Evaluation Report
proficiency test

DLA ptCO03 (2021)

Cosmetic Products III:

Coenzyme Q10, Panthenol and Tocopherol

in Skin Cream

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

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EP-Bericht Freigabe *PT-Report Authorization*	Dr. Matthias Besler-Scharf (Technischer Leiter / Technical Manager) *- gezeichnet / signed M. Besler-Scharf* Alexandra Scharf MSc. (QM-Beauftragte / Quality Manager) *- gezeichnet / signed A. Scharf* Datum / Date: 14 March 2022
Unteraufträge *Subcontractors*	Im Rahmen dieser Eignungsprüfung wurden nachstehende Leistungen im Unterauftrag vergeben: Keine As part of the present proficency test the following services were subcontracted: none
Vertraulichkeit *Confidentiality*	Die Teilnehmerergebnisse sind im EP-Bericht in anonymisierter Form mit Auswertenummern benannt. Daten einzelner Teilnehmer werden ausschließlich nach vorheriger Zustimmung des Teilnehmers an Dritte weitergegeben. Participant result are named anonymously with evaluation numbers in the PT report. Data of individual participants will be passed on to third parties only with prior consent of the participant.

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1. Introduction

The participation in proficiency testing schemes (PT) 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 commercially available body lotions and a hand cream from European suppliers.

The materials were mixed and homogenized. The composition of the PT samples (list of ingredients) is shown in table 1.

Afterwards, the samples were portioned to approximately 25 g into 28 ml plastic containers, sealed in metallised PET film bags and chronologically numbered.

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Table 1: Composition of DLA-Samples

PT-Samples Skin Cream
Skin cream / Body lotion 1 Ingredients: Aqua, Glycerin, Isopropyl Palmitate, Alcohol Denat., Glyceryl Stearate SE, Glyceryl Stearate, C12-15 Alkyl Benzoate, Ubiquinone, Sodium Ascorbyl Phosphate, Butyrospermum Parkii Butter, Dimethicone, Sodium Cetearyl Sulfate, Carbomer, Sodium Hydroxide, Trisodium EDTA, Phenoxyethanol, Ethylhexylglycerin, Linalool, Citronellol, Benzyl Alcohol, Alpha-Isomethyl Ionone, Limonene, Parfum
Skin cream / Body lotion 2 Ingredients: Aqua, Glycerin, Helianthus Annuus Hybrid Oil, Cetearyl Alcohol, Glyceryl Stearate, Sorbitol, Butyrospermum Parkii Butter, Ethylhexyl Stearate, Isopropyl Palmitate, Ubiquinone, Parfum, Sodium Cetearyl Sulfate, Xanthan Gum, Phenoxyethanol, Benzyl Alcohol, Linalool, Hexyl Cinnamal, Limonene, Citral, Citronellol, Sodium Chloride, Citric Acid, Sodium Hydroxide
Skin cream / Body lotion 3 Ingredients: Aqua, Ethylhexyl Stearate, Glycerin, Caprylic Triglyceride, Prunus Armeniaca Kernel Oil, Glyceryl Stearate Citrate, Phenoxyethanol, Panthenol, Cetearyl Alcohol, Carbomer, Parfum, Butyrospermum Parkii Butter, Tocopheryl Acetate, Sodium Hydroxide, Ethylhexylglycerin
Skin cream / Hand cream Ingredients: Aqua, Isopropyl Palmitate, Glycerin, Glyceryl Stearate SE, Cetyl Alcohol, Panthenol, Butyrospermum Parkii Butter, Octyldodecanol, Vitis Vinifera Seed Oil, Glycine Soja Oil, Tocopheryl Acetate, Sodium Cetearyl Sulfate, Sodium Acrylates/C10-30 Alkyl Acrylate Crosspolymer, Phenoxyethanol, Methylparaben, Ethylparaben, Alpha-Isomethyl Ionone, Citronellol, Geraniol, Linalool, Limonene, Benzyl Alcohol, Parfum

Note: *The metrological traceability of temperature, mass and volume during production of the PT samples is ensured by DAkkS calibrated reference materials.*

2.1.1 Homogeneity

The calculation of the **repeatability standard deviation S_r of the participants' double determination** was used as an indicator of homogeneity. The repeatability standard deviation was in the range of 0,81% to 4,78% (see Tab. 2) and thus in the normal to lower range of comparable methods. The repeatability standard deviations of the participants' results are given in the documentation in the statistic data (see 4.1 to 4.3).

Table 2: Repeatability standard deviation S_r of double determinations of the participants (coefficient of variation CV_r in %)

Parameter	**CV_r**
Coenzyme Q10	4,78 %
Panthenol	0,81 %
DL-alpha-Tocopheryl Acetate	3,17 %

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 Homogeneity).

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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

Experience has shown that unopened preserved skin creams are stable for several years. For the products, the manufacturers gave a shelf life of 6 or 12 months after opening. 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 48th week of 2021. The testing method was optional. The tests should be finished at 28 January 2022 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 mixture of common in commerce skin creams with the parameters Coenzym Q10 (Ubiquinone), Panthenol and Tocopherol (Tocopheryl acetate) to be determined.

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

Please note the attached information on the proficiency test.
(see documentation, section 5.3 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.

Of 10 participants, 9 submitted their results.
1 participant did not submit any results.

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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.

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3.4 Reproducibility standard deviation

The reproducibility standard deviation S_R represents an 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 PTs 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, if single results are available. 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.

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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.

To evaluate the results, the target standard deviation according to the general model of Horwitz (see 3.6.1) was used for coenzyme Q10, panthenol and tocopherol (calculated as DL-alpha-tocopheryl acetate) in the present PT.

Additionally, for DL-alpha-tocopheryl acetate 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)

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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]:

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

For the determination of coenzyme Q10, panthenol and tocopherol in cosmetic products to our knowledge currently there are no sufficient data available on relative repeatability standard deviations (RSD_r) and relative reproducibility standard deviations (RSD_R) from interlaboratory comparisons or ring trials.

