Proficiency Tests
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Evaluation Report
proficiency test

DLA 50/2018

Total Amino Acid Composition:

in Infant Food Drink Powder

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Allgemeine Informationen zur Eignungsprüfung (EP)
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.

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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 dietetic infant (drink powder, from >2 years of age on) with a protein content of < 20% based on milkprotein from a EU suppliers. The raw materials were sieved by means of a centrifugal mill (mesh 500 µm), mixed and homogenized.

Afterwards the samples were portioned to approximately 10 g into metal-lised PET film bags and chronologically numbered.

The composition (list of ingredients) is given in table 1.

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Table 1: Composition of DLA-Samples

Infant Milk Powder from 2 years on

Drink powder 1:

Ingredients:
Skimmed milk, whey product (demineralized whey, whey protein concentrate), oligosaccharides (galactooligosaccharides, fructo-oligosaccharides), palm oil, lactose, sunflower oil, rapeseed oil, potassium orthophosphate, calcium orthophosphate, fish oil, magnesium citrate, calcium carbonate, emulsifier (soya lecithin), vitamin C, ferrous sulfate, magnesium orthophosphate , vitamin E, vitamin D, vitamin B12, pantothenic acid, folic acid, vitamin A, vitamin B2, biotin, niacin, vitamin B6, vitamin B1, potassium iodide, vitamin K

Nutrients per 100 g: Fat 19 g, carbohydrates 45 g thereof sugar 43 g, fiber 8,2 g, protein 16 g, salt 0,6 g

Drink powder 2:

Ingredients:
Skimmed milk, lactose, palm oil, vegetable oil (rapeseed oil, sunflower oil), calcium citrate, calcium orthophosphate, emulsifier lecithin (soya), vitamin C, vitamin E, pantothenic acid, niacin, vitamin B1 (thiamine), vitamin A, vitamin B6, vitamin B2, folic acid, vitamin K, biotin, vitamin D, vitamin B12, potassium iodide

Nutrients per 100 g: Fat 16 g, carbohydrates 60 g thereof sugar 55 g, protein 17 g, salt 0,5 g

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

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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 86%. Additionally particle number results were converted into concentrations, statistically evaluated according to normal distribution and compared to the standard deviation according to Horwitz. For the assessment HorRat values between 0,3 and 1,3 are to be accepted under repeat conditions (measurements within the laboratory) [16, 17]. This gave a HorRat value of 0,8. The results of microtracer analysis 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 14 analytes it is < 2,4% (0,9% - 2,3%), for cystine, histidine and methionine in the range of 3,2% - 8,6% and for tryptophan increased with approx. 23%. Therefore with exception of tryptophan the repeatability standard deviations are similar to precision data of the referring standardized methods (e.g. ASU §64, s. 3.6.2) (see Tab. 3) [18-20]. The repeatability standard deviations of the participants' results are given in the tables of statistic data (see 4.1 to 4.19).

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

Parameter	CV_r	Parameter	CV_r
L-Alanine	2,31 %	L-Lysine	1,48 %
L-Arginine	1,77 %	L-Methionine	4,03 %
L-Aspartic acid	2,21 %	L-Phenylalanine	2,03 %
L-Cystine	8,56 %	L-Proline	0,91 %
L-Glutamic acid	1,49 %	L-Serine	1,43 %
Glycine	1,95 %	L-Threonine	1,55 %
L-Histidine	3,21 %	L-Tryptophan	22,7 %
L-Isoleucine	1,92 %	L-Tyrosine	2,09 %
L-Leucine	1,42 %	L-Valine	1,78 %

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

In case the criterion for sufficient homogeneity of the test items is not fulfilled the impact on the target standard deviation will be verified. If necessary the evaluation of results will be done considering the standard uncertainty of the assigned value by z'-scores (s. 3.8 and 3.11) [3].

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2.1.2 Stability

A water activity (a_W) of < 0,5 is an important factor to ensure the stability of dry or dried products during storage. Optimum conditions for storage is the a_W value range of 0,15 - 0,3. In this range the lowest possible degradation rate is to be expected [16].

The experience with various DLA test materials showed good storage stability with respect to the durability of the sample (spoilage) and the content of the PT parameters for comparable food matrices and water activity (a_W value <0,5).
The a_W value of the PT samples was approx. 0,21 (23,2°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 37th week of 2018. The testing method was optional. The tests should be finished at 26th October 2018 the latest.

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

The two portions contain identical samples of an infant milk drinking powder for children from 2 years on. Parameters to be determined are amino acids after protein hydrolysis. The protein equivalent content is < 20%. The analysis method is optional.

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 13 participants submitted their results in time.

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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^x) was calculated. The calculation was done according to algorithm A as described in annex C of ISO 13528 [3].

3.3 Repeatability standard deviation

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

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

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

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

3.4 Reproducibility standard deviation

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

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

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

3.5 Exclusion of results and outliers

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

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

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

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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.

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 valuation of all following parameters the target standard deviation according to the general model of Horwitz was applied (see 3.6.1): Glycine and L-Phenylalanine.

For the valuation of the following parameters the target standard deviation from precision experiments (s. 3.6.2) (German official ASU §64 [18, 19]) was applied: L-Alanine, L-Arginine, L-Aspartic Acid, L-Cystine, L-Glutamic Acid, L-Histidine, L-Isoleucine, L-Leucine, L-Lysine, L-Methionine, L-Proline, L-Serine, L-Threonine, L-Tryptophan, L-Tyrosine and L-Valine.

Additionally for L-Tryptophan the standard uncertainty was considered by evaluation using z'-scores (see 3.6.8).

Due to the low number of < 7 the results of Cysteine were not evaluated by means of z-scores.

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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 values given in Table 3 relative repeatability standard deviation (RSD_r) and relative reproducibility standard deviation (RSD_R) were determined in collaborative trials using the specified methods. The in the table indicated resulting target standard deviation σ_{pt} is additionally given in the evaluation for information.

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Table 3: Relative repeatability standard deviations (RSD_r) and relative reproducibility standard deviations (RSD_R) from precision experiments and resulting target standard deviations σ_{pt} [18-20]

Parameter	**Matrix**	**Mean [g/100g]**	**RSD_r**	**RSD_R**	**σ_{pt}**	**Method / Literature**
L-Alanine	Dietetic food	2,15	2,33%	5,12%	4,8% [1]	ASU 49.07-2
L-Arginine	Dietetic food	1,86	2,69%	6,99%	6,7% [1]	ASU 49.07-2
L-Aspartic acid	Dietetic food	4,16	1,92%	7,45%	7,3% [1]	ASU 49.07-2
L-Cysteine / L-Cystine	Dietetic food Protein concentrate	1,07 0,506	5,61% 2,6%	15,0% 12,3%	14,5% [1] 12,2%	ASU 49.07-2 Reg 152/ 2009/EC
L-Glutamic acid	Dietetic food	4,72	1,91%	5,08%	4,9% [1]	ASU 49.07-2
Glycine	Dietetic food	1,60	2,50%	6,88%	6,6% [1]	ASU 49.07-2
L-Histidine	Dietetic food	1,10	2,73%	10,90%	10,7% [1]	ASU 49.07-2
L-Isoleucine	Dietetic food	1,91	2,09%	5,24%	5,0% [1]	ASU 49.07-2
L-Leucine	Dietetic food	3,09	1,62%	5,50%	5,4% [1]	ASU 49.07-2
L-Lysine	Dietetic food Protein concentrate	3,61 4,77	1,94% 2,4%	8,95% 3,0%	8,8% [1] 2,5%	ASU 49.07-2 Reg 152/ 2009/EC
L-Methionine	Dietetic food Protein concentrate	1,30 1,2	5,38% 2,2%	7,69% 13%	6,7% [1] 12,9%	ASU 49.07-2 Reg 152/ 2009/EC
L-Phenylalanine	Dietetic food	-	-	-	-	ASU 49.07-2
L-Proline	Dietetic food	2,33	3,00%	8,15%	7,9% [1]	ASU 49.07-2
L-Serine	Dietetic food	2,28	2,63%	4,82%	4,4% [1]	ASU 49.07-2
L-Threonine	Dietetic food Protein concentrate	2,74 2,23	2,19% 2,7%	5,84% 3,8%	5,6% [1] 3,3%	ASU 49.07-2 Reg 152/ 2009/EC
L-Tryptophan	Dietetic food	0,30/0,24	3,75%	7,50%	7,0% [1]	ASU 49.07-3
L-Tyrosine	Dietetic food	3,05	2,62%	6,89%	6,6% [1]	ASU 49.07-2
L-Valine	Dietetic food	2,48	2,02%	6,05%	5,9% [1]	ASU 49.07-2

[1] *used/given in evaluation (s. chapter 4)*

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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 4 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. 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].

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Table 4: Characteristics of the present PT (on dark grey) in comparison to previous PTs since 2015 (SD = standard deviation, CV = coefficient of variation)

Parameter	Matrix (Powder)	robust Mean [g/100g]	rob. SD (S*) [g/100g]	rel. SD (CV_{S*}) [%]	Quotient S^*/σ_{pt}	DLA-Report
L-Alanine	Dietetic food	0,432	0,0150	3,47	0,77	DLA 42/2015
L-Alanine	Dietetic food	1,70	0,0562	3,31	0,90	DLA 59/2016
L-Alanine	Dietetic food	0,593	0,0528	8,91	1,68	DLA 50/2018
L-Arginine	Dietetic food	0,282	0,0384	13,6	1,69	DLA 42/2015
L-Arginine	Dietetic food	2,72	0,218	8,01	1,19	DLA 59/2016
L-Arginine	Dietetic food	0,524	0,0511	9,75	1,45	DLA 50/2018
L-Aspartic acid	Dietetic food	0,915	0,0369	4,03	0,55	DLA 42/2015
L-Aspartic acid	Dietetic food	4,55	0,130	2,86	0,90	DLA 59/2016
L-Aspartic acid	Dietetic food	1,38	0,131	9,49	1,30	DLA 50/2018
L-Cystine	Dietetic food	-	-	-	-	DLA 42/2015
L-Cystine	Dietetic food	0,487	0,133	27,3	1,89	DLA 59/2016
L-Cystine	Dietetic food	0,165	0,0229	13,9	0,89	DLA 50/2018
L-Glut-aminsäure	Dietetic food	1,906	0,124	6,51	1,36	DLA 42/2015
L-Glut-amic acid	Dietetic food	8,29	0,502	6,05	1,23	DLA 59/2016
L-Glut-amic acid	Dietetic food	3,52	0,253	7,17	1,29	DLA 50/2018
Glycine	Dietetic food	0,191	0,0116	6,07	1,20	DLA 42/2015
Glycine	Dietetic food	1,54	0,0933	6,06	1,62	DLA 59/2016
Glycine	Dietetic food	0,325	0,0300	9,20	1,94	DLA 50/2018
L-Histid-ine	Dietetic food	0,246	0,0662	26,9	1,62	DLA 42/2015
L-Histid-ine	Dietetic food	1,10	0,0915	8,32	0,78	DLA 59/2016
L-Histid-ine	Dietetic food	0,429	0,0485	11,3	1,05	DLA 50/2018

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Parameter	**Matrix** (Powder)	**robust Mean** [g/100g]	**rob. SD (S*)** [g/100g]	**rel. SD (CV_{S*})** [%]	**Quotient S^*/σ_{pt}**	**DLA-Report**
L-Isoleucine	Dietetic food	0,540	0,0276	5,11	1,16	DLA 42/2015
L-Isoleucine	Dietetic food	1,94	0,173	8,92	1,78	DLA 59/2016
L-Isoleucine	Dietetic food	0,862	0,0635	7,37	1,31	DLA 50/2018
L-Leucine	Dietetic food	1,02	0,0388	3,80	0,95	DLA 42/2015
L-Leucine	Dietetic food	3,45	0,248	7,19	1,34	DLA 59/2016
L-Leucine	Dietetic food	1,61	0,104	6,47	1,20	DLA 50/2018
L-Lysine	Dietetic food	0,817	0,0529	6,47	1,57	DLA 42/2015
L-Lysine	Dietetic food	2,71	0,168	6,20	1,81	DLA 59/2016
L-Lysine	Dietetic food	1,33	0,0968	7,30	0,83	DLA 50/2018
L-Methionine	Dietetic food	0,215	0,0123	5,72	0,94	DLA 42/2015
L-Methionine	Dietetic food	0,572	0,0608	10,6	1,59	DLA 59/2016
L-Methionine	Dietetic food	0,398	0,0486	12,2	1,54	DLA 50/2018
L-Phenylalanine	Dietetic food	0,408	0,0201	4,93	1,07	DLA 42/2015
L-Phenylalanine	Dietetic food	2,14	0,144	6,73	1,88	DLA 59/2016
L-Phenylalanine	Dietetic food	0,761	0,0551	7,24	1,74	DLA 50/2018
L-Proline	Dietetic food	0,744	0,0329	4,42	0,57	DLA 42/2015
L-Proline	Dietetic food	2,42	0,161	6,65	0,85	DLA 59/2016
L-Proline	Dietetic food	1,54	0,0726	4,71	0,60	DLA 50/2018
L-Serine	Dietetic food	0,538	0,0762	14,2	1,77	DLA 42/2015
L-Serine	Dietetic food	2,23	0,110	4,93	1,39	DLA 59/2016
L-Serine	Dietetic food	0,897	0,0739	8,24	1,54	DLA 50/2018
L-Threonine	Dietetic food	0,535	0,0203	3,79	0,86	DLA 42/2015

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Parameter	**Matrix** (Powder)	**robust Mean** [g/100g]	**rob. SD (S*)** [g/100g]	**rel. SD (CV_{S*})** [%]	**Quotient S^*/σ_{pt}**	**DLA-Report**
L-Threonine	Dietetic food	1,60	0,0914	5,71	1,53	DLA 59/2016
L-Threonine	Dietetic food	0,761	0,0516	6,79	1,21	DLA 50/2018
L-Tryptophan	Dietetic food	-	-	-	-	DLA 42/2015
L-Tryptophan	Dietetic food	0,563	0,0590	10,5	1,49	DLA 59/2016
L-Tryptophan	Dietetic food	0,213	0,0512	24,0	2,03	DLA 50/2018
L-Tyrosine	Dietetic food	0,314	0,0560	17,8	1,67	DLA 42/2015
L-Tyrosine	Dietetic food	1,56	0,166	10,6	1,61	DLA 59/2016
L-Tyrosine	Dietetic food	0,653	0,0669	10,2	1,35	DLA 50/2018
L-Valine	Dietetic food	0,598	0,0335	5,60	1,29	DLA 42/2015
L-Valine	Dietetic food	2,06	0,197	9,56	1,63	DLA 59/2016
L-Valine	Dietetic food	1,01	0,0600	5,92	0,95	DLA 50/2018

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3.8 z'-Score

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

The calculation is performed by:

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

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

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

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

For warning and action signals see 3.7.1.

3.9 Reproducibility cofficient of variation (CV_R)

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

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

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

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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.

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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, outliers or values outside the target range.
In the case of tryptophan a distribution of results with two peaks can be seen. However, the information provided by the participants on the methods gave no obvious indications of such an array of results.
When using the robust standard deviation as an estimator h, the distributions are converted into single peak distributions, so that an statistical evaluation has been carried out.

Comments to the statistic data:

For cysteine there were < 7 results, therefore no statistical evaluation could be done.

The target standard deviation was calculated for all other elements according to the model of Horwitz or according to the data of a precision experiment (ASU §64 method). The evaluation according to Horwitz was preferably used as long as the quotients S^*/σ_{pt} were in the range of ≤ 2,0. In all other cases, the standard deviation calculated from ASU §64 precision data was used (see 3.6).

For all parameters except tryptophan the distribution of results showed a normal variability. The quotients S^*/σ_{pt} were all below 2,0 in the range of 0,60 to 1,9 (see table 4).

For tryptophan the distribution of results showed an increased variability with a quotient above 2,0. Therefore tryptophan was evaluated considering the standard uncertainty by z´-scores. The quotient S^*/σ_{pt}' was 2,0 then (see table 4).

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

The comparability of results is given.

60% to 100% of results were in the respective target range.

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

In the first table the characteristics are listed:

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

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

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

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

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

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4.1 L-Alanin/L-Alanine in g/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	13
Number of outliers	-
Mean	0,596
Median	0,593
Robust Mean (X)	**0,593**
Robust standard deviation (S*)	**0,0528**
Number with 2 replicates	12
Repeatability SD (S_r)	0,0135
Repeatability (CV_r)	2,31%
Reproducibility SD (S_R)	0,0489
Reproducibility (CV_R)	8,40%
Target range:	
Target standard deviation σ_{pt}	**0,0287**
Target standard deviation (for Information)	0,0256
lower limit of target range	**0,535**
upper limit of target range	**0,650**
Quotient S/σ_{pt}*	*1,8*
Standard uncertainty $U_{(Xpt)}$	*0,0183*
Results in the target range	*9*
Percent in the target range	*69%*

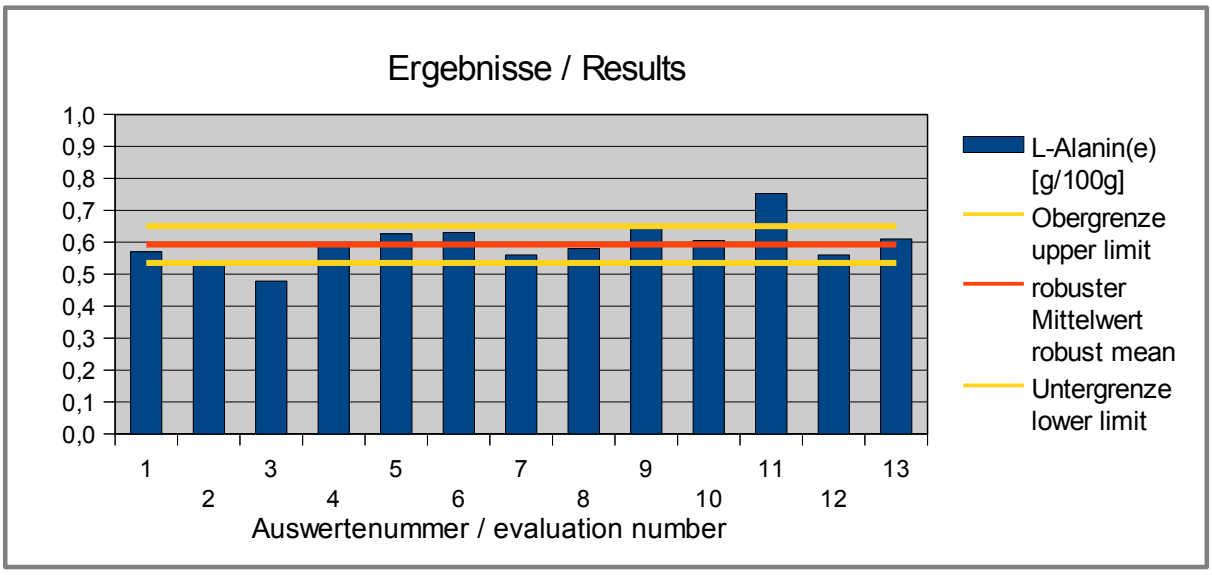

Abb. / Fig. 1: Ergebnisse L-Alanin / Results L-Alanine

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Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	L-Alanin(e) [g/100g]	Abweichung [g/100g] Deviation [g/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	0,570	-0,023	-0,8	-0,9	
2	0,530	-0,063	-2,2	-2,4	
3	0,478	-0,11	-4,0	-4,5	
4	0,593	0,00	0,0	0,0	
5	0,626	0,033	1,2	1,3	
6	0,630	0,037	1,3	1,5	
7	0,560	-0,033	-1,1	-1,3	
8	0,580	-0,013	-0,4	-0,5	
9	0,654	0,061	2,1	2,4	
10	0,605	0,012	0,4	0,5	
11	0,753	0,16	5,6	6,2	
12	0,560	-0,033	-1,1	-1,3	
13	0,610	0,017	0,6	0,7	

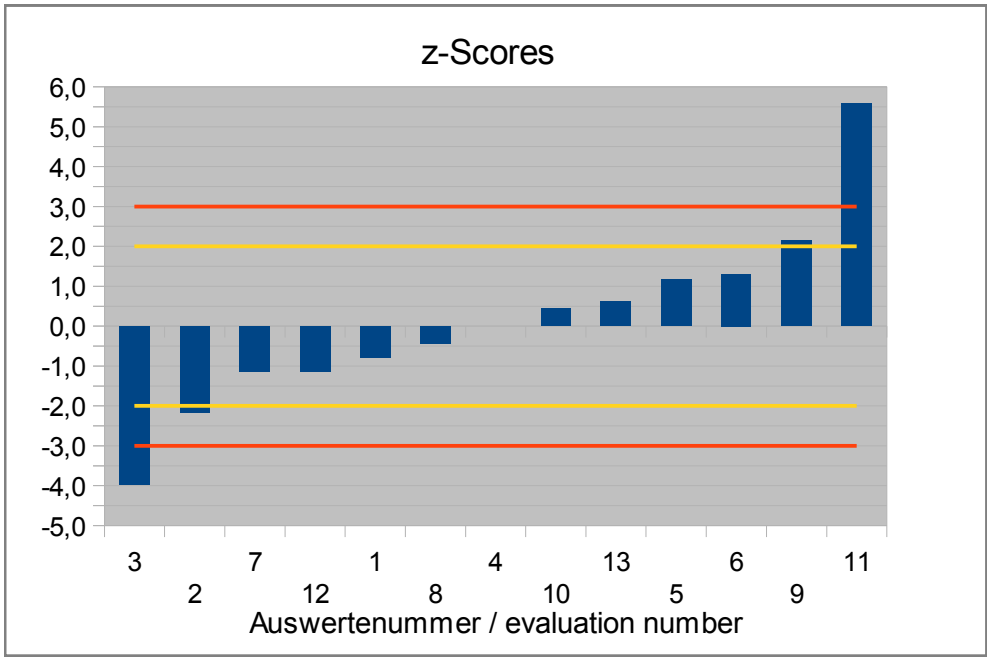

Abb. / Fig. 2: z-Scores L-Alanin / L-Alanine

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4.2 L-Arginin/L-Arginine in g/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	12
Number of outliers	0
Mean	0,524
Median	0,517
Robust Mean (X)	**0,524**
Robust standard deviation (S*)	**0,0511**
Number with 2 replicates	12
Repeatability SD (S_r)	0,0093
Repeatability (CV_r)	1,77%
Reproducibility SD (S_R)	0,0617
Reproducibility (CV_R)	11,8%
Target range:	
Target standard deviation σ_{pt}	**0,0352**
Target standard deviation (for Information)	0,0231
lower limit of target range	**0,454**
upper limit of target range	**0,595**
Quotient S/σ_{pt}*	*1,4*
Standard uncertainty $U_{(Xpt)}$	*0,0184*
Results in the target range	*9*
Percent in the target range	*75%*

