Evaluation Report
proficiency test

DLA ptSU06 (2020)

Dietetic Product I:

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

in Drink Powder

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

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Unteraufträge *Subcontractors*	Im Rahmen dieser Eignungsprüfung wurden nachstehende Leistungen im Unterauftrag vergeben: Keine As part of the present proficency test the following services were subcontracted: none
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.

Contents

1. Introduction

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

2. Realisation

2.1 Test material

The test material is a mixture of common in commerce dietetic products (drink powders) as meal replacements from European suppliers.
The raw materials were ground and sieved, mixed and homogenized.

Afterwards the samples were portioned to approximately 50g into metal-lised PET film bags and chronologically numbered.

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

Table 1: Composition of DLA-Samples

PT-Sample Drink Powder

Drink powder

Ingredients: Milk protein, soy protein isolate, dextrin, maltodextrin, defatted poppy seed flour, thickener sodium carboxymethyl cellulose, flavor, dipotassium phosphate, magnesium hydroxide, L-ascorbic acid, turmeric powder, sweetener sodium saccharin, anti-caking agent silicon dioxide, iron-III-pyrophocosphate, natural flavor, DL-alpha-ocopheryl acetate, nicotinamide, natural vanilla flavor, zinc oxide, calcium D-pantothenate, manganese sulfate, copper carbonate, cholecalciferol, pyridoxine hydrochloride, riboflavin, thiamine mononitrate, retiny acetate, folic acid, sodium iodide, sodium selenite, phylachinone, D-biotin, cyanocobalamin.

Meal replacement, classic

Ingredients: soy protein isolate, honey, soy oil, skimmed milk powder (10%), milk protein, yoghurt powder, glucose syrup, potassium chloride, trimagnesium dicitrate, tri-calcium phosphate, anti-caking agent silicon dioxide, flavor, maltodextrin, L-ascorbic acid, iron-III-diphosphate, D-alpha tocopherol acetate, nicotinamide, zinc oxide, antioxidant DL-alpha tocopherol, calcium D-pantothenate, manganese-II-sulfate, copper carbonate, pyridoxine hydrochloride, riboflavin, thiamine mononitrate, retinylacetate, pteroylmonoglutamic acid, sodium iodide, sodium selenite, phylloquinone, D-biotin, cholecalciferol, cyanocobalamin.

Meal replacement, vanilla

Ingredients: soy protein isolate, honey, soy oil, skimmed milk powder, milk protein, yoghurt powder, flavor, glucose syrup, magnesium dicitrate, tri-calcium phosphate, potassium citrate, potassium phosphate, anti-caking agent silicon dioxide, potassium chloride, vanilla pod powder, ascorbic acid, sweetener steviol glycosides, iron-III-diphosphate, nicotinamide, D-alpha tocopheryl acetate, zinc oxide, antioxidant DL-alpha tocopherol, calcium D-pantothenate, manganese-II-sulfate, copper carbonate, pyridoxine hydrochloride, riboflavin, thiamine monlaconitrate, dye beta-carotene, retynyl acetate, pteroylmonoglutamic acid, sodium iodide, sodium selenite, phylloquinone, D-biotin, cholecalciferol, cyanocobalamin.

Meal replacement, vanilla 2

Ingredients: soy protein isolate, skimmed milk yoghurt powder, honey, potassium citrate, tricalcium phosphate, flavor, magnesium hydroxide, anti-caking agent: silicon dioxide; iron fumarate, L-ascorbic acid, sweetener: sucralose; DL-alpha-tocopheryl acetate, nicotinamide, zinc oxide, calcium D-pantothenate, copper gluconate, manganese sulfate, pyridoxine hydrochloride, thiamine mononitrate, riboflavin, cholecalciferol, retinylacetate, folic acid, sodium selenite, sodium iodide, maltodextrin, phylloquinone, D-biotin, cyanocobalamin.

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 100g
Vitamin B1	1,28 mg
Vitamin B2	2,00 mg
Vitamin B6	1,73 mg
Vitamin B12	2,28 µg
Biotin	61,0 µg
Vitamin C	73,4 mg
Folic acid	264 µg
Niacinamide	17,8 mg
Pantothenic acid	5,71 mg

2.1.1 Homogeneity

The **mixture homogeneity before bottling** was examined 8-fold by **microtracer analysis**. It is a standardized method that is part of the international GMP certification system for feed [14]. Before mixing dye coated iron particles of µm size are added to the sample and the number of particles is determined after homogenization in taken aliquots. The evaluation of the mixture homogeneity is based on the Poisson distribution using the chi-square test. A probability of ≥ 5 % is equivalent to a good homogeneous mixture and of ≥ 25% to an excellent mixture [14, 15].
The microtracer analysis of the present PT sample showed a probability of 90%. Additionally particle number results were converted into concentrations, statistically evaluated according to normal distribution and compared to the standard deviation according to Horwitz. For the assessment HorRat values between 0,3 and 1,3 are to be accepted under repeat conditions (measurements within the laboratory) [16, 17]. This gave a HorRat value of 0,8 for the present PT sample. The results of microtracer 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 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- and EN-Methods, s. 3.6.2) [18-26]. 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**	**Parameter**	**CV_r**
Vitamin B1	7,67 %	Vitamin C	7,49 %
Vitamin B2	2,23 %	Folic acid	8,68 %
Vitamin B6	3,65 %	Niacin	3,34 %
Vitamin B12	6,86 %	Pantothenic	9,59 %
Biotin	-	acid	

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. 0,38 (19,4°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 15th week of 2020. The testing method was optional. The tests should be finished at 19th June 2020 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 dietetic food (meal replacement) with above mentioned parameters in the matrix of drink 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 5.4 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 11 participants submitted their results in time.

3. Evaluation

3.1 Consensus value from participants (assigned value)

The robust mean of the submitted results was used as assigned value (X_{pt}) („consensus value from participants") providing a normal distribution. The calculation was done according to algorithm A as described in annex C of ISO 13528 [3]. If there are < 12 quantitative results and an increased difference between robust mean and median, the median may be used as the assigned value (criterion: Δ median - rob. mean > 0,3 σ_{pt}) [3].
The condition is that the majority of the participants' results show a normal distribution or are distributed unimodal and symmetrically. To this end, an examination of the distribution is carried out, inter alia, using the kernel density estimate [3, 12].
In case there are indications for sources of higher variability such as a bimodal distribution of results, a cause analysis is performed. Frequently different analytical methods may cause an anomaly in results' distribution. If this is the case, separate evaluations with own assigned values (X_{pti}) are made whenever possible.

The statistical evaluation is carried out for all the parameters for a minimum of 7 values are present, in justified cases, an evaluation may also be carried out from 5 results onwards.
The actual measurement results will be drafted. Individual results, which are outside the specified measurement range of the participating laboratory (for example with the result > 25 mg/kg or < 2,5 mg/kg) or the indicating "0" will not be considered for the statistic evaluation [3].

3.2 Robust standard deviation

For comparison to the target standard deviation σ_{pt} (standard deviation for proficiency assessment) a robust standard deviation (S*) 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): vitamin B2, B6 and B12, folic acid, niacin and pantothenic acid.
The target standard deviation of the evaluation by a precision experiment (s. 3.6.2) was considered for the following parameters (ASU §64/ EN-Norms) [23]: vitamin B1 and vitamin C.

Additionally for vitamin B1 and B6 and for pantothenic acid the standard uncertainty was considered by evaluation using z'-scores (see 3.6.8).

No statistical evaluation using z-scores was carried out for biotin, because only 5 results with increased variation were available.

3.6.1 General model (Horwitz)

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

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

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

3.6.2 Value by precision experiment

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

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

The relative repeatability standard deviations (RSD_r) and relative reproducibility standard deviation (RSD_R) given in Table 4 were determined in ring tests using the indicated methods.
The resulting target standard deviations σ_{pt}, which were identified there, were used to evaluate the results and to provide additional information for the statistical data.

Table 4: Relative repeatability standard deviations (RSD_r) and relative reproducibility standard deviations (RSD_R) according to selected evaluations of tests for precision and the resulting target standard deviation σ_{pt} [18-26]

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

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

3.6.3 Value by perception

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

For the present evaluation the target standard deviation according to 3.6.1 or 3.6.2 was regarded suitable.
Table 5 shows selected statistic data of participants results of present PT compared to PT results of previous years.

3.7 z-Score

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

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

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

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

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

3.7.1 Warning and action signals

In accordance with the norm ISO 13528 it is recommended that a result that gives rise to a *z*-score above 3,0 or below −3,0, shall be considered to give an "action signal" [3]. Likewise, a *z*-score above 2,0 or below −2,0 shall be considered to give a "warning signal". A single "action signal", or "warning signal" in two successive PT-rounds, shall be taken as evidence that an anomaly has occurred which requires investigation.
An error or cause analysis can be carried out by checking the analysis process including understanding and implementation of the measurement by the staff, details of the measurement procedure, calibration of equipment and composition of reagents, transmission error or an error in the calculation, in the trueness and precision and use of reference material. If necessary, the problems must be addressed through appropriate corrective action [3].

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

Table 5: Characteristics of the present PT (on dark gray) in comparison to previous PTs (SD = standard deviation, CV = coefficient of variation, MV = Multivitamin)

Parameter	**Matrix (Powder)**	**robust Mean**	**rob. SD (S*)**	**rel. SD (CV_{S*}) [%]**	**Quotient S*/σpt**	**DLA-report**
Vitamin B1	MV-drink powder	3,11 mg/100g	0,606 mg/100g	19,5%	1,7*	DLA 32/2015
Vitamin B1	MV-capsule powder	690 mg/100g	98,1 mg/100g	14,2%	1,0	DLA 46/2019
Vitamin B1	Drink powder	1,25 mg/100g	0,412 mg/100g	33,1%	1,6*	ptSU06/2020
Vitamin B2	MV-drink powder	2,89 mg/100g	0,890 mg/100g	30,8%	2,0*	DLA 32/2015
Vitamin B2	MV-capsule powder	783 mg/100g	58,3 mg/100g	7,45%	1,2	DLA 46/2019
Vitamin B2	Drink powder	1,72 mg/100g	0,136 mg/100g	7,89%	0,76	ptSU06/2020
Vitamin B6	MV-drink powder	3,86 mg/100g	0,329 mg/100g	8,5%	0,93	DLA 32/2015
Vitamin B6	MV-capsule powder	749 mg/100g	58,4 mg/100g	7,80%	1,3	DLA 46/2019
Vitamin B6	Drink powder	1,50 mg/100g**	0,67 mg/100g	44,70%	1,6*	ptSU06/2020
Vitamin B12	MV-drink powder	7,90 µg/100g	2,66 µg/100g	33,7%	1,4	DLA 32/2015
Vitamin B12	MV-capsule powder	1018 µg/100g	102 µg/100g	10,0%	0,89	DLA 46/2019
Vitamin B12	Drink powder	2,46 µg/100g	1,11 µg/100g	45,2%	1,6	ptSU06/2020
Biotin	MV-capsule powder	11200 µg/100g	1190 µg/100g	10,6%	1,4	DLA 48/2016
Biotin	MV-capsule powder	67368 µg/100g	9709 µg/100g	14,4%	0,84	DLA 46/2019
Biotin	Drink powder	54,3 µg/100g**	42,1 µg/100g	-	-	ptSU06/2020
Folsäure	MV-drink powder	710 µg/100g	148 µg/100g	20,8%	1,8	DLA 32/2015
Folsäure	MV-capsule powder	88412 µg/100g	9691 µg/100g	11,0%	1,9	DLA 46/2019
Folsäure	Drink powder	256 µg/100g	23,8 µg/100g	9,27%	0,67	ptSU06/2020
Niacin	MV-capsule powder	1530 mg/100g	107 mg/100g	6,98%	1,9	DLA 48/2016
Niacin	MV-capsule powder	8062 mg/100g	324 mg/100g	4,02%	1,4	DLA 46/2019
Niacin	Drink powder	17,9 mg/100g	1,96 mg/100g	11,0%	1,5	ptSU06/2020

Continuation next page

Continuation Table 5:

Parameter	Matrix (Powder)	robust Mean	rob. SD (S*)	rel. SD (CV_{s*}) [%]	Quotient S*/σpt	DLA-report
Pantothen-ic acid	MV-capsule powder	598 mg/100g	41,1 mg/100g	6,88%	1,6	DLA 48/2016
Pantothen-ic acid	MV-capsule powder	2667 mg/100g	196 mg/100g	7,35%	1,8*	DLA 46/2019
Pantothen-ic acid	Drink powder	5,76 mg/100g	0,955 mg/100g	16,6%	1,4*	ptSU06/2020
Vitamin C	MV-capsule powder	6133 mg/100g	365 mg/100g	5,96%	1,4	DLA 48/2016
Vitamin C	MV-capsule powder	34257 mg/100g	2644 mg/100g	7,72%	0,74	DLA 46/2019
Vitamin C	Drink powder	80,0 mg/100g	13,0 mg/100g	16,3%	1,6	ptSU06/2020

** with target standard deviation σpt'*
*** assigned value (Xpt): Median*

3.8 z'-Score

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

The calculation is performed by:

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

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

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

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

For warning and action signals see 3.7.1.

