Proficiency Tests
DLA
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Evaluation Report
proficiency test

DLA 45/2018

Food Supplement I:

Vitamins A, D3, E, K1, β-Carotene, Coenzyme Q10 (Ubiquinone) and alpha-Liponic Acid

in Multi Vitamin Capsule Powder

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

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

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1. Introduction

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

2. Realisation

2.1 Test material

The test material is a mixture of common in commerce food supplements without capsule shells and maltodextrin as bulking agent from European suppliers.
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 (1. food supplement): Calcium carbonate, maltodextrin, magnesium oxide, ascorbic acid, lemon bioflavonoids, green tea extract, choline bitartrate, grape seed extract, lutein, ferrous sulfate, thiamine HCl, pyridoxine HCl, lycopene, vitamin E (Dl-alpha tocopherol acetate), calcium D-pantothenate, silica, riboflavin, nicotinamide, inositol, quercetin, zinc oxide, cyanocobalamin, vitamin D3 (cholecalciferol), coenzyme Q10, black pepper extract, vitamin A (vitamin A acetate), lactobacillus acidophilus, vitamin K (phylloquinone), sodium tetraborate, folic acid, chromium-III-chloride, manganese sulfate, copper sulfate, sodium selenite, D-biotin. Ingredients without capsule shell (2. food supplement): Vitamin C, buling agent rice starch, nicotinamide, vitamin E acetate, calcium D-pantothenate, vitamin B6 HCl, riboflavin, vitamin B1 nitrate, release agent magnesium salts of fatty acids, beta-carotene, biotin, folic acid, vitamin B12. Ingredients (3. supplement): alpha-Liponic acid, bulking agents: lactose, E 1202, microcrystalline cellulose, cellulose powder, E 1420, E 464, anti-caking agents: silicon dioxide, stearic acid and magnesium stearate, carrier: E 553b and E 1521, colors: E 171 and E 172. 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 A	5500 µg
Vitamin D3	340 µg
Vitamin E	740 mg
Vitamin K1	410 µg
beta-Carotene	33 mg
Coenzyme Q10	140 mg
alpha-Liponic Acid	460 mg

2.1.1 Homogeneity

The **mixture homogeneity before bottling** was examined 5-fold by determination of the paramater vitamin D3 by HPLC/UV. The repeatability standard deviations was with 1,0% below the range of repeatability standard deviations of the standardized methods (4,0 % - 12 %, see table 4) [21].
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 the repeatability standard deviation was < 7,0% (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) [21-25].
The repeatability standard deviations of the participants' results are given in the documentation in the statistic data (see 4.1 to 4.7).

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

Parameter	**CV_r**
Vitamin A	7,0 %
Vitamin D3	4,8 %
Vitamin E	6,8 %
Vitamin K1	0,82 %
beta-Carotene	5,5 %
Coenzyme Q10	6,2 %
alpha-Liponic Acid	4,8 %

Furthermore, the homogeneity was graphically characterized for information by the **trend line function of participants' results for chronological bottled single samples** (s. 5.2.1).

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 PT samples was approx. 0,22 (22,4°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 22nd week of 2018. The testing method was optional. The tests should be finished at 13th July 2018 the latest.

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

The two portions contain identical samples of a food supplement with the above mentioned parameters in the matrix of capsule powder (without capsule shells). The analysis method is optional. The results of the vitamins should be given as the sum of the equivalents in the form of the vitamin compound indicated in the result submission file.

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

2.3 Submission of results

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

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

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

All 17 participants submitted results in time.

3. Evaluation

3.1 Consensus value from participants (assigned value)

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

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

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

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

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

3.2 Robust standard deviation

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

3.3 Repeatability standard deviation

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

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

The relative repeatability standard deviation as a percentage of the mean value is indicated as coefficient of variation CV_r in the table of stat-

istical characteristics in the results section in case single results from participants are available.

3.4 Reproducibility standard deviation

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

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

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

3.5 Exclusion of results and outliers

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

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

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

3.6 Target standard deviation (for proficiency assessment)

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

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

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

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

For valuation of all following paramaters in the present PT the target standard deviation according to the general model of Horwitz was applied (see 3.6.1): Vitamin A, Vitamin D3 and Coenzyme Q10.

The target standard deviation of the evaluation by precision experiment (s. 3.6.2) was considered for the following parameters: (ASU §64 / EN-standard methods) [22, 24]: Vitamin E and beta-Carotene.

Additionally for beta-Carotene and Coenzyme Q10 the standard uncertainty was considered by evaluation using z'-scores (see 3.6.8).

Wherein the results of alpha Liponic Acid and Vitamin K were not evaluated by means of z-scores due to the number of < 7.

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} [18-25]

Parameter	**Matrix**	**Mean**	**RSD_r**	**RSD_R**	**σ_{pt}**	**Method / Literature**
Vitamin A	milk powder	653 µg/100 g	2,1%	3,4%	3,06%[1]	HPLC [23]
Vitamin D3	milk powder	14,30 µg/100 g	5,2%	5,5%	4,09%	HPLC [21]
Vitamin D3	milk powder	9,95 µg/100 g	8,2%	13,6%	12,3%[1]	HPLC [21b]
Vitamin D3	infant food, liquid	1,38 µg/100 g	5,9%	12,1%	11,4%	HPLC [21]
Vitamin D3	infant food, powder	10,1 µg/100 g	2,4%	7,1%	6,89%	HPLC [21]
Vitamin E	oat powder	0,279 mg/100g	9,0%	16,8%	15,5%	HPLC [22]
Vitamin E	milk powder	9,89 mg/100 g	4,0%	7,0%	6,40%	HPLC [22]
Vitamin E	milk powder	10,2 mg/100 g	3,0%	12,8%	12,6%[1]	HPLC [22]
Vitamin K1	6 infant food (mean)	77,37 µg/100 g	4,47%	5,91%	4,99%[1]	HPLC [25]
β-Carotene	mixed vegetables	18,05 mg/100g	3,9%	15%	14,7%[1]	HPLC [24]
β-Carotene	pudding powder	1,531 mg/100g	5,6%	9,3%	8,42%	HPLC [24]
β-Carotene	vitamin drink	2,248 mg/100g	2,9%	6,5%	6,17%	HPLC [24]
Coenzyme Q10	Raw Materials and Food Supplements	42-1000 mg/g	2,2 - 5,0 %	-	-	HPLC-UV [20]

[1] *used for evaluation or given for information (s. chapter 4)*

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.

Table 5: Characteristics of the present PT (on dark gray) in comparison to previous PTs since 2014 (SD = standard deviation, CV = coefficient of variation)

Parameter	**Matrix** (Powder)	**robust Mean**	**rob. SD (S*)**	**rel. SD (VK_{S*}) [%]**	**Quotient S^*/σ_{pt}**	**DLA-Report**
Vitamin A	Multivitam-in-Powder	690 µg/100g	180 µg/100g	26,1%	2,2	DLA 29/2014
Vitamin A	Multivitam-in-Capsule Powder	21900 µg/100g	2870 µg/100g	13,1%	1,8	DLA 47/2016
Vitamin A	Multivitam-in-Capsule Powder	7131 µg/100g	1058 µg/100g	14,8%	1,8	DLA 45/2018
Vitamin D3	Multivitam-in-Powder	28,6 µg/100g	11,2 µg/100g	39,2%	2,0	DLA 29/2014
Vitamin D3	Multivitam-in-Capsule Powder	146 µg/100g	10,3 µg/100g	7,05%	0,46	DLA 47/2016
Vitamin D3	Multivitam-in-Capsule Powder	455 µg/100g	74,4 µg/100g	16,4%	1,3	DLA 45/2018
Vitamin E	Multivitam-in-Powder	92,7 mg/100g	16,3 mg/100g	17,6%	1,4	DLA 29/2014
Vitamin E	Multivitam-in-Capsule Powder	988 mg/100g	211 mg/100g	21,4%	1,7	DLA 47/2016
Vitamin E	Multivitam-in-Capsule Powder	760 mg/100g	148 mg/100g	19,5%	1,5	DLA 45/2018
Vitamin K1	Multivitam-in-Powder	233 µg/100g	21,3 µg/100g	9,14%	0,7	DLA 29/2014
Vitamin K1	Multivitam-in-Capsule Powder	933 µg/100g	121 µg/100g	13,0%	1,1	DLA 47/2016
Vitamin K1	Multivitam-in-Capsule Powder	954 µg/100g	632 µg/100g	66,2%	-	DLA 45/2018
β-Carotene	Multivitam-in-Powder	0,509 mg/100g	0,160 mg/100g	31,4%	2,5	DLA 29/2014
β-Carotene	Multivitam-in-Capsule Powder	32,2 mg/100g	9,70 mg/100g	30,1%	2,0	DLA 47/2016
β-Carotene	Multivitam-in-Capsule Powder	27,7 mg/100g	8,45 mg/100g	30,5%	1,6	DLA 45/2018
Coenzyme Q10	Multivitam-in-Tablets	241 mg/100g	15 mg/100g	6,22%	1,3	DLA 49/2016
Coenzyme Q10	Multivitam-in-Capsule Powder	103 mg/100g	14,0 mg/100g	13,6%	1,7	DLA 45/2018

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].

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_R)

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

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

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

3.10 Quotient S*/σ_{pt}

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

3.11 Standard uncertainty of the assigned value

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

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

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

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

4. Results

Comments to the distribution of the results:

The kernel density plots showed for all parameters nearly a symmetrical distribution of results (figures see documentation 5.3). Partly slight shoulders and separated smaller peaks can be seen, which are due to individual results and outliers. On the basis of the kernel density plots single results were excluded before statistic evaluation.
In the case of vitamin E the kernel density estimation showed a second smaller peak. However, the information provided by the participants on the methods gave no obvious indications of such an array of results. When using the robust standard deviation as an estimator h, the distribution is converted into a single peak distribution, so that a statistical evaluation has been carried out.

