DLA
Dienstleistung
Lebensmittel
Analytik GbR

Evaluation Report
proficiency test

DLA 49/2016

Food Supplement III

Coenzyme Q10 and alpha-Liponic Acid

in Tablet Powder

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1. Introduction

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

2. Realisation

2.1 Test material

The test material is a mixture of common in commerce crushed tablets of a food supplement containing coenzyme Q10 and a pharmaceutical product containing alpha-liponic acid (thioctic acid) and additional lactose from EU suppliers. The materials were crushed, sieved, mixed and homogenized. After homogenization the samples were portioned to approximately 25 g into lightproof metallised PET film bags. The portions were numbered chronologically.

The composition (list of ingredients) of the samples is given in table 1. The contents of analytes were calculated according to the labeled values as given in table 2.

Table 1: Composition of DLA-Samples

Supplement tablets
Ingredients (1. supplement): Bulking agents: lactose and microcrystalline cellulose, **coenzyme Q10**, anti-caking agents: magnesium stearate and silicon dioxide. Ingredients (2. supplement): **alpha-Liponic acid**, bulking agents: lactose, E 1202, microcrystalline cellulose, cellulose powder, E 1420, E 464, anti-caking agents: silicon dioxide, stearic acid and magnesium stearate, carrier: E 553b and E 1521, colors: E 171 and E 172. additional ingredient: Bulking agent: lactose

Table 2: Calculated amounts according to labeled values of the analytes

Nutrient	**Content per 100 g**
Coenzyme Q10	278 mg
alpha-Liponic Acid	1.600 mg

2.1.1 Homogeneity

The **homogeneity of bottled numbered DLA-samples** was checked by 5fold determination of coenzyme Q10 by HPLC/UV. The repeatability standard deviation is 1,7% and is in the lower range of the repeatability standard deviations of comparable methods (see 3.6.2) [16, 17, 18]. The results of the homogeneity test are given in the documentation.

The calculation of the **repeatability standard deviation S_r of the participants** was also used as an indicator of homogeneity. For coenzyme Q10 it is 1,1% and below the repeatability standard deviations of of comparable methods (see Tab. 2) [16, 17, 18]. The repeatability standard deviation of the participants' results is given in the documentation of homogeneity testing (5.2) and in the table of statistic data (see 4.1).

Furthermore, the homogeneity was characterized by the **trend line function of participants' results for chronological bottled single samples**. The maximum deviations from the mean value of the trend line was in the range of 10% of the target standard deviation σ_{pt} (s. 5.2 homogeneity) and can therefore be regarded as low.

If the criteria for sufficient homogeneity of the test material are not fulfilled on a particular parameter, the impact on the target standard deviation is checked and optionally the evaluation of the results of the participants will be done using the z'-score considering the standard uncertainty of the assigned value (see 3.8 and 3.11) [3]. Even though criteria were fulfilled for the evaluation of inulin the z'-score was applied.

2.2 Sample shipment and information to the test

Two portions of test material were sent to every participating laboratory in the 11th week of 2016. The testing method was optional. The tests should be finished at 29th April 2016 the latest.

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

*The samples are two identical portions of food supplement (ground tablets) containing the analytes **Coenzyme Q10** and **Alpha-Liponic Acid**. The recommendation is to take 2,5 g per day. Each sample bag contains 25 g (10 daily intake doses). The material was tested for homogeneity and is intended for laboratory use only. The methods for determination are optional (e.g. HPLC).*
In general we recommend to homogenize a representative sample amount before analysis according to good laboratory practice, especially in case of low sample weights.

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.

From the 10 registered laboratories 8 participants submitted results. One laboratory canceled its participation and one participant submitted no results.

3. Evaluation

3.1 Consensus value from participants (assigned value)

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

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

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

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

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

3.2 Robust standard deviation

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

3.3 Repeatability standard deviation

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

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.

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.

The calculation of the reproducibility standard deviation S_R is performed by: [3, 4].

The relative reproducibility 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. Its meaning is explained in more detail in 3.9.

3.5 Exclusion of results and outliers

Before statistical evaluation obvious blunders, such as those with incorrect units, decimal point errors, and results for a another proficiency test item can be removed from the data set [2]. All results should be given at least with 2 significant digits. Specifying 3 significant digits is usually sufficient.

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

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

3.6 Target standard deviation (for proficiency assessment)

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

If an acceptable quotient S^*/σ_{pt} is present, the target standard deviation of the general model by Horwitz is preferably used for the proficiency assessment. It is usually suitable for for evaluation of interlaboratory studies, where different analytical 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.