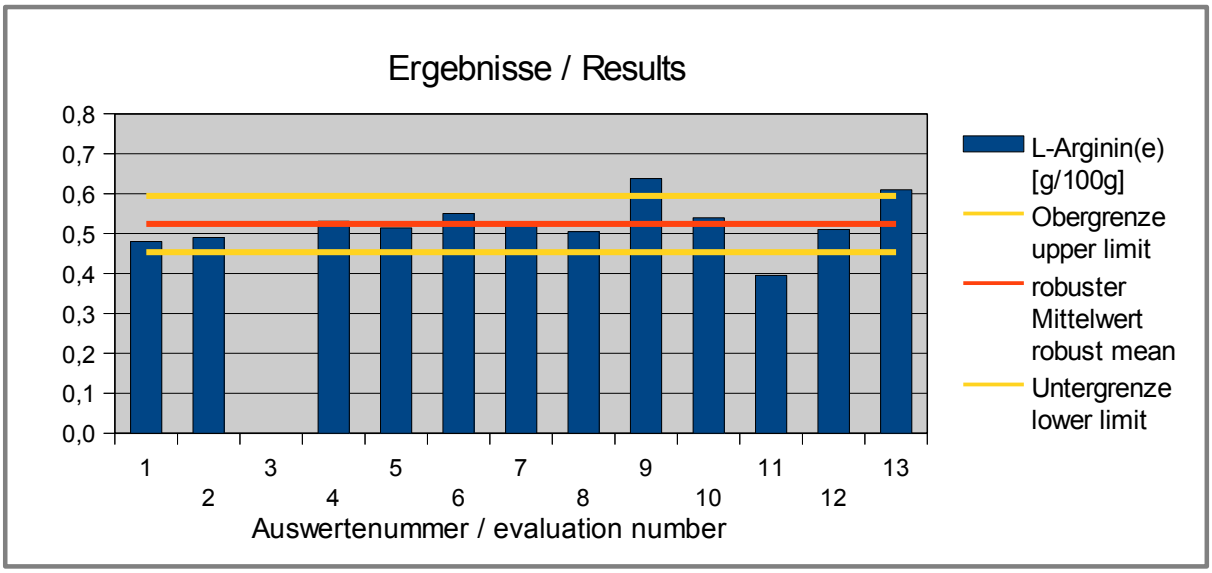

Abb. / Fig. 3: Ergebnisse L-Arginin / Results L-Arginine

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Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	L-Arginin(e) [g/100g]	Abweichung [g/100g] Deviation [g/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	0,480	-0,044	-1,2	-1,9	
2	0,490	-0,034	-1,0	-1,5	
3					
4	0,531	0,007	0,2	0,3	
5	0,514	-0,010	-0,3	-0,4	
6	0,550	0,026	0,7	1,1	
7	0,520	-0,004	-0,1	-0,2	
8	0,505	-0,019	-0,5	-0,8	
9	0,638	0,114	3,2	4,9	
10	0,540	0,016	0,5	0,7	
11	0,396	-0,129	-3,6	-5,6	
12	0,510	-0,014	-0,4	-0,6	
13	0,610	0,086	2,4	3,7	

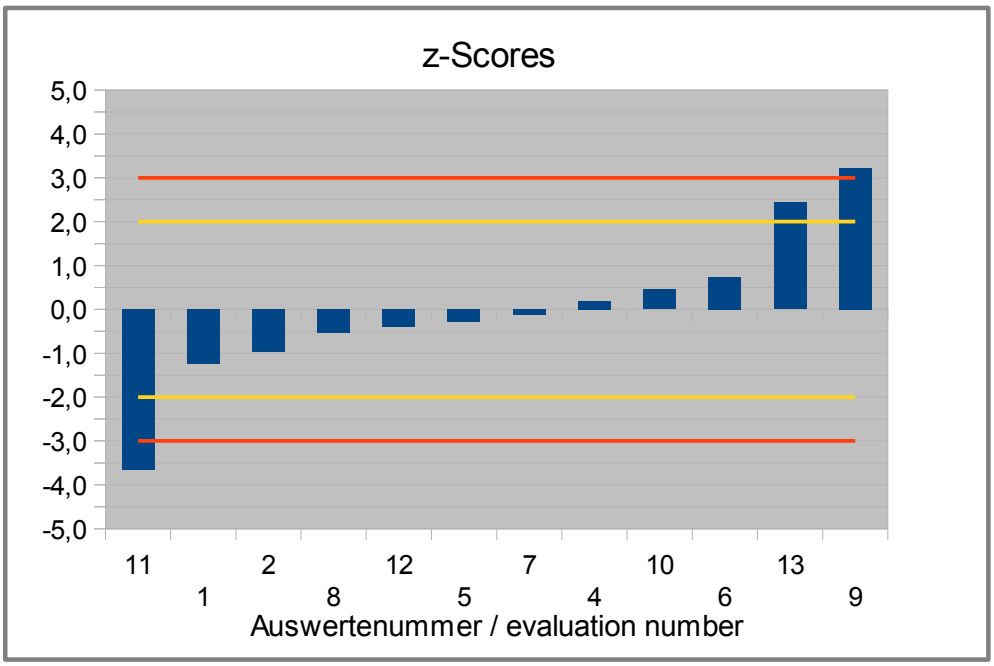

Abb. / Fig. 4: z-Scores L-Arginin / L-Arginine

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4.3 L-Asparaginsäure/L-Aspartic acid in g/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	13
Number of outliers	0
Mean	1,39
Median	1,34
Robust Mean (X)	**1,38**
Robust standard deviation (S*)	**0,131**
Number with 2 replicates	13
Repeatability SD (S_r)	0,0308
Repeatability (CV_r)	2,21%
Reproducibility SD (S_R)	0,135
Reproducibility (CV_R)	9,72%
Target range:	
Target standard deviation σ_{pt}	**0,101**
Target standard deviation (for Information)	0,0527
lower limit of target range	**1,18**
upper limit of target range	**1,59**
Quotient S/σ_{pt}*	*1,3*
Standard uncertainty $U_{(Xpt)}$	*0,0455*
Results in the target range	*12*
Percent in the target range	*92%*

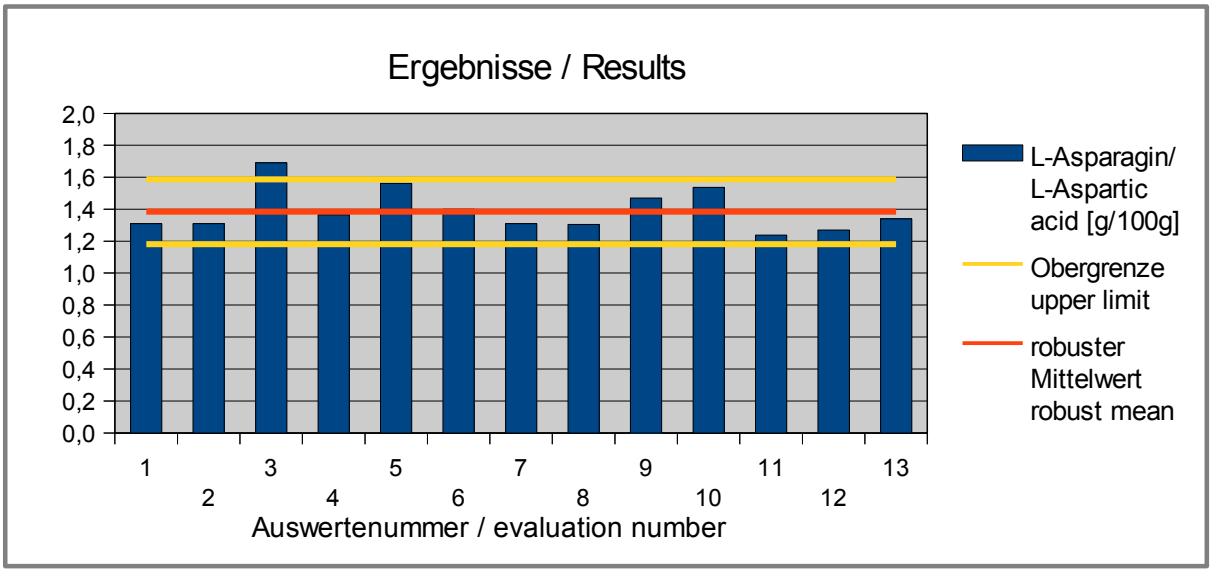

Abb. / Fig. 5: Ergebnisse L-Asparaginsäure / Results L-Aspartic acid

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Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	L-Asparagin / L-Aspartic acid [g/100g]	Abweichung [g/100g] Deviation [g/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	1,31	-0,074	-0,7	-1,4	
2	1,31	-0,074	-0,7	-1,4	
3	1,69	0,306	3,0	5,8	
4	1,37	-0,019	-0,2	-0,4	
5	1,56	0,178	1,8	3,4	
6	1,40	0,016	0,2	0,3	
7	1,31	-0,074	-0,7	-1,4	
8	1,31	-0,079	-0,8	-1,5	
9	1,47	0,086	0,8	1,6	
10	1,54	0,152	1,5	2,9	
11	1,24	-0,147	-1,5	-2,8	
12	1,27	-0,114	-1,1	-2,2	
13	1,34	-0,044	-0,4	-0,8	

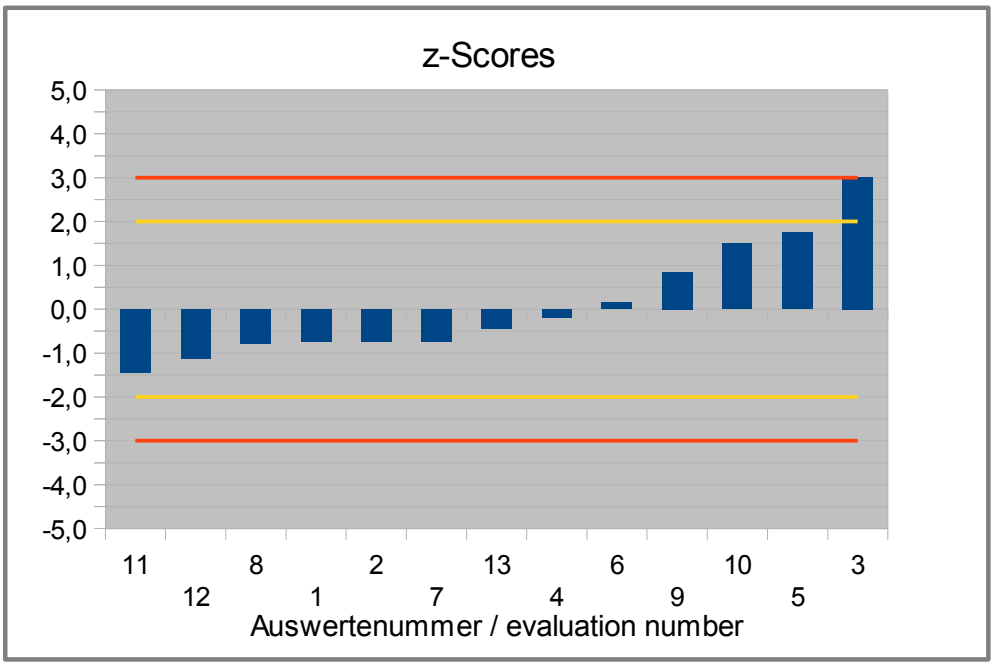

Abb. / Fig. 6: z-Scores L-Asparaginsäure / L-Aspartic acid

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4.4 L-Cystein/L-Cysteine in g/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	2
Number of outliers	0
Mean	0,172
Median	0,172
Robust Mean (X)	**0,172**
Robust standard deviation (S*)	**0,0192**
Number with 2 replicates	
Repeatability SD (S_r)	
Repeatability (CV_r)	
Reproducibility SD (S_R)	
Reproducibility (CV_R)	
Target range:	
Target standard deviation σ_{pt}	
Target standard deviation (for Information)	
lower limit of target range	
upper limit of target range	
Quotient S/σ_{pt}*	
Standard uncertainty $U_{(Xpt)}$	
Results in the target range	
Percent in the target range	

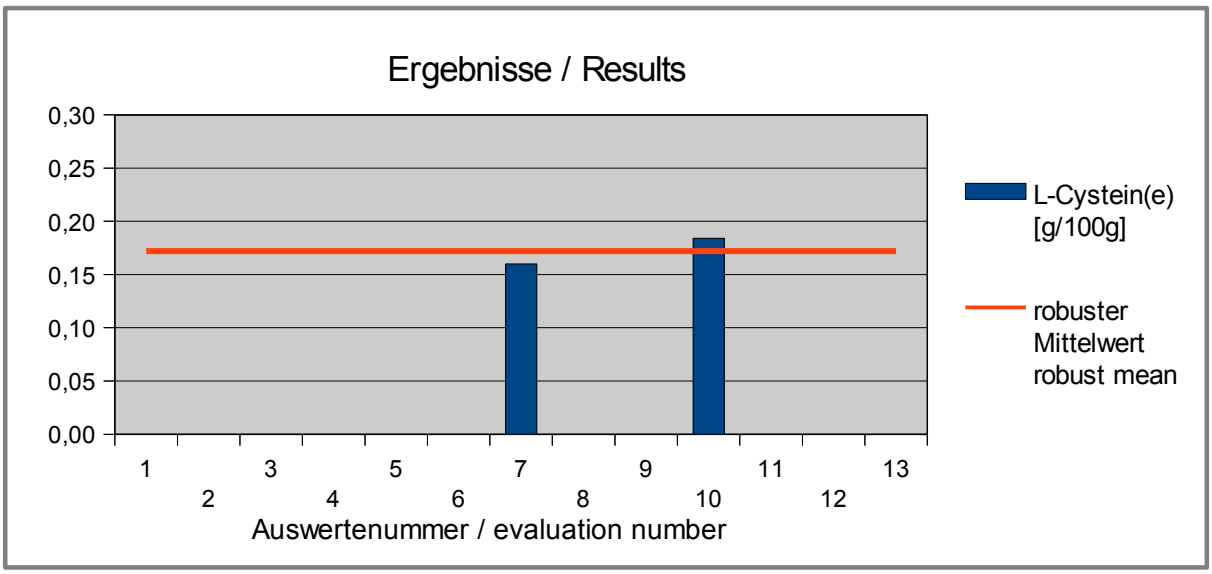

Abb. / Fig. 7: Ergebnisse L-Cystein / Results L-Cysteine

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Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	L-Cystein(e) [g/100g]	Abweichung [g/100g] Deviation [g/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1					
2					
3					
4					
5					
6					
7	0,160	-0,0120			
8					
9					
10	0,184	0,0120			
11					
12					
13					

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4.5 L-Cystin/L-Cystine in g/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	8
Number of outliers	0
Mean	0,163
Median	0,165
Robust Mean (X)	**0,165**
Robust standard deviation (S*)	**0,0229**
Number with 2 replicates	8
Repeatability SD (S_r)	0,0138
Repeatability (CV_r)	8,56%
Reproducibility SD (S_R)	0,0261
Reproducibility (CV_R)	16,1%
Target range:	
Target standard deviation σ_{pt}	**0,0238**
Target standard deviation (for Information)	0,00864
lower limit of target range	**0,117**
upper limit of target range	**0,212**
Quotient S/σ_{pt}*	*0,96*
Standard uncertainty $U_{(Xpt)}$	*0,0101*
Results in the target range	*7*
Percent in the target range	*88%*

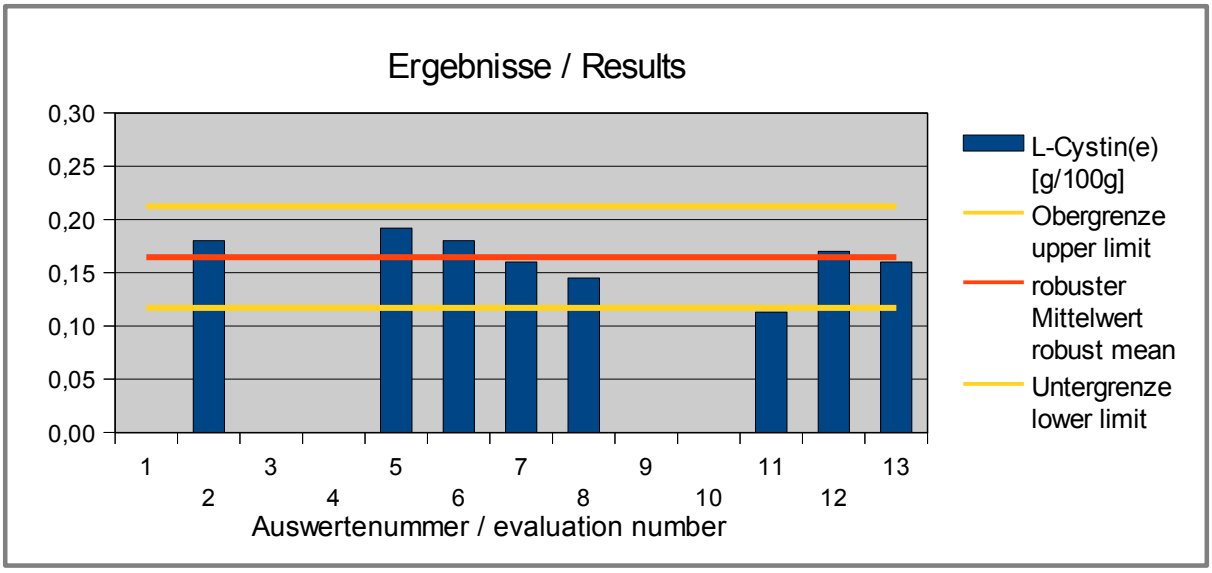

Abb. / Fig. 8: Ergebnisse L-Cystin / Results L-Cystine

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Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	L-Cystin(e) [g/100g]	Abweichung [g/100g] Deviation [g/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1					
2	0,180	0,015	0,6	1,8	
3					
4					
5	0,192	0,027	1,1	3,2	
6	0,180	0,015	0,6	1,8	
7	0,160	-0,005	-0,2	-0,5	
8	0,145	-0,020	-0,8	-2,3	
9					
10					
11	0,113	-0,052	-2,2	-6,0	
12	0,170	0,005	0,2	0,6	
13	0,160	-0,005	-0,2	-0,5	

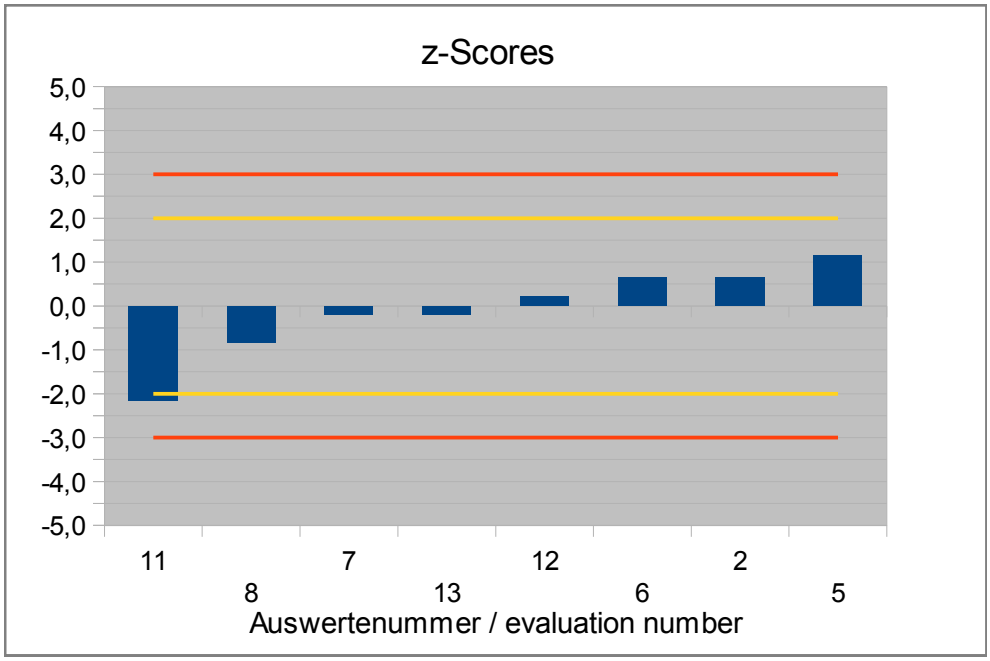

Abb. / Fig. 9: z-Scores L-Cystin / L-Cystine

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4.6 L-Glutaminsäure/L-Glutamic acid in g/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	12
Number of outliers	0
Mean	3,50
Median	3,50
Robust Mean (X)	**3,52**
Robust standard deviation (S*)	**0,253**
Number with 2 replicates	12
Repeatability SD (S_r)	0,0519
Repeatability (CV_r)	1,49%
Reproducibility SD (S_R)	0,274
Reproducibility (CV_R)	7,83%
Target range:	
Target standard deviation σ_{pt}	**0,173**
Target standard deviation (for Information)	0,117
lower limit of target range	**3,18**
upper limit of target range	**3,87**
Quotient S/σ_{pt}*	*1,5*
Standard uncertainty $U_{(Xpt)}$	*0,0912*
Results in the target range	*10*
Percent in the target range	*83%*

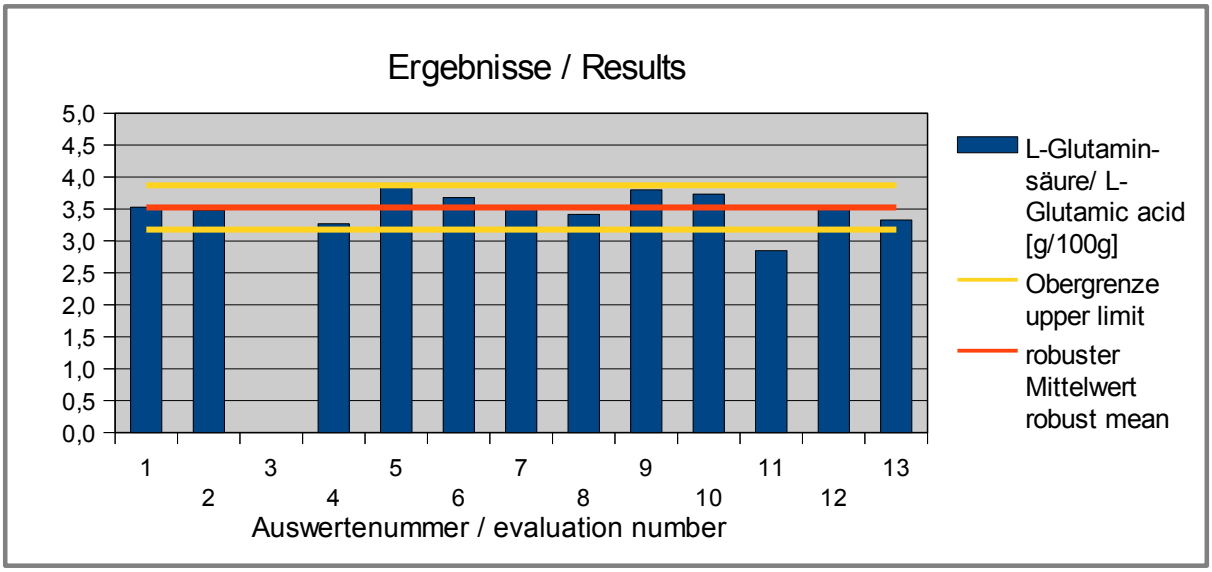

Abb. / Fig. 10: Ergebnisse L-Glutaminsäure / Results L-Glutamic acid

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Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	L-Glutamin-säure / L-Glutamic acid [g/100g]	Abweichung [g/100g] Deviation [g/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	3,53	0,006	0,0	0,1	
2	3,48	-0,044	-0,3	-0,4	
3					
4	3,27	-0,253	-1,5	-2,2	
5	3,90	0,375	2,2	3,2	
6	3,68	0,156	0,9	1,3	
7	3,51	-0,014	-0,1	-0,1	
8	3,42	-0,109	-0,6	-0,9	
9	3,80	0,276	1,6	2,4	
10	3,73	0,210	1,2	1,8	
11	2,85	-0,679	-3,9	-5,8	
12	3,49	-0,034	-0,2	-0,3	
13	3,33	-0,194	-1,1	-1,7	

