Proficiency Tests

DLA

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Evaluation Report
proficiency test

DLA 59/2016

Total Amino Acid Composition

in Dietetic Food as Meal Replacement (Powder)

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1ˢᵗ Correction 18/01/2017:

The evaluation of *L-Glutamic Acid* was replaced, because the first version contained a false data set of participants results. Pages below were changed:
- page 10: target standard deviation (now from precision experiment, German ASU §64)
- page 14: characteristic data in table 4
- page 18: last paragraph (L-Glutamic Acid is within 94% to 112% of amino acid content indicated by the manufacturer)
- pages 30-31: statistical evaluation of L-Glutamic Acid
- page 77: figure of kernel density estimation of L-Glutamic Acid

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

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Unteraufträge *Subcontractors*	Die Prüfung der Gehalte, Homogenität und Stabilität von EP-Parametern wird von DLA im Unterauftrag vergeben. The analysis of the content, homogeneity and stability of PT-parameters are subcontracted by DLA.

Inhalt / Content

1. Introduction

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

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

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

2. Realisation

2.1 Test material

The test material is a common in commerce dietetic food as a meal replacement (drink powder) with a protein content of > 20% based on soy- and milkprotein from a EU supplier. The raw materials were sieved by means of a centrifugal mill (mesh 500 µm), mixed and homogenized.

Afterwards the samples were portioned to approximately 10 g into 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

Dietetic Drink Powder as Meal Replacement
Ingredients: Soy protein isolate (41%), honey (20%), skimmed milk powder (12%), yoghurt powder (6%), maltodextrin, soybean oil, inulin, milk protein, di-potassium phosphate, tri-calcium phosphate, natural flavoring agent, release agent silica, magnesium hydroxide, emulsifier soy lecithins, L-ascorbic acid, iron diphosphate, sweetener steviol glycosides, niacin, DL alpha-tocopherol, zinc oxide, manganese (II) sulphate, copper carbonate, calcium D-pantothenate, dye beta carotene, pyridoxine hydrochloride, thiamine mononitrate, riboflavin, retinyl acetate, pteroylmonoglutamic acid, potassium iodide, sodium selenite, D-biotin, cholecalciferol, cyanocobalamin Nutrients per 100 g: Protein 42 g, carbohydrates 33 g thereof sugar 27 g, fat 6,4 g

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 according to labelled values of vitamins

Parameter	Content per 100 g	Parameter	Content per 100 g
L-Alanine	1,8 g	L-Lysine	2,7 g
L-Arginine	2,9 g	L-Methionine	0,55 g
L-Aspartic acid	4,6 g	L-Phenylalanine	2,2 g
L-Cysteine	0,80 g	L-Proline	2,4 g
L-Glutamic acid	7,9 g	L-Serine	2,0 g
Glycine	1,6 g	L-Threonine	1,6 g
L-Histidine	1,6 g	L-Tryptophan	0,50 g
L-Isoleucine	2,0 g	L-Tyrosine	1,6 g
L-Leucine	3,4 g	L-Valine	2,0 g

2.1.1 Homogeneity

The **mixture homogeneity before bottling** was examined 8-fold by **microtracer analysis**. It is a standardized method that is part of the international GMP certification system for feed [14]. Before mixing dye coated iron particles of µm size are added to the sample and the number of particles is determined after homogenization in taken aliquots. The evaluation of the mixture homogeneity is based on the Poisson distribution using the chi-square test. A probability of ≥ 5 % is equivalent to a good homogeneous mixture and of ≥ 25% to an excellent mixture [14, 15].
The microtracer analysis of the present PT sample showed a probability of 67%. Additionally particle number results were converted into concentrations, statistically evaluated according to normal distribution and compared to the standard deviation according to Horwitz. This gave a HorRat value of 1,2. The results of microtracer analysis are given in the documentation.

The calculation of the **repeatability standard deviation S_r of the participants** was also used as an indicator of homogeneity. It is < 2,7% (1,13% - 2,68%) for all analytes except for methionine, tryptophan and valine (3,78% - 6,71%). Therefore the repeatability standard deviations are similar to precision data of the referring standardized methods (e.g. ASU §64, s. 3.6.2) (see Tab. 3) [16-19]. The repeatability standard deviations of the participants' results are given in the tables of statistic data (see 4.1 to 4.19).

Furthermore, the homogeneity was characterized by the **trend line function of participants' results for chronological bottled single samples**. The maximum deviation from the mean value of the trend line for alanine and methionine was < 30% of the target standard deviation σ_{pt} (s. 5.2 homogeneity) and can therefore be regarded as low.

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

2.2 Sample shipment and information to the test

Two portions of test material were sent to every participating laboratory in the 37th week of 2016. The testing method was optional. The tests should be finished at 28th October 2016 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 dietetic food (drink powder as a meal replacement) based on soy and milk proteins. The protein content is > 20%. Any suitable method for determination of the analytes can be applied.
In general we recommend to homogenize a representative sample amount before analysis according to good laboratory practice, especially in case of low sample weights.

2.3 Submission of results

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

The finally calculated concentrations of the parameters 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 10 participants submitted the result 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^x) was calculated. The calculation was done according to algorithm A as described in annex C of ISO 13528 [3].

3.3 Repeatability standard deviation

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

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

The relative repeatability standard deviation as a percentage of the mean value is indicated as coefficient of variation CV_r in the table of 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 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. Its meaning is explained in more detail in 3.9.

3.5 Exclusion of results and outliers

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

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

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

3.6 Target standard deviation (for proficiency assessment)

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

If an acceptable quotient S^*/σ_{pt} is present, the target standard deviation of the general model by Horwitz is preferably used for the proficiency assessment. It is usually suitable for for evaluation of interlaboratory studies, where different analytical methods are applied by the participants. On the other hand the target standard deviation from the evaluation of precision data of an precision experiment is derived from collaborative studies with specified analytical methods.

In cases where both above-mentioned models are not suitable, the target standard deviation is determined based on values by perception, see under 3.6.3.

For information the z-scores of both models are given in the evaluation, if available.

In the present PT for valuation of all following parameters the target standard deviation according to the <u>general model of Horwitz</u> was applied (see 3.6.1): Alanine, Aspartic acid, Glycine, Lysine, Phenylalanine and Serine.

For the valuation of the following parameters the target standard devi-ation from <u>precision experiments</u> (s. 3.6.2) (German official ASU §64 method: 17, 18) was applied: Arginine, Cystine, Glutamic acid, Histid-ine, Isoleucine, Leucine, Methionine, Proline, Threonine, Tryptophan, Tyrosine and Valine.

Due to the low number of < 7 the results of <u>Cysteine</u> 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 µg/kg
$\sigma_R = 0{,}02c^{0,8495}$	$1{,}2 \times 10^{-7} \leq c \leq 0{,}138$	≥ 120 µg/kg
$\sigma_R = 0{,}01c^{0,5}$	$c > 0{,}138$	$> 13{,}8$ g/100g

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

3.6.2 Value by precision experiment

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

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

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

Table 3: Relative repeatability standard deviations (RSD_r) and relative reproducibility standard deviations (RSD_R) from precision experiments and resulting target standard deviations σ_{pt} [16-19]

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

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

3.6.3 Value by perception

The target standard deviation for proficiency assessment can be set at a value that corresponds to the level of performance that the coordinator would wish laboratories to be able to achieve [3].

For the present evaluation the target standard deviation according to 3.6.1 and 3.6.2 were regarded suitable.
Table 4 shows selected characteristics of participants results of the present PT in comparison to the previous year.

3.7 z-Score

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

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

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

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

The z-score valid for the PT evaluation is designated z-score (σ_{pt}), while the value of z-score (Info) is for information only. The two z-scores are calculated using the different target standard deviations according to 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].

<u>Table 4:</u> Characteristics of the present PT (on dark gray) 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	0,432	0,0150	3,47	0,77	DLA 42/2015
L-Alanine	Dietetic food	1,70	0,0562	3,31	0,90	DLA 59/2016
L-Arginine	Dietetic food	0,282	0,0384	13,6	1,69	DLA 42/2015
L-Arginine	Dietetic food	2,72	0,218	8,01	1,19	DLA 59/2016
L-Aspartic acid	Dietetic food	0,915	0,0369	4,03	0,55	DLA 42/2015
L-Aspartic acid	Dietetic food	4,55	0,130	2,86	0,90	DLA 59/2016
L-Cystine	Dietetic food	–	–	–	–	DLA 42/2015
L-Cystine	Dietetic food	0,487	0,133	27,3	1,89	DLA 59/2016
L-Glutamic acid	Dietetic food	1,906	0,124	6,51	1,36	DLA 42/2015
L-Glutamic acid	Dietetic food	8,29	0,502	6,05	1,23	DLA 59/2016
Glycine	Dietetic food	0,191	0,0116	6,07	1,20	DLA 42/2015
Glycine	Dietetic food	1,54	0,0933	6,06	1,62	DLA 59/2016
L-Histidin	Dietetic food	0,246	0,0662	26,9	1,62	DLA 42/2015
L-Histid-ine	Dietetic food	1,10	0,0915	8,32	0,78	DLA 59/2016
L-Isoleu-cine	Dietetic food	0,540	0,0276	5,11	1,16	DLA 42/2015
L-Isoleu-cine	Dietetic food	1,94	0,173	8,92	1,78	DLA 59/2016
L-Methion-ine	Dietetic food	0,215	0,0123	5,72	0,94	DLA 42/2015
L-Methion-ine	Dietetic food	0,572	0,0608	10,6	1,59	DLA 59/2016

Table 4 continued:

Parameter	Matrix (Powder)	rob. Mean [g/100g]	rob. SD (S*) [g/100g]	rel. SD (VK$_{S*}$) [%]	Quotient S*/σ$_{pt}$	DLA Report
L-Leucine	Dietetic food	1,02	0,0388	3,80	0,95	DLA 42/2015
L-Leucine	Dietetic food	3,45	0,248	7,19	1,34	DLA 59/2016
L-Lysine	Dietetic food	0,817	0,0529	6,47	1,57	DLA 42/2015
L-Lysine	Dietetic food	2,71	0,168	6,20	1,81	DLA 59/2016
L-Phenyl-alanine	Dietetic food	0,408	0,0201	4,93	1,07	DLA 42/2015
L-Phenyl-alanine	Dietetic food	2,14	0,144	6,73	1,88	DLA 59/2016
L-Proline	Dietetic food	0,744	0,0329	4,42	0,57	DLA 42/2015
L-Proline	Dietetic food	2,42	0,161	6,65	0,85	DLA 59/2016
L-Serine	Dietetic food	0,538	0,0762	14,2	1,77	DLA 42/2015
L-Serine	Dietetic food	2,23	0,110	4,93	1,39	DLA 59/2016
L-Threon-ine	Dietetic food	0,535	0,0203	3,79	0,86	DLA 42/2015
L-Threon-ine	Dietetic food	1,60	0,0914	5,71	1,53	DLA 59/2016
L-Trypto-phan	Dietetic food	–	–	–	–	DLA 42/2015
L-Trypto-phan	Dietetic food	0,563	0,0590	10,5	1,49	DLA 59/2016
L-Tyrosine	Dietetic food	0,314	0,0560	17,8	1,67	DLA 42/2015
L-Tyrosine	Dietetic food	1,56	0,166	10,6	1,61	DLA 59/2016
L-Valine	Dietetic food	0,598	0,0335	5,60	1,29	DLA 42/2015
L-Valine	Dietetic food	2,06	0,197	9,56	1,63	DLA 59/2016

3.8 z'-Score

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

The calculation is performed by:

$$z'_i = \frac{x_i - x_{pt}}{\sqrt{\sigma^2_{pt} + u^2_{(x_{pt})}}}$$

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$_R$)

The coefficient of variation (CV$_R$) of the reproducibility (= relative re-producibility 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 vari-ability the CV$_K$ gives the relative variability within a data region. While a low CV$_R$, e.g. < 5-10% can be taken as evidence for a homogeneous set of results, a CV$_R$ of more than 50% indicates a "strong inhomogeneity of statistical mass", so that the suitability for certain applications such as the assessment of exceeded maximum values or the performance evaluation of the participants possibly can not be done [3].