3.9 Reproducibility cofficient of variation (CV_R)

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

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

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

3.10 Quotient S*/σ_{pt}

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

3.11 Standard uncertainty of the assigned value

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

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

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

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

4. Results

Comments to the distribution of the results:

The kernel density plots showed for the parameters niacin, vitamin B1 and vitamin C 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.

In the case of vitamin B2, B6 and B12 as well as biotin, folic acid and pantothenic acid a kernel density estimation was not made due to the number of < 8 results.

Comments to the statistic data:

For biotin only 5 results with a high variation were available, so that no statistical analysis could be carried out. The evaluations for vitamin B6 and pantothenic acid were provided for information only due to the small number of results.

The target standard deviation was calculated according to the general model of Horwitz or by data from precision experiments (ASU §64 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 (s. 3.6).

For vitamin B1, B6 and pantothenic acid the distribution of results showed an increased variability with a quotient above 2,0. The parameters were evaluated by z´-scores considering the standard uncertainty. Then the quotients S^*/σ_{pt}' were below 2,0 (s. Tab. 5).
For all other parameters the distribution showed a normal variability of results. The quotients S^*/σ_{pt} were in the range of 0,67 to 1,6 (s. Tab. 5).

The median was used as the assigned value for the parameters biotin and vitamin B6.

The robust standard deviations and the repeatability and reproducibility standard deviation were in the range of of established values for the used determination methods (s. 3.6.2), or for vitamins B1, B6 and B12 in the upper range.
The comparability of results is given.

67% to 100% of results were in the respective target range.

The robust means or medians of the participant results were for all parameters in the range of 86% to 108% 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**
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°	8
Number of outliers	1
Mean	1,25
Median	1,31
Robust Mean (x_{pt})	**1,25**
Robust standard deviation (S*)	**0,412**
Number with 2 replicates	8
Repeatability SD (S_r)	0,0955
Repeatability (CV_r)	7,67%
Reproducibility SD (S_R)	0,370
Reproducibility (CV_R)	29,7%
Target range:	
Target standard deviation σ_{pt}'	**0,255**
Target standard deviation (for Information)	0,136
lower limit of target range	**0,736**
upper limit of target range	**1,76**
Quotient S/σ_{pt}'*	*1,6*
Standard uncertainty $U_{(xpt)}$	*0,182*
Results in the target range	*7*
Percent in the target range	*88%*

° *without outliers (results no. 10)*

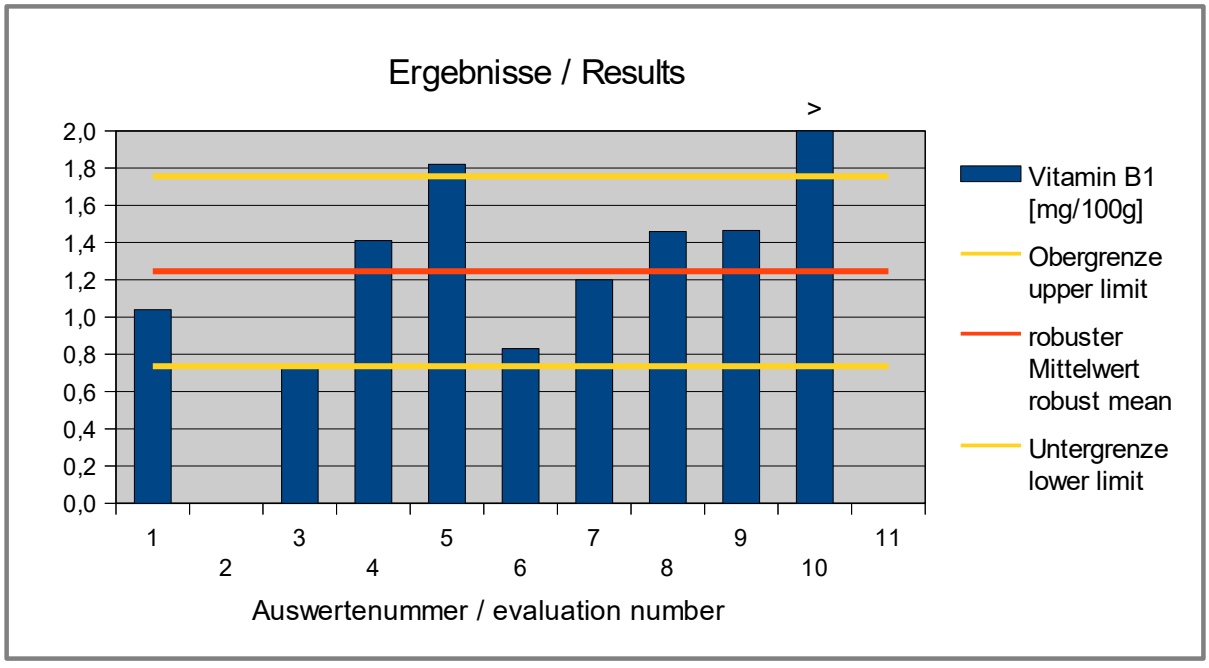


Abb. / Fig. 1: Ergebnisse Vitamin B1 / Results Vitamin B1

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Vitamin B1 [mg/100g]	Abweichung [mg/100g] Deviation [mg/100g]	z'-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	1,04	-0,206	-0,81	-1,5	
2					
3	0,740	-0,506	-2,0	-3,7	
4	1,41	0,164	0,64	1,2	
5	1,82	0,574	2,3	4,2	
6	0,830	-0,416	-1,6	-3,0	
7	1,20	-0,046	-0,18	-0,33	
8	1,46 *	0,214	0,84	1,6	
9	1,47 *	0,219	0,86	1,6	
10	13,1				Ausreißer ausgeschlossen / Outlier excluded
11					

* Mean calculated by DLA

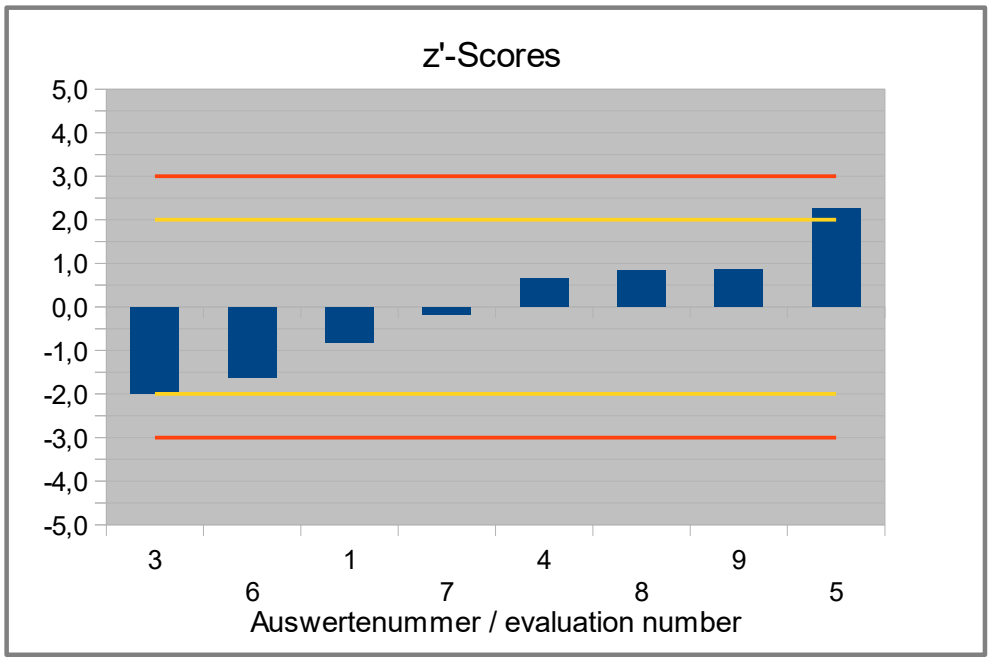


Abb. / Fig. 2: z'-Scores Vitamin B1

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

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	7
Number of outliers	-
Mean	1,67
Median	1,70
Robust Mean (X_{pt})	**1,72**
Robust standard deviation (S*)	**0,136**
Number with 2 replicates	6
Repeatability SD (S_r)	0,0390
Repeatability (CV_r)	2,23%
Reproducibility SD (S_R)	0,107
Reproducibility (CV_R)	6,15%
Target range:	
Target standard deviation σ_{pt}	**0,179**
Target standard deviation (for Information)	0,107
lower limit of target range	**1,36**
upper limit of target range	**2,08**
Quotient S/σ_{pt}*	*0,76*
Standard uncertainty $U_{(Xpt)}$	*0,0640*
Results in the target range	*6*
Percent in the target range	*86%*

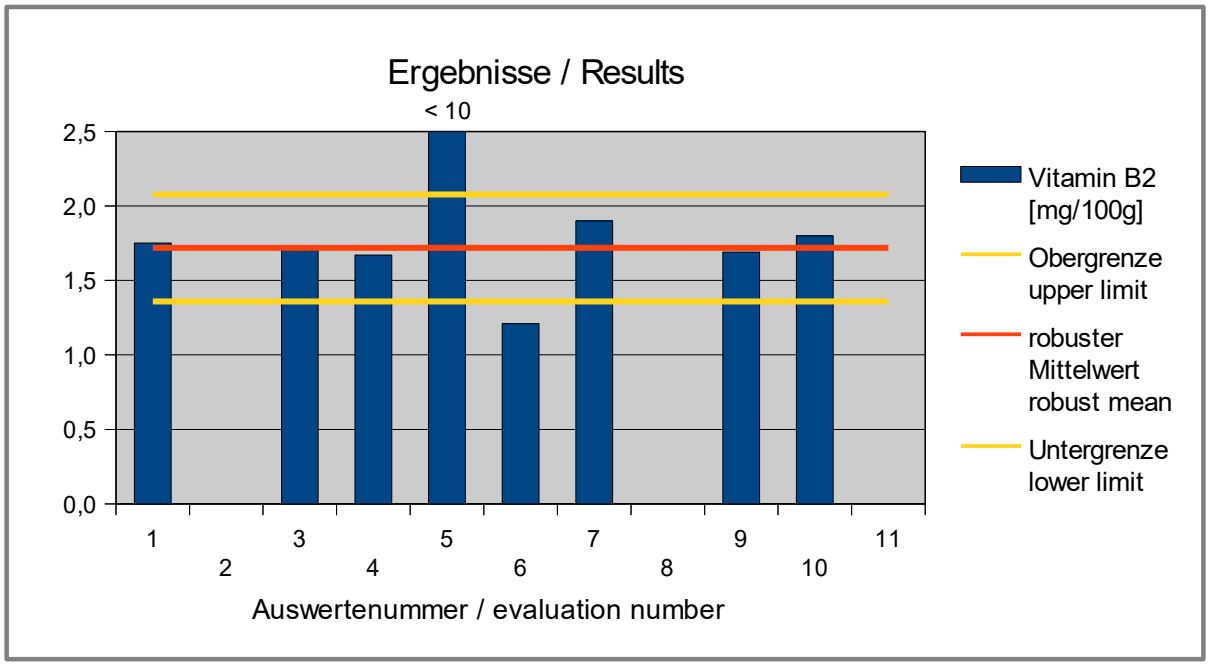


Abb. / Fig. 3: Ergebnisse Vitamin B2 / Results Vitamin B2

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Vitamin B2 [mg/100g]	Abweichung [mg/100g] Deviation [mg/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	1,75	0,032	0,18	0,30	
2					
3	1,70	-0,018	-0,10	-0,17	
4	1,67	-0,048	-0,27	-0,45	
5	<10				
6	1,21	-0,508	-2,8	-4,8	
7	1,90	0,182	1,0	1,7	
8					
9	1,69 *	-0,028	-0,16	-0,26	
10	1,80	0,082	0,46	0,77	
11					