Comments to the statistic data:

For vitamin K and alpha-liponic acid there were < 7 results, therefore no statistical evaluation could be done.

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 beta-carotene and coenzyme Q10 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}' were then below 2,0 (see table 5).

For all other parameters the distribution of results showed a normal variability. The quotients S^*/σ_{pt} were in the range of 1,3 to 1,8 (see table 5).

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

The comparability of results is given.

80% to 92% of results were in the respective target range.

The robust means of the participant results were for all evaluated parameters in the range of 74% to 134% of the vitamin contents according to the manufacturer specifications (see table 2).

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

In the first table the characteristics are listed:

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

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

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

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

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

4.1 Vitamin A (as Retinol in µg/100g)

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	11
Number of outliers	-
Mean	7380
Median	7250
Robust Mean (X_{pt})	**7130**
Robust standard deviation (S*)	**1060**
Number with 2 replicates	10
Repeatability SD (S_r)	487
Repeatability (CV_r)	6,98%
Reproducibility SD (S_R)	882
Reproducibility (CV_R)	12,6%
Target range:	
Target standard deviation σ_{pt}	**600**
Target standard deviation (for Information)	218
lower limit of target range	**5930**
upper limit of target range	**8330**
Quotient S/σ_{pt}*	*1,8*
Standard uncertainty $U_{(Xpt)}$	*399*
Results in the target range	*9*
Percent in the target range	*82%*

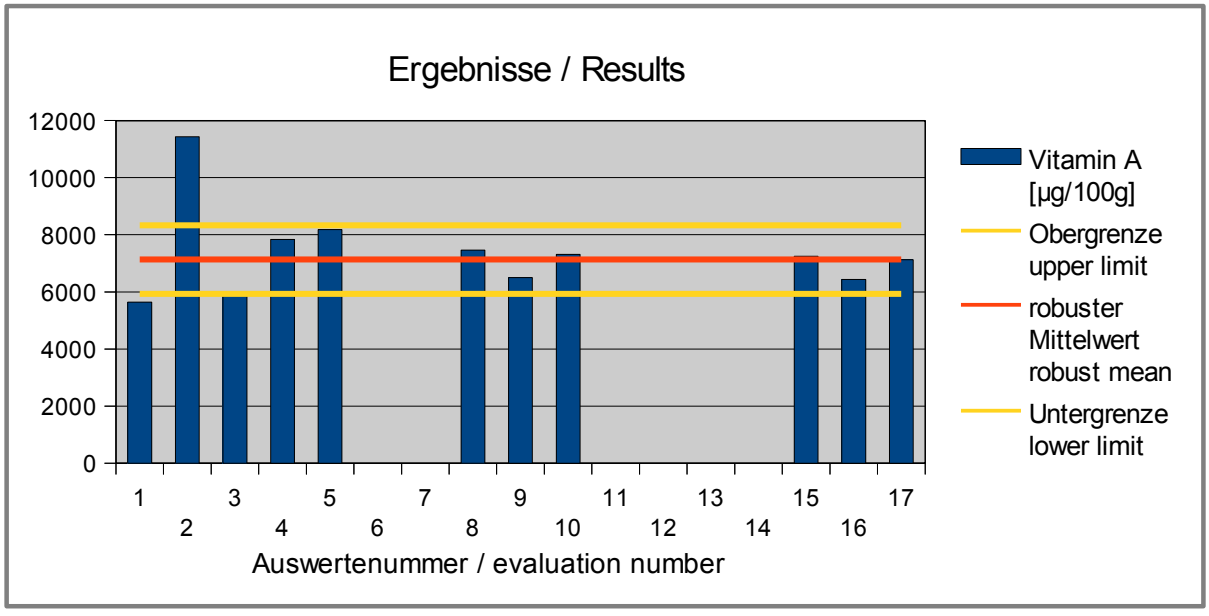


Abb. / Fig. 1: Ergebnisse Vitamin A/ Results vitamin A

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Vitamin A [µg/100g]	Abweichung [µg/100g] Deviation [µg/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	5640	-1490	-2,5	-6,8	
2	11400	4300	7,2	20	
3	5970	-1160	-1,9	-5,3	
4	7830	699	1,2	3,2	
5	8180	1050	1,7	4,8	
6					
7					
8	7470	334	0,56	1,5	
9	6500	-628	-1,0	-2,9	
10	7310	184	0,31	0,84	
11					
12					
13					
14					
15	7250	119	0,20	0,55	
16	6440	-696	-1,2	-3,2	
17	7130	-3	-0,01	-0,01	

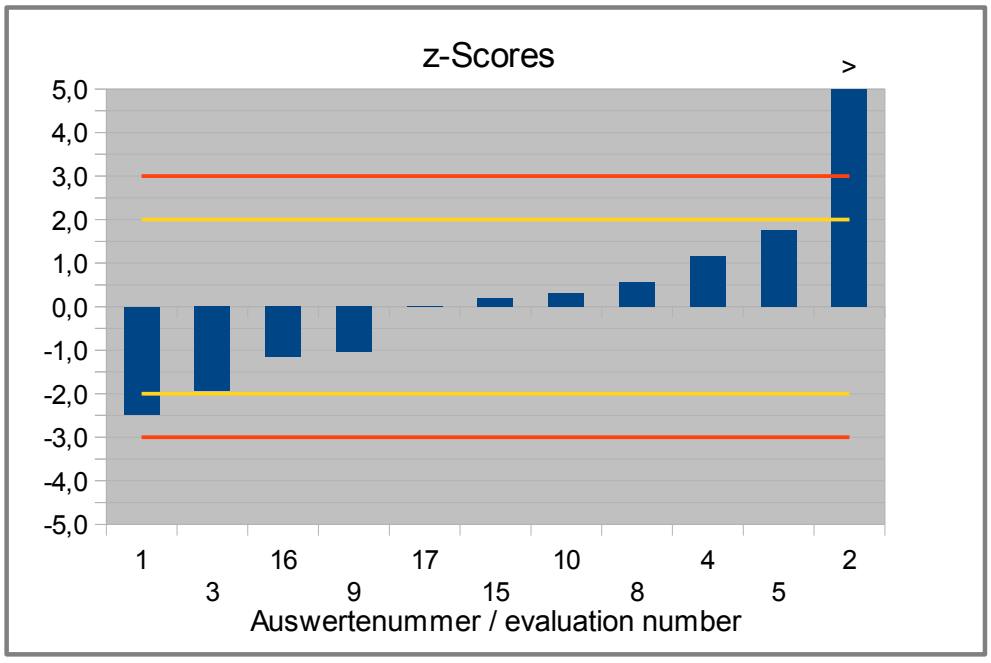


Abb. / Fig. 2: z-Scores Vitamin A / vitamin A

4.2 Vitamin D3 (as Cholecalciferol in µg/100g)

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	12
Number of outliers	-
Mean	448
Median	467
Robust Mean (X_{pt})	**455**
Robust standard deviation (S*)	**74,4**
Number with 2 replicates	11
Repeatability SD (S_r)	22,7
Repeatability (CV_r)	4,81%
Reproducibility SD (S_R)	77
Reproducibility (CV_R)	16,2%
Target range:	
Target standard deviation σ_{pt}	**58,0**
Target standard deviation (for Information)	56,0
lower limit of target range	**339**
upper limit of target range	**571**
Quotient S/σ_{pt}*	*1,3*
Standard uncertainty $U_{(Xpt)}$	*26,9*
Results in the target range	*10*
Percent in the target range	*83%*

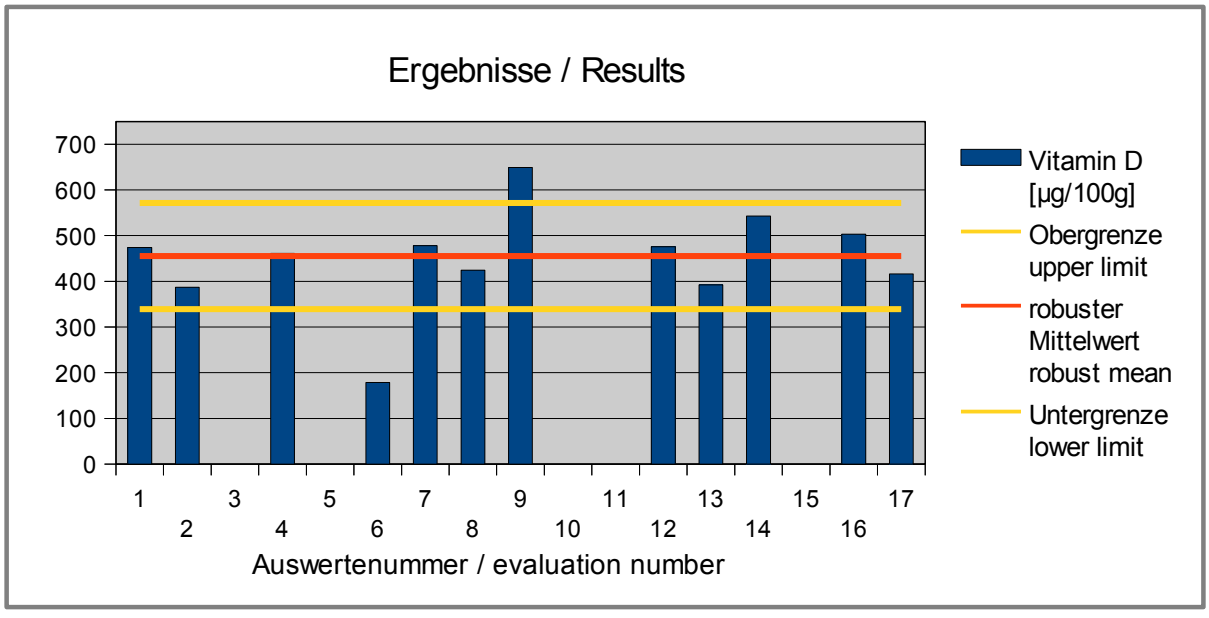


Abb. / Fig. 3: Ergebnisse Vitamin D3/ Results vitamin D3

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Vitamin D [µg/100g]	Abweichung [µg/100g] Deviation [µg/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	474	18,3	0,32	0,33	
2	387	-68,3	-1,2	-1,2	
3					
4	461	5,7	0,10	0,10	
5					
6	178	-277	-4,8	-4,9	
7	478	22,7	0,39	0,41	
8	424	-31,3	-0,54	-0,56	
9	649	194	3,3	3,5	
10					
11					
12	475	20,1	0,35	0,36	
13	392	-63,3	-1,1	-1,1	
14	543	87,7	1,5	1,6	
15					
16	503	47,7	0,82	0,85	
17	416	-39,3	-0,7	-0,7	