3.10 Quotient S*/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

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

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

If U(Xpt) ≤ 0,3 σ_{pt} the standard uncertainty of the consensus value needs not to be included in the interpretation of the results of the PT [3]. A clear exceeded the value of 0,3 is an indication that the target standard deviation was possibly set too low for the standard uncertainty of the assigned value.

The quotient U(X_{pt})/σ_{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 5.3). Partly slight shoulders and separate smaller peaks can be seen, which are due to individual values and outliers.
In the case of lysine and valine a distribution of results with two peaks can be seen. However, the information provided by the participants on the methods gave no obvious indications of such an array of results.
When using the robust standard deviation as an estimator h, the distributions are converted into single peak distributions, so that an statistical evaluation has been carried out.

Comments to the statistic data:

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

The target standard deviation was calculated for all other elements according to the model of Horwitz or according to the data of a precision experiment (ASU §64 method). The evaluation according to Horwitz was preferably used as long as the quotients $S*/\sigma_{pt}$ were in the range of ≤ 2,0. In all other cases, the standard deviation calculated from ASU §64 precision data was used. For proline and threonine 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 $S*/\sigma_{pt}$ were all below 2,0 in the range of 0,78 to 1,9 (see table 4).

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

The comparability of results is given.

The quotient $U_{(Xpt)}/\sigma_{pt}$ was increased > 0,3 for all parameters (0,31 bis 0,84), from which 6 quotients were in a slightly increased range of < 0,5 and 8 quotients > 0,5.

70% to 100% of results were in the target range.

The robust means of the participant results, with the exception of histidine, ranged from 94% to 112% 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
Number of results
Number of outliers
Mean
Median
Robust mean(X_{pt})
Robust standard deviation (S*)
Number with m replicate measurements
Repeatability standard deviation (S_r)
Coefficient of Variation (CV_r)in %
Reproducibility standard deviation (S_R)
Coefficient of Variation (CV_R)in %
Target range:
Target standard deviation σ_{pt} or σ_{pt}'
Target standard deviation for information
lower limit of target range $(X_{pt} - 2\sigma_{pt})$ or $(X_{pt} - 2\sigma_{pt}')$ *
upper limit of target range $(X_{pt} + 2\sigma_{pt})$ or $(X_{pt} + 2\sigma_{pt}')$ *
Variation coefficient V_K in %
Quotient S/σ_{pt} or S*/σ_{pt}'*
Standard uncertainty U(X_{pt})
Quotient U(X_{pt})/σ_{pt} or U(X_{pt})/σ_{pt}'
Number of results in the target range
Percent in the target range

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

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

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

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

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	10
Number of outliers	0
Mean	1,70
Median	1,69
Robust Mean (X)	**1,70**
Robust standard deviation (S*)	**0,0562**
Number with 2 replicates	10
Repeatability SD (S_r)	0,0254
Repeatability (CV_r)	1,49%
Reproducibility SD (S_R)	0,0602
Reproducibility (CV_R)	3,54%
Target range:	
Target standard deviation σ_{pt}	**0,0627**
Target standard deviation (for Information)	0,0822
lower limit of target range	**1,57**
upper limit of target range	**1,82**
Quotient S*/σ_{pt}	*0,90*
Standard uncertainty U(Xpt)	*0,0222*
Quotient U(Xpt)/σ_{pt}	*0,35*
Results in the target range	*10*
Percent in the target range	*100%*
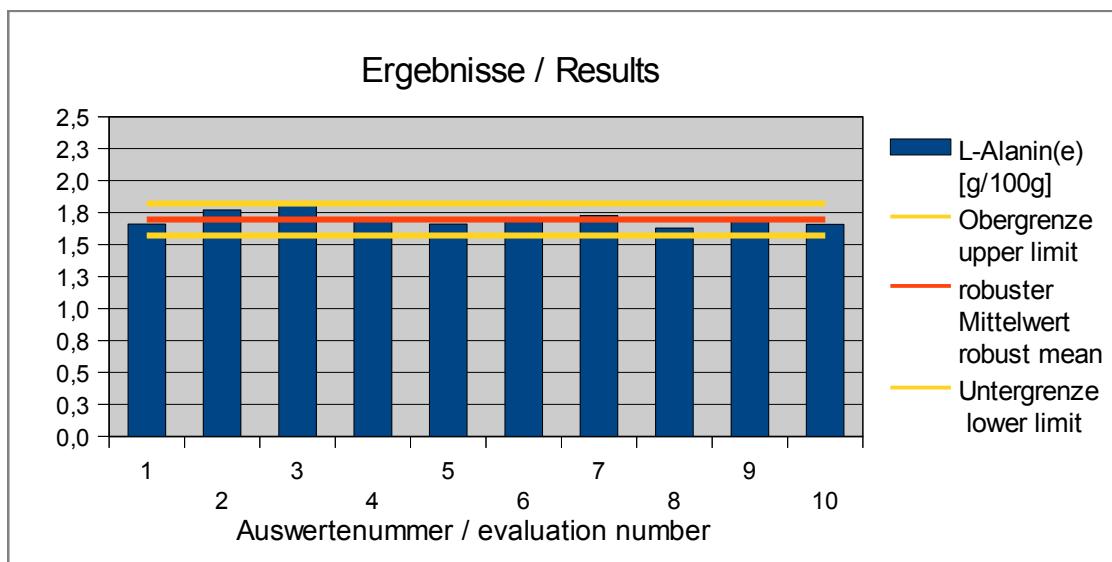
Abb. / Fig. 1: Ergebnisse L-Alanin **/** Results L-Alanine

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	L-Alanin / L-Alanine [g/100g]	Abweichung [g/100g] Deviation [g/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	1,66	−0,0364	−0,6	−0,4	
2	1,77	0,0736	1,2	0,9	
3	1,82	0,1236	2,0	1,5	
4	1,69	−0,0064	−0,1	−0,1	
5	1,66	−0,0364	−0,6	−0,4	
6	1,70	0,0036	0,1	0,0	
7	1,73	0,0312	0,5	0,4	
8	1,63	−0,0664	−1,1	−0,8	
9	1,69	−0,0094	−0,2	−0,1	
10	1,66	−0,0374	−0,6	−0,5	
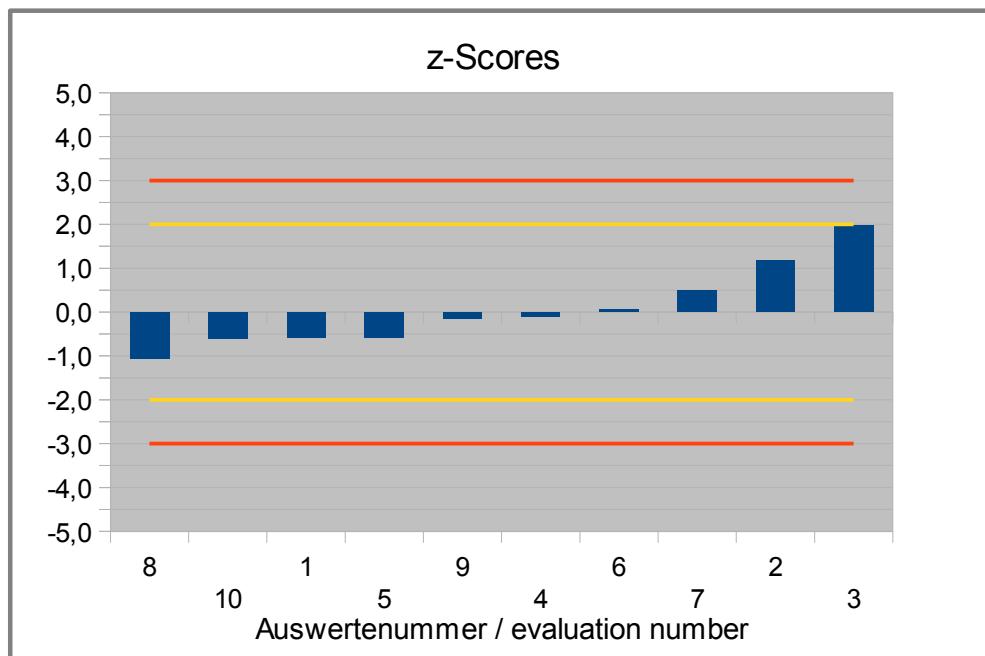
Abb. / Fig. 2:   Z-Scores L-Alanin / L-Alanine