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.

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}).

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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].

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 ($u_{(x_{pt})}$) [3].

The calculation is performed by:

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

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

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

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

For warning and action signals see 3.7.1.

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

3.10 Quotient S*/σ_{pt}

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

3.11 Standard uncertainty of the assigned value

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

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

If $u_{(x_{pt})} \leq 0{,}3\ \sigma_{pt}$, 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.

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4. 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}'
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**	**Hinweis** **Remark**

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

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4.1 Coenzyme Q10 (Ubiquinone) in mg/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	8
Number of outliers	0
Mean	6,70
Median	6,60
Robust Mean (X_{pt})	**6,66**
Robust standard deviation (S*)	**0,698**
Number with 2 replicates	8
Repeatability SD (S_r)	0,320
Repeatability (CV_r)	4,78%
Reproducibility SD (S_R)	0,742
Reproducibility (CV_R)	11,1%
Target range:	
Target standard deviation σ_{pt}	**0,566**
lower limit of target range	**5,52**
upper limit of target range	**7,79**
Quotient S/σ_{pt}*	*1,2*
Standard uncertainty $U_{(Xpt)}$	*0,309*
Results in the target range	*7*
Percent in the target range	*88%*

Comments to the statistic data:

The target standard deviation was calculated according to the general model of Horwitz (s. 3.6.1).

The distribution of the results showed a normal variability, with a quotient S/σ_{pt} below 2,0. The comparability of results is given.*

88% of results were in the target range.

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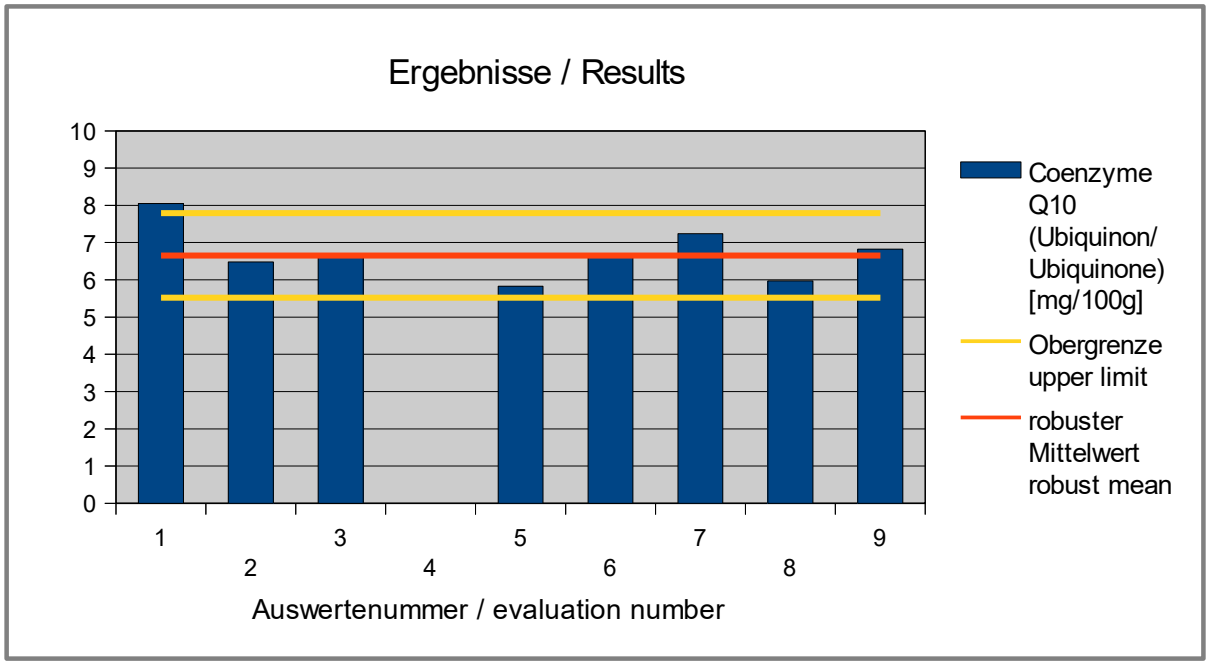

Abb. / Fig. 1: Ergebnisse Coenzym Q10 (Ubiquinon)/ Results Coenzyme Q10 (Ubiquinone)

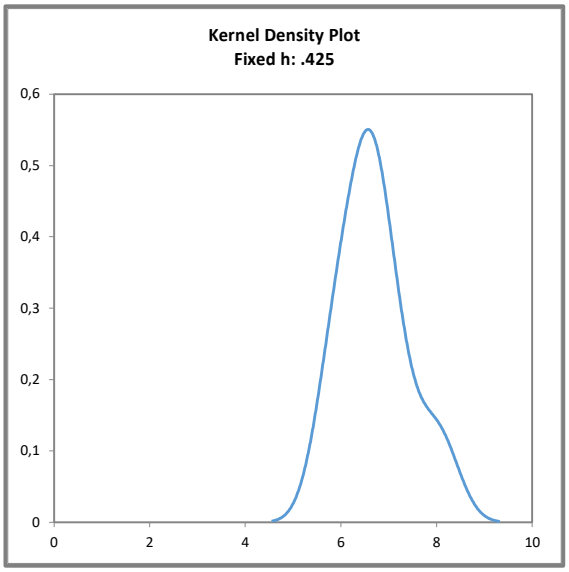

Abb. / Fig. 2:
Kerndichte-Schätzung der Ergebnisse (mit h = 0,75 x σ_{pt} von Xpt)

Kernel density plot of results (with h = 0,75 x σ_{pt} of Xpt)

Comment:
The kernel density estimation shows an approximately symmetrical distribution of results with a shoulder at approximately 8 mg/100g due to a participant result outside the target range.