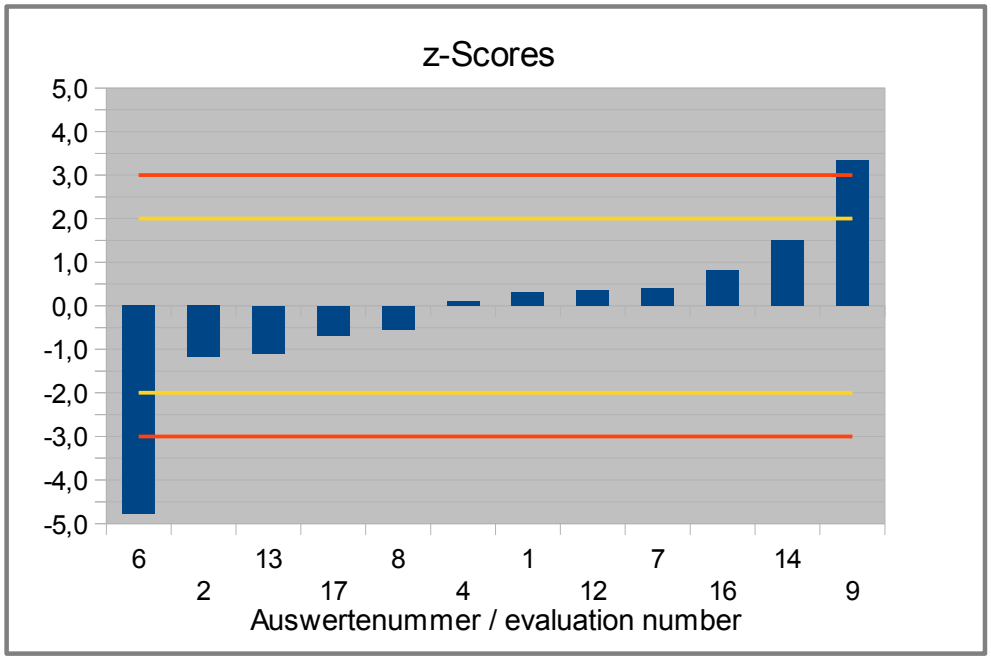


Abb. / Fig. 4: z-Scores Vitamin D3 / vitamin D3

4.3 Vitamin E (as D-alpha-Tocopherol in mg/100g)

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results°	13
Number of outliers	2
Mean	760
Median	791
Robust Mean (X_{pt})	**760**
Robust standard deviation (S*)	**148**
Number with 2 replicates	11
Repeatability SD (S_r)	51,4
Repeatability (CV_r)	6,76%
Reproducibility SD (S_R)	132
Reproducibility (CV_R)	17,4%
Target range:	
Target standard deviation σ_{pt}	**96,0**
Target standard deviation (for Information)	31,7
lower limit of target range	**568**
upper limit of target range	**952**
Quotient S/σ_{pt}*	*1,5*
Standard uncertainty $U_{(Xpt)}$	*51,4*
Results in the target range	*12*
Percent in the target range	*92%*

° number of results without outliers (no. 6 and 11)

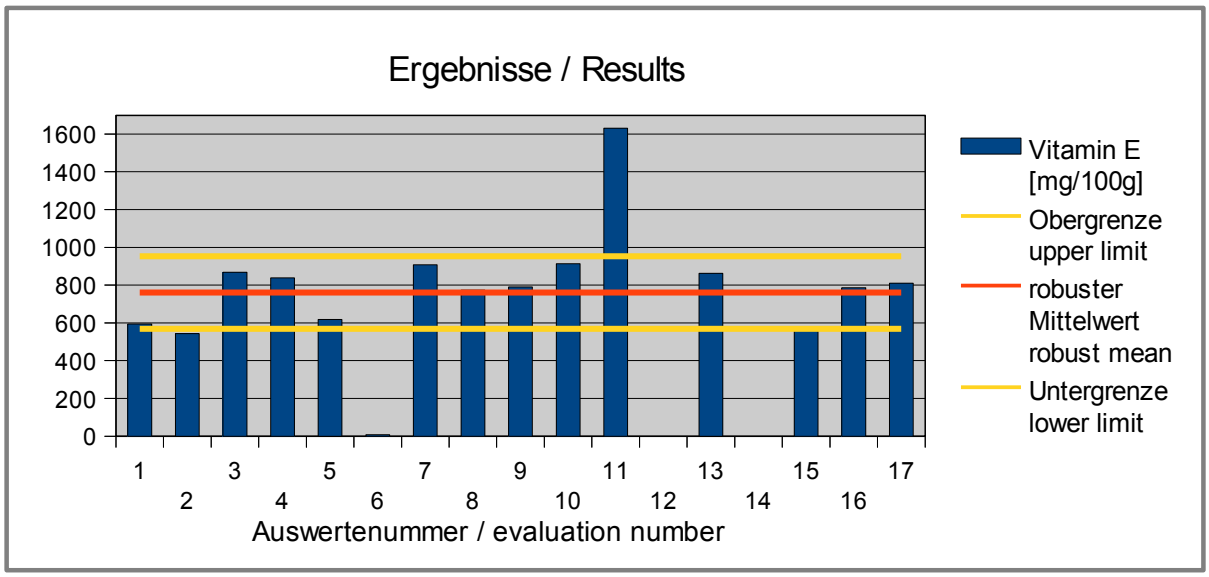


Abb. / Fig. 5: Ergebnisse Vitamin E / Results vitamin E

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Vitamin E [mg/100g]	Abweichung [mg/100g] Deviation [mg/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	594	-166	-1,7	-5,2	
2	544	-216	-2,3	-6,8	
3	868	108	1,1	3,4	
4	838	77,6	0,81	2,4	
5	618	-142	-1,5	-4,5	
6	7,7				Ergebnis ausgeschlossen / Result excluded
7	909	148	1,5	4,7	
8	773	12,6	0,13	0,40	
9	791	30,6	0,32	1,0	
10	913	152	1,6	4,8	
11	1630				Ergebnis ausgeschlossen / Result excluded
12					
13	862	102	1,1	3,2	
14					
15	579	-181	-1,9	-5,7	
16	786	25,6	0,27	0,81	
17	811	50,1	0,52	1,6	

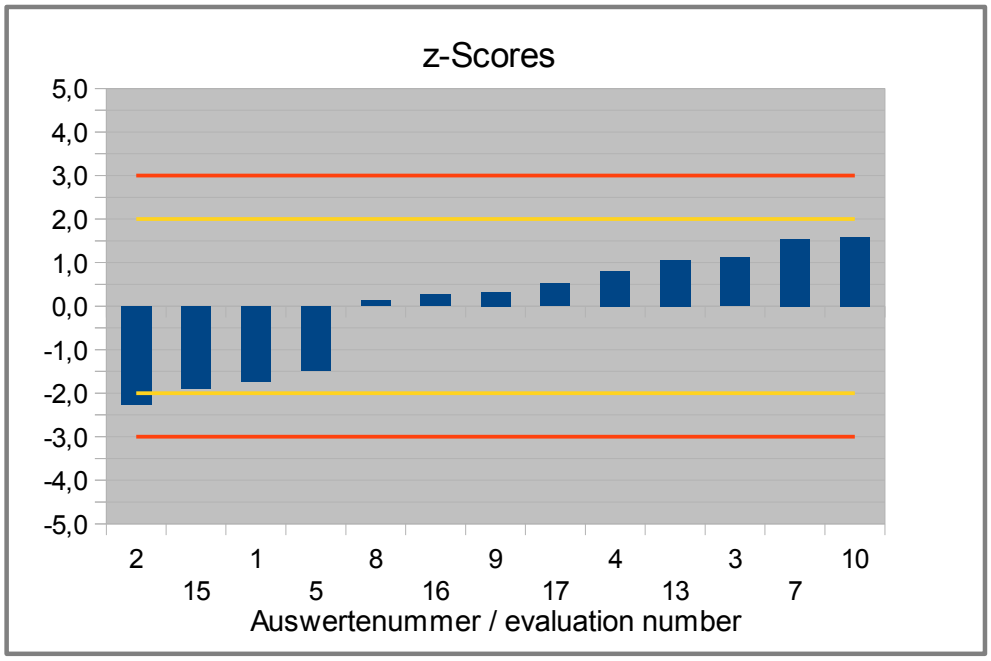


Abb. / Fig. 6: z-Scores Vitamin E / vitamin E

4.4 Vitamin K1 (as Phyllochinone in µg/100g)

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	5
Number of outliers	-
Mean	954
Median	858
Robust Mean	**954**
Robust standard deviation (S*)	**632**
Number with 2 replicates	5
Repeatability SD (S_r)	7,82
Repeatability (CV_r)	0,820%
Reproducibility SD (S_R)	557
Reproducibility (CV_R)	58,4%
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	