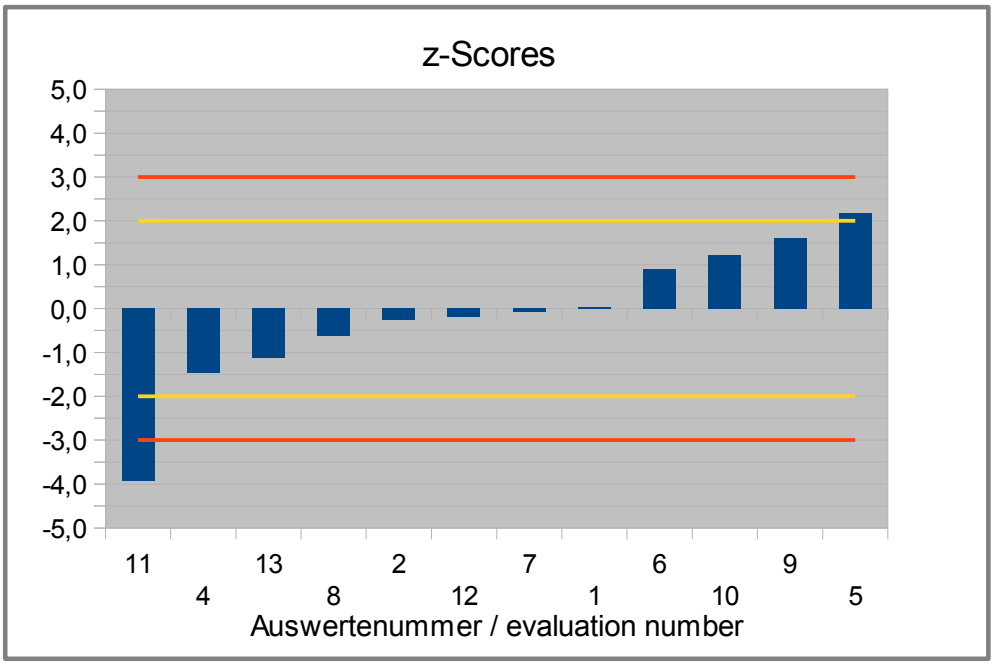

Abb. / Fig. 11: z-Scores L-Glutaminsäure / L-Glutamic acid

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4.7 Glycin/Glycine in g/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	13
Number of outliers	-
Mean	0,330
Median	0,325
Robust Mean (X)	**0,325**
Robust standard deviation (S*)	**0,0300**
Number with 2 replicates	10
Repeatability SD (S_r)	0,00624
Repeatability (CV_r)	1,95%
Reproducibility SD (S_R)	0,0168
Reproducibility (CV_R)	5,23%
Target range:	
Target standard deviation σ_{pt}	**0,0154**
Target standard deviation (for Information)	0,0216
lower limit of target range	**0,295**
upper limit of target range	**0,356**
Quotient S/σ_{pt}*	*1,9*
Standard uncertainty $U_{(Xpt)}$	*0,0104*
Results in the target range	*10*
Percent in the target range	*77%*

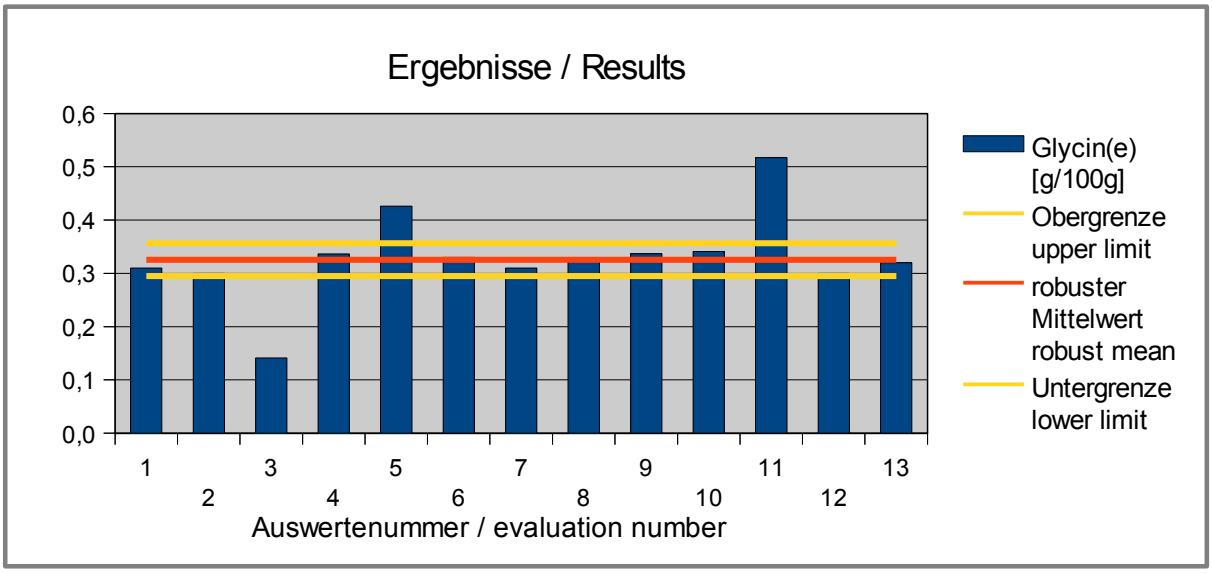

Abb. / Fig. 12: Ergebnisse Glycin / Results Glycine

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Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Glycin(e) [g/100g]	Abweichung [g/100g] Deviation [g/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	0,310	-0,0154	-1,0	-0,7	
2	0,300	-0,0254	-1,7	-1,2	
3	0,141	-0,1844	-12,0	-8,5	
4	0,337	0,0111	0,7	0,5	
5	0,426	0,1006	6,5	4,6	
6	0,330	0,0046	0,3	0,2	
7	0,310	-0,0154	-1,0	-0,7	
8	0,325	-0,0004	0,0	0,0	
9	0,337	0,0116	0,7	0,5	
10	0,341	0,0156	1,0	0,7	
11	0,517	0,192	12,4	8,9	
12	0,300	-0,0254	-1,7	-1,2	
13	0,320	-0,0054	-0,4	-0,3	

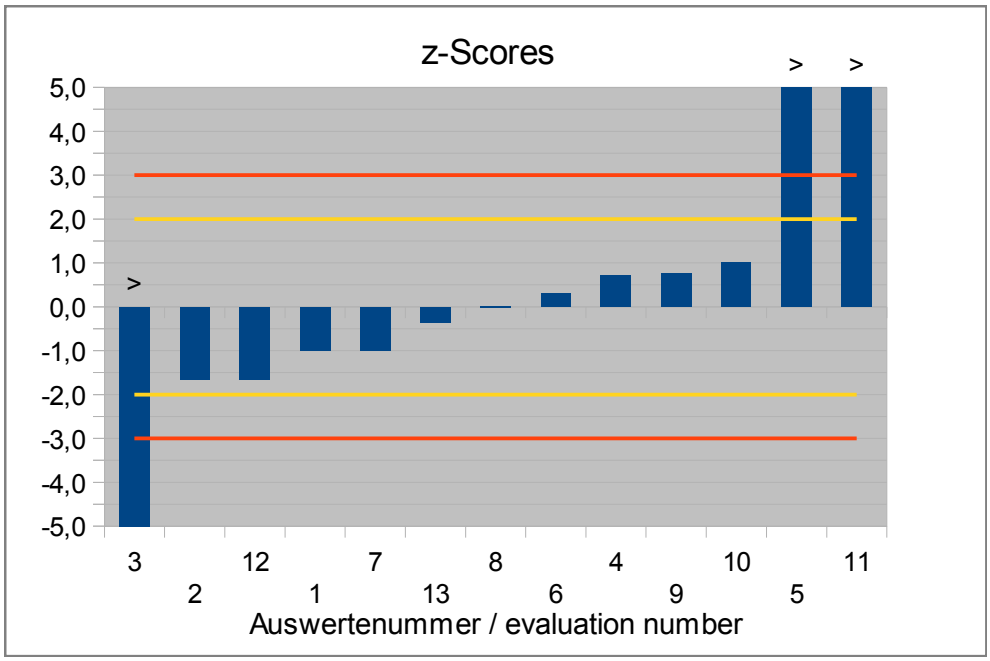

Abb. / Fig. 13: z-Scores Glycin / Glycine

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4.8 L-Histidin/L-Histidine in g/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	12
Number of outliers	0
Mean	0,430
Median	0,423
Robust Mean (X)	**0,429**
Robust standard deviation (S*)	**0,0485**
Number with 2 replicates	12
Repeatability SD (S_r)	0,0138
Repeatability (CV_r)	3,21%
Reproducibility SD (S_R)	0,0469
Reproducibility (CV_R)	10,9%
Target range:	
Target standard deviation σ_{pt}	**0,0460**
Target standard deviation (for Information)	0,0195
lower limit of target range	**0,337**
upper limit of target range	**0,520**
Quotient S/σ_{pt}*	*1,1*
Standard uncertainty $U_{(Xpt)}$	*0,0175*
Results in the target range	*12*
Percent in the target range	*100%*

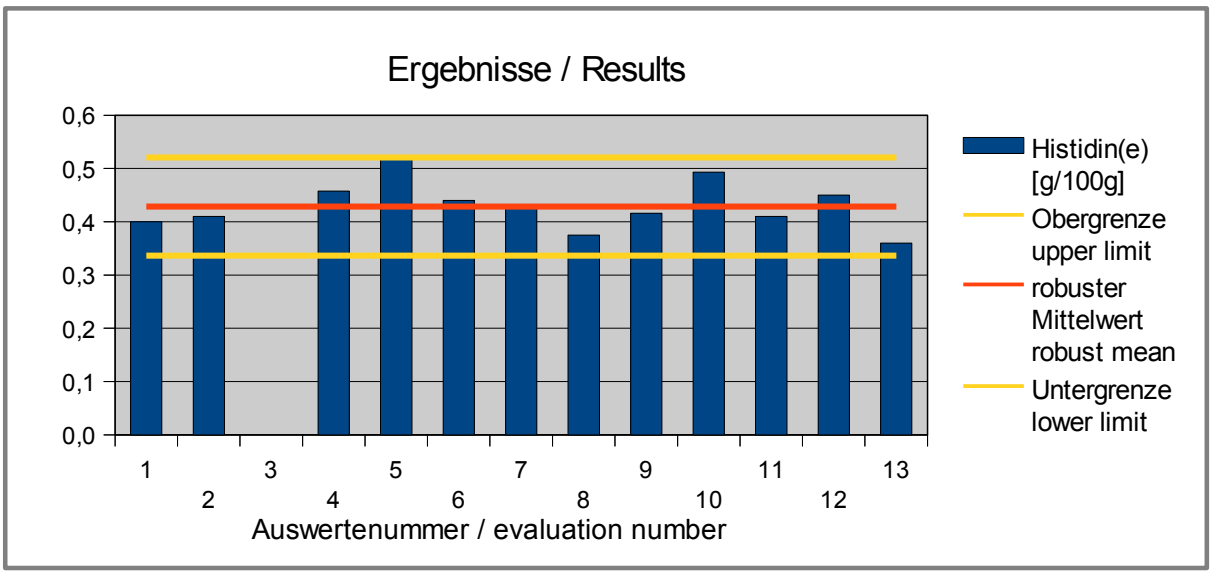

Abb. / Fig. 14: Ergebnisse L-Histidin / Results L-Histidine

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Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Histidin(e) [g/100g]	Abweichung [g/100g] Deviation [g/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	0,400	-0,0285	-0,6	-1,5	
2	0,410	-0,0185	-0,4	-1,0	
3					
4	0,458	0,0290	0,6	1,5	
5	0,518	0,0895	1,9	4,6	
6	0,440	0,0115	0,2	0,6	
7	0,430	0,0015	0,0	0,1	
8	0,375	-0,0535	-1,2	-2,7	
9	0,416	-0,0125	-0,3	-0,6	
10	0,493	0,0645	1,4	3,3	
11	0,410	-0,0187	-0,4	-1,0	
12	0,450	0,0215	0,5	1,1	
13	0,360	-0,0685	-1,5	-3,5	

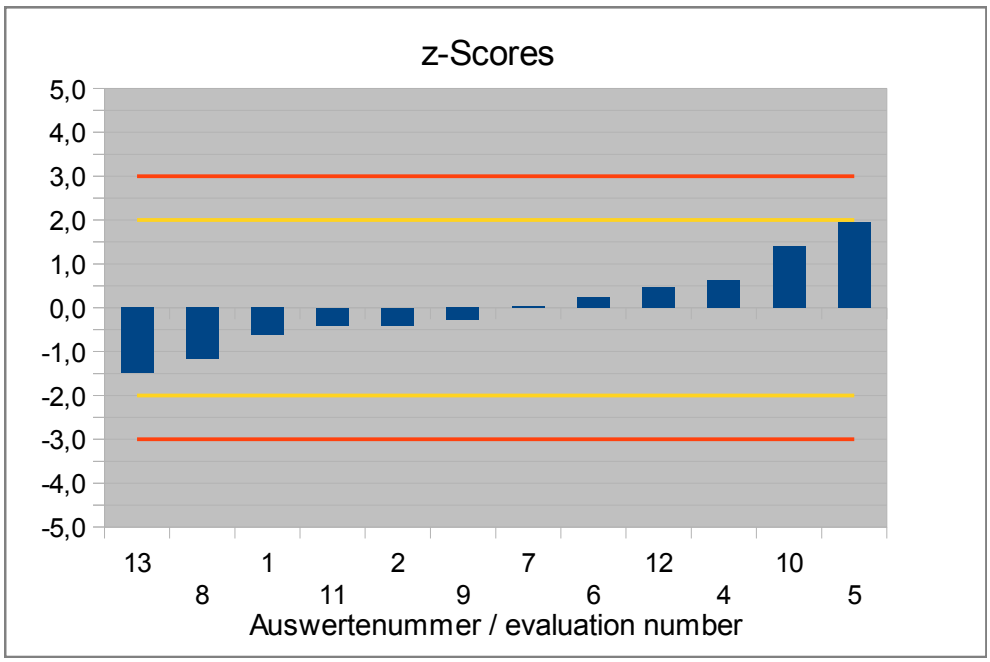

Abb. / Fig. 15: z-Scores L-Histidin / L-Histidine

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4.9 L-Isoleucin/L-Isoleucine in g/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	13
Number of outliers	0
Mean	0,862
Median	0,860
Robust Mean (X)	**0,862**
Robust standard deviation (S*)	**0,0635**
Number with 2 replicates	13
Repeatability SD (S_r)	0,0166
Repeatability (CV_r)	1,92%
Reproducibility SD (S_R)	0,0659
Reproducibility (CV_R)	7,65%
Target range:	
Target standard deviation σ_{pt}	**0,0433**
Target standard deviation (for Information)	0,0353
lower limit of target range	**0,775**
upper limit of target range	**0,949**
Quotient S/σ_{pt}*	*1,5*
Standard uncertainty $U_{(Xpt)}$	*0,0220*
Results in the target range	*10*
Percent in the target range	*77%*

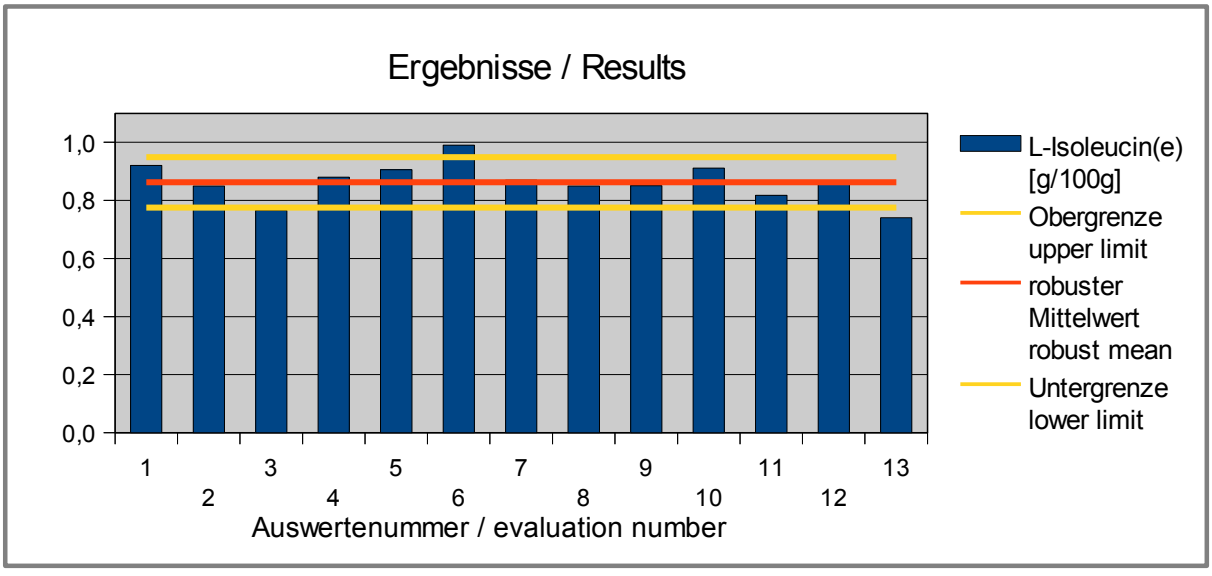

Abb. / Fig. 16: Ergebnisse L-Isoleucin / Results L-Isoleucine

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Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	L-Isoleucin(e) [g/100g]	Abweichung [g/100g] Deviation [g/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	0,920	0,0579	1,3	1,6	
2	0,850	-0,0121	-0,3	-0,3	
3	0,765	-0,0971	-2,2	-2,8	
4	0,880	0,0179	0,4	0,5	
5	0,906	0,0439	1,0	1,2	
6	0,990	0,1279	3,0	3,6	
7	0,870	0,0079	0,2	0,2	
8	0,850	-0,0121	-0,3	-0,3	
9	0,851	-0,0111	-0,3	-0,3	
10	0,911	0,0489	1,1	1,4	
11	0,818	-0,0441	-1,0	-1,2	
12	0,860	-0,0021	0,0	-0,1	
13	0,740	-0,122	-2,8	-3,5	

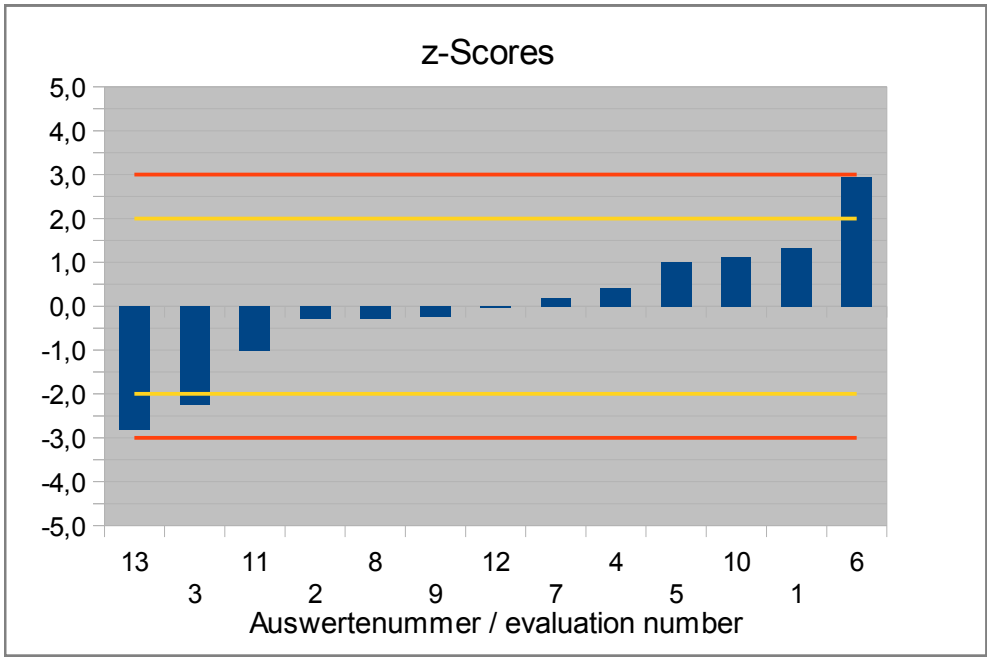

Abb. / Fig. 17: z-Scores L-Isoleucin / L-Isoleucine

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4.10 L-Leucin/L-Leucine in g/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	13
Number of outliers	0
Mean	1,61
Median	1,61
Robust Mean (X)	**1,61**
Robust standard deviation (S*)	**0,104**
Number with 2 replicates	13
Repeatability SD (S_r)	0,0228
Repeatability (CV_r)	1,42%
Reproducibility SD (S_R)	0,0936
Reproducibility (CV_R)	5,82%
Target range:	
Target standard deviation σ_{pt}	**0,0865**
Target standard deviation (for Information)	0,0599
lower limit of target range	**1,43**
upper limit of target range	**1,78**
Quotient S/σ_{pt}*	*1,2*
Standard uncertainty $U_{(Xpt)}$	*0,0361*
Results in the target range	*13*
Percent in the target range	*100%*

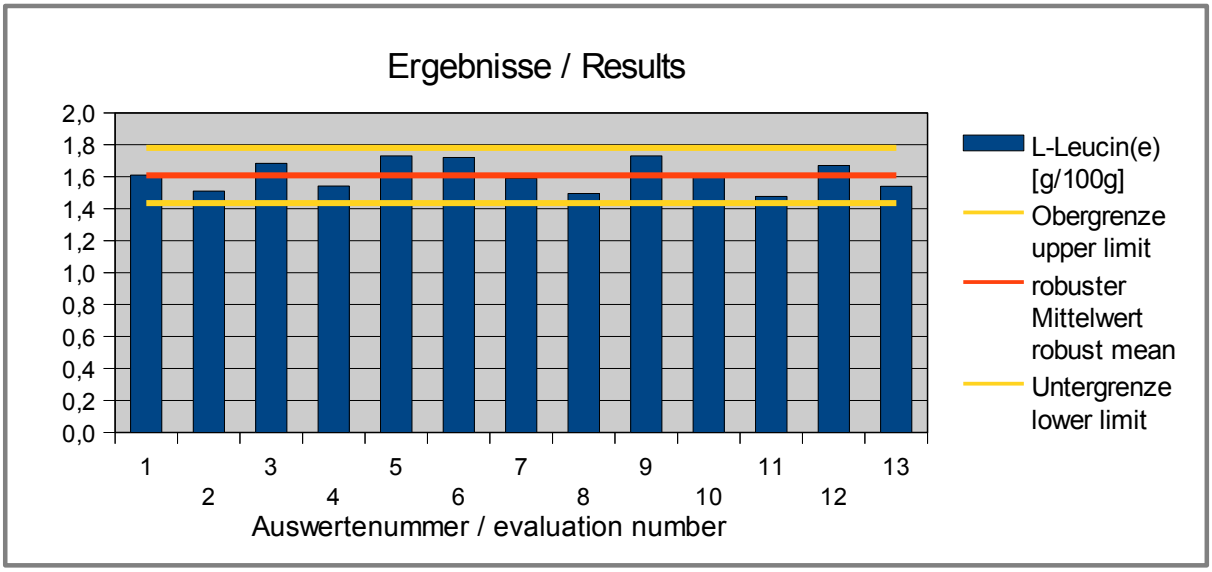

Abb. / Fig. 18: Ergebnisse L-Leucin / Results L-Leucine

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Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	L-Leucin(e) [g/100g]	Abweichung [g/100g] Deviation [g/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	1,61	0,002	0,0	0,0	
2	1,51	-0,098	-1,1	-1,6	
3	1,68	0,076	0,9	1,3	
4	1,54	-0,067	-0,8	-1,1	
5	1,73	0,122	1,4	2,0	
6	1,72	0,112	1,3	1,9	
7	1,59	-0,018	-0,2	-0,3	
8	1,50	-0,113	-1,3	-1,9	
9	1,73	0,122	1,4	2,0	
10	1,61	-0,003	0,0	0,0	
11	1,48	-0,131	-1,5	-2,2	
12	1,67	0,062	0,7	1,0	
13	1,54	-0,068	-0,8	-1,1	

