes	
	15	House method		HPLC-DAD	external, Sigma Aldrich		yes	
	16	LC-MSMS					yes	
	17		as per test instructions	ELISA R 2103 FAST Vitamin B 12			no	
	18							
	19	Microbiological Microtiter-Platetest, VitaFast® Vitamin B12 - Cyanocobalamin, r-biopharm, Art. Nr.: P1002			N.I.S.T. SRM 3280		yes	
	20							
	21	HPLC	-	-	-	no	no	
	22	HPLC-UV	N/A	n/a	n/a	no	yes	
	23	internal Method ELISA (P4-02-04-01-0501)	as per test kit isntructions	as per test kit instructions			yes	RIDASCREEN®FAST Vitamin B12
	24	In house method	Weighed 1g of sample in 50ml added 30ml diluent (Buffer: methanol) pH 3.55 sonicate 10 mts, made up filter & inject.	AACC-89.90,FEFANA -by HPLC	Vitamin B12	no	yes	as Cyanocobalamine
	25							

Analyte	Participant	Method description	Sample preparation	Measuring method	Calibration / Reference material	Recovery with same matrix	Method accredited	Further remarks
Biotin	1	In house developed method	Extraction through sonication	LC-MS/MS	External Standard	no	no	
	2							
	3	House method Görtler A.S. 07 2005-07	-	HPLC	Calibration	yes	yes	
	4							
	5		N/A	Biacore	N/A	no	yes	
	6							
	7	---	---	---	---	---	---	---
	8	HPLC-DAD				no	yes / no	
	9	ELISA, Testkit r-biopharm			yes	yes	yes	
	10	cancelled	cancelled	cancelled	cancelled	cancelled	cancelled	cancelled
	11							
	12	House method: L138	Immunuaffinity coulmns clean up	HPLC - DAD	Standard: Biotin	no	yes	
	13	LC-MS/MS (House method)			calibration with 8 points	yes	not yet	
	14			HPLC		no	yes	
	15	House method		HPLC-DAD	external, Sigma Aldrich		yes	
	16	LC-MSMS					yes	
	17							
	18							
	19							
	20							
	21	LC-MS	-	-	-	no	yes	
	22	HPLC-UV	n/a	n/a	n/a	no	yes	yes
	23	Internal Method Microbiology (P2-02-02-02-0393)	as per test kit instructions	as per test kit instructions			yes	Vita Fast®Vitamin B7 (Biotin)
	24	In house method	Weighed about 1g of spl in 50ml added 1.5% opa 30ml and boiled 10 mts at 70°C,pipetted 5ml acetonitrile madeup with 1.5% opa	AACC-89.90,FEFANA -mittels HPLC	Biotin	no	yes	as Biotin
	25							

Analyte	Participant	Method description	Sample preparation	Measuring method	Calibration / Reference material	Recovery with same matrix	Method accredited	Further remarks
Vitamin C	1	In house developed method	Extraction through sonication	UPLC-DAD	External Standard	no	no	no
	2	DIN EN 14130:2003 mod.					yes	
	3	SLMB 62/14.2.1 2000-03	-	HPLC	Calibration	yes	yes	
	4	HPLC					yes	
	5		N/A	HPLC	N/A	no	yes	
	6	LAV 21.0052-01; HPLC-DAD					yes	
	7	enzymatic after r-biopharm	---	---	L-Ascorbic acid	no	yes	---
	8	HPLC-DAD				no	yes / no	
	9	enzymatic, Testkit r-biopharm			yes	yes	yes	
	10	Vitamin C in food stuff, HPLC-FLD	noen	none	PT-Material	yes	yes	none
	11	SLMB 703	Soluted in 100ml ultrapure water + H2SO4 solution + potassium iodide solution	Measurement of Redoxpotential			yes	
	12	House method: P44	Acidic dissolved	HPLC - DAD	Standard: Ascorbic acid	no	yes	
	13	HPLC UV (House method)			calibration with 8 points	yes	no	
	14			HPLC		no	yes	
	15	House method	Reduction to ascorbic acid	HPLC-UV	external, Sigma Aldrich		yes	
	16	HPLC-UV					yes	
	17		as per test isntruction	VITA FAST P1010 Vitamin C			no	
	18	HPLC/UV	Extraction with aqueous solvent	none	ext. Calibration with Reference standard Ascorbic acid	no	no	none
	19							
	20	HPLC					yes	
	21	USP 36	-	-	no	yes	yes	
	22	titration	N/A	n/a	n/a	no	yes	
	23	Internal Method Enzymatic (P4-02-01-09-1521)	Sample was soluted in meta-Phosphoric acid	enzymatic determination, automated with Arena 20 XT	Sigma 99,5%	no	yes	
	24		same as above	AACC-89.90,FEFANA -by HPLC	Vitamin C	no	yes	as Ascorbic acid
	25	HPLC-UV	Extraction with Water			no	no	

