

Proficiency Tests

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Evaluation Report

proficiency test

DLA 47/2017

Free Amino Acids

**in Food Supplement
(Capsule Powder)**

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

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

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1. Introduction

The participation in proficiency testing schemes is an essential element of the quality-management-system of every laboratory testing food and feed, cosmetics and food contact materials. The implementation of proficiency tests enables the participating laboratories to prove their own analytical competence under realistic conditions. At the same time they receive valuable data regarding the verification and/or validation of the particular testing method [1, 5].

The purpose of DLA is to offer proficiency tests for selected parameters in concentrations with practical relevance.

Realisation and evaluation of the present proficiency test follows the technical requirements of DIN EN ISO/IEC 17043 (2010) and DIN ISO 13528:2009 / ISO 13528:2015 [2, 3].

2. Realisation

2.1 Test material

The test material is a mixture of common in commerce food supplements with free amino acids from EU suppliers with a protein equivalent content of > 20%. Further additives are L-glutamic acid and maltodextrin. The capsule shells of the food supplements were removed, all raw materials sieved (mesh 600 µm), mixed and homogenized.

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

The composition (list of ingredients) and the amounts of amino acids as calculated from the manufacturers specifications are given in table 1 and table 2 respectively.

Table 1: Composition of DLA-Samples

Amino Acid Capsule Powder
<p><u>Ingredients</u> (1. Food Supplement, without capsule shell): Bulking agent Dicalcium phosphate, L-Leucine, L-Lysine, L-Cysteine, L-Valine, L-Isoleucine, L-Arginine, L-Tyrosine, L-Histidine, L-Threonine, L-Phenylalanine, L-Tryptophan, L-Methionine, Release agents Magnesium salts of fatty acids and Silica, Dye Titanium dioxide.</p>
<p><u>Ingredients</u> (2. Food Supplement, without capsule shell): L-Lysine HCl, L-Leucine, L-Aspartic acid, L-Isoleucine, L-Tryptophan, Bulking agent: Rice flour, L-Proline, L-Serine, L-Alanine, L-Threonine, L-Valine, L-Phenylalanine, L-Methionine, Glycine, L-Histidine.</p>
<p><u>Further Ingredient:</u> L-Glutaminic acid, maltodextrin</p>

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

Table 2: Calculated amounts of PT parameters according to the manufacturers specifications

Parameter	Content per 100 g	Parameter	Content per 100 g
L-Alanine	1,7 g	L-Lysine	5,0 g
L-Arginine	1,5 g	L-Methionine	1,1 g
L-Aspartic acid	3,4 g	L-Phenylalanine	1,8 g
L-Cysteine	1,5 g	L-Proline	2,1 g
L-Glutamic acid	1,5 g	L-Serine	1,7 g
Glycine	0,68 g	L-Threonine	2,6 g
L-Histidine	1,8 g	L-Tryptophan	2,7 g
L-Isoleucine	3,8 g	L-Tyrosine	1,2 g
L-Leucine	6,6 g	L-Valine	3,0 g

2.1.1 Homogeneity

The **mixture homogeneity before bottling** was examined 8-fold by **micro-tracer 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 $\geq 5\%$ is equivalent to a good homogeneous mixture and of $\geq 25\%$ to an excellent mixture [14, 15].

The microtracer analysis of the present PT sample showed a probability of 90%. 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 0,64. The results of microtracer analysis are given in the documentation.

The calculation of the **repeatability standard deviations S_r of the participants** was also used as an indicator of homogeneity. For all parameters they are in the range of $< 10\%$ (from 2,8% to 9,8%) with the exception of L-alanine and L-serine (s. Tab. 3). Thus they were similar to the repeatability standard deviations of the corresponding official methods (e.g. ASU methods, s. 3.6.2) (see Tab. 4) [18]. The repeatability standard deviations of the participants' results are given in the documentation in the statistic data (see 4.1 and 4.19).

Table 3: 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	16,7 %	L-Lysine	5,3 %
L-Arginine	2,8 %	L-Methionine	3,9 %
L-Aspartic acid	5,3 %	L-Phenylalanine	3,0 %
L-Cysteine	-	L-Proline	3,8 %
L-Glutamic acid	8,1 %	L-Serine	11,5 %
Glycine	13,7 %	L-Threonine	2,9 %
L-Histidine	9,8 %	L-Tryptophan	3,6 %
L-Isoleucine	3,6 %	L-Tyrosine	3,6 %
L-Leucine	3,2 %	L-Valine	4,4 %

Furthermore, the homogeneity was characterized by the **trend line function of participants' results for chronological bottled single samples**. The maximum deviations from the mean value of the trend line were in the range of 8,8% to 13,4% of the target standard deviation σ_{pt} or σ_{pt}' (s. 5.2 Homogeneity) for L-Arginine, L-Phenylalanine and L-Tyrosine and thus can be regarded as low.

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

2.1.2 Stability

The experience with various DLA reference 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 stability of the sample material is therefore given during the investigation period under consideration of given storage conditions.

Furthermore, **participants' results** were compared with the corresponding **date of analysis** to characterize the stability of the PT-material during the whole time of analysis of the present PT (40 days) by using the trendline-functions.

The maximum deviations from the mean value of the trend line for L-Phenylalanine and L-Tyrosine were at 12% and 2,4% of the target standard deviation σ_{pt} or σ_{pt}' (see documentation section 5.3 Stability) and thus can be regarded as low. For L-Arginine the maximum deviation from the trend line was 58%, which can be due to the higher reproducibility standard deviation of the participants' results (CV_R 14,8%).

2.2 Sample shipment and information to the test

Two portions of test material were sent to every participating laboratory in the 38th week of 2017. The testing method was optional. The tests should be finished at 3rd November 2017 the latest.

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

The two portions contain identical samples of a food supplement with the parameters of free amino acids in the matrix of capsule powder. The protein equivalent content is > 20%. The analysis method is optional.

Please note the attached information on the proficiency test.

(see documentation, section 5.5 Information on the PT)

2.3 Submission of results

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

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

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

All 17 participants submitted their results in time.

3. Evaluation

3.1 Consensus value from participants (assigned value)

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

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

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

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

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

3.2 Robust standard deviation

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

3.3 Repeatability standard deviation

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

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

The relative repeatability standard deviation as a percentage of the mean value is indicated as coefficient of variation CV_r in the table of statistical characteristics in the results section in case single results from participants are available.

3.4 Reproducibility standard deviation

The reproducibility standard deviation S_R represents a inter-laboratory estimate of the standard deviation for the determination of each parameter on the bases of (outlier free) individual participant results. It takes into account both the repeatability standard deviation S_r and the within-laboratory standard deviation S_s . Reproducibility standard deviations of PT's may differ from reproducibility standard deviations of ring trials, because the participating laboratories of a PT generally use different internal conditions and methods for determining the measured values.

In the present evaluation, the specification of the reproducibility standard deviation, therefore, does not refer to a specific method, but characterizes approximately the comparability of results between the laboratories, assumed the effect of homogeneity and stability of the sample are negligible.

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

The relative reproducibility standard deviation VK_R in percent of the mean is given as variation coefficient in the statistical data of participant for each parameter. The significance of VK_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, and results for a another proficiency test item can be removed from the data set [2]. Even if a result clearly deviates from the robust mean (e.g. factor >10) and has an influence on the robust statistics, a result can be excluded from statistical evaluation [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 identified as outliers by the use of robust statistics. If a value deviates from the robust mean by more than 3 times the robust standard deviation, it is classified as an outlier [3]. Detected outliers are stated for information only, when z-score are < -2 or > 2. Due to the use of robust statistics outliers are not excluded, provided that no other reasons are present [3].

3.6 Target standard deviation (for proficiency assessment)

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

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

Alanine and Arginine.

For the valuation of the following parameters the target standard deviation from precision experiments (s. 3.6.2) (German official ASU §64 method: 18) was applied:

Aspartic acid, Glutamic acid, Glycine, Histidine, Isoleucine, Leucine, Lysine, Phenylalanine, Proline, Serine, Threonine, Tryptophan, Tyrosine and Valine.

Additionally for Alanine, Aspartic acid, Glycine, Isoleucine, Serine and Valine 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 and Cystin were not evaluated by means of z-scores.

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
$\sigma_R = 0,22c$	$c < 1,2 \times 10^{-7}$	$< 120 \mu\text{g}/\text{kg}$
$\sigma_R = 0,02c^{0,8495}$	$1,2 \times 10^{-7} \leq c \leq 0,138$	$\geq 120 \mu\text{g}/\text{kg}$
$\sigma_R = 0,01c^{0,5}$	$c > 0,138$	$> 13,8 \text{ g}/100\text{g}$

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

3.6.2 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]:

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

The relative repeatability standard deviations (RSD_r) and relative reproducibility standard deviation (RSD_R) given in Table 4 were determined in ring tests using the indicated methods.

The resulting target standard deviations σ_{pt} , which were identified there, were used to evaluate the results and to provide additional information for the statistical data.

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

Parameter	Matrix (Amino acid mixture)	Mean [mg/kg]	RSD_r	RSD_R	σ_{pt}	Method / Literature
L-Alanine	Dietetic food	3,09	2,75%	4,37%	3,91% ¹	ASU 49.07-1
L-Arginine	Dietetic food	2,94	3,41%	13,8%	13,6% ¹	ASU 49.07-1
L-Aspartic acid	Dietetic food	7,48	2,41%	5,88%	5,62% ¹	ASU 49.07-1
L-Cysteine	Dietetic food	2,21	7,71%	18,6%	17,8% ¹	ASU 49.07-1
L-Cystine	Dietetic food	2,21	7,92%	18,4%	17,5% ¹	ASU 49.07-1
L-Glutamic acid	Dietetic food	16,6	2,50%	6,93%	6,70% ¹	ASU 49.07-1
Glycine	Dietetic food	1,79	3,07%	5,59%	5,15% ¹	ASU 49.07-1
L-Histidine	Dietetic food	1,92	4,69%	16,4%	16,1% ¹	ASU 49.07-1
L-Isoleucine	Dietetic food	4,56	2,63%	4,93%	4,57% ¹	ASU 49.07-1
L-Methionine	Dietetic food	1,88	4,79%	7,45%	6,63% ¹	ASU 49.07-1
L-Leucine	Dietetic food	7,35	2,59%	4,69%	4,32% ¹	ASU 49.07-1
L-Lysine	Dietetic food	5,87	2,81%	12,9%	12,7% ¹	ASU 49.07-1
L-Phenylalanine	Dietetic food	3,19	5,64%	7,21%	6,01% ¹	ASU 49.07-1
L-Proline	Dietetic food	7,09	3,60%	6,56%	6,05% ¹	ASU 49.07-1
L-Serine	Dietetic food	4,00	2,63%	7,63%	7,40% ¹	ASU 49.07-1
L-Threonine	Dietetic food	3,59	3,34%	6,41%	5,96% ¹	ASU 49.07-1
L-Tryptophan	Dietetic food	1,32	4,92%	20,1%	19,8% ¹	ASU 49.07-1
L-Tyrosine	Dietetic food	4,04	5,07%	8,17%	7,34% ¹	ASU 49.07-1
L-Valine	Dietetic food	5,20	2,60%	5,20%	4,86% ¹	ASU 49.07-1

¹ used in evaluation or given for information (s. chapter 4)

The given values from ASU 49.07-1 are mean values of samples I and II (exceptions: Methionine sample I, Phenylalanine sample II only)

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 and 3.6.2 were regarded suitable.

Table 5 shows selected statistic data of participants results of present PT compared to PT results of previous years.

3.7 z-Score

To assess the results of the participants the z-score is used. It indicates about which multiple of the target standard deviation (σ_{pt}) the result (x_i) of the participant is deviating from the assigned value (X_{pt}) [3].

Participants' z-scores are derived from:

$$z_i = \frac{(x_i - x_{pt})}{\sigma_{pt}}$$

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].

Table 5: 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)	rob. Mean [g/100g]	rob. SD (S*) [g/100g]	rel. SD (VK _{S*}) [%]	Quotient S*/σ _{pt}	DLA Report
L-Alanine	Dietetic food	0,460	0,0369	8,02	1,8	DLA 43/2015
L-Alanine	Dietetic food	1,95	0,170	8,75	1,9 ¹	DLA 47/2017
L-Arginine	Dietetic food	0,749	0,0616	8,22	0,61	DLA 43/2015
L-Arginine	Dietetic food	1,42	0,222	15,6	1,1	DLA 47/2017
L-Aspartic acid	Dietetic food	0,740	0,0332	4,49	1,1	DLA 43/2015
L-Aspartic acid	Dietetic food	3,86	0,470	12,2	1,8 ¹	DLA 47/2017
L-Cystine	Dietetic food	**	-	-	-	DLA 43/2015
L-Cystine	Dietetic food	**	-	-	-	DLA 47/2017
L-Glutamic acid	Dietetic food	**	-	-	-	DLA 43/2015
L-Glutamic acid	Dietetic food	1,48	0,177	12,0	1,8	DLA 47/2017
Glycine	Dietetic food	0,688	0,0964	14,0	1,9 ¹	DLA 43/2015
Glycine	Dietetic food	0,755	0,0863	11,4	1,8 ¹	DLA 47/2017
L-Histidine	Dietetic food	0,435	0,0354	8,14	0,51	DLA 43/2015
L-Histidine	Dietetic food	1,66	0,404	24,4	1,5	DLA 47/2017
L-Isoleucine	Dietetic food	0,686	0,0246	3,59	0,85	DLA 43/2015
L-Isoleucine	Dietetic food	3,76	0,381	10,1	1,8 ¹	DLA 47/2017
L-Leucine	Dietetic food	1,17	0,0237	2,03	0,52	DLA 43/2015
L-Leucine	Dietetic food	6,56	0,532	8,12	1,9	DLA 47/2017

Table 5 continued:

Parameter	Matrix (Powder)	rob. Mean [g/100g]	rob. SD (S*) [g/100g]	rel. SD (VK _{s*}) [%]	Quotient S*/ σ_{pt}	DLA Report
L-Lysine	Dietetic food	0,797	0,0666	8,36	0,66	DLA 43/2015
L-Lysine	Dietetic food	4,01	0,435	10,9	0,85	DLA 47/2017
L-Methionine	Dietetic food	0,179	0,0164	9,16	1,8	DLA 43/2015
L-Methionine	Dietetic food	1,17	0,108	9,21	1,4	DLA 47/2017
L-Phenylalanine	Dietetic food	0,515	0,0273	5,30	1,8	DLA 43/2015
L-Phenylalanine	Dietetic food	1,89	0,157	8,28	1,4	DLA 47/2017
L-Proline	Dietetic food	0,837	0,0915	10,9	1,9	DLA 43/2015
L-Proline	Dietetic food	2,23	0,263	11,8	2,0	DLA 47/2017
L-Serine	Dietetic food	0,500	0,0594	11,9	1,7	DLA 43/2015
L-Serine	Dietetic food	1,80	0,256	14,2	1,6 ¹	DLA 47/2017
L-Threonine	Dietetic food	0,608	0,0666	11,0	1,9	DLA 43/2015
L-Threonine	Dietetic food	2,77	0,236	8,52	1,4	DLA 47/2017
L-Tryptophan	Dietetic food	**	-	-	-	DLA 43/2015
L-Tryptophan	Dietetic food	2,75	0,199	7,25	0,37	DLA 47/2017
L-Tyrosine	Dietetic food	0,524	0,0213	4,06	0,92	DLA 43/2015
L-Tyrosine	Dietetic food	1,18	0,0878	7,47	1,0	DLA 47/2017
L-Valine	Dietetic food	0,762	0,0320	4,20	1,0	DLA 43/2015
L-Valine	Dietetic food	3,04	0,327	10,7	1,8 ¹	DLA 47/2017

¹ with target standard deviation σ_{pt}

** no statistical evaluation (< 7 results)

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

The calculation is performed by:

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

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

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

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

For warning and action signals see 3.7.1.

3.9 Reproducibility coefficient of variation (CV)

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

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

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

3.10 Quotient S^*/σ_{pt}

Following the HorRat-value the results of a proficiency-test (PT) can be considered convincing, if the quotient of robust standard deviation S^* and target standard deviation σ_{pt} does not exceed the value of 2.

A value > 2 means an insufficient precision, i.e. the analytical method is too variable, or the variation between the test participants is higher than estimated. Thus the comparability of the results is not given [3].

3.11 Standard uncertainty of the assigned value

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

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

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

The Quotient $U_{(x_{pt})}/\sigma_{pt}$ is reported in the characteristics of the test.

4. Results

Comments to the distribution of the results:

The kernel density plots showed for all parameters nearly a normal distribution of results (figures see documentation 5.4). Partly slight shoulders and separate smaller peaks can be seen, which are due to individual values and outliers.

In the case of isoleucine and leucine 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/cystine there were < 7 plausible 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 $\leq 2,0$. In all other cases, the standard deviation calculated from ASU §64 precision data was used. For tyrosine the target standard deviation was calculated according to ASU § 64, because the Horwitz evaluation was considered too strict for these results.

For all parameters the distribution of results showed a normal variability. The quotients S^*/σ_{pt} were all in the range of 0,37 to 2,0 (see table 5).

The robust standard deviation as well as the repeatability and reproducibility standard deviations were in the range of established values for the applied methods (see 3.6.2).

The comparability of results is given.

The quotient $U(X_{pt})/\sigma_{pt}$ or $U(X_{pt})/\sigma_{pt}'$ was $< 0,3$ for lysine and tryptophan. For all other parameters it was slightly increased in the range of 0,38 to 0,61. This was considered acceptable because of the other characteristics.

71% to 93% of results were in the target range.

The robust means of the participant results ranged from 80% to 120% of the amino acid content according to the manufacturer's specification (see Table 2).

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

In the first table the characteristics are listed:

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

* 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	Parameter [Einheit / Unit]	Abweichung	z-Score	z-Score	Hinweis
Evaluation number		Deviation	σ_{pt}	(Info)	Remark

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

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

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	15
Number of outliers	1
Mean	1,98
Median	1,92
Robust Mean (X)	1,95
Robust standard deviation (S*)	0,170
Number with 2 replicates	14
Repeatability SD (S_r)	0,323
Repeatability (CV_r)	16,7%
Reproducibility SD (S_R)	-
Reproducibility (CV_R)	-
Target range:	
Target standard deviation σ_{pt}'	0,0893
Target standard deviation (for Information)	0,0762
lower limit of target range	1,77
upper limit of target range	2,13
Quotient S^*/σ_{pt}'	1,91
Standard uncertainty $U(x_{pt})$	0,0550
Quotient $U(x_{pt})/\sigma_{pt}'$	0,62
Results in the target range	12
Percent in the target range	80%

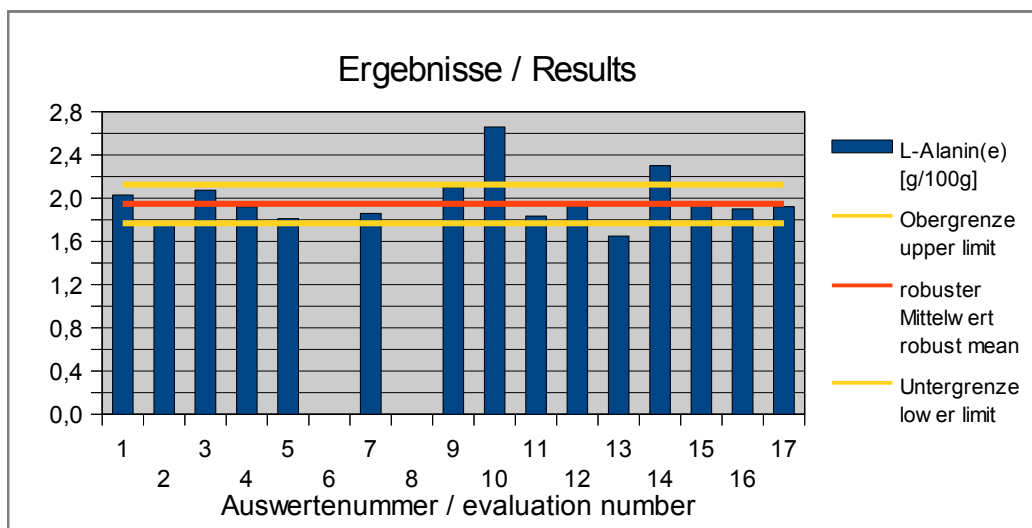


Abb. / Fig. 1: Ergebnisse L-Alanin / Results L-Alanine

**Ergebnisse der Teilnehmer:
Results of Participants:**

Auswertenummer	L-Alanin(e) [g/100g]	Abweichung [g/100g]	z'-Score (σ_{pt})	z-Score (Info)	Hinweis
Evaluation number		Deviation [g/100g]			Remark
1	2,03	0,0835	0,9	1,1	
2	1,77	-0,177	-2,0	-2,3	
3	2,07	0,126	1,4	1,7	
4	1,92	-0,0265	-0,3	-0,3	
5	1,81	-0,137	-1,5	-1,8	
6					
7	1,86	-0,0865	-1,0	-1,1	
8					
9	2,10	0,153	1,7	2,0	
10	2,66	0,713	8,0	9,4	Ausreisser / Outlier
11	1,84	-0,111	-1,2	-1,5	
12	1,93	-0,0215	-0,2	-0,3	
13	1,65	-0,297	-3,3	-3,9	
14	2,30	0,353	4,0	4,6	
15	1,96	0,0135	0,2	0,2	
16	1,90	-0,0465	-0,5	-0,6	
17	1,92	-0,0265	-0,3	-0,3	

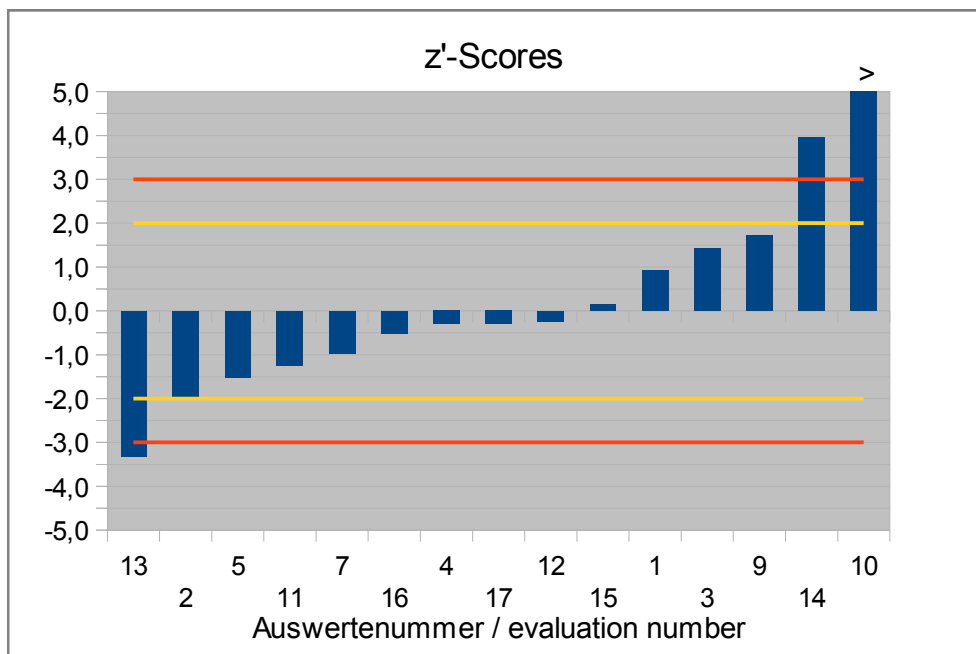


Abb. / Fig. 2: z'-Scores L-Alanin / L-Alanine

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

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results*	14
Number of outliers	0
Mean	1,41
Median	1,44
Robust Mean (X)	1,42
Robust standard deviation (S*)	0,222
Number with 2 replicates	14
Repeatability SD (S_r)	0,0399
Repeatability (CV_r)	2,82%
Reproducibility SD (S_R)	0,209
Reproducibility (CV_R)	14,8%
Target range:	
Target standard deviation σ_{pt}	0,193
Target standard deviation (for Information)	0,0539
lower limit of target range	1,03
upper limit of target range	1,81
Quotient S^*/σ_{pt}	1,1
Standard uncertainty $U(x_{pt})$	0,0741
Quotient $U(x_{pt})/\sigma_{pt}$	0,38
Results in the target range	13
Percent in the target range	93%

* without result of participant no. 10

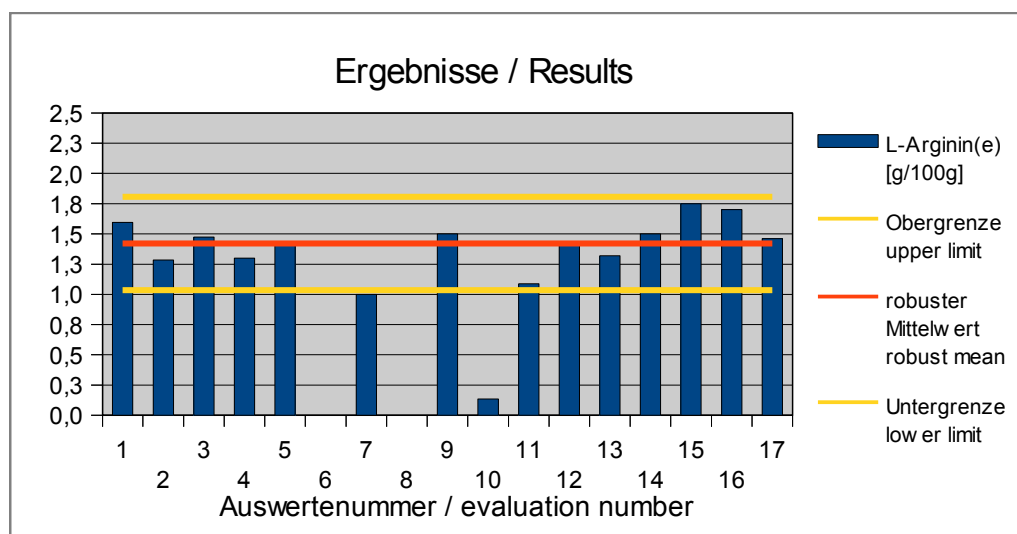


Abb. / Fig. 3: Ergebnisse L-Arginin / Results L-Arginine

Ergebnisse der Teilnehmer:
Results of Participants:

Auswertenummer	L-Arginin(e) [g/100g]	Abweichung [g/100g]	z-Score (σ _{pt})	z-Score (Info)	Hinweis
Evaluation number		Deviation [g/100g]		(Info)	Remark
1	1,60	0,174	0,90	3,2	
2	1,29	-0,136	-0,70	-2,5	
3	1,47	0,0525	0,27	1,0	
4	1,30	-0,121	-0,63	-2,2	
5	1,41	-0,0107	-0,056	-0,20	
6					
7	1,00	-0,421	-2,2	-7,8	
8					
9	1,50	0,0793	0,41	1,5	
10	0,134				Ergebnis ausgeschlossen / Result excluded
11	1,09	-0,333	-1,7	-6,2	
12	1,42	-0,0007	0,00	-0,01	
13	1,32	-0,101	-0,52	-1,9	
14	1,50	0,0793	0,41	1,5	
15	1,75	0,329	1,7	6,1	
16	1,70	0,279	1,4	5,2	
17	1,46	0,0393	0,2	0,7	

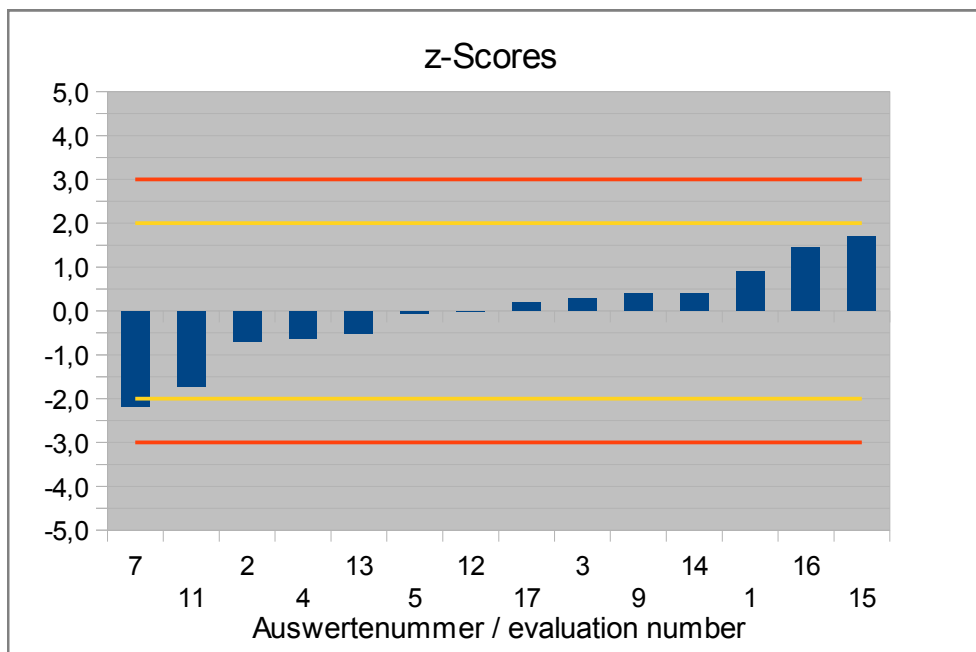


Abb. / Fig. 4: Z-Scores L-Arginin / L-Arginine

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

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results*	14
Number of outliers	0
Mean	3,88
Median	3,82
Robust Mean (X)	3,86
Robust standard deviation (S*)	0,470
Number with 2 replicates	14
Repeatability SD (S_r)	0,204
Repeatability (CV_r)	5,26%
Reproducibility SD (S_R)	0,547
Reproducibility (CV_R)	14,1%
Target range:	
Target standard deviation σ_{pt}'	0,268
Target standard deviation (for Information)	0,126
lower limit of target range	3
upper limit of target range	4
Quotient S^*/σ_{pt}'	1,8
Standard uncertainty $U(x_{pt})$	0,157
Quotient $U(x_{pt})/\sigma_{pt}'$	0,59
Results in the target range	10
Percent in the target range	71%

* without result of participant no. 10 and no.15

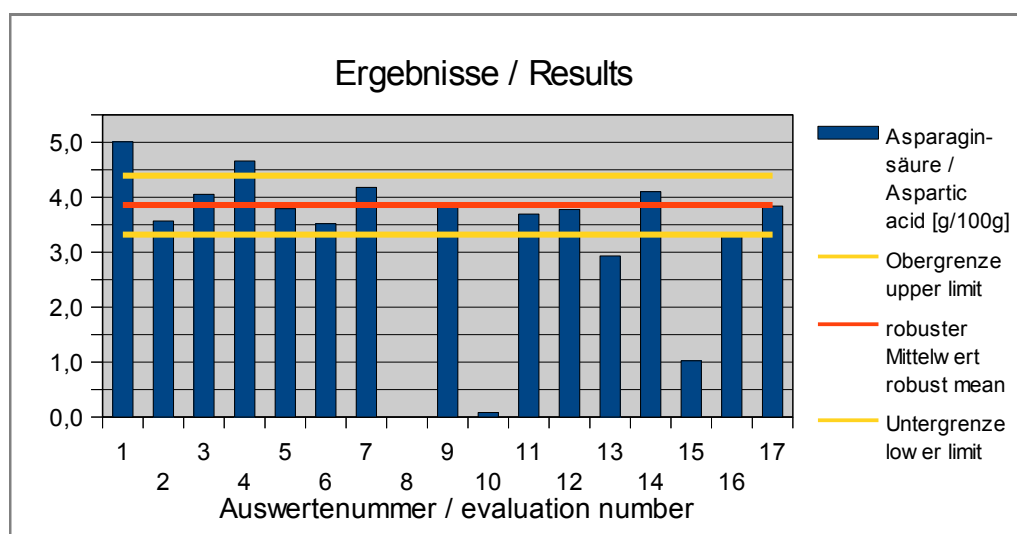


Abb. / Fig. 5: Ergebnisse L-Asparaginsäure / Results L-Aspartic acid

Ergebnisse der Teilnehmer:
Results of Participants:

Auswertenummer Evaluation number	Asparaginsäure / Aspartic acid [g/100g]	Abweichung [g/100g] Deviation [g/100g]	z'-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	5,01	1,15	4,3	9,2	
2	3,57	-0,291	-1,1	-2,3	
3	4,05	0,197	0,7	1,6	
4	4,66	0,804	3,0	6,4	
5	3,79	-0,066	-0,2	-0,5	
6	3,52	-0,336	-1,3	-2,7	
7	4,18	0,324	1,2	2,6	
8					
9	3,90	0,044	0,2	0,3	
10	0,0800				Ergebnis ausgeschlossen / Result excluded
11	3,69	-0,165	-0,6	-1,3	
12	3,78	-0,081	-0,3	-0,6	
13	2,93	-0,926	-3,5	-7,4	
14	4,10	0,244	0,9	1,9	
15	1,02				Ergebnis ausgeschlossen / Result excluded
16	3,30	-0,556	-2,1	-4,4	
17	3,84	-0,016	-0,1	-0,1	

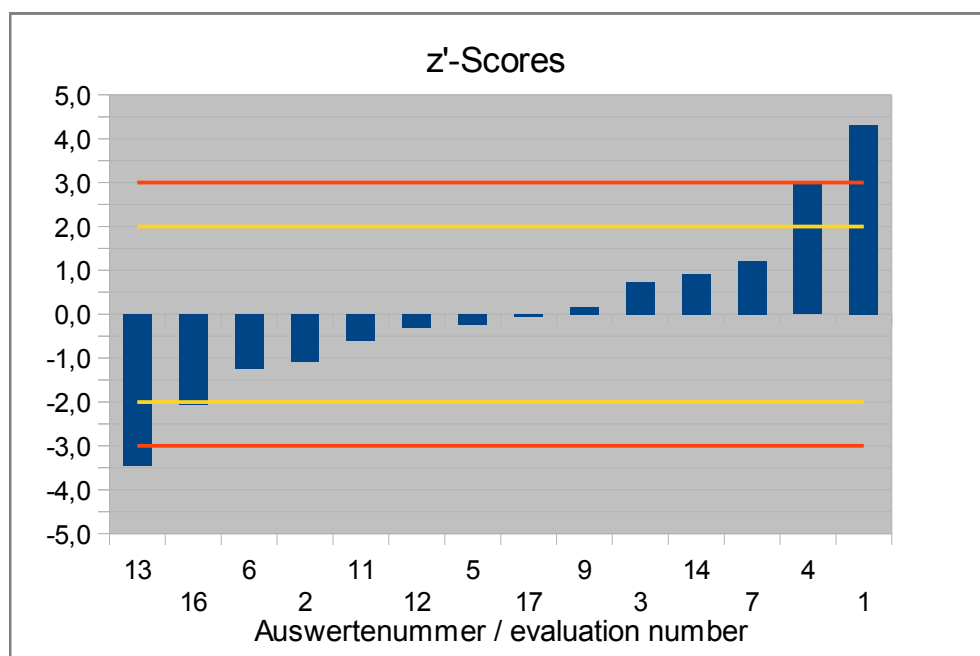


Abb. / Fig. 6: z'-Scores L-Asparaginsäure / L-Aspartic acid

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

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	4
Number of outliers	0
Mean	1,11
Median	1,30
Robust Mean (X)	1,11
Robust standard deviation (S*)	0,734
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 $U(x_{pt})$	
Quotient $U(x_{pt})/\sigma_{pt}$	
Results in the target range	
Percent in the target range	

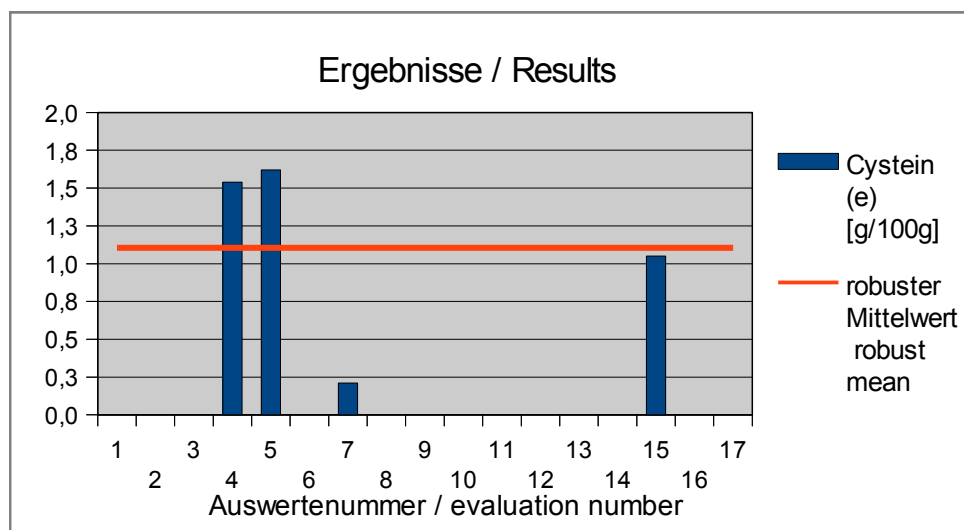


Abb. / Fig. 7: Ergebnisse L-Cystein / Results L-Cysteine

Ergebnisse der Teilnehmer:

Results of Participants:

Auswertenummer	Cystein (e) [g/100g]	Abweichung [g/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis
Evaluation number		Deviation [g/100g]			Remark
1					
2					
3					
4	1,54	0,435			
5	1,62	0,515			
6					
7	0,210	-0,895			
8					
9					
10					
11					
12					
13					
14					
15	1,05	-0,055			
16					
17					

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

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results*	6
Number of outliers	0
Mean	0,247
Median	0,199
Robust Mean (X)	0,216
Robust standard deviation (S*)	0,192
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 $U(x_{pt})$	
Quotient $U(x_{pt})/\sigma_{pt}$	
Results in the target range	
Percent in the target range	

* without result of participant no. 16

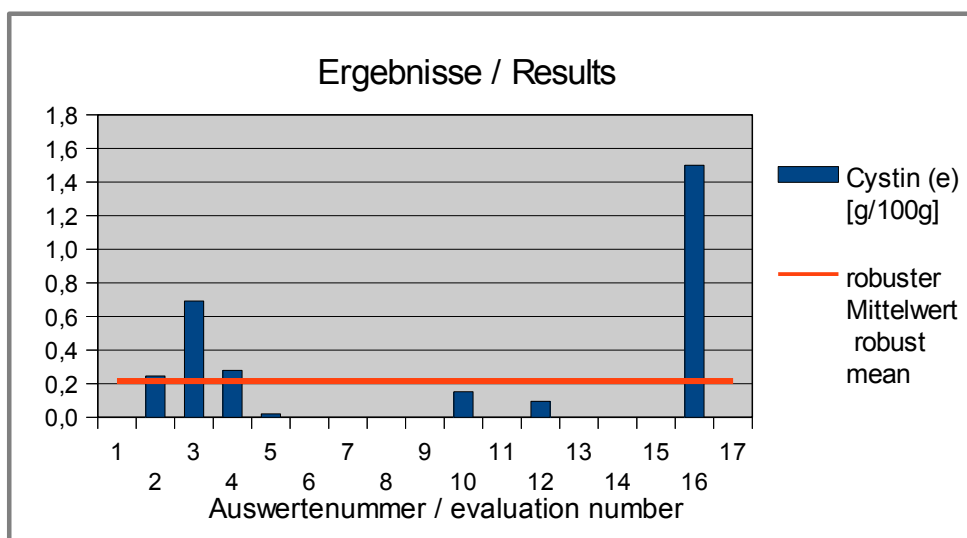


Abb. / Fig. 8: Ergebnisse L-Cystin / Results L-Cystine

Ergebnisse der Teilnehmer:

Results of Participants:

Auswerte- nummer	Cystin (e) [g/100g]	Abweichung [g/100g]	z-Score	z-Score	Hinweis
Evaluation number		Deviation [g/100g]	(σ_{pt})	(Info)	Remark
1					
2	0,246	0,030			
3	0,692	0,476			
4	0,280	0,064			
5	0,0200	-0,196			
6					
7					
8					
9					
10	0,152	-0,064			
11					
12	0,0950	-0,121			
13					
14					
15					
16	1,50				Summe Cystein + Cystin Ergebnis ausgeschlossen / Result excluded
17					

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

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results*	14
Number of outliers	0
Mean	1,49
Median	1,48
Robust Mean (X)	1,48
Robust standard deviation (S*)	0,177
Number with 2 replicates	14
Repeatability SD (S_r)	0,121
Repeatability (CV_r)	8,11%
Reproducibility SD (S_R)	0,199
Reproducibility (CV_R)	13,3%
<i>Target range:</i>	
Target standard deviation σ_{pt}	0,0991
Target standard deviation (for Information)	0,0558
lower limit of target range	1
upper limit of target range	2
Quotient S^*/σ_{pt}	1,8
Standard uncertainty $U(x_{pt})$	0,0592
Quotient $U(x_{pt})/\sigma_{pt}$	0,60
Results in the target range	11
Percent in the target range	79%

* without result of participant no. 10

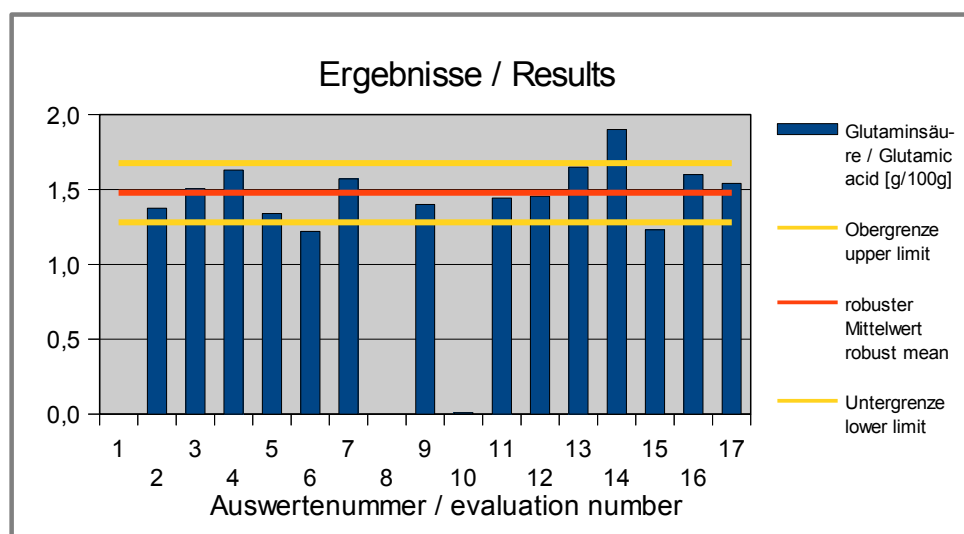


Abb. / Fig. 9: Ergebnisse L-Glutaminsäure/ Results L-Glutamic acid

Ergebnisse der Teilnehmer:
Results of Participants:

Auswertenummer	Glutaminsäure / Glutamic acid [g/100g]	Abweichung [g/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis
Evaluation number		Deviation [g/100g]		(Info)	Remark
1					
2	1,38	-0,104	-1,0	-1,9	
3	1,50	0,0259	0,3	0,5	
4	1,63	0,151	1,5	2,7	
5	1,34	-0,139	-1,4	-2,5	
6	1,22	-0,259	-2,6	-4,6	
7	1,57	0,0914	0,9	1,6	
8					
9	1,40	-0,0786	-0,8	-1,4	
10	0,0100				Ergebnis ausgeschlossen / Result excluded
11	1,44	-0,0367	-0,4	-0,7	
12	1,46	-0,0236	-0,2	-0,4	
13	1,65	0,171	1,7	3,1	
14	1,90	0,421	4,3	7,6	
15	1,23	-0,249	-2,5	-4,5	
16	1,60	0,121	1,2	2,2	
17	1,54	0,0614	0,6	1,1	

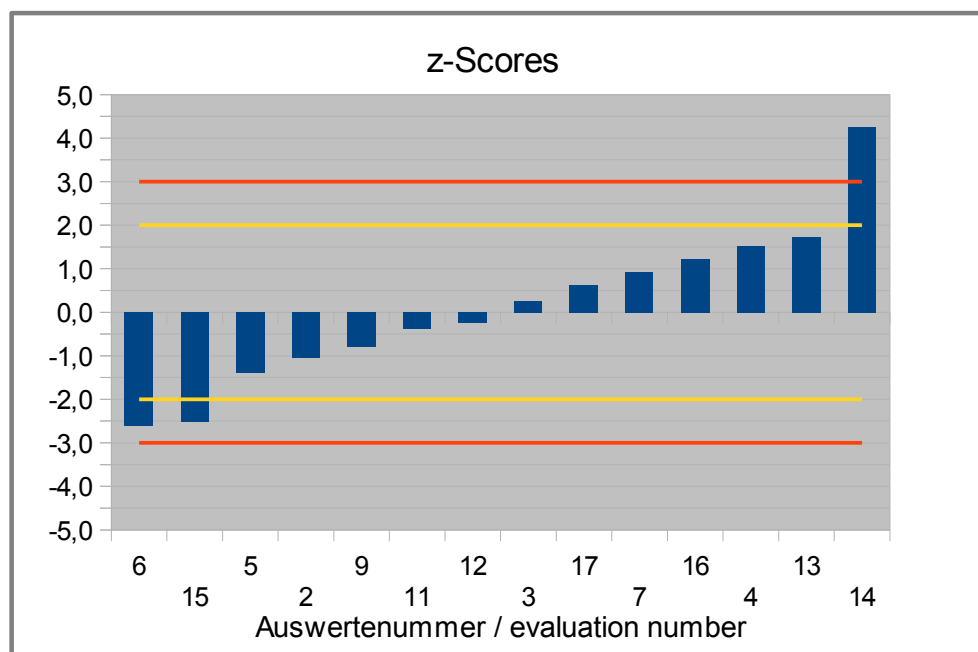


Abb. / Fig. 10: Z-Scores L-Glutaminsäure / L-Glutamic acid

4.7 Glycin/Glycine in g/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	14
Number of outliers	0
Mean	0,757
Median	0,754
Robust Mean (X)	0,755
Robust standard deviation (S*)	0,0863
Number with 2 replicates	14
Repeatability SD (S_r)	0,104
Repeatability (CV_r)	13,7%
Reproducibility SD (S_R)	0,109
Reproducibility (CV_R)	14,4%
Target range:	
Target standard deviation σ_{pt}'	0,0484
Target standard deviation (for Information)	0,0315
lower limit of target range	0,658
upper limit of target range	0,852
Quotient S^*/σ_{pt}'	1,8
Standard uncertainty $U(x_{pt})$	0,0288
Quotient $U(x_{pt})/\sigma_{pt}'$	0,60
Results in the target range	10
Percent in the target range	71%

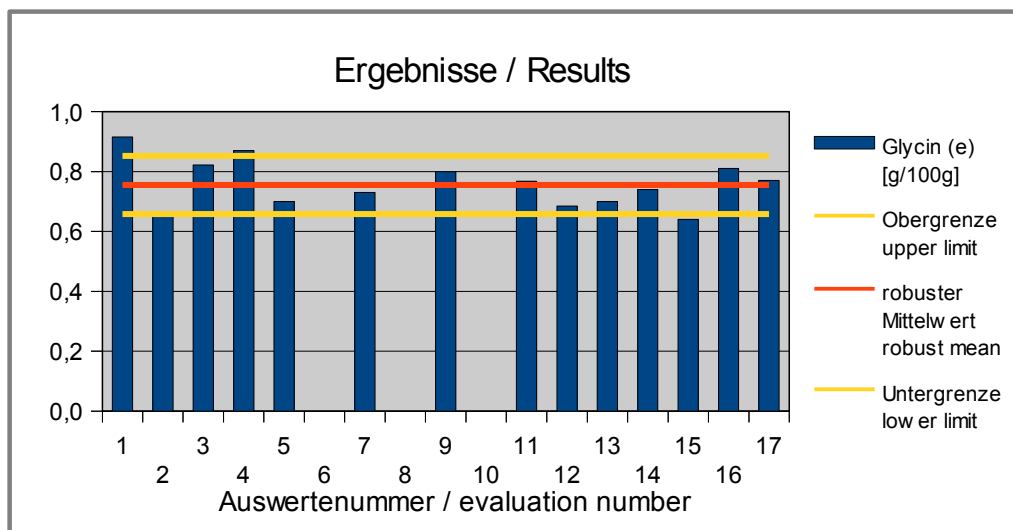


Abb. / Fig. 11: Ergebnisse Glycin/ Results Glycine

Ergebnisse der Teilnehmer:

Results of Participants:

Auswertenummer	Glycin (e) [g/100g]	Abweichung [g/100g]	z'-Score (σ_{pt})	z-Score (Info)	Hinweis
Evaluation number		Deviation [g/100g]		(Info)	Remark
1	0,915	0,160	3,3	5,1	
2	0,654	-0,102	-2,1	-3,2	
3	0,822	0,067	1,4	2,1	
4	0,870	0,115	2,4	3,6	
5	0,700	-0,055	-1,1	-1,8	
6					
7	0,730	-0,025	-0,5	-0,8	
8					
9	0,800	0,045	0,9	1,4	
10					
11	0,768	0,012	0,3	0,4	
12	0,685	-0,070	-1,5	-2,2	
13	0,700	-0,055	-1,1	-1,8	
14	0,740	-0,015	-0,3	-0,5	
15	0,640	-0,115	-2,4	-3,7	
16	0,810	0,055	1,1	1,7	
17	0,770	0,015	0,3	0,5	

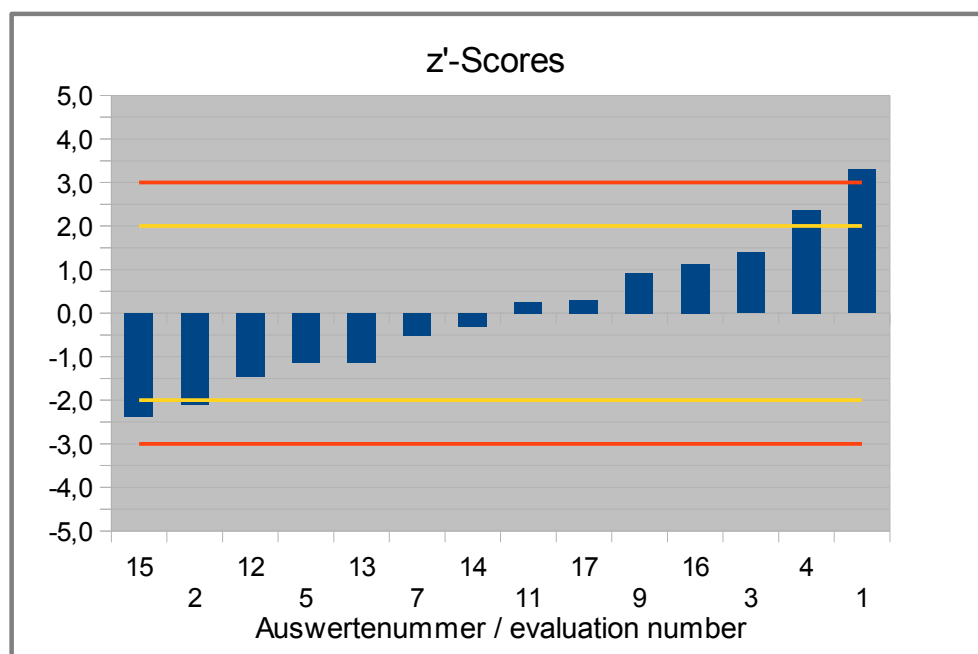


Abb. / Fig. 12: z'-Scores Glycin / Glycine

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

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	14
Number of outliers	2
Mean	1,63
Median	1,80
Robust Mean (X)	1,66
Robust standard deviation (S*)	0,404
Number with 2 replicates	12
Repeatability SD (S_r)	0,169
Repeatability (CV_r)	9,82%
Reproducibility SD (S_R)	0,252
Reproducibility (CV_R)	14,7%
Target range:	
Target standard deviation σ_{pt}	0,267
Target standard deviation (for Information)	0,174
lower limit of target range	1,13
upper limit of target range	2,19
Quotient S^*/σ_{pt}	1,5
Standard uncertainty $U(x_{pt})$	0,135
Quotient $U(x_{pt})/\sigma_{pt}$	0,51
Results in the target range	11
Percent in the target range	79%

