Evaluation Report
proficiency test

DLA ptSU02 (2020)

Food Supplements I:

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

in Multivitamin Capsule Powder

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General Information on the proficiency test (PT)

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

Content

1. Introduction

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

2. Realisation

2.1 Test material

The test material is a mixture of common in commerce food supplements (without capsule shells) with added maltodextrin as bulking agent from European suppliers.
The raw materials were sieved (mesh 600µm), 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 specification.

Table 1: Composition of DLA-Samples

PT-Sample Multivitamin Powder
Multivitamin powder (1. food supplement) Ingredients: calcium carbonate, maltodextrin, magnesium oxide, ascorbic acid, lemon bioflavonides, green tea extract, choline bitartrate, grape seed extract, lutein, iron sulfate, thiamine HCl, pyridoxine HCl, lycopene (tomato extract), vitamin E (DL-alpha tocopherol acetate), calcium pantothenate, silicon dioxide, riboflavin, nicotinamide, inositol, quercetin, zinc oxide, cyanocobalamin, vitamin D3, coenzyme Q10, black pepper extract, vitamin A, lactobacillus acidophilus, vitamin K (phylloquinone), sodium tetraborate, folic acid, chromium-III-chloride, manganese sulfate, copper sulfate, sodium selenite, D-biotin.
Multivitamin powder (2. food supplement) Ingredients: acidifying agent citric acid, dextrose, dicalcium phosphate, maize dextrin, calcium carbonate, flavors, magnesium citrate, L-ascorbic acid, zinc picolinate, sucralose and acesulfame-K, potassium chloride (contains silicon dioxide), DL-alpha-tocopheryl acetate, choline bitartrate, citrus bioflavonoids, ginseng root extract, nicotinamide, alpha-liponic acid, royal jelly, calcium D-pantothenate, spirulina powder, ginkgo biloba extract, beta carotene, inositol, green tea extract, retinyl acetate, manganese-II-sulfate, potassium iodide, grape seed extract, nettle extract, Pyridoxine hydrochloride, riboflavin, coenzyme Q10, cholecalciferol, copper-II-citrate, pteroylmonoglutamic acid, chromium-III-chloride, sodium selenite, D-biotin, sodium molybdate, phylloquinone, methylcobalamin, biotin, niacin.
Multivitamin powder (3. food supplement) Ingredients: Green tea leaf extract, ascorbyl palmitate, calcium ascorbate, magnesium ascorbate, d-alpha tocopheryl acetate, L-glutathione, L-methionine, L-glutamine, grape seed extract, soy lecithin, selenium methionine, beta-carotene, lycopene, lutein, cellulose powder.
Alpha liponic acid powder (4. food supplements) Ingredients: alpha liponic acid 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

Parameter	**Content per 100g**
Alpha liponic acid	350 mg
Beta-Carotene	5,1 mg
Coenzyme Q10 (Ubiquinone)	160 mg
Vitamin A (as Retinol, without Provitamins)	(8000 µg) *
Vitamin D3 (as Cholecalciferol)	410 µg
Vitamin E (as D-α-Tocopherol)	230 mg
Vitamin K1 (as Phylloquinone)	1600 µg

* Note: The declared value is very different from the analysis results.

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 95%. 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 0,71 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 < 12,5% (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**
Alpha Liponic Acid	2,70 %
Beta-Carotene	12,5 %
Coenzyme Q10	2,14 %
Vitamin A	8,40 %
Vitamin D3	3,47 %
Vitamin E	4,66 %
Vitamin K1	2,54 %

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. 0,28 (18,3°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 10th week of 2020. The testing method was optional. The tests should be finished at 15th May 2020 the latest (extended).

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

The two portions contain identical samples of a food supplement with above mentioned parameters in the matrix of capsule powder (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 20 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 paramaters in the present PT the target standard deviation according to the general model of Horwitz was applied (see 3.6.1): alpha liponic acid, coenzyme Q10, vitamin A, vitamin D3 and vitamin K1.

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) [22, 24]: beta-carotene and vitamin E.

Additionally for beta-carotene, coenzyme Q10, vitamin E and K1 the standard uncertainty was considered by evaluation using z'-scores (see 3.6.8).

3.6.1 General model (Horwitz)

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

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

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

3.6.2 Value by precision experiment

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

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

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

Table 4: Relative repeatability standard deviations (RSD_r) and relative reproducibility standard deviations (RSD_R) according to selected evaluations of tests for precision and the resulting target standard deviation σ_{pt} [18-25]

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 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 since 2014 (SD = standard deviation, CV = coefficient of variation, MV = Multivitamin)

Parameter	**Matrix** (Powder)	**robust Mean**	**rob. SD (S*)**	**rel. SD (VK_{S*})**	**Quotient S^*/σ_{pt}**	**DLA-Report**
Vitamin A	MV-Capsule Powder	21900 µg/100g	2870 µg/100g	13,1%	1,8	DLA 47/2016
Vitamin A	MV-Capsule Powder	7131 µg/100g	1058 µg/100g	14,8%	1,8	DLA 45/2018
Vitamin A	MV-Capsule Powder	50071 µg/100g	6345 µg/100g	12,7%	2,0	DLA ptSU02 2020
Vitamin D3	MV-Capsule Powder	146 µg/100g	10,3 µg/100g	7,05%	0,46	DLA 47/2016
Vitamin D3	MV-Capsule Powder	455 µg/100g	74,4 µg/100g	16,4%	1,3	DLA 45/2018
Vitamin D3	MV-Capsule Powder	515 µg/100g	117 µg/100g	22,8%	1,8	DLA ptSU02 2020
Vitamin E	MV-Capsule Powder	988 mg/100g	211 mg/100g	21,4%	1,7	DLA 47/2016
Vitamin E	MV-Capsule Powder	760 mg/100g	148 mg/100g	19,5%	1,5	DLA 45/2018
Vitamin E	MV-Capsule Powder	234 mg/100g	64,0 mg/100g	27,4%	1,8*	DLA ptSU02 2020
Vitamin K1	MV-Capsule Powder	933 µg/100g	121 µg/100g	13,0%	1,1	DLA 47/2016
Vitamin K1	MV-Capsule Powder	954 µg/100g	632 µg/100g	66,2%	-	DLA 45/2018
Vitamin K1	MV-Capsule Powder	1039 µg/100g°	604 µg/100g	49,8%	2,1*	DLA ptSU02 2020
α-Liponic acid	MV-Capsule Powder	393 mg/100g°	21,5 mg/100g	5,32%	1,2	DLA ptSU02 2020
β-Carotene	MV-Capsule Powder	32,2 mg/100g	9,70 mg/100g	30,1%	2,0	DLA 47/2016
β-Carotene	MV-Capsule Powder	27,7 mg/100g	8,45 mg/100g	30,5%	1,6*	DLA 45/2018
β-Carotene	MV-Capsule Powder	4,26 mg/100g	2,11 mg/100g	49,4%	2,0*	DLA ptSU02 2020
Coenzyme Q10	MV-Tablets	241 mg/100g	15 mg/100g	6,22%	1,3	DLA 49/2016
Coenzyme Q10	MV-Capsule Powder	103 mg/100g	14,0 mg/100g	13,6%	1,7*	DLA 45/2018
Coenzyme Q10	MV-Capsule Powder	131 mg/100g	30,1 mg/100g	23,0%	2,1*	DLA ptSU02 2020

°assigned value (Xpt): Median

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

3.8 z'-Score

The z'-score can be used for the valuation of the results of the participants, in cases the standard uncertainty has to be considered (s. 3.11). The z'-score represents the relation of the deviation of the result (x_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 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 and outlier testing single results were excluded before statistic evaluation.

In the case of alpha liponic acid, a kernel density estimation was not made due to the number of < 8 results. With respect to vitamin D3 and E, the very high outliers are not shown in the figures (see 5.3).

Comments to the statistic data:

There were <7 results for alpha liponic acid, so the statistical evaluation was provided for information only.

The median was used as the assigned value for the parameters alpha liponic acid and vitamin K1, and the robust mean for all other parameters (cf. 3.1).

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 A and D3 and alpha liponic acid the distribution showed a normal variability of results. The quotients S^*/σ_{pt} were in the range of 1,2 to 2,0 (s. Tab. 5).
For beta-carotene, coenzyme Q10, vitamin E and K1 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 or 2,1 for coenzym Q10 and vitamin K1 (s. Tab. 5).

The repeatability 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.

67% to 80% of results were in the respective target range.
Except for vitamin A the robust means or medians of the participant results were for all parameters in the range of 64% to 126% of the vitamin contents according to the manufacturer specifications (s. Tab. 2). The declared value for vitamin A is very different from the analytical results.

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 σ_{pt}**	**z-Score** (Info)	**Hinweis** / **Remark**

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

4.1 Alpha Liponic Acid (in mg/100g)

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	5
Number of outliers	–
Mean	413
Robust Mean	404
Median (X_{pt})	**393**
Robust standard deviation (S*)	**21,5**
Number with 2 replicates	4
Repeatability SD (S_r)	10,7
Repeatability (CV_r)	2,70%
Reproducibility SD (S_R)	–
Reproducibility (CV_R)	–%
Target range:	
Target standard deviation σ_{pt}	**18,1**
lower limit of target range	**357**
upper limit of target range	**429**
Quotient S/σ_{pt}*	*1,2*
Standard uncertainty $U_{(Xpt)}$	*12,0*
Results in the target range	*4*
Percent in the target range	*80%*

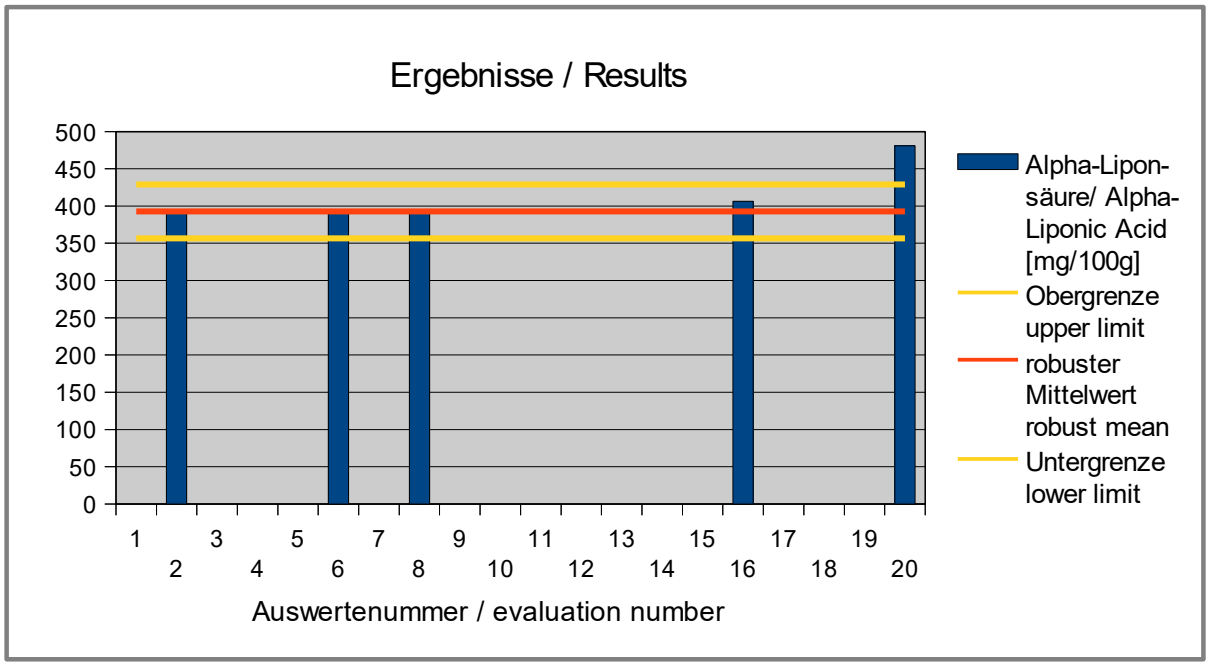


Abb. / Fig. 1: Ergebnisse Alpha-Liponsäure/ Results Alpha Liponic Acid

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Alpha-Lipon-säure/ Alpha-Liponic Acid [mg/100g]	Abweichung [mg/100g] Deviation [mg/100g]	z-Score (σ_{pt})	Hinweis Remark
1				
2	391	-1,7	-0,09	
3				
4				
5				
6	392	-0,7	-0,04	
7				
8	393	0,0	0,00	
9				
10				
11				
12				
13				
14				
15				
16	407	13,8	0,76	
17				
18				
19				
20	481	88,3	4,9	