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

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	10
Number of outliers	0
Mean	2,72
Median	2,75
Robust Mean (X)	**2,72**
Robust standard deviation (S*)	**0,218**
Number with 2 replicates	9
Repeatability SD (S_r)	0,0495
Repeatability (CV_r)	1,83%
Reproducibility SD (S_R)	0,206
Reproducibility (CV_R)	7,64%
Target range:	
Target standard deviation σ_{pt}	**0,183**
Target standard deviation (for Information)	0,0937
lower limit of target range	**2,36**
upper limit of target range	**3,09**
Quotient S*/σ_{pt}	*1,19*
Standard uncertainty U$_{(Xpt)}$	*0,0860*
Quotient U$_{(Xpt)}$/σ_{pt}	*0,47*
Results in the target range	*9*
Percent in the target range	*90%*
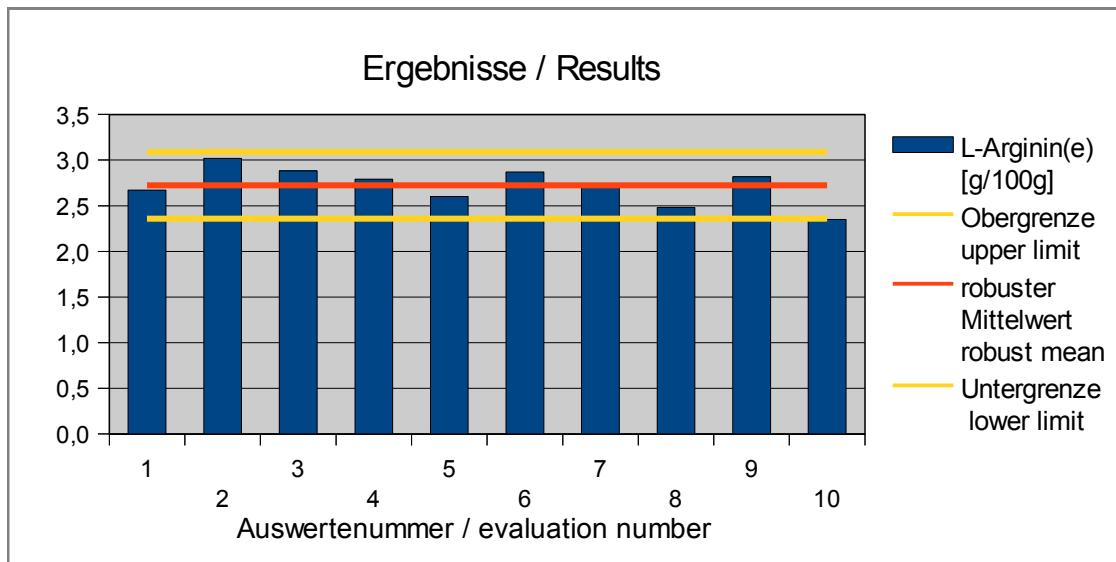
Abb. / Fig. 3: Ergebnisse L-Arginin / Results L-Arginine

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer / Evaluation number	L-Arginin / L-Arginine [g/100g]	Abweichung [g/100g] / Deviation [g/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis / Remark
1	2,67	−0,0536	−0,3	−0,6	
2	3,02	0,2964	1,6	3,2	
3	2,88	0,1564	0,9	1,7	
4	2,79	0,0664	0,4	0,7	
5	2,60	−0,1236	−0,7	−1,3	
6	2,87	0,1464	0,8	1,6	
7	2,71	−0,0131	−0,1	−0,1	
8	2,48	−0,2436	−1,3	−2,6	
9	2,82	0,0944	0,5	1,0	
10	2,35	−0,3746	−2,0	−4,0	

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	9
Number of outliers	1
Mean	4,51
Median	4,53
Robust Mean (X)	**4,55**
Robust standard deviation (S*)	**0,130**
Number with 2 replicates	9
Repeatability SD (S_r)	0,0868
Repeatability (CV_r)	1,98%
Reproducibility SD (S_R)	0,405
Reproducibility (CV_R)	9,23%
Target range:	
Target standard deviation σ_{pt}	**0,145**
Target standard deviation (for Information)	0,333
lower limit of target range	**4,26**
upper limit of target range	**4,84**
Quotient S*/σ_{pt}	*0,90*
Standard uncertainty U(Xpt)	*0,0541*
Quotient U(Xpt)/σ_{pt}	*0,37*
Results in the target range	*8*
Percent in the target range	*89%*
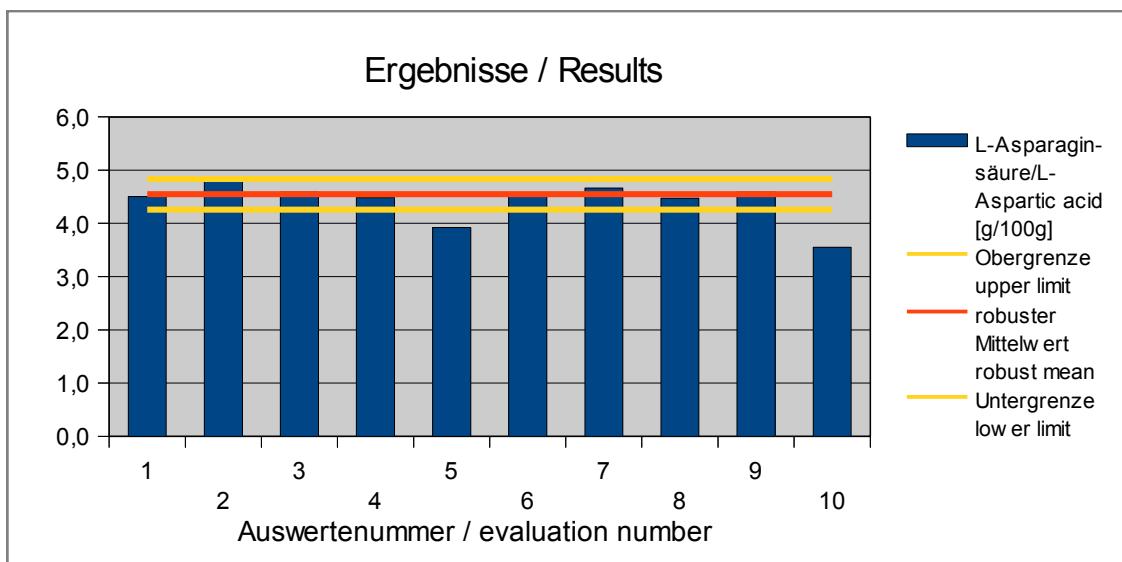
Abb. / Fig. 5: Ergebnisse L-Assparginsäure **/** Results L-Aspartic acid

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer / Evaluation number	L-Asparagin-säure / L-Aspartic acid [g/100g]	Abweichung [g/100g] Deviation [g/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	4,50	−0,0457	−0,3	−0,1	
2	4,83	0,2843	2,0	0,9	
3	4,59	0,0443	0,3	0,1	
4	4,48	−0,0657	−0,5	−0,2	
5	3,92	−0,6257	−4,3	−1,9	Ausreisser / Outlier
6	4,53	−0,0157	−0,1	0,0	
7	4,66	0,1179	0,8	0,4	
8	4,47	−0,0757	−0,5	−0,2	
9	4,59	0,0403	0,3	0,1	
10	3,55	−0,9533			Ausreisser ausgeschlossen* / Outlier excluded*

* Einfluss auf robuste Statistik / * influence on robust statistics
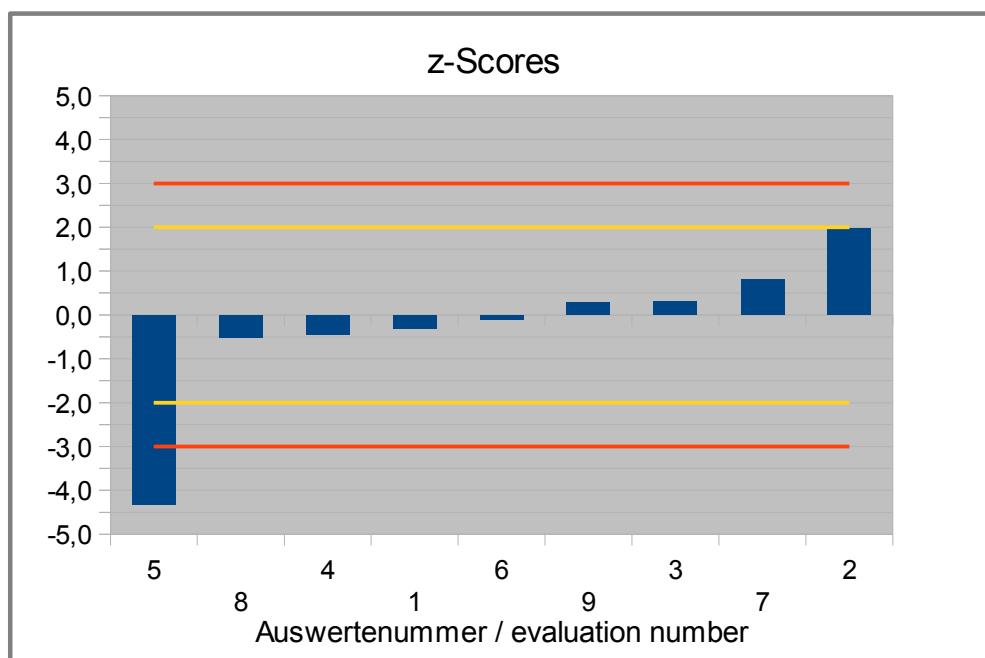
Abb. / Fig. 6:   Z-Scores L-Asparaginsäure / L-Aspartic acid

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

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	2
Number of outliers	0
Mean	0,482
Median	0,482
Robust Mean (X)	**0,482**
Robust standard deviation (S*)	**0,0617**
Number with 2 replicates	
Repeatability SD (S$_r$)	
Repeatability (CV$_r$)	
Reproducibility SD (S$_R$)	
Reproducibility (CV$_R$)	
Target range:	
Target standard deviation σ_{pt}	
Target standard deviation (for Information)	
lower limit of target range	
upper limit of target range	
Quotient S*/σ_{pt}	
Standard uncertainty U$_{(Xpt)}$	
Quotient U$_{(Xpt)}$/σ_{pt}	
Results in the target range	
Percent in the target range	
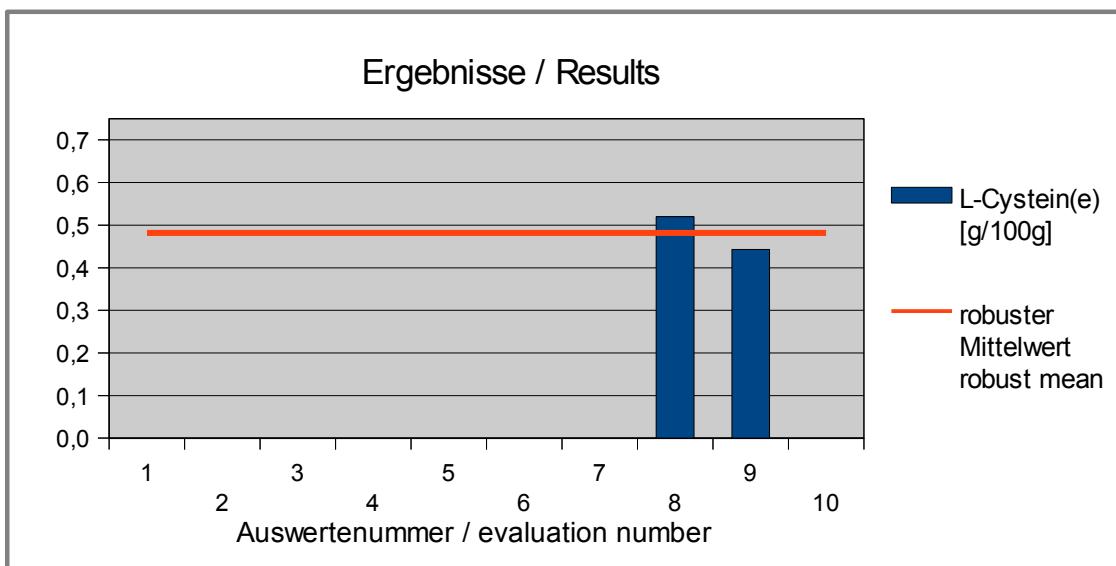
Abb. / Fig. 7: Ergebnisse L-Cystein / Results L-Cysteine