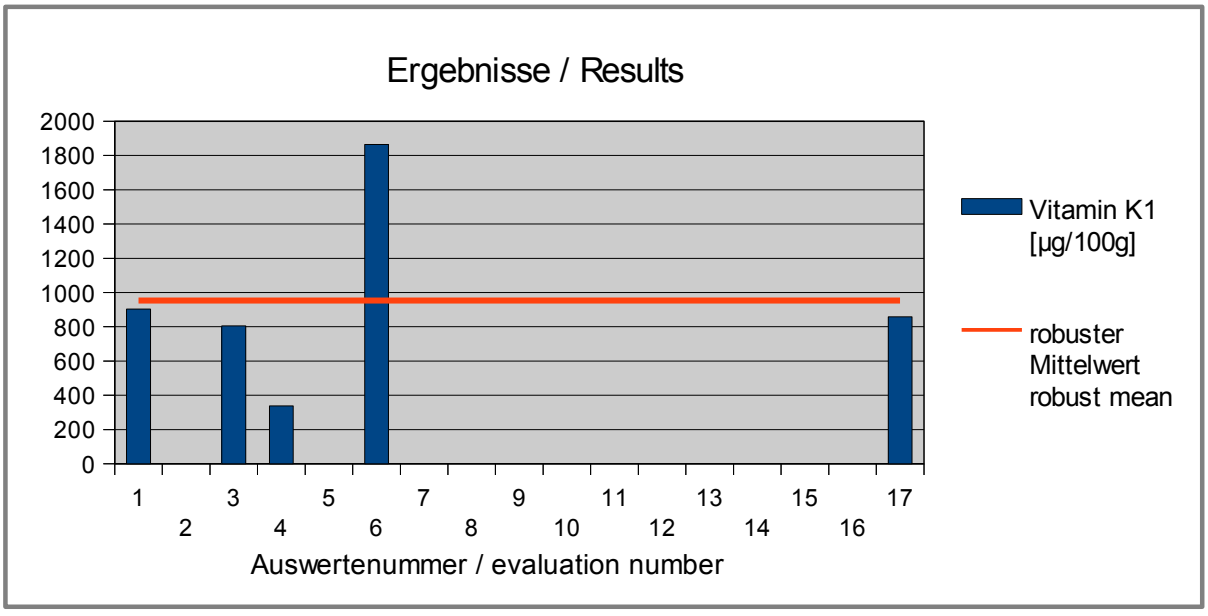


Abb. / Fig. 7: Ergebnisse Vitamin K1 / Results vitamin K1

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Vitamin K1 [µg/100g]	Abweichung [µg/100g] Deviation [µg/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	903	-50,9			
2					
3	805	-149			
4	339	-615			
5					
6	1870	911			
7					
8					
9					
10					
11					
12					
13					
14					
15					
16					
17	858	-96,3			

4.5 Beta-Carotene (in mg/100g)

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results°	10
Number of outliers	1
Mean	27,6
Median	28,4
Robust Mean (X_{pt})	**27,6**
Robust standard deviation (S*)	**8,45**
Number with 2 replicates	9
Repeatability SD (S_r)	1,58
Repeatability (CV_r)	5,47%
Reproducibility SD (S_R)	6,53
Reproducibility (CV_R)	22,5%
Target range:	
Target standard deviation σ_{pt}'	**5,27**
Target standard deviation (for Information)	1,90
lower limit of target range	**17,1**
upper limit of target range	**38,2**
Quotient S/σ_{pt}'*	*1,6*
Standard uncertainty $U_{(Xpt)}$	*3,34*
Results in the target range	*8*
Percent in the target range	*80%*

° number of results without outliers (no. 2)

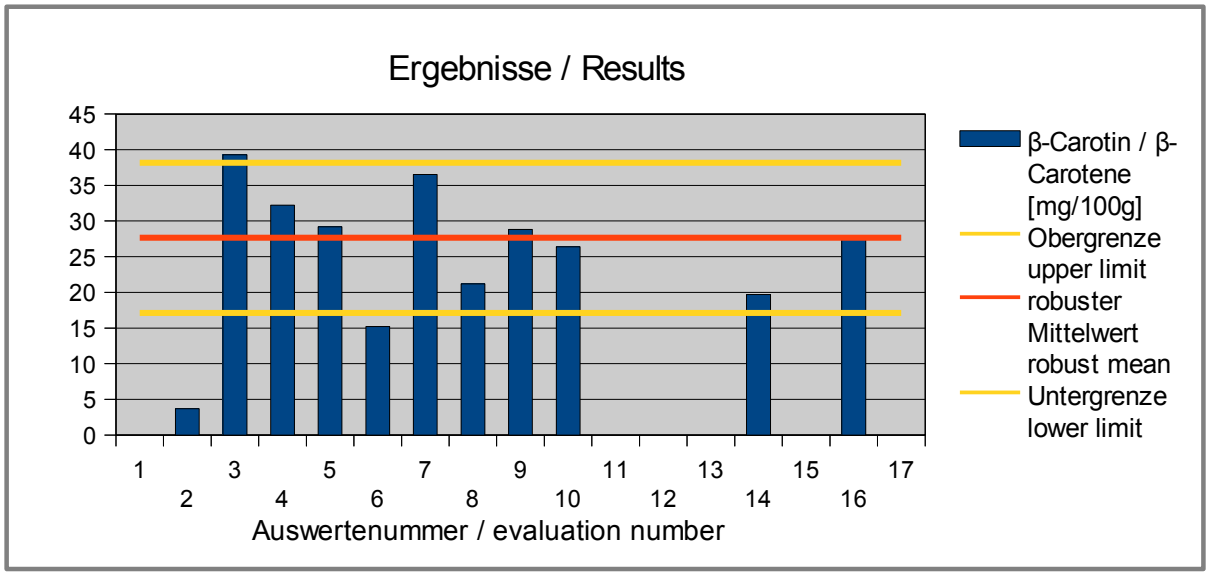


Abb. / Fig. 8: Ergebnisse β-Carotin / Results β-Carotene

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	β-Carotin / β-Carotene [mg/100g]	Abweichung [mg/100g] Deviation [mg/100g]	z'-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1					
2	3,70				Ergebnis ausgeschlossen / Result excluded
3	39,3	11,7	2,2	6,1	
4	32,2	4,56	0,87	2,4	
5	29,2	1,56	0,30	0,82	
6	15,2	-12,4	-2,4	-6,6	
7	36,5	8,86	1,7	4,7	
8	21,2	-6,44	-1,2	-3,4	
9	28,8	1,16	0,22	0,61	
10	26,4	-1,26	-0,24	-0,66	
11					
12					
13					
14	19,7	-7,94	-1,5	-4,2	
15					
16	27,9	0,26	0,05	0,14	
17					

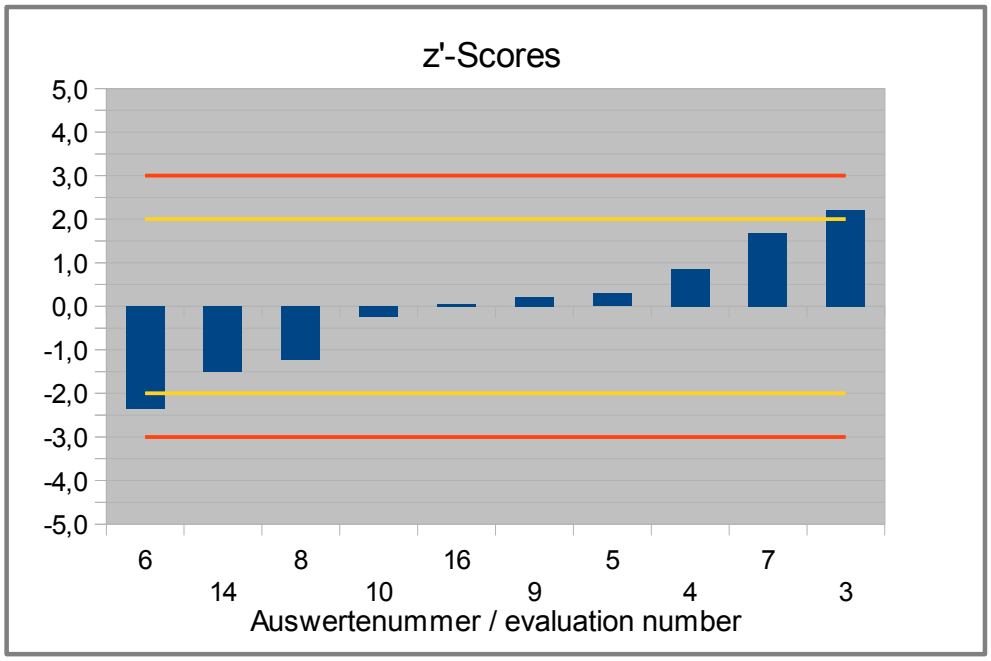


Abb. / Fig. 9: z'-Scores β-Carotin / β-Carotene

4.6 Coenzyme Q10 (in mg/100g)

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results°	9
Number of outliers	1
Mean	106
Median	99
Robust Mean (X_{pt})	**103**
Robust standard deviation (S*)	**14,0**
Number with 2 replicates	10
Repeatability SD (S_r)	7,09
Repeatability (CV_r)	6,17%
Reproducibility SD (S_R)	34,1
Reproducibility (CV_R)	29,7%
Target range:	
Target standard deviation σ_{pt}'	**8,23**
lower limit of target range	**86,7**
upper limit of target range	**120**
Quotient S^*/σ_{pt}'	*1,7*
Standard uncertainty $U_{(Xpt)}$	*5,83*
Results in the target range	*8*
Percent in the target range	*89%*