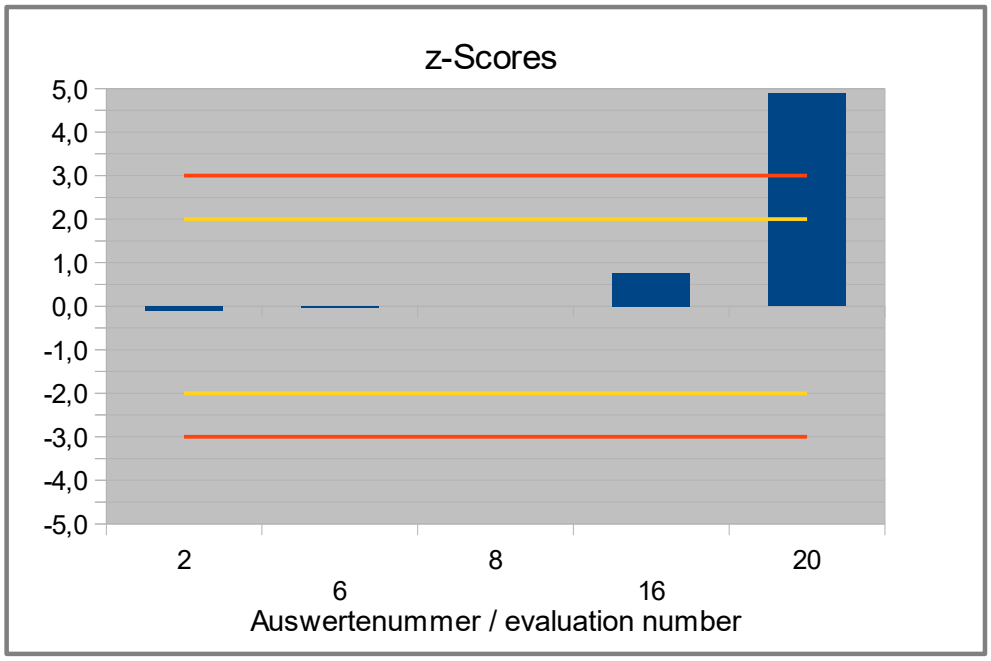


Abb. / Fig. 2: z-Scores Alpha-Liponsäure/ Alpha Liponic Acid

4.2 Beta-Carotene (without other provitamins in mg/100g)

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results°	8
Number of outliers	2
Mean	4,35
Median	4,13
Robust Mean (X_{pt})	**4,26**
Robust standard deviation (S*)	**2,11**
Number with 2 replicates	6
Repeatability SD (S_r)	0,443
Repeatability (CV_r)	12,5%
Reproducibility SD (S_R)	1,46
Reproducibility (CV_R)	41,5%
Target range:	
Target standard deviation σ_{pt}'	**1,12**
Target standard deviation (for Information)	0,388
lower limit of target range	**2,02**
upper limit of target range	**6,51**
Quotient S^*/σ_{pt}'	*1,9*
Standard uncertainty $U_{(Xpt)}$	*0,932*
Results in the target range	*6*
Percent in the target range	*75%*

°results without outliers (results no. 4 and 8)

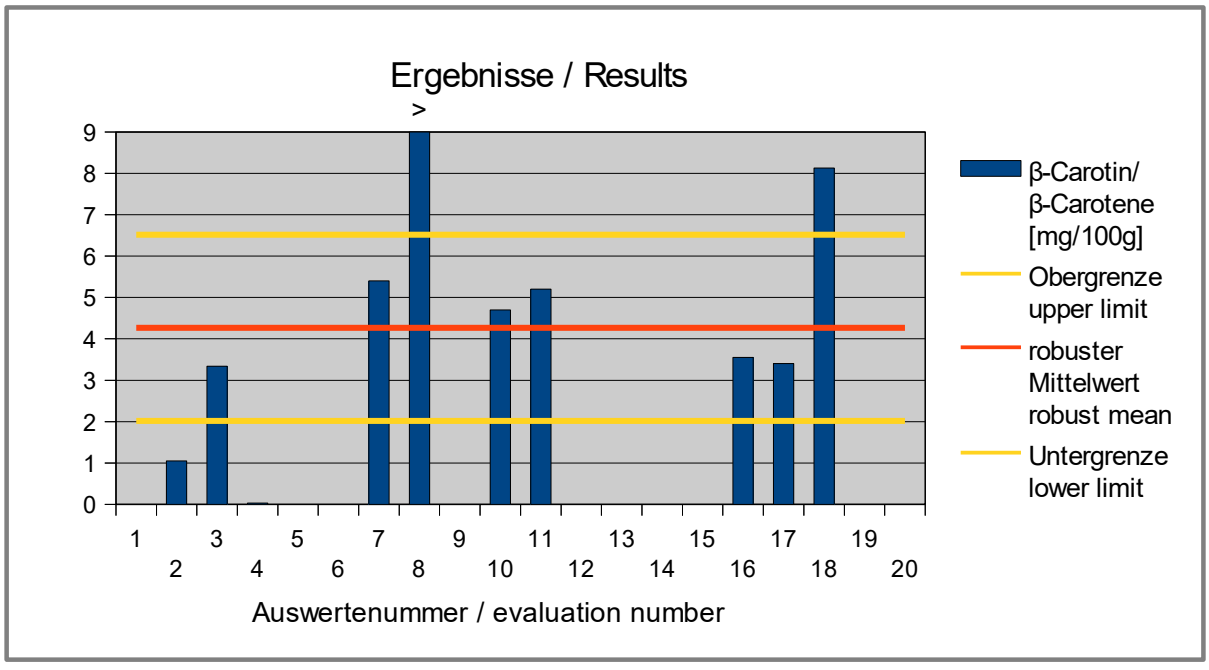


Abb. / Fig. 3: Ergebnisse Beta-Carotin/ Results Beta-Carotene

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	1,05	-3,21	-2,9	-8,3	
3	3,34	-0,92	-0,82	-2,4	
4	0,0300				Ausreißer ausgeschlossen / Outlier excluded
5					
6					
7	5,40	1,14	1,0	2,9	
8	24,9				Ausreißer ausgeschlossen / Outlier excluded
9					
10	4,70	0,44	0,39	1,1	
11	5,20	0,94	0,83	2,4	
12					
13					
14					
15					
16	3,55	-0,71	-0,64	-1,8	
17	3,40	-0,86	-0,77	-2,2	
18	8,13	3,87	3,4	10	
19					
20					

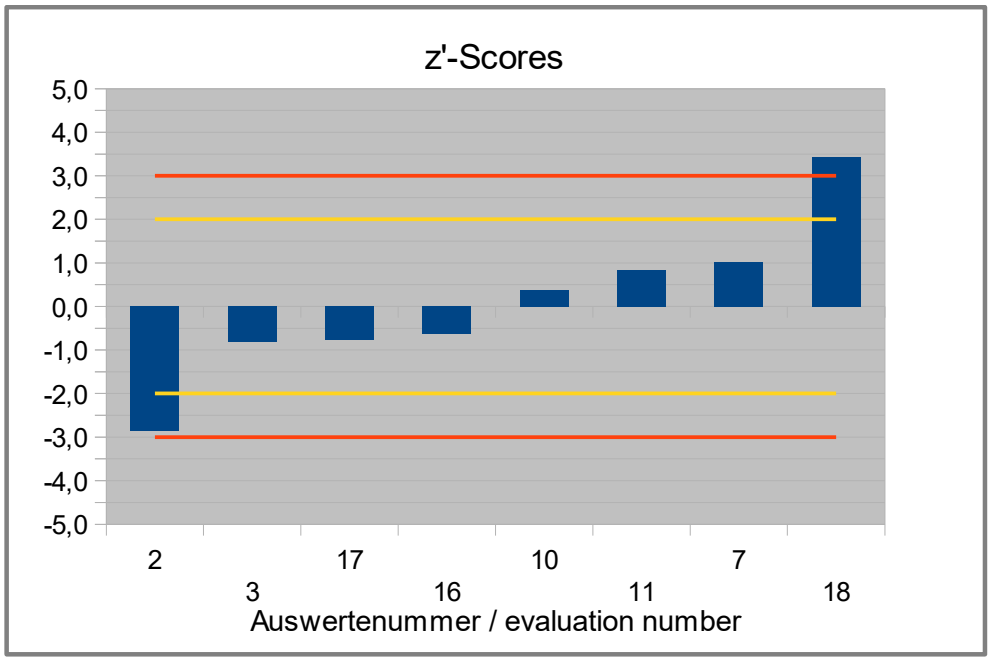


Abb. / Fig. 4: z'-Scores Beta-Carotin/ Beta-Carotene

4.3 Coenzyme Q10 (Ubiquinone in mg/100g)

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	9
Number of outliers	0
Mean	130
Median	126
Robust Mean (x_{pt})	**131**
Robust standard deviation (S*)	**30,1**
Number with 2 replicates	8
Repeatability SD (S_r)	2,68
Repeatability (CV_r)	2,14%
Reproducibility SD (S_R)	27,7
Reproducibility (CV_R)	22,1%
Target range:	
Target standard deviation σ_{pt}'	**14,4**
lower limit of target range	**102**
upper limit of target range	**160**
Quotient S/σ_{pt}'*	*2,1*
Standard uncertainty $U_{(xpt)}$	*12,6*
Results in the target range	*6*
Percent in the target range	*67%*