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte- nummer Evaluation number	L-Cystein(e) [g/100g]	Abweichung [g/100g] Deviation [g/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1					
2					
3					
4					
5					
6					
7					
8	0,520	0,0385			
9	0,443	−0,0385			
10					

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

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	8
Number of outliers	0
Mean	0,487
Median	0,473
Robust Mean (X)	**0,487**
Robust standard deviation (S*)	**0,133**
Number with 2 replicates	7
Repeatability SD (S$_r$)	0,0129
Repeatability (CV$_r$)	2,68%
Reproducibility SD (S$_R$)	0,142
Reproducibility (CV$_R$)	29,4%
Target range:	
Target standard deviation σ_{pt}	**0,0705**
Target standard deviation (for Information)	0,0217
lower limit of target range	**0,35**
upper limit of target range	**0,63**
Quotient S/σ_{pt}*	*1,89*
Standard uncertainty U$_{(Xpt)}$	*0,0590*
Quotient U$_{(Xpt)}$/σ_{pt}	*0,84*
Results in the target range	*6*
Percent in the target range	*75%*
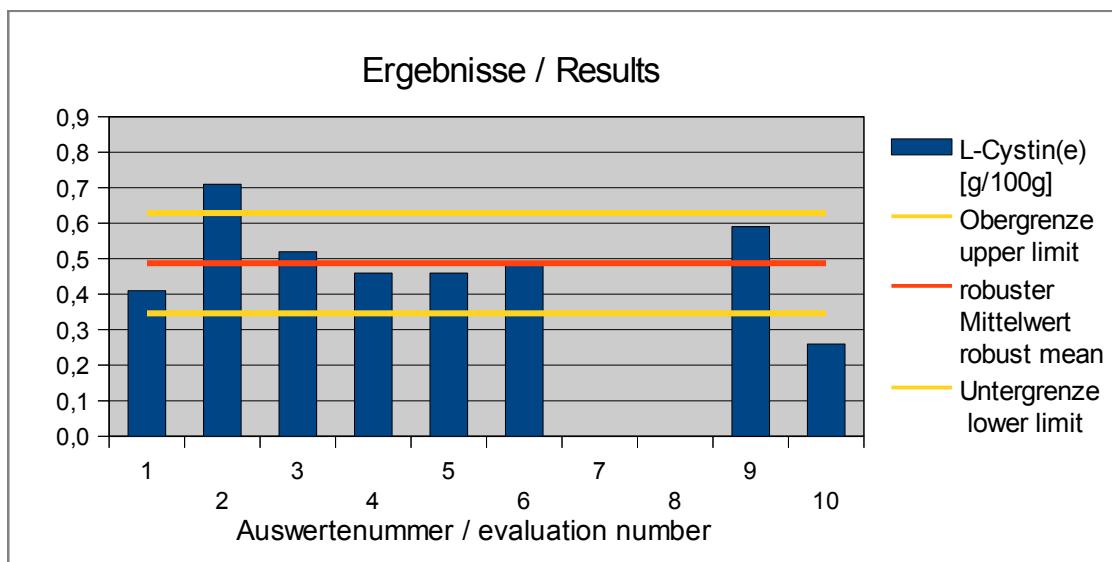
Abb. / Fig. 8: Ergebnisse L-Cystin **/** Results L-Cystine

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer	L-Cystin(e) [g/100g]	Abweichung [g/100g]	z-Score	z-Score	Hinweis
Evaluation number		Deviation [g/100g]	(σ_{pt})	(Info)	Remark
1	0,410	-0,0775	-1,1	-3,6	
2	0,710	0,2225	3,2	10,2	
3	0,520	0,0325	0,5	1,5	
4	0,460	-0,0275	-0,4	-1,3	
5	0,460	-0,0275	-0,4	-1,3	
6	0,485	-0,0025	0,0	-0,1	
7					
8					
9	0,590	0,1025	1,5	4,7	
10	0,260	-0,2275	-3,2	-10,5	
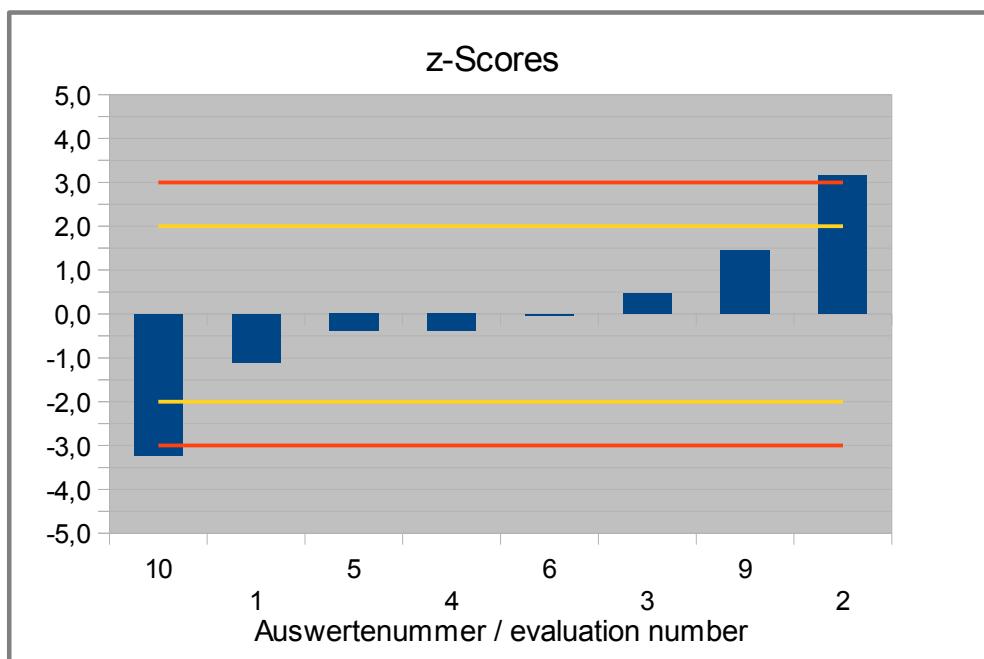
Abb. / Fig. 9:   Z-Scores L-Cystin / L-Cystine

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

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	10
Number of outliers	0
Mean	8,29
Median	8,14
Robust Mean (X)	**8,29**
Robust standard deviation (S*)	**0,502**
Number with 2 replicates	9
Repeatability SD (S$_r$)	0,140
Repeatability (CV$_r$)	1,70%
Reproducibility SD (S$_R$)	0,474
Reproducibility (CV$_R$)	5,74%
Target range:	
Target standard deviation σ_{pt}	**0,406**
Target standard deviation (for Information)	0,241
lower limit of target range	**7,48**
upper limit of target range	**9,11**
Quotient S/σ_{pt}*	*1,2*
Standard uncertainty U$_{(Xpt)}$	*0,198*
Quotient U$_{(Xpt)}$/σ_{pt}	*0,49*
Results in the target range	*10*
Percent in the target range	*100%*
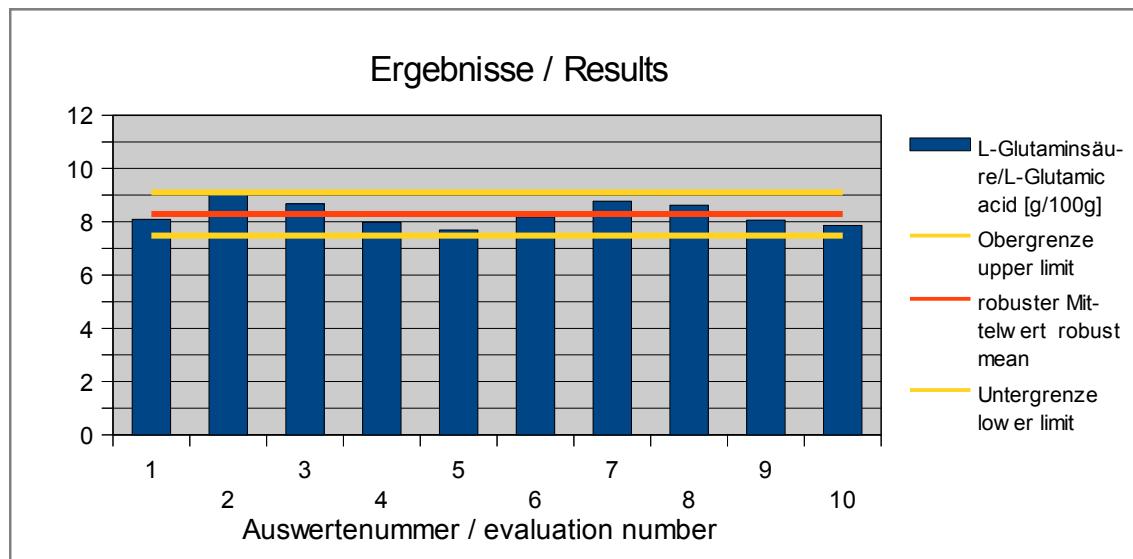
Abb. / Fig. 10: Ergebnisse L-Glutaminsäure/ Results L-Glutamic acid