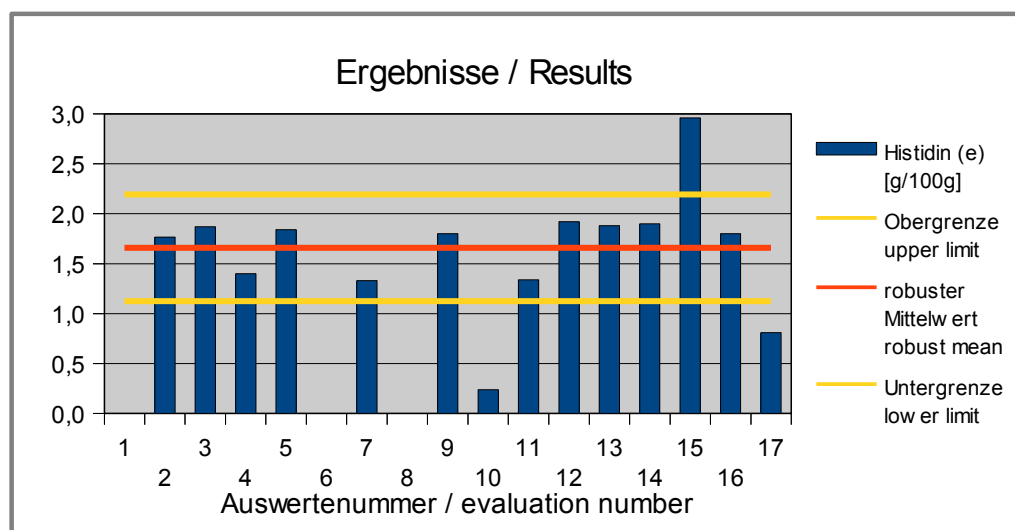


Abb. / Fig. 13: Ergebnisse L-Histidin/ Results L-Histidine

Ergebnisse der Teilnehmer:

Results of Participants:

Auswertenummer	Histidin (e) [g/100g]	Abweichung [g/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis
Evaluation number		Deviation [g/100g]		(Info)	Remark
1					
2	1,77	0,106	0,40	0,61	
3	1,87	0,210	0,79	1,2	
4	1,40	-0,259	-1,0	-1,5	
5	1,84	0,181	0,68	1,0	
6					
7	1,33	-0,329	-1,2	-1,9	
8					
9	1,80	0,141	0,53	0,81	
10	0,239	-1,420	-5,3	-8,2	Ausreisser / Outlier
11	1,34	-0,322	-1,2	-1,8	
12	1,92	0,261	1,0	1,5	
13	1,88	0,221	0,83	1,3	
14	1,90	0,241	0,90	1,4	
15	2,96	1,301	4,9	7,5	Ausreisser / Outlier
16	1,80	0,141	0,53	0,81	
17	0,810	-0,849	-3,2	-4,9	

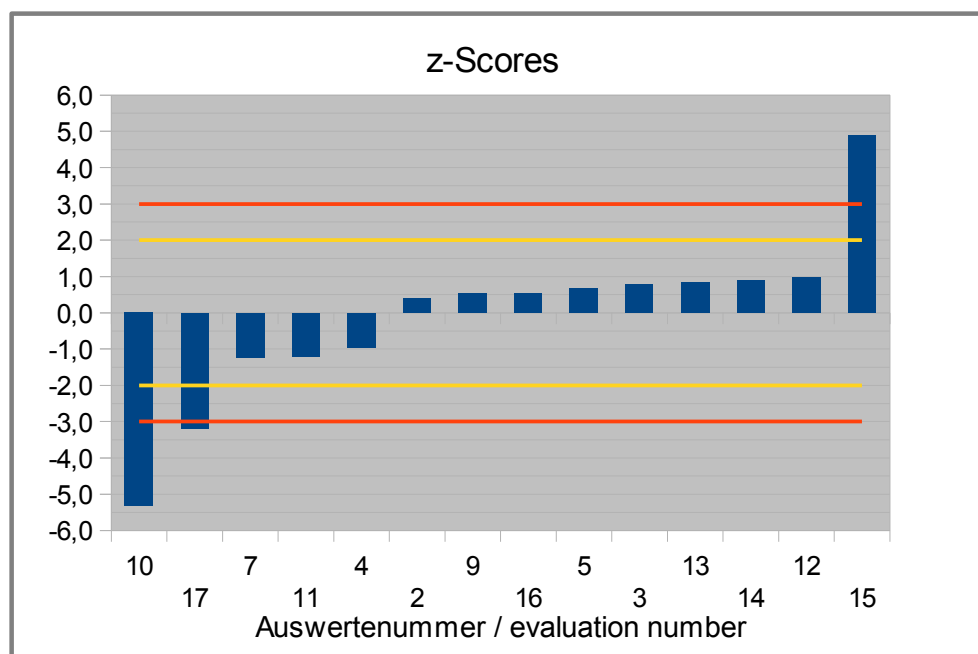


Abb. / Fig. 14: Z-Scores L-Histidin / L-Histidine

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

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results*	15
Number of outliers	0
Mean	3,75
Median	3,83
Robust Mean (X)	3,76
Robust standard deviation (S*)	0,381
Number with 2 replicates	15
Repeatability SD (S_r)	0,133
Repeatability (CV_r)	3,55%
Reproducibility SD (S_R)	0,378
Reproducibility (CV_R)	10,1%
Target range:	
Target standard deviation σ_{pt}'	0,211
Target standard deviation (for Information)	0,123
lower limit of target range	3,34
upper limit of target range	4,18
Quotient S^*/σ_{pt}'	1,8
Standard uncertainty $U(x_{pt})$	0,123
Quotient $U(x_{pt})/\sigma_{pt}'$	0,58
Results in the target range	11
Percent in the target range	73%

* without result of participant no. 10

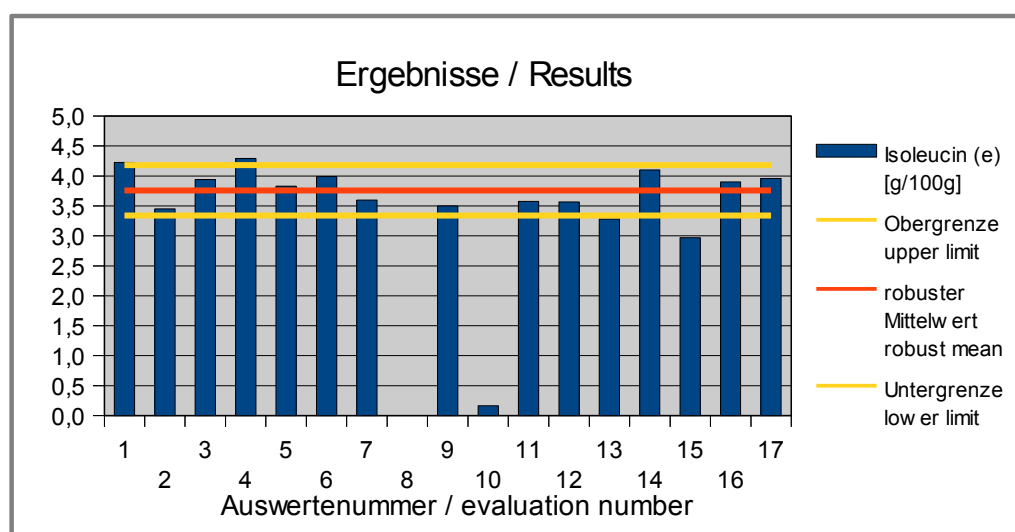


Abb. / Fig. 15: Ergebnisse L-Isoleucin/ Results L-Isoleucine

Ergebnisse der Teilnehmer:
Results of Participants:

Auswertenummer	Isoleucin (e) [g/100g]	Abweichung [g/100g]	z'-Score (σ_{pt})	z-Score (Info)	Hinweis
Evaluation number		Deviation [g/100g]		(Info)	Remark
1	4,23	0,465	2,2	3,8	
2	3,45	-0,310	-1,5	-2,5	
3	3,94	0,182	0,9	1,5	
4	4,29	0,530	2,5	4,3	
5	3,83	0,070	0,3	0,6	
6	3,99	0,230	1,1	1,9	
7	3,60	-0,160	-0,8	-1,3	
8					
9	3,50	-0,260	-1,2	-2,1	
10	0,165				Ergebnis ausgeschlossen / Result excluded
11	3,58	-0,182	-0,9	-1,5	
12	3,57	-0,195	-0,9	-1,6	
13	3,28	-0,480	-2,3	-3,9	
14	4,10	0,340	1,6	2,8	
15	2,97	-0,790	-3,7	-6,4	
16	3,90	0,140	0,7	1,1	
17	3,96	0,200	0,9	1,6	

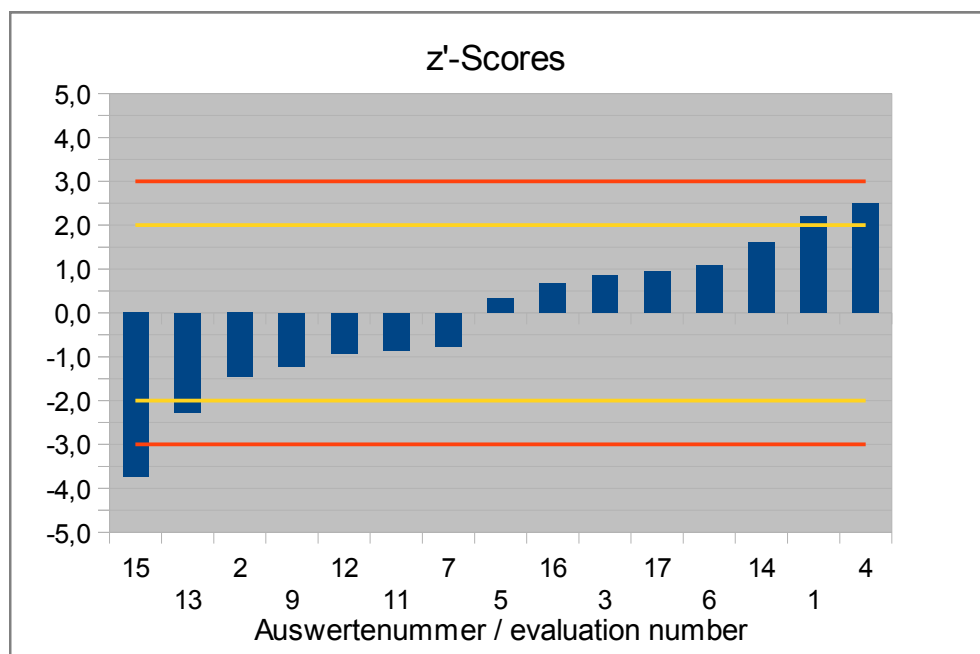


Abb. / Fig. 16: z'-Scores L-Isoleucin / L-Isoleucine

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

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results*	15
Number of outliers	0
Mean	6,55
Median	6,58
Robust Mean (X)	6,56
Robust standard deviation (S*)	0,532
Number with 2 replicates	15
Repeatability SD (S_r)	0,206
Repeatability (CV_r)	3,15%
Reproducibility SD (S_R)	0,557
Reproducibility (CV_R)	8,52%
Target range:	
Target standard deviation σ_{pt}	0,283
Target standard deviation (for Information)	0,198
lower limit of target range	6
upper limit of target range	7
Quotient S^*/σ_{pt}	1,9
Standard uncertainty $U(x_{pt})$	0,172
Quotient $U(x_{pt})/\sigma_{pt}$	0,61
Results in the target range	12
Percent in the target range	80%

* without result of participant no. 10

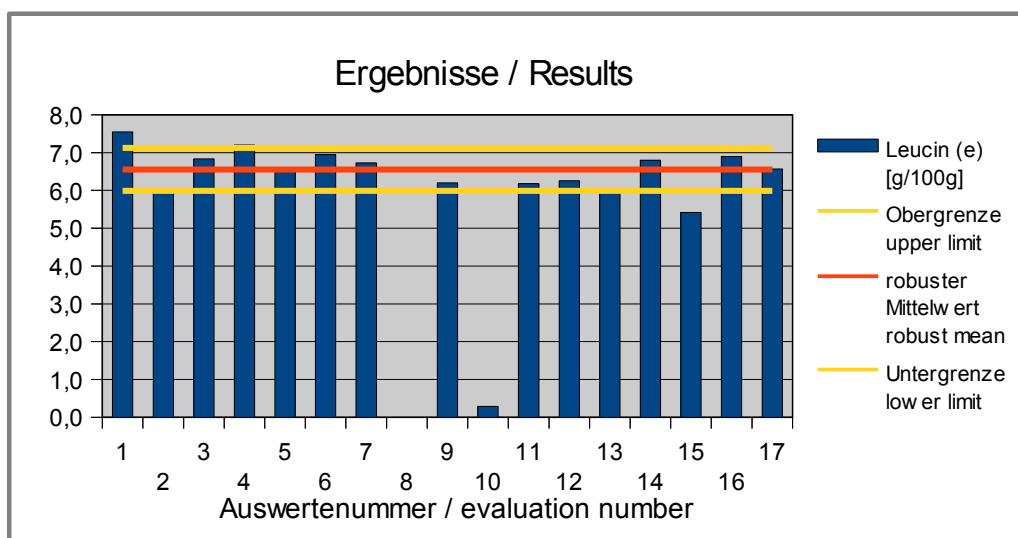


Abb. / Fig. 17: Ergebnisse L-Leucin/ Results L-Leucine

Ergebnisse der Teilnehmer:
Results of Participants:

Auswertenummer	Leucin (e) [g/100g]	Abweichung [g/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis
Evaluation number		Deviation [g/100g]		(Info)	Remark
1	7,55	0,995	3,51	5,03	
2	5,99	-0,565	-2,00	-2,86	
3	6,83	0,276	0,97	1,40	
4	7,20	0,645	2,27	3,26	
5	6,58	0,025	0,09	0,12	
6	6,95	0,395	1,39	2,00	
7	6,73	0,175	0,62	0,88	
8					
9	6,20	-0,355	-1,25	-1,80	
10	0,292				Ergebnis ausgeschlossen / Result excluded
11	6,18	-0,376	-1,33	-1,90	
12	6,26	-0,295	-1,04	-1,50	
13	6,03	-0,525	-1,85	-2,66	
14	6,80	0,245	0,86	1,24	
15	5,42	-1,14	-4,01	-5,75	
16	6,90	0,345	1,22	1,74	
17	6,57	0,015	0,05	0,07	

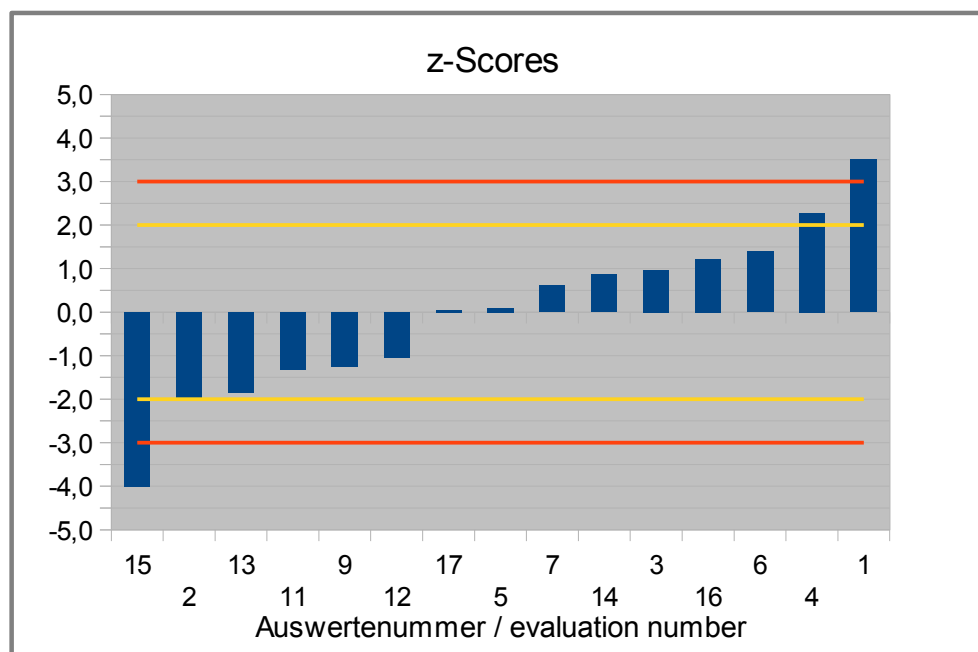


Abb. / Fig. 18: Z-Scores L-Leucin / L-Leucine

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

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	16
Number of outliers	1
Mean	3,94
Median	4,02
Robust Mean (X)	4,01
Robust standard deviation (S*)	0,435
Number with 2 replicates	15
Repeatability SD (S_r)	0,2
Repeatability (CV_r)	5,26%
Reproducibility SD (S_R)	0,4
Reproducibility (CV_R)	10,9%
Target range:	
Target standard deviation σ_{pt}	0,510
Target standard deviation (for Information)	0,130
lower limit of target range	2,99
upper limit of target range	5,03
Quotient S^*/σ_{pt}	0,85
Standard uncertainty $U(x_{pt})$	0,136
Quotient $U(x_{pt})/\sigma_{pt}$	0,27
Results in the target range	14
Percent in the target range	88%

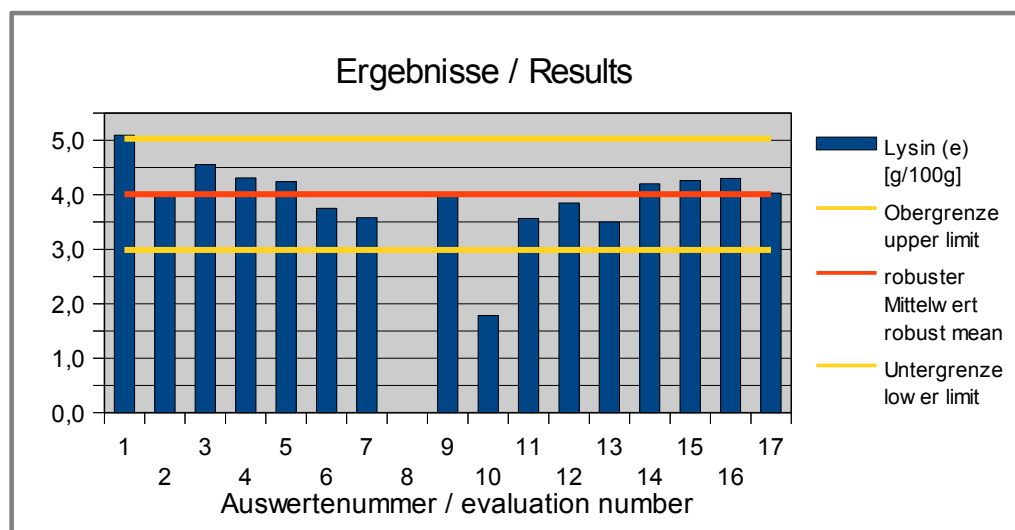


Abb. / Fig. 19: Ergebnisse L-Lysin/ Results L-Lysine

Ergebnisse der Teilnehmer:
Results of Participants:

Auswertenummer	Lysin (e) [g/100g]	Abweichung [g/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis
Evaluation number		Deviation [g/100g]		(Info)	Remark
1	5,10	1,09	2,1	8,3	
2	4,00	-0,015	0,0	-0,1	
3	4,55	0,544	1,1	4,2	
4	4,31	0,300	0,6	2,3	
5	4,24	0,230	0,5	1,8	
6	3,75	-0,260	-0,5	-2,0	
7	3,58	-0,430	-0,8	-3,3	
8					
9	4,00	-0,010	0,0	-0,1	
10	1,78	-2,23	-4,4	-17,1	Ausreisser / Outlier
11	3,57	-0,443	-0,9	-3,4	
12	3,85	-0,160	-0,3	-1,2	
13	3,50	-0,510	-1,0	-3,9	
14	4,20	0,190	0,4	1,5	
15	4,26	0,250	0,5	1,9	
16	4,30	0,290	0,6	2,2	
17	4,03	0,020	0,0	0,2	

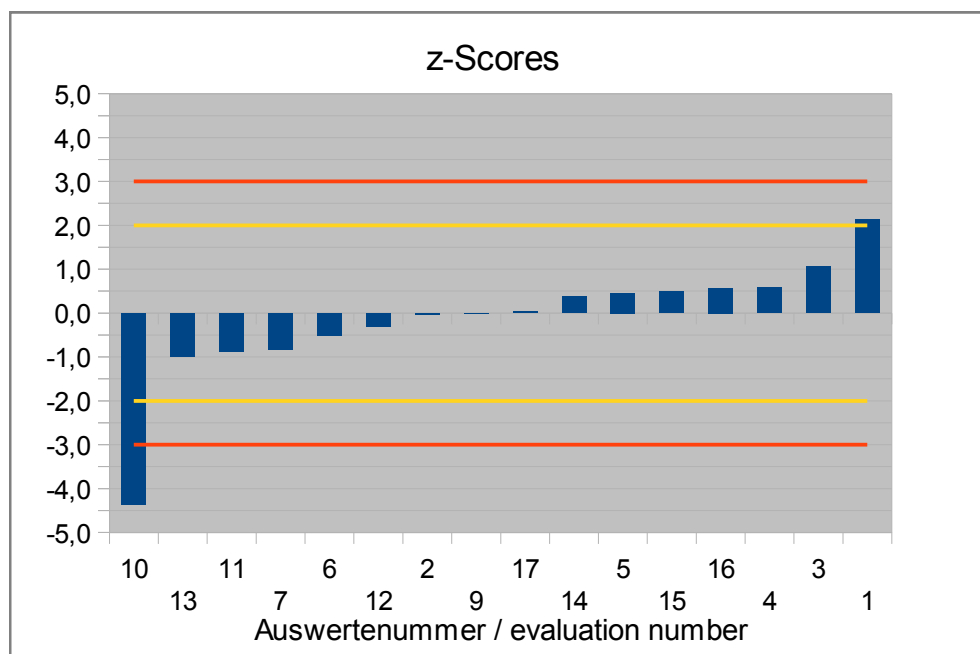


Abb. / Fig. 20: Z-Scores L-Lysin / L-Lysine

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

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results*	14
Number of outliers	0
Mean	1,18
Median	1,17
Robust Mean (X)	1,17
Robust standard deviation (S*)	0,108
Number with 2 replicates	14
Repeatability SD (S_r)	0,0
Repeatability (CV_r)	3,85%
Reproducibility SD (S_R)	0,127
Reproducibility (CV_R)	10,9%
Target range:	
Target standard deviation σ_{pt}	0,0779
Target standard deviation (for Information)	0,0458
lower limit of target range	1,02
upper limit of target range	1,33
Quotient S^*/σ_{pt}	1,4
Standard uncertainty $U(X_{pt})$	0,0361
Quotient $U(X_{pt})/\sigma_{pt}$	0,46
Results in the target range	11
Percent in the target range	79%

* without result of participant no. 10

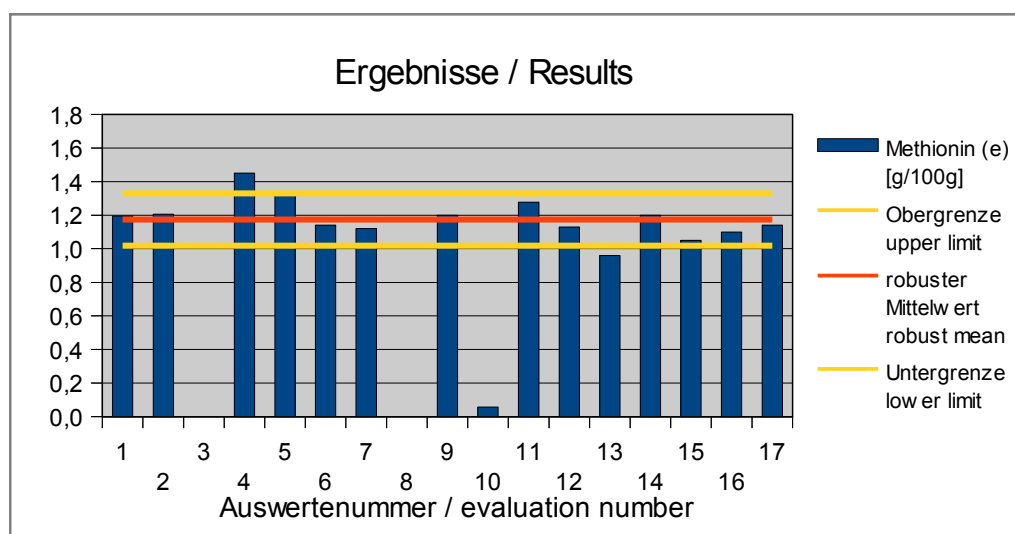


Abb. / Fig. 21: Ergebnisse L-Methionin / Results L-Methionine

Ergebnisse der Teilnehmer:
Results of Participants:

Auswertenummer	Methionin (e) [g/100g]	Abweichung [g/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis
Evaluation number		Deviation [g/100g]		(Info)	Remark
1	1,20	0,0210	0,3	0,5	
2	1,21	0,0310	0,4	0,7	
3					
4	1,45	0,276	3,5	6,0	
5	1,33	0,156	2,0	3,4	
6	1,14	-0,0340	-0,4	-0,7	
7	1,12	-0,0540	-0,7	-1,2	
8					
9	1,20	0,0260	0,3	0,6	
10	0,0575				Ergebnis ausgeschlossen / Result excluded
11	1,28	0,104	1,3	2,3	
12	1,13	-0,0440	-0,6	-1,0	
13	0,960	-0,214	-2,7	-4,7	
14	1,20	0,0260	0,3	0,6	
15	1,05	-0,124	-1,6	-2,7	
16	1,10	-0,0740	-0,9	-1,6	
17	1,14	-0,0340	-0,4	-0,7	

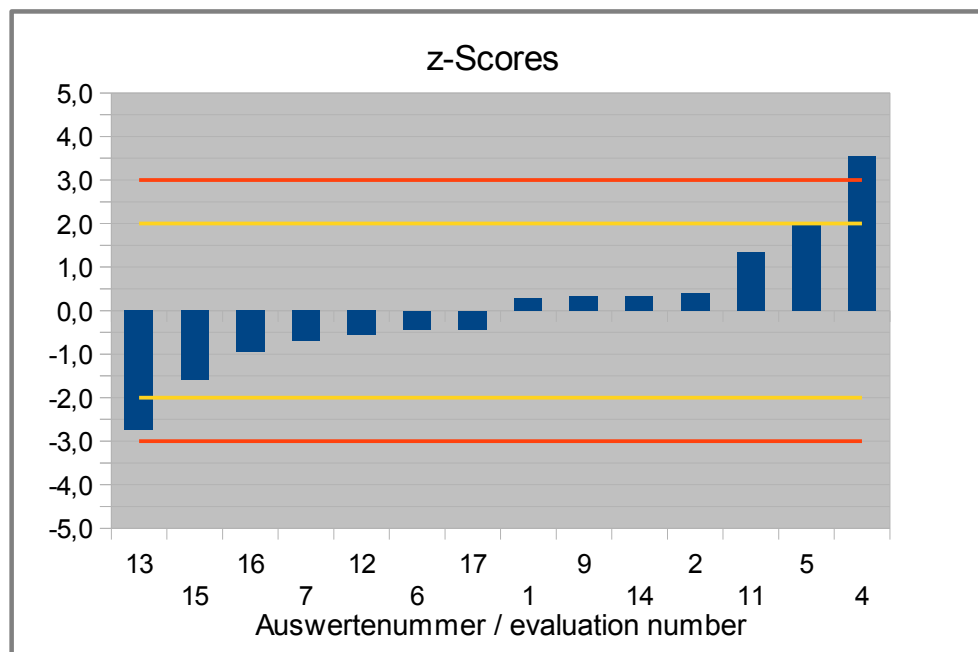


Abb. / Fig. 22: z-Scores L-Methionin / L-Methionine

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

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results*	16
Number of outliers	2
Mean	1,93
Median	1,90
Robust Mean (X)	1,89
Robust standard deviation (S*)	0,157
Number with 2 replicates	14
Repeatability SD (S_r)	0,0550
Repeatability (CV_r)	2,96%
Reproducibility SD (S_R)	0,113
Reproducibility (CV_R)	6,07%
Target range:	
Target standard deviation σ_{pt}	0,114
Target standard deviation (for Information)	0,0687
lower limit of target range	1,66
upper limit of target range	2,12
Quotient S^*/σ_{pt}	1,4
Standard uncertainty $U(x_{pt})$	0,0489
Quotient $U(x_{pt})/\sigma_{pt}$	0,43
Results in the target range	13
Percent in the target range	81%