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Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Coenzyme Q10 (Ubiquinon/ Ubiquinone) [mg/100g]	Abweichung [mg/100g] Deviation [mg/100g]	z-Score (σ_{pt})	Hinweis Remark
1	8,05 *	1,39	2,5	
2	6,48	-0,175	-0,31	
3	6,60	-0,055	-0,10	
4				
5	5,83	-0,825	-1,5	
6	6,60	-0,055	-0,10	
7	7,24	0,585	1,0	
8	5,97	-0,685	-1,2	
9	6,82	0,165	0,29	

* Mean calculated by DLA

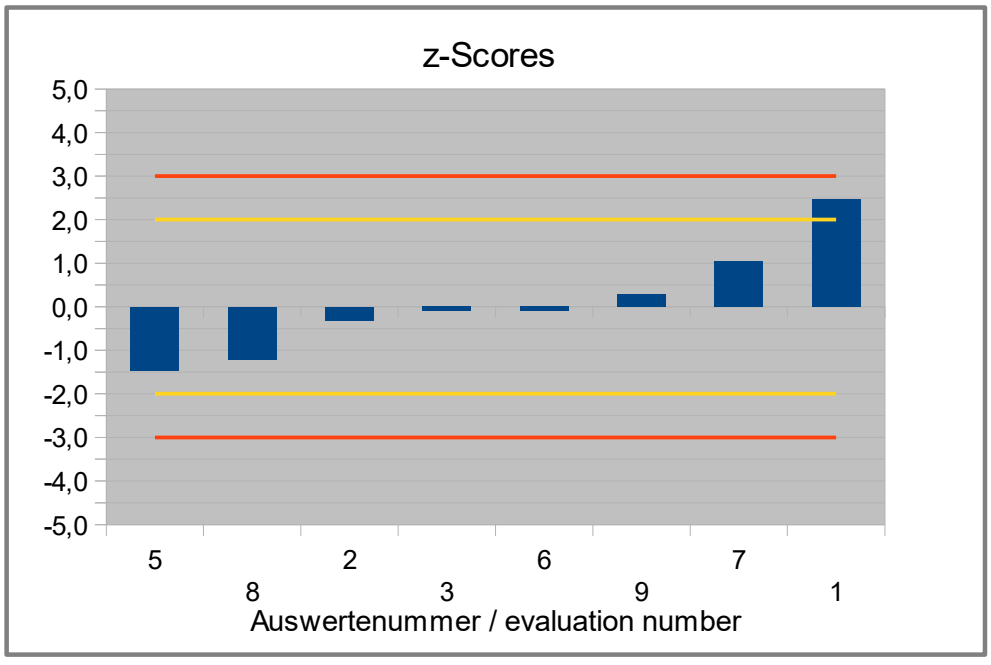

Abb. / Fig. 3: z-Scores Coenzyme Q10 (Ubiquinon / Ubiquinone)

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4.2 Panthenol in mg/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	7
Number of outliers	-
Mean	441
Median	403
Robust Mean (X_{pt})	**399**
Robust standard deviation (S*)	**20,5**
Number with 2 replicates	6
Repeatability SD (S_r)	3,18
Repeatability (CV_r)	0,807%
Reproducibility SD (S_R)	13,3
Reproducibility (CV_R)	3,37%
Target range:	
Target standard deviation σ_{pt}	**18,3**
lower limit of target range	**362**
upper limit of target range	**436**
Quotient S/σ_{pt}*	*1,1*
Standard uncertainty $U_{(Xpt)}$	*9,70*
Results in the target range	*6*
Percent in the target range	*86%*

Comments to the statistic data:

The target standard deviation was calculated according to the general model of Horwitz (s. 3.6.1).

The distribution of the results showed a normal variability, with a quotient S/σ_{pt} below 2,0. The comparability of results is given.*

86% of results were in the target range.

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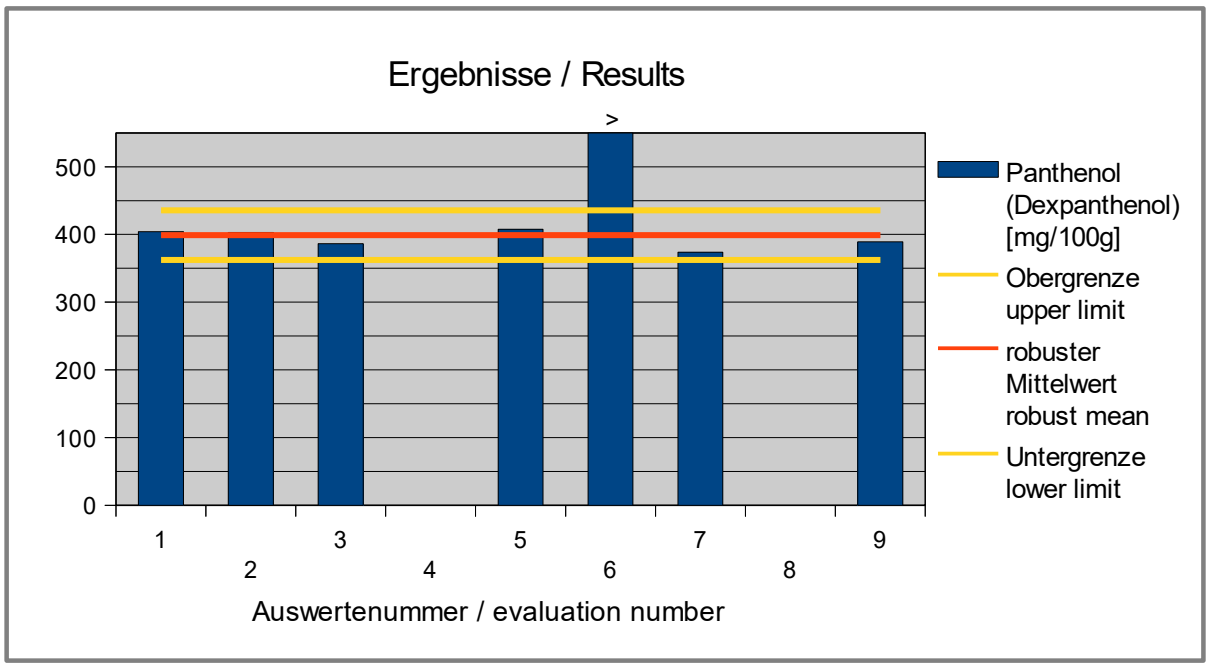

Abb. / Fig. 4: Ergebnisse / Results Panthenol

Comment:
Due to the number of < 8 results, no kernel density estimation was carried out.