Analyte	Participant	Method description	Sample preparation	Measuring method	Calibration / Reference material	Recovery with same matrix	Method accredited	Further remarks
Folsäure / Folic Acid	1							
	2	SLMB 62/11.2.2 mod.					yes	
	3	House method Görtler A.S.08 2005-07	-	HPLC	Calibration	yes	yes	
	4	VitaFast					yes	
	5		N/A	Biacore	N/A	no	yes	
	6	LAV 21.0017-02; HPLC-DAD					yes	
	7	Vitafast	---	---	Folic acid	no	no	---
	8	HPLC-DAD				no	yes / no	
	9	LC-MS/MS, House method			yes	yes	yes	
	10	R-Biopharm VitaFast Vitamin Folic acid P1001, 2011-06	none	none	PT-Material	yes	yes	none
	11		Sample soluted in H2O	LC-MS/MS		yes	yes	
	12	House method: L138	alkaline dissolved	HPLC - DAD	Standard: Folic acid	no	yes	
	13	LC-MS/MS (House method)			6-Point Calibration	yes	no	
	14			HPLC		no	yes	
	15	House method		HPLC-DAD	external, Sigma Aldrich		yes	
	16	LC-MSMS					yes	
	17							
	18							
	19	Microbiological Microtiter-Platetest, VitaFast® Folic acid, r-biopharm, Art. Nr.: P1001			N.I.S.T. SRM 3280		yes	
	20							
	21	USP 36	-	-	no	yes	yes	
	22	HPLC-UV	n/a	n/a	n/a	no	yes	yes
	23	internal Method Microbiology (P2-02-02-02-0390)	as per test kit instructions	as per test kit instructions			yes	Vita Fast®Folsäure
	24	In house method	Weighed about 2g of spl in 250ml added 5ml of 1M NaOH and diluent (750 water:240ml methanol:10ml gla acetic acid) sonicated and boiled 10 mts at 70°C, cool made up	AACC-89.90,FEFANA-by HPLC	Folic acid	no	yes	as Folic acid
	25	HPLC-UV	Extraction with Water			no	no	

Analyte	Participant	Method description	Sample preparation	Measuring method	Calibration / Reference material	Recovery with same matrix	Method accredited	Further remarks
Niacin	1	In house developed method	Extraction through sonication	UPLC-DAD	External standard	no	yes	Results include Niacin and Niacinamide
	2							
	3	House method Görtler A.S. 09 2005-07	-	HPLC	Calibration	yes	yes	
	4	HPLC					yes	
	5	Sum Niacin + Niacinamide	N/A	HPLC	N/A	no	yes	
	6	LAV 21.0017-02; HPLC-DAD					yes	
	7	2.019/016/02	---	---	Nicotin acid	no	no	---
	8	HPLC-DAD				no	yes / no	
	9	HPLC, House method			yes	yes	yes	
	10	Vitamins water soluble in food supplements, HPLC-DAD	none	none	PT-Material	yes	yes	none
	11		Sample soluted in H2O	LC-MS/MS		yes	yes	
	12	House method: L138	acidic dissolved	HPLC - DAD	Standard: Niacinamide	no	yes	
	13	HPLC-Fluo (NF EN 15652)			calibration with 6 points	yes	yes	
	14			HPLC		no	yes	
	15	House method		HPLC-DAD	external, Sigma Aldrich		yes	
	16	HPLC-UV					yes	
	17		as per test instructions	VITA FAST P1004 Niacin			no	
	18							
	19							
	20							
	21	USP 36	-	-	yes	no	yes	
	22	HPLC-UV	N/A	n/a	n/a	no	yes	Results are for Niacinamide and not as Niacin as indicated; not associated Peak for Niacin
	23	internal Method Microbiology (P2-02-02-02-0390)	as per test instructions	as per test instructions			yes	Vita Fast®Folic acid
	24	In house method	same as above	AACC-89.90,FEFANA -by HPLC	Niacin und Nicotin acid	no	yes	Niacinamide: Sample A: 26030mg/100g, Sample B: 26260mg/100g, Mean 26150 mg/100g
	25	HPLC-UV	Extraction with Water			no	no	