* without result of participant no. 10

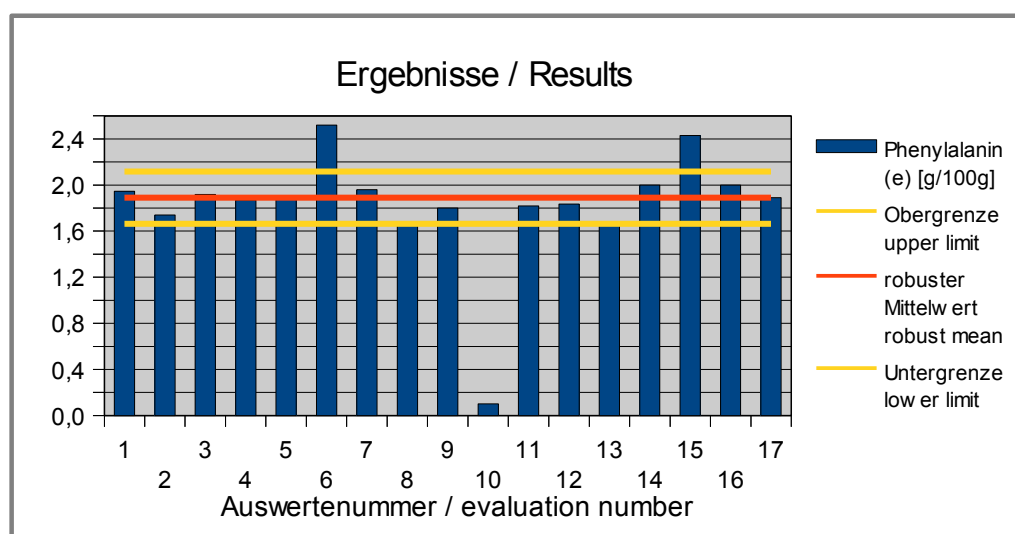


Abb. / Fig. 23: Ergebnisse L-Phenylalanin/ Results L-Phenylalanine

Ergebnisse der Teilnehmer:
Results of Participants:

Auswertenummer	Phenylalanin(e) [g/100g]	Abweichung [g/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis
Evaluation number	[g/100g]	Deviation [g/100g]	(σ_{pt})	(Info)	Remark
1	1,95	0,0541	0,5	0,8	
2	1,74	-0,151	-1,3	-2,2	
3	1,92	0,0281	0,2	0,4	
4	1,87	-0,0209	-0,2	-0,3	
5	1,90	0,0091	0,1	0,1	
6	2,52	0,629	5,5	9,2	Ausreisser / Outlier
7	1,96	0,0691	0,6	1,0	
8	1,67	-0,221	-1,9	-3,2	
9	1,80	-0,0909	-0,8	-1,3	
10	0,102				Ergebnis ausgeschlossen / Result excluded
11	1,82	-0,0727	-0,6	-1,1	
12	1,84	-0,0559	-0,5	-0,8	
13	1,65	-0,241	-2,1	-3,5	
14	2,00	0,109	1,0	1,6	
15	2,43	0,539	4,7	7,8	Ausreisser / Outlier
16	2,00	0,109	1,0	1,6	
17	1,89	-0,0009	0,0	0,0	

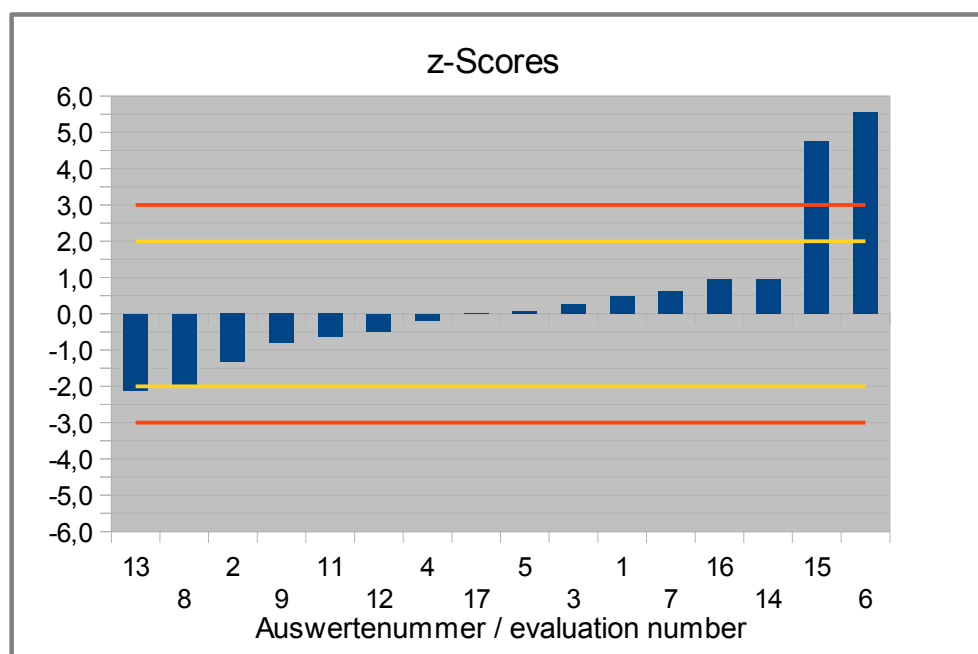


Abb. / Fig. 24: Z-Scores L-Phenylalanin / L-Phenylalanine

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

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	15
Number of outliers	1
Mean	2,28
Median	2,22
Robust Mean (X)	2,23
Robust standard deviation (S*)	0,263
Number with 2 replicates	14
Repeatability SD (S_r)	0,0833
Repeatability (CV_r)	3,77%
Reproducibility SD (S_R)	0,258
Reproducibility (CV_R)	11,7%
Target range:	
Target standard deviation σ_{pt}	0,135
Target standard deviation (for Information)	0,0790
lower limit of target range	1,96
upper limit of target range	2,50
Quotient S^*/σ_{pt}	2,0
Standard uncertainty $U(x_{pt})$	0,0848
Quotient $U(x_{pt})/\sigma_{pt}$	0,63
Results in the target range	12
Percent in the target range	80%

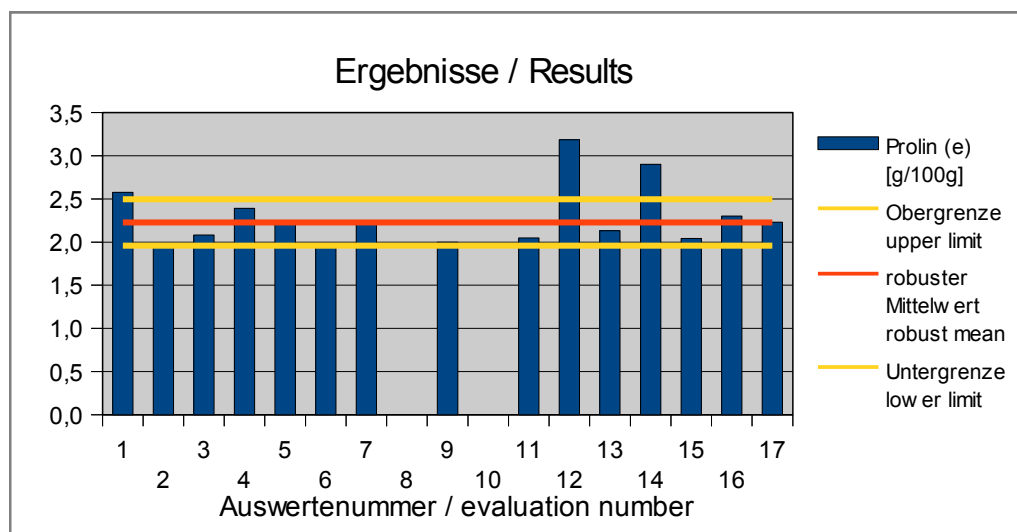


Abb. / Fig. 25: Ergebnisse L-Prolin/ Results L-Proline

Ergebnisse der Teilnehmer:
Results of Participants:

Auswertenummer	Prolin (e) [g/100g]	Abweichung [g/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis
Evaluation number		Deviation [g/100g]		(Info)	Remark
1	2,58	0,347	2,6	4,4	
2	1,97	-0,258	-1,9	-3,3	
3	2,08	-0,144	-1,1	-1,8	
4	2,39	0,162	1,2	2,1	
5	2,23	0,0021	0,0	0,0	
6	1,96	-0,268	-2,0	-3,4	
7	2,22	-0,0079	-0,1	-0,1	
8					
9	2,00	-0,228	-1,7	-2,9	
10					
11	2,05	-0,181	-1,3	-2,3	
12	3,19	0,957	7,1	12,1	Ausreisser / Outlier
13	2,13	-0,0979	-0,7	-1,2	
14	2,90	0,672	5,0	8,5	
15	2,04	-0,188	-1,4	-2,4	
16	2,30	0,0721	0,5	0,9	
17	2,23	0,0021	0,0	0,0	

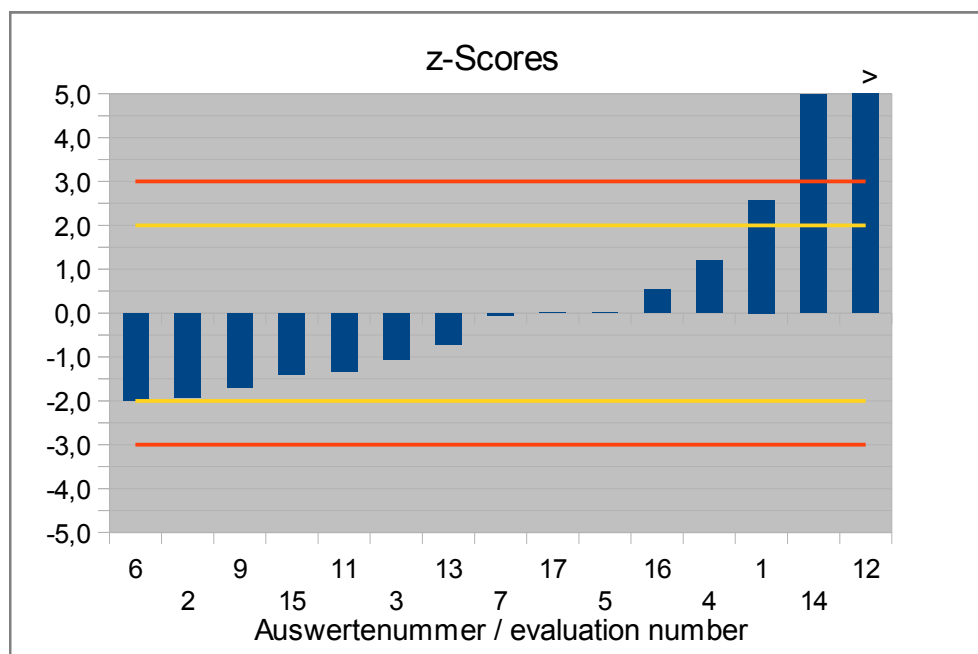


Abb. / Fig. 26: Z-Scores L-Prolin / L-Proline

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

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results*	15
Number of outliers	0
Mean	1,79
Median	1,82
Robust Mean (X)	1,80
Robust standard deviation (S*)	0,256
Number with 2 replicates	15
Repeatability SD (S_r)	0,205
Repeatability (CV_r)	11,5%
Reproducibility SD (S_R)	0,294
Reproducibility (CV_R)	16,5%
Target range:	
Target standard deviation σ_{pt}'	0,157
Target standard deviation (for Information)	0,066
lower limit of target range	1,49
upper limit of target range	2,12
Quotient S^*/σ_{pt}'	1,6
Standard uncertainty $U(x_{pt})$	0,0826
Quotient $U(x_{pt})/\sigma_{pt}'$	0,53
Results in the target range	13
Percent in the target range	87%

* without result of participant no. 10

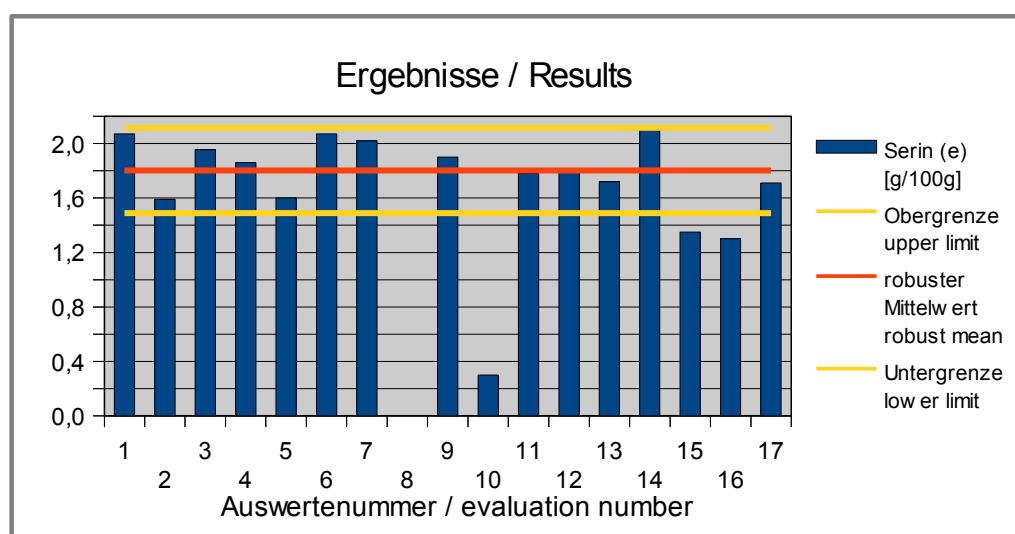


Abb. / Fig. 27: Ergebnisse L-Serin/ Results L-Serine

**Ergebnisse der Teilnehmer:
Results of Participants:**

Auswertenummer	Serin (e) [g/100g]	Abweichung [g/100g]	z'-Score	z-Score	Hinweis
Evaluation number		Deviation [g/100g]	(σ_{pt})	(Info)	Remark
1	2,07	0,268	1,7	4,1	
2	1,59	-0,212	-1,3	-3,2	
3	1,95	0,152	1,0	2,3	
4	1,86	0,0584	0,4	0,9	
5	1,60	-0,202	-1,3	-3,1	
6	2,07	0,268	1,7	4,1	
7	2,02	0,218	1,4	3,3	
8					
9	1,90	0,0984	0,6	1,5	
10	0,300				Ergebnis ausgeschlossen / Result excluded
11	1,78	-0,0210	-0,1	-0,3	
12	1,82	0,0134	0,1	0,2	
13	1,72	-0,0816	-0,5	-1,2	
14	2,10	0,298	1,9	4,5	
15	1,35	-0,452	-2,9	-6,8	
16	1,30	-0,502	-3,2	-7,6	
17	1,71	-0,0916	-0,6	-1,4	

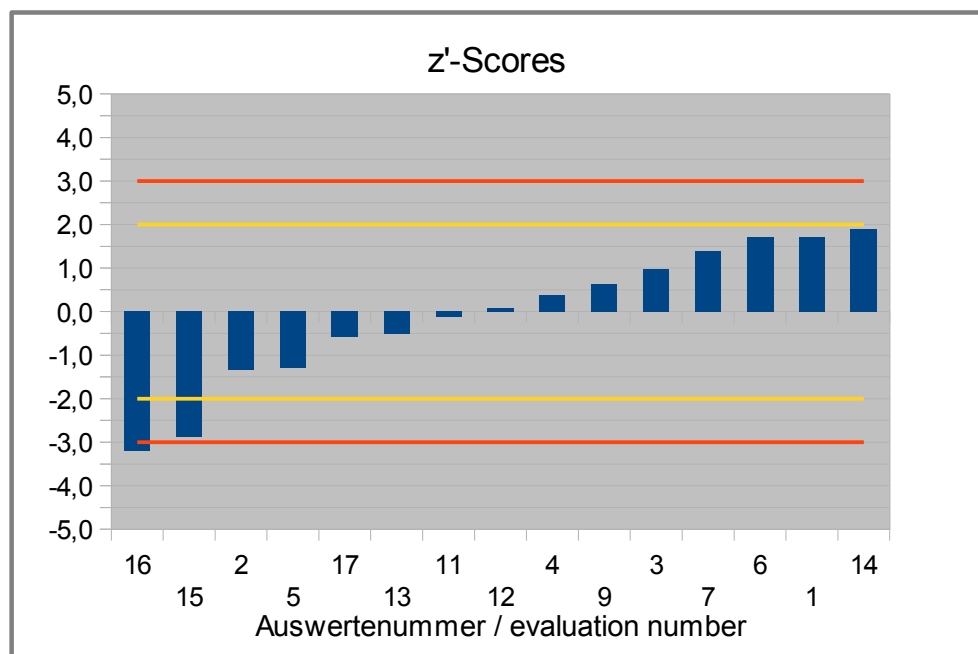


Abb. / Fig. 28: z'-Scores L-Serin/ L-Serine

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

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results*	14
Number of outliers	0
Mean	2,77
Median	2,74
Robust Mean (X)	2,77
Robust standard deviation (S*)	0,236
Number with 2 replicates	14
Repeatability SD (S_r)	0,0800
Repeatability (CV_r)	2,89%
Reproducibility SD (S_R)	0,231
Reproducibility (CV_R)	8,35%
Target range:	
Target standard deviation σ_{pt}	0,165
Target standard deviation (for Information)	0,0949
lower limit of target range	2,44
upper limit of target range	3,09
Quotient S^*/σ_{pt}	1,4
Standard uncertainty $U(x_{pt})$	0,0787
Quotient $U(x_{pt})/\sigma_{pt}$	0,48
Results in the target range	12
Percent in the target range	86%

* without result of participant no. 10

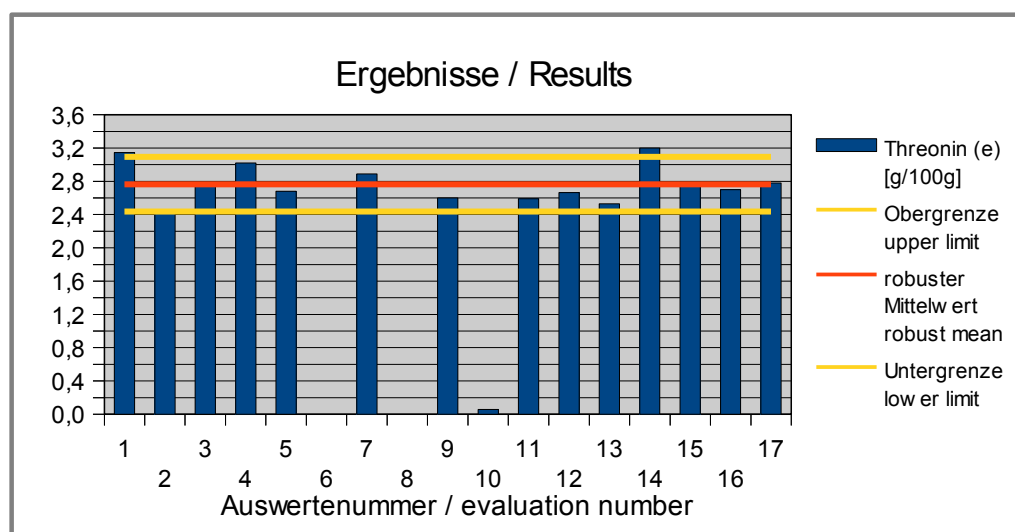


Abb. / Fig. 29: Ergebnisse L-Threonin/ Results L-Threonine

Ergebnisse der Teilnehmer:
Results of Participants:

Auswertenummer	Threonin (e) [g/100g]	Abweichung [g/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis
Evaluation number		Deviation [g/100g]		(Info)	Remark
1	3,15	0,380	2,3	4,0	
2	2,46	-0,310	-1,9	-3,3	
3	2,77	0,0084	0,1	0,1	
4	3,02	0,255	1,5	2,7	
5	2,68	-0,0851	-0,5	-0,9	
6					
7	2,89	0,125	0,8	1,3	
8					
9	2,60	-0,165	-1,0	-1,7	
10	0,0587				Ergebnis ausgeschlossen / Result excluded
11	2,59	-0,173	-1,1	-1,8	
12	2,67	-0,100	-0,6	-1,1	
13	2,53	-0,235	-1,4	-2,5	
14	3,20	0,435	2,6	4,6	
15	2,79	0,0249	0,2	0,3	
16	2,70	-0,0651	-0,4	-0,7	
17	2,78	0,0149	0,1	0,2	

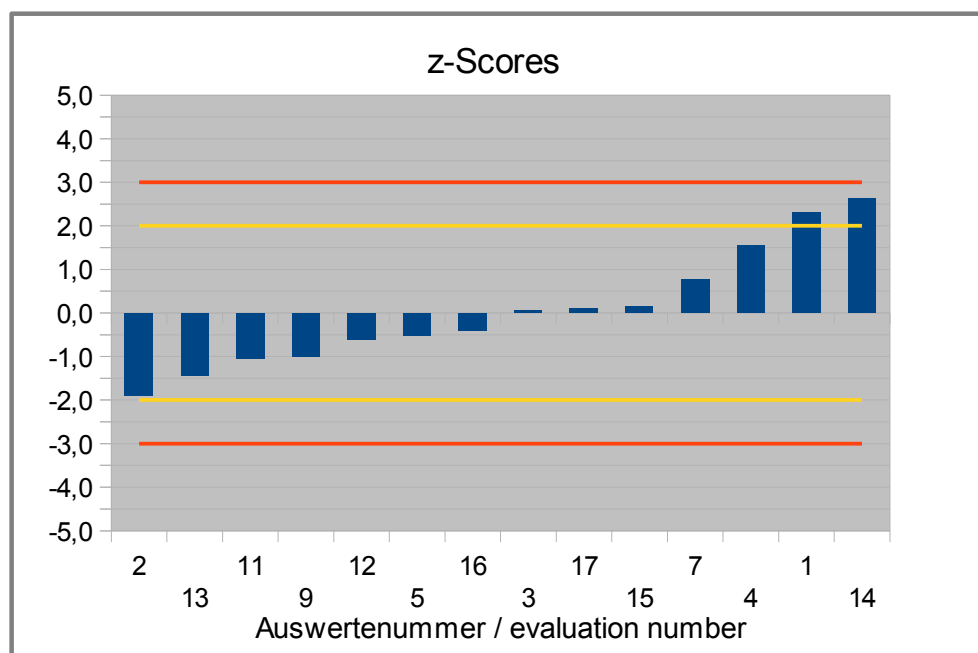


Abb. / Fig. 30: z-Scores L-Threonin / L-Threonine

4.17 L-Tryptophan in g/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	13
Number of outliers	1
Mean	2,81
Median	2,79
Robust Mean (X)	2,75
Robust standard deviation (S*)	0,199
Number with 2 replicates	12
Repeatability SD (S_r)	0,0974
Repeatability (CV_r)	3,58%
Reproducibility SD (S_R)	0,180
Reproducibility (CV_R)	6,61%
Target range:	
Target standard deviation σ_{pt}	0,543
Target standard deviation (for Information)	0,0944
lower limit of target range	1,66
upper limit of target range	3,83
Quotient S^*/σ_{pt}	0,37
Standard uncertainty $U(x_{pt})$	0,0690
Quotient $U(x_{pt})/\sigma_{pt}$	0,13
Results in the target range	12
Percent in the target range	92%

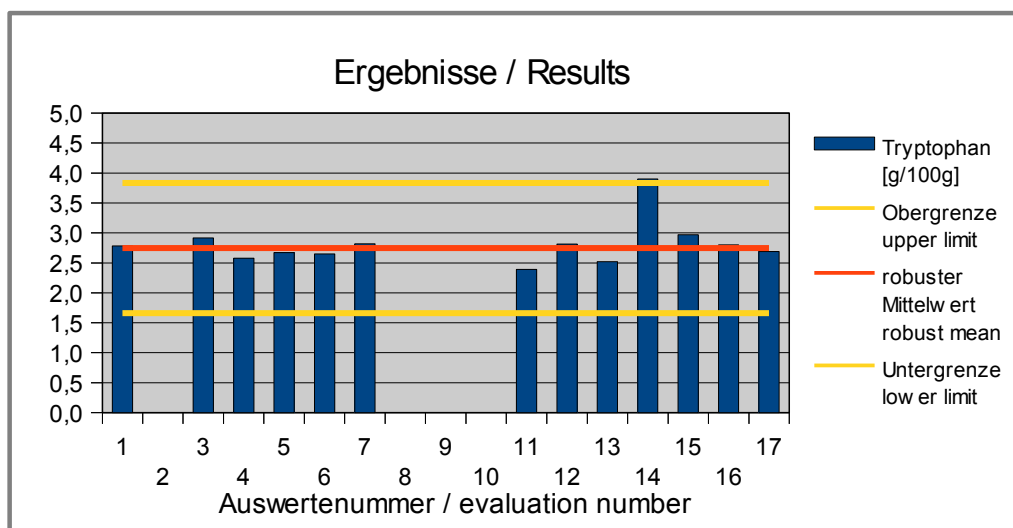


Abb. / Fig. 31: Ergebnisse L-Tryptophan / Results L-Tryptophan

Ergebnisse der Teilnehmer:
Results of Participants:

Auswertenummer	Tryptophan [g/100g]	Abweichung [g/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis
Evaluation number		Deviation [g/100g]		(Info)	Remark
1	2,79	0,0378	0,1	0,4	
2					
3	2,92	0,172	0,3	1,8	
4	2,58	-0,167	-0,3	-1,8	
5	2,67	-0,0772	-0,1	-0,8	
6	2,65	-0,0972	-0,2	-1,0	
7	2,82	0,0728	0,1	0,8	
8					
9					
10					
11	2,39	-0,356	-0,7	-3,8	
12	2,82	0,0678	0,1	0,7	
13	2,52	-0,227	-0,4	-2,4	
14	3,90	1,15	2,1	12,2	Ausreisser / Outlier
15	2,97	0,223	0,4	2,4	
16	2,80	0,0528	0,1	0,6	
17	2,69	-0,0572	-0,1	-0,6	