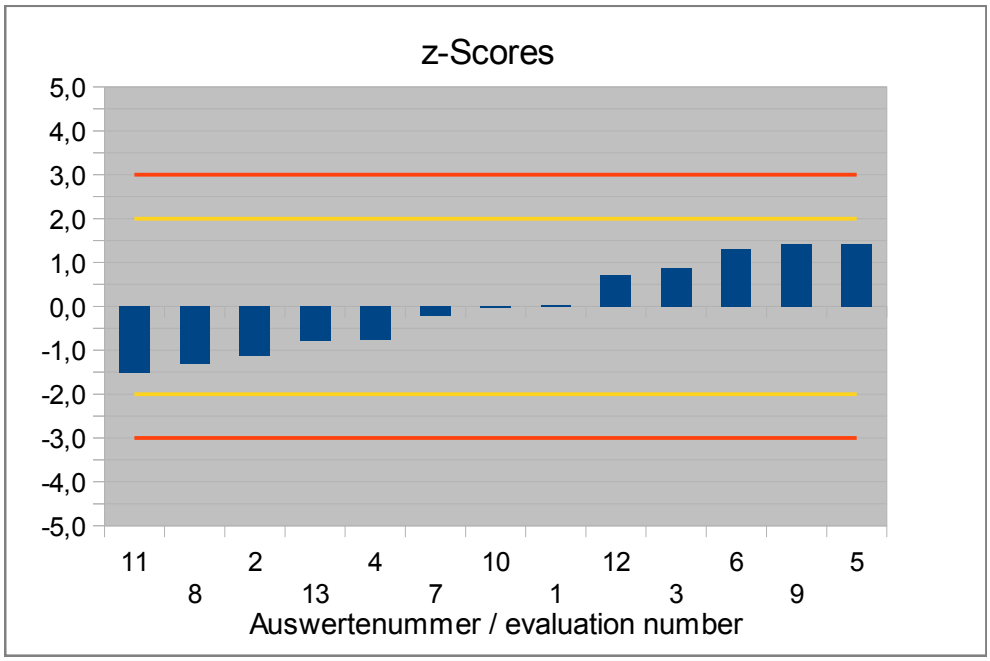

Abb. / Fig. 19: z-Scores L-Leucin / L-Leucine

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4.11 L-Lysin/L-Lysine in g/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	12
Number of outliers	1
Mean	1,31
Median	1,32
Robust Mean (X)	**1,33**
Robust standard deviation (S*)	**0,0968**
Number with 2 replicates	11
Repeatability SD (S_r)	0,0198
Repeatability (CV_r)	1,48%
Reproducibility SD (S_R)	0,0768
Reproducibility (CV_R)	5,73%
Target range:	
Target standard deviation σ_{pt}	**0,117**
Target standard deviation (for Information)	0,0508
lower limit of target range	**1,09**
upper limit of target range	**1,56**
Quotient S/σ_{pt}*	*0,83*
Standard uncertainty $U_{(Xpt)}$	*0,0349*
Results in the target range	*11*
Percent in the target range	*92%*

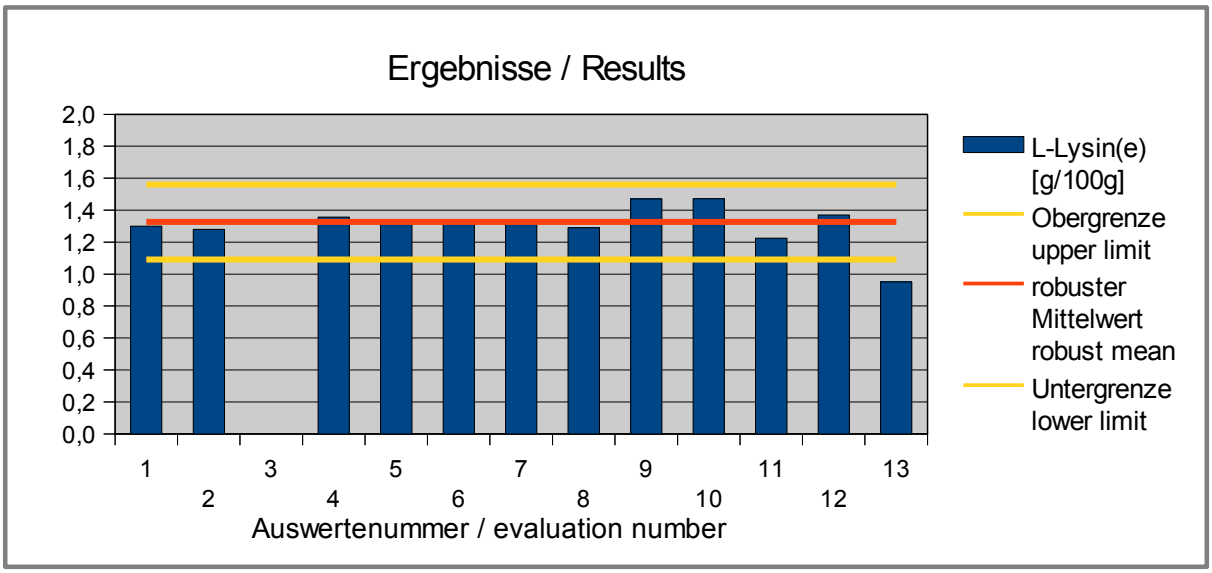

Abb. / Fig. 20: Ergebnisse L-Lysin / Results L-Lysine

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Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	L-Lysin(e) [g/100g]	Abweichung [g/100g] Deviation [g/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	1,30	-0,0261	-0,2	-0,5	
2	1,28	-0,0461	-0,4	-0,9	
3					
4	1,36	0,0304	0,3	0,6	
5	1,32	-0,0061	-0,1	-0,1	
6	1,33	0,0039	0,0	0,1	
7	1,32	-0,0061	-0,1	-0,1	
8	1,29	-0,0361	-0,3	-0,7	
9	1,47	0,144	1,2	2,8	
10	1,47	0,146	1,2	2,9	
11	1,22	-0,101	-0,9	-2,0	
12	1,37	0,0439	0,4	0,9	
13	0,950	-0,376	-3,2	-7,4	

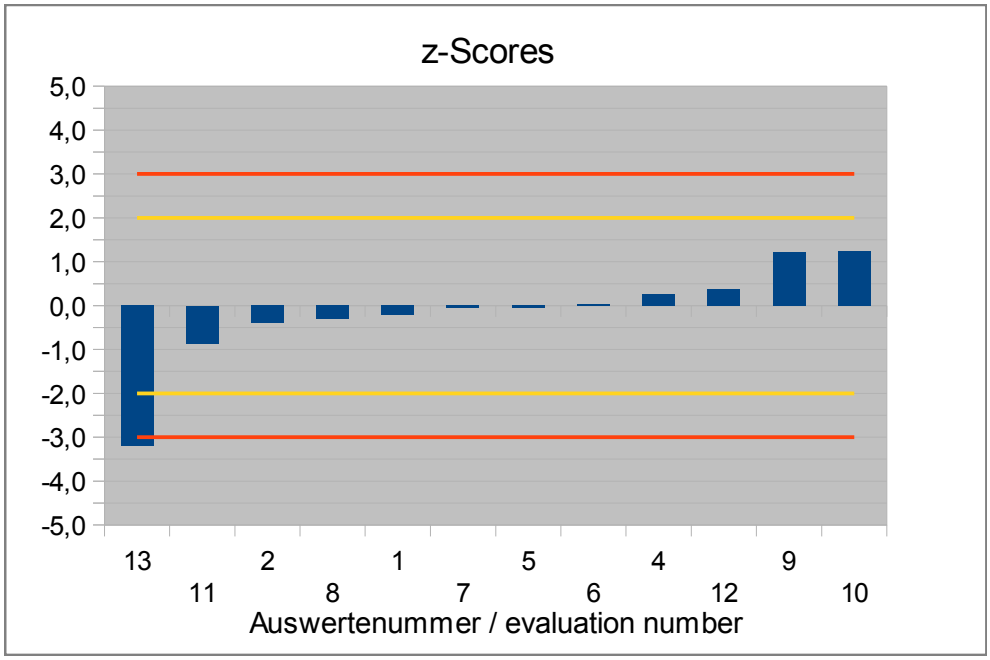

Abb. / Fig. 21: z-Scores L-Lysin / L-Lysine

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4.12 L-Methionin/L-Methionine in g/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	13
Number of outliers	0
Mean	0,396
Median	0,390
Robust Mean (X)	**0,398**
Robust standard deviation (S*)	**0,0486**
Number with 2 replicates	13
Repeatability SD (S_r)	0,0159
Repeatability (CV_r)	4,03%
Reproducibility SD (S_R)	0,0474
Reproducibility (CV_R)	12,0%
Target range:	
Target standard deviation σ_{pt}	**0,0266**
Target standard deviation (for Information)	0,0183
lower limit of target range	**0,345**
upper limit of target range	**0,451**
Quotient S/σ_{pt}*	*1,8*
Standard uncertainty $U_{(Xpt)}$	*0,0168*
Results in the target range	*10*
Percent in the target range	*77%*

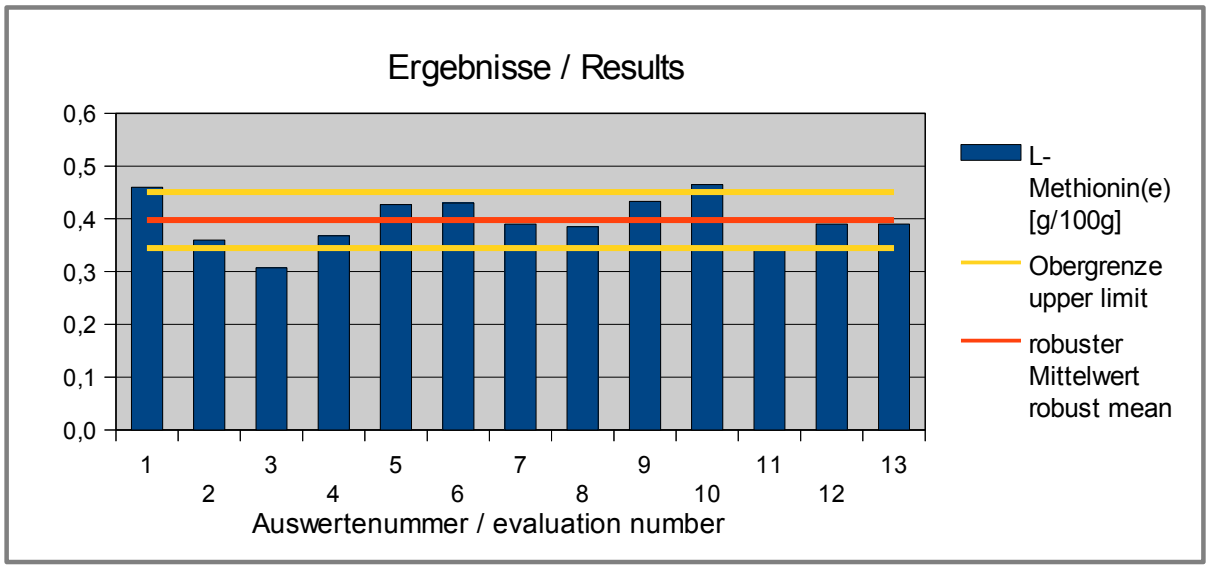

Abb. / Fig. 22: Ergebnisse L-Methionin / Results L-Methionine

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Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	L-Methionin(e) [g/100g]	Abweichung [g/100g] Deviation [g/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	0,460	0,0623	2,3	3,4	
2	0,360	-0,0377	-1,4	-2,1	
3	0,307	-0,0907	-3,4	-5,0	
4	0,368	-0,0297	-1,1	-1,6	
5	0,427	0,0293	1,1	1,6	
6	0,430	0,0323	1,2	1,8	
7	0,390	-0,0077	-0,3	-0,4	
8	0,385	-0,0127	-0,5	-0,7	
9	0,433	0,0353	1,3	1,9	
10	0,465	0,0673	2,5	3,7	
11	0,348	-0,0502	-1,9	-2,7	
12	0,390	-0,0077	-0,3	-0,4	
13	0,390	-0,0077	-0,3	-0,4	

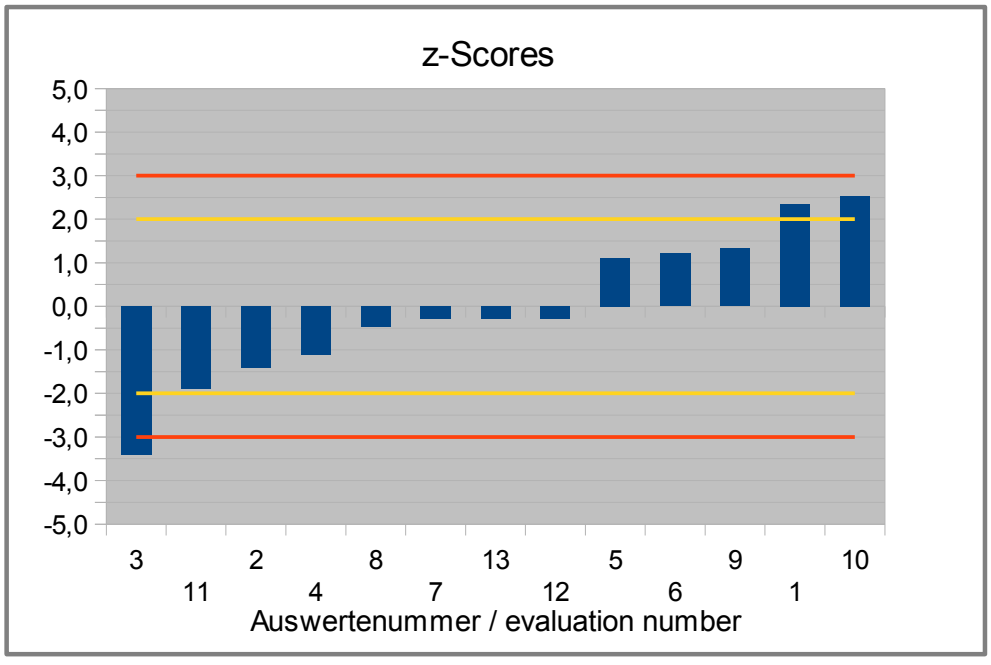

Abb. / Fig. 23: z-Scores L-Methionin / L-Methionine

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4.13 L-Phenylalanin/L-Phenylalanine in g/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	13
Number of outliers	0
Mean	0,760
Median	0,767
Robust Mean (X)	**0,761**
Robust standard deviation (S*)	**0,0551**
Number with 2 replicates	13
Repeatability SD (S_r)	0,0154
Repeatability (CV_r)	2,03%
Reproducibility SD (S_R)	0,0672
Reproducibility (CV_R)	8,86%
Target range:	
Target standard deviation σ_{pt}	**0,0317**
Target standard deviation (for Information)	0,0370
lower limit of target range	**0,698**
upper limit of target range	**0,825**
Quotient S/σ_{pt}*	*1,7*
Standard uncertainty $U_{(Xpt)}$	*0,0191*
Results in the target range	*10*
Percent in the target range	*77%*

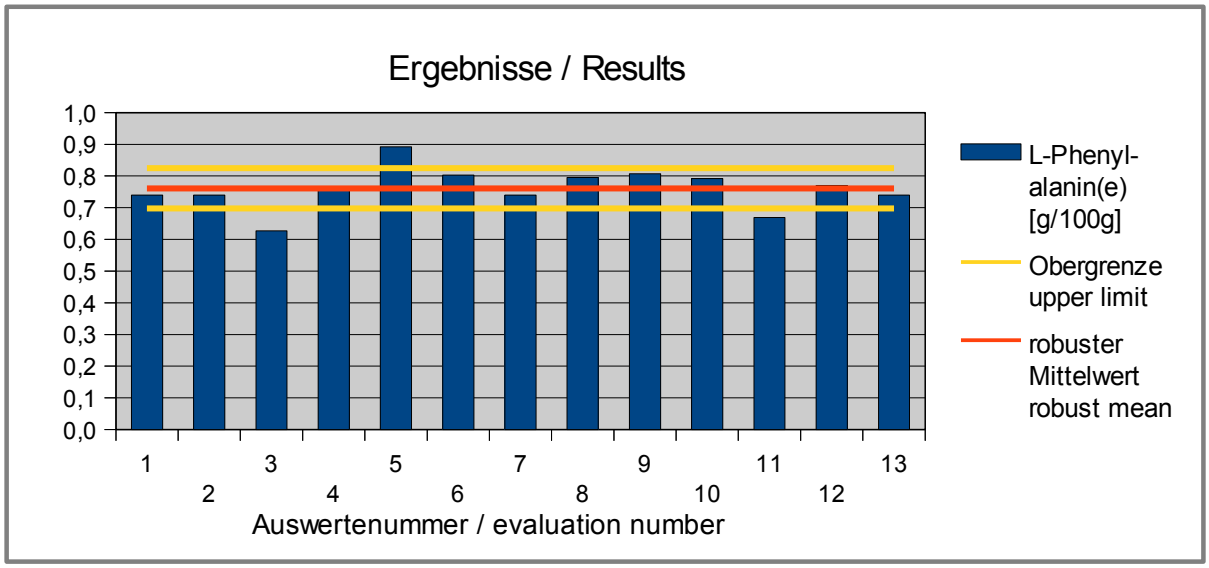

Abb. / Fig. 24: Ergebnisse L-Phenylalanin / Results L-Phenylalanine

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Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	L-Phenyl-alanin(e) [g/100g]	Abweichung [g/100g] Deviation [g/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	0,740	-0,0211	-0,7	-0,6	
2	0,740	-0,0211	-0,7	-0,6	
3	0,627	-0,1341	-4,2	-3,6	
4	0,767	0,0054	0,2	0,1	
5	0,892	0,1309	4,1	3,5	
6	0,803	0,0419	1,3	1,1	
7	0,740	-0,0211	-0,7	-0,6	
8	0,795	0,0339	1,1	0,9	
9	0,807	0,0459	1,4	1,2	
10	0,792	0,0309	1,0	0,8	
11	0,669	-0,0918	-2,9	-2,5	
12	0,770	0,0089	0,3	0,2	
13	0,740	-0,0211	-0,7	-0,6	

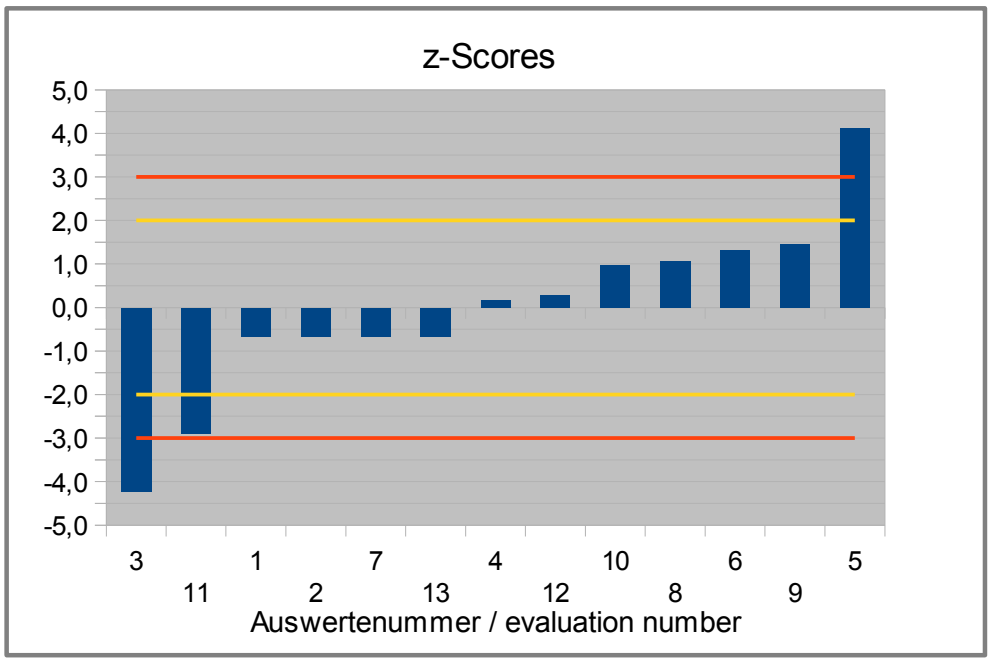

Abb. / Fig. 25: z-Scores L-Phenylalanin / L-Phenylalanine

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4.14 L-Prolin/L-Proline in g/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	13
Number of outliers	-
Mean	1,61
Median	1,55
Robust Mean (X)	**1,54**
Robust standard deviation (S*)	**0,0726**
Number with 2 replicates	11
Repeatability SD (S_r)	0,0139
Repeatability (CV_r)	0,91%
Reproducibility SD (S_R)	0,0633
Reproducibility (CV_R)	4,12%
Target range:	
Target standard deviation σ_{pt}	**0,121**
Target standard deviation (for Information)	0,0578
lower limit of target range	**1,30**
upper limit of target range	**1,79**
Quotient S/σ_{pt}*	*0,60*
Standard uncertainty $U_{(Xpt)}$	*0,0252*
Results in the target range	*12*
Percent in the target range	*92%*

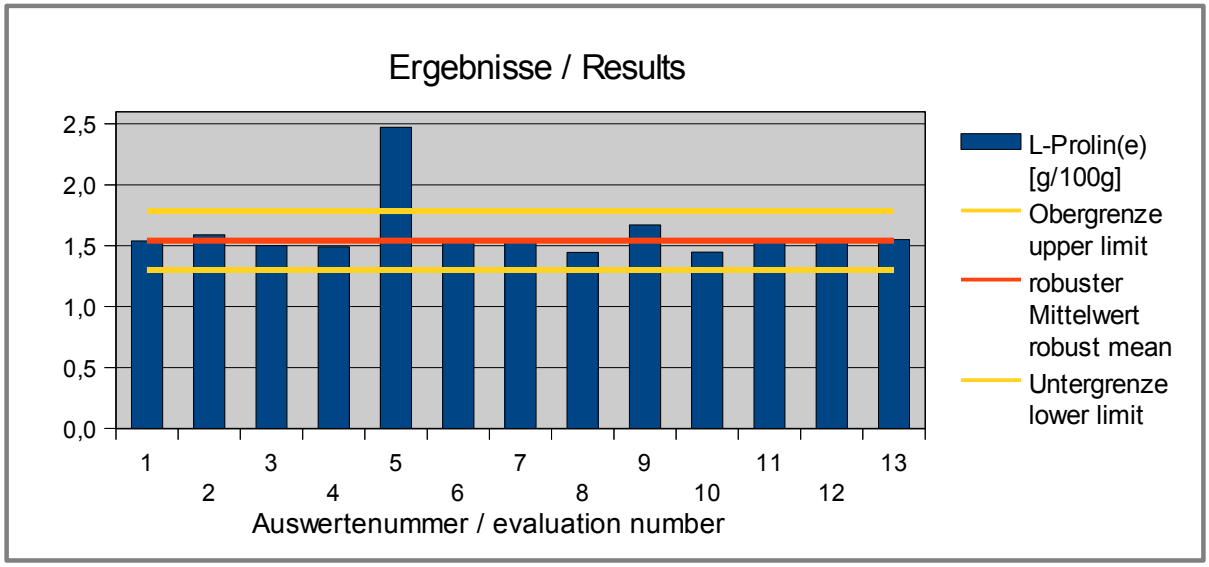

Abb. / Fig. 26: Ergebnisse L-Prolin / Results L-Proline

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Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	L-Prolin(e) [g/100g]	Abweichung [g/100g] Deviation [g/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	1,54	-0,002	0,0	0,0	
2	1,59	0,048	0,4	0,8	
3	1,50	-0,043	-0,4	-0,8	
4	1,49	-0,050	-0,4	-0,9	
5	2,47	0,930	7,7	16,1	
6	1,55	0,008	0,1	0,1	
7	1,55	0,008	0,1	0,1	
8	1,45	-0,097	-0,8	-1,7	
9	1,67	0,128	1,1	2,2	
10	1,45	-0,095	-0,8	-1,7	
11	1,55	0,003	0,0	0,1	
12	1,54	-0,002	0,0	0,0	
13	1,55	0,008	0,1	0,1	