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Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Panthenol (Dexpanthenol) [mg/100g]	Abweichung [mg/100g] Deviation [mg/100g]	z-Score (σ_{pt})	Hinweis Remark
1	404 *	4,9	0,27	
2	403	3,9	0,22	
3	387	-12,6	-0,68	
4				
5	408	8,4	0,46	
6	722	323	18	
7	374	-25,5	-1,4	
8				
9	389	-10,1	-0,55	

* Mean calculated by DLA

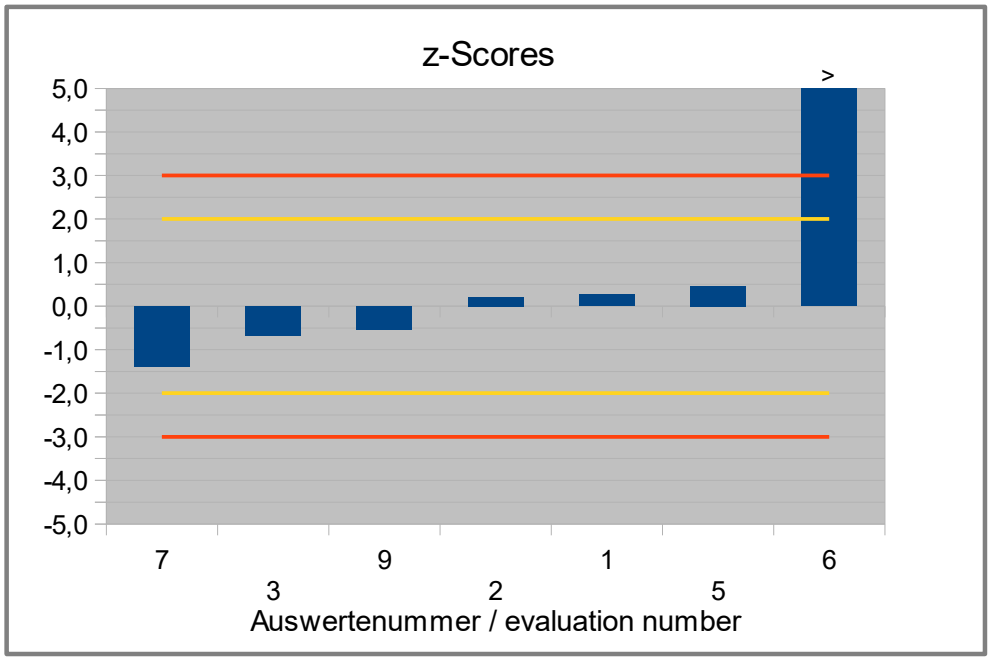

Abb. / Fig. 5: z-Scores Panthenol

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4.3 DL-alpha-Tocopheryl Acetate in mg/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	8
Number of outliers	0
Mean	40,1
Robust Mean	40,2
Median (X_{pt})	**41,9**
Robust standard deviation (S*)	**9,47**
Number with 2 replicates	8
Repeatability SD (S_r)	1,27
Repeatability (CV_r)	3,17%
Reproducibility SD (S_R)	8,51
Reproducibility (CV_R)	21,2%
Target range:	
Target standard deviation σ_{pt}'	**4,98**
lower limit of target range	**31,9**
upper limit of target range	**51,9**
Quotient S^*/σ_{pt}'	*1,9*
Standard uncertainty $U_{(Xpt)}$	*4,19*
Results in the target range	*6*
Percent in the target range	*75%*

Assigned value (Xpt): Median (see 3.1)

Comments to the statistic data:

The target standard deviation was calculated according to the general model of Horwitz (s. 3.6.1).

The distribution of the results showed a slightly increased variability with a quotient $S^*/\sigma_{pt} > 2,0$. Therefore, the z'-score was used for the evaluation, taking into account the standard uncertainty. The quotient S^*/σ_{pt}' was then 1,9.

The evaluation showed a normal variability of results with a quotient S^*/σ_{pt}' below 2,0. The comparability of results is given.

75% of results were in the target range.