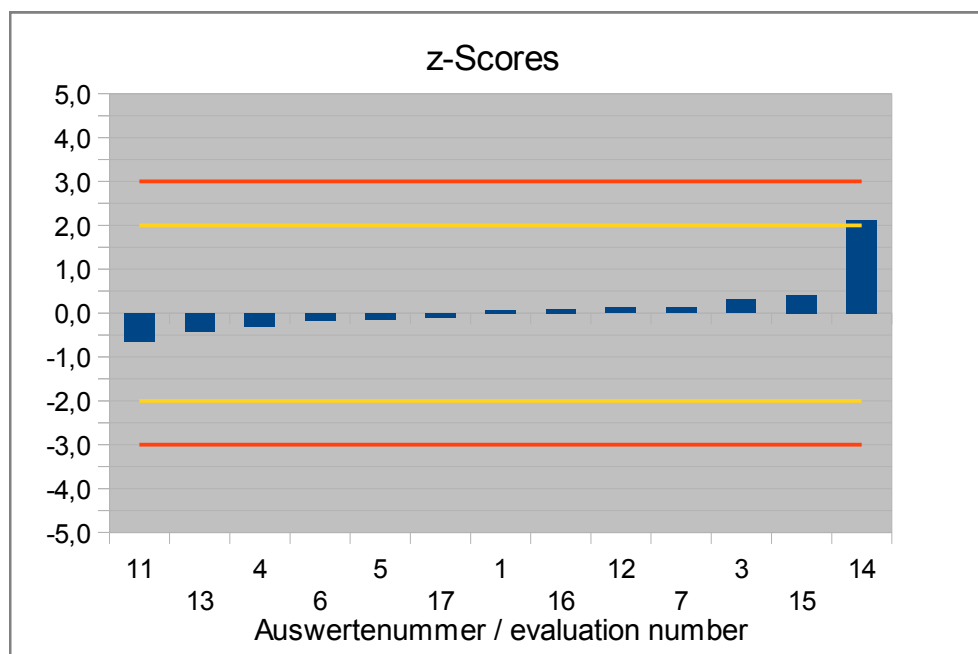


Abb. / Fig. 32: Z-Scores L-Tryptophan

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

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results*	15
Number of outliers	1
Mean	1,15
Median	1,19
Robust Mean (X)	1,18
Robust standard deviation (S*)	0,0878
Number with 2 replicates	14
Repeatability SD (S_r)	0,0422
Repeatability (CV_r)	3,57%
Reproducibility SD (S_R)	0,0709
Reproducibility (CV_R)	5,99%
Target range:	
Target standard deviation σ_{pt}	0,0862
Target standard deviation (for Information)	0,0459
lower limit of target range	1,00
upper limit of target range	1,35
Quotient S^*/σ_{pt}	1,0
Standard uncertainty $U(x_{pt})$	0,0283
Quotient $U(x_{pt})/\sigma_{pt}$	0,33
Results in the target range	14
Percent in the target range	93%

* without result of participant no. 10

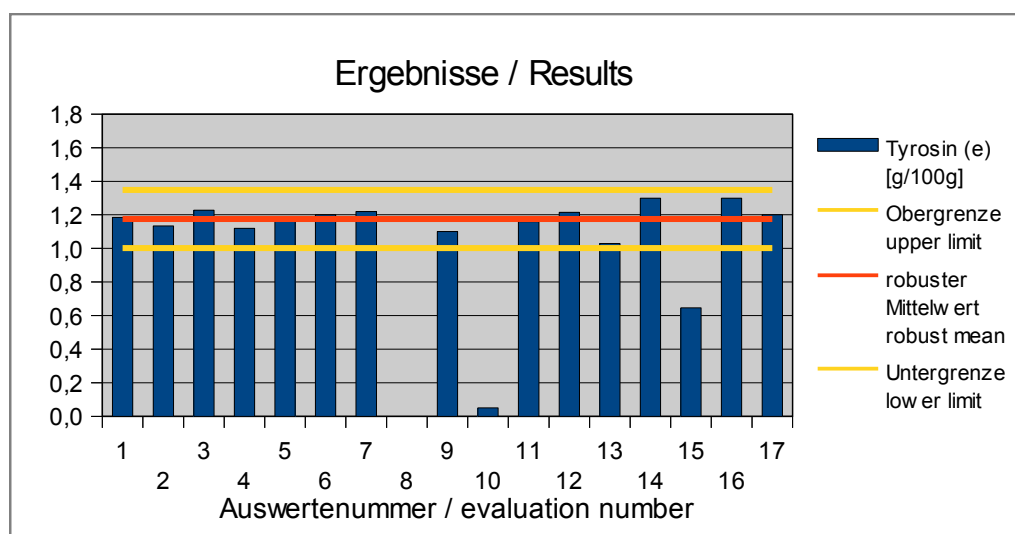


Abb. / Fig. 33: Ergebnisse L-Tyrosin/ Results L-Tyrosine

Ergebnisse der Teilnehmer:
Results of Participants:

Auswertenummer	Tyrosin (e) [g/100g]	Abweichung [g/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis
Evaluation number		Deviation [g/100g]		(Info)	Remark
1	1,19	0,0096	0,1	0,2	
2	1,14	-0,0404	-0,5	-0,9	
3	1,23	0,0530	0,6	1,2	
4	1,12	-0,0554	-0,6	-1,2	
5	1,18	0,0046	0,1	0,1	
6	1,20	0,0246	0,3	0,5	
7	1,22	0,0446	0,5	1,0	
8					
9	1,10	-0,0754	-0,9	-1,6	
10	0,0504				Ergebnis ausgeschlossen / Result excluded
11	1,16	-0,0160	-0,2	-0,3	
12	1,22	0,0396	0,5	0,9	
13	1,03	-0,145	-1,7	-3,2	
14	1,30	0,125	1,4	2,7	
15	0,647	-0,528	-6,1	-11,5	Ausreisser / Outlier
16	1,30	0,125	1,4	2,7	
17	1,20	0,0246	0,3	0,5	

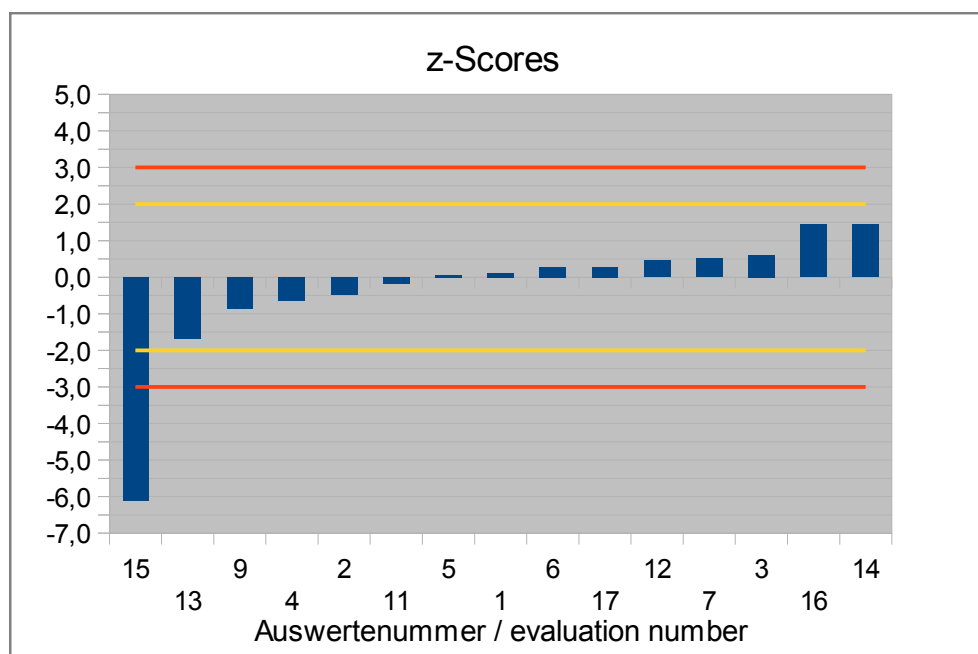


Abb. / Fig. 34: z-Scores L-Tyrosin/ L-Tyrosine

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

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results*	13
Number of outliers	0
Mean	3,05
Median	2,94
Robust Mean (X)	3,04
Robust standard deviation (S*)	0,327
Number with 2 replicates	13
Repeatability SD (S_r)	0,134
Repeatability (CV_r)	4,39%
Reproducibility SD (S_R)	0,319
Reproducibility (CV_R)	10,4%
Target range:	
Target standard deviation σ_{pt}'	0,186
Target standard deviation (for Information)	0,103
lower limit of target range	2,67
upper limit of target range	3,41
Quotient S^*/σ_{pt}'	1,8
Standard uncertainty $U(x_{pt})$	0,113
Quotient $U(x_{pt})/\sigma_{pt}'$	0,61
Results in the target range	10
Percent in the target range	77%

* without result of participant no. 10

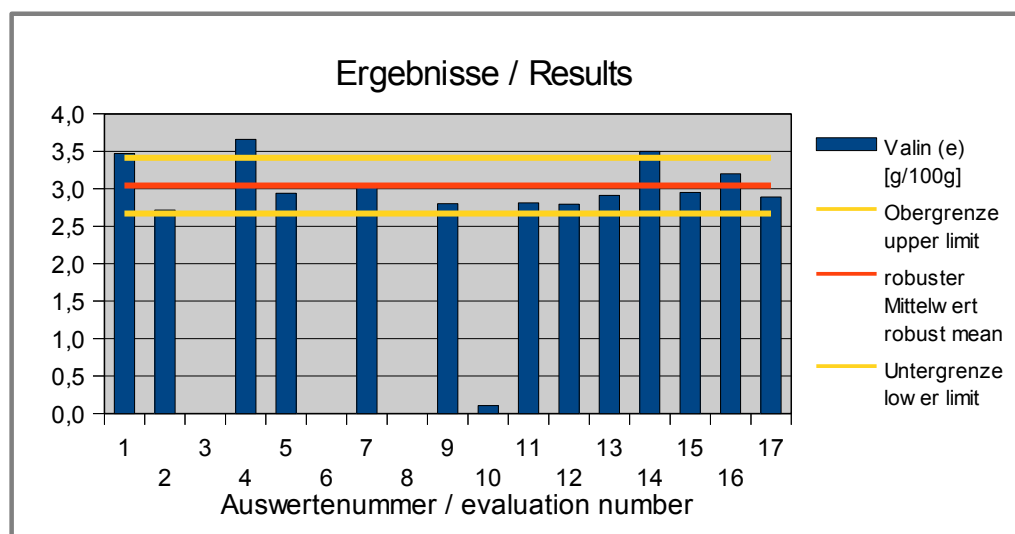


Abb. / Fig. 35: Ergebnisse L-Valin/ Results L-Valine

Ergebnisse der Teilnehmer:
Results of Participants:

Auswertenummer	Valin (e) [g/100g]	Abweichung [g/100g]	z'-Score (σ_{pt})	z-Score (Info)	Hinweis
Evaluation number		Deviation [g/100g]		(Info)	Remark
1	3,47	0,428	2,3	4,2	
2	2,72	-0,327	-1,8	-3,2	
3					
4	3,66	0,618	3,3	6,0	
5	2,94	-0,102	-0,5	-1,0	
6					
7	3,03	-0,0117	-0,1	-0,1	
8					
9	2,80	-0,242	-1,3	-2,3	
10	0,1080				Ergebnis ausgeschlossen / Result excluded
11	2,81	-0,230	-1,2	-2,2	
12	2,80	-0,247	-1,3	-2,4	
13	2,91	-0,132	-0,7	-1,3	
14	3,50	0,458	2,5	4,5	
15	2,95	-0,092	-0,5	-0,9	
16	3,20	0,158	0,8	1,5	
17	2,89	-0,152	-0,8	-1,5	

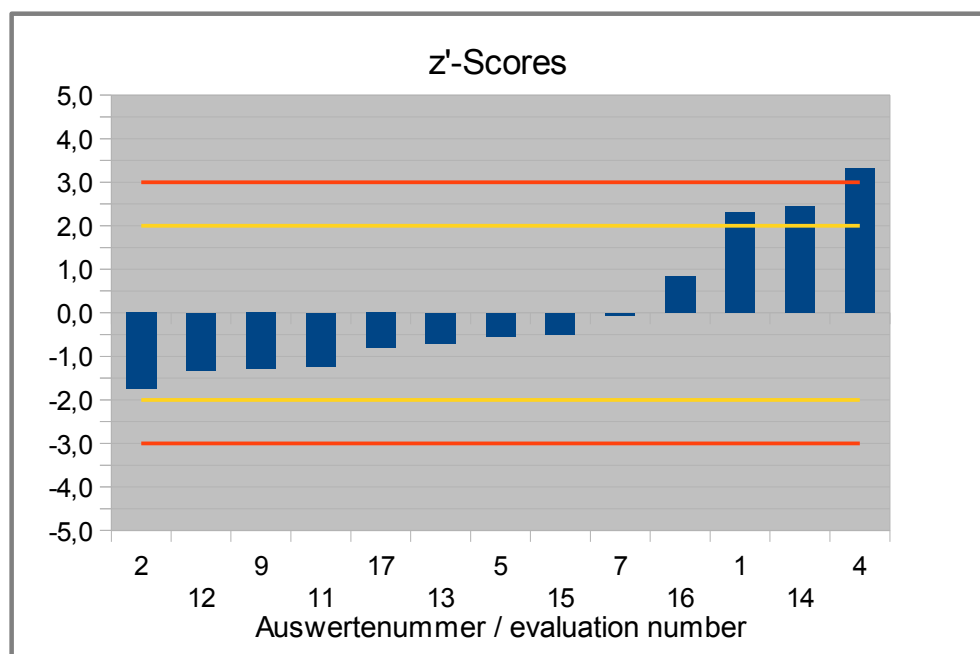


Abb. / Fig. 36: z'-Scores L-Valin/ L-Valine

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	Teilnehmer	Einheit	Proben-Nr. A	Proben-Nr. B	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis A	Ergebnis A	Bestimmungsgrenze	Inkl. WF	Wiederfindungsrate [%]
Analyte	Participant	Unit	Sample No. A	Sample No. B	Date of analysis	Result (Mean)	Result A	Result A	LOQ	Incl. RR	Recovery rate [%]
L-Alanin(e)	1	g/100g	6	86	27.10.17	2,03	2,8	1,26		No	
	2	g/100g	42	50	04.10.17	1,77	1,73	1,81			
	3	g/100g	22	70	03.11.17	2,0729	2,1931	1,9528	0,3	no	-
	4	g/100g	25	67	27.10.17	1,92	1,93	1,91	0,01	no	
	5	g/100g	40	52	27.10.17	1,81	1,91	1,71	0,01	no	not determined
	6	g/100g	8	84	25.10.17					no	
	7	g/100g	21	71	25.10.17	1,86	2,08	1,64		no	92,6
	8	g/100g	14	78							
	9	g/100g	15	77	01.11.17	2,1	2	2,2	0,03	no	
	10	g/100g	26	66	02.11.17	2,6595	2,9110	2,4080			
	11	g/100g	28	64	01.10.17	1,8353	1,7720	1,8984	0,02	no	
	12	g/100g	19	73	23.09.17	1,925	1,76	2,09	<0,01	yes	101,5
	13	g/100g	3	89	10.10.17	1,65	1,65	1,64	<0,1	no	
	14	g/100g	37	55	14.10.17	2,3	2,2	2,4		no	-
	15	g/100g	44	48		1,96	1,98	1,93	0,00001		100
	16	g/100g	11	81	18.10.17	1,9	2	1,8	0,05	no	80-120
	17	g/100g	43	49	4.10.	1,92	1,86	1,97		no	no

Parameter	Teilnehmer	Einheit	Proben-Nr. A	Proben-Nr. B	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis A	Ergebnis A	Bestimmungsgrenze	Inkl. WF	Wiederfindungsrate [%]
Analyte	Participant	Unit	Sample No. A	Sample No. B	Date of analysis	Result (Mean)	Result A	Result A	LOQ	Incl. RR	Recovery rate [%]
L-Arginin(e)	1	g/100g	6	86	27.10.17	1,595	1,53	1,66		No	
	2	g/100g	42	50	04.10.17	1,285	1,28	1,29			
	3	g/100g	22	70	03.11.17	1,47321	1,45710	1,48932	0,3	no	-
	4	g/100g	25	67	27.10.17	1,3	1,24	1,37	0,01	no	
	5	g/100g	40	52	27.10.17	1,41	1,42	1,4	0,01	no	
	6	g/100g	8	84							
	7	g/100g	21	71		1	1,02	0,99		no	94,7
	8	g/100g	14	78							
	9	g/100g	15	77	01.11.17	1,5	1,5	1,5	0,07	no	
	10	g/100g	26	66	02.11.17	0,1335	0,142	0,125			
	11	g/100g	28	64	01.10.17	1,0878	1,1116	1,0640	0,03	no	
	12	g/100g	19	73	23.09.17	1,42	1,4	1,44	<0,01	yes	
	13	g/100g	3	89	10.10.17	1,32	1,33	1,31	<0,1	no	
	14	g/100g	37	55		1,5	1,5	1,5			
	15	g/100g	44	48		1,75	1,78	1,72	0,00001		100
	16	g/100g	11	81	18.10.17	1,7	1,7	1,7	0,05		
	17	g/100g	43	49	4.10.	1,46	1,45	1,47			

Parameter	Teilnehmer	Einheit	Proben-Nr. A	Proben-Nr. B	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis A	Ergebnis A	Bestimmungsgrenze	Inkl. WF	Wiederfindungsrate [%]
Analyte	Participant	Unit	Sample No. A	Sample No. B	Date of analysis	Result (Mean)	Result A	Result A	LOQ	Incl. RR	Recovery rate [%]
L-Asparaginsäure / L-Aspartic aci	1	g/100g	6	86	27.10.17	5,01	5,36	4,66		No	
	2	g/100g	42	50	04.10.17	3,565	3,66	3,47			
	3	g/100g	22	70	03.11.17	4,05316	4,10548	4,00084	0,3	no	-
	4	g/100g	25	67	27.10.17	4,66	4,38	4,93	0,01	no	
	5	g/100g	40	52	27.10.17	3,79	3,84	3,74	0,01	no	
	6	g/100g	8	84		3,52	3,49	3,55	0,03		
	7	g/100g	21	71		4,18	4,12	4,23		no	91,8
	8	g/100g	14	78							
	9	g/100g	15	77	01.11.17	3,9	3,9	3,9	0,07	no	
	10	g/100g	26	66	02.11.17	0,079	0,086	0,072			
	11	g/100g	28	64	01.10.17	3,69083	3,70040	3,68127	0,03	no	
	12	g/100g	19	73	23.09.17	3,775	3,85	3,7	<0,01	yes	
	13	g/100g	3	89	10.10.17	2,93	2,87	2,98	<0,1	no	
	14	g/100g	37	55		4,1	4,1	4			
	15	g/100g	44	48		1,02	0,93	1,1	0,00001		100
	16	g/100g	11	81	18.10.17	3,3	3	3,5	0,05		
	17	g/100g	43	49	4.10.	3,84	3,83	3,84			

Parameter	Teilnehmer	Einheit	Proben-Nr. A	Proben-Nr. B	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis A	Ergebnis A	Bestimmungsgrenze	Inkl. WF	Wiederfindungsrate [%]
Analyte	Participant	Unit	Sample No. A	Sample No. B	Date of analysis	Result (Mean)	Result A	Result A	LOQ	Incl. RR	Recovery rate [%]
L-Cystein(e)	1	g/100g	6	86	27.10.17					No	
	2	g/100g	42	50							
	3	g/100g	22	70	-	NT	NT	NT	-	-	-
	4	g/100g	25	67	27.10.17	1,54	1,22	1,86	0,01	no	
	5	g/100g	40	52		1,62	1,59	1,65	0,01	no	
	6	g/100g	8	84							
	7	g/100g	21	71		0,21	0,2	0,21		no	102,8
	8	g/100g	14	78							
	9	g/100g	15	77	01.11.17	< 0,20	< 0,20	< 0,20	0,07	no	
	10	g/100g	26	66	02.11.17	-	-	-			
	11	g/100g	28	64							
	12	g/100g	19	73	23.09.17					yes	
	13	g/100g	3	89							
	14	g/100g	37	55							
	15	g/100g	44	48		1,05	1,11	0,982	0,00001		100
	16	g/100g	11	81	18.10.17						
	17	g/100g	43	49	4.10.						

Parameter	Teilnehmer	Einheit	Proben-Nr. A	Proben-Nr. B	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis A	Ergebnis A	Bestimmungsgrenze	Inkl. WF	Wiederfindungsrate [%]
Analyte	Participant	Unit	Sample No. A	Sample No. B	Date of analysis	Result (Mean)	Result A	Result A	LOQ	Incl. RR	Recovery rate [%]
L-Cystin(e)	1	g/100g	6	86	27.10.17					No	
	2	g/100g	42	50	04.10.17	0,2455	0,235	0,26			
	3	g/100g	22	70	03.11.17	0,69212	0,67815	0,70608	0,3	no	-
	4	g/100g	25	67	27.10.17	0,28	0,3	0,26	0,01	no	
	5	g/100g	40	52	27.10.17	0,02	0,02	0,02	0,01	no	
	6	g/100g	8	84							
	7	g/100g	21	71							
	8	g/100g	14	78							
	9	g/100g	15	77							
	10	g/100g	26	66	02.11.17	0,1515	0,169	0,134			
	11	g/100g	28	64							
	12	g/100g	19	73	23.09.17	0,1	0,05	0,14	<0,01	yes	
	13	g/100g	3	89	10.10.17	<0,1	<0,1	<0,1	<0,1	no	
	14	g/100g	37	55		nb	nb	nb	0,4		
	15	g/100g	44	48					0,00001		100
	16	g/100g	11	81	18.10.17	1,5	1,5	1,4	0,05		
	17	g/100g	43	49	4.10.						

Parameter	Teilnehmer	Einheit	Proben-Nr. A	Proben-Nr. B	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis A	Ergebnis A	Bestimmungsgrenze	Inkl. WF	Wiederfindungsrate [%]
Analyte	Participant	Unit	Sample No. A	Sample No. B	Date of analysis	Result (Mean)	Result A	Result A	LOQ	Incl. RR	Recovery rate [%]
L-Glutaminsäure / L-Glutaminc acid	1	g/100g	6	86	27.10.17					No	
	2	g/100g	42	50	04.10.17	1,375	1,39	1,36			
	3	g/100g	22	70	03.11.17	1,50451	1,53441	1,47460	0,3	no	-
	4	g/100g	25	67	27.10.17	1,63	1,59	1,67	0,01	no	
	5	g/100g	40	52	27.10.17	1,34	1,36	1,32	0,01	no	
	6	g/100g	8	84		1,22	1,23	1,2	0,03		
	7	g/100g	21	71		1,57	1,55	1,58		no	97,1
	8	g/100g	14	78							
	9	g/100g	15	77	01.11.17	1,4	1,4	1,5	0,07	no	
	10	g/100g	26	66	02.11.17	0,01165	0,0097	0,0136			
	11	g/100g	28	64	01.10.17	1,44188	1,47947	1,40430	0,07	no	
	12	g/100g	19	73	23.09.17	1,455	1,4	1,51	<0,01	yes	
	13	g/100g	3	89	10.10.17	1,65	1,93	1,37	<0,1	no	
	14	g/100g	37	55		1,9	1,9	1,9			
	15	g/100g	44	48		1,23	1,17	1,29	0		100
	16	g/100g	11	81	18.10.17	1,6	1,7	1,5	0,05		
	17	g/100g	43	49	4.10.	1,54	1,53	1,55			

Parameter	Teilnehmer	Einheit	Proben-Nr. A	Proben-Nr. B	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis A	Ergebnis A	Bestimmungsgrenze	Inkl. WF	Wiederfindungsrate [%]
Analyte	Participant	Unit	Sample No. A	Sample No. B	Date of analysis	Result (Mean)	Result A	Result A	LOQ	Incl. RR	Recovery rate [%]
Glycin(e)	1	g/100g	6	86	27.10.17	0,915	1	0,83		No	
	2	g/100g	42	50	04.10.17	0,6535	0,629	0,678			
	3	g/100g	22	70	03.11.17	0,82238	0,85379	0,79097	0,3	no	-
	4	g/100g	25	67	27.10.17	0,87	0,7	1,04	0,01	no	
	5	g/100g	40	52	27.10.17	0,7	0,77	0,63	0,01	no	
	6	g/100g	8	84							
	7	g/100g	21	71		0,73	0,8	0,67		no	93,1
	8	g/100g	14	78							
	9	g/100g	15	77	01.11.17	0,8	0,74	0,85	0,07	no	
	10	g/100g	26	66	02.11.17	-	-	-			
	11	g/100g	28	64	01.10.17	0,7676	0,7817	0,7535	0,02	no	
	12	g/100g	19	73	23.09.17	0,685	0,7	0,67	<0,01	yes	
	13	g/100g	3	89	10.10.17	0,7	0,65	0,75	<0,1	no	
	14	g/100g	37	55		0,74	0,73	0,75			
	15	g/100g	44	48		0,64	0,58	0,7	0,00001		100
	16	g/100g	11	81	18.10.17	0,81	0,68	0,94	0,05		
	17	g/100g	43	49	4.10.	0,77	0,81	0,72			

Parameter	Teilnehmer	Einheit	Proben-Nr. A	Proben-Nr. B	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis A	Ergebnis A	Bestimmungsgrenze	Inkl. WF	Wiederfindungsrate [%]
Analyte	Participant	Unit	Sample No. A	Sample No. B	Date of analysis	Result (Mean)	Result A	Result A	LOQ	Incl. RR	Recovery rate [%]
L-Histidin(e)	1	g/100g	6	86	27.10.17					No	
	2	g/100g	42	50	04.10.17	1,765	1,69	1,84			
	3	g/100g	22	70	03.11.17	1,86902	1,88642	1,85163	0,3	no	-
	4	g/100g	25	67	27.10.17	1,4	1,2	1,6	0,01	no	
	5	g/100g	40	52	27.10.17	1,84	1,89	1,79	0,01	no	
	6	g/100g	8	84							
	7	g/100g	21	71		1,33	1,2	1,46		no	95,1
	8	g/100g	14	78							
	9	g/100g	15	77	01.11.17	1,8	1,9	1,7	0,07	no	
	10	g/100g	26	66	02.11.17	0,2385	0,244	0,233			
	11	g/100g	28	64	01.10.17	1,34	1,28	1,4	0,04	no	
	12	g/100g	19	73	23.09.17	1,92	2,07	1,77	<0,01	yes	
	13	g/100g	3	89	10.10.17	1,88	1,81	1,94	<0,1	no	
	14	g/100g	37	55		1,9	1,7	2			
	15	g/100g	44	48		2,96	2,86	3,06	0,00001		100
	16	g/100g	11	81	18.10.17	1,8	1,6	2	0,05		
	17	g/100g	43	49	4.10.	0,81	1,86	1,76			