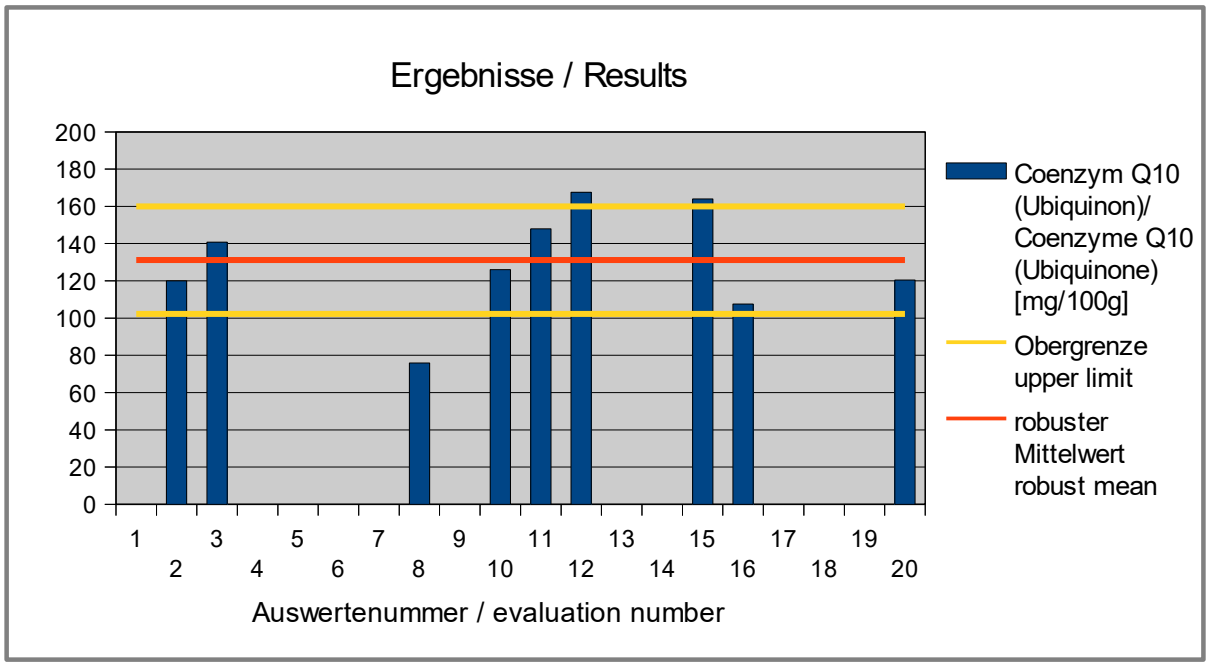


Abb. / Fig. 5: Ergebnisse Coenzym Q10 (Ubiquinon)/ Results Coenzyme Q10 (Ubiquinone)

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Coenzym Q10 (Ubiquinon)/ Coenzyme Q10 (Ubiquinone) [mg/100g]	Abweichung [mg/100g] Deviation [mg/100g]	z'-Score (σ_{pt})	Hinweis Remark
1				
2	120	-11,1	-0,77	
3	141	9,6	0,67	
4				
5				
6				
7				
8	75,9	-55,2	-3,8	
9				
10	126	-5,1	-0,36	
11	148	16,9	1,2	
12	168	36,5	2,5	
13				
14				
15	164	32,9	2,3	
16	108	-23,6	-1,6	
17				
18				
19				
20	121	-10,6	-0,74	

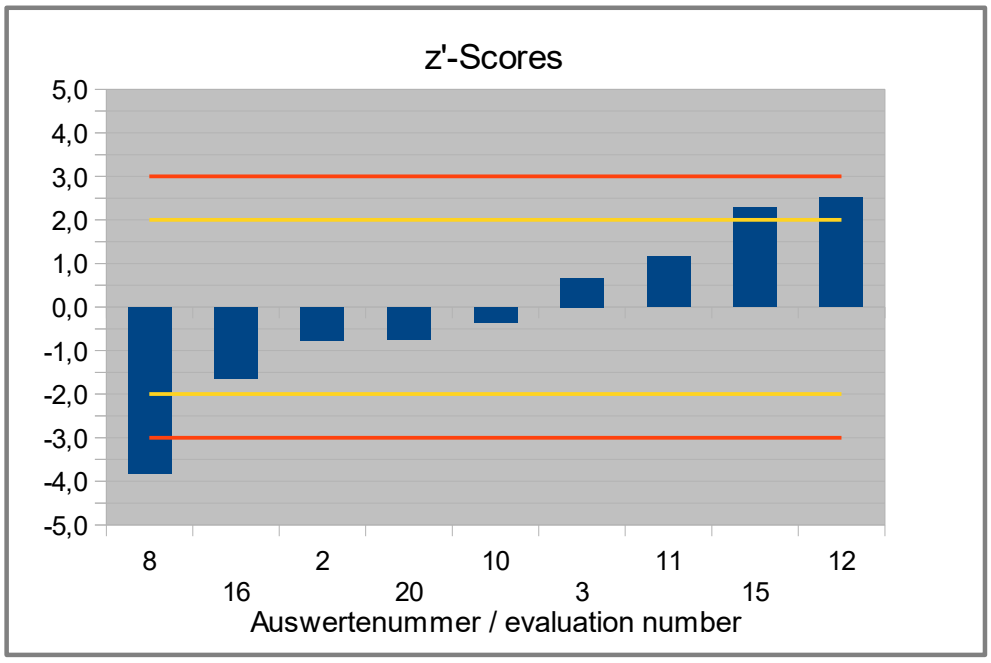


Abb. / Fig. 6: z'-Scores Coenzym Q10 (Ubiquinon)/ Coenzyme Q10 (Ubiquinone)

4.4 Vitamin A (as retinol without provitamins in µg/100g)

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results°	14
Number of outliers	3
Mean	50800
Median	47800
Robust Mean (x_{pt})	**50100**
Robust standard deviation (S*)	**6350**
Number with 2 replicates	13
Repeatability SD (S_r)	4140
Repeatability (CV_r)	8,40%
Reproducibility SD (S_R)	5860
Reproducibility (CV_R)	11,9%
Target range:	
Target standard deviation σ_{pt}	**3140**
Target standard deviation (for Information)	1530
lower limit of target range	**43800**
upper limit of target range	**56400**
Quotient S/σ_{pt}*	*2,0*
Standard uncertainty $U_{(xpt)}$	*2120*
Results in the target range	*11*
Percent in the target range	*79%*

°results without outliers (results no. 9, 14 and 15)

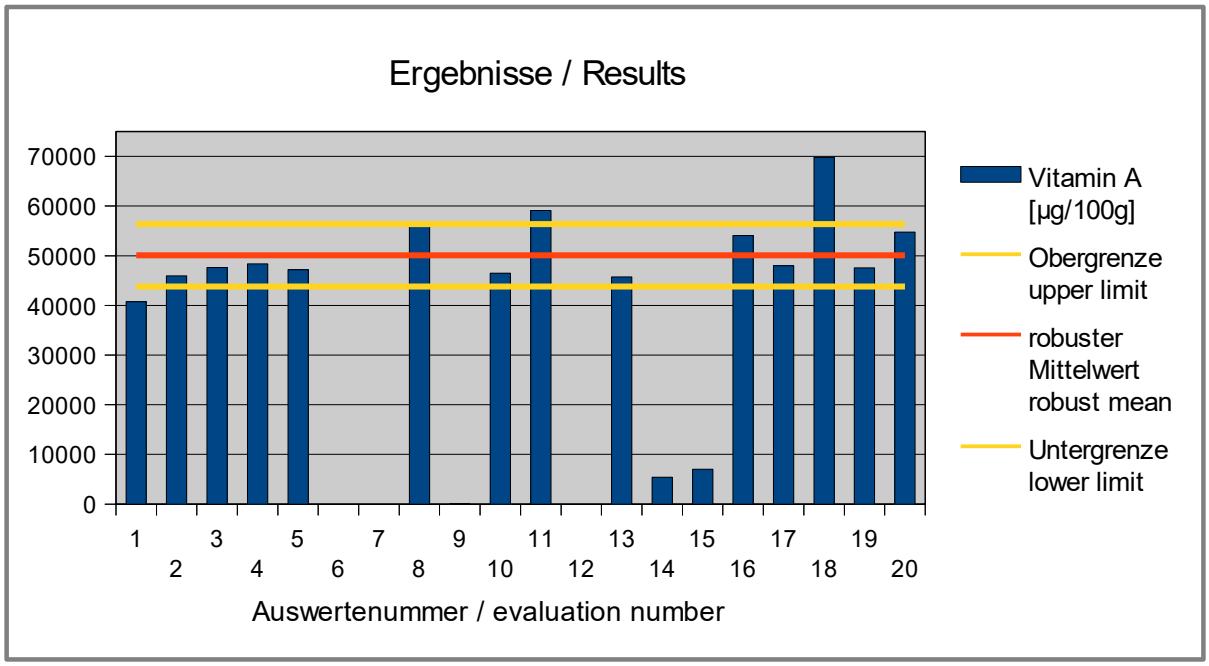


Abb. / Fig. 7: Ergebnisse Vitamin A/ Results Vitamin A

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	40750	-9321	-3,0	-6,1	
2	45950	-4121	-1,3	-2,7	
3	47615	-2456	-0,78	-1,6	
4	48350	-1721	-0,55	-1,1	
5	47155	-2916	-0,93	-1,9	
6					
7					
8	55883	5812	1,8	3,8	
9	30,4				Ausreißer ausgeschlossen / Outlier excluded
10	46500 *	-3571	-1,1	-2,3	
11	59100	9029	2,9	5,9	
12					
13	45750	-4320	-1,4	-2,8	
14	5408				Ausreißer ausgeschlossen / Outlier excluded
15	7025				Ausreißer ausgeschlossen / Outlier excluded
16	54050	3979	1,3	2,6	
17	48000	-2071	-0,66	-1,4	
18	69800	19729	6,3	13	
19	47550	-2521	-0,80	-1,6	
20	54750	4679	1,5	3,1	