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer	L-Glutamin-säure/	Abweichung [g/100g]	z-Score	z-Score	Hinweis
Evaluation number	L-Glutamic acid [g/100g]	Deviation [g/100g]	(σ_{pt})	(Info)	Remark
1	8,09	−0,204	−0,5	−0,8	
2	9,02	0,726	1,8	3,0	
3	8,67	0,376	0,9	1,6	
4	7,98	−0,314	−0,8	−1,3	
5	7,69	−0,604	−1,5	−2,5	
6	8,18	−0,114	−0,3	−0,5	
7	8,77	0,474	1,2	2,0	
8	8,62	0,326	0,8	1,4	
9	8,06	−0,233	−0,6	−1,0	
10	7,86	−0,435	−1,1	−1,8	
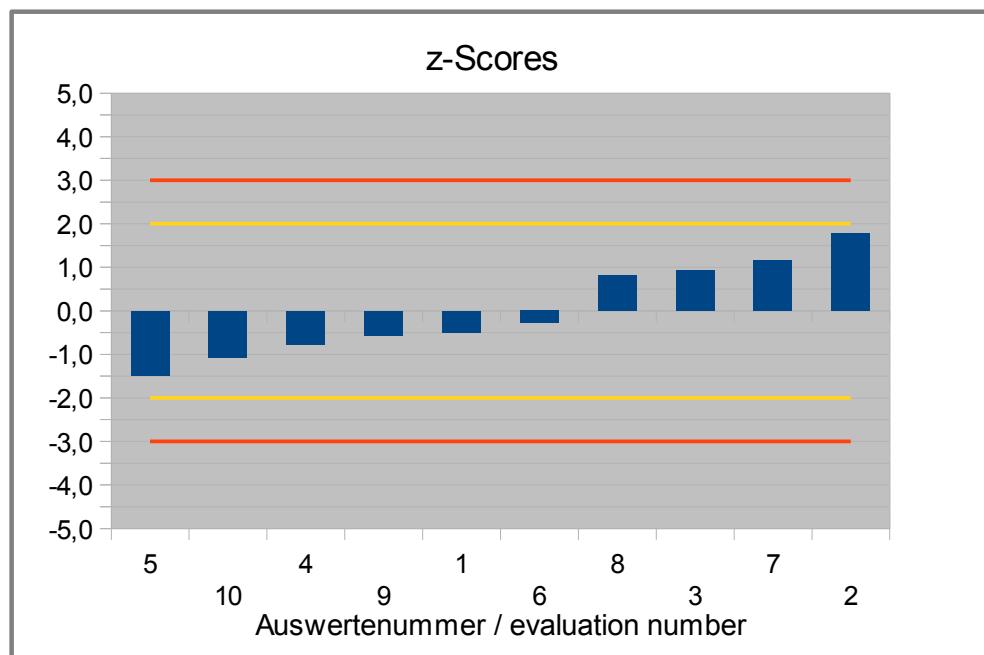
Abb. / Fig. 11:   Z-Scores L-Glutaminsäure / L-Glutamic acid

4.7 Glycin/Glycine in g/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	10
Number of outliers	0
Mean	1,54
Median	1,57
Robust Mean (X)	**1,54**
Robust standard deviation (S*)	**0,0933**
Number with 2 replicates	9
Repeatability SD (S$_r$)	0,0210
Repeatability (CV$_r$)	1,38%
Reproducibility SD (S$_R$)	0,0852
Reproducibility (CV$_R$)	5,57%
Target range:	
Target standard deviation σ_{pt}	**0,0576**
Target standard deviation (for Information)	0,102
lower limit of target range	**1,42**
upper limit of target range	**1,65**
Quotient S*/σ_{pt}	*1,62*
Standard uncertainty U$_{(Xpt)}$	*0,0369*
Quotient U$_{(Xpt)}$/σ_{pt}	*0,64*
Results in the target range	7
Percent in the target range	70%
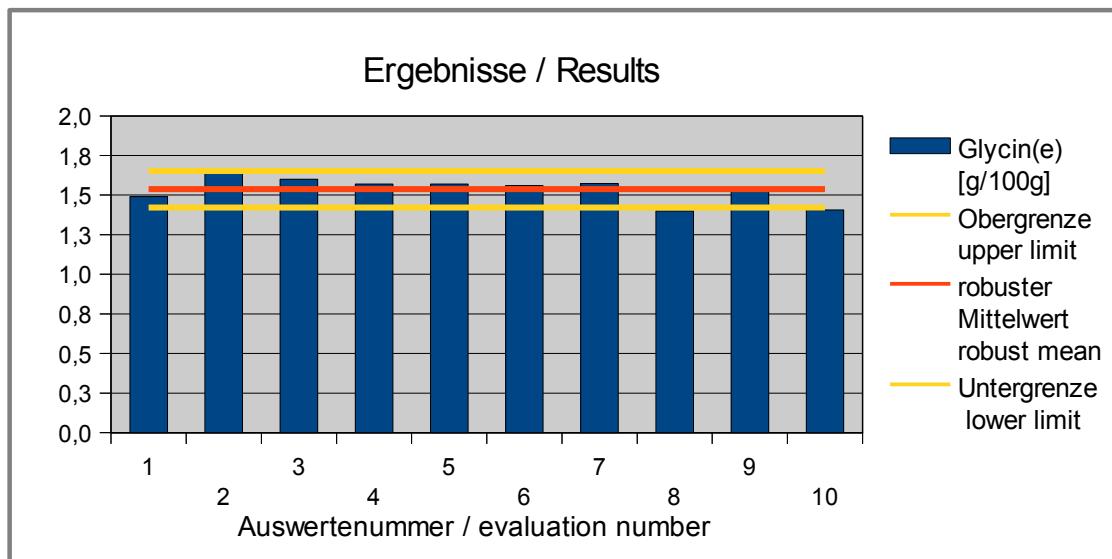
Abb. / Fig. 12: Ergebnisse Glycin**/** Results Glycine

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Glycin(e) [g/100g]	Abweichung [g/100g] Deviation [g/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	1,49	−0,0474	−0,8	−0,5	
2	1,66	0,1226	2,1	1,2	
3	1,60	0,0626	1,1	0,6	
4	1,57	0,0326	0,6	0,3	
5	1,57	0,0326	0,6	0,3	
6	1,56	0,0226	0,4	0,2	
7	1,57	0,0352	0,6	0,3	
8	1,40	−0,1374	−2,4	−1,3	
9	1,54	0,0066	0,1	0,1	
10	1,41	−0,1304	−2,3	−1,3	
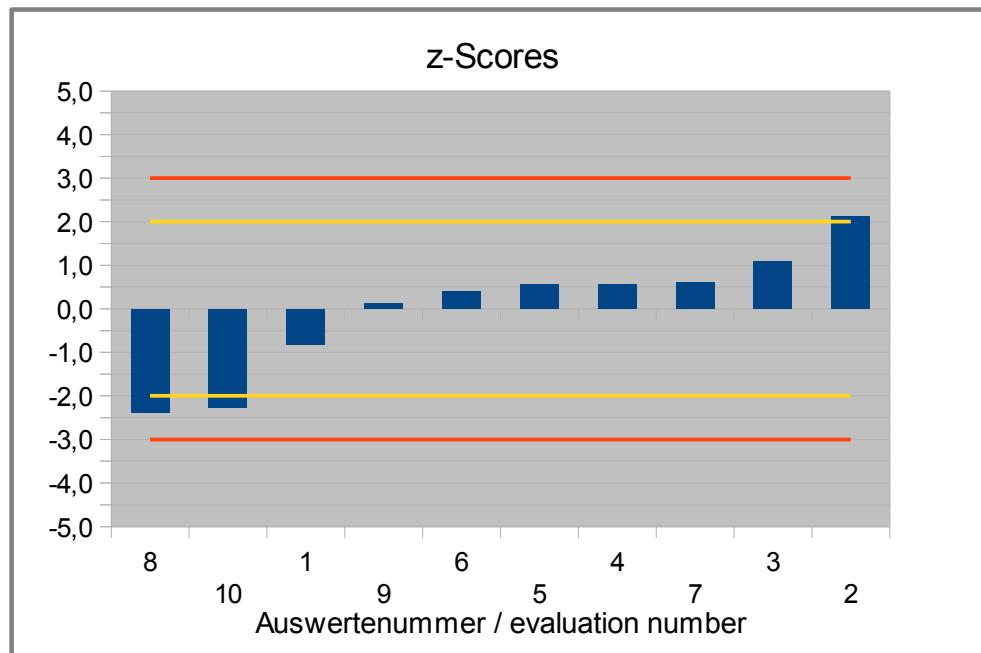
Abb. / Fig. 13:   Z-Scores Glycin / Glycine

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

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	10
Number of outliers	0
Mean	1,10
Median	1,07
Robust Mean (X)	**1,10**
Robust standard deviation (S*)	**0,0915**
Number with 2 replicates	9
Repeatability SD (S$_r$)	0,0220
Repeatability (CV$_r$)	2,03%
Reproducibility SD (S$_R$)	0,0748
Reproducibility (CV$_R$)	6,91%
Target range:	
Target standard deviation σ_{pt}	**0,118**
Target standard deviation (for Information)	0,0433
lower limit of target range	**0,86**
upper limit of target range	**1,33**
Quotient S*/σ_{pt}	*0,78*
Standard uncertainty U$_{(Xpt)}$	*0,0362*
Quotient U$_{(Xpt)}$/σ_{pt}	*0,31*
Results in the target range	*10*
Percent in the target range	*100%*
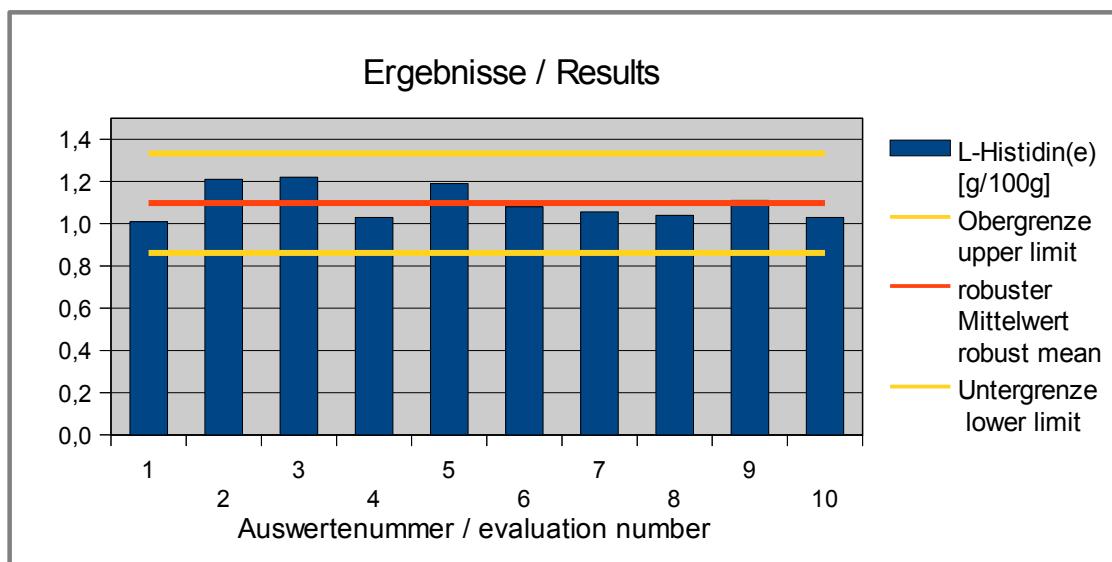
Abb. / Fig. 14: Ergebnisse L-Histidin**/** Results L-Histidine