Parameter	Teilnehmer	Einheit	Proben-Nr. A	Proben-Nr. B	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis A	Ergebnis A	Bestimmungsgrenze	Inkl. WF	Wiederfindungsrate [%]
Analyte	Participant	Unit	Sample No. A	Sample No. B	Date of analysis	Result (Mean)	Result A	Result A	LOQ	Incl. RR	Recovery rate [%]
L-Isoleucin(e)	1	g/100g	6	86	27.10.17	4,225	4,25	4,2		No	
	2	g/100g	42	50	04.10.17	3,45	3,48	3,42			
	3	g/100g	22	70	03.11.17	3,94139	3,99108	3,89171	0,3	no	-
	4	g/100g	25	67	27.10.17	4,29	4,12	4,47	0,01	no	
	5	g/100g	40	52	27.10.17	3,83	3,84	3,82	0,01	no	
	6	g/100g	8	84		3,99	3,75	4,22	0,03		
	7	g/100g	21	71		3,6	3,67	3,54		no	90,4
	8	g/100g	14	78							
	9	g/100g	15	77	01.11.17	3,5	3,4	3,6	0,03	no	
	10	g/100g	26	66	02.11.17	0,1645	0,155	0,174			
	11	g/100g	28	64	01.10.17	3,57755	3,6534	3,5017	0,04	no	
	12	g/100g	19	73	23.09.17	3,57	3,61	3,52	<0,01	yes	
	13	g/100g	3	89	10.10.17	3,28	3,21	3,35	<0,1	no	
	14	g/100g	37	55		4,1	4,1	4,1			
	15	g/100g	44	48		2,97	2,97	2,97	0		100
	16	g/100g	11	81	18.10.17	3,9	3,9	3,9	0,05		
	17	g/100g	43	49	4.10.	3,96	3,83	4,08			

Parameter	Teilnehmer	Einheit	Proben-Nr. A	Proben-Nr. B	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis A	Ergebnis A	Bestimmungsgrenze	Inkl. WF	Wiederfindungsrate [%]
Analyte	Participant	Unit	Sample No. A	Sample No. B	Date of analysis	Result (Mean)	Result A	Result A	LOQ	Incl. RR	Recovery rate [%]
L-Leucin(e)	1	g/100g	6	86	27.10.17	7,55	7,57	7,53		No	
	2	g/100g	42	50	04.10.17	5,99	6,01	5,97			
	3	g/100g	22	70	03.11.17	6,83159	6,93853	6,72465	0,3	no	-
	4	g/100g	25	67	27.10.17	7,2	7,09	7,31	0,01	no	
	5	g/100g	40	52	27.10.17	6,58	6,47	6,69	0,01	no	
	6	g/100g	8	84		6,95	6,95	6,95	0,03		
	7	g/100g	21	71		6,73	6,67	6,79		no	83,7
	8	g/100g	14	78							
	9	g/100g	15	77	01.11.17	6,2	6,1	6,3	0,03	no	
	10	g/100g	26	66	02.11.17	0,292	0,279	0,304			
	11	g/100g	28	64	01.10.17	6,17938	6,27047	6,0883	0,05	no	
	12	g/100g	19	73	23.09.17	6,26	6,32	6,2	<0,01	yes	
	13	g/100g	3	89	10.10.17	6,03	6,01	6,04	<0,1	no	
	14	g/100g	37	55		6,8	6,9	6,6			
	15	g/100g	44	48		5,42	5,81	5,02	0,00001		100
	16	g/100g	11	81	18.10.17	6,9	7,1	6,6	0,05		
	17	g/100g	43	49	4.10.	6,57	6,45	6,69			

Parameter	Teilnehmer	Einheit	Proben-Nr. A	Proben-Nr. B	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis A	Ergebnis A	Bestimmungsgrenze	Inkl. WF	Wiederfindungsrate [%]
Analyte	Participant	Unit	Sample No. A	Sample No. B	Date of analysis	Result (Mean)	Result A	Result A	LOQ	Incl. RR	Recovery rate [%]
L-Lysin(e)	1	g/100g	6	86	27.10.17	5,095	5,39	4,8		No	
	2	g/100g	42	50	04.10.17	3,995	3,89	4,1			
	3	g/100g	22	70	03.11.17	4,55332	4,73719	4,36944	0,3	no	-
	4	g/100g	25	67	27.10.17	4,31	4,1	4,53	0,01	no	
	5	g/100g	40	52	27.10.17	4,24	4,3	4,17	0,01	no	
	6	g/100g	8	84		3,75	3,66	3,84	0,03		
	7	g/100g	21	71		3,58	3,82	3,35		no	92,7
	8	g/100g	14	78							
	9	g/100g	15	77	01.11.17	4	3,8	4,2	0,05	no	
	10	g/100g	26	66	02.11.17	1,782	1,935	1,629			
	11	g/100g	28	64	01.10.17	3,56617	3,54530	3,58703	0,02	no	
	12	g/100g	19	73	23.09.17	3,85	4,05	3,65	<0,01	yes	
	13	g/100g	3	89	10.10.17	3,5	3,45	3,55	<0,1	no	
	14	g/100g	37	55		4,2	4,1	4,2			
	15	g/100g	44	48		4,26	4,36	4,16	0,00001		100
	16	g/100g	11	81	18.10.17	4,3	4,2	4,3	0,05		
	17	g/100g	43	49	4.10.	4,03	4,05	4			

Parameter	Teilnehmer	Einheit	Proben-Nr. A	Proben-Nr. B	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis A	Ergebnis A	Bestimmungsgrenze	Inkl. WF	Wiederfindungsrate [%]
Analyte	Participant	Unit	Sample No. A	Sample No. B	Date of analysis	Result (Mean)	Result A	Result A	LOQ	Incl. RR	Recovery rate [%]
L-Methionin(e)	1	g/100g	6	86	27.10.17	1,195	1,22	1,17		No	
	2	g/100g	42	50	04.10.17	1,205	1,21	1,2			
	3	g/100g	22	70	-	NT	NT	NT	-	-	-
	4	g/100g	25	67	27.10.17	1,45	1,43	1,47	0,01	no	
	5	g/100g	40	52	27.10.17	1,33	1,32	1,34	0,01	no	
	6	g/100g	8	84		1,14	1,18	1,09	0,03		
	7	g/100g	21	71		1,12	1,12	1,11		no	94,9
	8	g/100g	14	78							
	9	g/100g	15	77	01.11.17	1,2	1,1	1,2	0,05	no	
	10	g/100g	26	66	02.11.17	0,0575	0,0593	0,0557			
	11	g/100g	28	64	01.10.17	1,27752	1,26383	1,2912	0,02	no	
	12	g/100g	19	73	23.09.17	1,13	1,1	1,16	<0,01	yes	
	13	g/100g	3	89	10.10.17	0,96	0,97	0,95	<0,1	no	
	14	g/100g	37	55		1,2	1,2	1,2			
	15	g/100g	44	48		1,05	1,03	1,08	0,00001		100
	16	g/100g	11	81	18.10.17	1,1	1	1,1	0,05		
	17	g/100g	43	49	4.10.	1,14	1,07	1,2			

Parameter	Teilnehmer	Einheit	Proben-Nr. A	Proben-Nr. B	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis A	Ergebnis A	Bestimmungsgrenze	Inkl. WF	Wiederfindungsrate [%]
Analyte	Participant	Unit	Sample No. A	Sample No. B	Date of analysis	Result (Mean)	Result A	Result A	LOQ	Incl. RR	Recovery rate [%]
L-Phenylalanin(e)	1	g/100g	6	86	27.10.17	1,945	1,97	1,92		No	
	2	g/100g	42	50	04.10.17	1,74	1,74	1,74			
	3	g/100g	22	70	03.11.17	1,91900	1,94783	1,89017	0,3	no	-
	4	g/100g	25	67	27.10.17	1,87	1,89	1,84	0,01	no	
	5	g/100g	40	52	27.10.17	1,9	1,91	1,88	0,01	no	
	6	g/100g	8	84		2,52	2,55	2,49	0,03		
	7	g/100g	21	71		1,96	1,99	1,93		no	93,7
	8	g/100g	14	78	02.11.17	1,67	1,68	1,66	0	no	80
	9	g/100g	15	77	01.11.17	1,8	1,8	1,9	0,03	no	
	10	g/100g	26	66	02.11.17	0,1015	0,1180	0,0850			
	11	g/100g	28	64	01.10.17	1,81815	1,84117	1,79513	0,03	no	
	12	g/100g	19	73	23.09.17	1,835	1,82	1,85	<0,01	yes	
	13	g/100g	3	89	10.10.17	1,65	1,62	1,69	<0,1	no	
	14	g/100g	37	55		2	1,9	2			
	15	g/100g	44	48		2,43	2,57	2,28	0,00001		100
	16	g/100g	11	81	18.10.17	2	2,1	1,9	0,05		
	17	g/100g	43	49	4.10.	1,89	1,86	1,92			

Parameter	Teilnehmer	Einheit	Proben-Nr. A	Proben-Nr. B	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis A	Ergebnis A	Bestimmungsgrenze	Inkl. WF	Wiederfindungsrate [%]
Analyte	Participant	Unit	Sample No. A	Sample No. B	Date of analysis	Result (Mean)	Result A	Result A	LOQ	Incl. RR	Recovery rate [%]
L-Prolin(e)	1	g/100g	6	86	27.10.17	2,575	2,56	2,59		No	
	2	g/100g	42	50	04.10.17	1,97	2,01	1,93			
	3	g/100g	22	70	03.11.17	2,08343	2,13239	2,03447	0,3	no	-
	4	g/100g	25	67	27.10.17	2,39	2,33	2,46	0,01	no	
	5	g/100g	40	52	27.10.17	2,23	2,21	2,24	0,01	no	
	6	g/100g	8	84		1,96	1,97	1,95	0,03		
	7	g/100g	21	71		2,22	2,17	2,28		no	93
	8	g/100g	14	78							
	9	g/100g	15	77	01.11.17	2	2	2	0,03	no	
	10	g/100g	26	66	02.11.17	-	-	-			
	11	g/100g	28	64	01.10.17	2,04653	2,07937	2,0137	0,06	no	
	12	g/100g	19	73	23.09.17	3,185	3,15	3,22	<0,01	yes	
	13	g/100g	3	89	10.10.17	2,13	2,16	2,1	<0,1	no	
	14	g/100g	37	55		2,9	2,8	2,9			
	15	g/100g	44	48		2,04	2,2	1,88	0,00001		100
	16	g/100g	11	81	18.10.17	2,3	2,3	2,2	0,05		
	17	g/100g	43	49	4.10.	2,23	2,16	2,29			

Parameter	Teilnehmer	Einheit	Proben-Nr. A	Proben-Nr. B	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis A	Ergebnis A	Bestimmungsgrenze	Inkl. WF	Wiederfindungsrate [%]
Analyte	Participant	Unit	Sample No. A	Sample No. B	Date of analysis	Result (Mean)	Result A	Result A	LOQ	Incl. RR	Recovery rate [%]
L-Serin(e)	1	g/100g	6	86	27.10.17	2,07	2,29	1,85		No	
	2	g/100g	42	50	04.10.17	1,59	1,38	1,8			
	3	g/100g	22	70	03.11.17	1,95413	1,92439	1,98387	0,3	no	-
	4	g/100g	25	67	27.10.17	1,86	1,53	2,2	0,01	no	
	5	g/100g	40	52	27.10.17	1,6	1,64	1,56	0,01	no	
	6	g/100g	8	84		2,07	2,09	2,05	0,03		
	7	g/100g	21	71		2,02	2,18	1,87		no	90,4
	8	g/100g	14	78							
	9	g/100g	15	77	01.11.17	1,9	1,9	1,8	0,07	no	
	10	g/100g	26	66	02.11.17	0,3	0,33	0,27			
	11	g/100g	28	64	01.10.17	1,78068	1,8945	1,66687	0,03	no	
	12	g/100g	19	73	23.09.17	1,82	1,91	1,72	<0,01	yes	
	13	g/100g	3	89	10.10.17	1,72	1,61	1,83	<0,1	no	
	14	g/100g	37	55		2,1	2,1	2,1			
	15	g/100g	44	48		1,35	1,26	1,44	0,00001		100
	16	g/100g	11	81	18.10.17	1,3	1,3	1,2	0,05		
	17	g/100g	43	49	4.10.	1,71	1,9	1,52			

Parameter	Teilnehmer	Einheit	Proben-Nr. A	Proben-Nr. B	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis A	Ergebnis A	Bestimmungsgrenze	Inkl. WF	Wiederfindungsrate [%]
Analyte	Participant	Unit	Sample No. A	Sample No. B	Date of analysis	Result (Mean)	Result A	Result A	LOQ	Incl. RR	Recovery rate [%]
L-Threonin(e)	1	g/100g	6	86	27.10.17	3,145	3,21	3,08		No	
	2	g/100g	42	50	04.10.17	2,455	2,48	2,43			
	3	g/100g	22	70	03.11.17	2,77351	2,78262	2,76441	0,3	no	-
	4	g/100g	25	67	27.10.17	3,02	2,92	3,12	0,01	no	
	5	g/100g	40	52	27.10.17	2,68	2,68	2,68	0,01	no	
	6	g/100g	8	84							
	7	g/100g	21	71		2,89	2,88	2,9		no	91,9
	8	g/100g	14	78							
	9	g/100g	15	77	01.11.17	2,6	2,6	2,6	0,03	no	
	10	g/100g	26	66	02.11.17	0,05865	0,06020	0,05710			
	11	g/100g	28	64	01.10.17	2,59197	2,6272	2,5567	0,03	no	
	12	g/100g	19	73	23.09.17	2,665	2,68	2,65	<0,01	yes	
	13	g/100g	3	89	10.10.17	2,53	2,47	2,59	<0,1	no	
	14	g/100g	37	55		3,2	3,1	3,3			
	15	g/100g	44	48		2,79	2,9	2,68	0,00001		100
	16	g/100g	11	81	18.10.17	2,7	2,7	2,6	0,05		
	17	g/100g	43	49	4.10.	2,78	2,79	2,77			

Parameter	Teilnehmer	Einheit	Proben-Nr. A	Proben-Nr. B	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis A	Ergebnis A	Bestimmungsgrenze	Inkl. WF	Wiederfindungsrate [%]
Analyte	Participant	Unit	Sample No. A	Sample No. B	Date of analysis	Result (Mean)	Result A	Result A	LOQ	Incl. RR	Recovery rate [%]
L-Tryptophan	1	g/100g	6	86	27.10.17	2,79	2,73	2,84		No	
	2	g/100g	42	50							
	3	g/100g	22	70	03.11.17	2,91873	3,00034	2,83712	0,3	no	-
	4	g/100g	25	67	27.10.17	2,58	2,61	2,55	0,01	no	
	5	g/100g	40	52	27.10.17	2,67	2,65	2,69	0,01	no	
	6	g/100g	8	84		2,65	2,62	2,68	0,03		
	7	g/100g	21	71		2,82	2,79	2,85		no	94
	8	g/100g	14	78							
	9	g/100g	15	77							
	10	g/100g	26	66	02.11.17	-	-	-			
	11	g/100g	28	64	01.10.17	2,39138	2,42680	2,35597	0,09	no	
	12	g/100g	19	73	23.09.17	2,815	2,78	2,85	<0,01	yes	101,4
	13	g/100g	3	89	10.10.17	2,52	2,52	2,53	<0,1	no	
	14	g/100g	37	55		3,9	3,5	4,3			
	15	g/100g	44	48		2,97	3	2,93	0,00001		100
	16	g/100g	11	81	18.10.17	2,8	3	2,6	0,05		
	17	g/100g	43	49	6.10.	2,69	2,71	2,67		no	no

Parameter	Teilnehmer	Einheit	Proben-Nr. A	Proben-Nr. B	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis A	Ergebnis A	Bestimmungsgrenze	Inkl. WF	Wiederfindungsrate [%]
Analyte	Participant	Unit	Sample No. A	Sample No. B	Date of analysis	Result (Mean)	Result A	Result A	LOQ	Incl. RR	Recovery rate [%]
L-Tyrosin(e)	1	g/100g	6	86	27.10.17	1,185	1,17	1,2		No	
	2	g/100g	42	50	04.10.17	1,135	1,12	1,15			
	3	g/100g	22	70	03.11.17	1,22837	1,25415	1,20259	0,3	no	-
	4	g/100g	25	67	27.10.17	1,12	1,07	1,18	0,01	no	
	5	g/100g	40	52	27.10.17	1,18	1,18	1,18	0,01	no	
	6	g/100g	8	84		1,2	1,17	1,23	0,03		
	7	g/100g	21	71		1,22	1,25	1,19		no	95
	8	g/100g	14	78							
	9	g/100g	15	77	01.11.17	1,1	1,1	1,2	0,03	no	
	10	g/100g	26	66	02.11.17	0,05035	0,0509	0,0498			
	11	g/100g	28	64	01.10.17	1,15938	1,17930	1,13947	0,02	no	
	12	g/100g	19	73	23.09.17	1,215	1,23	1,2	<0,01	yes	
	13	g/100g	3	89	10.10.17	1,03	1,06	1	<0,1	no	
	14	g/100g	37	55		1,3	1,2	1,3			
	15	g/100g	44	48		0,647	0,657	0,636	0,00001		100
	16	g/100g	11	81	18.10.17	1,3	1,3	1,3	0,05		
	17	g/100g	43	49	4.10.	1,2	1,19	1,2			

Parameter	Teilnehmer	Einheit	Proben-Nr. A	Proben-Nr. B	Datum d. Analyse	Ergebnis (Mittel)	Ergebnis A	Ergebnis A	Bestimmungsgrenze	Inkl. WF	Wiederfindungsrate [%]
Analyte	Participant	Unit	Sample No. A	Sample No. B	Date of analysis	Result (Mean)	Result A	Result A	LOQ	Incl. RR	Recovery rate [%]
L-Valin(e)	1	g/100g	6	86	27.10.17	3,47	3,47	3,47		No	
	2	g/100g	42	50	04.10.17	2,715	2,74	2,69			
	3	g/100g	22	70	-	NT	NT	NT	-	-	-
	4	g/100g	25	67	27.10.17	3,66	3,35	3,97	0,01	no	
	5	g/100g	40	52	27.10.17	2,94	2,93	2,95	0,01	no	
	6	g/100g	8	84							
	7	g/100g	21	71		3,03	3	3,06		no	90,9
	8	g/100g	14	78							
	9	g/100g	15	77	01.11.17	2,8	2,8	2,9	0,03	no	
	10	g/100g	26	66	02.11.17	0,10815	0,1084	0,1079			
	11	g/100g	28	64	01.10.17	2,81217	2,86793	2,75640	0,02	no	
	12	g/100g	19	73	23.09.17	2,795	2,79	2,8	<0,01	yes	
	13	g/100g	3	89	10.10.17	2,91	2,9	2,93	<0,1	no	
	14	g/100g	37	55		3,5	3,5	3,5			
	15	g/100g	44	48		2,95	2,97	2,93	0,00001		100
	16	g/100g	11	81	18.10.17	3,2	3,3	3,1	0,05		
	17	g/100g	43	49	4.10.	2,89	2,83	2,94			

5.1.2 Analytical Methods

Parameter	Teilnehmer	Methodenangabe, wie in Prüfbericht / Norm / Literatur	Hinweise zu Probenvorbereitung und -aufarbeitung	Hinweise zur Messmethode	Kalibrierung und Referenzmaterial	Wiederfindung mit gleicher Matrix	Methode akkr. ISO/IEC 17025	Sonstige Hinweise	
Analyte	Participant	Method description, like in a analysis report / norm / literature	Notes to sample preparation	Notes to analytical method	Calibration and reference material	Recovery w ith same matrix	Method accr. ISO/IEC 17025	Further Remarks	
L-Alanin(e)	1	Waters AccqTag Ultra Derivization Method			External Standard Calibration	No	No		
	2						No		
	3	-	water extraction	derivatization, HPLC-DAD/ HPLC-FLD/ HPLC-MS-MS	-	no	yes	-	
	4	SOP M 3123	Extraction and dilution	LCMS/MS with ISTD	div. reference materials / control chart material	no	yes		
	5	Determination of free amino acids by amino acid analyser (in-house method AS 02).	ground by mortar	following Ph. Eur. 2.2.56, Method 1, post column derivatisation with ninhydrin	amino acid mix solution 79248 (Sigma-Aldrich), single substances		yes		
	6	HPLC-IPAD			external Standard, AAS 18 Sigma and single substances		yes		
	7		sample weight: 125 mg; acidic extraction with HCl 0.01 M, incl. Carrez precipitation, filtration and ACCQ Tag Derivatization Kit from Waters	UPLC with UV PDA Detection	1 Point calibration with Waters Std+IS (Norvalin), Tau, Trp, B-Ala and Orn.	yes	no		
	8								
	9	Waters - ACCQ.TAG Chemistry Package – Instruction Manual, WAT 052 874 TP, rev. 0 April, 1993				Thermo Scientific Amino Acids Standard H	no	yes	
	10							yes	By IC
	11	Determination of free amino acids by HPLC with post column derivatisation	aquaous extract after protein precipitation with 5-sulfosalicylic acid solution	Ion exchange chromatographie with post column derivatisation (ninhydrin)	Extemer Standard, Einpunkt-Kalibration, Teewurst			yes	
	12	Determination of free amino acids in foods by amino acid analyser with ninhydrin according to ASU §64 LFGB L 49.07-2	Extraction with 0,1 M HCl	post column derivatisation with ninhydrin	internal reference material; internal standard multi-point calibration	yes	yes		
	13	ASU §64 LFGB L49.07-2/ mod.	sample wheigt 0,5g	amino acid analyser	calibration with internal standard		yes		
	14	Determination of free amino acids by amino acid analyser (P 20247 02x)	-	-	interner Standard	-	yes	-	
	15	EZ:faat-Kit Phenomenex, LC-MS/MS	acidic extraction	LC-MS/MS	multi-point calibration, internal calibration, in-house LST	no	yes		
	16	HPLC/FLD after derivatisation	derivatisation with AQC		Ext. calibr. with internal standard	no	yes		
	17	free amino acids (according to regulation (EC) 152/2009)	0,1 mol/L HCl, at 40°C in ultra sonic bath	approx. 0,2 g sample wheigt	internal Std., 1-Point calibration, food supplement		yes		

Parameter	Teilnehmer	Methodenangabe, wie in Prüfbericht / Norm / Literatur	Hinweise zu Probenvorbereitung und -aufarbeitung	Hinweise zur Messmethode	Kalibrierung und Referenzmaterial	Wiederfindung mit gleicher Matrix	Methode akkr. ISO/IEC 17025	Sonstige Hinweise	
Analyte	Participant	Method description, like in a analysis report / norm / literature	Notes to sample preparation	Notes to analytical method	Calibration and reference material	Recovery w ith same matrix	Method accr. ISO/IEC 17025	Further Remarks	
L-Arginin(e)	1								
	2						No		
	3	-	water extraction	derivatization, HPLC-DAD/ HPLC-FLD/ HPLC-MS-MS	-	no	yes	-	
	4	SOP M 3123	dto.	dto.	dto.	no	yes		
	5						yes		
	6								
	7						yes		
	8							no	
	9		Waters - ACCQ.TAG Chemistry Package – Instruction Manual, WAT 052 874 TP, rev. 0 April, 1994			Thermo Scientific Amino Acids Standard H	no	yes	
	10							yes	By IC
	11								
	12								
	13		ASU §64 LFGB L49.07-2/ mod.	sample wheigt 0,5g	amino acid analyser	calibration with internal standard		yes	
	14								
	15								
	16								
	17								

Parameter	Teilnehmer	Methodenangabe, wie in Prüfbericht / Norm / Literatur	Hinweise zu Probenvorbereitung und -aufarbeitung	Hinweise zur Messmethode	Kalibrierung und Referenzmaterial	Wiederfindung mit gleicher Matrix	Methode akkr. ISO/IEC 17025	Sonstige Hinweise	
Analyte	Participant	Method description, like in a analysis report / norm / literature	Notes to sample preparation	Notes to analytical method	Calibration and reference material	Recovery with same matrix	Method accr. ISO/IEC 17025	Further Remarks	
L-Asparaginsäure / L-Aspartic Acid	1								
	2						No		
	3	-	water extraction	derivatization, HPLC-DAD/ HPLC-FLD/ HPLC-MS-MS	-	no	yes	-	
	4	SOP M 3123	dto.	dto.	dto.	no	yes		
	5						yes		
	6								
	7						yes	no	
	8								
	9		Waters - ACCQ.TAG Chemistry Package – Instruction Manual, WAT 052 874 TP, rev. 0 April, 1995			Thermo Scientific Amino Acids Standard H	no	yes	
	10							yes	By IC
	11								
	12								
	13		ASU §64 LFGB L49.07-2/ mod.	sample weight 0,5g	amino acid analyser	calibration with internal standard		yes	
	14								
	15								
	16								
	17								

Parameter	Teilnehmer	Methodenangabe, wie in Prüfbericht / Norm / Literatur	Hinweise zu Probenvorbereitung und -aufarbeitung	Hinweise zur Messmethode	Kalibrierung und Referenzmaterial	Wiederfindung mit gleicher Matrix	Methode akkr. ISO/IEC 17025	Sonstige Hinweise	
Analyte	Participant	Method description, like in a analysis report / norm / literature	Notes to sample preparation	Notes to analytical method	Calibration and reference material	Recovery w with same matrix	Method accr. ISO/IEC 17025	Further Remarks	
L-Cstein(e)	1								
	2								
	3	-	water extraction	derivatization, HPLC-DAD/ HPLC-FLD/ HPLC-MS-MS	-	-	yes	-	
	4	SOP M 3123	dto.	dto.	dto.	no	yes		
	5	Free L-Cysteine determined by amino acid analyser (in-house method AS 11).	ground by mortar, stabilised by sodium sulfite		single reference substance		yes		
	6								
	7						yes	no	
	8								
	9	Waters - ACCQ.TAG Chemistry Package – Instruction Manual, WAT 052 874 TP, rev. 0 April, 1996				Thermo Scientific Amino Acids Standard H	no	yes	
	10								
	11								
	12								
	13							yes	
	14								
	15								
	16								
	17								