° number of results without outliers (no. 11)

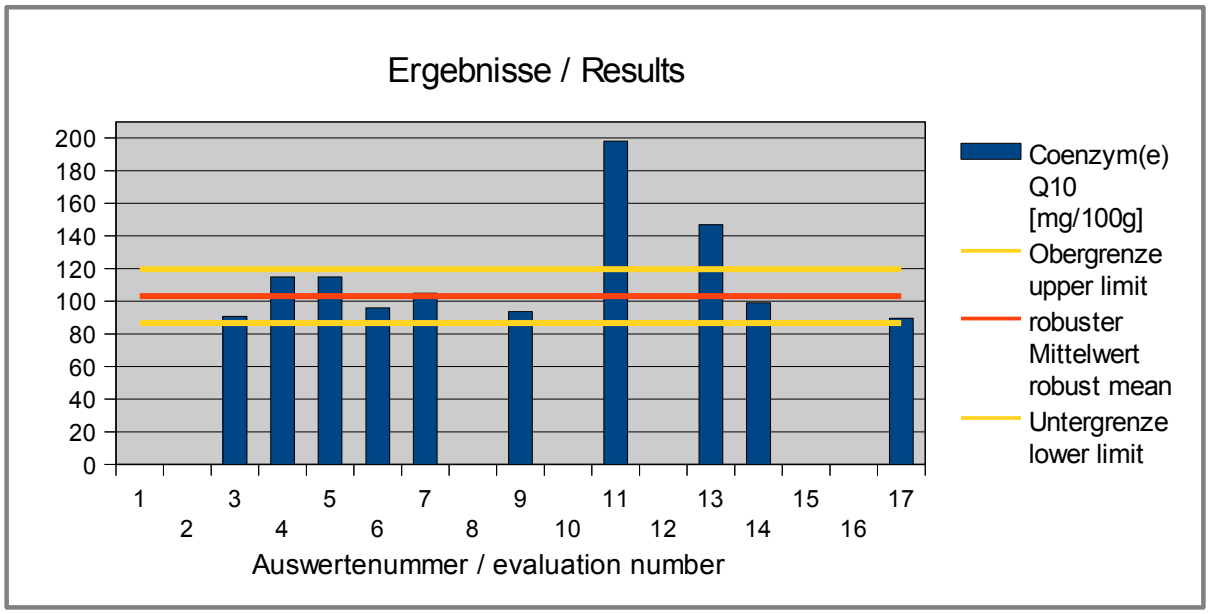


Abb. / Fig. 10: Ergebnisse Coenzym Q10 / Results coenzyme Q10

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Coenzym(e) Q10 [mg/100g]	Abweichung [mg/100g] Deviation [mg/100g]	z'-Score (σ_{pt})	Hinweis Remark
1				
2				
3	90,8	-12,3	-1,5	
4	115	11,9	1,4	
5	115	11,9	1,4	
6	95,9	-7,2	-0,9	
7	105	1,9	0,23	
8				
9	93,7	-9,4	-1,1	
10				
11	198			Ergebnis ausgeschlossen / Result excluded
12				
13	147	43,9	5,3	
14	99,0	-4,1	-0,50	
15				
16				
17	89,6	-13,5	-1,6	

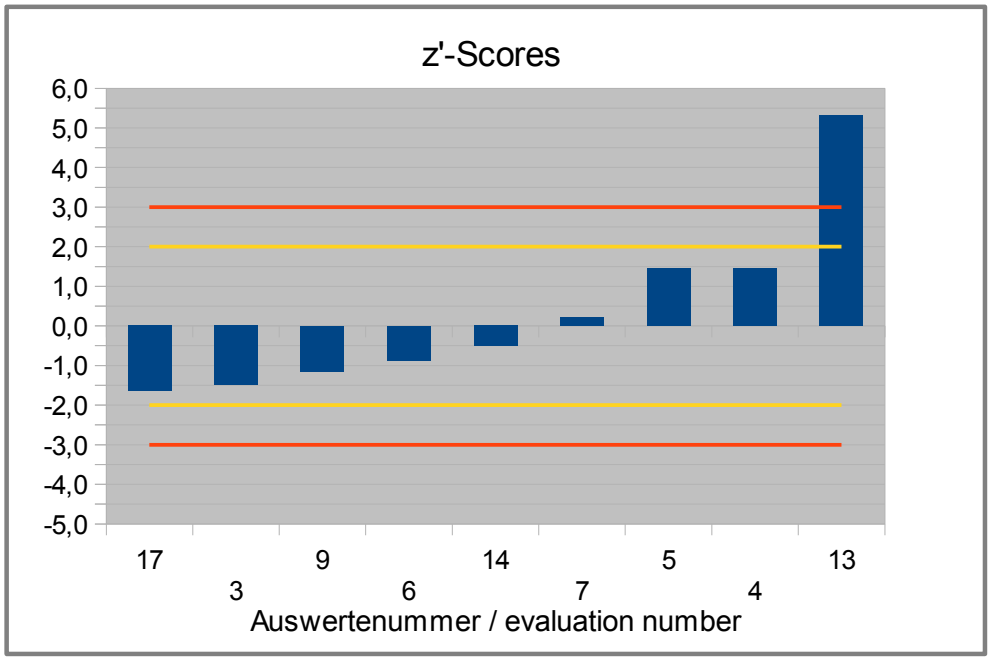


Abb. / Fig. 11: z'-Scores Coenzym Q10 / coenzyme Q10

4.7 Alpha-Liponic Acid (in mg/100g)

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	3
Number of outliers	-
Mean	433
Median	416
Robust Mean	**433**
Robust standard deviation (S*)	**45,5**
Number with 2 replicates	3
Repeatability SD (S_r)	20,8
Repeatability (CV_r)	4,81%
Reproducibility SD (S_R)	42,7
Reproducibility (CV_R)	9,86%
Target range:	
Target standard deviation σ_{pt}	
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	

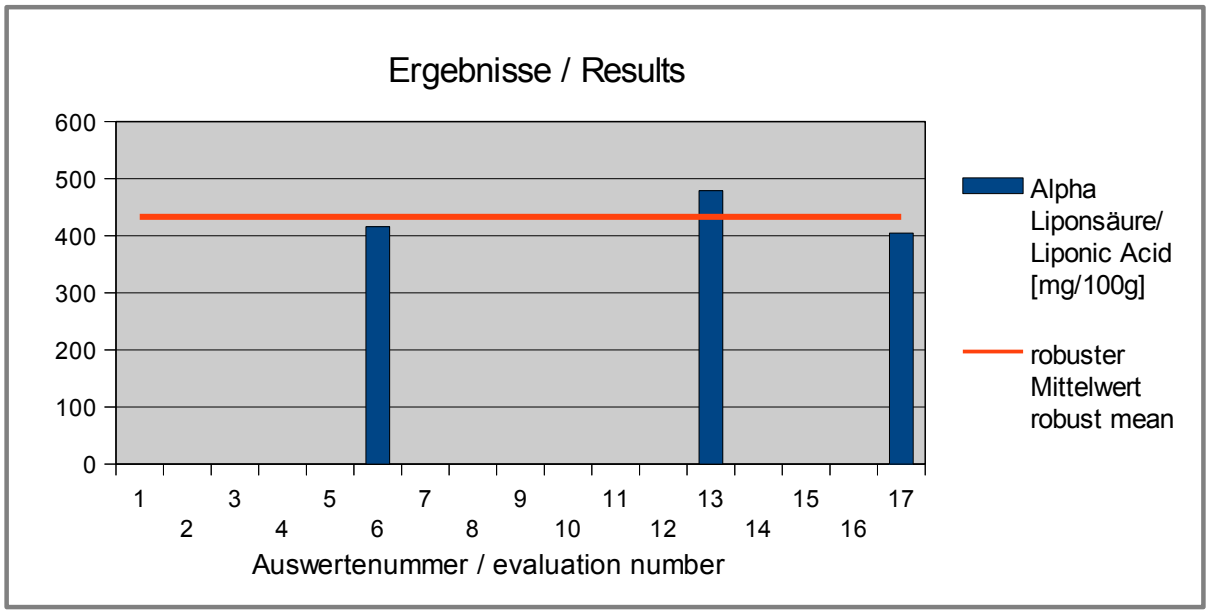


Abb. / Fig. 12: Ergebnisse α-Liponsäure / Results α-liponic acid

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Alpha-Liponsäure/ Liponic Acid [mg/100g]	Abweichung [mg/100g] Deviation [mg/100g]	z-Score (σ_{pt})	Hinweis Remark
1				
2				
3				
4				
5				
6	416	-17		
7				
8				
9				
10				
11				
12				
13	479	46		
14				
15				
16				
17	405	-29		

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 A (calculated as Retinol, without Provitamins)	1	µg/100g	33	35		5.644,86	5681,08	5608,64		no	
	2	µg/100g	11	57	19. Jun	11431	11288	11573	0,02	no	
	3	µg/100g	13	45	20./06.	5970	5090	6850	10	yes	75
	4	µg/100g	32	36	22.06.	7830	8160	7500	950	no	101,4
	5	µg/100g	18	50	03. + 04.July	8180	8315	8045		no	108
	6	µg/100g	16	52	-	-	-	-	-	-	-
	7	µg/100g	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
	8	µg/100g	4	64	04./05.06.	7465	7398	7532	822	no	
	9	µg/100g			18.06.2018	6503	6595	6410	10	no	95,6
	10	µg/100g			29/30.5.18	7314,5	7285,1	7343,8	35	no	100,10%
	11	µg/100g	12	56							
	12	µg/100g	6	62							
	13	µg/100g	17	51							
	14	µg/100g	21	47							
	15	µg/100g	29	39	20. Jun	7250	7600	6900	3000	no	
	16	µg/100g	28	40	26. Jun	6435	6750	6120		no	
	17	µg/100g	31	37	31.05.2018	7127,5	7341	6914			