Analyte	Participant	Method description	Sample preparation	Measuring method	Calibration / Reference material	Recovery with same matrix	Method accredited	Further remarks
Pantothensäure / Pantothenic Acid	1	House method	In house developed method	Extraction through sonication	External standard	no	no	no
	2							
	3	House method Görtler A.S. 10 2005-07		HPLC	Calibration	yes	yes	yes
	4	Vitafast					yes	
	5		N/A		N/A			
	6	LAV 21.0017-02; HPLC-DAD					yes	
	7	Vitafast	---	---	Pantothenic acid	no	no	---
	8	HPLC-DAD				no	yes / no	
	9	ELISA, Testkit r-biopharm			yes	yes	yes	
	10	R-Biopharm VitaFast Vitamin Pantothenic acid P1005, 2011-06	none	none	PT material	yes	yes	none
	11		sample soluted in H2O	LC-MS/MS		yes	yes	
	12	House method: L138	acidic dissolved	HPLC - DAD	Standard: D-Pantothenic acid hemicalcium salt	no	yes	
	13	HPLC UV (in house method)			7-Poitn Calibration	yes	no	
	14			HPLC		no	yes	
	15	House method		HPLC-DAD	external, Sigma Aldrich		yes	
	16	LC-MSMS					yes	
	17		as per test instructions	VITA FAST P1005 Pantothenic acid			no	
	18							
	19							
	20							
	21	USP 36	-	-	no	yes	yes	
	22	HPLC-UV	N/A	n/a	n/a	no	yes	yes
	23	Internal Method Microbiology (P2-02-02-02-0391)	as per test instructions	as per test instructions			yes	VitaFast® Pantothenic acid
	24	In house method	Weigh 2g of spl in 50ml added diluent (Buffer:methanol) pH 3.2, sonicate 10mts made up with diluent	AACC-89.90,FEFANA -by HPLC	Calcium Pantothenate	no	yes	as Pantothenic acid
	25	HPLC-UV	Extaction with Water			no	no	

5.2 Homogeneity

5.2.1 Homogeneity of bottled PT-samples

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

Folic acid

Indepedant Samples	g/kg
1	2,43
2	2,42
3	2,44
4	2,43
5	2,44
6	2,44
7	2,44
8	2,43
9	2,33
10	2,47

General Mean	2,43	
Repeatability standard deviation	0,0366	1,51%

Niacinamide

Indepedant Samples	g/kg
1	144
2	144
3	143
4	143
5	142
6	144
7	145
8	145
9	143
10	144

General Mean	144	
Repeatability standard deviation	0,766	0,53%

Pantothenic acid

Indepedant Samples	g/kg
1	67,9
2	68,9
3	68,8
4	67,8
5	67,5
6	68,7
7	67,9
8	69,2
9	69,5
10	69,2

General Mean	68,5	
Repeatability standard deviation	0,724	1,06%

Vitamin B1

Indepedant Samples	g/kg
1	11,3
2	11,5
3	11,3
4	11,2
5	11,2
6	11,1
7	11,5
8	11,3
9	11,3
10	11,4

General Mean	11,3	
Repeatability standard deviation	0,129	1,14%

Vitamin B6

Indepedant Samples	g/kg
1	3,56
2	3,70
3	3,54
4	3,61
5	3,42
6	3,62
7	3,54
8	3,64
9	3,71
10	3,49

General Mean	3,58	
Repeatability standard deviation	0,0911	2,54%

Vitamin B2

Indepedant Samples	g/kg
1	11,9
2	12,3
3	12,1
4	12,8
5	11,9
6	11,6
7	11,9
8	13,1
9	12,4
10	11,8

General Mean	12,2	
Repeatability standard deviation	0,489	4,01%

5.2.2 Comparision of sample numbers / test results and trend line

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

Vitamin B6				
Target standard deviation σ_{pt}	26,1			mg/100g
Sample numbers	6 – 114			
Total numbers of samples	40			
Slope	0,368			
Trend line range	374	-	359	mg/100g
Deviation trend line	366	±	7,35	mg/100g
Percent of σpt	28,2	%		