Parameter	Teilnehmer	Methodenangabe, wie in Prüfbericht / Norm / Literatur	Hinweise zu Probenvorbereitung und -aufarbeitung	Hinweise zur Messmethode	Kalibrierung und Referenzmaterial	Wiederfindung mit gleicher Matrix	Methode akkr. ISO/IEC 17025	Sonstige Hinweise	
Analyte	Participant	Method description, like in a analysis report / norm / literature	Notes to sample preparation	Notes to analytical method	Calibration and reference material	Recovery with same matrix	Method accr. ISO/IEC 17025	Further Remarks	
L-Cystin(e)	1								
	2						No		
	3	-	water extraction	derivatization, HPLC-DAD/ HPLC-FLD/ HPLC-MS-MS	-	no	yes	-	
	4	SOP M 3123	dto.	dto.	dto.	no	yes		
	5	Determination of free amino acids by amino acid analyser (in-house method AS 02).	ground by mortar		amino acid mix solution 79248 (Sigma-Aldrich), single substances		yes		
	6								
	7					yes	no		
	8								
	9								
	10							yes	By IC
	11								
	12								
	13	ASU §64 LFGB L49.07-2/ mod.	sample weight 0,5g	amino acid analyser	calibration with internal standard		yes		
	14								
	15								
	16								Sum Cysteine/Cystine
	17								

Parameter	Teilnehmer	Methodenangabe, wie in Prüfbericht / Norm / Literatur	Hinweise zu Probenvorbereitung und -aufarbeitung	Hinweise zur Messmethode	Kalibrierung und Referenzmaterial	Wiederfindung mit gleicher Matrix	Methode akkr. ISO/IEC 17025	Sonstige Hinweise	
Analyte	Participant	Method description, like in a analysis report / norm / literature	Notes to sample preparation	Notes to analytical method	Calibration and reference material	Recovery w ith same matrix	Method accr. ISO/IEC 17025	Further Remarks	
L-Glutaminsäure / L-Glutamic Acid	1								
	2						No		
	3	-	water extraction	derivatization, HPLC-DAD/ HPLC-FLD/ HPLC-MS-MS	-	no	yes	-	
	4	SOP M 3123	dto.	dto.	dto.	no	yes		
	5						yes		
	6								
	7						yes	no	
	8								
	9	Waters - ACCQ.TAG Chemistry Package – Instruction Manual, WAT 052 874 TP, rev. 0 April, 1998				Thermo Scientific Amino Acids Standard H	no	yes	
	10							yes	By IC
	11								
	12								
	13	ASU §64 LFGB L49.07-2/ mod.	sample wheight 0,5g	amino acid analyser	calibration with internal standard			yes	
	14								
	15								
	16								
	17								

Parameter	Teilnehmer	Methodenangabe, wie in Prüfbericht / Norm / Literatur	Hinweise zu Probenvorbereitung und -aufarbeitung	Hinweise zur Messmethode	Kalibrierung und Referenzmaterial	Wiederfindung mit gleicher Matrix	Methode akkr. ISO/IEC 17025	Sonstige Hinweise	
Analyte	Participant	Method description, like in a analysis report / norm / literature	Notes to sample preparation	Notes to analytical method	Calibration and reference material	Recovery w ith same matrix	Method accr. ISO/IEC 17025	Further Remarks	
Glycin	1								
	2						No		
	3	-	water extraction	derivatization, HPLC-DAD/ HPLC-FLD/ HPLC-MS-MS	-	no	yes	-	
	4	SOP M 3123	dto.	dto.	dto.	no	yes		
	5						yes		
	6								
	7						yes	no	
	8								
	9		Waters - ACCQ.TAG Chemistry Package – Instruction Manual, WAT 052 874 TP, rev. 0 April, 1999			Thermo Scientific Amino Acids Standard H	no	yes	
	10								
	11								
	12								
	13		ASU §64 LFGB L49.07-2/ mod.	sample wheigt 0,5g	amino acid analyser	calibration with internal standard		yes	
	14								
	15								
	16								
	17								

Parameter	Teilnehmer	Methodenangabe, wie in Prüfbericht / Norm / Literatur	Hinweise zu Probenvorbereitung und -aufarbeitung	Hinweise zur Messmethode	Kalibrierung und Referenzmaterial	Wiederfindung mit gleicher Matrix	Methode akkr. ISO/IEC 17025	Sonstige Hinweise	
Analyte	Participant	Method description, like in a analysis report / norm / literature	Notes to sample preparation	Notes to analytical method	Calibration and reference material	Recovery w ith same matrix	Method accr. ISO/IEC 17025	Further Remarks	
L-Histidin(e)	1								
	2						No		
	3	-	water extraction	derivatization, HPLC-DAD/ HPLC-FLD/ HPLC-MS-MS	-	no	yes	-	
	4	SOP M 3123	dto.	dto.	dto.	no	yes		
	5						yes		
	6								
	7						yes		
	8							no	
	9		Waters - ACCQ.TAG Chemistry Package – Instruction Manual, WAT 052 874 TP, rev. 0 April, 2000			Thermo Scientific Amino Acids Standard H	no	yes	
	10							yes	By IC
	11								
	12								
	13		ASU §64 LFGB L49.07-2/ mod.	sample wheigt 0,5g	amino acid analyser	calibration with internal standard		yes	
	14								
	15								
	16								
	17								

Parameter	Teilnehmer	Methodenangabe, wie in Prüfbericht / Norm / Literatur	Hinweise zu Probenvorbereitung und -aufarbeitung	Hinweise zur Messmethode	Kalibrierung und Referenzmaterial	Wiederfindung mit gleicher Matrix	Methode akkr. ISO/IEC 17025	Sonstige Hinweise	
Analyte	Participant	Method description, like in a analysis report / norm / literature	Notes to sample preparation	Notes to analytical method	Calibration and reference material	Recovery w ith same matrix	Method accr. ISO/IEC 17025	Further Remarks	
L-Isoleucin(e)	1								
	2						No		
	3	-	water extraction	derivatization, HPLC-DAD/ HPLC-FLD/ HPLC-MS-MS	-	no	yes	-	
	4	SOP M 3123	dto.	dto.	dto.	no	yes		
	5						yes		
	6								
	7						yes	no	
	8								
	9	Waters - ACCQ.TAG Chemistry Package – Instruction Manual, WAT 052 874 TP, rev. 0 April, 2001				Thermo Scientific Amino Acids Standard H	no	yes	
	10							yes	By IC
	11								
	12								
	13	ASU §64 LFGB L49.07-2/ mod.	sample wheigt 0,5g	amino acid analyser	calibration with internal standard			yes	
	14								
	15								
	16								
	17								

Parameter	Teilnehmer	Methodenangabe, wie in Prüfbericht / Norm / Literatur	Hinweise zu Probenvorbereitung und -aufarbeitung	Hinweise zur Messmethode	Kalibrierung und Referenzmaterial	Wiederfindung mit gleicher Matrix	Methode akkr. ISO/IEC 17025	Sonstige Hinweise	
Analyte	Participant	Method description, like in a analysis report / norm / literature	Notes to sample preparation	Notes to analytical method	Calibration and reference material	Recovery w ith same matrix	Method accr. ISO/IEC 17025	Further Remarks	
L-Leucin(e)	1								
	2						No		
	3	-	water extraction	derivatization, HPLC-DAD/ HPLC-FLD/ HPLC-MS-MS	-	no	yes	-	
	4	SOP M 3123	dto.	dto.	dto.	no	yes		
	5						yes		
	6								
	7						yes		
	8								
	9		Waters - ACCQ.TAG Chemistry Package – Instruction Manual, WAT 052 874 TP, rev. 0 April, 2002			Thermo Scientific Amino Acids Standard H	no	yes	
	10							yes	By IC
	11								
	12								
	13		ASU §64 LFGB L49.07-2/ mod.	sample wheigt 0,5g	amino acid analyser	calibration with internal standard		yes	
	14								
	15								
	16								
	17								

Parameter	Teilnehmer	Methodenangabe, wie in Prüfbericht / Norm / Literatur	Hinweise zu Probenvorbereitung und -aufarbeitung	Hinweise zur Messmethode	Kalibrierung und Referenzmaterial	Wiederfindung mit gleicher Matrix	Methode akkr. ISO/IEC 17025	Sonstige Hinweise	
Analyte	Participant	Method description, like in a analysis report / norm / literature	Notes to sample preparation	Notes to analytical method	Calibration and reference material	Recovery w ith same matrix	Method accr. ISO/IEC 17025	Further Remarks	
L-Lysin(e)	1								
	2						No		
	3	-	water extraction	derivatization, HPLC-DAD/ HPLC-FLD/ HPLC-MS-MS	-	no	yes	-	
	4	SOP M 3123	dto.	dto.	dto.	no	yes		
	5						yes		
	6								
	7						yes	no	
	8								
	9	Waters - ACCQ.TAG Chemistry Package – Instruction Manual, WAT 052 874 TP, rev. 0 April, 2003				Thermo Scientific Amino Acids Standard H	no	yes	
	10							yes	By IC
	11								
	12								
	13	ASU §64 LFGB L49.07-2/ mod.	sample wheigt 0,5g	amino acid analyser	calibration with internal standard			yes	
	14								
	15								
	16								
	17								

Parameter	Teilnehmer	Methodenangabe, wie in Prüfbericht / Norm / Literatur	Hinweise zu Probenvorbereitung und -aufarbeitung	Hinweise zur Messmethode	Kalibrierung und Referenzmaterial	Wiederfindung mit gleicher Matrix	Methode akkr. ISO/IEC 17025	Sonstige Hinweise	
Analyte	Participant	Method description, like in a analysis report / norm / literature	Notes to sample preparation	Notes to analytical method	Calibration and reference material	Recovery with same matrix	Method ac cr. ISO/IEC 17025	Further Remarks	
L-Methionin(e)	1								
	2						No		
	3	-	water extraction	derivatization, HPLC-DAD/ HPLC-FLD/ HPLC-MS-MS	-	-	yes	-	
	4	SOP M 3123	dto.	dto.	dto.	no	yes		
	5						yes		
	6								
	7						yes	no	
	8								
	9		Waters - ACCQ.TAG Chemistry Package – Instruction Manual, WAT 052 874 TP, rev. 0 April, 2004			Thermo Scientific Amino Acids Standard H	no	yes	
	10							yes	By IC
	11								
	12								
	13		ASU §64 LFGB L49.07-2/ mod.	sample weight 0,5g	amino acid analyser	calibration with internal standard		yes	
	14								
	15								
	16								
	17								

Parameter	Teilnehmer	Methodenangabe, wie in Prüfbericht / Norm / Literatur	Hinweise zu Probenvorbereitung und -aufarbeitung	Hinweise zur Messmethode	Kalibrierung und Referenzmaterial	Wiederfindung mit gleicher Matrix	Methode akkr. ISO/IEC 17025	Sonstige Hinweise	
Analyte	Participant	Method description, like in a analysis report / norm / literature	Notes to sample preparation	Notes to analytical method	Calibration and reference material	Recovery with same matrix	Method accr. ISO/IEC 17025	Further Remarks	
L-Phenylalanin(e)	1								
	2						No		
	3	-	water extraction	derivatization, HPLC-DAD/ HPLC-FLD/ HPLC-MS-MS	-	no	yes	-	
	4	SOP M 3123	dto.	dto.	dto.	no	yes		
	5						yes		
	6								
	7						yes	no	
	8	HPLC-MS/MS	No ISO 13903	none	sigma	no	yes	Sample A in PT label is 14 and sample 78	
	9	Waters - ACCQ.TAG Chemistry Package – Instruction Manual, WAT 052 874 TP, rev. 0 April, 2005				Thermo Scientific Amino Acids Standard H	no	yes	
	10							yes	By IC
	11								
	12								
	13	ASU §64 LFGB L49.07-2/ mod.	sample wheight 0,5g	amino acid analyser	calibration with internal standard			yes	
	14								
	15								
	16								
	17								

Parameter	Teilnehmer	Methodenangabe, wie in Prüfbericht / Norm / Literatur	Hinweise zu Probenvorbereitung und -aufarbeitung	Hinweise zur Messmethode	Kalibrierung und Referenzmaterial	Wiederfindung mit gleicher Matrix	Methode akkr. ISO/IEC 17025	Sonstige Hinweise	
Analyte	Participant	Method description, like in a analysis report / norm / literature	Notes to sample preparation	Notes to analytical method	Calibration and reference material	Recovery with same matrix	Method accr. ISO/IEC 17025	Further Remarks	
L-Prolin(e)	1								
	2						No		
	3	-	water extraction	derivatization, HPLC-DAD/ HPLC-FLD/ HPLC-MS-MS	-	no	yes	-	
	4	SOP M 3123	dto.	dto.	dto.	no	yes		
	5						yes		
	6								
	7						yes	no	
	8								
	9		Waters - ACCQ.TAG Chemistry Package – Instruction Manual, WAT 052 874 TP, rev. 0 April, 2006			Thermo Scientific Amino Acids Standard H	no	yes	
	10								
	11								
	12								
	13		ASU §64 LFGB L49.07-2/ mod.	sample weight 0,5g	amino acid analyser	calibration with internal standard		yes	
	14								
	15								
	16								
	17								

Parameter	Teilnehmer	Methodenangabe, wie in Prüfbericht / Norm / Literatur	Hinweise zu Probenvorbereitung und -aufarbeitung	Hinweise zur Messmethode	Kalibrierung und Referenzmaterial	Wiederfindung mit gleicher Matrix	Methode akkr. ISO/IEC 17025	Sonstige Hinweise	
Analyte	Participant	Method description, like in a analysis report / norm / literature	Notes to sample preparation	Notes to analytical method	Calibration and reference material	Recovery w ith same matrix	Method accr. ISO/IEC 17025	Further Remarks	
L-Serin(e)	1								
	2						No		
	3	-	water extraction	derivatization, HPLC-DAD/ HPLC-FLD/ HPLC-MS-MS	-	no	yes	-	
	4	SOP M 3123	dto.	dto.	dto.	no	yes		
	5						yes		
	6								
	7						yes	no	
	8								
	9	Waters - ACCQ.TAG Chemistry Package – Instruction Manual, WAT 052 874 TP, rev. 0 April, 2007				Thermo Scientific Amino Acids Standard H	no	yes	
	10							yes	By IC
	11								
	12								
	13	ASU §64 LFGB L49.07-2/ mod.	sample wheigt 0,5g	amino acid analyser	calibration with internal standard			yes	
	14								
	15								
	16								
	17								

Parameter	Teilnehmer	Methodenangabe, wie in Prüfbericht / Norm / Literatur	Hinweise zu Probenvorbereitung und -aufarbeitung	Hinweise zur Messmethode	Kalibrierung und Referenzmaterial	Wiederfindung mit gleicher Matrix	Methode akkr. ISO/IEC 17025	Sonstige Hinweise	
Analyte	Participant	Method description, like in a analysis report / norm / literature	Notes to sample preparation	Notes to analytical method	Calibration and reference material	Recovery w ith same matrix	Method accr. ISO/IEC 17025	Further Remarks	
L-Threonin(e)	1								
	2						No		
	3	-	water extraction	derivatization, HPLC-DAD/ HPLC-FLD/ HPLC-MS-MS	-	no	yes	-	
	4	SOP M 3123	dto.	dto.	dto.	no	yes		
	5						yes		
	6								
	7						yes	no	
	8								
	9	Waters - ACCQ.TAG Chemistry Package – Instruction Manual, WAT 052 874 TP, rev. 0 April, 2008				Thermo Scientific Amino Acids Standard H	no	yes	
	10							yes	By IC
	11								
	12								
	13	ASU §64 LFGB L49.07-2/ mod.	sample wheigt 0,5g	amino acid analyser	calibration with internal standard			yes	
	14								
	15								
	16								
	17								

Parameter	Teilnehmer	Methodenangabe, wie in Prüfbericht / Norm / Literatur	Hinweise zu Probenvorbereitung und -aufarbeitung	Hinweise zur Messmethode	Kalibrierung und Referenzmaterial	Wiederfindung mit gleicher Matrix	Methode akkr. ISO/IEC 17025	Sonstige Hinweise	
Analyte	Participant	Method description, like in a analysis report / norm / literature	Notes to sample preparation	Notes to analytical method	Calibration and reference material	Recovery w ith same matrix	Method accr. ISO/IEC 17025	Further Remarks	
L-Tryptohan	1								
	2								
	3	-	water extraction	derivatization, HPLC-DAD/ HPLC-FLD/ HPLC-MS-MS	-	no	yes	-	
	4	SOP M 3123	dto.	dto.	dto.	no	yes		
	5						yes		
	6								
	7						yes	no	
	8								
	9								
	10								
	11								
	12						yes	yes	
	13	ASU §64 LFGB L49.07-2/ mod.	sample wheigt 0,5g	amino acid analyser	calibration with internal standard			yes	
	14								
	15								
	16								
	17		HPLC (according to no. 4.11.2 VDLUFA Method Book III, 2. Amend. 1988)			3-Point calibration, feed		yes	

Parameter	Teilnehmer	Methodenangabe, wie in Prüfbericht / Norm / Literatur	Hinweise zu Probenvorbereitung und -aufarbeitung	Hinweise zur Messmethode	Kalibrierung und Referenzmaterial	Wiederfindung mit gleicher Matrix	Methode akkr. ISO/IEC 17025	Sonstige Hinweise	
Analyte	Participant	Method description, like in a analysis report / norm / literature	Notes to sample preparation	Notes to analytical method	Calibration and reference material	Recovery w ith same matrix	Method accr. ISO/IEC 17025	Further Remarks	
L-Tyrosin(e)	1								
	2						No		
	3	-	water extraction	derivatization, HPLC-DAD/ HPLC-FLD/ HPLC-MS-MS	-	no	yes	-	
	4	SOP M 3123	dto.	dto.	dto.	no	yes		
	5						yes		
	6								
	7						yes	no	
	8								
	9	Waters - ACCQ.TAG Chemistry Package – Instruction Manual, WAT 052 874 TP, rev. 0 April, 2010				Thermo Scientific Amino Acids Standard H	no	yes	
	10							yes	By IC
	11								
	12								
	13	ASU §64 LFGB L49.07-2/ mod.	sample wheight 0,5g	amino acid analyser	calibration with internal standard			yes	
	14								
	15								
	16								
	17								

Parameter	Teilnehmer	Methodenangabe, wie in Prüfbericht / Norm / Literatur	Hinweise zu Probenvorbereitung und -aufarbeitung	Hinweise zur Messmethode	Kalibrierung und Referenzmaterial	Wiederfindung mit gleicher Matrix	Methode akkr. ISO/IEC 17025	Sonstige Hinweise	
Analyte	Participant	Method description, like in a analysis report / norm / literature	Notes to sample preparation	Notes to analytical method	Calibration and reference material	Recovery w ith same matrix	Method accr. ISO/IEC 17025	Further Remarks	
L-Valin(e)	1								
	2						No		
	3	-	water extraction	derivatization, HPLC-DAD/ HPLC-FLD/ HPLC-MS-MS	-	-	yes	-	
	4	SOP M 3123	dto.	dto.	dto.	no	yes		
	5						yes		
	6								
	7						yes	no	
	8								
	9	Waters - ACCQ.TAG Chemistry Package – Instruction Manual, WAT 052 874 TP, rev. 0 April, 2011				Thermo Scientific Amino Acids Standard H	no	yes	
	10							yes	By IC
	11								
	12								
	13	ASU §64 LFGB L49.07-2/ mod.	sample wheigt 0,5g	amino acid analyser	calibration with internal standard			yes	
	14								
	15								
	16								
	17								

5.2 Homogeneity

5.2.1 Mixture homogeneity before bottling

Microtracer Homogeneity Test

DLA 47-2017

Weight whole sample	1,06	kg
Microtracer	FSS-rot lake	
Particle size	75 – 300	µm
Weight per particle	2,0	µg
Addition of tracer	42,0	mg/kg

Result of analysis

Sample	Weight [g]	Particle number	Particles [mg/kg]
1	5,06	131	51,8
2	4,99	117	46,9
3	5,02	125	49,8
4	5,06	121	47,8
5	5,06	118	46,6
6	5,04	116	46,0
7	4,96	127	51,2
8	4,99	134	53,7

Poisson distribution

Number of samples	8	
Degree of freedom	7	
Mean	123,6	Particles
Standard deviation	7,05	Particles
χ^2 (CHI-Quadrat)	2,81	
Probability	90	%
Recovery rate	117	%

Normal distribution

Number of samples	8	
Mean	49,2	mg/kg
Standard deviation	2,81	mg/kg
rel. Standard deviaton	5,70	%
Horwitz standard deviation	8,90	%
HorRat-value	0,64	
Recovery rate	117	%

5.2.2 Comparison 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 item can be characterized with the help of the trend line function:

L-Arginine			
Target standard deviation σ_{pt}	0,193		g/100g
Sample numbers	3 - 89		
Total numbers of samples	28		
Slope	0,00124		
Trend line range	1,397	-	1,432 g/100g
Deviation trend line	1,415	±	0,0174 g/100g
Percent of σ_{pt}	9,0	%	

without results with z-Scores > |3,0|

L-Phenylalanine			
Target standard deviation σ_{pt}	0,114		g/100g
Sample numbers	3 - 89		
Total numbers of samples	28		
Slope	-0,00109		
Trend line range	1,872	-	1,842 g/100g
Deviation trend line	1,857	±	0,0153 g/100g
Percent of σ_{pt}	13,4	%	

without results with z-Scores > |3,0|

L-Tyrosine			
Target standard deviation σ_{pt}	0,0862		g/100g
Sample numbers	3 - 89		
Total numbers of samples	28		
Slope	0,00054		
Trend line range	1,176	-	1,191 g/100g
Deviation trend line	1,184	±	0,00756 g/100g
Percent of σ_{pt}	8,8	%	

without results with z-Scores > |3,0|

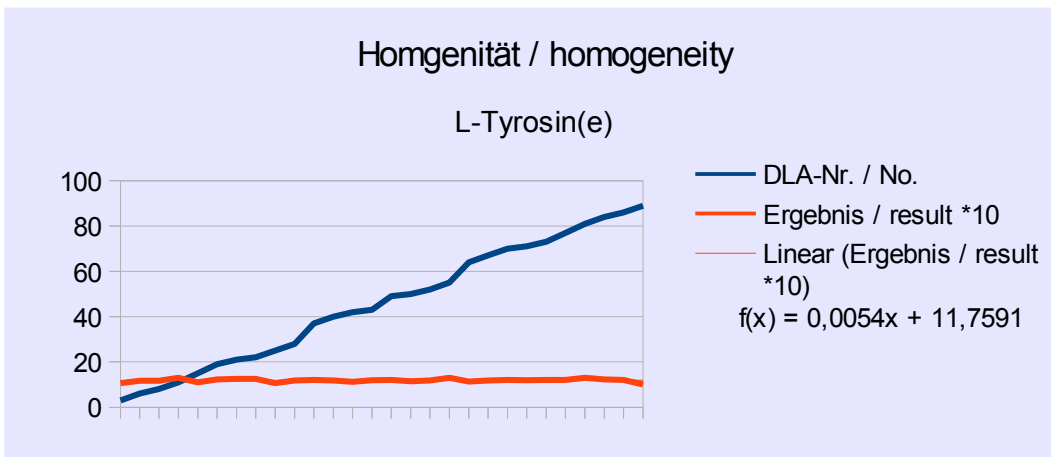
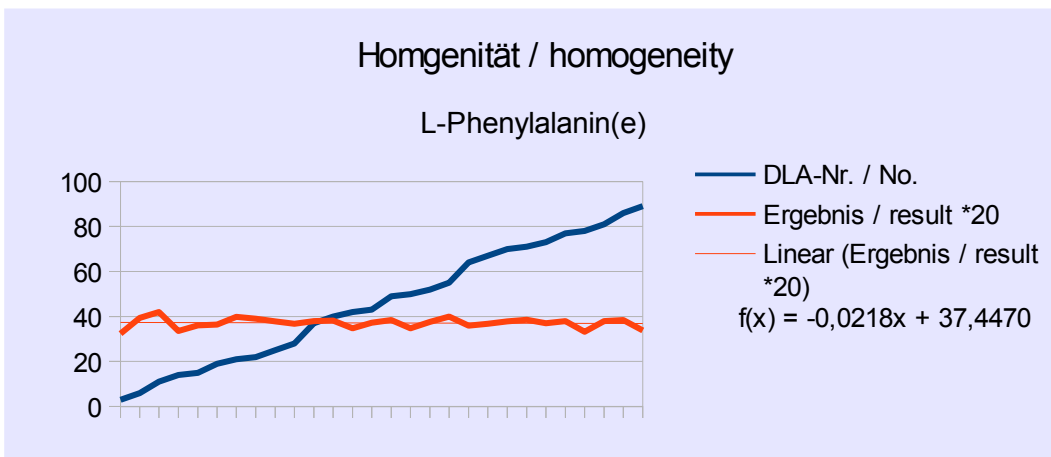
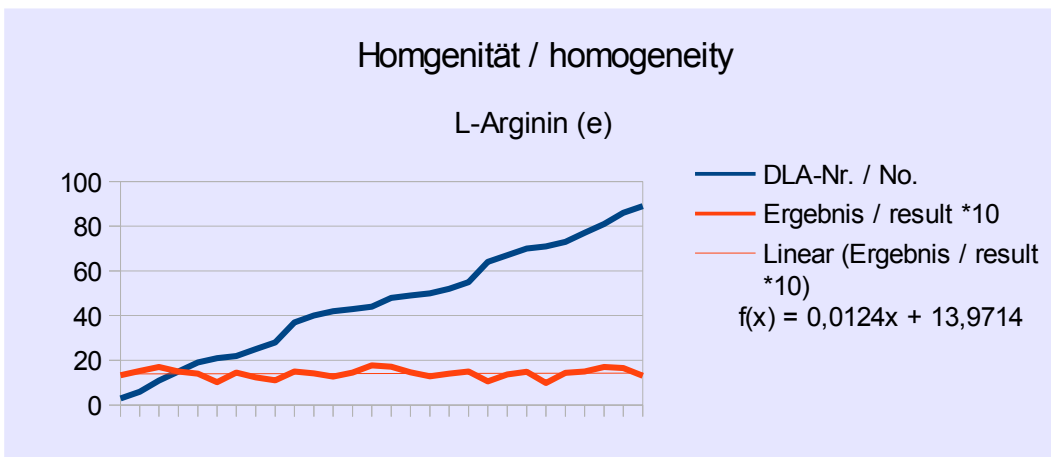


Abb./Fig. 37:

Trendfunktion Probennummern vs. Ergebnisse: L-Arginin, L-Phenylalanin und L-Tyrosin (x10, x20 und x10 dargestellt)
trend line function sample number vs. results: L-arginine, L-phenylalanine and L-tyrosine (x10, x20 and x10 shown)