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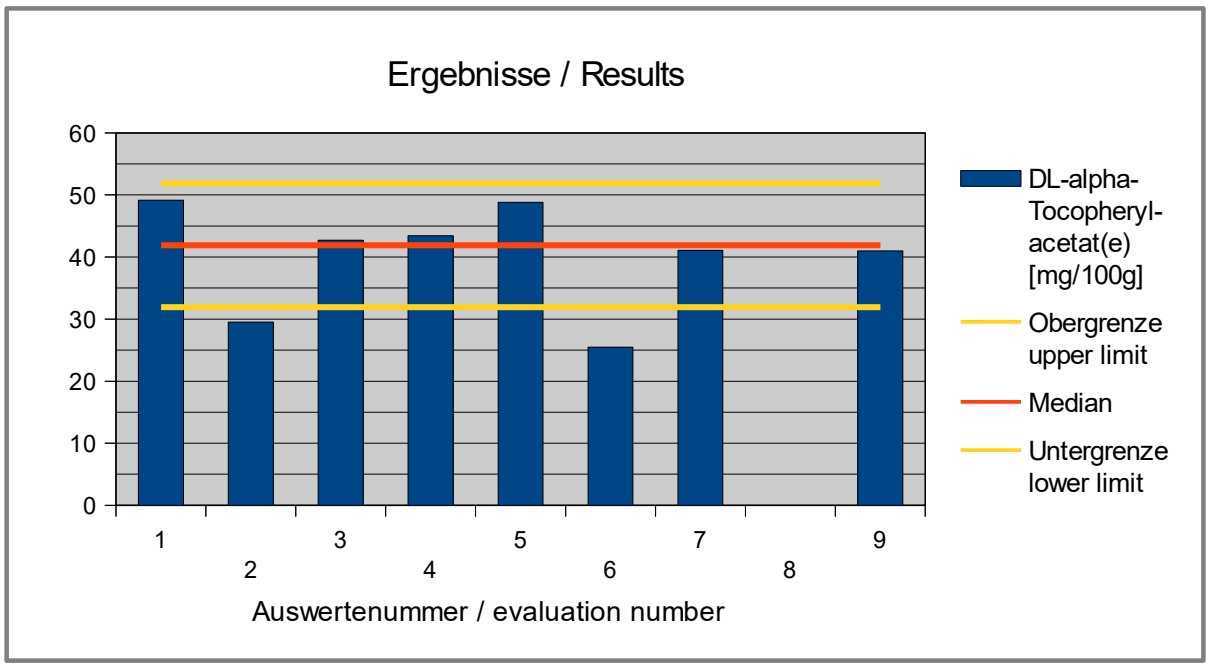

Abb. / Fig. 6: Ergebnisse DL-alpha-Tocopherylacetat / Results DL-alpha-tocopheryl acetate

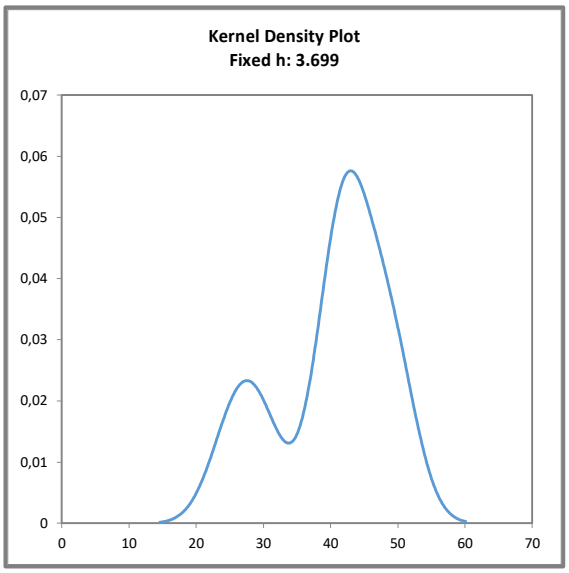

Abb. / Fig. 7:
Kerndichte-Schätzung der Ergebnisse (mit h = 0,75 x σ_{pt} von Xpt)

Kernel density plot of results (with h = 0,75 x σ_{pt} von Xpt)

Comment:
The kernel density estimation shows an approximately symmetrical distribution of results with a secondary peak at approximately 28 mg/100g due to two participant results outside the target range.

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Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	DL-alpha-Tocopheryl-acetat(e) [mg/100g]	Abweichung [mg/100g] Deviation [mg/100g]	z'-Score (σ_{pt})	Hinweis Remark
1	49,2 *	7,25	1,5	
2	29,5	-12,4	-2,5	
3	42,7	0,80	0,16	
4	43,4	1,53	0,31	
5	48,8	6,90	1,4	
6	25,5	-16,4	-3,3	
7	41,1	-0,81	-0,16	
8				
9	41,0	-0,90	-0,18	

* Mittelwert von DLA berechnet

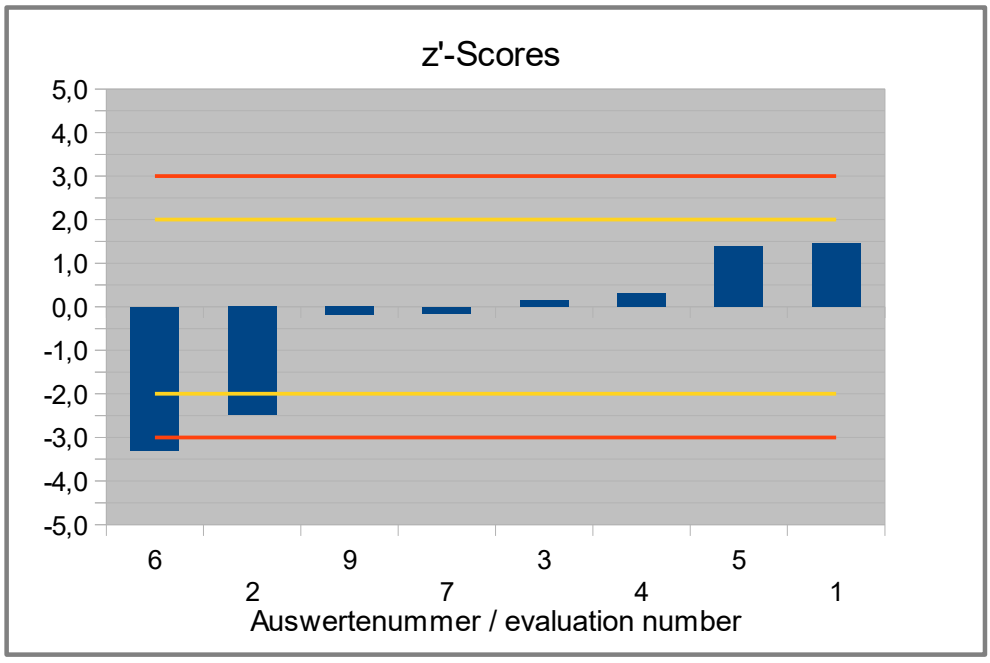

Abb. / Fig. 8: z'-Scores DL-alpha-Tocopherylacetat / DL-alpha-tocopheryl acetate

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4.4 Participants' z-Scores: Overview table

Evaluation number	Coenzyme Q10	Panthenol	D,L-alpha-Tocopheryl Acetate
	z-Score	z-Score	z'-Score
1	2,5	0,27	1,5
2	-0,31	0,22	-2,5
3	-0,10	-0,68	0,16
4			0,31
5	-1,5	0,46	1,4
6	-0,10	18	-3,3
7	1,0	-1,4	-0,16
8	-1,2		
9	0,29	-0,55	-0,18

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)

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5. Documentation

Note: Information given in German were translated by DLA to the best of our knowledge (without guarantee of correctness).