* Mean calculated by DLA

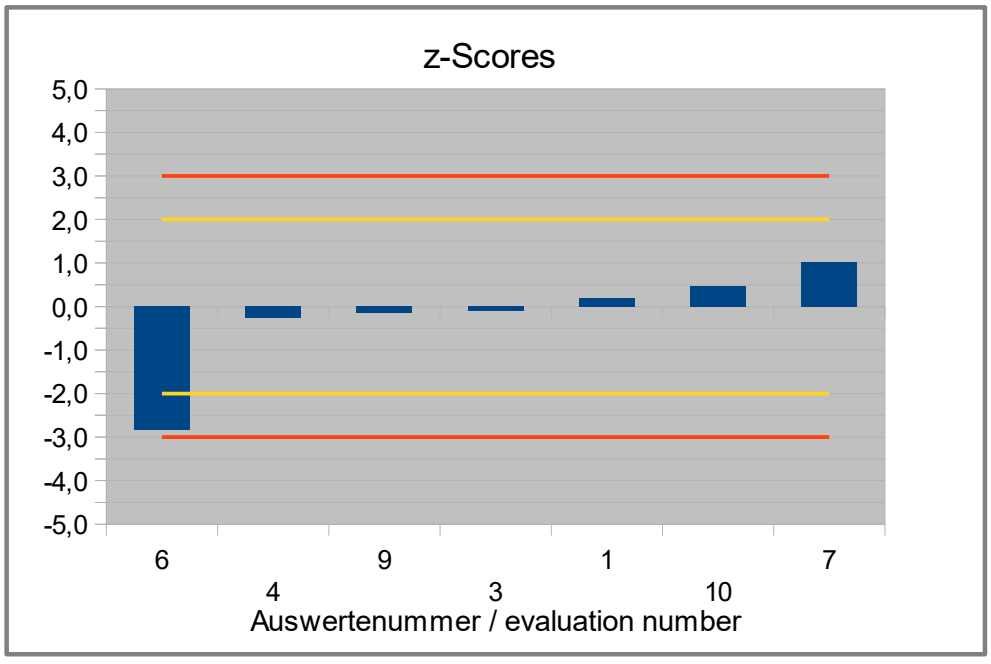


Abb. / Fig. 4: z-Scores Vitamin B2

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

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results°	5
Number of outliers	2
Mean	1,73
Robust Mean	2,13
Median (x_{pt})	**1,50**
Robust standard deviation (S*)	**0,67**
Number with 2 replicates	5
Repeatability SD (S_r)	0,0628
Repeatability (CV_r)	3,65%
Reproducibility SD (S_R)	0,732
Reproducibility (CV_R)	42,5%
Target range:	
Target standard deviation σ_{pt}'	**0,407**
Target standard deviation (for Information)	0,0841
lower limit of target range	**0,686**
upper limit of target range	**2,31**
Quotient S/σ_{pt}'*	*1,6*
Standard uncertainty $U_{(Xpt)}$	*0,374*
Results in the target range	*4*
Percent in the target range	*80%*

° *without outliers (results no. 1 + 10)*

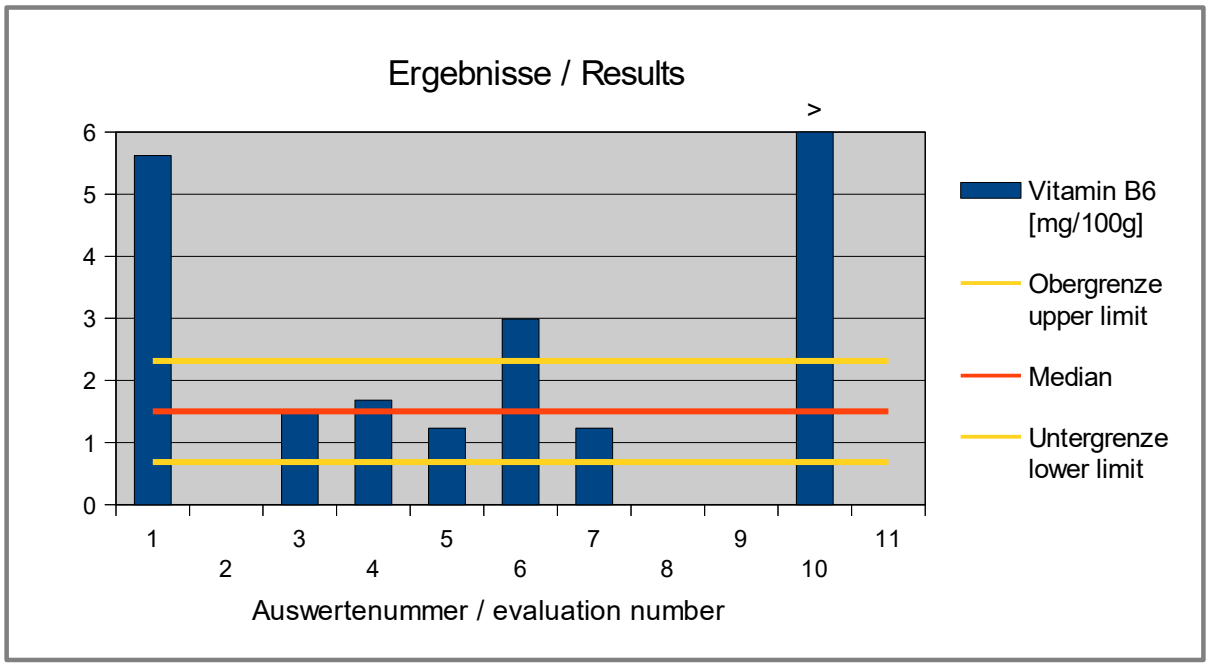


Abb. / Fig. 5: Ergebnisse Vitamin B6 / Results Vitamin B6

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Vitamin B6 [mg/100g]	Abweichung [mg/100g] Deviation [mg/100g]	z'-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	5,62				Ausreißer ausgeschlossen / Outlier excluded
2					
3	1,50	0,00	0,00	0,0	
4	1,68	0,18	0,44	2,1	
5	1,23	-0,27	-0,66	-3,2	
6	2,99	1,49	3,7	18	
7	1,23	-0,27	-0,66	-3,2	
8					
9					
10	18,6				Ausreißer ausgeschlossen / Outlier excluded
11					

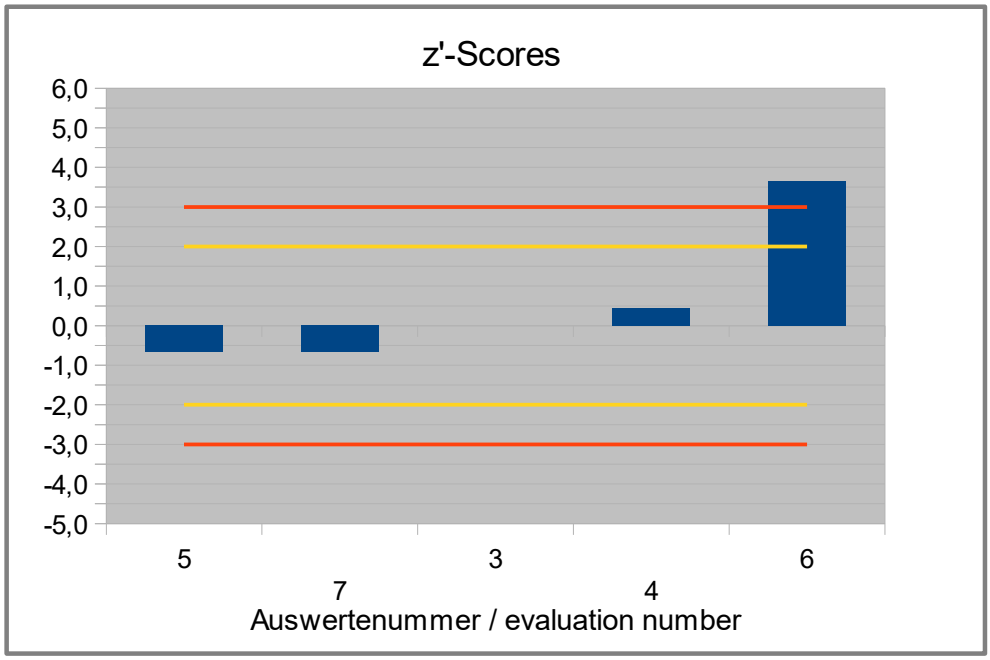


Abb. / Fig. 6: z'-Scores Vitamin B6

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

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results°	6
Number of outliers	1
Mean	2,46
Median	2,50
Robust Mean (x_{pt})	**2,46**
Robust standard deviation (S*)	**1,11**
Number with 2 replicates	6
Repeatability SD (S_r)	0,171
Repeatability (CV_r)	6,86%
Reproducibility SD (S_R)	1,04
Reproducibility (CV_R)	41,6%
Target range:	
Target standard deviation σ_{pt}	**0,688**
lower limit of target range	**1,09**
upper limit of target range	**3,84**
Quotient S/σ_{pt}*	*1,6*
Standard uncertainty $U_{(Xpt)}$	*0,567*
Results in the target range	*4*
Percent in the target range	*67%*

° *without outliers (results no. 10)*

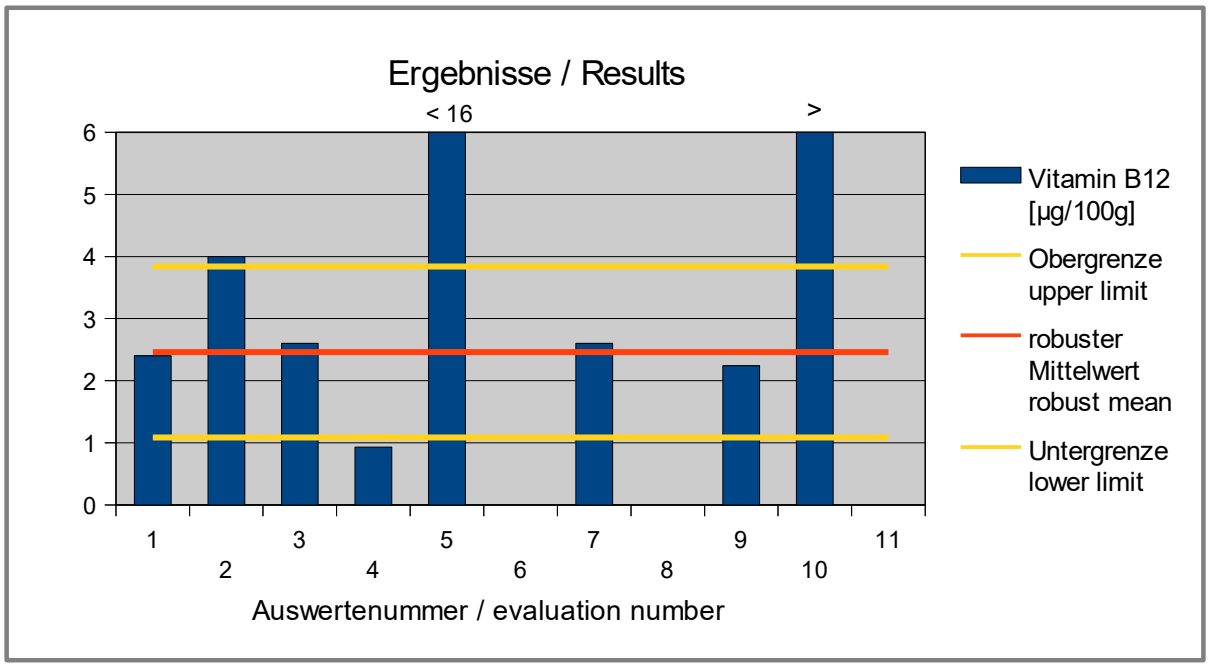


Abb. / Fig. 7: Ergebnisse Vitamin B12/ Results Vitamin B12

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Vitamin B12 [µg/100g]	Abweichung [µg/100g] Deviation [µg/100g]	z-Score (σ_{pt})	Hinweis Remark
1	2,40	-0,06	-0,09	
2	4,00	1,54	2,2	
3	2,60	0,14	0,20	
4	0,930	-1,53	-2,2	
5	<16			
6				
7	2,60	0,14	0,20	
8				
9	2,24	-0,22	-0,32	
10	1122			Ausreißer ausgeschlossen / Outlier excluded
11				