In the present PT for the valuation of coenzyme Q10 the target standard deviation according to the general model of Horwitz was applied (3.6.1).

For alpha-liponic acid there were less than 7 quantitative results, thus no statistical valuation was done.

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]:

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

For coenzyme Q10 there are no results from precision experiments of proficiency-tests or official methods.
The repeatability standard deviations given in table 2 were obtained by studies from single laboratories or working groups.

Table 2: Relative repeatability standard deviations (RSD_r) of published methods [16, 17, 18]

Method	Parameter	Matrix	RSD_r	Literature
HPLC	Coenzyme Q10	Pharmaceutical Soy Oil Product	2,0 %	Andersson (1992)
HPLC-UV	Coenzyme Q10	Dairy Product	3,0 %	Strazisar et al. (2005)
HPLC-MS	Coenzyme Q10	Dairy Product	4,0 %	Strazisar et al. (2005)
HPLC-UV	Coenzyme Q10	Raw Materials and Food Supplements	2,2 - 5,0 %	Orozco et al. (2007)

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

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

3.7.1 Warning and action signals

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

trueness and precision must be performed and if necessary appropriate corrective measures should be applied [3].

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

3.8 z'-Score

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

The calculation is performed by:

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

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

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

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

For warning and action signals see 3.7.1.

3.9 Reproducibility coefficient of variation (CK_R)

The coefficient of variation (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 V_K 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 values or the performance evaluation of the participants possibly can not be done [3].

3.10 Quotient S*/σpt

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

The consensus value has a standard uncertainty $U(X_{pt})$ that depends on the analytical method, differences between the analytical methods used, the test material, the number of participant laboratories (P) and perhaps on other factors. The standard uncertainty of the assigned value ($U(X_{pt})$) 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 consensus value needs not to be included in the interpretation of the results of the PT [3]. A clear exceeded the value of 0,3 is an indication that the target standard deviation was possibly set too low for the standard uncertainty of the assigned value. The quotient $U(X_{pt})/\sigma_{pt}$ is reported in the characteristics of the test.

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}'
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 ($X_{pt} + 2\sigma_{pt}$´) *
Variation coefficient V_K in %
Quotient S*/σ_{pt} *or* S*/σ_{pt}'
Standard uncertainty $U(X_{pt})$
Quotient $U(X_{pt})/\sigma_{pt}$ *or* $U(X_{pt})/\sigma_{pt}$'
Number of results in the target range
Percent in the target range

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

In the second table the individual results of the participating laboratories are listed:

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

4.1 Coenzyme Q10 (Ubiquinone) in mg/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	8
Number of outliers	0
Mean	241
Median	245
Robust Mean (X)	**241**
Robust standard deviation (S*)	**15,0**
Number with 2 replicates	7
Repeatability SD (S_r)	2,69
Repeatability (CV_r)	1,13%
Reproducibility SD (S_R)	12,2
Reproducibility (CV_R)	5,11%
Target range:	
Target standard deviation σ_{pt}	**12,0**
lower limit of target range	**217**
upper limit of target range	**265**
Quotient S/σ_{pt}*	*1,3*
Standard uncertainty $U_{(Xpt)}$	*6,63*
Quotient $U_{(Xpt)}/\sigma_{pt}$	*0,55*
Results in the target range	*8*
Percent in the target range	*100%*

Comments to the statistic data:

The target standard deviation was calculated according to the general model of Horwitz.

The evaluation showed a normal to low variability of results. The quotient S^*/σ_{pt} was clearly below 2,0. The robust standard deviation as well as the repeatability and reproducibility standard deviations were in the range of established values for the applied methods (see 3.6.2). The comparability of results is given.

The quotient $U_{(Xpt)}/\sigma_{pt}$ was 0,54. Although it was not below 0,3 it is acceptable due to the other statistical data and the use of different analytical methods.

All results were in the target range.