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 D3 (calculated as Cholecalciferol)	1	µg/100g	33	35		473,59	469,91	477,28		no	
	2	µg/100g	11	57	19. Jun	387	361	413	0,02	no	
	3	µg/100g	13	45	19./06.	not tested	not tested	not tested	not tested	not tested	not tested
	4	µg/100g	32	36	28.06.	461	449	473	20	no	99,6
	5	µg/100g	18	50							
	6	µg/100g	16	52	22.06.2018	178,3	178,1	178,4	-	no	-
	7	µg/100g	3	65	21. Jun	478	489	467	N/A	No	N/A
	8	µg/100g	4	64	11./12.06.	424	405	442		no	
	9	µg/100g			08.06.2018	649	628	669	0,1	no	
	10	µg/100g									
	11	µg/100g	12	56							
	12	µg/100g	6	62	11.06.	475,4	465,5	485,2		ja	84,1
	13	µg/100g	17	51	20.06.18	392	386	398		no	
	14	µg/100g	21	47	19. Jun	543	543	543	4	no	
	15	µg/100g	29	39	20. Jun	<BG			400	no	
	16	µg/100g	28	40	26. Jun	503	476	529		no	
	17	µg/100g	31	37	04. Jun	416	433	399			

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 E (calculated as D-alpha Tocopherol)	1	mg/100g	33	35		594,03	599,49	588,57		no	
	2	mg/100g	11	57	21. Jun	544	535	552	0,09	no	
	3	mg/100g	13	45	15./06.	868	972	764	0,01	yes	81,1
	4	mg/100g	32	36	21.06.	838	821	855	119	no	102,3
	5	mg/100g	18	50	03. +04. Juli	618	628	608		no	66
	6	mg/100g	16	52	02.07.2018	7,72	7,61	7,84	1 mg/L	no	-
	7	mg/100g	3	65	29. Jun	908,5	962	855	N/A	No	N/A
	8	mg/100g	4	64	04./05.06.	773	762	784	49	no	
	9	mg/100g			18.06.2018	791	800	782	1	no	101
	10	mg/100g			29/30.5.18	912,8	907,14	918,38	1,43	no	98,73
	11	mg/100g	12	56	24. Jun	1631	1649	1613	0,5	yes	97
	12	mg/100g	6	62							
	13	mg/100g	17	51	29.06.18	862	867	858		no	
	14	mg/100g	21	47							
	15	mg/100g	29	39	20. Jun	579	570	587	63	no	
	16	mg/100g	28	40	26. Jun	786	797	775		no	
	17	mg/100g	31	37	06. Jun	810,5	805	816			

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 K1 (calculated as Phylloquinone)	1	µg/100g	33	35		902,88	902,9	902,85		no	
	2	µg/100g	11	57							
	3	µg/100g	13	45	21./06.	805	813	798	200	no	
	4	µg/100g	32	36	07.06.	338,5	347	330	86	no	78,5
	5	µg/100g	18	50							
	6	µg/100g	16	52	22.06.2018	1865	1868	1861	-	no	-
	7	µg/100g	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
	8	µg/100g	4	64							
	9	µg/100g									
	10	µg/100g									
	11	µg/100g	12	56							
	12	µg/100g	6	62							
	13	µg/100g	17	51							
	14	µg/100g	21	47							
	15	µg/100g	29	39	20. Jun	<BG			8000	no	
	16	µg/100g	28	40							
	17	µg/100g	31	37	04. Jun	857,5	861	854			

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 [%]
β-Carotene (calculated as β-Carotene, without other Provitamins)	1	mg/100g	33	35							
	2	mg/100g	11	57	19. Jun	3,7	3,7	3,7	0,02	no	
	3	mg/100g	13	45	15./06.	39,3	37,6	41,1	0,2	yes	90,9
	4	mg/100g	32	36	20.06.	32,2	31,9	32,5	1,83	no	106
	5	mg/100g	18	50	19. Juni +06. Juli	29,2	28,7	29,6		no	93
	6	mg/100g	16	52	03.07.2018	15,2	11,2	19,2	-	no	-
	7	mg/100g	3	65	20. Jun	36,5	35	38	N/A	No	N/A
	8	mg/100g	4	64	30.05./ 06.06	21,2	22	20,4	3	no	
	9	mg/100g			30.05.2018	28,8	27,9	29,6		no	98,5
	10	mg/100g			22./25.6.18	26,38	26,8	25,3	0,15	no	81
	11	mg/100g	12	56							
	12	mg/100g	6	62							
	13	mg/100g	17	51							
	14	mg/100g	21	47	27. Jun	19,7	17,8	21,6	1	no	
	15	mg/100g	29	39							
	16	mg/100g	28	40	26. Jun	27,9	28,3	27,5		no	
	17	mg/100g	31	37							

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 [%]
Coenzyme Q10 (Ubiquinone)	1	mg/100g	33	35							
	2	mg/100g	11	57							
	3	mg/100g	13	45	21./06.	90,8	78,6	103	20	yes	78,9
	4	mg/100g	32	36	05.06.	115	112	118	0,48	no	87,5
	5	mg/100g	18	50	28. + 31. May	115	116	115		no	96
	6	mg/100g	16	52	02.07.2018	95,9	99,7	92,1	-	no	-
	7	mg/100g	3	65	02. Jul	105	102	108	N/A	No	N/A
	8	mg/100g	4	64							
	9	mg/100g			21.06.2018	93,7	99,5	87,9		no	97
	10	mg/100g									
	11	mg/100g	12	56	24. Jun	197,8	197,4	198,2	0,5	yes	98
	12	mg/100g	6	62							
	13	mg/100g	17	51	04.06.18	147	144	149		no	
	14	mg/100g	21	47	29.05.2018	99	101	96,8	25	no	100,4
	15	mg/100g	29	39							
	16	mg/100g	28	40							
	17	mg/100g	31	37	07. Jun	89,6	84,6	94,6			

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 [%]
Alpha-Liponic Acid	1	mg/100g	33	35							
	2	mg/100g	11	57							
	3	mg/100g	13	45	not tested	not tested	not tested	not tested	not tested	not tested	not tested
	4	mg/100g	32	36	---	---	---	---	---	---	---
	5	mg/100g	18	50							
	6	mg/100g	16	52	28.06.2018	416,1	438,4	393,8	-	no	-
	7	mg/100g	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
	8	mg/100g	4	64							
	9	mg/100g									
	10	mg/100g									
	11	mg/100g	12	56							
	12	mg/100g	6	62							
	13	mg/100g	17	51	12.06.18	479	471	487		no	
	14	mg/100g	21	47							
	15	mg/100g	29	39							
	16	mg/100g	28	40							
	17	mg/100g	31	37	07. Jun	404,5	414	395			

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 A (calculated as Retinol, without Provitamins)	1			HPLC			yes	
	2	L 00.00-63/1		HPLC-DAD			yes	
	3	MSZ EN 12823:1:2014	saponification	LUNA CN	ESTD	no	yes	
	4	2.020/002-03	---	HPLC	Retinol palmitate	yes	yes	---
	5	Vitamins A and E in food, HPLC-FLD, 03-32-MAA-M-VITAE, 2015-08	none	none	PT Material	yes	yes	none
	6	-	-	-	-	-	-	-
	7	N/A	N/A	N/A	N/A	N/A	N/A	N/A
	8	ASU L 49.00-3, HPLC-DAD					yes	sample material apparently inhomogeneous
	9	Vitamin A and E; determination in food and food supplements by HPLC according to ASU §64 methods with modified saponification temperature				yes	yes	
	10	L49.00-3,modified	Saponification, Liquid-Liquid Extraction	HPLC	Standards, Content determination	no	yes	
	11							
	12							
	13							
	14							
	15	in-house method		HPLC-DAD	external, Sigma Aldrich		yes	
	16	in-house, HPLC-UV					no	
	17							

Analyte	Participant	Method description	Sample preparation	Measuring method	Calibration / Reference material	Recovery with same matrix	Method accredited	Further remarks
Vitamin D3 (calculated as Cholecalciferol)	1			HPLC			yes	
	2	L 00.00-61		HPLC-DAD			yes	
	3	not tested	not tested	not tested	not tested	not tested	not tested	not tested
	4	2.020/004-03	---	HPLC	Cholecalciferol, Ergocalciferol	yes	yes	---
	5							
	6	in-house method	liquid extraction	HPLC-DAD	-	-	nein	-
	7	MQLTM-0508 by HPLC	N/A	N/A	N/A	N/A	YES	N/A
	8	LAV 36.3002-02, LC-MS/MS					yes	sample material apparently inhomogeneous
	9	Determination of Vitamin D in edible fats and oils and food supplements						Distribution of results is high for the kind of sample material (Inhomogeneity of material?)
	10							
	11							
	12	ASU L 00.00-61: 2010-01			yes	yes	yes	
	13	according to ASU § 64 LFGB L 49.00-1: 1991-06 by HPLC/UV				yes		
	14	ČSN EN 12821	preparative analyses, internal standard	HPLC/DAD (DETECTION 265)	Sigma		yes	
	15	in-house method		HPLC-DAD	external, Sigma Aldrich		yes	
	16	in-house, HPLC-UV					yes	
	17							