* Mean calculated by DLA

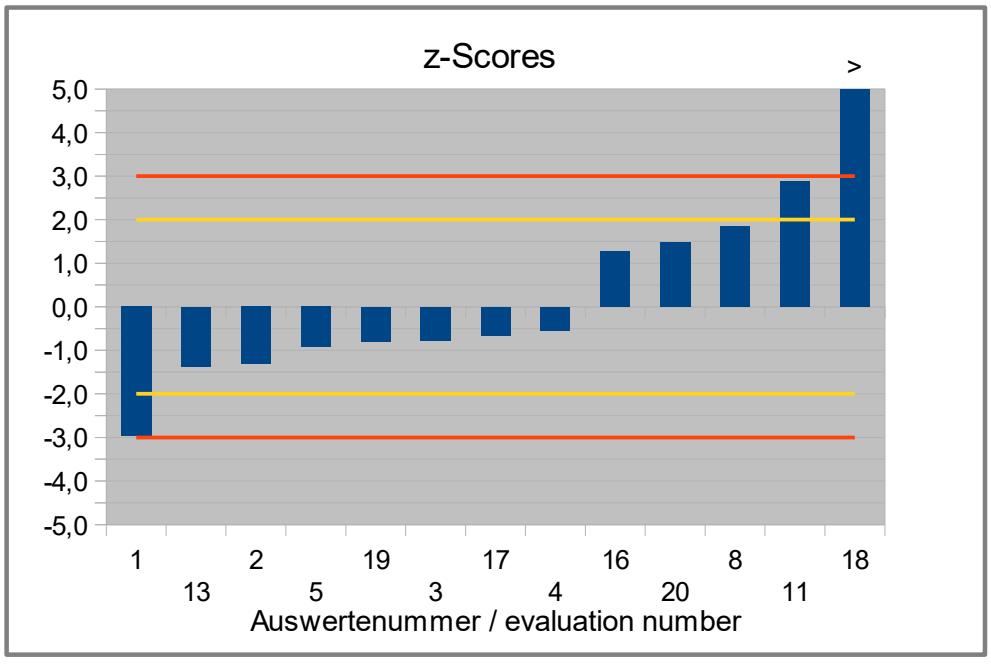


Abb. / Fig. 8: z-Scores Vitamin A

4.5 Vitamin D3 (as cholecalciferol in µg/100g)

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results°	14
Number of outliers	1
Mean	503
Median	549
Robust Mean (x_{pt})	**515**
Robust standard deviation (S*)	**117**
Number with 2 replicates	11
Repeatability SD (S_r)	17,2
Repeatability (CV_r)	3,47%
Reproducibility SD (S_R)	138
Reproducibility (CV_R)	27,9%
Target range:	
Target standard deviation σ_{pt}	**64,4**
Target standard deviation (for Information)	63,4
lower limit of target range	**386**
upper limit of target range	**644**
Quotient S/σ_{pt}*	*1,8*
Standard uncertainty $U_{(Xpt)}$	*39,2*
Results in the target range	*10*
Percent in the target range	*71%*

°*results without outliers (result no. 5)*

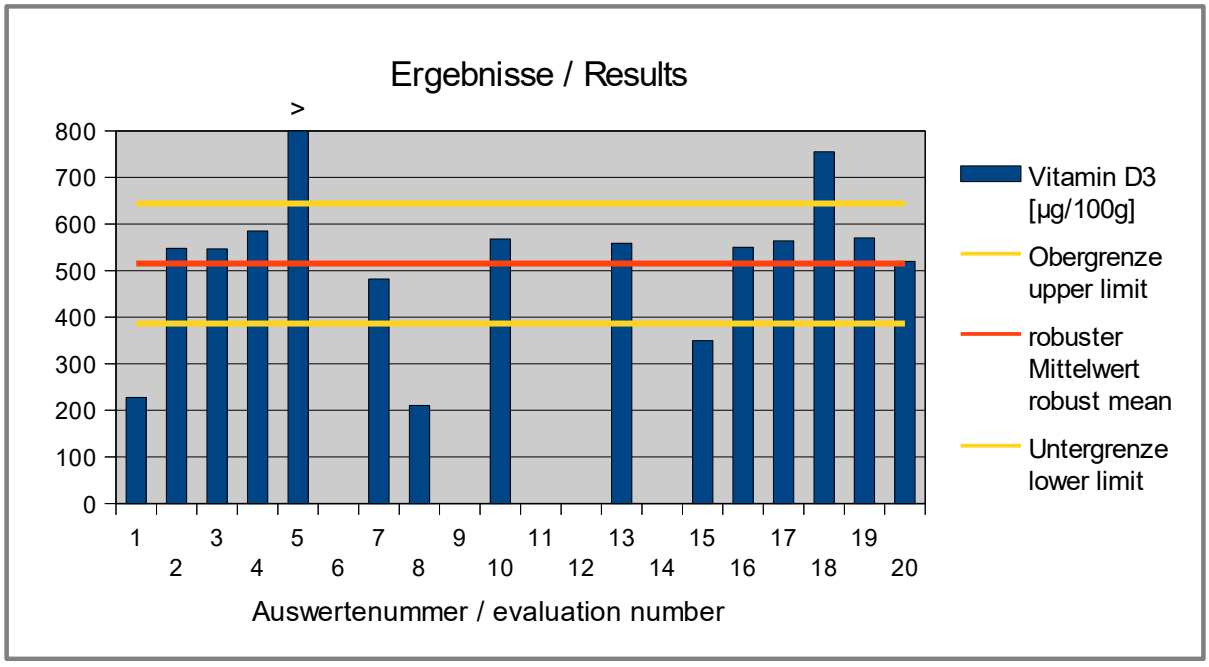


Abb. / Fig. 9: Ergebnisse Vitamin D3/ Results Vitamin D3

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Vitamin D3 [µg/100g]	Abweichung [µg/100g] Deviation [µg/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	228	-288	-4,5	-4,5	
2	548	33	0,51	0,52	
3	547	31	0,49	0,50	
4	585	70	1,1	1,1	
5	620000				Ausreißer ausgeschlossen / Outlier excluded
6					
7	482	-33	-0,52	-0,52	
8	211	-305	-4,7	-4,8	
9					
10	568	53	0,82	0,83	
11					
12					
13	559	44	0,68	0,69	
14					
15	350	-165	-2,6	-2,6	
16	550	35	0,54	0,55	
17	564	49	0,76	0,77	
18	755	240	3,7	3,8	
19	570	55	0,85	0,86	
20	520	5	0,07	0,08	

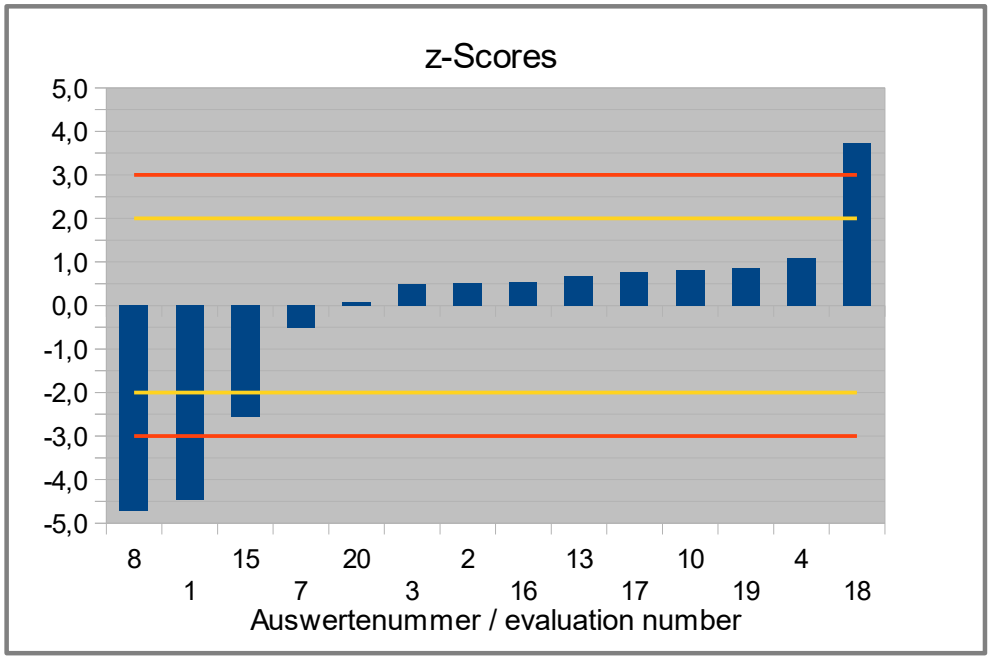


Abb. / Fig. 10: z-Scores Vitamin D3

4.6 Vitamin E (as D-α-tocopherol in mg/100g)

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results°	17
Number of outliers	2
Mean	235
Median	234
Robust Mean (X_{pt})	**234**
Robust standard deviation (S*)	**64,0**
Number with 2 replicates	14
Repeatability SD (S_r)	11,2
Repeatability (CV_r)	4,66%
Reproducibility SD (S_R)	76,1
Reproducibility (CV_R)	31,5%
Target range:	
Target standard deviation σ_{pt}'	**35,3**
Target standard deviation (for Information)	11,6
lower limit of target range	**163**
upper limit of target range	**305**
Quotient S/σ_{pt}'*	*1,8*
Standard uncertainty $U_{(Xpt)}$	*19,4*
Results in the target range	*12*
Percent in the target range	*71%*

°results without outliers (results no. 8 and 16)

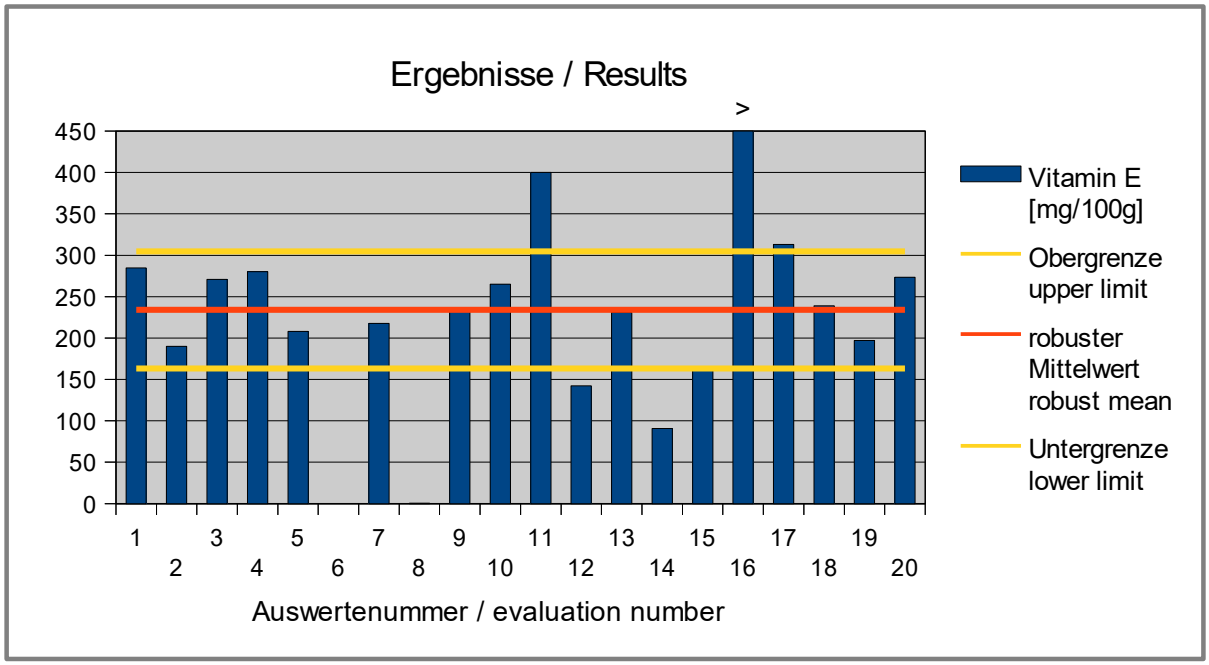


Abb. / Fig. 11: Ergebnisse Vitamin E / Results Vitamin E

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	285	50,6	1,4	4,3	
2	190	-43,9	-1,2	-3,8	
3	271	37,0	1,0	3,2	
4	280	46,1	1,3	4,0	
5	208	-25,9	-0,73	-2,2	
6					
7	218	-16,2	-0,46	-1,4	Result converted
8	0,526				Ausreißer ausgeschlossen / Outlier excluded
9	234	0,2	0,01	0,02	
10	265	31,1	0,88	2,7	
11	400	166,1	4,7	14,3	
12	142	-91,6	-2,6	-7,9	
13	231	-3,1	-0,09	-0,27	
14	90,7	-143,2	-4,1	-12,3	
15	163	-71,0	-2,0	-6,1	
16	284000				Ausreißer ausgeschlossen / Outlier excluded
17	313	79,1	2,2	6,8	
18	239	5,1	0,14	0,44	
19	197	-36,9	-1,0	-3,2	
20	274	39,6	1,1	3,4	