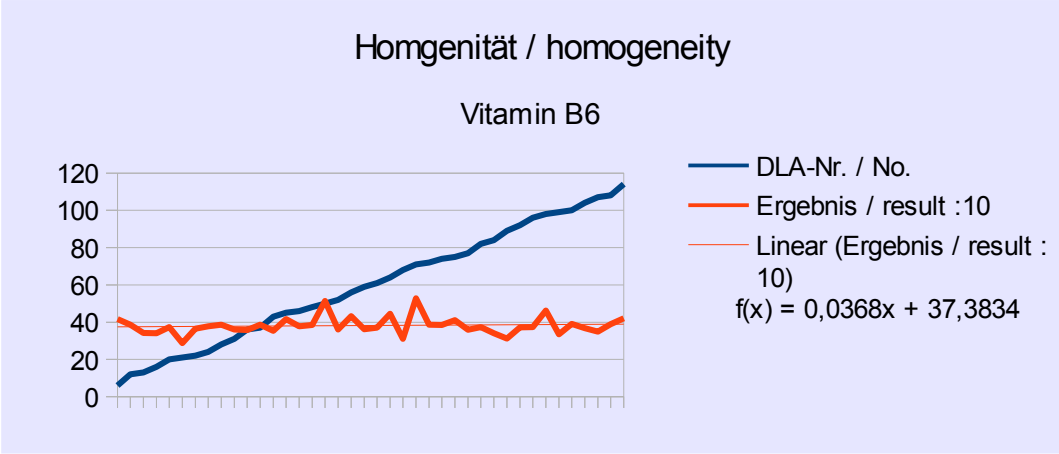
* without results with z-Scores > |3,0|

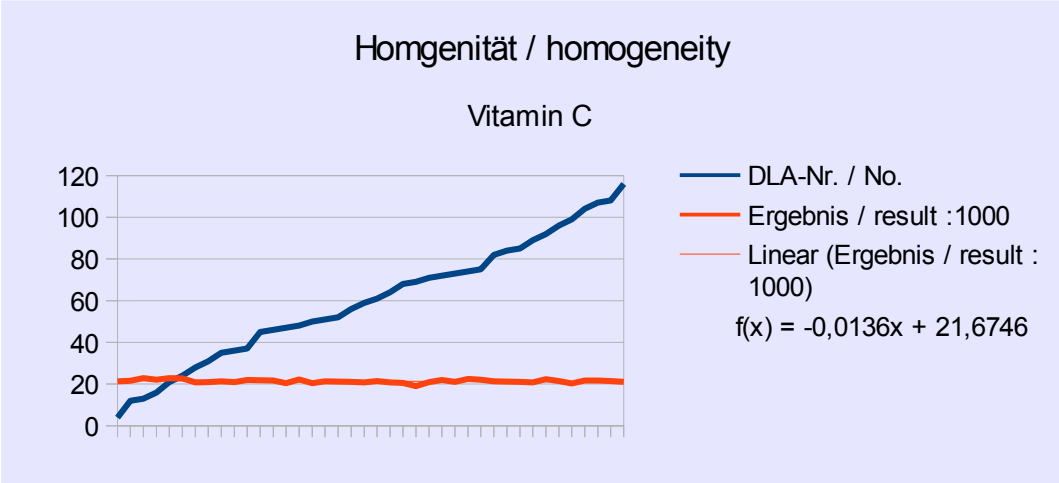
Vitamin C				
Target standard deviation σ_{pt}	535			mg/100g
Sample numbers	4 – 116			
Total numbers of samples	40			
Slope	-13,6			
Trend line range	21675	-	21130	mg/100g
Deviation trend line	21403	±	272	mg/100g
Percent of σpt	50,9	%		

* without results with z-Scores > |3,0|

Folic acid				
Target standard deviation σ_{pt}	36682			µg/100g
Sample numbers	11 – 109			
Total numbers of samples	32			
Slope	42,9			
Trend line range	224377	-	225750	µg/100g
Deviation trend line	225063	±	686	µg/100g
Percent of σpt	1,9	%		

* without results with z-Scores > |3,0|





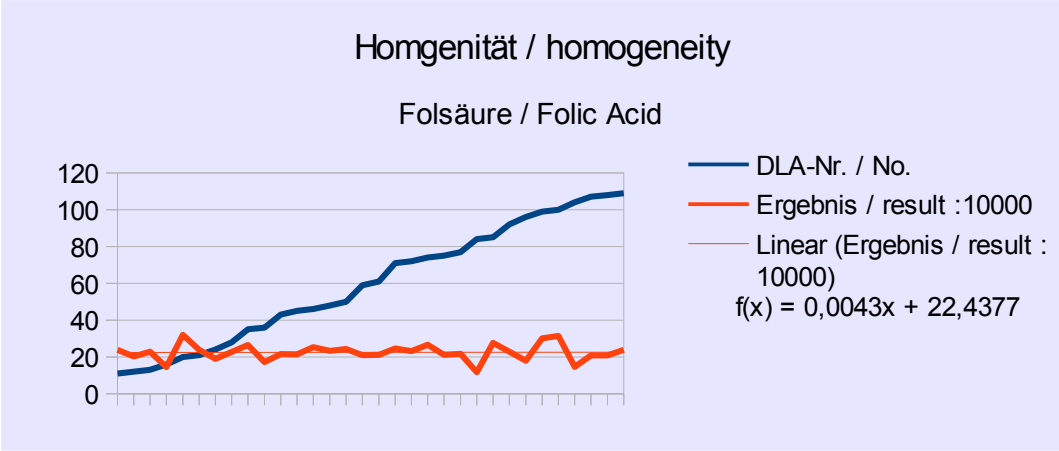


Abb./Fig. 19:
Trendfunktion Probennummern vs. Ergebnisse: Vitamin B6, Vitamin C und Folsäure (1/10, 1/000 und 1/10000 dargestellt)
trend line function sample number vs. results: vitamin B6, vitamin C and folic acid (1/10, 1/000 and 1/10000 shown)