Analyte	Participant	Method description	Sample preparation	Measuring method	Calibration / Reference material	Recovery with same matrix	Method accredited	Further remarks
Vitamin E (calculated as D-alpha Tocopherol)	1			HPLC			yes	
	2	L 00.00-62		HPLC-FLD			yes	
	3	MSZ EN 12822:2014	saponification	LUNA CN	ESTD	no	yes	
	4	2.020/003-03	---	HPLC	alpha-Tocopherol	yes	yes	---
	5	Vitamins A and E in food, HPLC-FLD, 03-32-MAA-M-VITAE, 2015-08	none	none	PT Material	yes	yes	Vitamin E, calculated from alpha- and gamma-Tocopherol
	6	in-house method	liquid extraction	HPLC-DAD	-	-	yes	-
	7	MQLTM-0100 by HPLC	N/A	N/A	N/A	N/A	YES	N/A
	8	ASU L 49.00-5, HPLC-DAD					yes	sample material apparently inhomogeneous
	9	Vitamins A and E; determination in food and food supplements by HPLC according to ASU §64 methods with modified saponification temperature				yes	yes	
	10	L00.00-62, modified	Saponification, Liquid-Liquid Extraction	HPLC	Standards, Content determination	no	yes	
	11	DGF standard method, F-II 4a (00)	Extraction with iso-Octane	HPLC-FLD		no	yes	
	12							
	13	according to ASU § 64 LFGB L 00.00-62: 2015-06						
	14							
	15	in-house method		HPLC-DAD	external, Sigma Aldrich		yes	
	16	in-house, HPLC-fluorescence					yes	
	17							

Analyte	Participant	Method description	Sample preparation	Measuring method	Calibration / Reference material	Recovery with same matrix	Method accredited	Further remarks
Vitamin K1 (calculated as Phylloquinone)	1			HPLC			yes	
	2							
	3	SM-SZ-223:2018	extraction	LUNA CN	ESTD	no	no	
	4	2.019/019-01	---	HPLC	Phyllochinone	yes	yes	---
	5							
	6	in-house method	liquid extraction	HPLC-DAD	-	-	no	-
	7	N/A	N/A	N/A	N/A	N/A	N/A	N/A
	8							
	9							
	10							
	11							
	12							
	13							
	14							
	15	in-house method		HPLC-DAD	external, Sigma Aldrich		yes	
	16							
	17							

Analyte	Participant	Method description	Sample preparation	Measuring method	Calibration / Reference material	Recovery with same matrix	Method accredited	Further remarks
β-Carotene (calculated as β-Carotene, without other Provitamins)	1							
	2	L 00.00-63/2		HPLC-DAD			yes	
	3	MSZ EN 12823-2:2000	saponification	LUNA CN	ESTD	no	yes	
	4	2.019/012-03	---	HPLC	beta-Carotene	yes	no	---
	5	Tota carotene in food directly, photometry, 03-32-MAA-M-CaroD, 2015-08	none	none	PT Material	yes	yes	none
	6	in-house method	liquid extraction	HPLC-DAD	-	-	no	-
	7	MQLTM-0101A by HPLC	N/A	N/A	N/A	N/A	YES	N/A
	8	LAV 21.0055-02, HPLC-DAD					yes	sample material apparently inhomogeneous
	9	Photometric determination of total carotinoides and beta-carotene in food and food supplements				yes	yes	
	10	L00.00-63/2, modified	Saponification, Liquid-Liquid Extraction	HPLC	Standards, Content determination	no	yes	
	11							
	12							
	13							
	14	ČSN EN 12823-2		HPLC/DAD (DETECTION 450 NM)	Sigma		yes	
	15							
	16	in-house, HPLC-UV					yes	
	17							

Analyte	Participant	Method description	Sample preparation	Measuring method	Calibration / Reference material	Recovery with same matrix	Method accredited	Further remarks
Coenzyme Q10 (Ubiquinone)	1							
	2							
	3	SM-SZ-224:2018	extraction	Kinetex C18	ESTD	no	no	
	4	2.019/017-02	---	HPLC	Coenzym Q10	yes	no	---
	5	Ubichinone Q10 in food supplements, HPLC-DAD, 03-32-MAA-M-Q10, 2015-08	none	none	PT Material	yes	yes	none
	6	in-house method	liquid extraction	HPLC-DAD	-	-	no	-
	7	MQLTM-0162 by HPLC	N/A	N/A	N/A	N/A	YES	N/A
	8							
	9	Determination of Coenzyme Q10 in Foods by HPLC				yes	yes	
	10							
	11	HPLC-DAD, in-house method	Extraction mit iso-Octane	HPLC-DAD		no	yes	
	12							
	13	after extraction by HPLC/UV						
	14	in-house method		HPLC/DAD (DETECTION 276 NM)	Sigma	yes	yes	
	15							
	16							
	17							

Analyte	Participant	Method description	Sample preparation	Measuring method	Calibration / Reference material	Recovery with same matrix	Method accredited	Further remarks
Alpha-Liponic Acid	1							
	2							
	3	not tested	not tested	not tested	not tested	not tested	not tested	not tested
	4	---	---	---	---	---	---	---
	5							
	6	in-house method	liquid extraction	HPLC-DAD	-	-	no	-
	7	N/A	N/A	N/A	N/A	N/A	N/A	N/A
	8							
	9							
	10							
	11							
	12							
	13	hplc/uv						
	14							
	15							
	16							
	17							

5.2 Homogeneity

5.2.1 Homogeneity of bottled PT-samples

Homogeneity test by determination of Vitamin D3 by HPLC/UV (ASU §64 49.00-1):

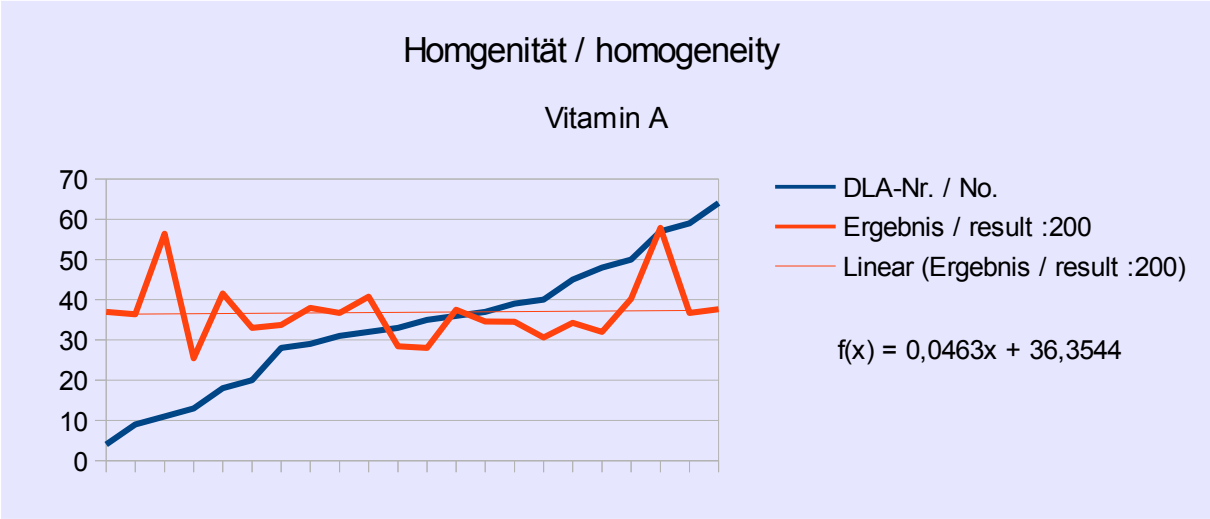
Vitamin D

Independant Samples	µg/100g
1	357
2	357
3	363
4	363
5	357

General Mean	359	
Repeatability standard deviation	3,65	1,02%

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 items can be shown by the trend line for information:



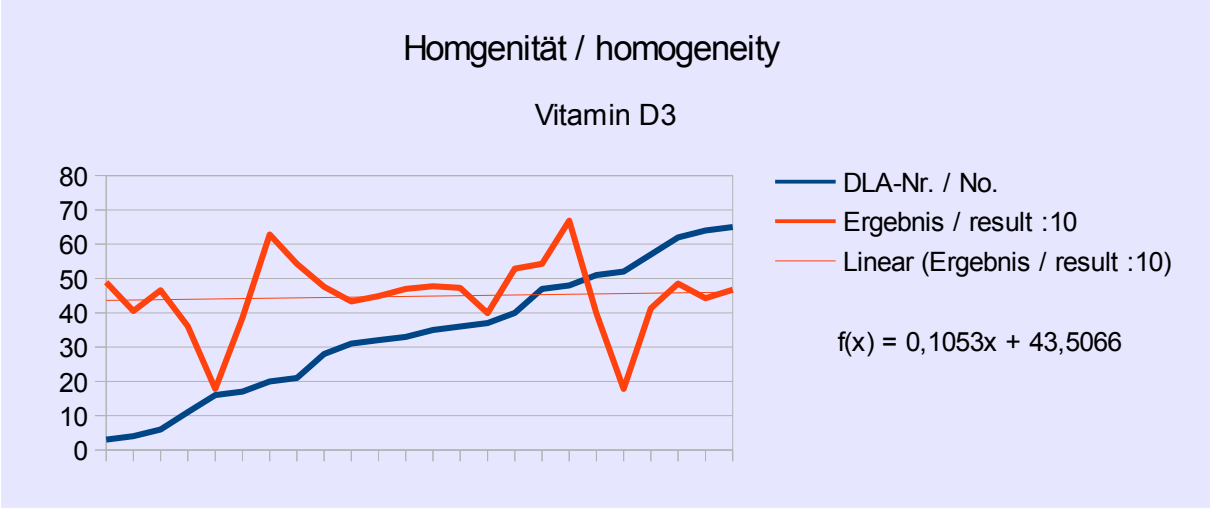


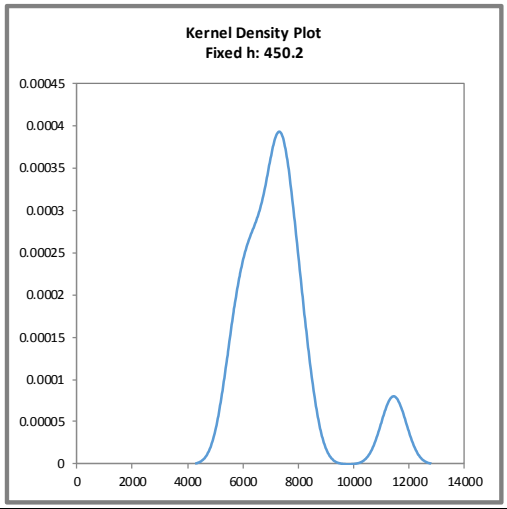
Abb./Fig. 13:
Trendfunktion Probennummern vs. Ergebnisse: Vitamin A und Vitamin D3 (1/200 und 1/10 dargestellt)
trend line function sample number vs. results: vitamin A and vitamin D3 (1/200 and 1/10 shown)