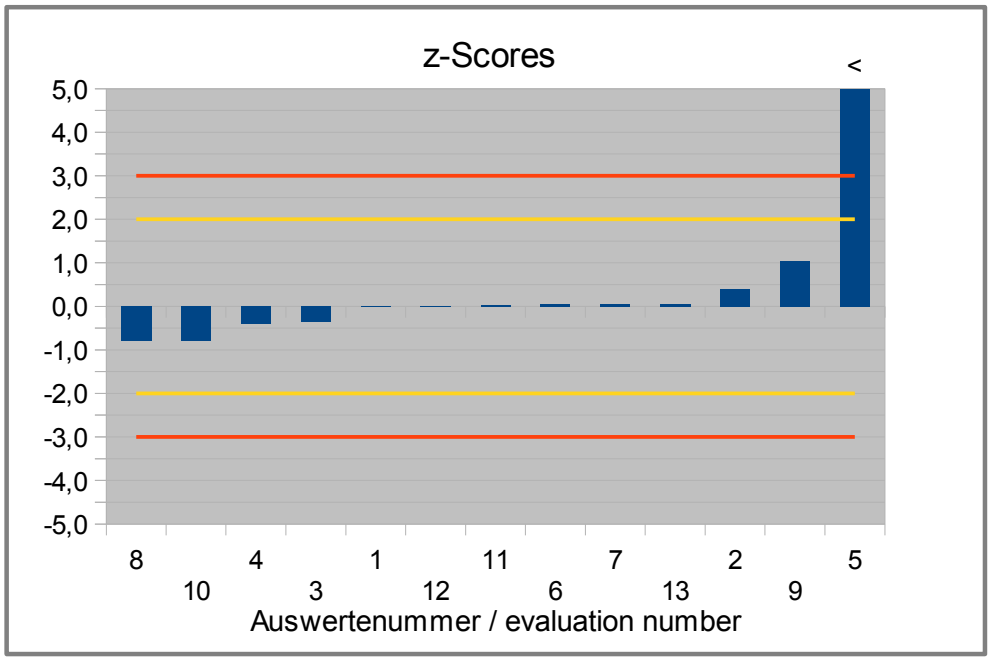

Abb. / Fig. 27: z-Scores L-Prolin / L-Proline

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4.15 L-Serin/L-Serine in g/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	12
Number of outliers	0
Mean	0,899
Median	0,891
Robust Mean (X)	**0,897**
Robust standard deviation (S*)	**0,0739**
Number with 2 replicates	12
Repeatability SD (S_r)	0,0128
Repeatability (CV_r)	1,43%
Reproducibility SD (S_R)	0,0691
Reproducibility (CV_R)	7,70%
Target range:	
Target standard deviation σ_{pt}	**0,0399**
Target standard deviation (for Information)	0,0365
lower limit of target range	**0,817**
upper limit of target range	**0,977**
Quotient S/σ_{pt}*	*1,9*
Standard uncertainty $U_{(Xpt)}$	*0,0267*
Results in the target range	*8*
Percent in the target range	*67%*

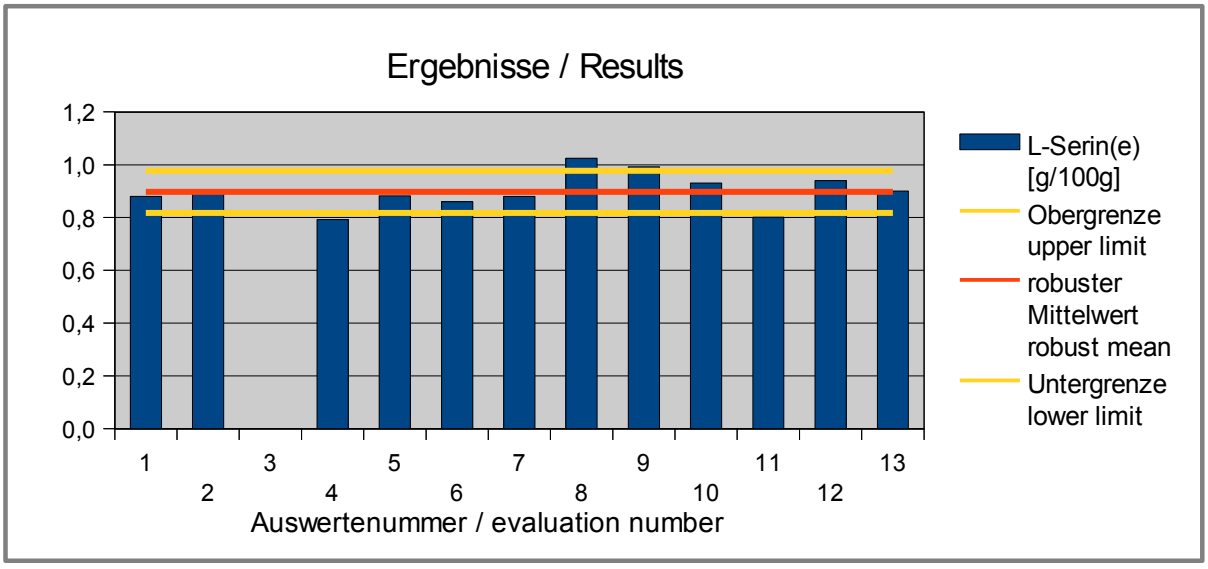

Abb. / Fig. 28: Ergebnisse L-Serin / Results L-Serine

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Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	L-Serin(e) [g/100g]	Abweichung [g/100g] Deviation [g/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	0,880	-0,0172	-0,4	-0,5	
2	0,900	0,0028	0,1	0,1	
3					
4	0,792	-0,1052	-2,6	-2,9	
5	0,882	-0,0152	-0,4	-0,4	
6	0,860	-0,0372	-0,9	-1,0	
7	0,880	-0,0172	-0,4	-0,5	
8	1,03	0,1278	3,2	3,5	
9	0,992	0,0948	2,4	2,6	
10	0,931	0,0338	0,8	0,9	
11	0,801	-0,0959	-2,4	-2,6	
12	0,940	0,0428	1,1	1,2	
13	0,900	0,0028	0,1	0,1	

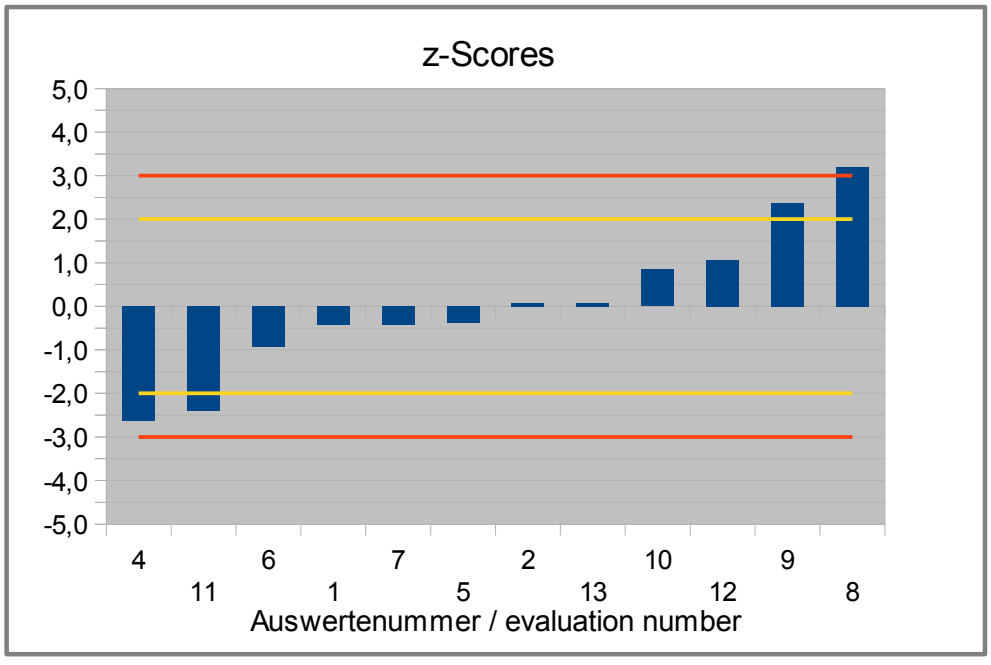

Abb. / Fig. 29: z-Scores L-Serin / L-Serine

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4.16 L-Threonin/L-Threonine in g/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results°	12
Number of outliers	1
Mean	0,761
Median	0,762
Robust Mean (X)	**0,761**
Robust standard deviation (S*)	**0,0516**
Number with 2 replicates	12
Repeatability SD (S_r)	0,0118
Repeatability (CV_r)	1,55%
Reproducibility SD (S_R)	0,0459
Reproducibility (CV_R)	6,04%
Target range:	
Target standard deviation σ_{pt}	**0,0428**
Target standard deviation (for Information)	0,0317
lower limit of target range	**0,675**
upper limit of target range	**0,846**
Quotient S/σ_{pt}*	*1,2*
Standard uncertainty $U_{(Xpt)}$	*0,0186*
Results in the target range	*12*
Percent in the target range	*100%*

° *without result no. 3 (excluded)*

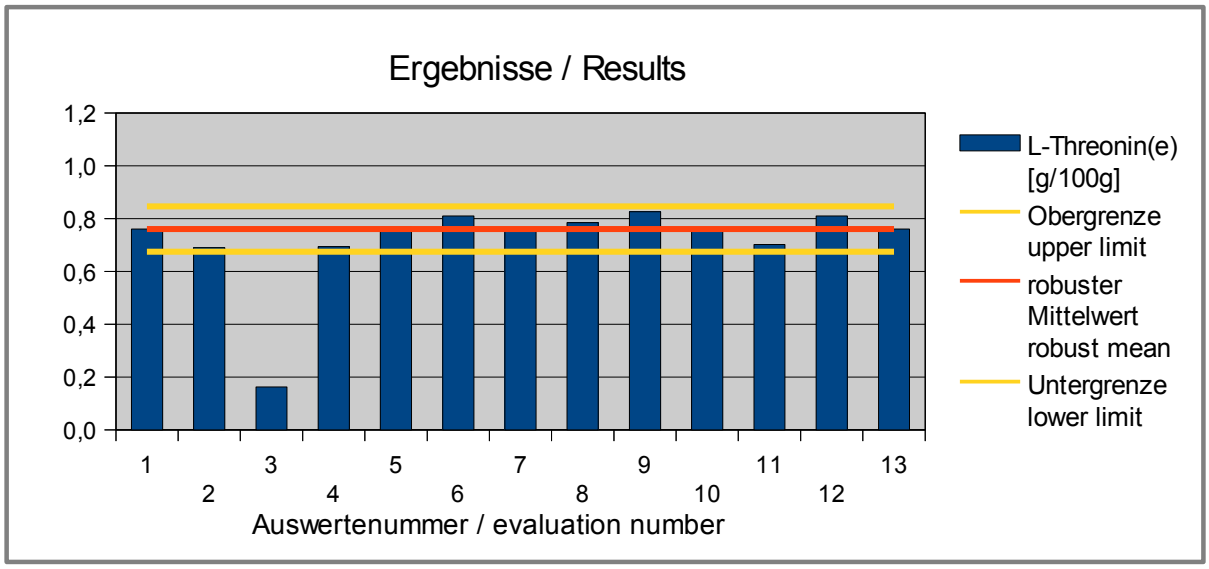

Abb. / Fig. 30: Ergebnisse L-Threonin / Results L-Threonine

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Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	L-Threonin(e) [g/100g]	Abweichung [g/100g] Deviation [g/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	0,760	-0,0006	0,0	0,0	
2	0,690	-0,0706	-1,6	-2,2	
3	0,162				Result excluded, mean calculated by DLA
4	0,694	-0,0671	-1,6	-2,1	
5	0,767	0,0064	0,1	0,2	
6	0,810	0,0494	1,2	1,6	
7	0,760	-0,0006	0,0	0,0	
8	0,785	0,0244	0,6	0,8	
9	0,826	0,0654	1,5	2,1	
10	0,764	0,0034	0,1	0,1	
11	0,702	-0,0591	-1,4	-1,9	
12	0,810	0,0494	1,2	1,6	
13	0,760	-0,0006	0,0	0,0	

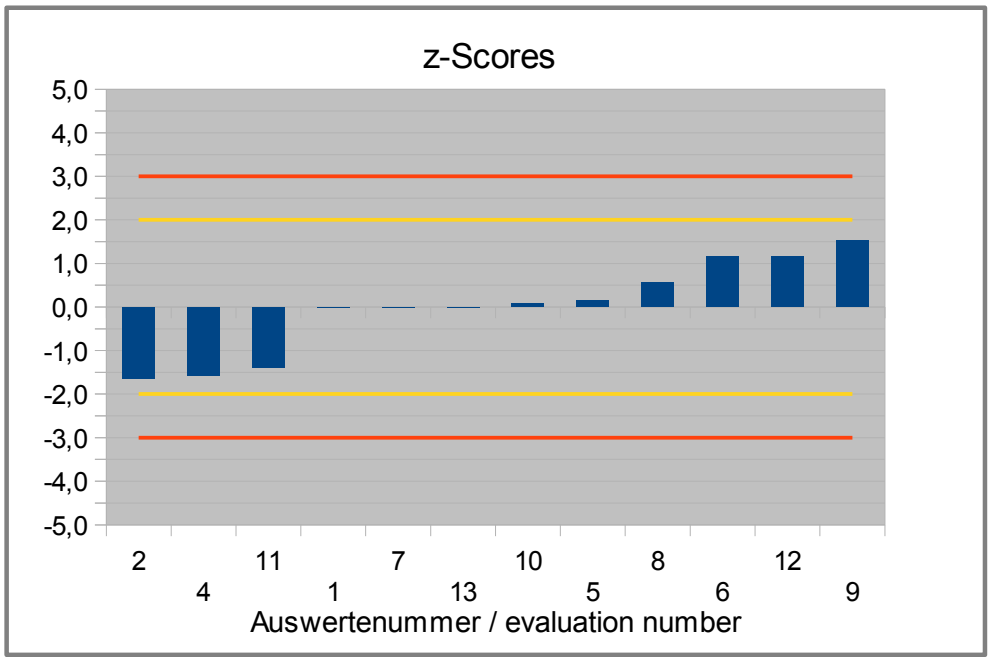

Abb. / Fig. 31: z-Scores L-Threonin / L-Threonine

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4.17 L-Thryptophan in g/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	10
Number of outliers	0
Mean	0,218
Median	0,210
Robust Mean (X)	**0,213**
Robust standard deviation (S*)	**0,0512**
Number with 2 replicates	10
Repeatability SD (S_r)	0,0493
Repeatability (CV_r)	22,7%
Reproducibility SD (S_R)	0,0664
Reproducibility (CV_R)	30,5%
Target range:	
Target standard deviation σ_{pt}'	**0,0252**
Target standard deviation (for Information)	0,0108
lower limit of target range	**0,163**
upper limit of target range	**0,264**
Quotient S/σ_{pt}'*	*2,0*
Standard uncertainty $U_{(Xpt)}$	*0,0203*
Results in the target range	*6*
Percent in the target range	*60%*

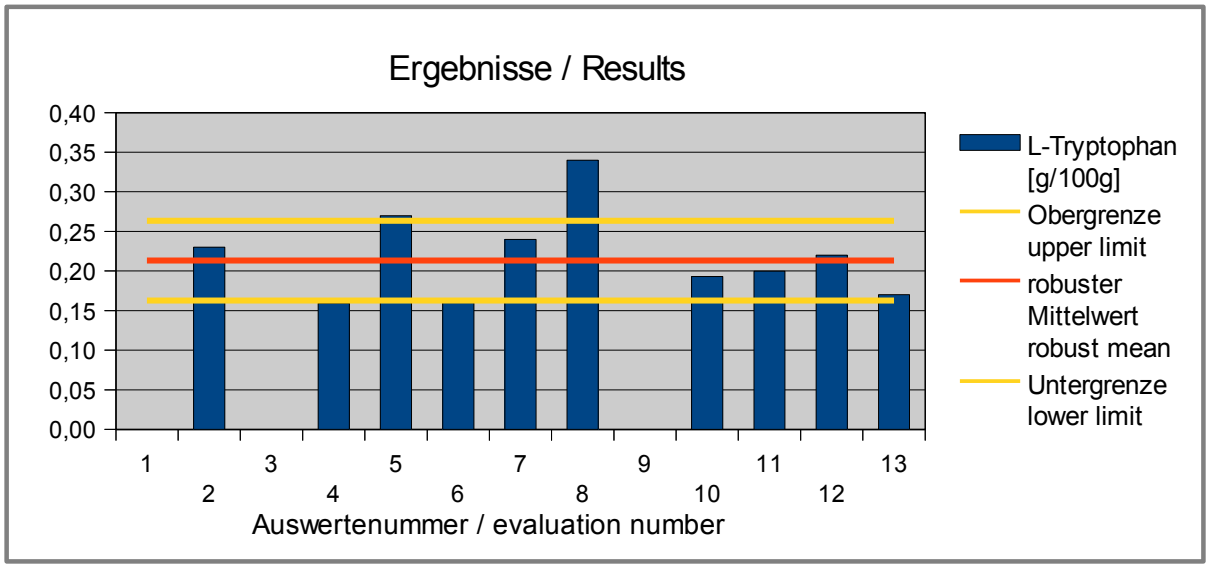

Abb. / Fig. 32: Ergebnisse / Results L-Tryptophan

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Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	L-Tryptophan [g/100g]	Abweichung [g/100g] Deviation [g/100g]	z'-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1					
2	0,230	0,0168	0,7	1,6	
3					
4	0,159	-0,0539	-2,1	-5,0	
5	0,270	0,0568	2,3	5,3	
6	0,160	-0,0532	-2,1	-4,9	
7	0,240	0,0268	1,1	2,5	
8	0,340	0,1268	5,0	11,8	
9					
10	0,193	-0,0202	-0,8	-1,9	
11	0,200	-0,0134	-0,5	-1,2	
12	0,220	0,0068	0,3	0,6	
13	0,170	-0,0432	-1,7	-4,0	

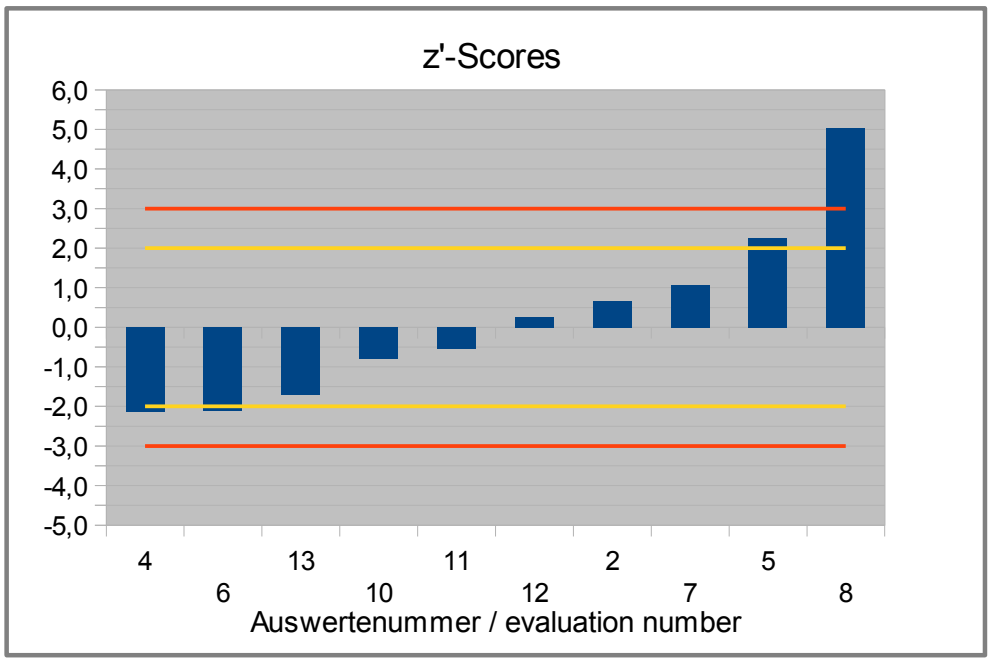

Abb. / Fig. 33: z-Scores L-Tryptophan

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4.18 L-Tyrosin/L-Tyrosine in g/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	12
Number of outliers	0
Mean	0,648
Median	0,660
Robust Mean (X)	**0,653**
Robust standard deviation (S*)	**0,0669**
Number with 2 replicates	12
Repeatability SD (S_r)	0,0136
Repeatability (CV_r)	2,09%
Reproducibility SD (S_R)	0,0703
Reproducibility (CV_R)	10,8%
Target range:	
Target standard deviation σ_{pt}	**0,0434**
Target standard deviation (for Information)	0,0279
lower limit of target range	**0,567**
upper limit of target range	**0,740**
Quotient S/σ_{pt}*	*1,5*
Standard uncertainty $U_{(Xpt)}$	*0,0241*
Results in the target range	*10*
Percent in the target range	*83%*