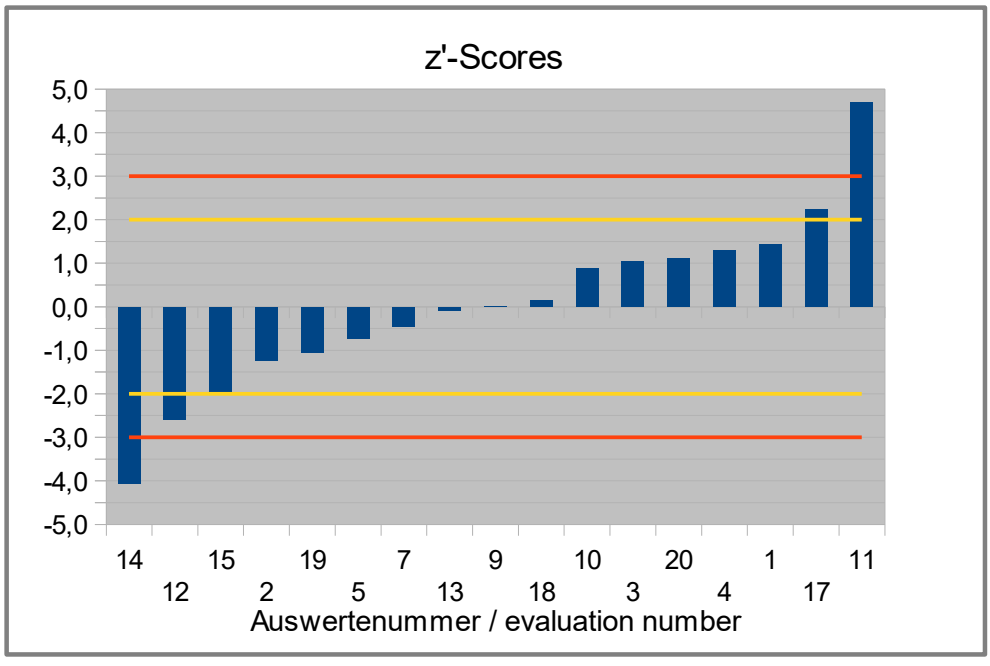


Abb. / Fig. 12: z'-Scores Vitamin E

4.7 Vitamin K1 (as phylloquinone in µg/100g)

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results°	8
Number of outliers	2
Mean	1310
Robust Mean	1210
Median (x_{pt})	**1040**
Robust standard deviation (S*)	**604**
Number with 2 replicates	7
Repeatability SD (S_r)	27,6
Repeatability (CV_r)	2,54%
Reproducibility SD (S_R)	418
Reproducibility (CV_R)	38,6%
Target range:	
Target standard deviation σ_{pt}'	**292**
Target standard deviation (for Information)	51,9
lower limit of target range	**456**
upper limit of target range	**1620**
Quotient S/σ_{pt}'*	*2,1*
Standard uncertainty $U_{(Xpt)}$	*267*
Results in the target range	*6*
Percent in the target range	*75%*

° *results without outliers (results no. 9 and 18)*

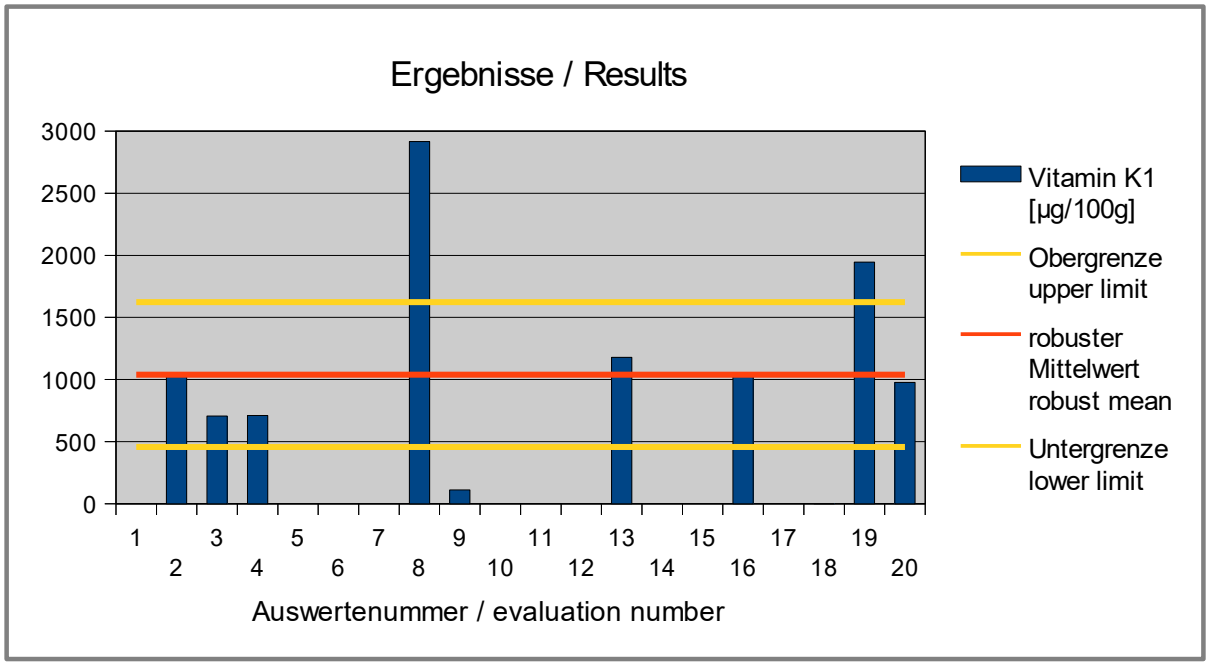


Abb. / Fig. 13: Ergebnisse Vitamin K1 / Results Vitamin K1

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					
2	1023	-16	-0,05	-0,31	
3	706	-333	-1,1	-6,4	
4	710	-329	-1,1	-6,3	
5					
6					
7					
8	2916	1877	6,4	36	
9	111				Ausreißer ausgeschlossen / Outlier excluded
10					
11					
12					
13	1179	140	0,48	2,7	
14					
15					
16	1055	16	0,05	0,31	
17					
18	1,07				Ausreißer ausgeschlossen / Outlier excluded
19	1945	906	3,1	17	
20	977	-63	-0,21	-1,2	

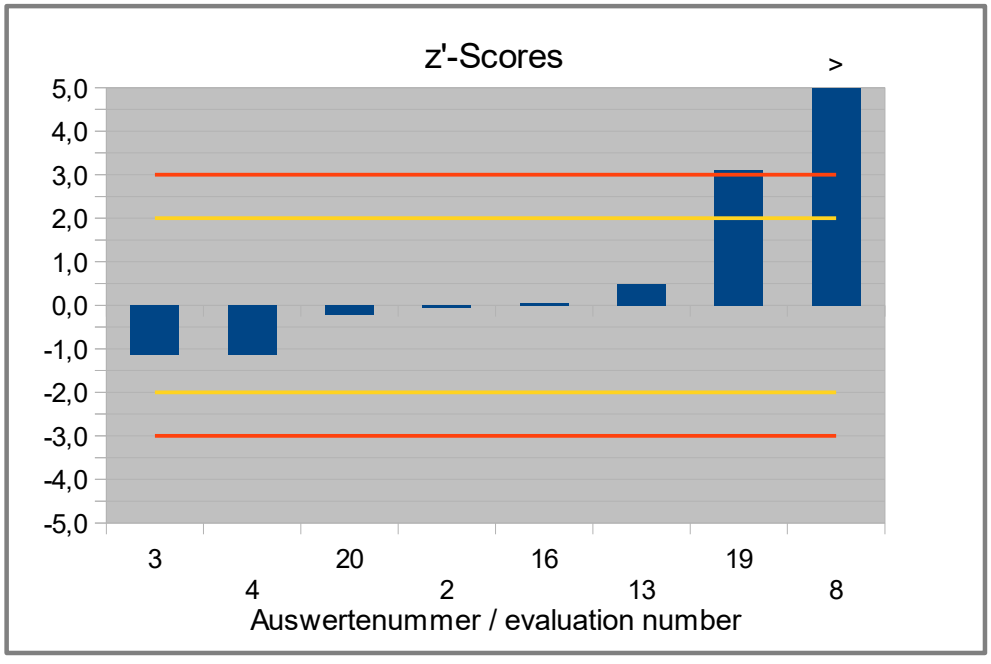


Abb. / Fig. 14: z'-Scores Vitamin K1

4.8 Participant z-Scores: overview table

Evaluation number	Alpha Lipo-nic Acid	Beta-Caro-tene	Coenzyme Q10	Vitamin A	Vitamin D3	Vitamin E	Vitamin K1
	z-Score	z'-Score	z'-Score	z-Score	z-Score	z'-Score	z'-Score
1				-3,0	-4,5	1,4	
2	-0,09	-2,9	-0,77	-1,3	0,51	-1,2	-0,05
3		-0,82	0,67	-0,78	0,49	1,0	-1,1
4				-0,55	1,1	1,3	-1,1
5				-0,93		-0,73	
6	-0,04						
7		1,0			-0,52	-0,46	
8	0,00		-3,8	1,8	-4,7		6,4
9						0,01	
10		0,39	-0,36	-1,1	0,82	0,88	
11		0,83	1,2	2,9		4,7	
12			2,5			-2,6	
13				-1,4	0,68	-0,09	0,48
14						-4,1	
15			2,3		-2,6	-2,0	
16	0,76	-0,64	-1,6	1,27	0,54		0,05
17		-0,77		-0,66	0,76	2,2	
18		3,4		6,3	3,7	0,14	
19				-0,80	0,85	-1,0	3,1
20	4,9		-0,74	1,5	0,07	1,1	-0,21