5.3 Kernel Density Plots of Results

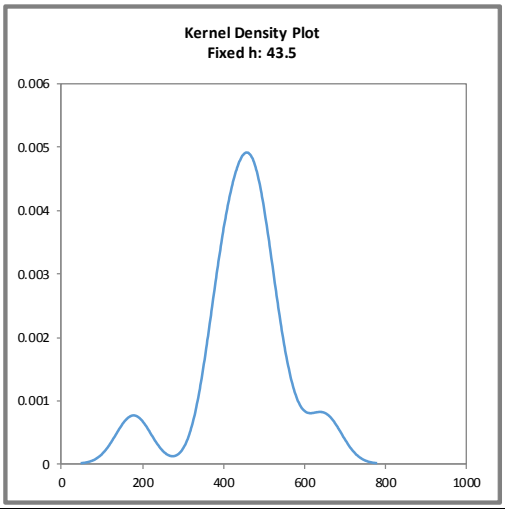
Abbildungen:
Kerndichte-Schätzungen
der Teilnehmerergebnisse
(mit h = 0,75 x σ_{pt} von Xpt)

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

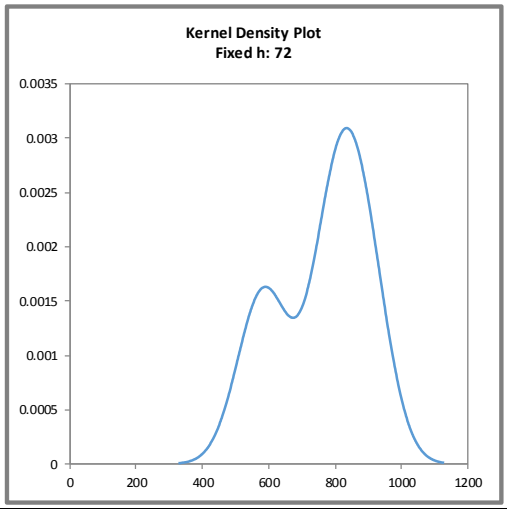
Vitamin A



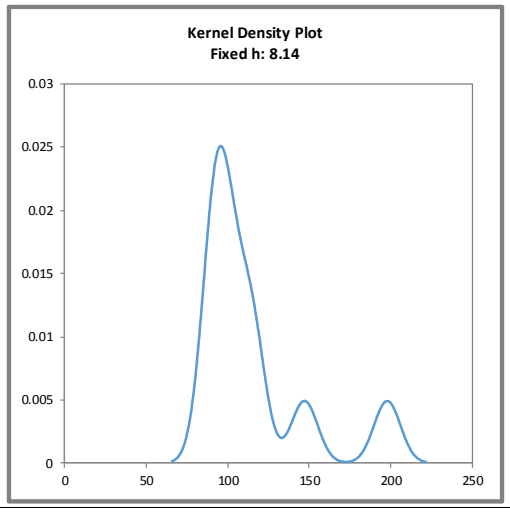
Vitamin D3



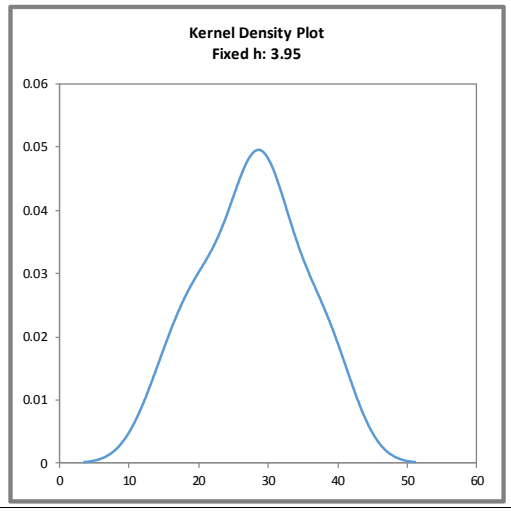
Vitamin E



Coenzym Q10 / Coenzyme Q10



Beta-Carotin(e)



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 45-2018***
PT name	***Food Supplement I: Vitamins A, E, D3, K1, β-Carotene, Coenzyme Q10 (Ubiquinone) and Alpha Liponic Acid***
*Sample matrix**	***Samples I + II:*** *Capsule powder (without capsule shells) / Ingredients: Maltodextrin, calcium carbonate, rice starch, further food additives and vitamins and minerals*
Number of samples and sample amount	*2 identical samples, 50 g each.*
Storage	*cooled 2 - 10°C (dry and dark)*
Intentional use	*Laboratory use only (quality control samples)*
Parameter	*quantitative: Vitamins A, E, D3, K1, β-Carotene and Coenzyme Q10 (Ubiquinone) and Alpha Liponic Acid* *Contents: The contents are of the order of the nutrient reference values per recommended daily dose (1-3 capsules approx. 0.5 - 6 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	*µg/100g and mg/100g*
Number of significant digits	*at least 2*
Further information	*For information please specify:* – *Date of analysis* – *DLA-sample-numbers (for sample I and I'I)* – *Limit of detection* – *Assignment incl. Recovery* – *Recovery with the same matrix* – *Method is accredited*
Result submission	*The result submission file should be sent by e-mail to:* ***pt@dla-lvu.de***
Deadline	***the latest 06th July 2018***
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-Scharf*

* 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
		UNITED KINGDOM
		HUNGARY
		Germany
		Germany
		Germany
		Germany
		CZECH REPUBLIC
		USA
		Germany
		Germany
		Germany
		Germany
		Germany
		Germany
		USA
		Germany
		Germany

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

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

7. Index of references

1. DIN EN ISO/IEC 17025:2005; Allgemeine Anforderungen an die Kompetenz von Prüf- und Kalibrierlaboratorien / General requirements for the competence of testing and calibration laboratories
2. DIN EN ISO/IEC 17043:2010; Konformitätsbewertung – Allgemeine Anforderungen an Eignungsprüfungen / Conformity assessment – General requirements for proficiency testing
3. ISO 13528:2015 & DIN ISO 13528:2009; Statistische Verfahren für Eignungsprüfungen durch Ringversuche / Statistical methods for use in proficiency testing by interlaboratory comparisons
4. ASU §64 LFGB: Planung und statistische Auswertung von Ringversuchen zur Methodenvalidierung / DIN ISO 5725 series part 1, 2 and 6 Accuracy (trueness and precision) of measurement methods and results
5. Verordnung / Regulation 882/2004/EU; Verordnung über über amtliche Kontrollen zur Überprüfung der Einhaltung des Lebensmittel- und Futtermittelrechts sowie der Bestimmungen über Tiergesundheit und Tierschutz / Regulation on official controls performed to ensure the verification of compliance with feed and food law, animal health and animal welfare rules
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21. ASU § 64 LFGB L 00.00-61 / DIN EN 12821:2009 [16b: 2000]: Bestimmung von

Vitamin D (Cholecalciferol (D_3) und Ergocalciferol (D_2)) in Lebensmitteln mittels HPLC / Foodstuffs. Determination of vitamin D by high performance liquid chromatography. Measurement of cholecalciferol (D3) or ergocalciferol (D2)

22. ASU § 64 LFGB L 00.00-62 / DIN EN 12822:2014: Bestimmung von Vitamin E (α-, β-, γ- und δ-Tocopherol) in Lebensmitteln mittels HPLC / Foodstuffs. Determination of vitamin E by high performance liquid chromatography. Measurement of α-, β-, γ- and δ-tocopherol
23. ASU § 64 LFGB L 00.00-63/1 / DIN EN 12823-1:2014: Bestimmung von Vitamin A in Lebensmitteln mittels HPLC, Teil 1: Bestimmung von all-trans-Retinol und 13-cis-Retinol / Foodstuffs. Determination of vitamin A by high performance liquid chromatography. Measurement of all-E-retinol and 13-Z-retinol
24. ASU § 64 LFGB L 00.00-63/2 / DIN EN 12823-2:2000: Bestimmung von Vitamin A in Lebensmitteln mittels HPLC, Teil 2: Bestimmung von β-Carotin / Foodstuffs. Determination of vitamin A by high performance liquid chromatography. Measurement of β-carotene
25. ASU § 64 LFGB L 00.00-86 / DIN EN 14148:2003: Untersuchung von Lebensmitteln - Bestimmung von Vitamin K_1 mit HPLC / Foodstuffs. Determination of vitamin K1 by HPLC