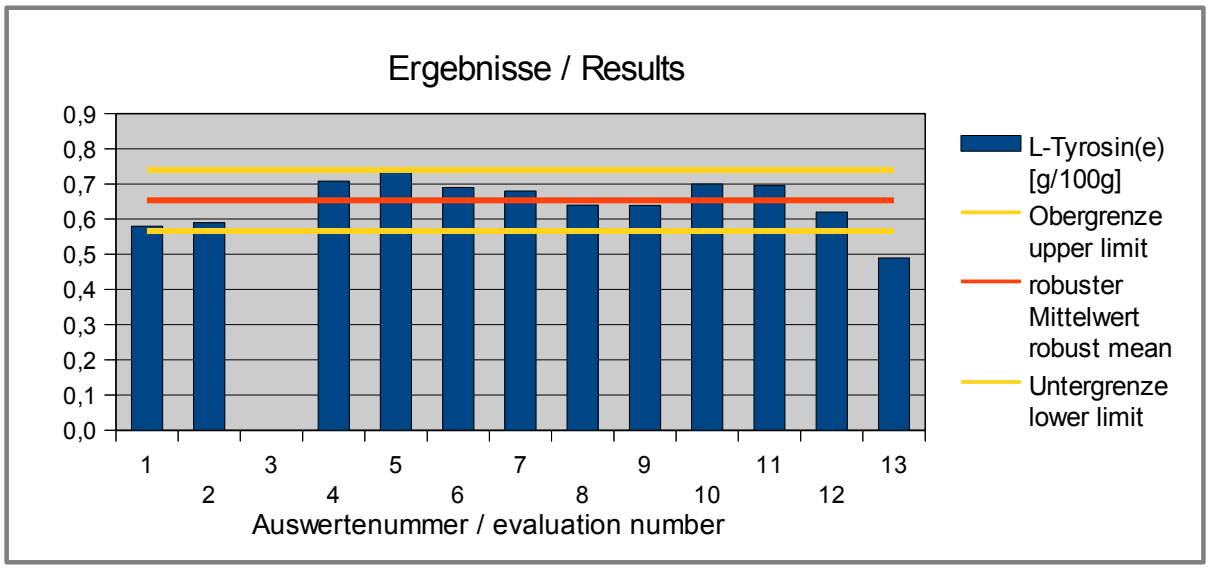

Abb. / Fig. 34: Ergebnisse L-Tyrosin / Results L-Tyrosine

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Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	L-Tyrosin(e) [g/100g]	Abweichung [g/100g] Deviation [g/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	0,580	-0,0733	-1,7	-2,6	
2	0,590	-0,0633	-1,5	-2,3	
3					
4	0,708	0,0542	1,2	1,9	
5	0,745	0,0917	2,1	3,3	
6	0,690	0,0367	0,8	1,3	
7	0,680	0,0267	0,6	1,0	
8	0,640	-0,0133	-0,3	-0,5	
9	0,639	-0,0143	-0,3	-0,5	
10	0,700	0,0467	1,1	1,7	
11	0,696	0,0422	1,0	1,5	
12	0,620	-0,0333	-0,8	-1,2	
13	0,490	-0,163	-3,8	-5,9	

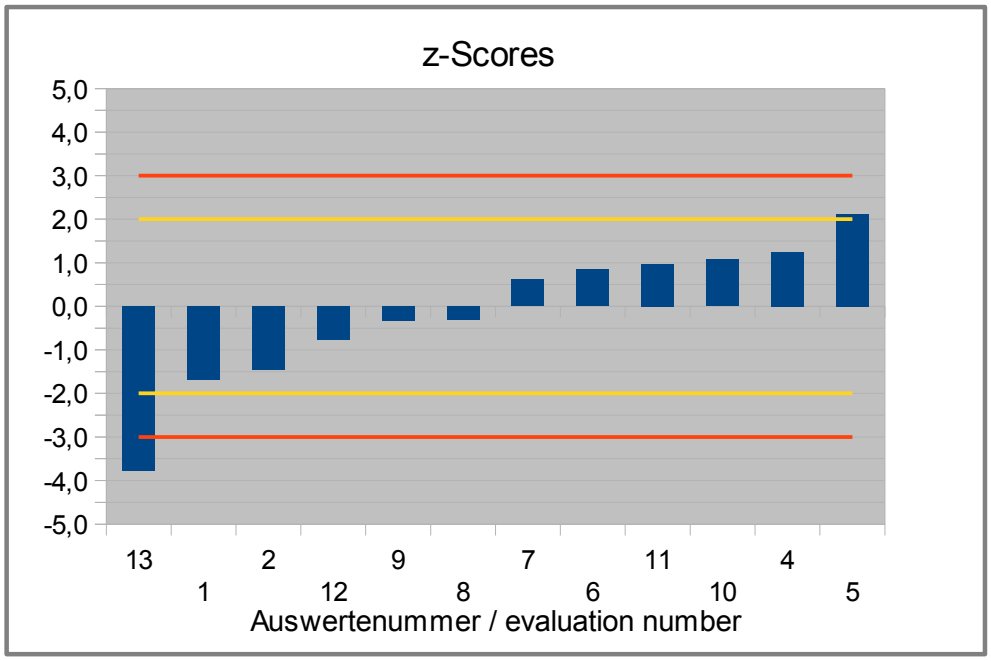

Abb. / Fig. 35: z-Scores L-Tyrosin / L-Tyrosine

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4.19 L-Valin/L-Valine in g/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	13
Number of outliers	0
Mean	1,01
Median	1,03
Robust Mean (X)	**1,01**
Robust standard deviation (S*)	**0,0600**
Number with 2 replicates	13
Repeatability SD (S_r)	0,0180
Repeatability (CV_r)	1,78%
Reproducibility SD (S_R)	0,0681
Reproducibility (CV_R)	6,73%
Target range:	
Target standard deviation σ_{pt}	**0,0596**
Target standard deviation (for Information)	0,0405
lower limit of target range	**0,895**
upper limit of target range	**1,13**
Quotient S/σ_{pt}*	*1,0*
Standard uncertainty $U_{(Xpt)}$	*0,0208*
Results in the target range	*11*
Percent in the target range	*85%*

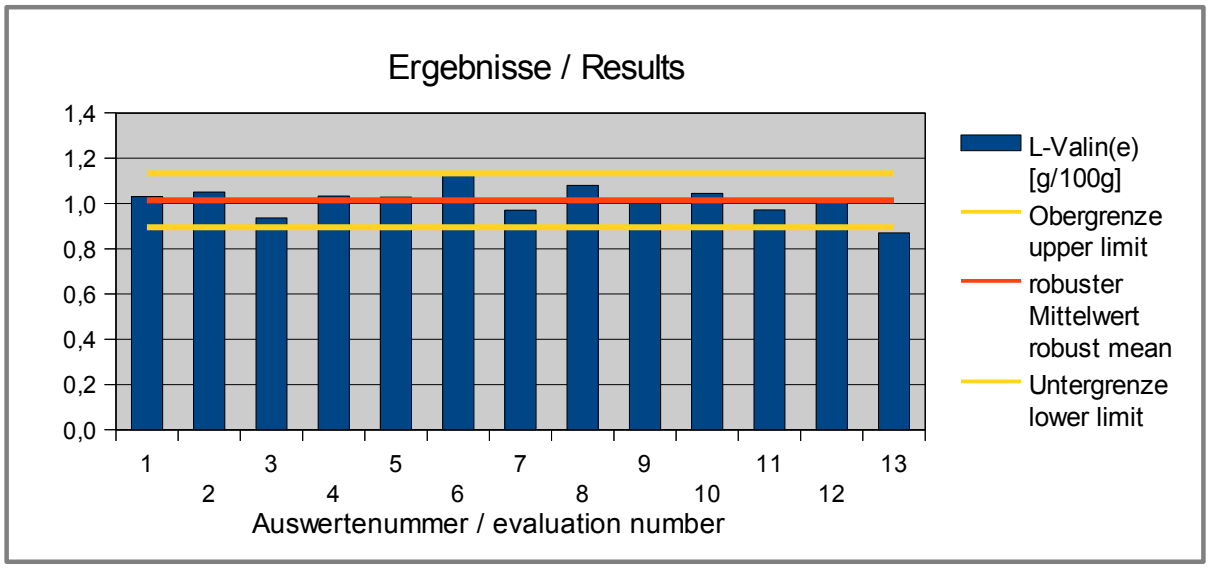

Abb. / Fig. 36: Ergebnisse L-Valin / Results L-Valine

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Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	L-Valin(e) [g/100g]	Abweichung [g/100g] Deviation [g/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	1,03	0,0160	0,3	0,4	
2	1,05	0,0360	0,6	0,9	
3	0,936	-0,0780	-1,3	-1,9	
4	1,03	0,0190	0,3	0,5	
5	1,03	0,0140	0,2	0,3	
6	1,14	0,126	2,1	3,1	
7	0,970	-0,0440	-0,7	-1,1	
8	1,08	0,0660	1,1	1,6	
9	1,00	-0,0140	-0,2	-0,3	
10	1,05	0,0310	0,5	0,8	
11	0,972	-0,0420	-0,7	-1,0	
12	1,01	-0,0040	-0,1	-0,1	
13	0,870	-0,144	-2,4	-3,6	

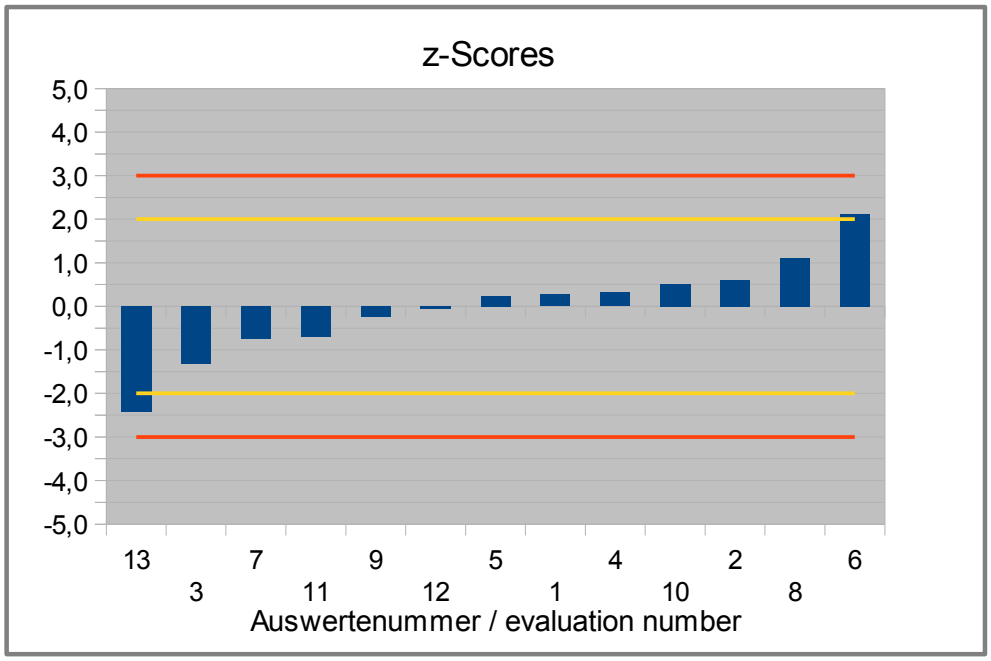

Abb. / Fig. 37: z-Scores L-Valin / L-Valine

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5. Documentation

5.1 Details by the participants

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

5.1.1 Primary Data

Parameter	Partici-pant	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana-lysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifi-cation	Incl. RR	Recovery rate
					day/month					yes/no	in %
L-Alanine	1	g/100g	8	78	13.10.	0,57	0,57	0,57		no	
	2	g/100g	52	34	29.09.18	0,53	0,53	0,53		no	
	3	g/100g	66	20	23.10.18	0,478	0,452	0,504		no	
	4	g/100g	1	85	15.10.18	0,5925	0,587	0,598	0,05	no	
	5	g/100g	18	68	27.09.18	0,626	0,628	0,624	0,006	no	x
	6	g/100g	10	76	19.10.18	0,63	0,63	0,63	0,03	no	
	7	g/100g	29	57	08.10.18	0,56	0,56	0,56	0,05	no	na
	8	g/100g	74	12	24.09.18	0,58	0,59	0,57		no	
	9	g/100g	30	56	10.10.18	0,654	0,651	0,658	0,2	no	
	10	g/100g	11	75	27.09.18	0,605	0,59	0,621	0,02	no	
	11	g/100g	37	49	24.09.18	0,7525	0,7525	0,7525	0,0018	no	
	12	g/100g	9	77	21.09.	0,56	0,56	0,56		no	
	13	g/100g	21	65	18/09	0,61	0,61	0,6	0,05	no	-

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Parameter	Partici-pant	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana-lysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifi-cation	Incl. RR	Recovery rate
					day/month					yes/no	in %
L-Arginine	1	g/100g	8	78	13.10.	0,48	0,48	0,49		no	
	2	g/100g	52	34		0,49	0,47	0,5			
	3	g/100g	66	20							
	4	g/100g	1	85	15.10.18	0,531	0,526	0,536	0,05	no	
	5	g/100g	18	68	04.10.18	0,514	0,507	0,52	0,013	no	x
	6	g/100g	10	76	19.10.18	0,55	0,55	0,54	0,07	no	
	7	g/100g	29	57	08.10.18	0,52	0,52	0,52	0,05	no	na
	8	g/100g	74	12	24.09.18	0,505	0,51	0,5		no	
	9	g/100g	30	56	10.10.18	0,638	0,641	0,635	0,2	no	
	10	g/100g	11	75	26.09.18	0,54	0,529	0,552	0,08		
	11	g/100g	37	49	24.09.18	0,3955	0,398	0,393	0,0096	no	
	12	g/100g	9	77	21.09.	0,51	0,51	0,51			
	13	g/100g	21	65	18/09	0,61	0,61	0,61	0,05	no	-

Parameter	Partici-pant	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana-lysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifi-cation	Incl. RR	Recovery rate
					day/month					yes/no	in %
L-Aspartic acid	1	g/100g	8	78	13.10.	1,31	1,31	1,32		no	
	2	g/100g	52	34		1,31	1,32	1,3			
	3	g/100g	66	20	23.10.18	1,69	1,743	1,636		no	
	4	g/100g	1	85	15.10.18	1,365	1,361	1,369	0,05	no	
	5	g/100g	18	68	25.09.18	1,562	1,559	1,565	0,009	no	x
	6	g/100g	10	76	19.10.18	1,4	1,4	1,39	0,04	no	
	7	g/100g	29	57	08.10.18	1,31	1,3	1,31	0,05	no	na
	8	g/100g	74	12	24.09.18	1,305	1,32	1,29		no	
	9	g/100g	30	56	10.10.18	1,47	1,49	1,46	0,1	no	
	10	g/100g	11	75	26.09.18	1,536	1,488	1,585	0,02		
	11	g/100g	37	49	24.09.18	1,237	1,2505	1,2235	0,0065	no	
	12	g/100g	9	77	21.09.	1,27	1,28	1,27			
	13	g/100g	21	65	18/09	1,34	1,35	1,33	0,05	no	-

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Parameter	Partici-pant	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana-lysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifi-cation	Incl. RR	Recovery rate
					day/month					yes/no	in %
L-Cysteine	1	g/100g	8	78							
	2	g/100g	52	34							
	3	g/100g	66	20							
	4	g/100g	1	85	15.10.18				0,05	no	
	5	g/100g	18	68							
	6	g/100g	10	76							
	7	g/100g	29	57	08.10.18	0,16	0,16	0,16	0,05	no	na
	8	g/100g	74	12							
	9	g/100g	30	56							
	10	g/100g	11	75	15.10.18	0,184	0,19	0,178	0,04		
	11	g/100g	37	49	24.09.18	N/A	N/A	N/A	N/A	no	
	12	g/100g	9	77							
	13	g/100g	21	65	-	-	-	-	-	-	-

Parameter	Partici-pant	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana-lysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifi-cation	Incl. RR	Recovery rate
					day/month					yes/no	in %
L-Cystine	1	g/100g	8	78							
	2	g/100g	52	34		0,18	0,17	0,18			
	3	g/100g	66	20							
	4	g/100g	1	85	15.10.18				0,05	no	
	5	g/100g	18	68	19.10.18	0,192	0,211	0,173	0,03	no	x
	6	g/100g	10	76	19.10.18	0,18	0,17	0,18	<0,1	no	
	7	g/100g	29	57	08.10.18	0,16	0,16	0,16	0,05	no	na
	8	g/100g	74	12	24.09.18	0,145	0,15	0,14		no	
	9	g/100g	30	56							
	10	g/100g	11	75							
	11	g/100g	37	49	24.09.18	0,113	0,1305	0,0955	0,0155	no	
	12	g/100g	9	77	26.09.	0,17	0,17	0,18			
	13	g/100g	21	65	18/09	0,16	0,16	0,16	0,05	no	-

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Parameter	Partici-pant	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana-lysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifi-cation	Incl. RR	Recovery rate
					day/month					yes/no	in %
L-Glutamic acid	1	g/100g	8	78	13.10.	3,53	3,52	3,54		no	
	2	g/100g	52	34		3,48	3,45	3,5			
	3	g/100g	66	20							
	4	g/100g	1	85	15.10.18	3,2705	3,296	3,245	0,05	no	
	5	g/100g	18	68	24.09.18	3,899	3,854	3,823	0,01	no	x
	6	g/100g	10	76	19.10.18	3,68	3,69	3,66	0,05	no	
	7	g/100g	29	57	08.10.18	3,51	3,5	3,51	0,05	no	na
	8	g/100g	74	12	24.09.18	3,415	3,39	3,44		no	
	9	g/100g	30	56	10.10.18	3,8	3,84	3,76	0,1	no	
	10	g/100g	11	75	26.09.18	3,734	3,632	3,837	0,03		
	11	g/100g	37	49	24.09.18	2,845	2,82	2,87	0,0038	no	
	12	g/100g	9	77	26.09.	3,49	3,5	3,49			
	13	g/100g	21	65	18/09	3,33	3,36	3,3	0,05	no	-

Parameter	Partici-pant	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana-lysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifi-cation	Incl. RR	Recovery rate
					day/month					yes/no	in %
Glycine	1	g/100g	8	78	13.10.	0,31	0,31	0,31		no	
	2	g/100g	52	34		0,3	0,3	0,29			
	3	g/100g	66	20	23.10.18	0,141	0,161	0,12		no	
	4	g/100g	1	85	15.10.18	0,3365	0,335	0,338	0,05	no	
	5	g/100g	18	68	17.10.18	0,426	0,426	0,425	0,006	no	x
	6	g/100g	10	76	19.10.18	0,33	0,33	0,33	0,03	no	
	7	g/100g	29	57	08.10.18	0,31	0,31	0,31	0,05	no	na
	8	g/100g	74	12	24.09.18	0,325	0,33	0,32		no	
	9	g/100g	30	56	10.10.18	0,337	0,336	0,337	0,2	no	
	10	g/100g	11	75	26.09.18	0,341	0,335	0,348	0,03		
	11	g/100g	37	49	24.09.18	0,517	0,5155	0,5185	0,0044	no	
	12	g/100g	9	77	21.09.	0,3	0,3	0,3			
	13	g/100g	21	65	18/09	0,32	0,33	0,31	0,05	no	-

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Parameter	Partici-pant	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana-lysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifi-cation	Incl. RR	Recovery rate
					day/month					yes/no	in %
L-Histidine	1	g/100g	8	78	13.10.	0,4	0,4	0,4		no	
	2	g/100g	52	34		0,41	0,38	0,43			
	3	g/100g	66	20							
	4	g/100g	1	85	15.10.18	0,4575	0,464	0,451	0,05	no	
	5	g/100g	18	68	17.10.18	0,518	0,506	0,529	0,009	no	x
	6	g/100g	10	76	19.10.18	0,44	0,44	0,44	0,07	no	
	7	g/100g	29	57	08.10.18	0,43	0,43	0,43	0,05	no	na
	8	g/100g	74	12	24.09.18	0,375	0,37	0,38		no	
	9	g/100g	30	56	10.10.18	0,416	0,423	0,41	0,2	no	
	10	g/100g	11	75	26.09.18	0,493	0,482	0,504	0,2		
	11	g/100g	37	49	24.09.18	0,4098	0,4045	0,415	0,0071	no	
	12	g/100g	9	77	21.09.	0,45	0,45	0,46			
	13	g/100g	21	65	18/09	0,36	0,37	0,35	0,05	no	-

Parameter	Partici-pant	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana-lysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifi-cation	Incl. RR	Recovery rate
					day/month					yes/no	in %
L-Isoleucine	1	g/100g	8	78	13.10.	0,92	0,91	0,92		no	
	2	g/100g	52	34		0,85	0,85	0,85			
	3	g/100g	66	20	23.10.18	0,765	0,77	0,761		no	
	4	g/100g	1	85	15.10.18	0,88	0,885	0,875	0,05	no	
	5	g/100g	18	68	27.09.18	0,906	0,928	0,883	0,009	no	x
	6	g/100g	10	76	19.10.18	0,99	0,99	0,99	0,05	no	
	7	g/100g	29	57	08.10.18	0,87	0,87	0,87	0,05	no	na
	8	g/100g	74	12	24.09.18	0,85	0,83	0,87		no	
	9	g/100g	30	56	10.10.18	0,851	0,859	0,843	0,1	no	
	10	g/100g	11	75	26.09.18	0,911	0,887	0,936	0,02		
	11	g/100g	37	49	24.09.18	0,818	0,822	0,814	0,0084	no	
	12	g/100g	9	77	21.09.	0,86	0,86	0,85			
	13	g/100g	21	65	18/09	0,74	0,75	0,73	0,05	no	-

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Parameter	Partici-pant	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana-lysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifi-cation	Incl. RR	Recovery rate
					day/month					yes/no	in %
L-Leucine	1	g/100g	8	78	13.10.	1,61	1,61	1,62		no	
	2	g/100g	52	34		1,51	1,51	1,51			
	3	g/100g	66	20	23.10.18	1,684	1,66	1,709		no	
	4	g/100g	1	85	15.10.18	1,541	1,522	1,56	0,05	no	
	5	g/100g	18	68	25.09.18	1,73	1,737	1,723	0,01	no	x
	6	g/100g	10	76	19.10.18	1,72	1,73	1,71	0,05	no	
	7	g/100g	29	57	08.10.18	1,59	1,58	1,59	0,05	no	na
	8	g/100g	74	12	24.09.18	1,495	1,48	1,51		no	
	9	g/100g	30	56	10.10.18	1,73	1,72	1,74	0,1	no	
	10	g/100g	11	75	26.09.18	1,605	1,565	1,645	0,02		
	11	g/100g	37	49	24.09.18	1,477	1,4895	1,4645	0,0054	no	
	12	g/100g	9	77	26.09.	1,67	1,68	1,66			
	13	g/100g	21	65	18/09	1,54	1,54	1,53	0,05	no	-

Parameter	Partici-pant	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana-lysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifi-cation	Incl. RR	Recovery rate
					day/month					yes/no	in %
L-Lysine	1	g/100g	8	78	13.10.	1,3	1,3	1,31		no	
	2	g/100g	52	34		1,28	1,28	1,28			
	3	g/100g	66	20							
	4	g/100g	1	85	15.10.18	1,3565	1,369	1,344	0,05	no	
	5	g/100g	18	68	27.09.18	1,32	1,301	1,339	0,06	no	x
	6	g/100g	10	76	19.10.18	1,33	1,33	1,32	0,06	no	
	7	g/100g	29	57	08.10.18	1,32	1,32	1,32	0,05	no	na
	8	g/100g	74	12	24.09.18	1,29	1,27	1,31		no	
	9	g/100g	30	56	10.10.18	1,47	1,48	1,46	0,15	no	
	10	g/100g	11	75	26.09.18	1,472	1,44	1,505	0,1		
	11	g/100g	37	49	24.09.18	1,2248	1,224	1,2255	0,007	no	
	12	g/100g	9	77	26.09.	1,37	1,37	1,36			
	13	g/100g	21	65	18/09	0,95	0,92	0,97	0,05	no	-

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Parameter	Partici-pant	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana-lysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifi-cation	Incl. RR	Recovery rate
					day/month					yes/no	in %
L-Methionine	1	g/100g	8	78	13.10.	0,46	0,45	0,47		no	
	2	g/100g	52	34		0,36	0,35	0,36			
	3	g/100g	66	20	23.10.18	0,307	0,303	0,31		no	
	4	g/100g	1	85	15.10.18	0,368	0,364	0,372	0,05	no	
	5	g/100g	18	68	04.10.18	0,427	0,418	0,435	0,03	no	x
	6	g/100g	10	76	19.10.18	0,43	0,397	0,46	<0,1	no	
	7	g/100g	29	57	08.10.18	0,39	0,39	0,38	0,05	no	na
	8	g/100g	74	12	24.09.18	0,385	0,39	0,38		no	
	9	g/100g	30	56	10.10.18	0,433	0,438	0,428	0,2	no	
	10	g/100g	11	75	15.10.18	0,465	0,448	0,482	0,03		
	11	g/100g	37	49	24.09.18	0,3475	0,341	0,354	0,0049	no	
	12	g/100g	9	77	26.09.	0,39	0,39	0,39			
	13	g/100g	21	65	18/09	0,39	0,39	0,38	0,05	no	-