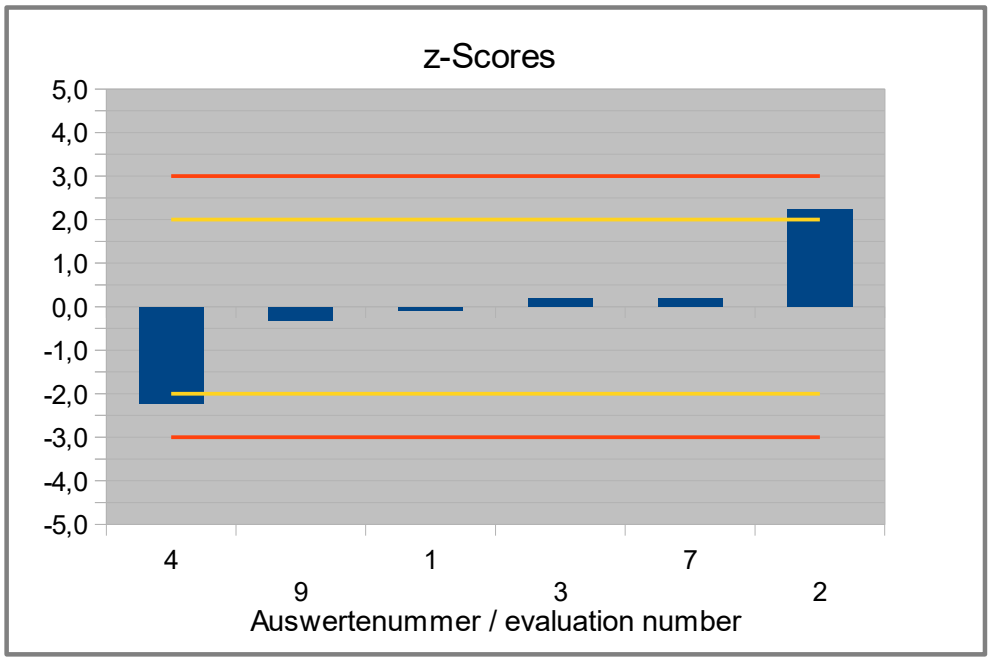


Abb. / Fig. 8: z-Scores Vitamin B12

4.5 Biotin (in µg/100g)

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	5
Number of outliers	
Mean	479
Robust Mean	59,0
Median (x_{pt})	**54,3**
Robust standard deviation (S*)	**42,1**
Number with 2 replicates	
Repeatability SD (S_r)	
Repeatability (CV_r)	
Reproducibility SD (S_R)	
Reproducibility (CV_R)	
Target range:	
Target standard deviation σ_{pt}	
Target standard deviation (for Information)	
lower limit of target range	
upper limit of target range	
Quotient S/σ_{pt}*	
Standard uncertainty $U_{(Xpt)}$	
Results in the target range	
Percent in the target range	

Note: Due to the small number and variation of the available results (5), no statistical analysis was carried out.

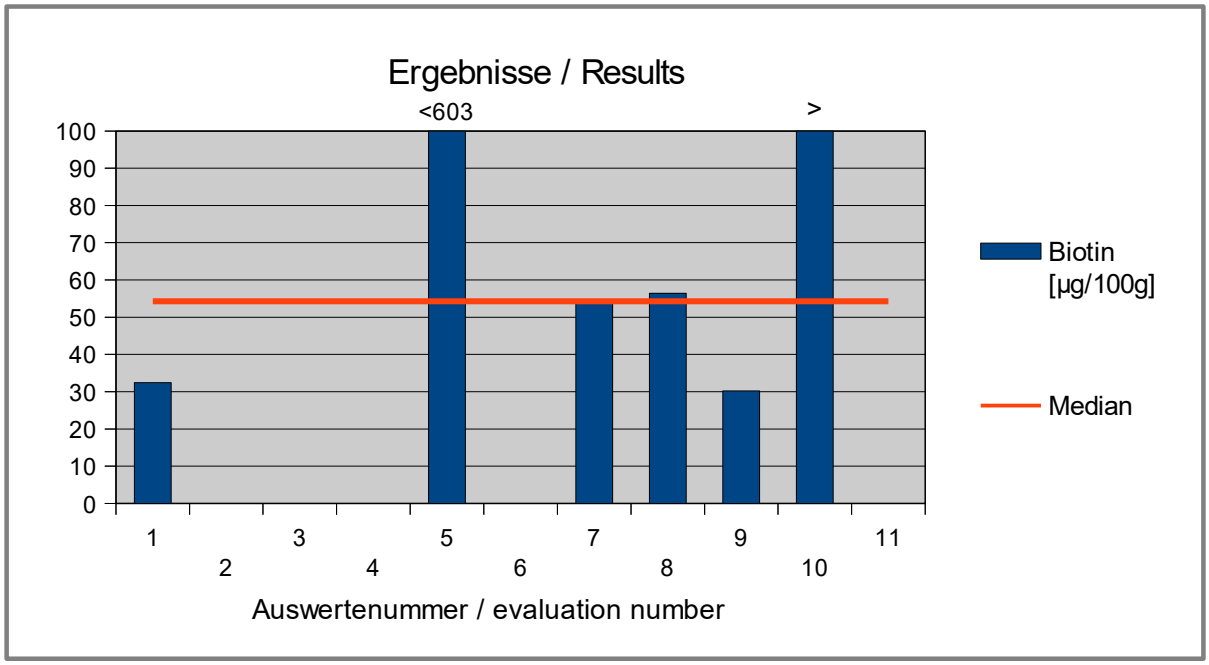


Abb. / Fig. 9: Ergebnisse Biotin/ Results Biotin

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Biotin [µg/100g]	Abweichung [µg/100g] Deviation [µg/100g]	z-Score (σ_{pt})	Hinweis Remark
1	32,4	-21,9		
2				
3				
4				
5	<603			
6				
7	54,3	0,0		
8	56,4 *	2,1		
9	30,2	-24,1		
10	2223	2168,4		
11				

* Mean calculated by DLA for information

4.6 Folic acid (as pteroylmonoglutamic acid in µg/100g)

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results°	6
Number of outliers	1
Mean	256
Median	254
Robust Mean (X_{pt})	**256**
Robust standard deviation (S*)	**23,8**
Number with 2 replicates	6
Repeatability SD (S_r)	22,3
Repeatability (CV_r)	8,68%
Reproducibility SD (S_R)	26,1
Reproducibility (CV_R)	10,2%
Target range:	
Target standard deviation σ_{pt}	**35,6**
lower limit of target range	**185**
upper limit of target range	**328**
Quotient S/σ_{pt}*	*0,67*
Standard uncertainty $U_{(Xpt)}$	*12,1*
Results in the target range	*6*
Percent in the target range	*100%*

° *without outliers (results no. 10)*

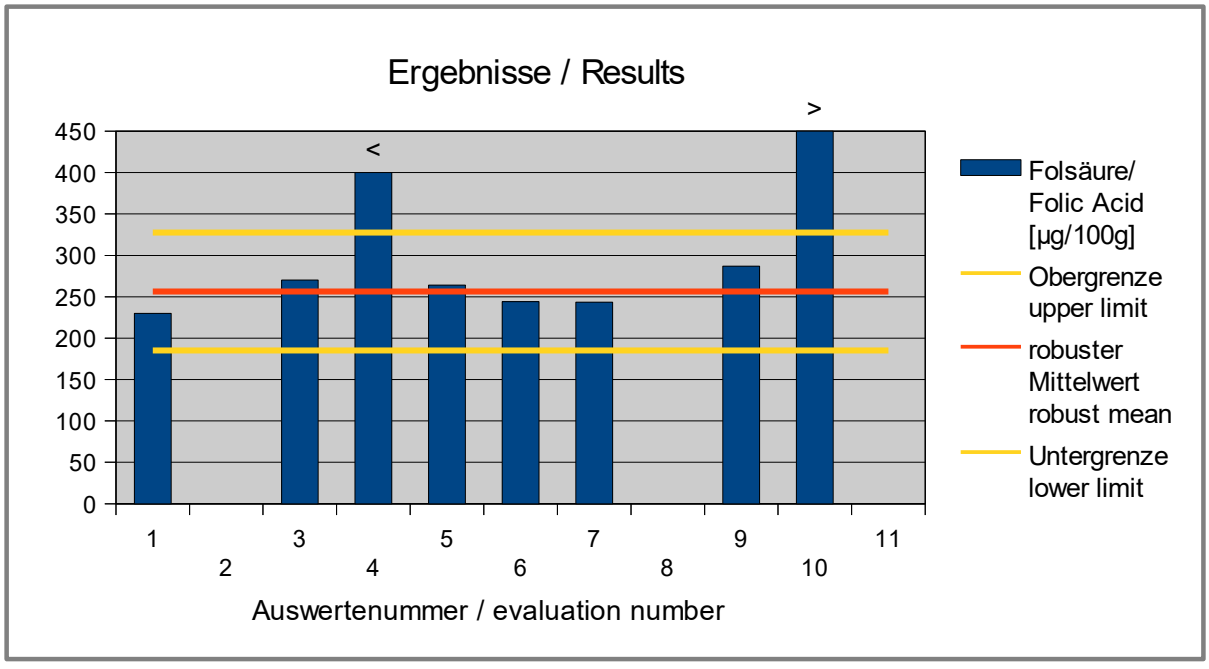


Abb. / Fig. 10: Ergebnisse Folsäure/ Results Folic Acid

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Folsäure/ Folic Acid [µg/100g]	Abweichung [µg/100g] Deviation [µg/100g]	z-Score (σ_{pt})	Hinweis Remark
1	230	-26,4	-0,74	
2				
3	270	13,6	0,38	
4	<400			
5	264	7,6	0,21	
6	244	-12,4	-0,35	
7	244	-12,9	-0,36	
8				
9	287	30,6	0,86	
10	7220			Ausreißer ausgeschlossen / Outlier excluded
11				