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	L-Histidin / L-Histidine [g/100g]	Abweichung [g/100g] Deviation [g/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	1,01	−0,0876	−0,7	−2,0	
2	1,21	0,1124	1,0	2,6	
3	1,22	0,1224	1,0	2,8	
4	1,03	−0,0676	−0,6	−1,6	
5	1,19	0,0924	0,8	2,1	
6	1,08	−0,0176	−0,1	−0,4	
7	1,06	−0,0410	−0,3	−0,9	
8	1,04	−0,0576	−0,5	−1,3	
9	1,11	0,0124	0,1	0,3	
10	1,03	−0,0686	−0,6	−1,6	
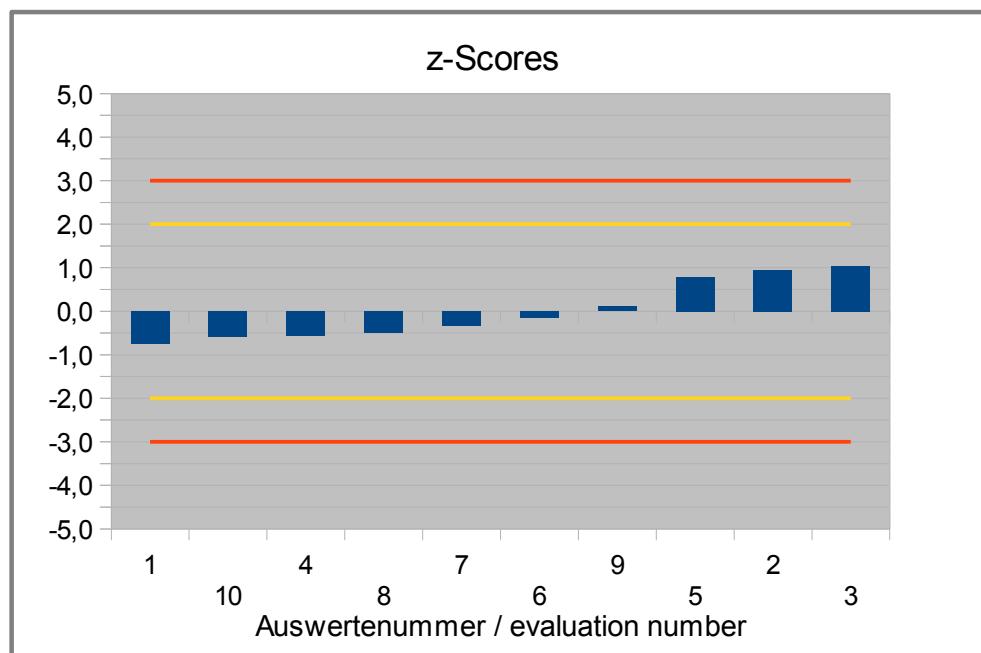
Abb. / Fig. 15:   Z-Scores L-Histidin / L-Histidine

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

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	10
Number of outliers	1
Mean	1,91
Median	1,97
Robust Mean (X)	**1,94**
Robust standard deviation (S*)	**0,173**
Number with 2 replicates	9
Repeatability SD (S$_r$)	0,0356
Repeatability (CV$_r$)	1,87%
Reproducibility SD (S$_R$)	0,241
Reproducibility (CV$_R$)	12,7%
Target range:	
Target standard deviation σ_{pt}	**0,0975**
Target standard deviation (for Information)	0,0702
lower limit of target range	**1,75**
upper limit of target range	**2,14**
Quotient S*/σ_{pt}	*1,78*
Standard uncertainty U$_{(Xpt)}$	*0,0686*
Quotient U$_{(Xpt)}$/σ_{pt}	*0,70*
Results in the target range	*9*
Percent in the target range	*90%*
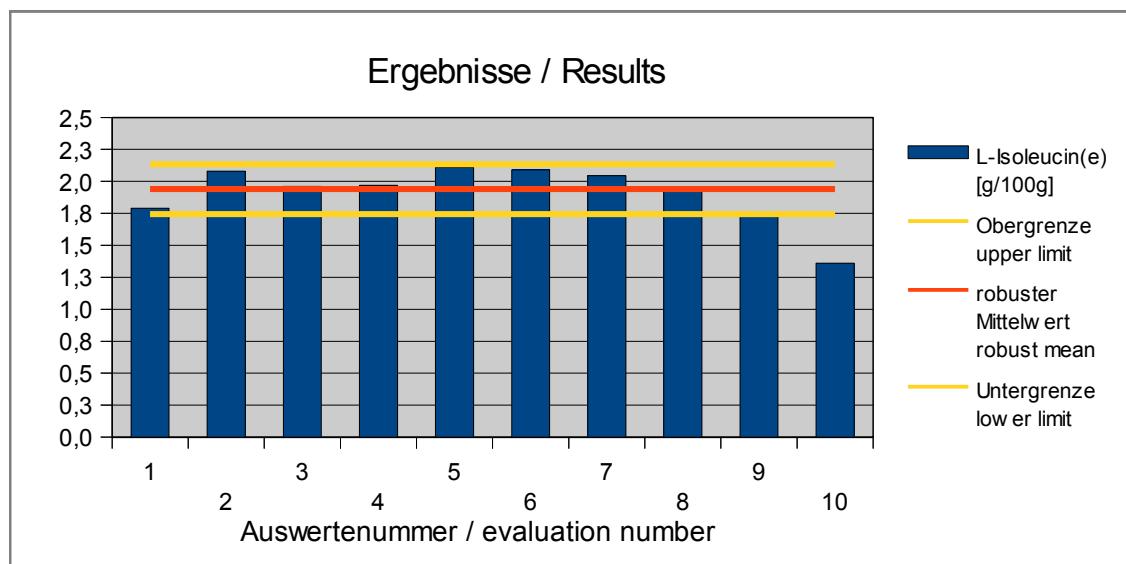
Abb. / Fig. 16: Ergebnisse L-Isoleucin**/** Results L-Isoleucine

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	L-Isoleucin / L-Isoleucine [g/100g]	Abweichung [g/100g] Deviation [g/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	1,79	−0,1501	−1,5	−2,1	
2	2,08	0,1399	1,4	2,0	
3	1,96	0,0199	0,2	0,3	
4	1,97	0,0299	0,3	0,4	
5	2,11	0,1699	1,7	2,4	
6	2,09	0,1499	1,5	2,1	
7	2,04	0,1037	1,1	1,5	
8	1,93	−0,0101	−0,1	−0,1	
9	1,75	−0,1931	−2,0	−2,7	
10	1,36	−0,5801	−5,9	−8,3	Ausreisser/Outlier
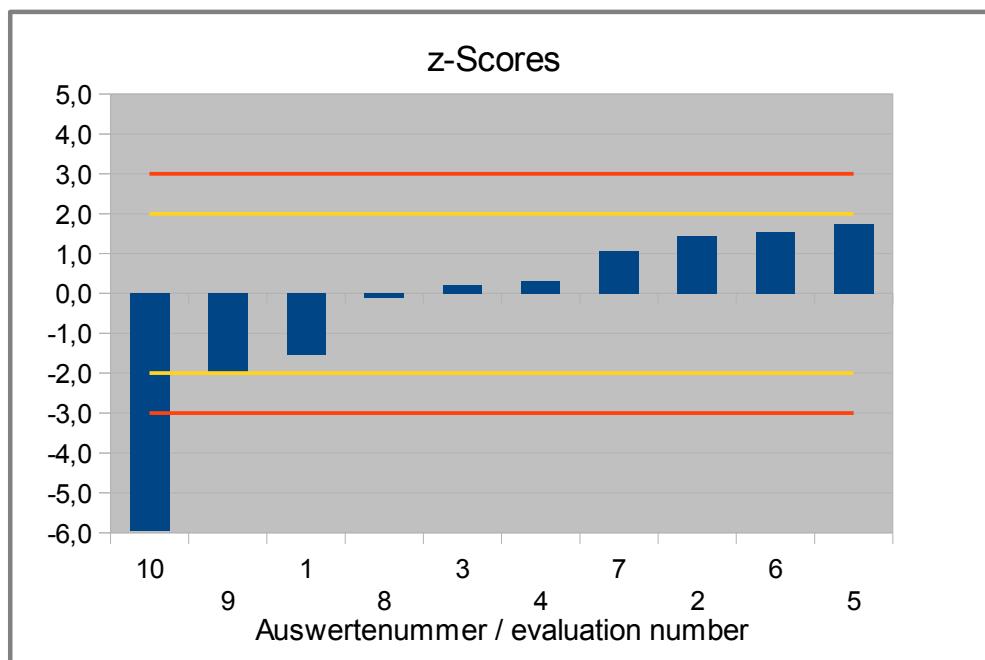
Abb. / Fig. 17:   Z-Scores L-Isoleucin / L-Isoleucine

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

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	10
Number of outliers	0
Mean	3,45
Median	3,41
Robust Mean (X)	**3,45**
Robust standard deviation (S*)	**0,248**
Number with 2 replicates	9
Repeatability SD (S$_r$)	0,0539
Repeatability (CV$_r$)	1,58%
Reproducibility SD (S$_R$)	0,231
Reproducibility (CV$_R$)	6,76%
Target range:	
Target standard deviation σ_{pt}	**0,186**
Target standard deviation (for Information)	0,115
lower limit of target range	**3,08**
upper limit of target range	**3,82**
Quotient S*/σ_{pt}	*1,34*
Standard uncertainty U$_{(Xpt)}$	*0,0981*
Quotient U$_{(Xpt)}$/σ_{pt}	*0,53*
Results in the target range	*9*
Percent in the target range	*90%*
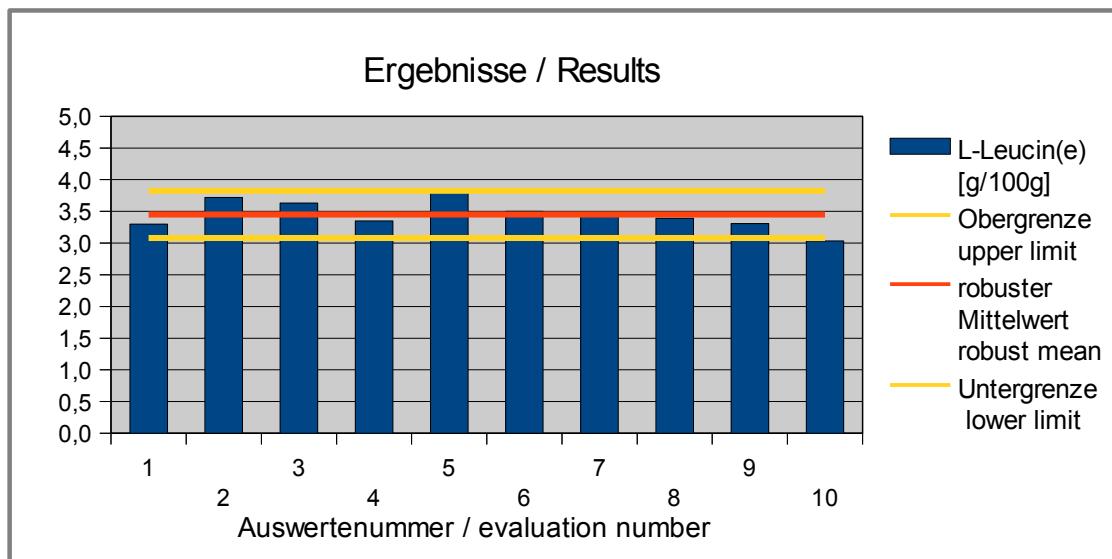
Abb. / Fig. 18: Ergebnisse L-Leucin/ Results L-Leucine