Parameter	Partici-pant	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana-lysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifi-cation	Incl. RR	Recovery rate
					day/month					yes/no	in %
L-Phenyl-alanine	1	g/100g	8	78	13.10.	0,74	0,74	0,74		nein	
	2	g/100g	52	34		0,74	0,74	0,73			
	3	g/100g	66	20	23.10.18	0,627	0,605	0,641		nein	
	4	g/100g	1	85	15.10.18	0,7665	0,76	0,773	0,05	nein	
	5	g/100g	18	68	17.10.18	0,892	0,876	0,907	0,01	nein	x
	6	g/100g	10	76	19.10.18	0,803	0,803	0,803	0,06	nein	
	7	g/100g	29	57	08.10.18	0,74	0,74	0,74	0,05	nein	na
	8	g/100g	74	12	24.09.18	0,795	0,78	0,81		nein	
	9	g/100g	30	56	10.10.18	0,807	0,81	0,805	0,1	nein	
	10	g/100g	11	75	26.09.18	0,792	0,769	0,815	0,03		
	11	g/100g	37	49	24.09.18	0,6693	0,675	0,6635	0,008	nein	
	12	g/100g	9	77	26.09.	0,77	0,78	0,76			
	13	g/100g	21	65	18/09	0,74	0,73	0,74	0,05	nein	-

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Parameter	Partici-pant	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana-lysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifi-cation	Incl. RR	Recovery rate
					day/month					yes/no	in %
L-Proline	1	g/100g	8	78	13.10.	1,54	1,54	1,54		no	
	2	g/100g	52	34		1,59	1,59	1,58			
	3	g/100g	66	20	23.10.18	1,499				no	
	4	g/100g	1	85	15.10.18	1,492	1,493	1,491	0,05	no	
	5	g/100g	18	68	17.10.18	2,472	2,502	2,442	0,01	no	x
	6	g/100g	10	76	19.10.18	1,55	1,55	1,55	0,04	no	
	7	g/100g	29	57	08.10.18	1,55	1,54	1,55	0,05	no	na
	8	g/100g	74	12	24.09.18	1,445	1,43	1,46		no	
	9	g/100g	30	56	10.10.18	1,67	1,67	1,67	0,1	no	
	10	g/100g	11	75	09.10.18	1,447	1,468	1,425	0,1		
	11	g/100g	37	49	24.09.18	1,5453	1,543	1,5475	0,0043	no	
	12	g/100g	9	77	21.09.	1,54	1,55	1,53			
	13	g/100g	21	65	18/09	1,55	1,53	1,56	0,05	no	-

Parameter	Partici-pant	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana-lysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifi-cation	Incl. RR	Recovery rate
					day/month					yes/no	in %
L-Serine	1	g/100g	8	78	13.10.	0,88	0,87	0,88		no	
	2	g/100g	52	34		0,9	0,91	0,89			
	3	g/100g	66	20							
	4	g/100g	1	85	15.10.18	0,792	0,798	0,786	0,05	no	
	5	g/100g	18	68	04.10.18	0,882	0,879	0,884	0,007	no	x
	6	g/100g	10	76	19.10.18	0,86	0,87	0,85	0,03	no	
	7	g/100g	29	57	08.10.18	0,88	0,87	0,88	0,05	no	na
	8	g/100g	74	12	24.09.18	1,025	1,02	1,03		no	
	9	g/100g	30	56	10.10.18	0,992	0,995	0,989	0,2	no	
	10	g/100g	11	75	26.09.18	0,931	0,91	0,952	0,02		
	11	g/100g	37	49	24.09.18	0,8013	0,811	0,7915	0,0031	no	
	12	g/100g	9	77	21.09.	0,94	0,94	0,95			
	13	g/100g	21	65	18/09	0,9	0,91	0,89	0,05	no	-

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Parameter	Partici-pant	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana-lysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifi-cation	Incl. RR	Recovery rate
					day/month					yes/no	in %
L-Threonine	1	g/100g	8	78	13.10.	0,76	0,75	0,77		no	
	2	g/100g	52	34		0,69	0,68	0,7			
	3	g/100g	66	20	23.10.18	1,611	0,158	0,166		no	
	4	g/100g	1	85	15.10.18	0,6935	0,699	0,688	0,05	no	
	5	g/100g	18	68	27.09.18	0,767	0,772	0,762	0,055	no	x
	6	g/100g	10	76	19.10.18	0,81	0,81	0,802	0,04	no	
	7	g/100g	29	57	08.10.18	0,76	0,76	0,76	0,05	no	na
	8	g/100g	74	12	24.09.18	0,785	0,78	0,79		no	
	9	g/100g	30	56	10.10.18	0,826	0,824	0,828	0,2	no	
	10	g/100g	11	75	26.09.18	0,764	0,748	0,781	0,05		
	11	g/100g	37	49	24.09.18	0,7015	0,7075	0,6955	0,0049	no	
	12	g/100g	9	77	26.09.	0,81	0,81	0,81			
	13	g/100g	21	65	18/09	0,76	0,77	0,74	0,05	no	-

Parameter	Partici-pant	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana-lysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifi-cation	Incl. RR	Recovery rate
					day/month					yes/no	in %
L-Tryptophan	1	g/100g	8	78							
	2	g/100g	52	34		0,23	0,23	0,23			
	3	g/100g	66	20							
	4	g/100g	1	85	15.10.18	0,1593	0,1575	0,161	0,05	no	
	5	g/100g	18	68	19.10.18	0,27	0,267	0,272	0,008	no	x
	6	g/100g	10	76	19.10.18	0,16	0,16	0,16	0,09	no	
	7	g/100g	29	57	28.09.18	0,24	0,24	0,24	0,02	no	na
	8	g/100g	74	12	24.09.18	0,34	0,23	0,45		no	
	9	g/100g	30	56							
	10	g/100g	11	75	04.10.18	0,193	0,198	0,188	0,01		
	11a	g/100g	37	49	24.09.18	0	0	0	0,0047	no	
	11b	g/100g	9	77	24.09.18	0,1998	0,196	0,2035	0,0047	no	
	12	g/100g	21	65	25.09.	0,22	0,22	0,22		no	
	13	g/100g			18/09	0,17	0,17	0,16	0,05	no	-

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Parameter	Partici-pant	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana-lysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifi-cation	Incl. RR	Recovery rate
					day/month					yes/no	in %
L-Tyrosine	1	g/100g	8	78	13.10.	0,58	0,58	0,59		no	
	2	g/100g	52	34		0,59	0,58	0,6			
	3	g/100g	66	20							
	4	g/100g	1	85	15.10.18	0,7075	0,725	0,69	0,05	no	
	5	g/100g	18	68	17.10.18	0,745	0,746	0,743	0,02	no	x
	6	g/100g	10	76	19.10.18	0,69	0,69	0,69	0,07	no	
	7	g/100g	29	57	08.10.18	0,68	0,67	0,68	0,05	no	na
	8	g/100g	74	12	24.09.18	0,64	0,63	0,65		no	
	9	g/100g	30	56	10.10.18	0,639	0,627	0,652	0,2	no	
	10	g/100g	11	75	26.09.18	0,7	0,684	0,717	0,04		
	11	g/100g	37	49	24.09.18	0,6955	0,686	0,705	0,0065	no	
	12	g/100g	9	77	21.09.	0,62	0,62	0,63			
	13	g/100g	21	65	18/09	0,49	0,49	0,49	0,05	no	-

Parameter	Partici-pant	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana-lysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifi-cation	Incl. RR	Recovery rate
					day/month					yes/no	in %
L-Valine	1	g/100g	8	78	13.10.	1,03	1,03	1,03		nein	
	2	g/100g	52	34		1,05	1,05	1,04			
	3	g/100g	66	20	23.10.18	0,936	0,927	0,946		nein	
	4	g/100g	1	85	15.10.18	1,033	1,032	1,034	0,05	nein	
	5	g/100g	18	68	27.09.18	1,028	1,04	1,015	0,008	nein	x
	6	g/100g	10	76	19.10.18	1,14	1,14	1,13	0,04	nein	
	7	g/100g	29	57	08.10.18	0,97	0,97	0,97	0,05	nein	na
	8	g/100g	74	12	24.09.18	1,08	1,06	1,1		nein	
	9	g/100g	30	56	10.10.18	1	1,01	0,991	0,15	nein	
	10	g/100g	11	75	26.09.18	1,045	1,011	1,079	0,02		
	11	g/100g	37	49	24.09.18	0,972	0,978	0,966	0,0052	nein	
	12	g/100g	9	77	21.09.	1,01	1	1,02			
	13	g/100g	21	65	18/09	0,87	0,86	0,87	0,05	nein	-

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Parameter	Partici-pant	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana-lysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifi-cation	Incl. RR	Recovery rate
					day/month					yes/no	in %
Taurin	11	g/100g			24.09.18	0,088	0,092	0,083	0,0049	no	

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5.1.2 Analytical Methods

* For application of the same method notes to the method could be given for L-Alanine only.

Parameter	Partici-pant	Method specification, as in test report / standard / literature *	Notes to sample preparation*	Notes to analytical method*	Calibration and reference material	Recovery with same matrix	Method accr. ISO / IEC 17025	Further remarks
						yes / no	yes / no	
L-Alanine	1	Total amino acids after acid hydrolysis by amino acid analyzer (with post coulmn derivatization)	acid hydrolysis with 6 M HCl, 24 h at 110°C	amino acid analyzer (with post coulmn derivatization, ninhydrin)	External standard, one point calibration, reference material: milk protein		yes	
	2	ASU § 64	Hydrolysis	amino acid analyzer	yes		yes	
	3	Phenomenex EZ faast GC FID	16 h at 110°C with 6 mol HCl	GC FID			no	
	4	SOP M 3123	sample weight in, digestion with microwave, dilution, derivatization, measurement				yes	
	5	§64 LFGB L 49.07-2	Hydrolysis with 6 N HCL	Derivatization with Ninhydrin	DLA 59 / 2016	no	yes	
	6	Determination by amino acid analyzer after acid hydrolysis			internal standard		yes	
	7	VO(EG) 152/2009, III, F : 2009-02	na	na	na	no	yes	
	8	acid hydrolysis followed by HPLC with fluorescence detection					accredited for total amino acids but not for free amino acids	
	9	Waters - ACCQ.TAG Chemistry Package – Instruction Manual, WAT 052 874 TP, rev. 0 April, 1993 *			Thermo Scientific Amino Acids Standard H		yes	
	10	HPLC-FLD	acid hydrolysis, OPA-deriv.	HPLC-FLD; ISTD	PT 2014	no	yes	
	11	AOAC (2000) 999.13 Ch4 p17	Alkali Hydrolysis	Internal Method QC0601	single point	no	yes	
	12	Total amino acids (according Regulation(EC) Nr. 152/2009)	Hydrolysis		internal std, 1-point calibration, feed		yes	
	13	HPLC/UV-FLD - internal method PNTQ1339			external calib. curve and internal RM	no	no	

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Parameter	Partici-pant	Method specification, as in test report / standard / literature *	Notes to sample preparation*	Notes to analytical method*	Calibration and reference material	Recovery with same matrix	Method accr. ISO / IEC 17025	Further remarks
						yes / no	yes / no	
L-Arginine	1						yes	
	2							
	3							
	4						yes	
	5		Hydrolysis with 6 N HCL	Derivatization with Ninhydrin	DLA 59 / 2016	no	yes	
	6	Determination by amino acid analyzer after acid hydrolysis			interner Standard		yes	
	7	VO(EG) 152/2009, III, F : 2009-02	na	na	na	no	yes	
	8							Argenine may present with high bias if taurine is present.
	9				Thermo Scientific Amino Acids Standard H		yes	
	10		saure Hydrolyse, OPA-Deriv.	HPLC-FLD; ISTD				
	11		Acid Hydrolysis		calibration and	no	yes	
	12		Hydrolyse					
	13	HPLC/UV-FLD - internal method PNTQ1339			external calib. curve and internal RM	no	no	

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Parameter	Participant	Method specification, as in test report / standard / literature *	Notes to sample preparation*	Notes to analytical method*	Calibration and reference material	Recovery with same matrix	Method accr. ISO / IEC 17025	Further remarks
						yes / no	yes / no	
L-Aspartic acid, Glycine, L-Isoleucine, L-Leucine, L-Phenyl-alanine, L-Threonine, L-Valine	1						yes	
	2							
	3						no	
	4						yes	
	5		Hydrolysis with 6 N HCL	Derivatization with Ninhydrin	DLA 59 / 2016	no	yes	
	6	Determination by amino acid analyzer after acid hydrolysis			internal standard		yes	
	7	VO(EG) 152/2009, III, F : 2009-02	na	na	na	no	yes	
	8							
	9				Thermo Scientific Amino Acids Standard H		yes	
	10		acid hydrolysis, OPA-deriv.	HPLC-FLD; ISTD				
	11		Acid Hydrolysis		internal QC used.	no	yes	
	12		Hydrolysis					
	13	HPLC/UV-FLD - internal method PNTQ1339			external calib. curve and internal RM	no	no	

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Parameter	Partici-pant	Method specification, as in test report / standard / literature *	Notes to sample preparation*	Notes to analytical method*	Calibration and reference material	Recovery with same matrix	Method accr. ISO / IEC 17025	Further remarks
						yes / no	yes / no	
L-Cysteine	1						yes	
	2							
	3							
	4						yes	
	5							
	6							
	7	VO(EG) 152/2009, III, F : 2009-02	na	na	na	no	yes	determined as cysteic acid
	8							
	9							
	10		acid hydrolysis, OPA-deriv.	HPLC-FLD; ISTD				
	11	N/A	N/A	N/A	N/A	N/A	N/A	Unable to determine at the present time
	12							
	13	-			-	-	-	

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Parameter	Partici-pant	Method specification, as in test report / standard / literature *	Notes to sample preparation*	Notes to analytical method*	Calibration and reference material	Recovery with same matrix	Method accr. ISO / IEC 17025	Further remarks
						yes / no	yes / no	
L-Cystine	1						yes	
	2							
	3							
	4						yes	
	5		Hydrolysis with 6 N HCL	Derivatization with Ninhydrin	DLA 59 / 2016	no	yes	
	6	Determination by amino acid analyzer after oxidation and acid hydrolysis			internal standard		yes	
	7	VO(EG) 152/2009, III, F : 2009-02	na	na	na	no	yes	determined as cysteic acid
	8	also oxidised prior to analysis in performic acid						
	9							
	10							
	11		Alkali Hydrolysis			no	yes	
	12		Oxidation					
	13	HPLC/UV-FLD - internal method PNTQ1339			external calib. curve and internal RM	no	no	

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Parameter	Partici-pant	Method specification, as in test report / standard / literature *	Notes to sample preparation*	Notes to analytical method*	Calibration and reference material	Recovery with same matrix	Method accr. ISO / IEC 17025	Further remarks
						yes / no	yes / no	
L-Glutamic acid	1						yes	
	2							
	3							
	4						yes	
	5		Hydrolysis with 6 N HCL	Derivatization with Ninhydrin	DLA 59 / 2016	no	yes	
	6	Determination by amino acid analyzer after acid hydrolysis			internal standard		yes	
	7	VO(EG) 152/2009, III, F : 2009-02	na	na	na	no	yes	
	8							
	9				Thermo Scientific Amino Acids Standard H		yes	
	10		acid hydrolysis, OPA-deriv.	HPLC-FLD; ISTD				
	11		Alkali Hydrolysis			no	yes	
	12		Hydrolysis					
	13	HPLC/UV-FLD - internal method PNTQ1339			external calib. curve and internal RM	no	no	

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Parameter	Partici-pant	Method specification, as in test report / standard / literature *	Notes to sample preparation*	Notes to analytical method*	Calibration and reference material	Recovery with same matrix	Method accr. ISO / IEC 17025	Further remarks
						yes / no	yes / no	
L-Histidine, L-Lysine, L-Serine	1						yes	
	2							
	3							
	4						yes	
	5		Hydrolysis with 6 N HCL	Derivatization with Ninhydrin	DLA 59 / 2016	no	yes	
	6	Determination by amino acid analyzer after acid hydrolysis			internal standard		yes	
	7	VO(EG) 152/2009, III, F : 2009-02	na	na	na	no	yes	
	8							
	9				Thermo Scientific Amino Acids Standard H		yes	
	10		acid hydrolysis, OPA-deriv.	HPLC-FLD; ISTD				
	11		acid hydrolysis			no	yes	
	12		Hydrolysis					
	13	HPLC/UV-FLD - internal method PNTQ1339			external calib. curve and internal RM	no	no	

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Parameter	Participant	Method specification, as in test report / standard / literature *	Notes to sample preparation*	Notes to analytical method*	Calibration and reference material	Recovery with same matrix	Method accr. ISO / IEC 17025	Further remarks
						yes / no	yes / no	
L-Methionine	1						ja	
	2							
	3						nein	
	4						ja	
	5		Hydrolysis with 6 N HCL	Derivatization with Ninhydrin	DLA 59 / 2016	nein	ja	
	6	Determination by amino acid analyzer after oxidation and acid hydrolysis			internal standard		ja	
	7	VO(EG) 152/2009, III, F : 2009-02	na	na	na	nein	ja	determined as methionine sulfon
	8	also oxidised prior to analysis in performic acid						
	9				Thermo Scientific Amino Acids Standard H		ja	
	10		Oxid./acid hydrolysis, OPA-deriv.	HPLC-FLD; ISTD				
	11		Alkali Hydrolysis			no	yes	
	12		Oxidation					
	13	HPLC/UV-FLD - internal method PNTQ1339			external calib. curve and internal RM	no	no	

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Parameter	Partici-pant	Method specification, as in test report / standard / literature *	Notes to sample preparation*	Notes to analytical method*	Calibration and reference material	Recovery with same matrix	Method accr. ISO / IEC 17025	Further remarks
						yes / no	yes / no	
L-Proline	1						yes	
	2							
	3						no	
	4						yes	
	5		Hydrolysis with 6 N HCL	Derivatization with Ninhydrin	DLA 59 / 2016	no	yes	
	6	Determination by amino acid analyzer after acid hydrolysis			internal standard		yes	
	7	VO(EG) 152/2009, III, F : 2009-02	na	na	na	no	yes	
	8							
	9				Thermo Scientific Amino Acids Standard H		yes	
	10		acid hydrolysis, deriv. with NBDCl	HPLC-FLD; ext.STD				
	11		Acid Hydrolysis			no	yes	
	12		Hydrolyse					
	13	HPLC/UV-FLD - internal method PNTQ1339			external calib. curve and internal RM	no	no	

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Parameter	Partici-pant	Method specification, as in test report / standard / literature *	Notes to sample preparation*	Notes to analytical method*	Calibration and reference material	Recovery with same matrix	Method accr. ISO / IEC 17025	Further remarks
						yes / no	yes / no	
L-Tryptophan	1						yes	
	2							
	2	RP-HPLC and fluorescence detection, in-house method						
	3							
	4						yes	
	4		Sample weight in with barium hydroxide, digestion in autoclave, diluted, derivatization and measurement				yes	
	5							
	5	VDLUFA III 4.11.2	alkali hydrolysis with LiOH	HPLC isocratic	DLA 59 / 2016	no	yes	
	6							
	6	Determination by amino acid analyzer after alkali hydrolysis			internal standard		yes	
	7							
	7	FO(EF) 152/2009 App. III, G : 2009-02	na	na	na	no	yes	
	8							
	8	alkaline hydrolysis followed by HPLC with fluorescence detection					accredited for total tryptophan only	
	9							
	9							
	10		alkali Hydrolysis	HPLC-FLD; ext.STD				
	11a		Acid Hydrolysis			no	yes	
	11b		Alkali Hydrolysis			no	yes	
	12							
	12	HPLC (analog Nr 4.11.2 VDLUFA Methodenbuch III, 2. Erg. 1988)	Hydrolyse		3 point calibration, feed		yes	
	13	-			-	-	-	
	13	HPLC/UV-FLD - internal method PNTQ1339			external calib. curve and internal RM	no	no	HPLC/UV-FLD - internal method PNTQ1339

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Parameter	Participant	Method specification, as in test report / standard / literature *	Notes to sample preparation*	Notes to analytical method*	Calibration and reference material	Recovery with same matrix	Method accr. ISO / IEC 17025	Further remarks
						yes / no	yes / no	
L-Tyrosine	1						yes	
	2							
	3							
	4						yes	
	5		Hydrolysis with 6 N HCL	Derivatization with Ninhydrin	DLA 59 / 2016	nein	yes	
	6	Determination by amino acid analyzer after acid hydrolysis			internal standard		yes	
	7	VO(EG) 152/2009, III, F : 2009-02	na	na	na	nein	yes	
	8							
	9				Thermo Scientific Amino Acids Standard H		yes	
	10		acid hydrolysis, OPA-deriv.	HPLC-FLD; ISTD				
	11		Alkali Hydrolysis			nein	yes	
	12		Hydrolysis					
	13	HPLC/UV-FLD - internal method PNTQ1339			external calib. curve and internal RM	no	no	

Parameter	Participant	Method specification, as in test report / standard / literature *	Notes to sample preparation*	Notes to analytical method*	Calibration and reference material	Recovery with same matrix	Method accr. ISO / IEC 17025	Further remarks
						yes / no	yes / no	
Taurine	11		acid Hydrolysis			no	yes	

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5.2 Homogeneity

5.2.1 Mixture homogeneity before bottling

Microtracer Homogeneity Test
DLA 50-2018

Weight whole sample	0,97	kg
Microtracer	FSS-rot lake	
Particle size	75 – 300	µm
Weight per particle	2,0	µg
Addition of tracer	33,9	mg/kg

Result of analysis

Sample	Weight [g]	Particle number	Particles [mg/kg]
1	5,00	98	39,2
2	5,03	101	40,2
3	5,03	92	36,6
4	5,02	86	34,3
5	5,05	99	39,2
6	5,02	97	38,6
7	5,00	90	36,0
8	5,00	82	32,8

Poisson distribution		
Number of samples	8	
Degree of freedom	7	
Mean	93,1	Particles
Standard deviation	6,61	Particles
χ^2 (CHI-Quadrat)	3,29	
Probability	**86**	%
Recovery rate	109	%

Normal distribution		
Number of samples	8	
Mean	37,1	mg/kg
Standard deviation	2,64	mg/kg
rel. Standard deviaton	7,10	%
Horwitz standard deviation	9,29	%
HorRat-value	**0,76**	
Recovery rate	109	%