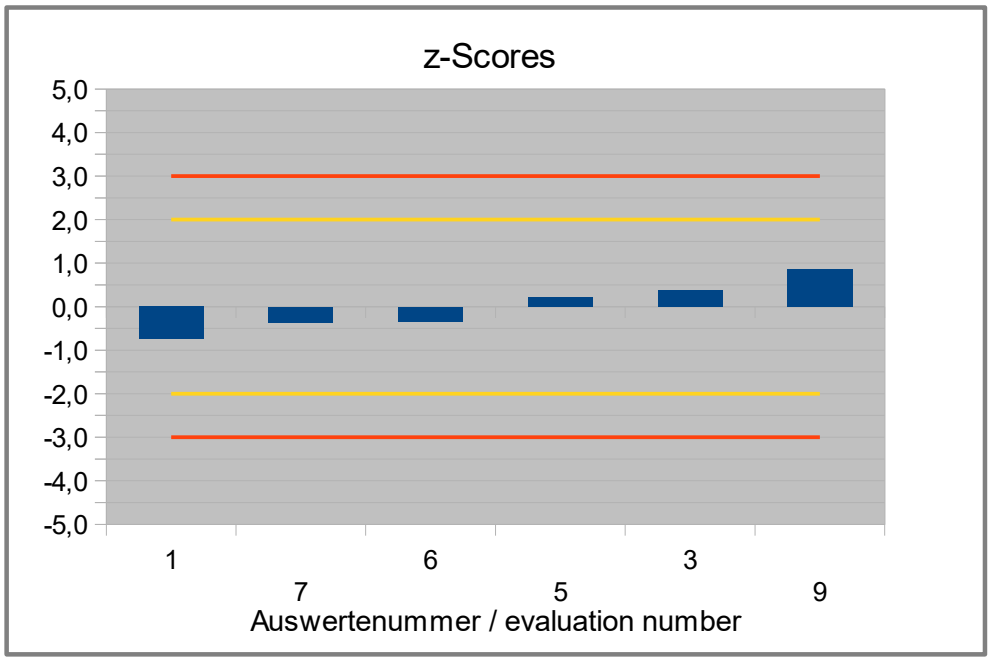


Abb. / Fig. 11: z-Scores Folsäure / Folic Acid

4.7 Niacin (in mg/100g)

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	8
Number of outliers	0
Mean	17,9
Median	18,3
Robust Mean (X_{pt})	**17,9**
Robust standard deviation (S*)	**1,96**
Number with 2 replicates	8
Repeatability SD (S_r)	0,597
Repeatability (CV_r)	3,34%
Reproducibility SD (S_R)	1,77
Reproducibility (CV_R)	9,92%
Target range:	
Target standard deviation σ_{pt}	**1,31**
Target standard deviation (for Information)	0,683
lower limit of target range	**15,3**
upper limit of target range	**20,5**
Quotient S/σ_{pt}*	*1,5*
Standard uncertainty $U_{(Xpt)}$	*0,868*
Results in the target range	*8*
Percent in the target range	*100%*

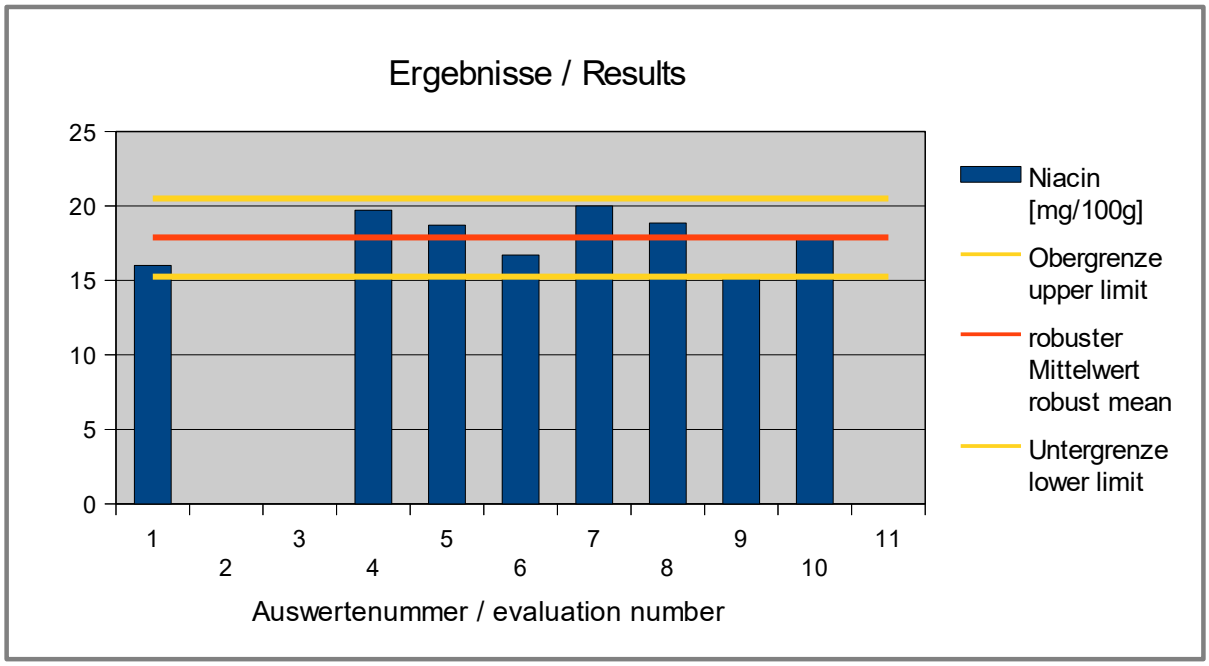


Abb. / Fig. 12: Ergebnisse Niacin / Results Niacin

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Niacin [mg/100g]	Abweichung [mg/100g] Deviation [mg/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	16,0	-1,88	-1,4	-2,8	
2					
3					
4	19,7	1,82	1,4	2,7	
5	18,7	0,82	0,62	1,2	
6	16,7	-1,18	-0,90	-1,7	
7	20,0	2,12	1,6	3,1	
8	18,9 *	0,97	0,74	1,4	
9	15,3	-2,58	-2,0	-3,8	
10	17,8	-0,08	-0,06	-0,12	
11					

* Mean calculated by DLA

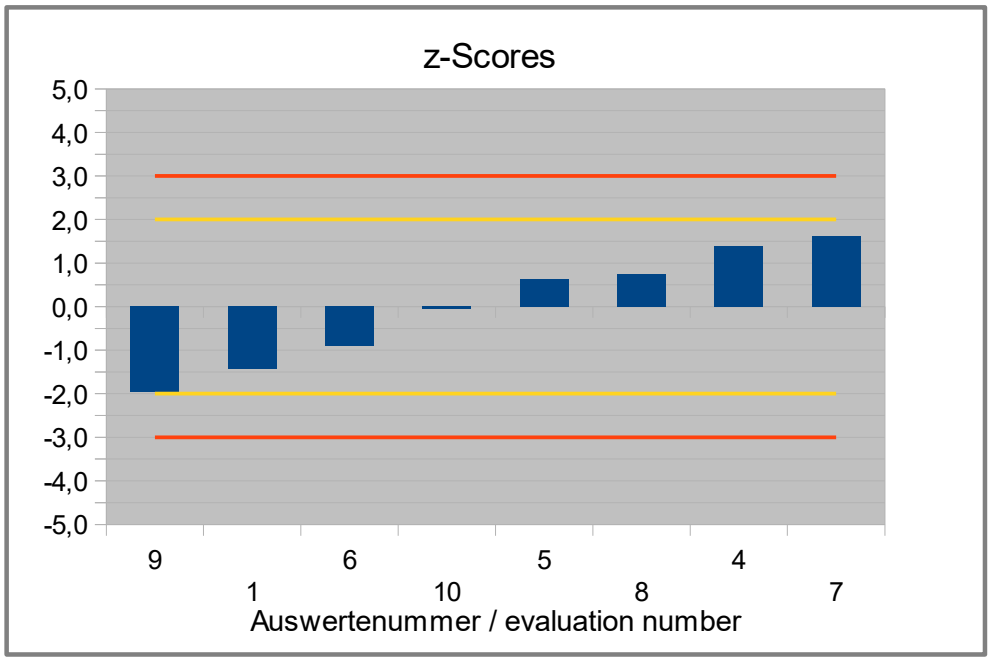


Abb. / Fig. 13: z-Scores Niacin

4.8 Pantothenic acid (in mg/100g)

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	6
Number of outliers	0
Mean	5,75
Median	5,84
Robust Mean (x_{pt})	**5,76**
Robust standard deviation (S*)	**0,955**
Number with 2 replicates	6
Repeatability SD (S_r)	0,551
Repeatability (CV_r)	9,59%
Reproducibility SD (S_R)	0,937
Reproducibility (CV_R)	16,3%
Target range:	
Target standard deviation σ_{pt}'	**0,699**
lower limit of target range	**4,36**
upper limit of target range	**7,15**
Quotient S/σ_{pt}'*	*1,4*
Standard uncertainty $U_{(Xpt)}$	*0,487*
Results in the target range	*5*
Percent in the target range	*83%*

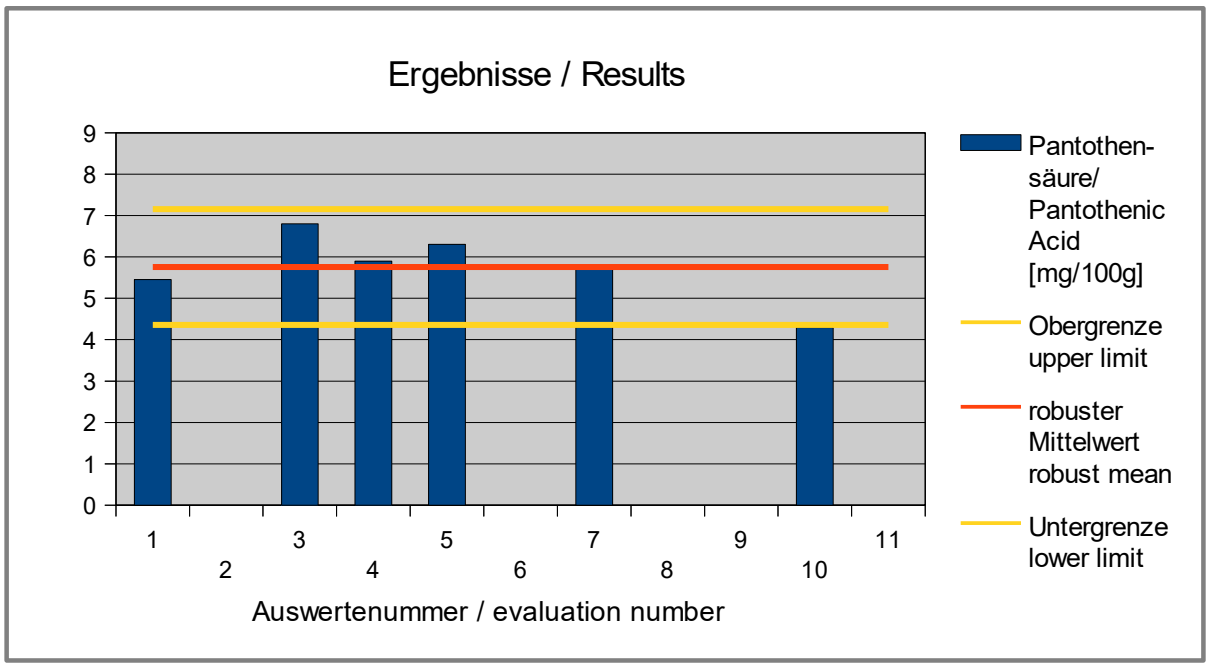


Abb. / Fig. 14: Ergebnisse Pantothensäure/ Results Pantothenic Acid

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Pantothen-säure/ Pantothenic Acid [mg/100g]	Abweichung [mg/100g] Deviation [mg/100g]	z'-Score (σ_{pt})	Hinweis Remark
1	5,45	-0,308	-0,44	
2				
3	6,80	1,042	1,5	
4	5,90	0,142	0,20	
5	6,30	0,542	0,78	
6				
7	5,77	0,012	0,02	
8				
9				
10	4,30	-1,463	-2,1	
11				