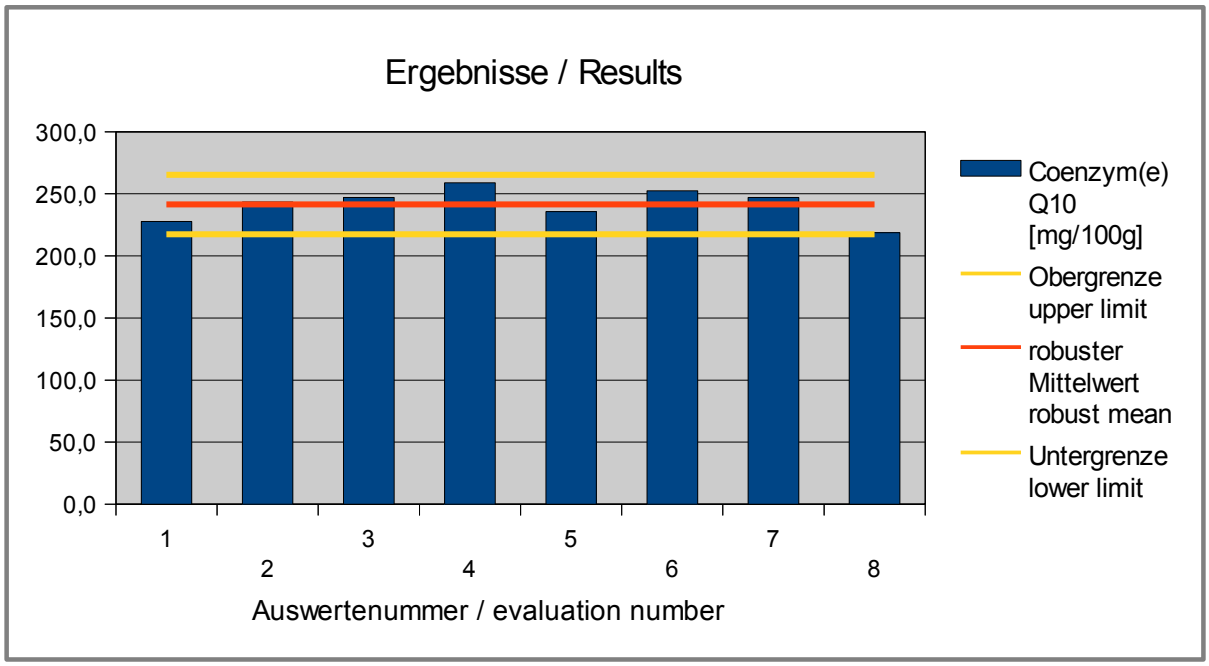

Abb. 1: Ergebnisse Coenzym Q10
Fig. 1: Results coenzyme Q10

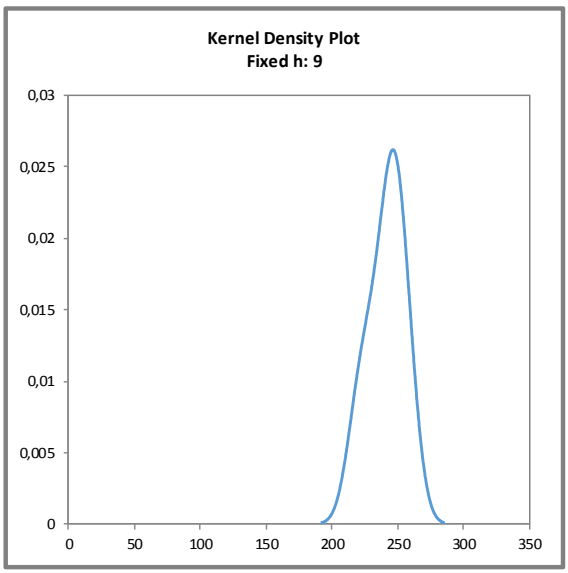

Abb. 2: Kerndichte-Schätzung der Ergebnisse für Coenzym Q10
(mit h = 0,75 x σ_{pt} von X_{pt})

Fig. 2: Kernel density plot of coenzyme Q10 results
(with h = 0,75 x σ_{pt} von X_{pt})

Comments:
The kernel density estimation shows almost a normal distribution (s. fig. 2).

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Coenzym(e) Q10 [mg/100g]	Abweichung [mg/100g] Deviation [mg/100g]	z-Score (σ_{pt})	Hinweis Remark
1	227,64	-13,7	-1,1	
2	243,4	2,0	0,2	
3	247	5,6	0,5	
4	259	17,6	1,5	
5	235,64	-5,7	-0,5	
6	252,3	10,9	0,9	
7	247	5,6	0,5	
8	218,7	-22,7	-1,9	