5.3 Stability

5.3.1 Trend line function of participant results

By comparison of the participant results with the corresponding date of analysis the stability of the PT-material can be characterized for the range of analysis time (36-41days) of the present PT by the trend line functions:

Vitamin B6				
Target standard deviation σ_{pt}	26,1			mg/100g
Period of PT analysis	40			Days
Number of results	16			
Slope	-0,916			
Trend line range	382	-	367	mg/100g
Deviation trend line	374	±	7,33	mg/100g
Percent of σ_{pt}	28,1	%		

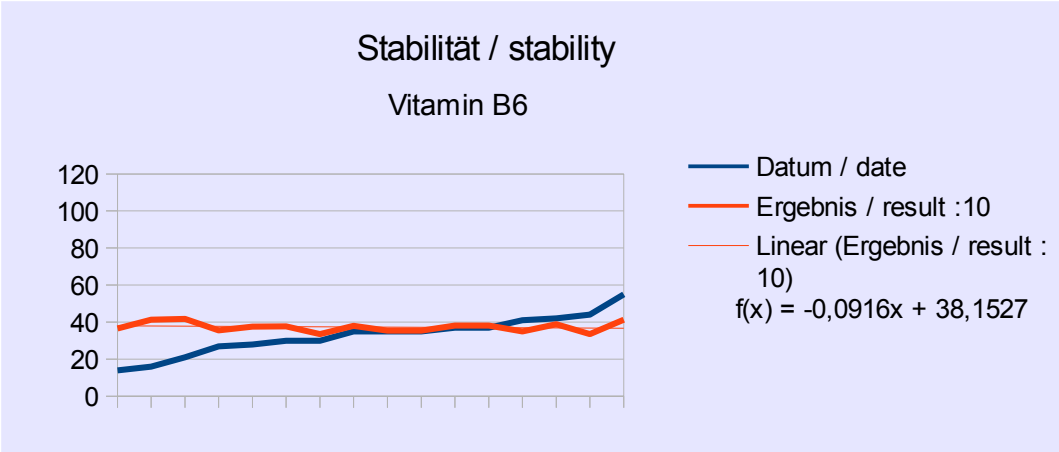
* without results with z-Scores > |2,0|

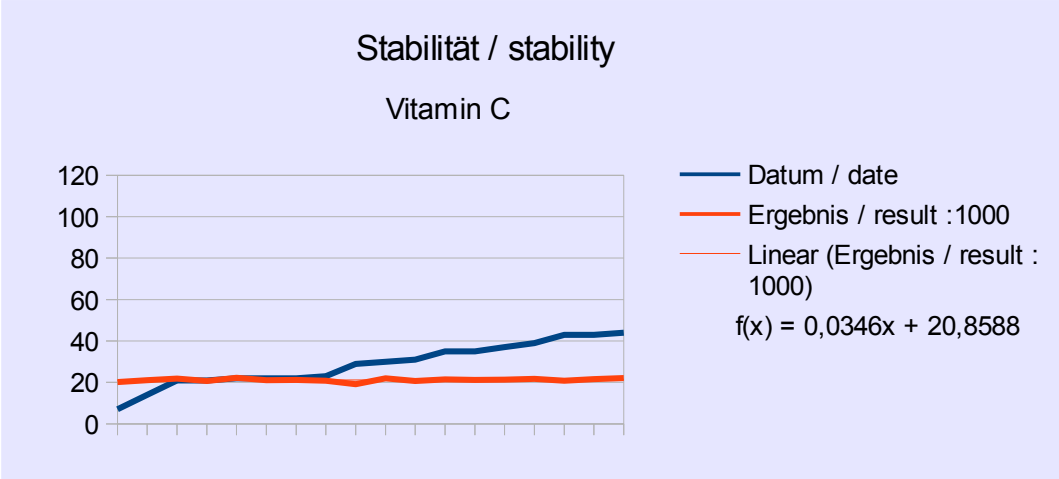
Vitamin C				
Target standard deviation σ_{pt}	535			mg/100g
Period of PT analysis	36			Days
Number of results	18			
Slope	34,6			
Trend line range	20548	-	21171	mg/100g
Deviation trend line	20860	±	312	mg/100g
Percent of σ_{pt}	58,3	%		

* without results with z-Scores > |3,0|

Folic acid				
Target standard deviation σ_{pt}	36682			µg/100g
Period of PT analysis	41			Days
Number of results	14			
Slope	1727			
Trend line range	212404	-	236582	µg/100g
Deviation trend line	224493	±	12089	µg/100g
Percent of σ_{pt}	33,0	%		