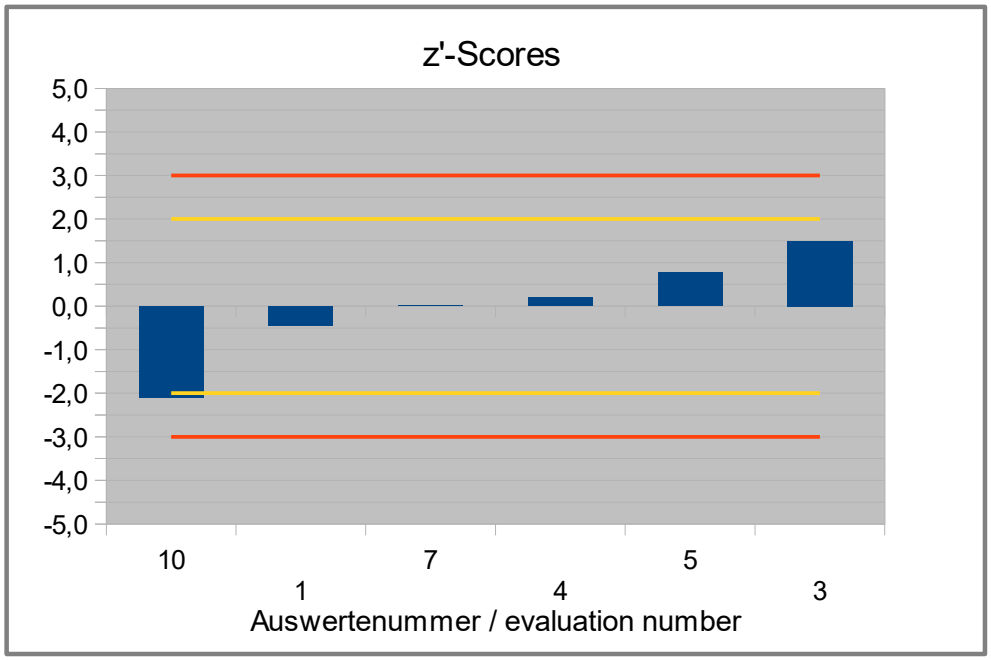


Abb. / Fig. 15: z'-Scores Pantothensäure/ Pantothenic Acid

4.9 Vitamin C (as ascorbic acid in mg/100g)

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	9
Number of outliers	–
Mean	98,6
Median	78,5
Robust Mean (x_{pt})	**80,0**
Robust standard deviation (S*)	**13,0**
Number with 2 replicates	8
Repeatability SD (S_r)	5,62
Repeatability (CV_r)	7,49%
Reproducibility SD (S_R)	15,7
Reproducibility (CV_R)	21,0%
Target range:	
Target standard deviation σ_{pt}	**8,40**
Target standard deviation (for Information)	4,68
lower limit of target range	**63,2**
upper limit of target range	**96,8**
Quotient S/σ_{pt}*	*1,6*
Standard uncertainty $U_{(Xpt)}$	*5,43*
Results in the target range	7
Percent in the target range	78%

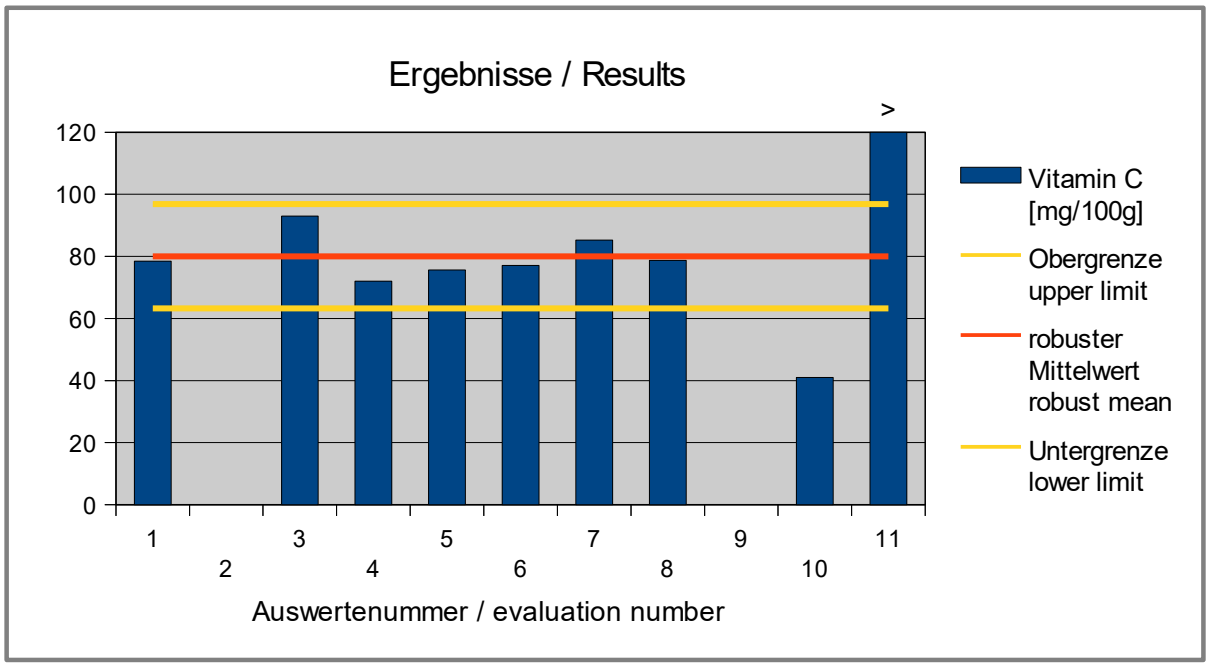


Abb. / Fig. 16: Ergebnisse Vitamin C/ Results Vitamin C

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Vitamin C [mg/100g]	Abweichung [mg/100g] Deviation [mg/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	78,5	-1,5	-0,18	-0,33	
2					
3	93,0	13,0	1,5	2,8	
4	72,0	-8,0	-0,96	-1,7	
5	75,6	-4,4	-0,53	-0,95	
6	77,1	-2,9	-0,35	-0,63	
7	85,3	5,2	0,62	1,1	
8	78,8 *	-1,3	-0,15	-0,27	
9					
10	41,0	-39,0	-4,6	-8,3	
11	286	206,0	25	44	

* Mean calculated by DLA

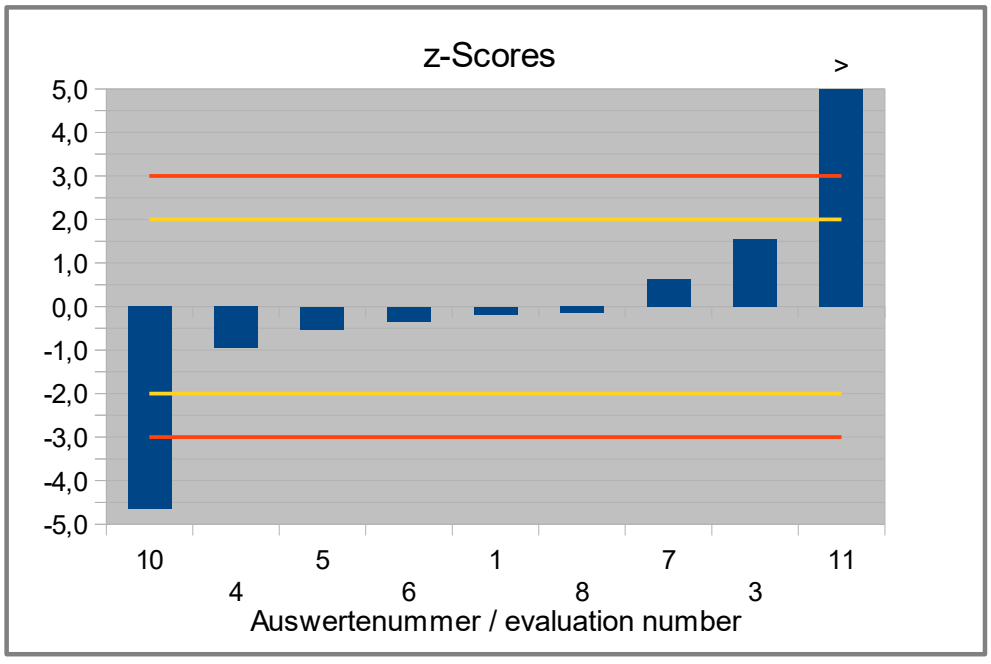


Abb. / Fig. 17: z-Scores Vitamin C

4.10 Participant z-Scores: overview table

Evaluation number	Vitamin B1	Vitamin B2	Vitamin B6	Vitamin B12	Folic Acid	Niacin	Pantothenic Acid	Vitamin C
	z'-Score	z-Score	z'-Score	z-Score	z-Score	z-Score	z-Score	z-Score
1	-0,81	0,18	-	-0,06	-0,74	-1,4	-0,44	-0,18
2	-	-	-	1,5	-	-	-	-
3	-2,0	-0,10	0,00	0,14	0,38	-	1,5	1,5
4	0,64	-0,27	0,44	-1,5	-	1,4	0,20	-0,96
5	2,3	-	-0,66	-	0,21	0,62	0,8	-0,53
6	-1,6	-2,8	3,7	-	-0,35	-0,90	-	-0,35
7	-0,18	1,0	-0,66	0,14	-0,36	1,6	0,02	0,62
8	0,84	-	-	-	-	0,74	-	-0,15
9	0,86	-0,16	-	-0,22	0,86	-2,0	-	-
10	-	0,46	-	-	-	-0,06	-2,1	-4,6
11	-	-	-	-	-	-	-	25

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

Parameter	Partici-pant	Unit	Sample I DLA No.	Sample II DLA No.	Date of analysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quan-tification	Incl. Recovery Rate	Recovery rate
					Day/Month					yes / no	in %
Vitamin B1 (als Thiamin-Kation/ as Thiamine-cation)	1	mg/100g	23	69	22.05.20	1,04	1,03	1,05	0,02	no	-
	2	mg/100g									
	3	mg/100g	39 + 29	53 + 63	28.04.2020 to 11.06.2020	0,74	0,7	0,77	0,2	no	97
	4	mg/100g	28	64	07.05.20	1,41	1,38	1,43		no	
	5	mg/100g	44	48	10.06.20	1,82	1,67	1,97	1,5	no	-
	6	mg/100g	5	87		0,83	0,93	0,73			
	7	mg/100g	19	73	22.04.20	1,2	1,158	1,247	0,002	no	
	8	mg/100g	65	66	16.06.20		1,46	1,46			
	9	mg/100g	68	24	10.06.20	18.06.20	1,47	1,46	0,01	no	-
	10	mg/100g	34	58	08.06.20	13,05	13,6	12,5	-	-	-
	11	mg/100g	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A

Parameter	Partici-pant	Unit	Sample I DLA No.	Sample II DLA No.	Date of analysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quan-tification	Incl. Recovery Rate	Recovery rate
					Day/Month					yes / no	in %
Vitamin B2 (als Riboflavin / as Riboflavin)	1	mg/100g	23	69	22.05.20	1,75	1,74	1,75	0,02	no	-
	2	mg/100g									
	3	mg/100g				1,7	1,7	1,6	0,2	no	83
	4	mg/100g	28	64	07.05.20	1,67	1,62	1,71		no	
	5	mg/100g	44	48	10.06.20	<10	<10	<10	10	no	-
	6	mg/100g	5	87		1,21	1,36	1,06			
	7	mg/100g	19	73	22.04.20	1,9	1,929	1,921	0,002	no	
	8	mg/100g	65	66	16.06.20						
	9	mg/100g	68	24	10.06.20	18.06.20	1,69	1,69	0,01	no	-
	10	mg/100g	34	58	08.06.20	1,8	1,8	1,8	-	-	-
	11	mg/100g	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A

Parameter	Partici-pant	Unit	Sample I DLA No.	Sample II DLA No.	Date of analysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quan-tification	Incl. Recovery Rate	Recovery rate
					Day/Month					yes / no	in %
Vitamin B6 (als Pyridoxin/ as Pyridoxine)	1	mg/100g	23	69	20.05.20	5,62	5,78	5,46	0,04	no	-
	2	mg/100g									
	3	mg/100g				1,5	1,5	1,5	0,1	no	97
	4	mg/100g	28	64	07.05.20	1,68	1,6	1,75		no	
	5	mg/100g	44	48	10.06.20	1,23	1,19	1,26	1	no	-
	6	mg/100g	5	87		2,99	3,04	2,93			
	7	mg/100g	19	73	17.06.20	1,23	1,23	1,23	0,002	no	
	8	mg/100g									
	9	mg/100g									
	10	mg/100g	34	58	08.06.20	18,55	18,4	18,7	-	-	-
	11	mg/100g	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A