5.1 Details by the participants

5.1.1 Primary data

Parameter	Partici-pant	Unit	Sample I DLA-No	Sample II DLA-No	Date of analysis	Final Result	Result Sample I	Result Sample II	LOQ (Limit of quantifi-cation)	Recovery included	Recovery Rate
					day/month					yes/no	in %
Coenzyme Q10 (Ubi-quinone)	1	mg/100g	30	12	17.12.		8	8,1	1	no	
	2	mg/100g	20	22	04.01.2022	6,48	6,39	6,57	2	no	
	3	mg/100g	5	37	03.01.2022	6,6	6,4	6,8	2	no	-
	4	mg/100g									
	5	mg/100g	3	39	21. Jan	5,83	5,27	6,38	0,1	no	not given
	6	mg/100g	8	34	23 Dec	6,6	6,8	6,4		no	
	7	mg/100g	2	40	05.01.2022	7,24	7,3	7,17	1	no	
	8	mg/100g	4	38	26.01.2022	5,97	6,05	5,88	1 mg/100g	no	100%
	9	mg/100g	ptCO03 No 15	ptCO03 No 27	9.12.	6,82	6,82	6,82	10mg/100g	no	99.8

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Parameter	Partici-pant	Unit	Sample I DLA-No	Sample II DLA-No	Date of analysis	Final Result	Result Sample I	Result Sample II	LOQ (Limit of quantifi-cation)	Recovery included	Recovery Rate
					day/month					yes/no	in %
Panthenol (Dexpan-thenol)	1	mg/100g	30	12	17.12.		403,5	404,4	1	no	
	2	mg/100g	20	22	28.12.2021	403	399	407	50	no	
	3	mg/100g	5	37	22.12.2021	386,5	387,7	385,2	20	no	-
	4	mg/100g									
	5	mg/100g	3	39	13. Jan	407,5	408	407	0,01	no	not given
	6	mg/100g	8	34	21. Jan	721,9	721,2	722,6		no	
	7	mg/100g	2	40	17.12.2021	373,58	373,32	373,84	6	no	
	8	mg/100g									
	9	mg/100g	ptCO03 No 15	ptCO03 No 27	7.12.	389	393	386	<0.5 g/kg	no	96.1

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Parameter	Partici-pant	Unit	Sample I DLA-No	Sample II DLA-No	Date of analysis	Final Result	Result Sample I	Result Sample II	LOQ (Limit of quantifi-cation)	Recovery included	Recovery Rate
					day/month					yes/no	in %
DL-alpha-Tocopheryl Acetate	1	mg/100g	30	12	17.12.		49	49,3	1	no	
	2	mg/100g	20	22	10.01.2022	29,5	30,3	28,7	10	no	
	3	mg/100g	5	37	21.12.2021	42,7	42,5	42,8	25	no	-
	4	mg/100g	32	10	06.01.	43,43	44	42,85	0,06	no	
	5	mg/100g	3	39	18. Jan	48,8	48,2	49,3	1	no	not given
	6	mg/100g	8	34	16 Dec	25,5	26,5	24,5		no	
	7	mg/100g	2	40	11.01.2022	41,09	40,72	41,47	0,9	no	
	8	mg/100g									
	9	mg/100g	ptCO03 No 15	ptCO03 No 27	8.12.	41	39	43	<0.01g/100 g	no	84.3

Parameter	Partici-pant	Unit	Sample I DLA-No	Sample II DLA-No	Date of analysis	Final Result	Result Sample I	Result Sample II	LOQ (Limit of quantifi-cation)	Recovery included	Recovery Rate
					day/month					yes/no	in %
optional: other Toco-pherol com-pounds	1	mg/100g	30	12	17.12.		1,5	1,5	1	no	
	2	mg/100g	20	22	10.01.2022	0,503	0,513	0,492	0,05	no	
	3	mg/100g									
	4	mg/100g									
	5	mg/100g									
	6	mg/100g									
	7	mg/100g	2	40							
	8	mg/100g									
	9	mg/100g									

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5.1.2 Analytical Methods

Parameter	Participant	Method description, like in an analysis report / norm / literature	Notes to sample preparation	Notes to analytical method	Calibration and reference material	Recovery with same matrix	Method accredited ISO/IEC 17025	Further Remarks
						yes / no	yes / no	
Coenzyme Q10 (Ubiquinone)	1	PV-SA-376				no	yes	
	2	In-house method HPLC-DAD	Extraction with 1mL THF, 10 min ultrasonic bath and 3 mL acetone, 10 min ultrasonic bath fill up with MeOH	RP-HPLC DAD	external	no	yes	Usually only acetone is used for extraction. The levels when extracted with THF are clearly higher.
	3	In-house method, HPLC-DAD	-	HPLC-DAD	external calibration	-	yes	
	4							
	5	SOP M 849, HPLC/UV		HPLC/UV in house method	available		no	
	6	HPLC in-house method					no	
	7	M 12.4113.01 (2010-02)		HPLC-DAD			yes	
	8	AOAC, Vol. 90, No. 5, 2007 / J AOAC Int. 2008; 91(4): 702–708	Extraction with acetonitrile/tetrahydrofuran/water	HPLC-DAD	Calibration: Sigma Aldrich C9538	yes (body lotion)	yes	
	9	Determination of the coenzyme Q10 in cosmetic products			five point calibration	no	yes	