* without results with z-Scores > |3,0|





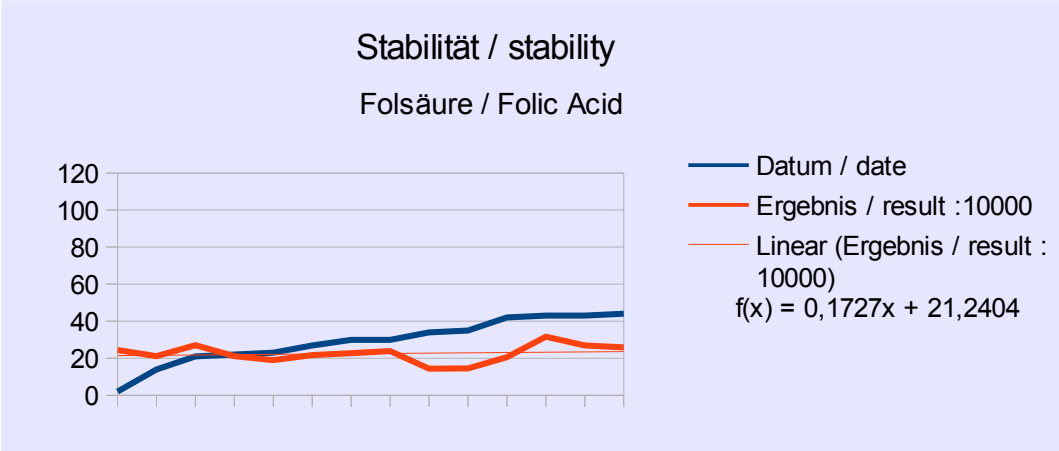


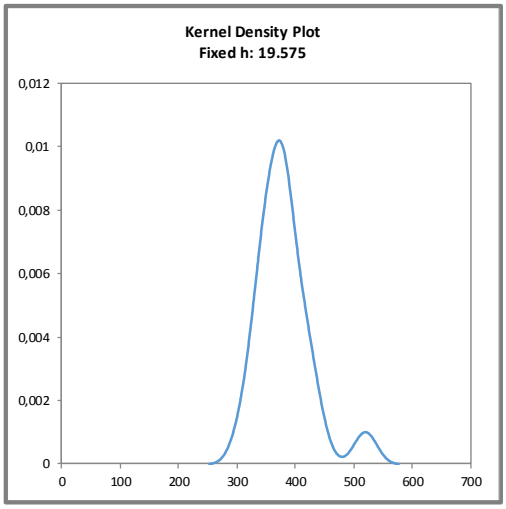
Abb./Fig. 20:
Trendfunktion Analysendatum vs. Ergebnisse: Vitamin B6, Vitamin C und Folsäure (1/10, 1/000 und 1/10000 dargestellt)
trend line function date of analysis vs. results: vitamin B6, vitamin C and folic acid (1/10, 1/000 and 1/10000 shown)

5.4 Kernel Density Plots of Results

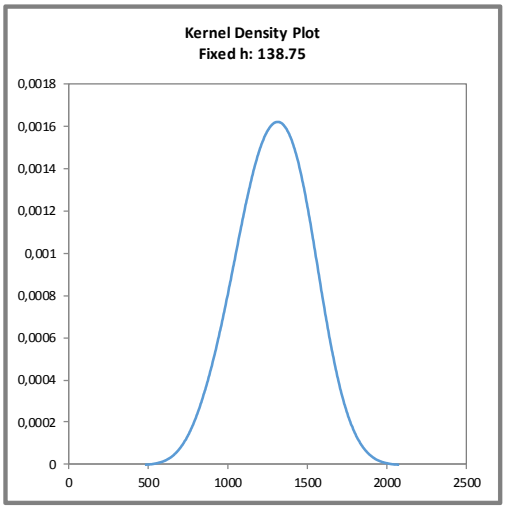
Abbildungen:
Kerndichte-Schätzungen
der Teilnehmerergebnisse
(mit h = 0,75 x σ_{pt} von Xpt)