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	L-Leucin/ L-Leucine [g/100g]	Abweichung [g/100g] Deviation [g/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	3,30	−0,1520	−0,8	−1,3	
2	3,72	0,2680	1,4	2,3	
3	3,63	0,1780	1,0	1,6	
4	3,35	−0,1020	−0,5	−0,9	
5	3,81	0,3580	1,9	3,1	
6	3,50	0,0480	0,3	0,4	
7	3,43	−0,0186	−0,1	−0,2	
8	3,39	−0,0620	−0,3	−0,5	
9	3,31	−0,1450	−0,8	−1,3	
10	3,03	−0,4200	−2,3	−3,7	
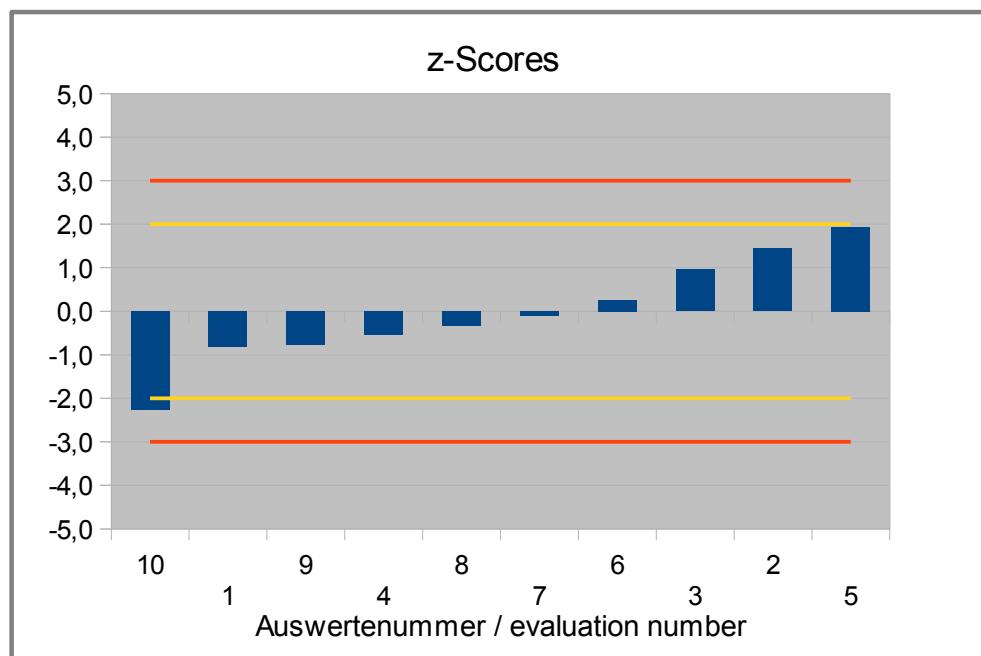
Abb. / Fig. 19:   Z-Scores L-Leucin / L-Leucine

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

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	10
Number of outliers	0
Mean	2,71
Median	2,73
Robust Mean (X)	**2,71**
Robust standard deviation (S*)	**0,168**
Number with 2 replicates	9
Repeatability SD (S$_r$)	0,0464
Repeatability (CV$_r$)	1,72%
Reproducibility SD (S$_R$)	0,156
Reproducibility (CV$_R$)	5,79%
Target range:	
Target standard deviation σ_{pt}	**0,0932**
Target standard deviation (for Information)	0,239
lower limit of target range	**2,52**
upper limit of target range	**2,89**
Quotient S*/σ_{pt}	*1,81*
Standard uncertainty U$_{(Xpt)}$	*0,0665*
Quotient U$_{(Xpt)}$/σ_{pt}	*0,71*
Results in the target range	*9*
Percent in the target range	*90%*
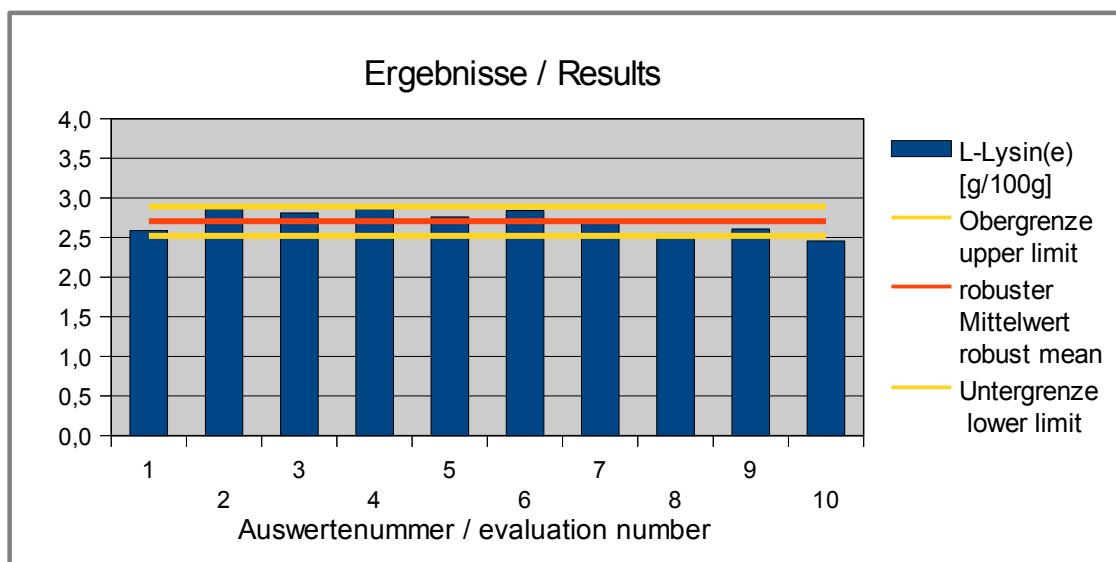
Abb. / Fig. 20: Ergebnisse L-Lysin/ Results L-Lysine

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	L-Lysin / L-Lysine [g/100g]	Abweichung [g/100g] Deviation [g/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	2,59	−0,1170	−1,3	−0,5	
2	2,86	0,1530	1,6	0,6	
3	2,81	0,1030	1,1	0,4	
4	2,89	0,1830	2,0	0,8	
5	2,76	0,0530	0,6	0,2	
6	2,84	0,1330	1,4	0,6	
7	2,70	−0,0026	0,0	0,0	
8	2,55	−0,1570	−1,7	−0,7	
9	2,61	−0,0990	−1,1	−0,4	
10	2,46	−0,2490	−2,7	−1,0	
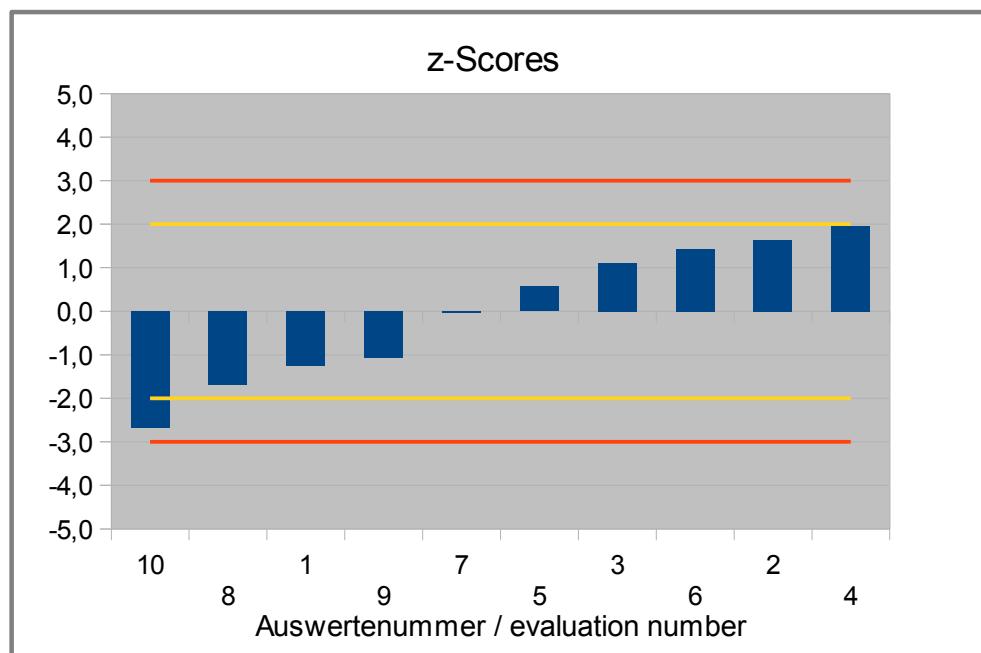
Abb. / Fig. 21:   Z-Scores L-Lysin / L-Lysine