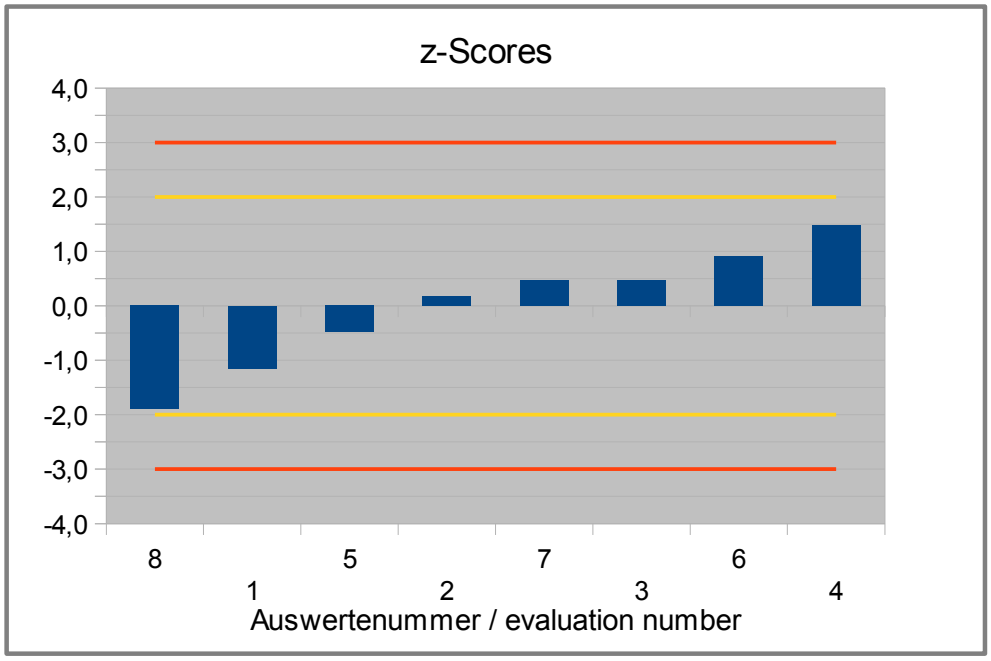

Abb. 3: Z-Scores Coenzym Q10
Fig. 3: Z-Scores coenzyme Q10

4.2 Alpha-Liponic Acid in mg/100g

Vergleichsuntersuchung / Proficiency Test

Only one result was submitted (participant 4: 1437 mg/100g).
Further details are given in the documentation.

5. Documentation

5.1 Primary data

Parameter	Teilnehmer	Einheit	Proben-Nr. A	Proben-Nr. B	Ergebnis (Mittel)	Ergebnis A	Ergebnis B	Wiederfindungsrate [%]
Analyte	**Participant**	**Unit**	**Sample No. A**	**Sample No. B**	**Result (Mean)**	**Result A**	**Result B**	**Recovery rate** [%]
Coenzym(e) Q10	1	mg/100g	**23**	**51**	227,64	236,92 (without recovery 227,44)	234,9 (without recovery 227,85)	97
	2	mg/100g	16	42	243,4	241	245,7	
	3	mg/100g	**31**	**45**	247	242 (without recovery 245)	244 (without recovery 248)	98,6
	4	mg/100g	37	6	259			
	5	mg/100g	26	39	235,64	236,22	235,06	n/a
	6	mg/100g	1	22	252,3	253,9	250,7	
	7	mg/100g	3	48	247	245	249	
	8	mg/100g	12	29	218,7	221,9	215,4	100
alpha-Liponsäure / alpha-Liponic Acid	1	mg/100g	**23**	**51**				
	2	mg/100g	16	42				
	3	mg/100g	**31**	**45**	k.A.	k.A.	k.A.	k.A.
	4	mg/100g	37	6	1437			
	5	mg/100g	26	39	n/a	n/a	n/a	n/a
	6	mg/100g	1	22				
	7	mg/100g	3	48				
	8	mg/100g	12	29				

5.2 Homogeneity

5.2.1 Homogeneity of bottled PT-samples

Homogeneity test of coenzyme Q10 by HPLC/UV:

Independant samples	mg/100g
1	272
2	280
3	282
4	284
5	277

Mean	279	
Repeatability Standard Deviation	4,7	1,7%

5.2.2 Repeatability standard deviation of replicate measurements of participants

The repeatability standard deviation S_r was calculated with the data documented in chapter 5.1 and given in the statistic data in 4.1.

It is S_r = 2,69 mg/100g and CV_r = 1,13 % of X for coenzyme Q10.