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

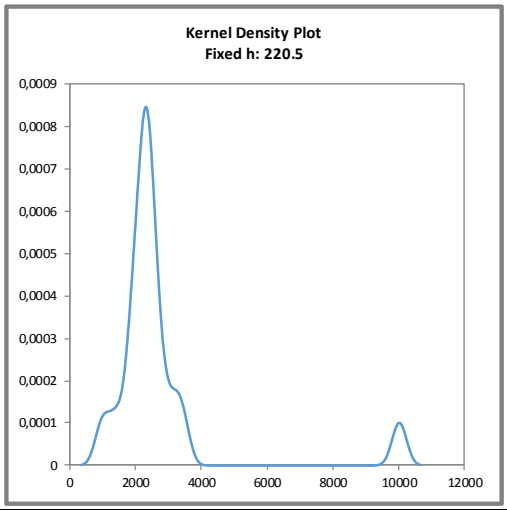
Vitamin B6



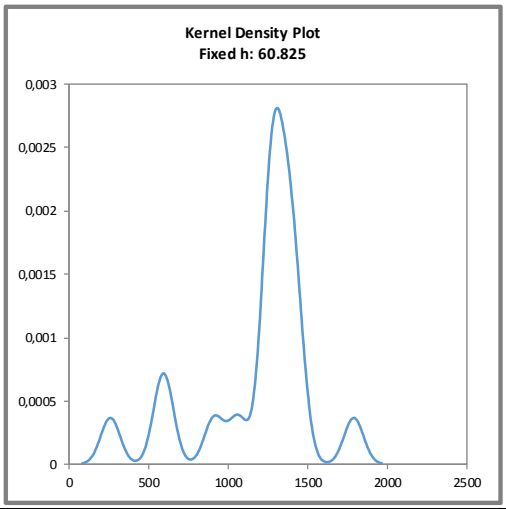
Vitamin B1



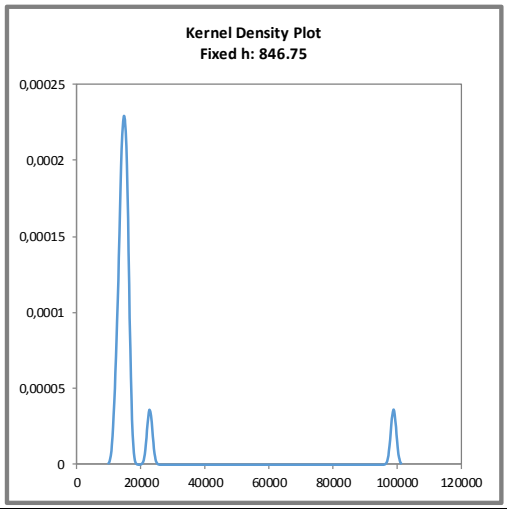
Vitamin B12



Vitamin B2



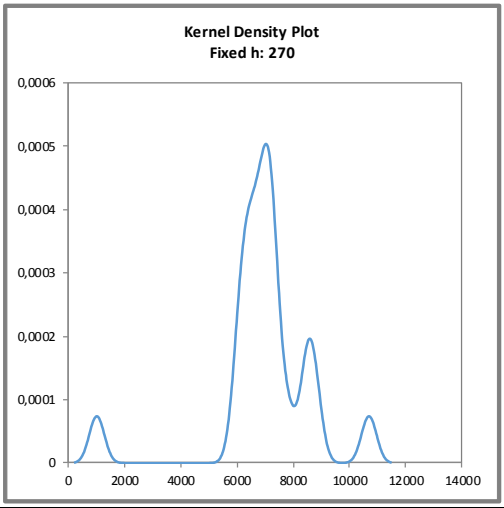
Biotin



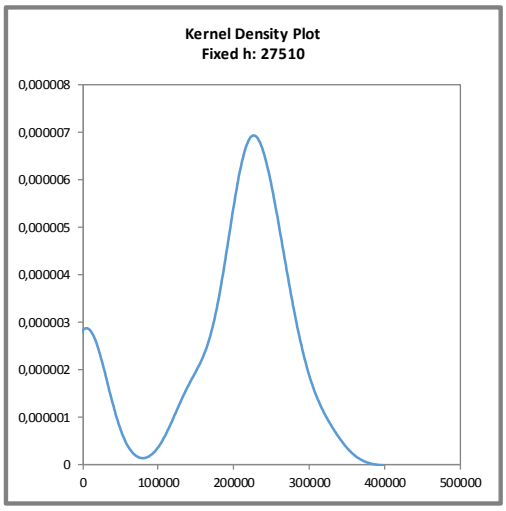
Abbildungen:
Kerndichte-Schätzungen
der Teilnehmerergebnisse
(mit h = 0,75 x σ_{pt} von X_{pt})