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	Participant	Unit	Sample I DLA No.	Sample II DLA No.	Date of analysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quanti-fication	Incl. RR	Recovery rate
					Day/Month					yes / no	in %
Alpha Liponsäure / Alpha Liponic Acid	1	mg/100g									
	2	mg/100g	15	45	06.04.20	391	384	397	0,1		
	3	mg/100g									
	4	mg/100g									
	5	mg/100g									
	6	mg/100g	16	44	13.04.20	392	394	390	N/A	N/A	N/A
	7	mg/100g	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
	8	mg/100g	17	43	27.03.20	392,7	382,2	403,2	-	no	-
	9	mg/100g									
	10	mg/100g									
	11	mg/100g									
	12	mg/100g									
	13	mg/100g									
	14	mg/100g									
	15	mg/100g									
	16	mg/100g	22	38	04.05	406,5	415	398		no	
	17	mg/100g									
	18	mg/100g									
	19	mg/100g									
	20	mg/100g	20	40	31. Mrz	481	502	460			

Parameter	Participant	Unit	Sample I DLA No.	Sample II DLA No.	Date of analysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quanti-fication	Incl. RR	Recovery rate
					Day/Month					yes / no	in %
β -Carotin (berechnet als β-Carotin, ohne andere Provitamine) / β-Carotene (calculated as β-Carotene, without other Provitamins)	1	mg/100g									
	2	mg/100g	15	45	08.04.20	1,05	1,05	1,04	0,05		
	3	mg/100g	28	32	16.03.20	3,34	3,35	3,32	0,004	no	-
	4	mg/100g	23	37	27.03.20	0,03	0,03	0,03	0,07	no	
	5	mg/100g									
	6	mg/100g									
	7	mg/100g	11	49	16.04.20	5,4	5,2	5,6	N/A	no	N/A
	8	mg/100g	17	43	31.03.20	24,9	21,5	28,2	-	no	-
	9	mg/100g									
	10	mg/100g	4,8	4,6	01.05.20	4,7				no	
	11	mg/100g	24	7	23.04.20	5,2	5,7	4,6	1,6	no	1,01
	12	mg/100g									
	13	mg/100g									
	14	mg/100g									
	15	mg/100g									
	16	mg/100g	22	38	05.05	3.55	3,39	3,71		no	
	17	mg/100g	34	26	19.03.20	3,4	3,9	2,9		no	
	18	mg/100g	51-A	51-B	28.04.20	8,13	7,9	8,35	2	no	
	19	mg/100g									
	20	mg/100g									

Parameter	Participant	Unit	Sample I DLA No.	Sample II DLA No.	Date of analysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quanti-fication	Incl. RR	Recovery rate
					Day/Month					yes / no	in %
Coenzym Q10 (Ubiquinon)/ Coenzyme Q10 (Ubiquinone)	1	mg/100g									
	2	mg/100g	15	45	02.04.20	120	121	118	0,1		
	3	mg/100g	28	32	02.04.20	140,75	139,93	141,57	0,5	no	-
	4	mg/100g									
	5	mg/100g									
	6	mg/100g									
	7	mg/100g	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
	8	mg/100g	17	43	02.04.20	75,91	76,07	75,74	-	no	-
	9	mg/100g									
	10	mg/100g	125	126	01.03.20	126				no	
	11	mg/100g	24	7	06.05.	148	150	146	15	no	1,09
	12	mg/100g	18	42	13.05.20	167,6	168,6	166,5	5 mg/kg		
	13	mg/100g									
	14	mg/100g									
	15	mg/100g			15/4	164			10	no	
	16	mg/100g	22	38	05.05	107,5	107	108	0,2	no	
	17	mg/100g									
	18	mg/100g									
	19	mg/100g									
	20	mg/100g	20	40	31. Mrz	120,5	125	116			

Parameter	Participant	Unit	Sample I DLA No.	Sample II DLA No.	Date of analysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quanti-fication	Incl. RR	Recovery rate
					Day/Month					yes / no	in %
Vitamin A (berechnet als Retinol ohne Provitamine) / Vitamin A (calculated as Retinol, without Provitamins)	1	µg/100g	13	47	14.04.2020	40750	40500	41000		no	
	2	µg/100g	15	45	13.03.20	45950	46900	45000	500		
	3	µg/100g	28	32	16.03.20	47614,96	48323,28	46906,63	4	no	-
	4	µg/100g	23	37	27.03.20	48350	48900	47800	50	no	96
	5	µg/100g	3	57		47155	47370	46940		no	
	6	µg/100g									
	7	µg/100g	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
	8	µg/100g	17	43	02.04.20	55883	55959,4	55806,2	-	no	-
	9	µg/100g	5	55		30,4	29,4	31,3		no	80,9
	10	µg/100g	46100	46900	01.03.20					no	
	11	µg/100g	24	7	28.04.20	59100	58600	59500	23000	no	0,99
	12	µg/100g									
	13	µg/100g	1	59		45750,4	46006,3	45494,5			
	14	µg/100g	12	48	13.05.20	5407,59	5078,58	5736,6	50	yes	
	15	µg/100g			16/4	7025			70	no	
	16	µg/100g	22	38	05.05	54050	54100	54000	10	no	
	17	µg/100g	34	26	31.03.20	48000	48300	47700		no	
	18	µg/100g	51-A	51-B	28.04.20	69800	66700	72800	1000	no	
	19	µg/100g	25	35	01.04.20	47550	47710	47390	3000	no	
	20	µg/100g	20	40	31. Mrz	54750	65200	44300			

Parameter	Participant	Unit	Sample I DLA No.	Sample II DLA No.	Date of analysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quanti-fication	Incl. RR	Recovery rate
					Day/Month					yes / no	in %
Vitamin D3 (berechnet als Cholecalciferol) / Vitamin D3 (calculated as Cholecalciferol)	1	µg/100g	13	47	11.03.2020	227,63	256,86	198,41	0,5	yes	97
	2	µg/100g	15	45	13.03.20	548	563	533	0,05		
	3	µg/100g	28	32	25.03.20	546,64	541,74	551,54	0,3	no	-
	4	µg/100g	23	37	31.03.20	585	580	590	1	no	
	5	µg/100g	3	57		620000	581000	659000		no	
	6	µg/100g									
	7	µg/100g	11	49	16.04.20	482	480	484	N/A	no	N/A
	8	µg/100g	17	43	02.04.20	210,71	212,5	209	-	no	-
	9	µg/100g									
	10	µg/100g	572	564	31.03.20	568				no	
	11	µg/100g									
	12	µg/100g									
	13	µg/100g	1	59		559	546,8	571,1			
	14	µg/100g									
	15	µg/100g			15/4	350			16	no	
	16	µg/100g	22	38	05.05	550	565	535		no	99
	17	µg/100g	34	26	16.03.20	564	565	563		no	
	18	µg/100g	51-A	51-B	28.04.20	755	773	737	60	no	
	19	µg/100g	25	35	01.04.20	570	580	560	400	no	
	20	µg/100g	20	40	31. Mrz	520	520	520			

Parameter	Participant	Unit	Sample I DLA No.	Sample II DLA No.	Date of analysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quanti-fication	Incl. RR	Recovery rate
					Day/Month					yes / no	in %
Vitamin E (berechnet als D-α-Tocopherol) / Vitamin E (calculated as D-α-Tocopherol)	1	mg/100g	13	47	14.04.2020	284,5	286	283		no	
	2	mg/100g	15	45	13.03.20	190	210	170	0,5		
	3	mg/100g	28	32	06.04.20	270,95	267,79	274,1	0,45	no	-
	4	mg/100g	23	37	27.03.20	280	276	283	0,05	no	103
	5	mg/100g	3	57		208	211,3	204,6		no	
	6	mg/100g									
	7	mg/100g	11	49	16.04.20	324,5	328	321	N/A	no	N/A
	8	mg/100g	17	43	02.04.20	0,526	0,498	0,553	-	no	-
	9	mg/100g	5	55		234,1	229	239,2		no	80,9
	10	mg/100g	262	267	31.03.20	265				no	
	11	mg/100g	24	7	28.04.	400	410	390	88	no	1,06
	12	mg/100g	18	42	13.05.20	142,31	142,6	142,1	5 mg/kg		
	13	mg/100g	1	59		230,8	227,4	234,2			
	14	mg/100g	12	48	14.05.20	90,72	87,07	94,38	0,05	yes	
	15	mg/100g			16/4	162,9			41,7	no	
	16	mg/100g	22	38	05.05	284000	285000	283000		no	
	17	mg/100g	34	26	17.03.20	313	309	317		no	
	18	mg/100g	51-A	51-B	28.04.20	239	224	253	2	no	
	19	mg/100g	25	35	01.04.20	197	199	195	63	no	
	20	mg/100g	20	40	31. Mrz	273,5	257	290			

Parameter	Participant	Unit	Sample I DLA No.	Sample II DLA No.	Date of analysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quanti-fication	Incl. RR	Recovery rate
					Day/Month					yes / no	in %
Vitamin K1 (berechnet als Phyllochinon) / Vitamin K1 (calculated as Phylloquinone)	1	µg/100g									
	2	µg/100g	15	45	13.03.20	1023	985	1060	0,1		
	3	µg/100g	28	32	06.04.20	705,55	698,64	712,46	2	no	-
	4	µg/100g	23	37	16.04.20	710	735	690	70	no	
	5	µg/100g									
	6	µg/100g									
	7	µg/100g	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
	8	µg/100g	17	43	02.04.20	2916,1	2958,2	2874,1	-	no	-
	9	µg/100g	5	55		110,5	106,7	114,2		no	
	10	µg/100g									
	11	µg/100g									
	12	µg/100g									
	13	µg/100g	1	59		1178,5	1163	1194			
	14	µg/100g									
	15	µg/100g									
	16	µg/100g	22	38	05.05	1055	1045	1065		no	106
	17	µg/100g									
	18	µg/100g	51-A	51-B	28.04.20	1,07	1,06	1,07	0,2	no	
	19	µg/100g	25	35	01.04.20	1945	1930	1960	3000	no	
	20	µg/100g	20	40	31. Mrz	976,5	965	988			

5.1.2 Analytical Methods

Parameter	Participant	Method description as in test report / norm / literature	Sample preparation and processing	Measuring method	Calibration / Reference material	Recovery rate with same matrix	Method accredited ISO/IEC 17025	Further Remarks
						yes / no	yes / no	
Alpha Liponsäure / Alpha Liponic Acid	1							
	2	SOP M3495, in house method, HPLC-UV					no	
	3							
	4							
	5							
	6	In house method- QCL686	Liquid extraction	UPLC	USP	N/A	Yes	
	7	N/A	N/A	N/A	N/A	N/A	N/A	N/A
	8	in house method	Liquid extraction	HPLC-DAD	-	-	no	-
	9							
	10							
	11							
	12							
	13							
	14							
	15							
	16	In-house method			Alfa Lipoic acid - Sigma		No	
	17							
	18							
	19							
	20	In house developed method, UPLC platform	Sonication in diluent.		External cal curve, Sigma Aldrich	No	No	