5.2.3 Comparison of sample numbers / test results and trend line

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

Coenzyme Q10
Sample numbers: 1 – 51
Measurement results: 14
Trend line range: 241,1 ± 1,13 mg/100g (= ± 0,094 x σ_{pt})
Maximum relative deviation to mean: ± 0,469%

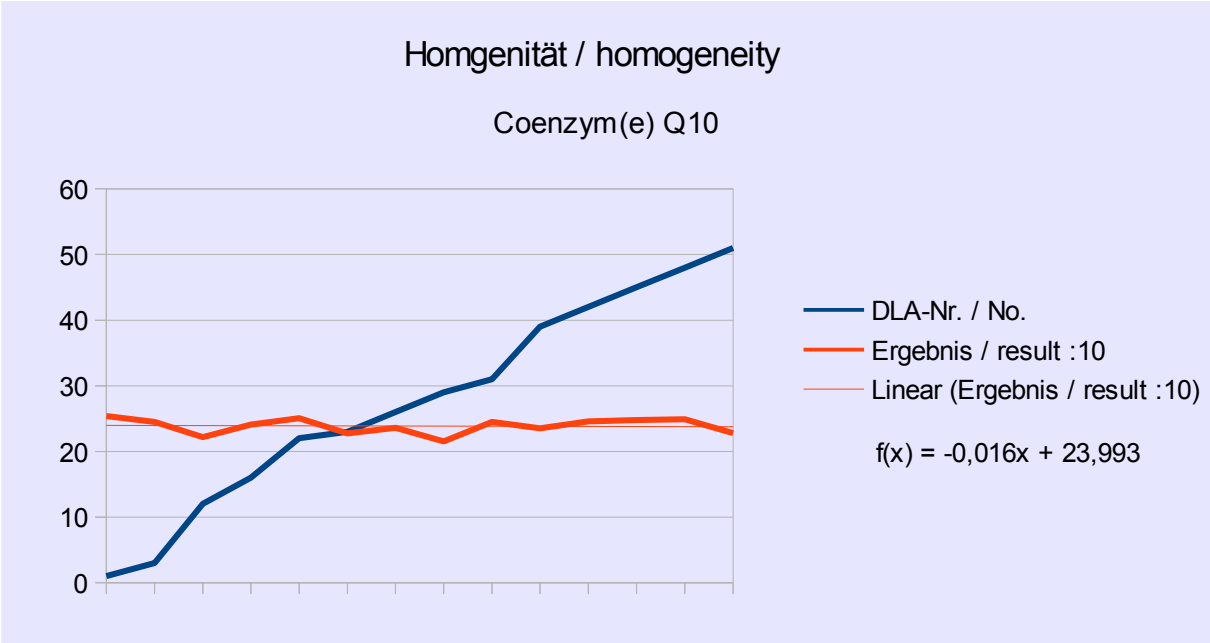

Abb. 4: Trendfunktion Probennummern / Coenzym Q10
Ergebnisse (: 10 dargestellt)
Fig. 4: trend line function sample number / coenzyme Q10
results (: 10 shown)

5.3 Analytical Methods

Details by the participants

Parameter	Teilnehmer	Methodenbeschreibung	Hinweise zur Analyse	NG	BG	Wiederfindung mit gleicher Matrix	Methode ist akkreditiert	Sonstige Hinweise
Analyte	**Participant**	**Method description**	**Notes to analysis**	**LOD**	**LOQ**	**Recovery with same matrix**	**Method accredited**	**Further remarks**
Coenzym(e) Q10	1	In-house method by Aquanova	-	0,04	0,4	no	yes	Recovery was determined by standard substance
	2	HPLC					no	without considering the recovery rate
	3	HPLC-DAD	none	8	30	yes / no	yes / no	
	4						no	
	5	RP-HPLC	n/a	n/a	n/a	n/a	yes	n/a
	6	HPLC DAD (210nm)	FI/FI Extraction with Isooctan/TBME	0,1 mg/100g	0,5 mg/100g	no	no	
	7	after extraction by LC-DAD	Extraction with aceton	17 mg/100 g	50 mg/100 g		yes	
	8	Determination of coenzyme Q10 content		0,2 mg/L	2,5 mg/L	yes	yes	

Parameter	Teilnehmer	Methodenbeschreibung	Hinweise zur Analyse	NG	BG	Wiederfindung mit gleicher Matrix	Methode ist akkreditiert	Sonstige Hinweise
Analyte	Participant	Method description	Notes to analysis	LOD	LOQ	Recovery with same matrix	Method accredited	Further remarks
alpha-Liponsäure / alpha-Liponic Acid	1							
	2							
	3							
	4						no	
	5	n/a	n/a	n/a	n/a	n/a	n/a	n/a
	6							
	7							
	8							

6. Index of participant laboratories in alphabetical order

Teilnehmer / Participant	Ort / Town	Land / Country
		Germany
		Germany
		Germany
		Germany
		USA
		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.]

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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