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

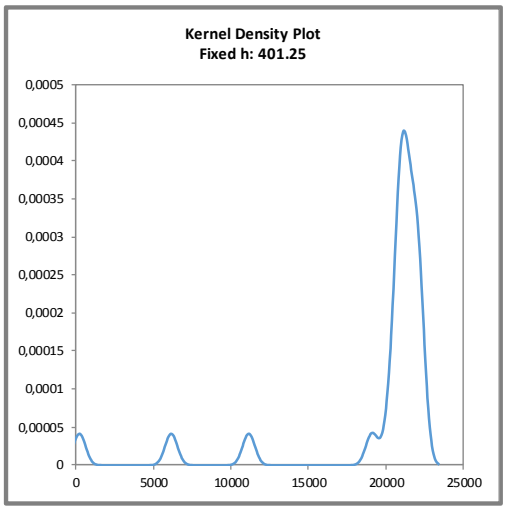
Pantothensäure / Pantothenic Acid



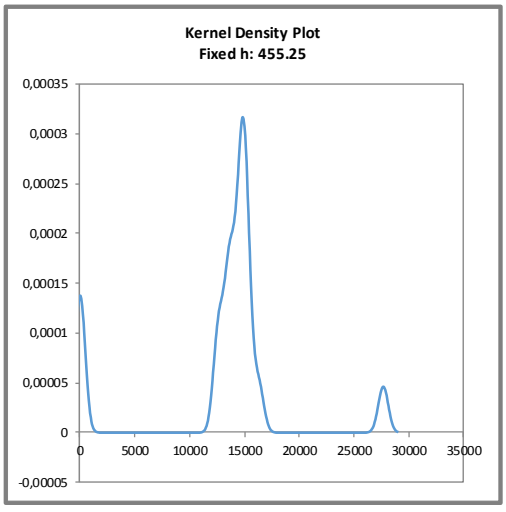
Folsäure / Folic Acid



Vitamin C



Niacin



5.5 Information on the Proficiency Test (PT)

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

PT number	***DLA 43-2017***
PT name	***Food Supplement I: Vitamins B1, B2, B6, B12, Biotin, Vitamin C, Folic Acid, Niacin and Pantothenic Acid***
*Sample matrix**	***Samples A + B:*** *Multi-vitamin capsule powder (without capsule shell) / ingredients: maltodextrin, vitamins and carrier: mannitol*
Number of samples and sample amount	*2 identical samples A + B, 50 g each.*
Storage	*Samples A + B: cooled 2 - 10°C (dark and dry)*
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. 0.5 - 1.5 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 A and B as well as the final results calculated as mean of the double determination (samples A and B) should be filled in the result submission file 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*
Number of significant digits	*at least 2*
Further information	*For information please specify:* – *Date of analysis* – *DLA-sample-numbers (for sample A and B)* – *Limit of quantification* – *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 June 30th 2017***
Evaluation report	*The evaluation report is expected to be completed 6 weeks after deadline of result submission and sent as PDF file by e-mail.*
Coordinator and contact person of PT	*Dr. Matthias Besler*

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

6. Index of participant laboratories in alphabetical order

Teilnehmer / Participant	Ort / Town	Land / Country
		FRANCE
		GREAT BRITAIN
		AUSTRIA
		Germany
		Germany
		Germany
		Germany
		Germany
		Germany
		USA
		Germany
		Germany
		Germany
		Germany
		Germany
		Germany
		USA
		Germany
		Germany
		Germany
		Germany
		CROATIA
		THAILAND
		INDIA

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

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

7. Index of references

1. DIN EN ISO/IEC 17025:2005; Allgemeine Anforderungen an die Kompetenz von Prüf- und Kalibrierlaboratorien / General requirements for the competence of testing and calibration laboratories
2. DIN EN ISO/IEC 17043:2010; Konformitätsbewertung – Allgemeine Anforderungen an Eignungsprüfungen / Conformity assessment – General requirements for proficiency testing
3. ISO 13528:2015 & DIN ISO 13528:2009; Statistische Verfahren für Eignungsprüfungen durch Ringversuche / Statistical methods for use in proficiency testing by interlaboratory comparisons
4. ASU §64 LFGB: Planung und statistische Auswertung von Ringversuchen zur Methodenvalidierung / DIN ISO 5725 series part 1, 2 and 6 Accuracy (trueness and precision) of measurement methods and results
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17. 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
18. 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
19. 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
20. ASU §64 LFGB: L 00.00-87 / EN 14131:2003 Mikrobiologische Bestimmung von Folat, Juli 2004 / Foodstuffs - Determination of folate by microbiological assay
21. EN 14130:2003; Untersuchung von Lebensmitteln: Bestimmung von Vitamin C mit HPLC (zurückgezogen) / Foodstuffs. Determination of vitamin C by HPLC (withdrawn)
22. EN 15607:2009; Untersuchung von Lebensmitteln: Bestimmung von D-Biotin mit HPLC / Foodstuffs. Determination of d-biotin by HPLC
23. EN 15652:2009; Untersuchung von Lebensmitteln: Bestimmung von Niacin mit HPLC / Foodstuffs. Determination of niacin by HPLC
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