Parameter	Partici-pant	Unit	Sample I DLA No.	Sample II DLA No.	Date of analysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quan-tification	Incl. Recovery Rate	Recovery rate
					Day/Month					yes / no	in %
Vitamin B12 (als Cyanoco-balamin/ as Cyanoco-balamine)	1	µg/100g	23	69	15.05.20	2,4	2,3	2,5	0,1	no	-
	2	µg/100g	22	70	03.06.20	4	4	4,3	1	no	82,4
	3	µg/100g				2,6	2,8	2,4	0,9	no	109
	4	µg/100g	28	64	07.05.20	0,93	0,95	0,91		no	
	5	µg/100g	44	48	08.05.20	<16	<16	<16	16	no	-
	6	µg/100g	n/a	n/a		n/a	n/a	n/a			
	7	µg/100g	19	73	07.05.20	2,6	2,72	2,48	0,03	no	
	8	µg/100g									
	9	µg/100g	68	24	14.05.	2,24	2,25	2,23	0,03	no	
	10	µg/100g	34	58	08.06.20	1122,05	1068,9	1175,2	-	-	-
	11	µg/100g	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A

Parameter	Partici-pant	Unit	Sample I DLA No.	Sample II DLA No.	Date of analysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quan-tification	Incl. Recovery Rate	Recovery rate
					Day/Month					yes / no	in %
Biotin	1	µg/100g	23	69	26.05.20	32,4	33,4	31,4	2	no	-
	2	µg/100g									
	3	µg/100g				not determined					
	4	µg/100g									
	5	µg/100g	44	48	12.06.20	<603	<603	<603	603	no	-
	6	µg/100g	n/a	n/a		n/a	n/a	n/a			
	7	µg/100g	19	73	20. Mai & 04. Juni	54,3	49,3	59,3	0,08	no	
	8	µg/100g	65	66	16.06.20		44,4	68,4			
	9	µg/100g	68	24	10.06.	30,2	31,5	28,9	0,08	no	
	10	µg/100g	34	58	08.06.20	2222,65	1847,1	2598,2	-	-	-
	11	µg/100g	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A

Parameter	Partici-pant	Unit	Sample I DLA No.	Sample II DLA No.	Date of analysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quan-tification	Incl. Recovery Rate	Recovery rate
					Day/Month					yes / no	in %
Folsäure (als Pteroylmono-glutaminsäure) Folic acid (as Pteroylmono-glutaminic acid)	1	µg/100g	23	69	21.05.20	230	200	260	1	no	-
	2	µg/100g									
	3	µg/100g				270	280	260	64	no	83
	4	µg/100g	28	64	07.05.20	<400	<400	<400		no	
	5	µg/100g	44	48	12.06.20	264	266	261	100	no	-
	6	µg/100g	5	87		244	254	234			
	7	µg/100g	19	73	06.05.20	243,5	256,5	230,5	0,2	no	
	8	µg/100g									
	9	µg/100g	68	24	11.06.	287	272	301	0,16	no	
	10	µg/100g	34	58	08.06.20	7219,9	7383,2	7056,6	-	-	-
	11	µg/100g	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A

Parameter	Partici-pant	Unit	Sample I DLA No.	Sample II DLA No.	Date of analysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quan-tification	Incl. Recovery Rate	Recovery rate
					Day/Month					yes / no	in %
Niacin	1	mg/100g	23	69	14.05.20	16	16,1	15,9	0,2	no	-
	2	mg/100g									
	3	mg/100g				not determined					
	4	mg/100g	28	64	07.05.20	19,7	19,5	19,8		no	
	5	mg/100g	44	48	10.06.20	18,7	19,1	18,2	2	no	-
	6	mg/100g	5	87		16,7	16,9	16,5			
	7	mg/100g	19	73	08.05.20	20	20	20	0,02	no	
	8	mg/100g	65	66	16.06.20		18,3	19,4			
	9	mg/100g	68	24	14.05.	15,3	14,4	16,2	0,016	no	
	10	mg/100g	34	58	08.06.20	17,8	17,6	18	-	-	-
	11	mg/100g	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A

Parameter	Partici-pant	Unit	Sample I DLA No.	Sample II DLA No.	Date of analysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quan-tification	Incl. Recovery Rate	Recovery rate
					Day/Month					yes / no	in %
Pantothensäure/ Pantothenic acid	1	mg/100g	23	69	21.05.20	5,45	5,53	5,36	0,05	no	-
	2	mg/100g									
	3	mg/100g				6,8	6,6	7	0,1	no	104
	4	mg/100g	28	64	07.05.20	5,9	6	5,8		no	
	5	mg/100g	44	48	10.06.20	6,3	6,64	5,95	1	no	-
	6	mg/100g	n/a	n/a		n/a	n/a	n/a			
	7	mg/100g	19	73	08.05.20	5,77	5,84	5,7	0,04	no	
	8	mg/100g									
	9	mg/100g									
	10	mg/100g	34	58	08.06.20	4,295	3,44	5,15	-	-	-
	11	mg/100g	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A

Parameter	Partici-pant	Unit	Sample I DLA No.	Sample II DLA No.	Date of analysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quan-tification	Incl. Recovery Rate	Recovery rate
					Day/Month					yes / no	in %
Vitamin C (als Ascorbinsäure/ as Ascorbic acid)	1	mg/100g	23	69	12.05.20	78,5	78,6	78,3	2	no	-
	2	mg/100g									
	3	mg/100g				93	93	93	1	no	132
	4	mg/100g	28	64	05.05.20	72	65,4	78,5		no	
	5	mg/100g	44	48	16.06.20	75,6	72	79,2	1	no	-
	6	mg/100g	5	87		77,1	83,1	71			
	7	mg/100g	19	73	08./09. Apr	85,27	87,85	82,69	2,5	no	
	8	mg/100g	65	66	16.06.20		74	83,5			
	9	mg/100g									
	10	mg/100g	34	58	18.06.20	41	38,8	43,2	-	-	-
	11	mg/100g	30	62	16.06.20	286	287	284	N/A	no	N/A

5.1.2 Analytical Methods

Parameter	Partici-pant	Method description as in test report / norm / literature	Sample preparation and processing	Measuring method	Calibration / Refe-rence material	Recovery rate with same matrix	Method accredited ISO/IEC 17025	Further Remarks
						yes / no	yes / no	
Vitamin B1 (als Thiamin-Kation/ as Thiamine-cation)	1	HPLC/FD - internal method PNTA0128			external calib. curve and internal RM	no	yes	
	2							
	3	yes			PT- material		yes	
	4	HPLC - DAD / FLD					yes	
	5	HPLC	-	HPLC-UV	external calibr.	no	no	-
	6							
	7	§64 LFGB L 00.00-83					yes	
	8							
	9	PV 0016-04 (ASU L 00.00-83 (2015-06))		HPLC	11.06.2020* / LVU 17-20 (MP)	no	yes	* Thiamine chlo-ride hydrochloride
	10	house method	Liquid extraction	HPLC-DAD	-	-	no	-
	11	N/A	N/A	N/A	N/A	N/A	N/A	N/A

Parameter	Partici-pant	Method description as in test report / norm / literature	Sample preparation and processing	Measuring method	Calibration / Refe-rence material	Recovery rate with same matrix	Method accredited ISO/IEC 17025	Further Remarks
						yes / no	yes / no	
Vitamin B2 (als Riboflavin / as Riboflavin)	1	HPLC/FD - internal method PNTA0128			external calib. curve and internal RM	no	yes	
	2							
	3	yes			PT- material		yes	
	4	HPLC - DAD / FLD					yes	
	5	HPLC	-	HPLC-UV	external calibr.	no	no	-
	6							
	7	§64 LFGB L 00.00-84					yes	
	8							
	9	PV 0017-03 (ASU L 00.00-84 (2015-06))		HPLC	02.03.2020* /LVU 17-20 (MP)	no	yes	*Riboflavins
	10	house method	Liquid extraction	HPLC-DAD	-	-	no	-
	11	N/A	N/A	N/A	N/A	N/A	N/A	N/A

Parameter	Partici-pant	Method description as in test report / norm / literature	Sample preparation and processing	Measuring method	Calibration / Refe-rence material	Recovery rate with same matrix	Method accredited ISO/IEC 17025	Further Remarks
						yes / no	yes / no	
Vitamin B6 (als Pyridoxin/ as Pyridoxine)	1	HPLC/FD - internal method PNTA0056			external calib. curve and internal RM	no	yes	
	2							
	3	yes			PT- material		yes	
	4	HPLC - DAD / FLD					yes	
	5	HPLC	-	HPLC-UV	external calibr.	no	no	-
	6							
	7	Vita Fast Vitamin B6 (RBiopharm)					no	Method not yet validated here
	8							
	9							
	10	house method	Liquid extraction	HPLC-DAD	-	-	no	-
	11	N/A	N/A	N/A	N/A	N/A	N/A	N/A

Parameter	Partici-pant	Method description as in test report / norm / literature	Sample preparation and processing	Measuring method	Calibration / Refe-rence material	Recovery rate with same matrix	Method accredited ISO/IEC 17025	Further Remarks
						yes / no	yes / no	
Vitamin B12 (als Cyanoco-balamin/ as Cyanoco-balamine)	1	HPLC/DAD - internal method PNTA0137			external calib. curve and internal RM	no	yes	
	2	Extraction with mix of water:methanol 60:40 by sonication. Analitycal determination with LC/MSMS.			Calibration range: 10-100 ug/l	YES	YES	
	3	yes			PT- material		yes	
	4	HPLC / VIS,MS					yes	
	5	HPLC	-	HPLC-UV	external calibr.	no	no	-
	6							
	7	Vita Fast Vitamin B12 (RBiopharm)					yes	
	8							
	9	Microbiological (VitaFast Vitamin B12 - Cyanocobalamin / r-biopharm Art. Nr. P1002)			SRM 3280	no	yes	
	10	house method	Liquid extraction	HPLC-DAD	-	-	no	-
	11	N/A	N/A	N/A	N/A	N/A	N/A	N/A

Parameter	Partici-pant	Method description as in test report / norm / literature	Sample preparation and processing	Measuring method	Calibration / Refe-rence material	Recovery rate with same matrix	Method accredited ISO/IEC 17025	Further Remarks
						yes / no	yes / no	
Biotin	1	LC/MS/MS - internal method PNTA0164			external calib. curve and internal RM	no	yes	
	2							
	3							
	4							
	5	HPLC	-	HPLC-UV	external calibr.	no	no	-
	6							
	7	Vita Fast Biotin (RBiopharm)					yes	Results inhomo-geneous, espe-cially noticeable deviations bet-ween the two samples, several processing / measurements necessary
	8							
	9	Microbiological (VitaFast Vitamin B7 - Biotin / r-biopharm Art. Nr. P1003)			SRM 3280	no	yes	
	10	house method	Liquid extraction	HPLC-DAD	-	-	no	-
	11	N/A	N/A	N/A	N/A	N/A	N/A	N/A

Parameter	Partici-pant	Method description as in test report / norm / literature	Sample preparation and processing	Measuring method	Calibration / Refe-rence material	Recovery rate with same matrix	Method accredited ISO/IEC 17025	Further Remarks
						yes / no	yes / no	
Folsäure (als Pteroylmono-glutaminsäure) Folic acid (as Pteroylmono-glutaminic acid)	1	HPLC/DAD - internal method PNTA0138			external calib. curve and internal RM	no	yes	
	2							
	3	yes			PT- material		yes	
	4	HPLC - DAD / FLD					yes	
	5	HPLC	-	HPLC-UV	external calibr.	no	no	-
	6							
	7	Vita Fast Folic acid (RBiopharm)					yes	
	8							
	9	Microbiological (VitaFast Vitamin B9 – Folic acid / r-biopharm Art. Nr. P1001)			SRM 3280	no	yes	
	10	house method	Liquid extraction	HPLC-DAD	-	-	no	-
	11	N/A	N/A	N/A	N/A	N/A	N/A	N/A

Parameter	Partici-pant	Method description as in test report / norm / literature	Sample preparation and processing	Measuring method	Calibration / Refe-rence material	Recovery rate with same matrix	Method accredited ISO/IEC 17025	Further Remarks
						yes / no	yes / no	
Niacin	1	HPLC/FD - internal method PNTA0147			external calib. curve and internal RM	no	yes	
	2							
	3							
	4	HPLC - DAD / FLD					yes	
	5	HPLC	-	HPLC-UV	external calibr.	no	no	-
	6							
	7	internal method HPLC		HPLC based on VDLUFA Method Book III No. 13.9.1			yes	
	8							
	9	Microbiological (VitaFast Vitamin B3 - Niacin / r-biopharm Art. Nr. P1004)			SRM 3280	no	yes	
	10	house method	Liquid extraction	HPLC-DAD	-	-	no	-
	11	N/A	N/A	N/A	N/A	N/A	N/A	N/A