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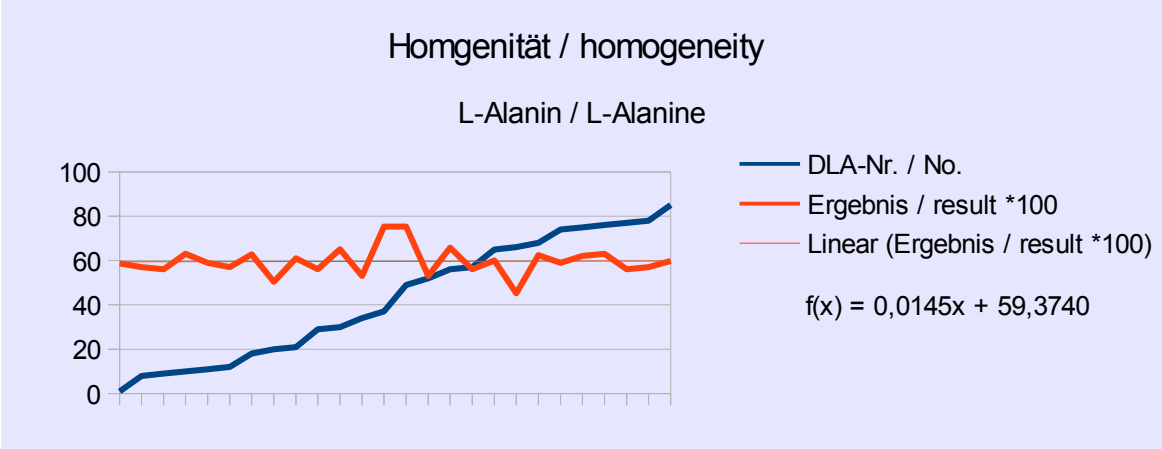
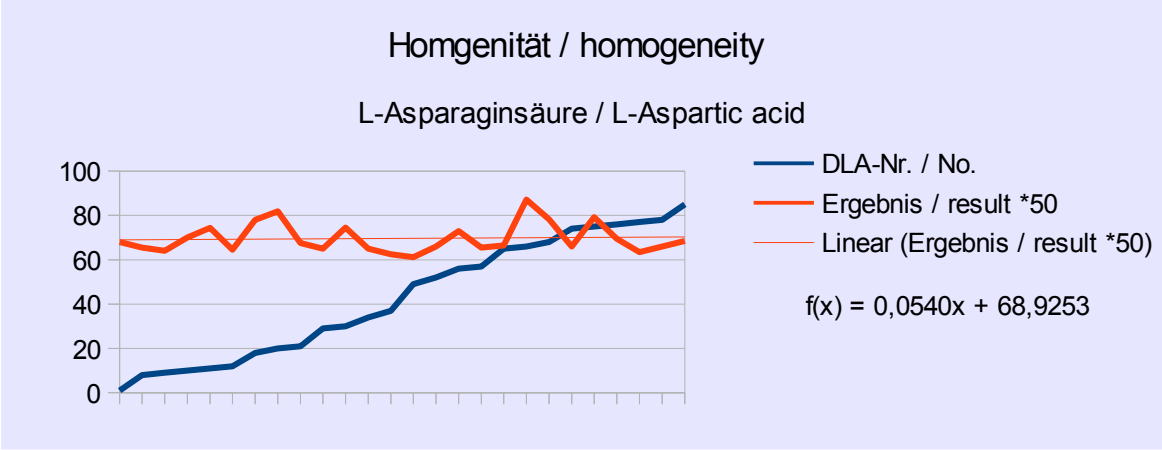
5.2.2 Comparision of sample numbers / test results and trend line

By comparison of the increasing sample numbers and the measurement results of participants, the homogeneity of the chronological bottled PT items can be shown by the trend line for information:





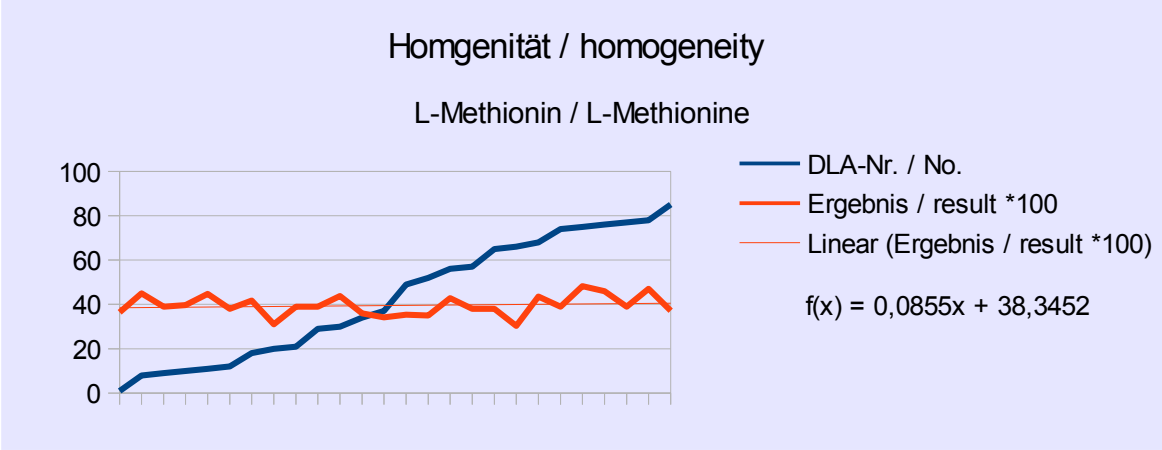


Abb./Fig. 38: Trendfunktion Probennummern vs. Ergebnisse: L-Alanin, L-Asparaginsäure und L-Methionin (1*100, 1*50 und 1*100 dargestellt)
trend line function sample number vs. results: L-Alanine, L-Aspartic acid and L-Methionine (1*100, 1*50 and 1*100 shown)

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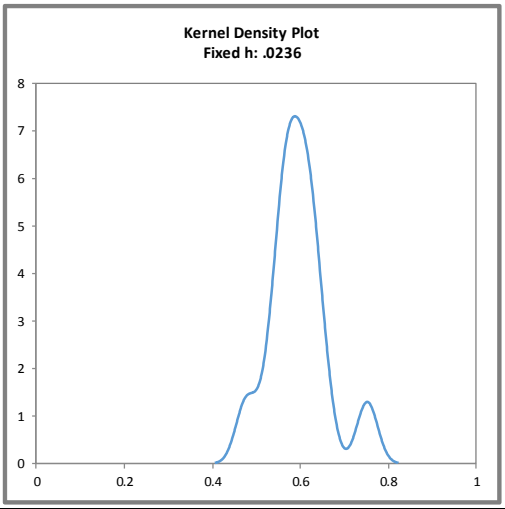
5.3 Kernel Density Plots of Results

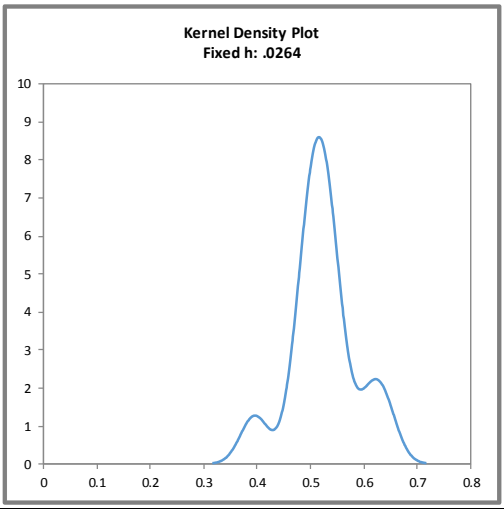
Abbildungen:
Kerndichte-Schätzungen
der Teilnehmerergebnisse
(mit h = 0,75 x σ_{pt} von Xpt)

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

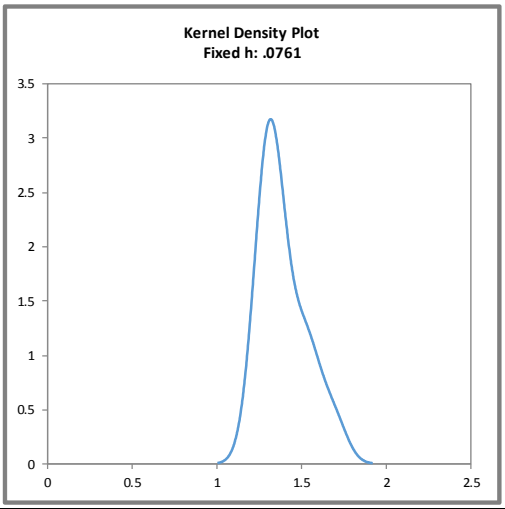
L-Alanin / L-Alanine



L-Arginin / L-Arginine



L-Asparaginsäure / L-Aspartic acid

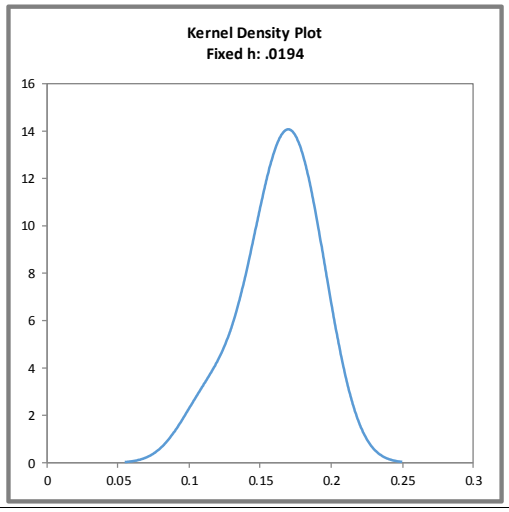


L-Cystein / L-Cysteine

<8 Ergebnisse

<8 Results

L-Cystin / L-Cystine



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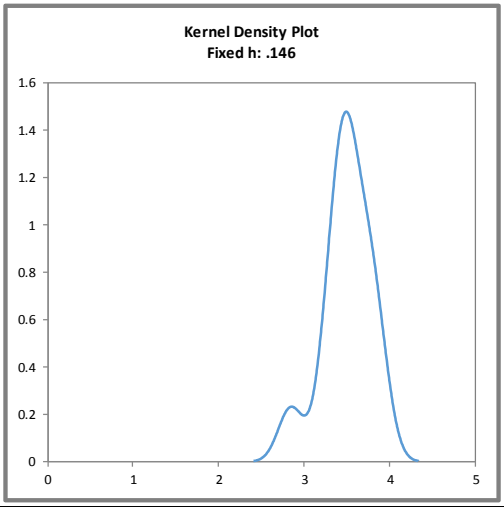
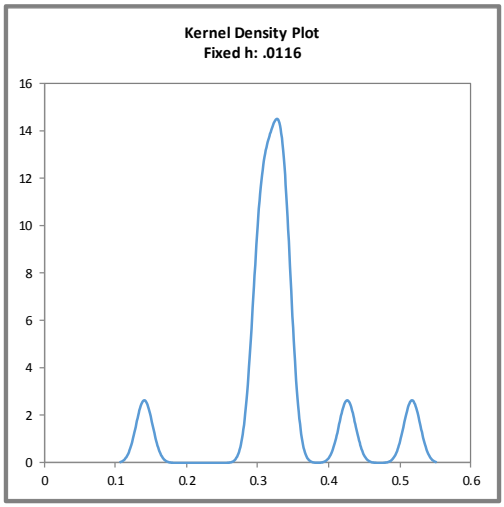
Abbildungen:
Kerndichte-Schätzungen
der Teilnehmerergebnisse
(mit h = 0,75 x σ_{pt} von Xpt)

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

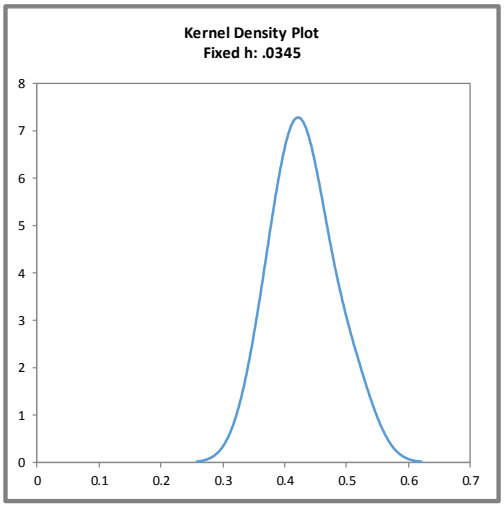
L-Glutaminsäure / L-Glutamic acid



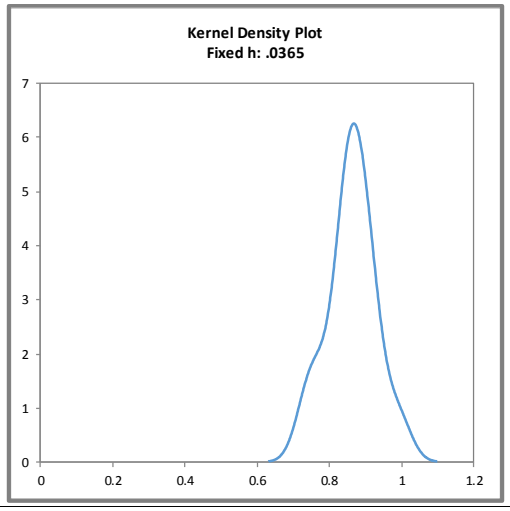
Glycin / Glycine



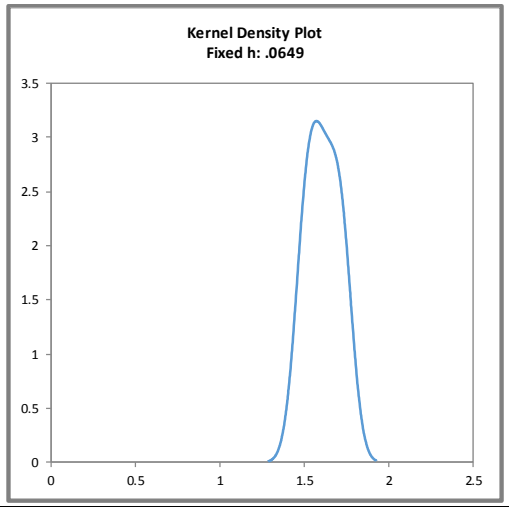
L-Histidin / L-Histidine



L-Isoleucin / L-Isoleucine



L-Leucin / L-Leucine



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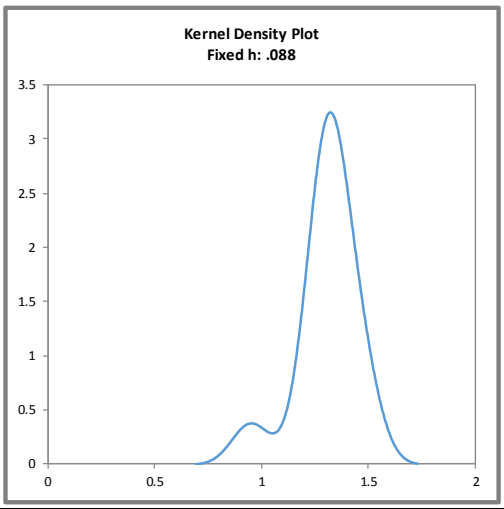
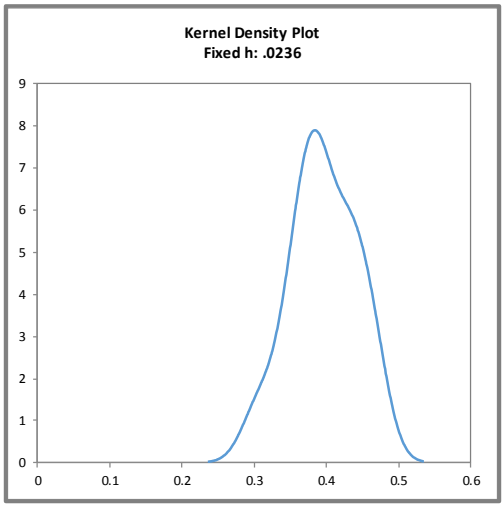
Abbildungen:
Kerndichte-Schätzungen
der Teilnehmerergebnisse
(mit h = 0,75 x σ_{pt} von X_{pt})

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

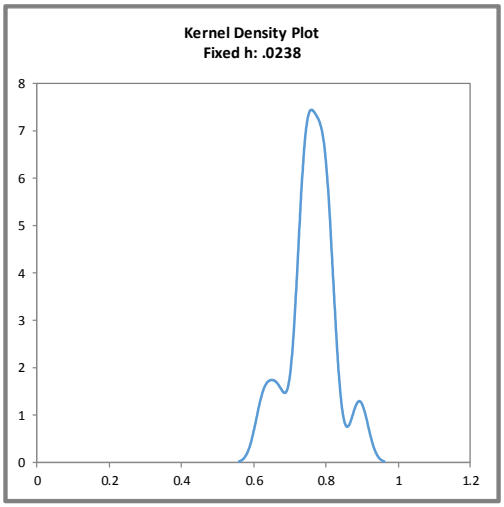
L-Lysin / L-Lysine



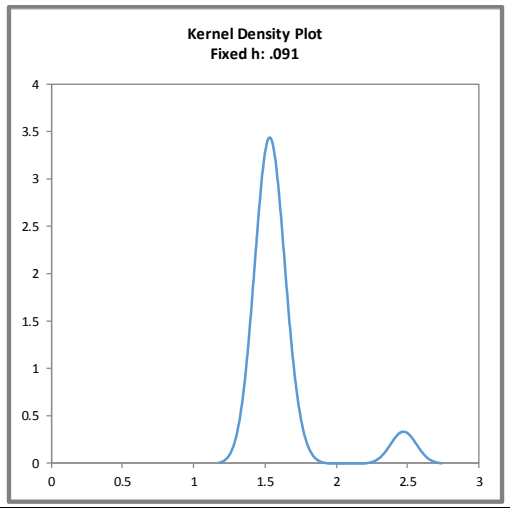
L-Methionin / L-Methionine



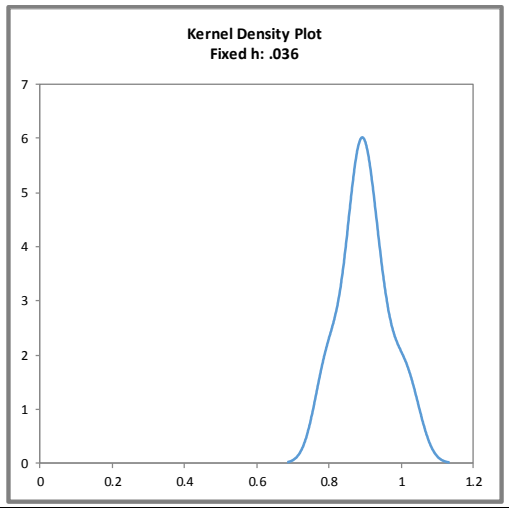
L-Phenylalanin / L-Phenylalanine



L-Prolin / L-Proline



L-Serin / L-Serine



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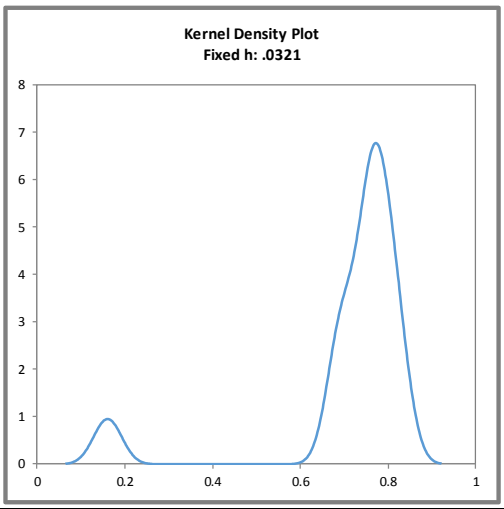
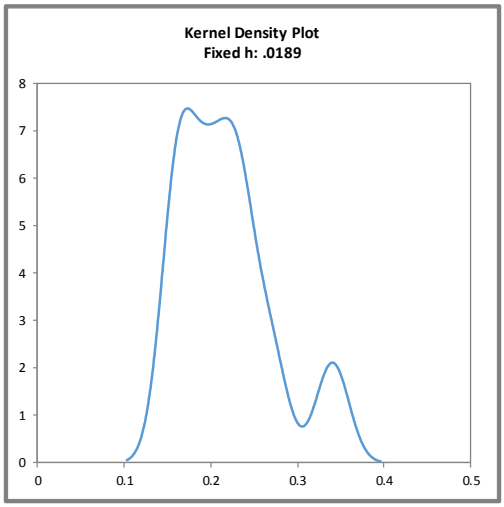
Abbildungen:
Kerndichte-Schätzungen
der Teilnehmerergebnisse
(mit h = 0,75 x σ_{pt} von Xpt)

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

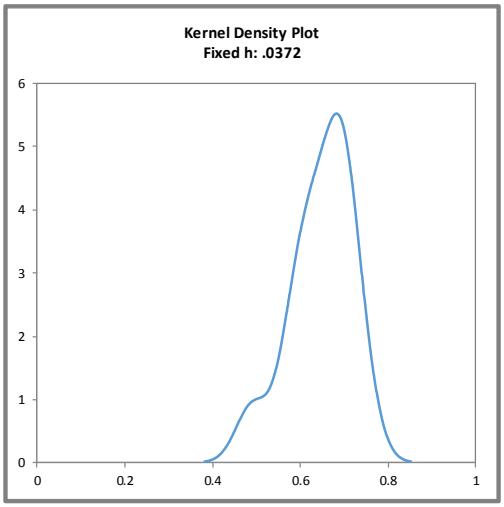
L-Threonin / L-Threonine



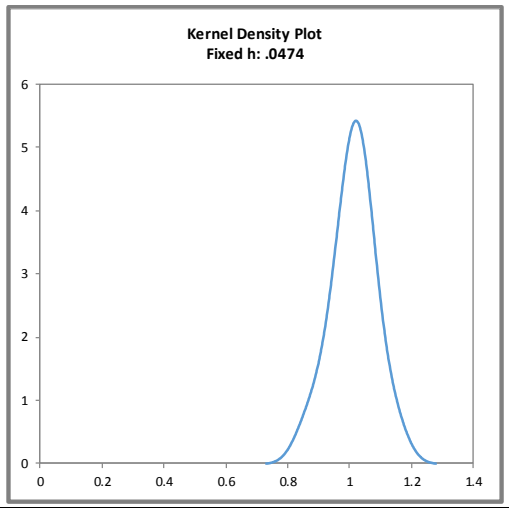
L-Tryptophan / L-Tryptophan



L-Tyrosin / L-Tyrosine



L-Valin / L-Valine



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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 50-2018***
PT name	***Total Amino Acids in Food***
*Sample matrix**	***Samples I + II:*** *infant food, drink powder with a protein content <20% from milk protein, ingredients: skimmed milk powder, whey products, oligosaccharides, fats, lactose, minerals and vitamins*
Number of samples and sample amount	*2 identical samples I + II, 10 g each.*
Storage	*Samples I + II: room temperature (dry and dark)*
Intentional use	*Laboratory use only (quality control samples)*
Parameter	*quantitative:* ***diverse amino acids after protein hydrolysis*** *(see result submission file)*
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	*g/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***
Deadline	***the latest 26th October 2018***
Evaluation report	*The evaluation report is expected to be completed 6 weeks after deadline of result submission and sent as PDF file by e-mail.*
Coordinator and contact person of PT	*Matthias Besler-Scharf, PhD*

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

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6. Index of participant laboratories in alphabetical order

Teilnehmer / Participant	Ort / Town	Land / Country
		GREAT BRITAIN
		GREAT BRITAIN
		CZECH REPUBLIC
		Germany
		Germany
		Germany
		Germany
		Germany
		Germany
		Germany
		Germany
		Germany
		Germany
		SPAIN

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

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

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20. Verordnung 152/2009/EG zur Festlegung der Probenahmeverfahren und Analysemethoden für die amtliche Untersuchung von Futtermitteln / Regulation 152/2009/EC laying down the methods of sampling and analysis for the official control of feed

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