Parameter	Participant	Method description as in test report / norm / literature	Sample preparation and processing	Measuring method	Calibration / Reference material	Recovery rate with same matrix	Method accredited ISO/IEC 17025	Further Remarks
						yes / no	yes / no	
β -Carotin (berechnet als β-Carotin, ohne andere Provitamine) / β-Carotene (calculated as β-Carotene, without other Provitamins)	1							
	2	SOP M932, in house method, HPLC-UV					yes	
	3	PV-196-Coenzyme Q10 (HPLC) : 2013-06 (a)	no	no	external calibration, RM material ok	-	yes	-
	4	EVS-EN 12823-2	extraction dichloromethane/hexane	HPLC-DAD	Fluka22040		yes	
	5							
	6							
	7	MQLTM-0101A By HPLC	N/A	N/A	N/A	N/A	Yes	N/A
	8	in house method	Liquid extraction	HPLC-DAD	-	-	no	-
	9							
	10							
	11	03-32-MAA-M-CaroA, 08-2015					yes	
	12							
	13							
	14							
	15							
	16	In-house method					No	
	17	In house developed by HPLC					Yes	
	18	LCMSMS						
	19							
	20							

Parameter	Participant	Method description as in test report / norm / literature	Sample preparation and processing	Measuring method	Calibration / Reference material	Recovery rate with same matrix	Method accredited ISO/IEC 17025	Further Remarks
						yes / no	yes / no	
Coenzym Q10 (Ubiquinon) / Coenzyme Q10 (Ubiquinone)	1							
	2	SOP M849, in house method, HPLC-UV					yes	
	3	DIN EN 12823-2 : 2007-07 (a)	no	no	external calibration, RM material ok	-	yes	-
	4							
	5							
	6							
	7	N/A	N/A	N/A	N/A	N/A	N/A	N/A
	8	in house method	Liquid extraction	HPLC-DAD	-	-	no	-
	9							
	10							
	11	03-32-MAA-M-Q10, 08-2015					yes	
	12	Determination of ubiquinone (coenzyme 10) in food supplements	Fl/Fl extr. with isooctane		Sigma Q10 98% HPLC		yes	
	13							
	14							
	15	HPLC, AOAC Official Method 2008.07				No	No	
	16	In-house method			Coenzima Q10- Sigma		No	
	17							
	18							
	19							
	20	In house developed method, UPLC platform	Sonication in diluent.		External cal curve, Sigma Aldrich	No	No	

Parameter	Participant	Method description as in test report / norm / literature	Sample preparation and processing	Measuring method	Calibration / Reference material	Recovery rate with same matrix	Method accredited ISO/IEC 17025	Further Remarks
						yes / no	yes / no	
Vitamin A (berechnet als Retinol ohne Provitamine) / Vitamin A (calculated as Retinol, without Provitamins)	1	QSA	Alkaline saponification	HPLC-FLD				
	2	SOP M840, in house method, HPLC-UV					yes	Determined retinyl acetate and calculated with a factor of 0.872 retinol
	3	DIN EN 12823-1 : 2014-08 (a)	no	no	external calibration, RM material ok	-	yes	-
	4	EVS-EN12823-1:2014	saponification with KOH, SPE	HPLC-FLD	Sigma R7632/muva kempten	no	yes	
	5	ASU L 00.00-63/1 2015-05					yes	
	6							
	7	N/A	N/A	N/A	N/A	N/A	N/A	N/A
	8	in house method	Liquid extraction	HPLC-DAD	-	-	no	-
	9	HPLC (ASU L 00.00-62 and -63/1 : 2015-06, modified)				no	yes	
	10							
	11	03-32-MAA-A-VitAE, 02-2020					yes	
	12							
	13			HPLC			yes	
	14			HPLC		yes		
	15	HPLC, HRN EN 12823-1:2014				No	No	
	16	In-house method					Yes	
	17	In house developed by HPLC					Yes	
	18	LCMSMS						
	19	in house method		HPLC-DAD			yes	
	20	In house developed method, UPLC platform	Sonication in diluent.		External cal curve, Sigma Aldrich	No	No	

Parameter	Participant	Method description as in test report / norm / literature	Sample preparation and processing	Measuring method	Calibration / Reference material	Recovery rate with same matrix	Method accredited ISO/IEC 17025	Further Remarks
						yes / no	yes / no	
Vitamin D3 (berechnet als Cholecalciferol) / Vitamin D3 (calculated as Cholecalciferol)	1	QSA	Samples were saponified, extracted with isooctane and then derivatized	LC-MS/MS	Calibration range: 10-1000 ng/ml; Reference material: SRM 1849a	yes	no	
	2	SOP M2885, in house method, LC-MS/MS					yes	
	3	DIN EN 12821 : 2009-08 (a)	no	no	external calibration, RM material ok	-	yes	-
	4	NMKL 167	saponification with KOH, liquid/liquid extr.	HPLC-DAD	Cholecaliferol/ Calciferol		yes	
	5	VDLUFA method book Volume III, 13.8.1 4.Erg. 1997					yes	
	6							
	7	MQLTM-0508 By HPLC	N/A	N/A	N/A	N/A	Yes	N/A
	8	in house method	Liquid extraction	HPLC-DAD	-	-	no	-
	9							
	10							
	11							
	12							
	13			HPLC			yes	
	14							
	15	HPLC, HRN ISO 14892:2003				No	No	
	16	In-house method			Reference Material Vit D3, Internal Standard Vit D2	Yes	Yes	
	17	In house developed by HPLC					Yes	
	18	LCMSMS						
	19	in house method		HPLC-DAD			yes	
	20	In house developed method, LC-MS/MS platform	Sonication in diluent.		External cal curve, Sigma Aldrich	No	No	

Parameter	Participant	Method description as in test report / norm / literature	Sample preparation and processing	Measuring method	Calibration / Reference material	Recovery rate with same matrix	Method accredited ISO/IEC 17025	Further Remarks
						yes / no	yes / no	
Vitamin E (berechnet als D-α-Tocopherol)/ Vitamin E (calculated as D-α-Tocopherol)	1	QSA	Alkaline saponification	HPLC-FLD				
	2	SOP M840, in house method, HPLC-UV					yes	Determined alpha-tocopherol acetate and calculated alpha-tocopherol with factor 0.671
	3	DIN EN 12822 : 2014-08 (a)	no	no	external calibration, RM material ok	-	yes	-
	4	EVS-EN12822:2014	saponification with KOH, SPE	HPLC-FLD	Calbiochem 613424/muva kempten	no	yes	
	5	ASU L 00.00-62 2015-06					yes	
	6							
	7	MQLTM-0100 By HPLC	N/A	N/A	N/A	N/A	Yes	reported as dl-alpha tocopheryl acetate
	8	in house method	Liquid extraction	HPLC-DAD	-	-	yes	-
	9	HPLC (ASU L 00.00-62 and -63/1 : 2015-06, modified)				no	yes	
	10							
	11	03-32-MAA-A-VitAE, 02-2020					yes	Only alpha tocopherol was evaluated; the sample also contains gamma and delta tocopherol
	12	Determination of tocopherols (vitamin E)	FI/FI extr. with isooctane		Merk Tocopherol Set		yes	
	13			HPLC			yes	
	14			HPLC		yes		
	15	HRN EN 12822:2014, modified P-DPBAT-09 Issue: 1/1				No	Yes	
	16	In-house method					Yes	
	17	In house developed by HPLC					Yes	
	18	LCMSMS						
	19	in house method		HPLC-DAD			yes	
	20	In house developed method, UPLC platform	Sonication in diluent.		External cal curve, Sigma Aldrich	No	No	

Parameter	Participant	Method description as in test report / norm / literature	Sample preparation and processing	Measuring method	Calibration / Reference material	Recovery rate with same matrix	Method accredited ISO/IEC 17025	Further Remarks
						yes / no	yes / no	
Vitamin K1 (berechnet als Phyllochinon)/ Vitamin K1 (calculated as Phylloquinone)	1							
	2	SOP M2986, in house method, HPLC-FLD					yes	
	3	DIN EN 14148 : 2003-10 (a)	no	no	external calibration, RM material ok	-	yes	-
	4	EVS-EN 14148	lipase, post-column der.	HPLC-FLD	Sigma95271		no	
	5							
	6							
	7	N/A	N/A	N/A	N/A	N/A	N/A	N/A
	8	in house method	Liquid extraction	HPLC-DAD	-	-	no	-
	9	HPLC (ASU L 00.00-86 : 2004-07)					yes	
	10							
	11							
	12							
	13			HPLC			yes	
	14							
	15							
	16	In-house method			Vitamina K1	Yes	No	
	17							
	18	LCMSMS						
	19	in house method		HPLC-DAD			yes	
	20	In house developed method, LC-MS/MS platform	Sonication in diluent.		External cal curve, Sigma Aldrich	No	No	

5.2 Homogeneity

5.2.1 Mixture homogeneity before bottling

Microtracer Homogeneity Test
DLA ptSU02

Weight whole sample	3,12	kg
Microtracer	FSS-rot lake	
Particle size	75 – 300	µm
Weight per particle	2,0	µg
Addition of tracer	21,9	mg/kg

Result of analysis

Sample	Weight [g]	Particle number	Particles [mg/kg]
1	5,04	70	27,8
2	5,04	68	27,0
3	5,03	60	23,9
4	5,00	62	24,8
5	4,99	67	26,9
6	5,05	71	28,1
7	4,98	68	27,3
8	4,98	74	29,7

Poisson distribution		
Number of samples	8	
Degree of freedom	7	
Mean	67,5	Particles
Standard deviation	4,65	Particles
χ^2 (CHI-Quadrat)	2,24	
Probability	**95**	%
Recovery rate	123	%

Normal distribution		
Number of samples	8	
Mean	26,9	mg/kg
Standard deviation	1,85	mg/kg
rel. Standard deviaton	6,88	%
Horwitz standard deviation	9,75	%
HorRat-value	**0,71**	
Recovery rate	123	%

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:

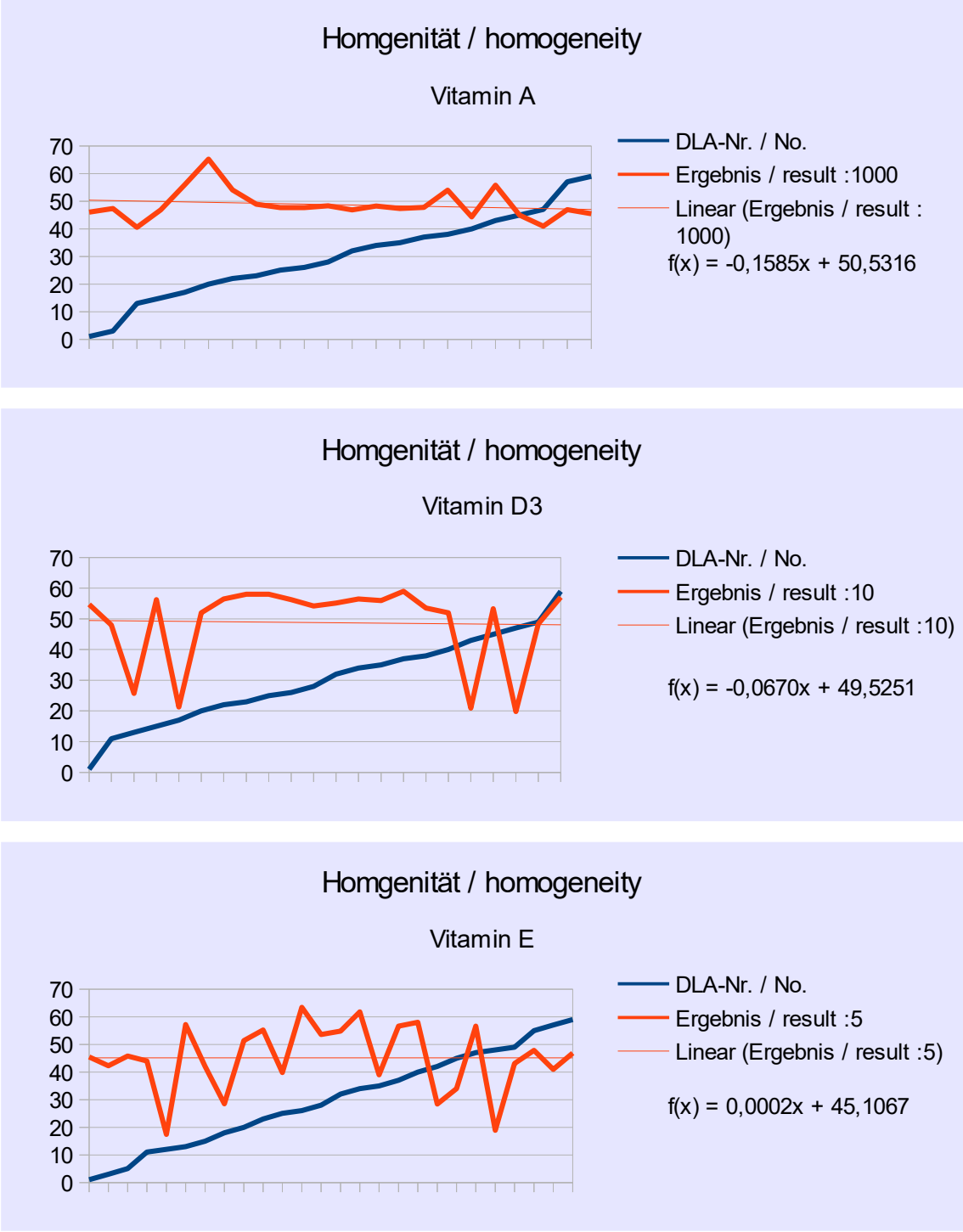


Abb./Fig. 15: Trendfunktion Probennummern vs. Ergebnisse: Vitamin A, D3 und E (1/1000, 1/10 und 1/5 dargestellt)
trend line function sample number vs. results: vitamin A, D3 and E (1/1000, 1/10 and 1/5 shown)

5.3 Kernel Density Plots of Results

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

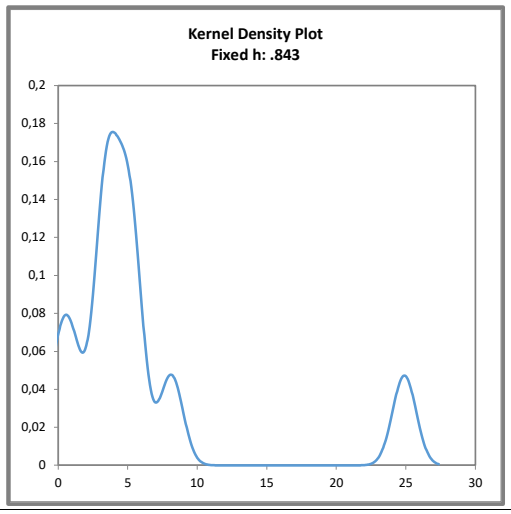
Figures:
Kernel density plots
of participants' results
(with h = 0,75 x σ_{pt} of Xpt)

Alpha-Liponsäure / Alpha Liponic Acid

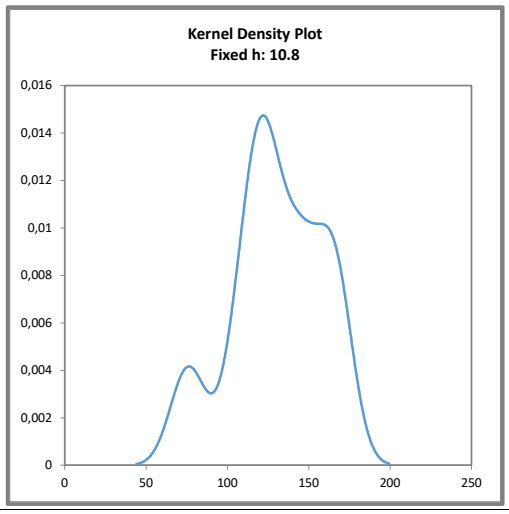
< 8 Ergebnisse

< 8 Results

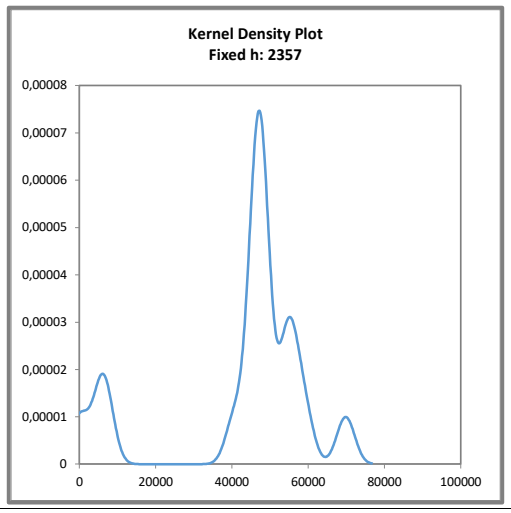
Beta-Carotin / Beta-Carotene



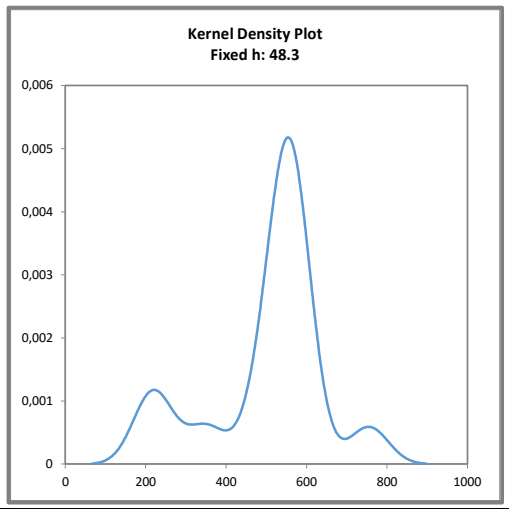
Coenzym Q10 / Coenzyme Q10



Vitamin A



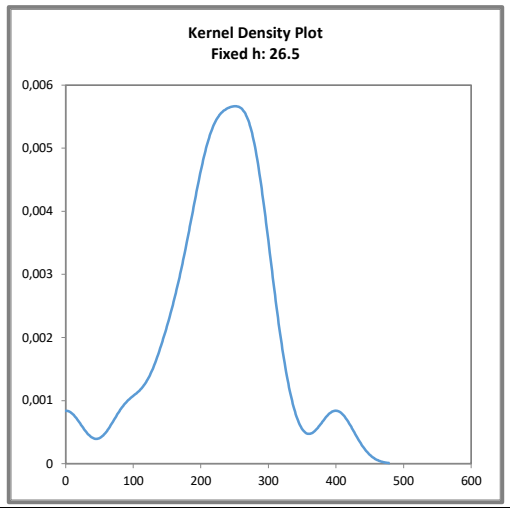
Vitamin D3



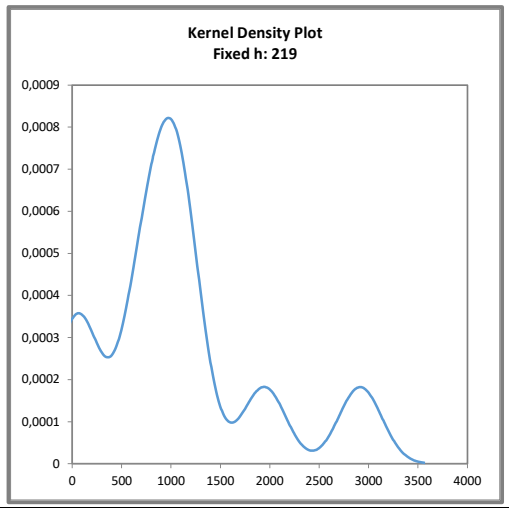
Abbildungen:
Kerndichte-Schätzungen
der Teilnehmerergebnisse
(mit h = 0,75 x σ_{pt} von X_{pt})

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

Vitamin E



Vitamin K1



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 ptSU02***
PT name	***Food Supplement I: Vitamins A, E, D3, K1, β-Carotene and Coenzym Q10 (Ubiquinone) and Alpha Liponic Acid***
*Sample matrix**	*Samples I + II: Capsule powder (without capsule shells) / Ingredients: Maltodextrin, further food additives, vitamins and minerals*
Number of samples and sample amount	*2 identical samples I + II, 50 g each.*
Storage	*Samples I + II: 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 Coenzym 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. 0.2 - 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 or 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 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 April 17th 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
		GREAT BRITAIN
		Germany
		Germany
		Germany
		Germany
		CROATIA
		USA
		Germany
		USA
		Germany
		Germany
		Germany
		BELGIUM
		Germany
		USA
		ITALY
		Germany
		ESTONIA
		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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7. The International Harmonised Protocol for the Proficiency Testing of Ananlytical Laboratories ; J.AOAC Int., 76(4), 926 – 940 (1993)
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14. GMP+ Feed Certification scheme, Module: Feed Safety Assurance, chapter 5.7 Checking procedure for the process accuracy of compound feed with micro tracers in GMP+ BA2 Control of residues, Version: 1st of January 2015 GMP+ International B.V.
15. MTSE SOP No. 010.01 (2014): Quantitative measurement of mixing uniformity and carry-over in powder mixtures with the rotary detector technique, MTSE Micro Tracers Services Europe GmbH
16. Homogeneity and stability of reference materials; Linsinger et al.; Accred Qual Assur, 6, 20-25 (2001)
17. AOAC Official Methods of Analysis: Guidelines for Standard Method Performance Requirements, Appendix F, p. 2, AOAC Int (2016)
18. Andersson (1992) Determination of coenzyme Q by non-aqueous reversed- phase liquid chromatography. J Chromatogr. 606(2):272-6
19. Strazisar et al. (2005) Quantitative determination of coenyzme Q10 by liquid chromatography and liquid chromatography/mass spectrometry in dairy products. J AOAC Int. 88(4):1020-7
20. Orozco et al. (2007) Determination of ubidecarenone (coenzyme Q10, ubiquinol-10) in raw materials and dietary supplements by high-performance liquid chromatography with ultraviolet detection: single-laboratory validation. J AOAC Int. 90(5):1227-36
21. ASU § 64 LFGB L 00.00-61 / DIN EN 12821:2009 [21b: 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 Le-

bensmitteln 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
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