Parameter	Partici-pant	Method description as in test report / norm / literature	Sample preparation and processing	Measuring method	Calibration / Refe-rence material	Recovery rate with same matrix	Method accredited ISO/IEC 17025	Further Remarks
						yes / no	yes / no	
Pantothensäure/ Pantothenic acid	1	LC/MS/MS - internal method PNTA0148			external calib. curve and internal RM	no	yes	
	2							
	3	yes			PT- material		yes	
	4	LC / MS					yes	
	5	HPLC	-	HPLC-UV	external calibr.	no	no	-
	6							
	7	Vita Fast Pantothenic acid (RBiopharm)					yes	
	8							
	9							
	10	house method	Liquid extraction	HPLC-DAD	-	-	no	-
	11	N/A	N/A	N/A	N/A	N/A	N/A	N/A

Parameter	Partici-pant	Method description as in test report / norm / literature	Sample preparation and processing	Measuring method	Calibration / Refe-rence material	Recovery rate with same matrix	Method accredited ISO/IEC 17025	Further Remarks
						yes / no	yes / no	
Vitamin C (als Ascorbinsäure/ as Ascorbic acid)	1	HPLC/DAD - internal method PNTA0127			external calib. curve and internal RM	no	yes	
	2							
	3	yes			PT- material		yes	
	4	HPLC - DAD					yes	
	5	HPLC	-	HPLC-UV	external calibr.	no	no	-
	6							
	7	internal method HPLC					yes	
	8							
	9							
	10	house method	Liquid extraction	HPLC-DAD	-	-	yes	-
	11	MQLTM-0149 by Titration	N/A	MQLTM-0149	N/A	N/A	Yes	N/A

5.2 Homogeneity

5.2.1 Mixture homogeneity before bottling

Microtracer Homogeneity Test
DLA ptSU06

Weight whole sample	4,72	kg
Microtracer	FSS-rot lake	
Particle size	75 – 300	µm
Weight per particle	2,0	µg
Addition of tracer	22,7	mg/kg

Result of analysis

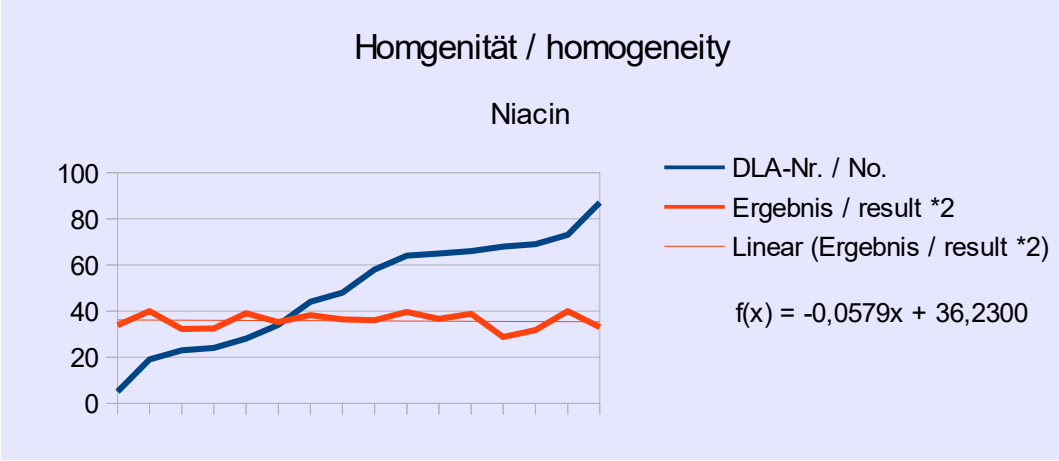
Sample	Weight [g]	Particle number	Particles [mg/kg]
1	5,05	58	23,0
2	5,00	57	22,8
3	5,01	67	26,7
4	5,06	71	28,1
5	5,03	63	25,0
6	5,01	65	25,9
7	4,98	61	24,5
8	5,05	70	27,7

Poisson distribution		
Number of samples	8	
Degree of freedom	7	
Mean	64,0	Particles
Standard deviation	5,04	Particles
χ^2 (CHI-Quadrat)	2,78	
Probability	**90**	%
Recovery rate	112	%

Normal distribution		
Number of samples	8	
Mean	25,5	mg/kg
Standard deviation	2,01	mg/kg
rel. Standard deviaton	7,87	%
Horwitz standard deviation	9,83	%
HorRat-value	**0,80**	
Recovery rate	112	%

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:



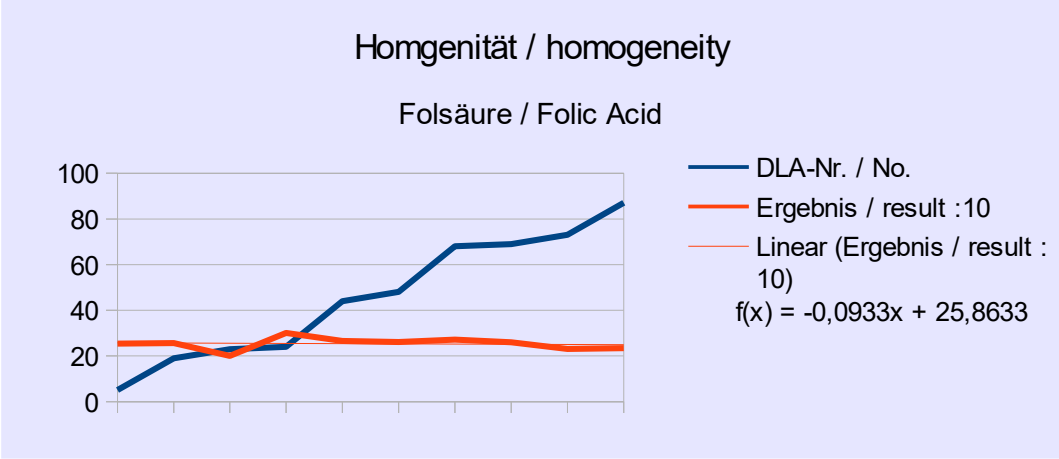


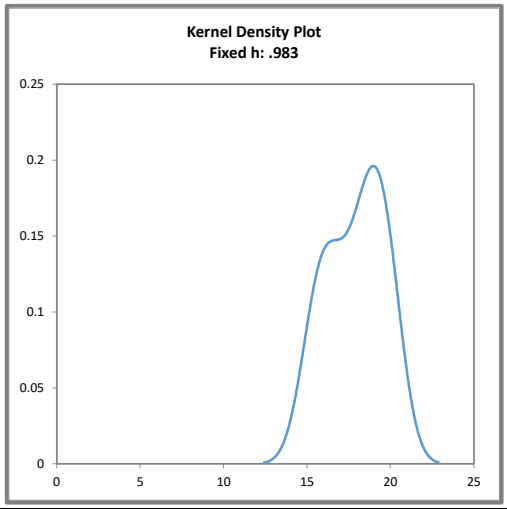
Abb./Fig. 18: Trendfunktion Probennummern vs. Ergebnisse: Niacin und Folsäure (1*2 und 1/10 dargestellt)
trend line function sample number vs. results: niacin and folic acid (1*2 and 1/10 shown)

5.3 Kernel Density Plots of Results

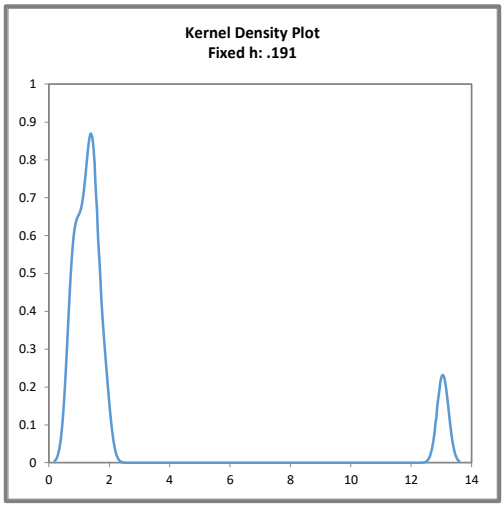
Abbildungen:
Kerndichte-Schätzungen
der Teilnehmerergebnisse
(mit h = 0,75 x σ_{pt} von Xpt)

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

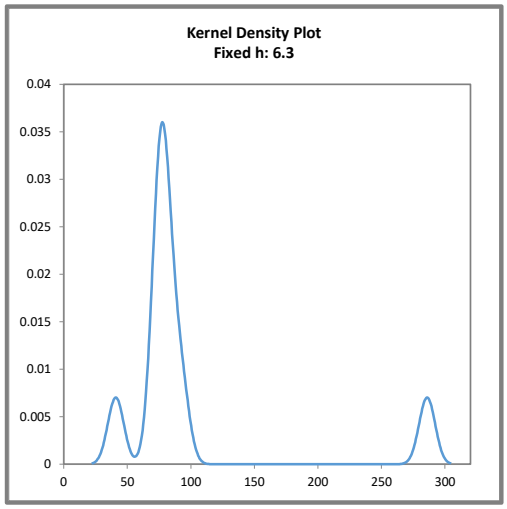
Niacin



Vitamin B1



Vitamin C



andere Parameter / other parameters

< 8 Ergebnisse

< 8 Results

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 ptSU06 (2020)***
PT name	***Dietetic-Product I: Vitamins B1, B2, B6, B12, Biotin, Vitamin C, Folic Acid , Niacin and Pantothenic Acid***
*Sample matrix**	***Samples I + II:*** *Dietetic food as a meal replacement (drink powder) / ingredients: soy protein isolate, milk protein, honey, skimmed milk powder, yoghurt powder, soybean oil, vitamins, minerals and other food additives*
Number of samples and sample amount	*2 identical samples I + II, 50 g each.*
Storage	*Samples I + II: cooled 2 - 10°C*
Intentional use	*Laboratory use only (quality control samples)*
Parameter	*quantitative: Vitamins B1, B2, B6, B12, Biotin, Vitamin C, Folic Acid, Niacin and Pantothenic Acid* *Contents: The contents are of the order of the nutrient reference values per recommended daily dose (approx. 25 g)*
Methods of analysis	*Analytical methods are optional*
Notes to analysis	*The analysis of PT samples should be performed like a routine laboratory analysis.* *In general we recommend to homogenize a representative sample amount before analysis according to good laboratory practice, especially in case of low sample weights.*
Result sheet	*The results for sample I and II as well as the final results calculated as mean of the double determination (samples I and II) should be filled in the result submission file. The recovery rates, if carried out, has to be included in the calculation.*
Units	*mg/100 g and µg/100 g , respectively (see results file)*
Number of significant digits	*at least 2*
Further information	*For information please specify:* – *Date of analysis* – *DLA-sample-numbers (for sample I and II)* – *Limit of detection* – *Assignment incl. Recovery* – *Recovery with the same matrix* – *Method is accredited*
Result submission	*The result submission file should be sent by e-mail to:* ***pt@dla-lvu.de***
Last Deadline	***the latest June 19th 2020***
Evaluation report	*The evaluation report is expected to be completed 6 weeks after deadline of result submission and sent as PDF file by e-mail.*
Coordinator and contact person of PT	*Matthias Besler-Scharf PhD*

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

6. Index of participant laboratories in alphabetical order

Teilnehmer / Participant	Ort / Town	Land / Country
		AUSTRIA
		FRANCE
		GREAT BRITAIN
		Germany
		Germany
		Germany
		USA
		ITALY
		Germany
		Germany
		SPAIN

[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.]

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20. ASU §64 LFGB: L 00.00-97 / EN 14663:2006 Bestimmung von Vitamin B6 (einschlieslich glucosidisch gebundener Verbindungen) in Lebensmitteln HPLC-Verfahren, Dezember 2006 / Foodstuffs - Determination of vitamin B6 (including its glycosylated forms) by HPLC
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