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Parameter	Participant	Method description, like in an analysis report / norm / literature	Notes to sample preparation	Notes to analytical method	Calibration and reference material	Recovery with same matrix	Method accredited ISO/IEC 17025	Further Remarks
						yes / no	yes / no	
Panthenol (Dexpanthenol)	1	PV-SA-344				no	yes	
	2	In-house method HPLC-DAD	Extraction with potassium hydrogen phosphate buffer (20 mmol, pH 3)	RP-HPLC DAD	external	no	yes	
	3	In-house method, HPLC-DAD	-	HPLC-DAD	external calibration	-	yes	
	4							
	5	SOP M 3656, LC-MS/MS		LC-MS/MS in-house method	available		no	
	6	HPLC in-house method					no	
	7	M 12.4112.06 (2018-11)		HPLC-DAD			yes	
	8							
	9	Determination of allantoin and panthenol in cosmetic products			five point calibration	no	yes	

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Parameter	Participant	Method description, like in an analysis report / norm / literature	Notes to sample preparation	Notes to analytical method	Calibration and reference material	Recovery with same matrix	Method accredited ISO/IEC 17025	Further Remarks
						yes / no	yes / no	
DL-alpha-Tocopheryl Acetate	1	PV-SA-376				no	yes	
	2	In-house method HPLC-FLD	Extraction with isopropyl	RP-HPLC FLD	external	no	yes	
	3	In-house method, HPLC-DAD	-	HPLC-DAD	external calibration	-	yes	
	4	Determination of fat soluble vitamins (vitamin A, E) by liquid chromatography method with FLD detection		HPLC with FLD				
	5	SOP M 659, HPLC/UV		HPLC/UV in-house method	available		yes	
	6	HPLC in-house method					no	
	7	M 12.3415.01 (2021-07)		HPLC-DAD			yes	
	8							
	9	Determination of vitamins A and E in cosmetic products			five point calibration	no	yes	

Parameter	Participant	Method description, like in an analysis report / norm / literature	Notes to sample preparation	Notes to analytical method	Calibration and reference material	Recovery with same matrix	Method accredited ISO/IEC 17025	Further Remarks
						yes / no	yes / no	
optional: other Tocopherol compounds	1	PV-SA-376				no	yes	
	2	In-house method HPLC-FLD	Extraction with isopropyl	RP-HPLC FLD	external	no	yes	Tocopherol
	3							
	4							
	5							
	6							
	7							
	8							
	9							

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5.2 Homogeneity

5.2.1 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:

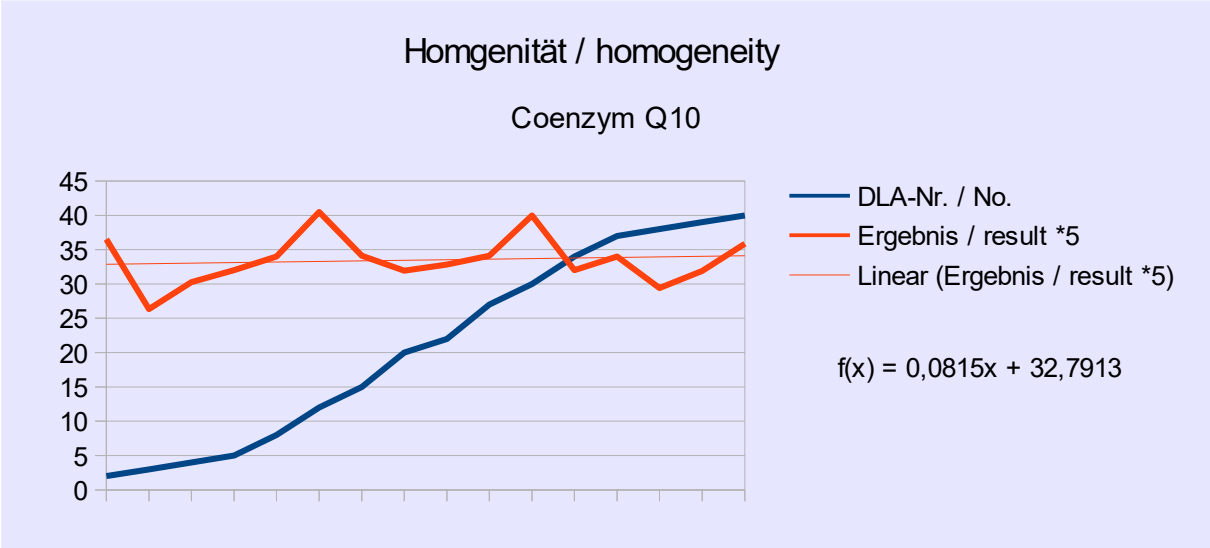

Abb./Fig. 9:
Trendfunktion Probennummern vs. Ergebnisse: Coenzym Q10 (1*5 dargestellt)
trend line function sample number vs. results: Coenzyme Q10(1*5 shown)