5.3 Stability

5.3.1 Trend line function of participant results

By comparison of the participant results with the corresponding date of analysis the stability of the PT-material can be characterized for the range of analysis time of the present PT by the trend line functions:

L-Arginine			
Target standard deviation σ_{pt}	0,193		g/100g
Time of analysis	40		days
Number of results	11		
Slope	0,0204		
Trend line range	1,291	-	1,516 g/100g
Deviation trend line	1,403	±	0,1122 g/100g
Percent of σ_{pt}	58,1	%	

L-Phenylalanine			
Target standard deviation σ_{pt}	0,114		g/100g
Time of analysis	40		days
Number of results	12		
Slope	0,00230		
Trend line range	1,821	-	1,849 g/100g
Deviation trend line	1,835	±	0,0138 g/100g
Percent of σ_{pt}	12,1	%	

without results with z-Scores > |3,0|

L-Tyrosine			
Target standard deviation σ_{pt}	0,0862		g/100g
Time of analysis	40		days
Number of results	11		
Slope	-0,00037		
Trend line range	1,171	-	1,167 g/100g
Deviation trend line	1,169	±	0,0020 g/100g
Percent of σ_{pt}	2,4	%	

without results with z-Scores > |3,0|

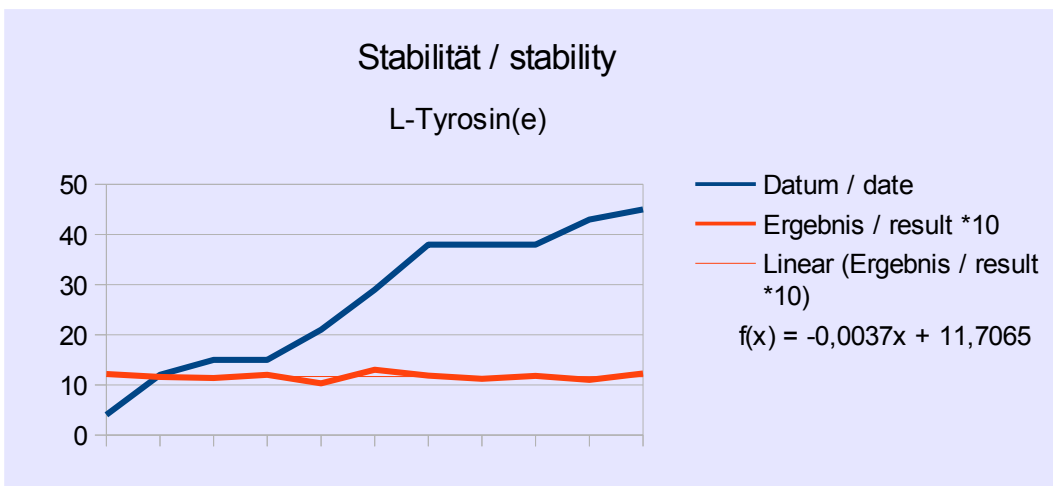
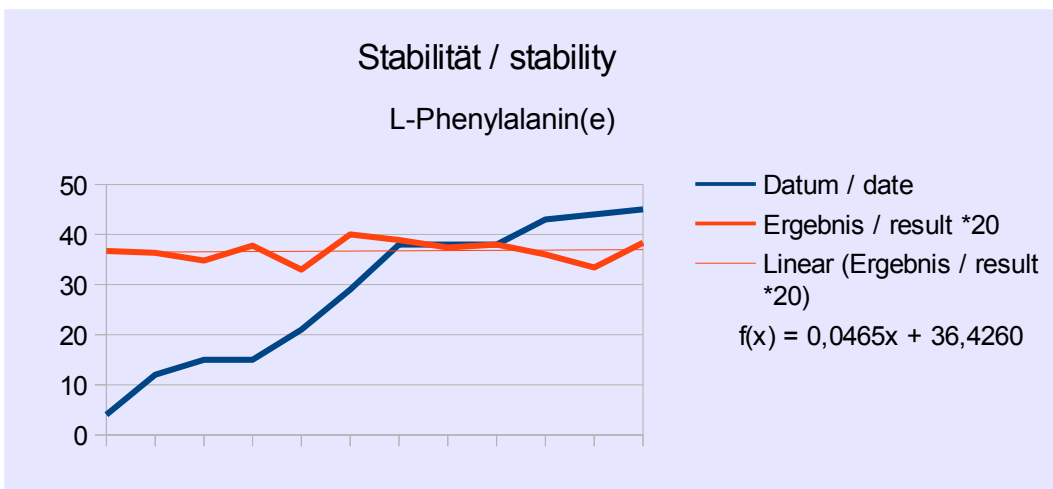
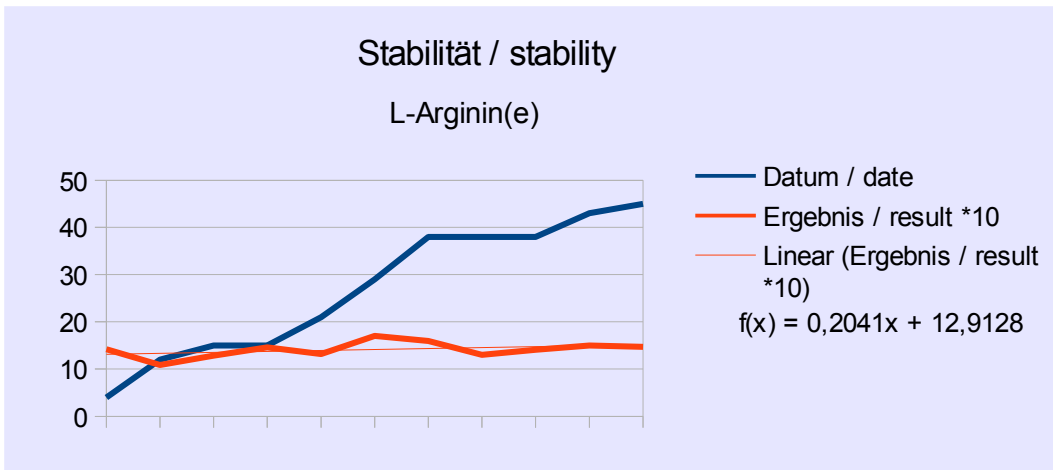


Abb./Fig. 38:

Trendfunktion Analysendatum vs. Ergebnisse: L-Arginin, L-Phenylalanin und L-Tyrosin (x10, x20 und x10 dargestellt)

trend line function date of analysis vs. results: L-arginine, L-phenylalanine and L-tyrosine (x10, x20 and x10 shown)

5.4 Kernel Density Plots of Results

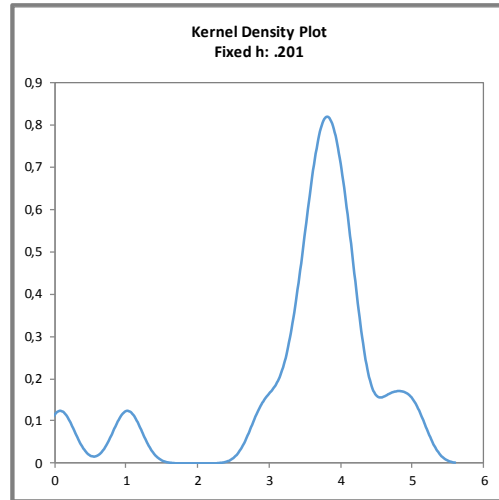
Abbildungen:

Kerndichte-Schätzungen der Teilnehmerergebnisse (mit $h = 0,75 \times \sigma_{pt}$ von X_{pt})

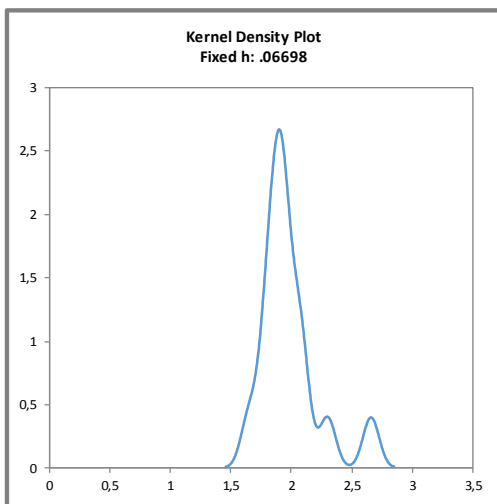
Figures:

Kernel density plots of participants' results (with $h = 0,75 \times \sigma_{pt}$ of X_{pt})

L-Asparaginsäure/L-Aspartic acid



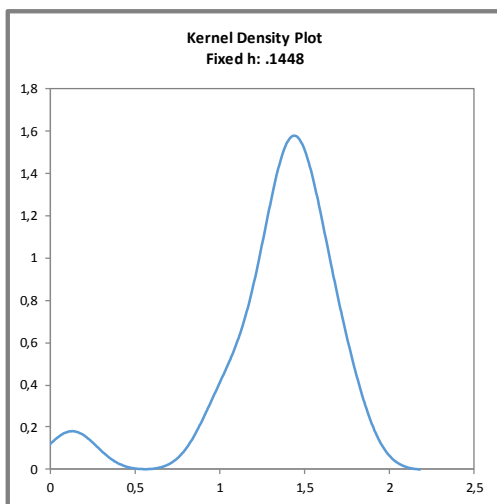
L-Alanin/L-Alanine



L-Cystein/L-Cysteine

<8 Ergebnisse
<8 Results

L-Arginin/L-Arginine



L-Cystin/L-Cystine

<8 Ergebnisse
<8 Results

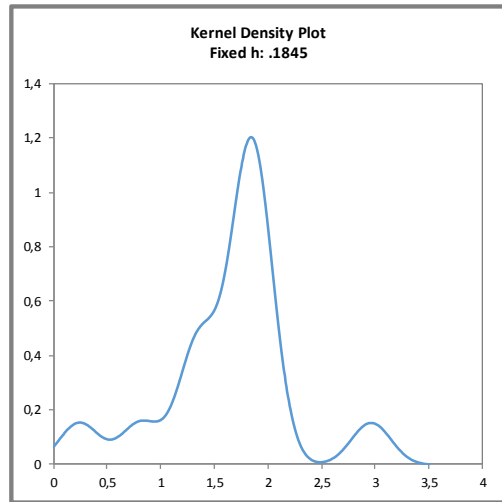
Abbildungen:

Kerndichte-Schätzungen der Teilnehmerergebnisse (mit $h = 0,75 \times \sigma_{pt}$ von X_{pt})

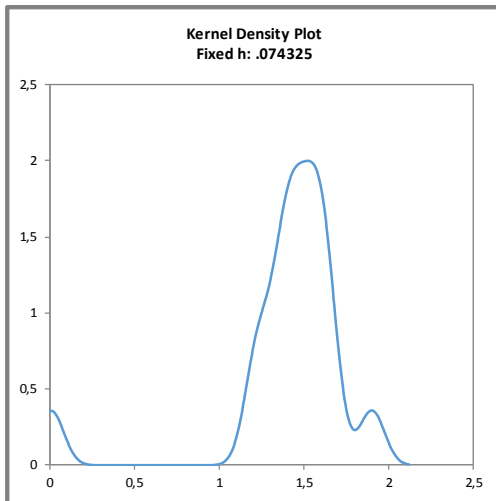
Figures:

Kernel density plots of participants' results (with $h = 0,75 \times \sigma_{pt}$ of X_{pt})

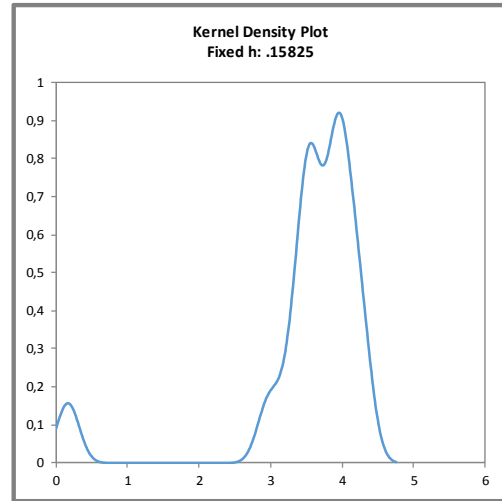
L-Histidin/L-Histidine



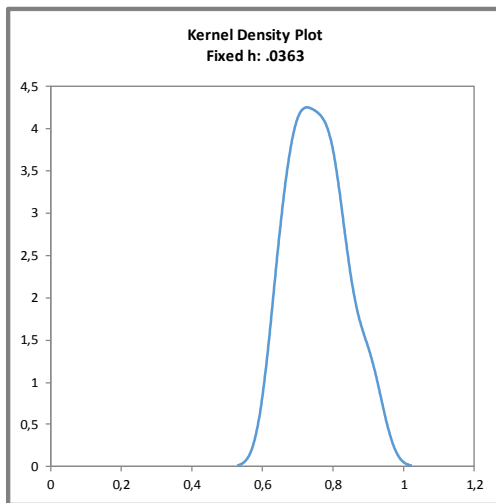
L-Glutaminsäure/L-Glutamic acid



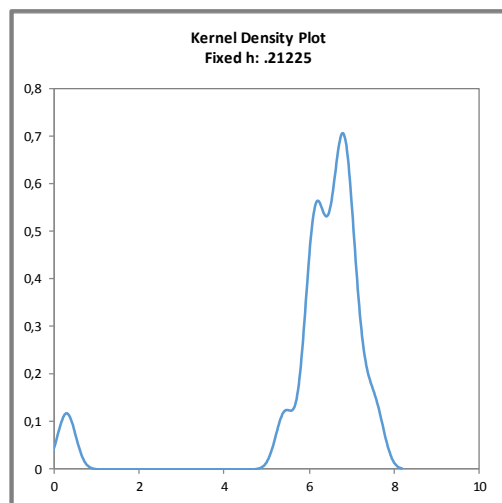
L-Isoleucin/L-Isoleucine



Glycin/Glycine



L-Leucin/L-Leucine



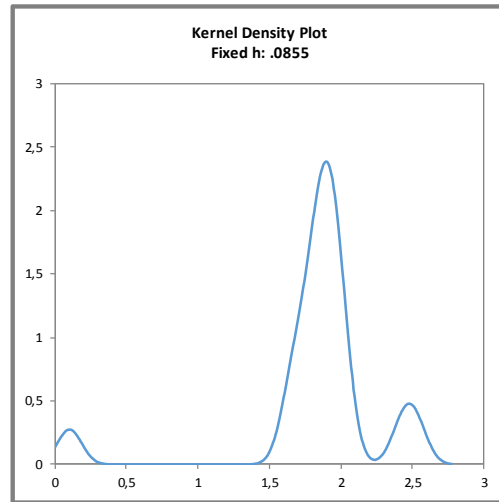
Abbildungen:

Kerndichte-Schätzungen der Teilnehmerergebnisse (mit $h = 0,75 \times \sigma_{pt}$ von X_{pt})

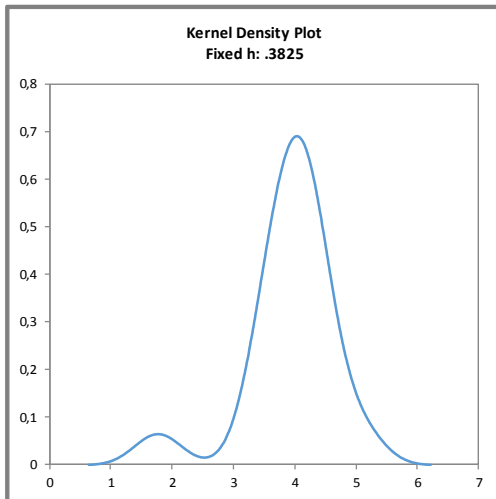
Figures:

Kernel density plots of participants' results (with $h = 0,75 \times \sigma_{pt}$ of X_{pt})

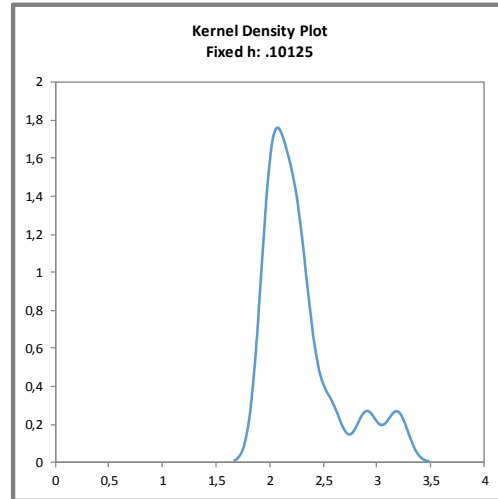
L-Phenylalanin/L-Phenylalanine



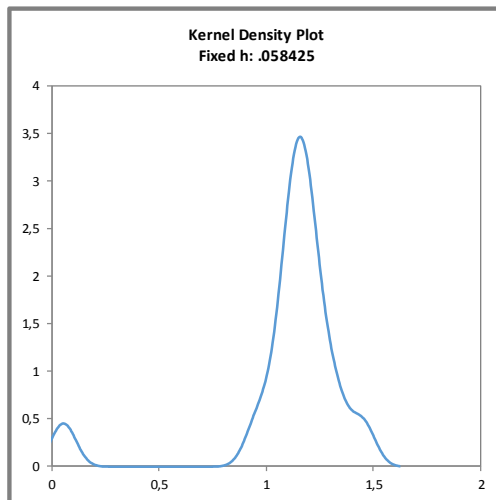
L-Lysin/L-Lysine



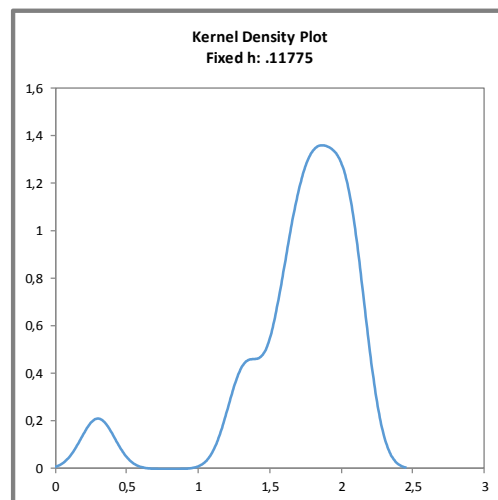
L-Prolin/L-Proline



L-Methionin/L-Methionine



L-Serin/L-Serine



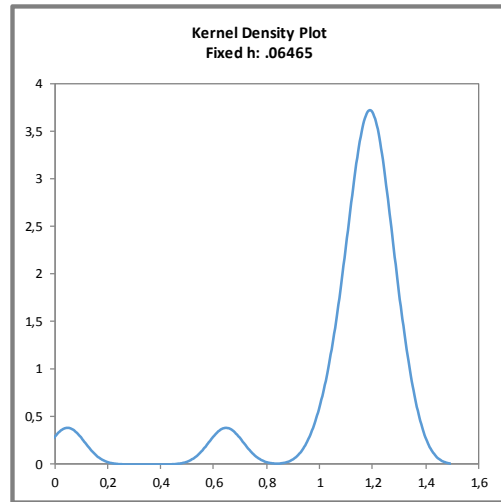
Abbildungen:

Kerndichte-Schätzungen der Teilnehmerergebnisse (mit $h = 0,75 \times \sigma_{pt}$ von X_{pt})

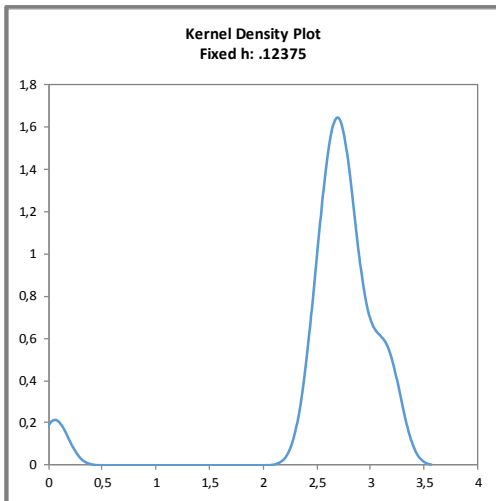
Figures:

Kernel density plots of participants' results (with $h = 0,75 \times \sigma_{pt}$ of X_{pt})

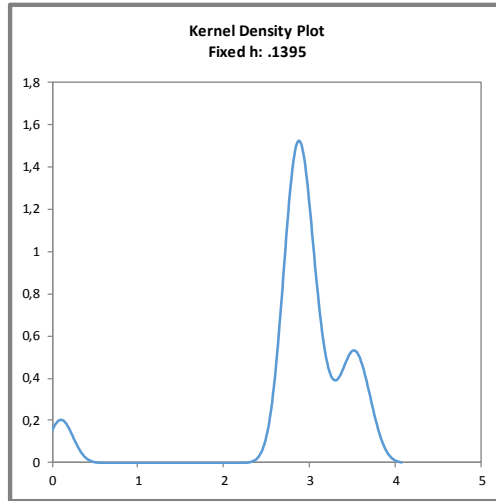
L-Tyrosin/L-Tyrosine



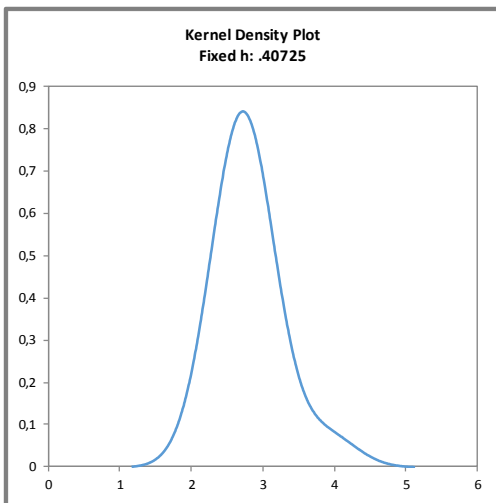
L-Threonin/L-Threonine



L-Valin/L-Valine



L-Tryptophan/L-Tryptophan



5.5 Information on the Proficiency Test (PT)

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

<i>PT number</i>	DLA 47-2017
<i>PT name</i>	Free Amino Acids in Food
<i>Sample matrix*</i>	Samples A + B: Food supplement capsule powder (without capsule shell) / ingredients: free amino acids (as food additives), maltodextrin and further food additives (bulking agents, separating agents, food dye titanium dioxide); Protein equivalent content is > 20%
<i>Number of samples and sample amount</i>	2 identical samples A + B: 10 g each.
<i>Storage</i>	Samples A + B: cooled 2 - 10°C (dry and dark)
<i>Intentional use</i>	Laboratory use only (quality control samples)
<i>Parameter</i>	quantitative: diverse free amino acids (see result submission file)
<i>Methods of analysis</i>	Analytical methods are optional
<i>Notes to analysis</i>	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.
<i>Result sheet</i>	The results for sample A and B as well as the final results calculated as mean of the double determination (samples A and B) should be filled in the result submission file. The recovery rates, if carried out, has to be included in the calculation.
<i>Units</i>	g/100g
<i>Number of significant digits</i>	at least 2
<i>Further information</i>	For information please specify: <ul style="list-style-type: none"> - Date of analysis - DLA-sample-numbers (for sample A and B) - Limit of detection - Assignment incl. Recovery - Recovery with the same matrix - Method is accredited
<i>Result submission</i>	The result submission file should be sent by e-mail to: pt@dla-lvu.de
<i>Deadline</i>	the latest <u>03rd November 2017</u>
<i>Evaluation report</i>	The evaluation report is expected to be completed 6 weeks after deadline of result submission and sent as PDF file by e-mail.
<i>Coordinator and contact person of PT</i>	Dr. Matthias Besler

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

6. Index of participant laboratories in alphabetical order

Teilnehmer / Participant	Ort / Town	Land / Country
		GREAT BRITAIN
		HUNGARY
		Germany
		CZECH REPUBLIC
		Germany
		Germany
		USA
		Germany
		Germany
		SWITZERLAND
		SPAIN
		Germany
		Germany
		Germany
		Germany
		Germany
		INDIA

[Die Adressdaten der Teilnehmer wurden für die allgemeine Veröffentlichung des Auswertebereichs nicht angegeben.]

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

7. Index of references

1. DIN EN ISO/IEC 17025:2005; Allgemeine Anforderungen an die Kompetenz von Prüf- und Kalibrierlaboratorien / General requirements for the competence of testing and calibration laboratories
2. DIN EN ISO/IEC 17043:2010; Konformitätsbewertung - Allgemeine Anforderungen an Eignungsprüfungen / Conformity assessment - General requirements for proficiency testing
3. ISO 13528:2015 & DIN ISO 13528:2009; Statistische Verfahren für Eignungsprüfungen durch Ringversuche / Statistical methods for use in proficiency testing by inter-laboratory comparisons
4. ASU §64 LFGB: Planung und statistische Auswertung von Ringversuchen zur Methodenvalidierung / DIN ISO 5725 series part 1, 2 and 6 Accuracy (trueness and precision) of measurement methods and results
5. Verordnung / Regulation 882/2004/EU; Verordnung über über amtliche Kontrollen zur Überprüfung der Einhaltung des Lebensmittel- und Futtermittelrechts sowie der Bestimmungen über Tiergesundheit und Tierschutz / Regulation on official controls performed to ensure the verification of compliance with feed and food law, animal health and animal welfare rules
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14. GMP+ Feed Certification scheme, Module: Feed Safety Assurance, chapter 5.7 Checking procedure for the process accuracy of compound feed with micro tracers in GMP+ BA2 Control of residues, Version: 1st of January 2015 GMP+ International B.V.
15. MTSE SOP No. 010.01 (2014): Quantitative measurement of mixing uniformity and carry-over in powder mixtures with the rotary detector technique, MTSE Micro Tracers Services Europe GmbH
16. Horwitz Equation as Quality Benchmark in ISO/IEC 17025 Testing Laboratory, Rivera & Rodriguez, Bufete de ingenieros industriales, S.C. (Corrigendum 2014)
17. AOAC Official Methods of Analysis: Guidelines for Standard Method Performance Requirements, Appendix F, p. 2, AOAC Int (2016)
- 18.
19. ASU §64 LFGB L 49.07-1 Bestimmung der Aminosäuren in Aminosäurengemischen (1985) [Determination of amino acids in amino acid mixtures]
20. ASU §64 LFGB L 49.07-2 Bestimmung der Aminosäuren in diätetischen Lebensmitteln auf Basis von Proteinhydrolysaten (1986) [Determination of amino acids in dietetic foods on the basis of protein hydrolysates]
21. ASU §64 LFGB L 49.07-3 Bestimmung des Tryptophangehaltes in diätetischen Lebensmitteln auf Basis von Proteinhydrolysaten (1989) [Determination of tryptophan in dietetic foods on the basis of protein hydrolysates]
22. 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