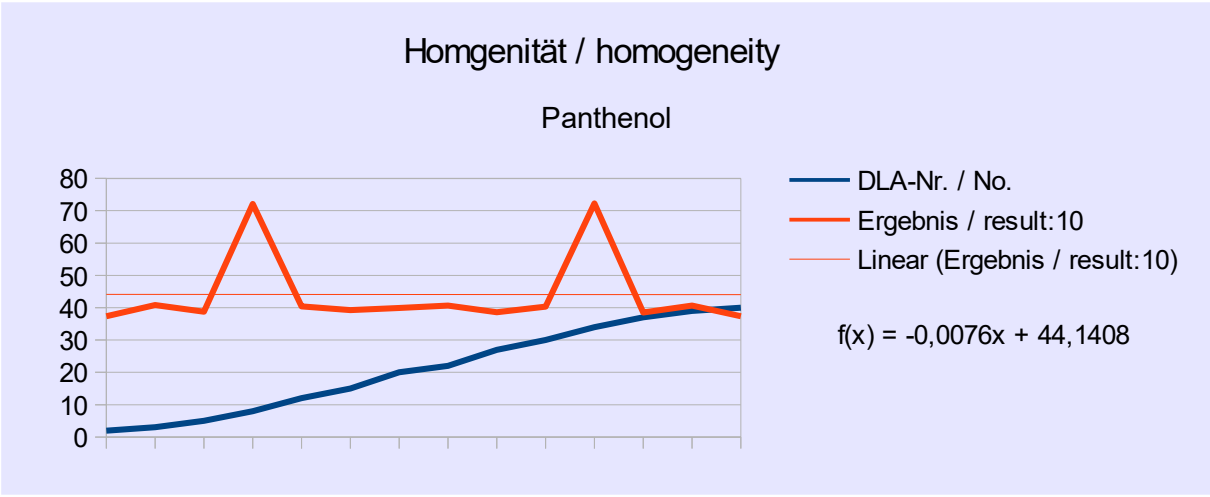

Abb./Fig. 10:
Trendfunktion Probennummern vs. Ergebnisse: Panthenol (1/10 dargestellt)
trend line function sample number vs. results: Panthenol (1/10 shown)

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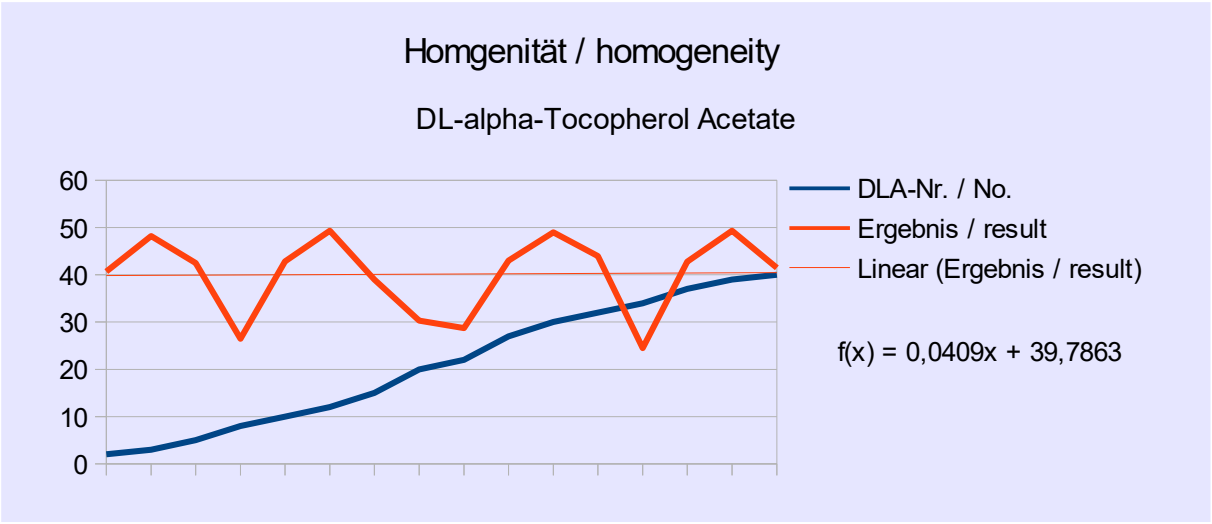

Abb./Fig. 11:
Trendfunktion Probennummern vs. Ergebnisse: DL-alpha Tocopherylacetat
trend line function sample number vs. results: DL-alpha Tocopheryl acetate

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5.3 Information on the Proficiency Test (PT)

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

PT number	***DLA ptCO03 - 2021***
PT name	***Cosmetic Products III: Coenzym Q10, Panthenol and Tocopherol in Skin Cream***
*Sample matrix**	*Samples I + II: Skin Cream, common in commerce ingredients*
Number of samples and sample amount	*2 identical samples I + II, 25 g each.*
Storage	*Samples I + II: cooled 2 - 10°C*
Intentional use	*Laboratory use only (quality control samples)*
Parameter	*quantitative: Coenzym Q10 (Ubiquinone), Panthenol and Tocopherol (Tocopheryl acetate)*
Methods of analysis	*Analytical methods are optional*
Notes to analysis	*The analysis of PT samples should be performed like a routine laboratory analysis.* *In general we recommend to homogenize a representative sample amount before analysis according to good laboratory practice, especially in case of low sample weights.*
Result sheet	*The results for sample I and II as well as the final results calculated as mean of the double determination (samples I and II) should be filled in the result submission file. The recovery rates, if carried out, has to be included in the calculation.*
Units	*mg/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 January 28th 2022***
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.*

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6. Index of participant laboratories in alphabetical order

Teilnehmer / Participant	Ort / Town	Land / Country
		USA
		AUSTRIA
		CZECH REPUBLIC
		Germany
		Germany
		Germany
		Germany
		Germany
		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.]

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7. Index of references

1. DIN EN ISO/IEC 17025:2005; Allgemeine Anforderungen an die Kompetenz von Prüf- und Kalibrierlaboratorien / General requirements for the competence of testing and calibration laboratories
2. DIN EN ISO/IEC 17043:2010; Konformitätsbewertung – Allgemeine Anforderungen an Eignungsprüfungen / Conformity assessment – General requirements for proficiency testing
3. ISO 13528:2015 & DIN ISO 13528:2009; Statistische Verfahren für Eignungsprüfungen durch Ringversuche / Statistical methods for use in proficiency testing by inter-laboratory comparisons
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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
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17. AOAC Official Methods of Analysis: Guidelines for Standard Method Performance Re-quirements, Appendix F, p. 2, AOAC Int (2016)

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