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

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	10
Number of outliers	0
Mean	0,565
Median	0,570
Robust Mean (X)	**0,572**
Robust standard deviation (S*)	**0,0608**
Number with 2 replicates	9
Repeatability SD (S$_r$)	0,0209
Repeatability (CV$_r$)	3,78%
Reproducibility SD (S$_R$)	0,0685
Reproducibility (CV$_R$)	12,4%
Target range:	
Target standard deviation σ_{pt}	**0,0382**
Target standard deviation (for Information)	0,0249
lower limit of target range	**0,50**
upper limit of target range	**0,65**
Quotient S*/σ_{pt}	*1,59*
Standard uncertainty U$_{(Xpt)}$	*0,0240*
Quotient U$_{(Xpt)}$/σ_{pt}	*0,63*
Results in the target range	*8*
Percent in the target range	*80%*
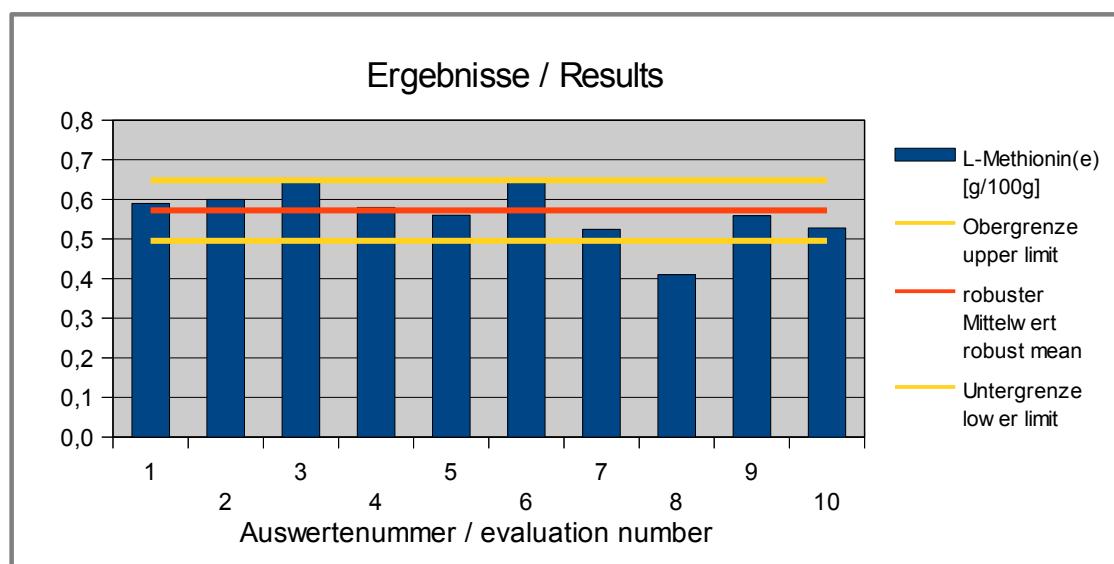
Abb. / Fig. 22: Ergebnisse L-Methionin**/** Results L-Methionine

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer / Evaluation number	L-Methionin(e) [g/100g]	Abweichung [g/100g] / Deviation [g/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis / Remark
1	0,590	0,0180	0,5	0,7	
2	0,600	0,0280	0,7	1,1	
3	0,650	0,0780	2,0	3,1	
4	0,580	0,0080	0,2	0,3	
5	0,560	-0,0120	-0,3	-0,5	
6	0,648	0,0760	2,0	3,1	
7	0,524	-0,0478	-1,3	-1,9	
8	0,410	-0,1620	-4,2	-6,5	
9	0,559	-0,0130	-0,3	-0,5	
10	0,528	-0,0440	-1,2	-1,8	
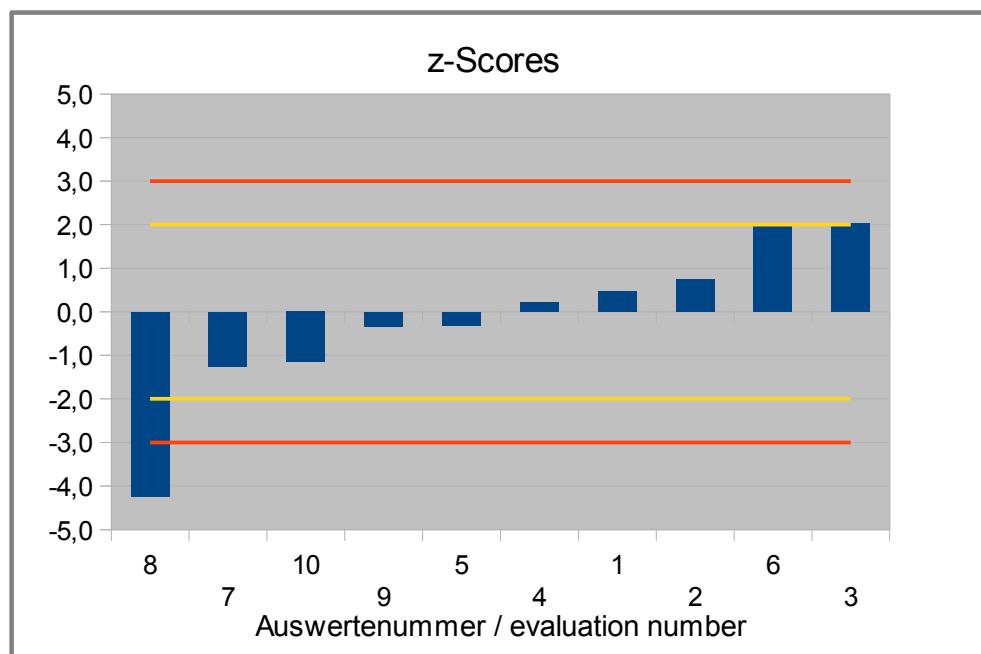
Abb. / Fig. 23:   Z-Scores L-Methionin / L-Methionine

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

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	10
Number of outliers	0
Mean	2,15
Median	2,14
Robust Mean (X)	**2,14**
Robust standard deviation (S*)	**0,144**
Number with 2 replicates	9
Repeatability SD (S$_r$)	0,0352
Repeatability (CV$_r$)	1,64%
Reproducibility SD (S$_R$)	0,178
Reproducibility (CV$_R$)	8,28%
Target range:	
Target standard deviation σ_{pt}	**0,0765**
lower limit of target range	**1,99**
upper limit of target range	**2,30**
Quotient S*/σ_{pt}	*1,88*
Standard uncertainty U$_{(Xpt)}$	*0,0569*
Quotient U$_{(Xpt)}$/σ_{pt}	*0,74*
Results in the target range	7
Percent in the target range	70%
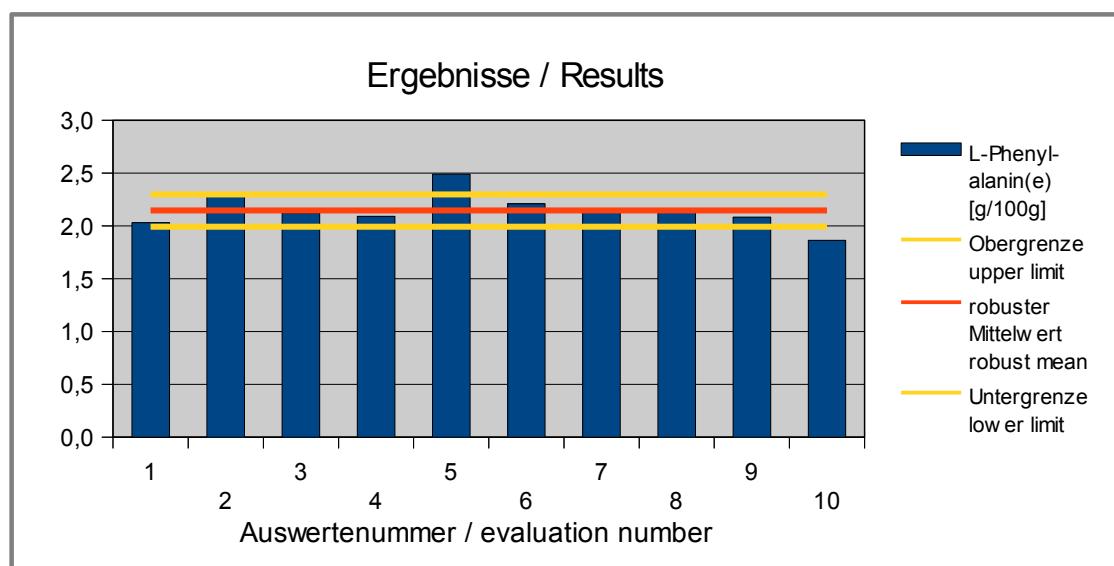
Abb. / Fig. 24: Ergebnisse L-Phenylalanin**/** Results L-Phenylalanine

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte- nummer / Evaluation number	L-Phenyl- alanin(e) [g/100g]	Abweichung [g/100g] / Deviation [g/100g]	z-Score (σ_{pt})	Hinweis / Remark
1	2,03	−0,1147	−1,5	
2	2,31	0,1653	2,2	
3	2,12	−0,0247	−0,3	
4	2,09	−0,0547	−0,7	
5	2,49	0,3453	4,5	
6	2,21	0,0653	0,9	
7	2,15	0,0099	0,1	
8	2,16	0,0153	0,2	
9	2,08	−0,0617	−0,8	
10	1,86	−0,2827	−3,7	
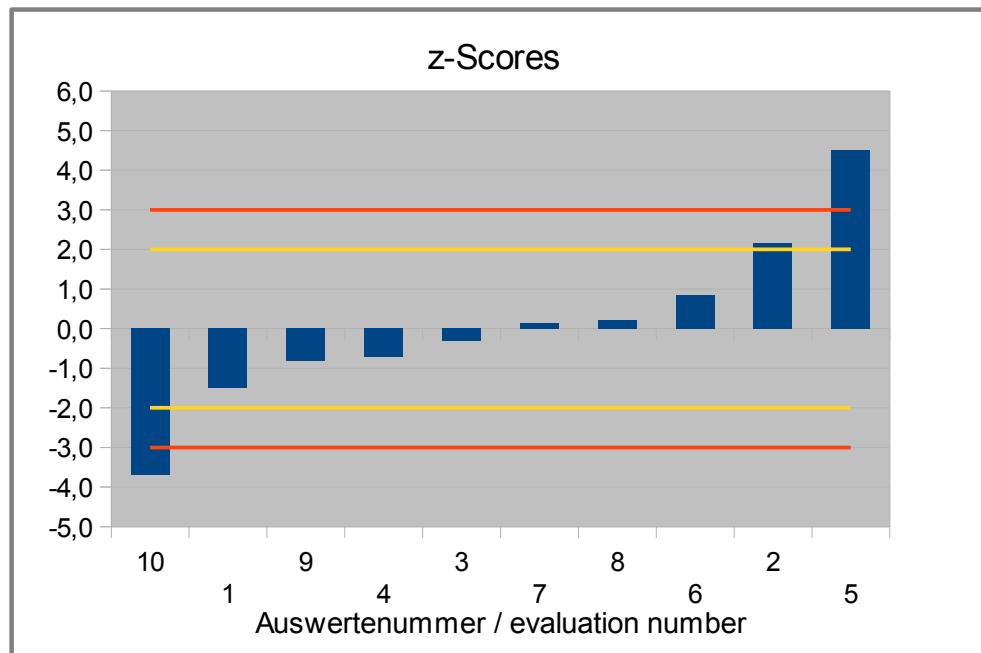
Abb. / Fig. 25:   Z-Scores L-Phenylalanin / L-Phenylalanine

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

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	10
Number of outliers	0
Mean	2,40
Median	2,43
Robust Mean (X)	**2,42**
Robust standard deviation (S*)	**0,161**
Number with 2 replicates	9
Repeatability SD (S_r)	0,0599
Repeatability (CV_r)	2,48%
Reproducibility SD (S_R)	0,191
Reproducibility (CV_R)	7,91%
Target range:	
Target standard deviation σ_{pt}	**0,190**
Target standard deviation (for Information)	0,0847
lower limit of target range	**2,04**
upper limit of target range	**2,80**
Quotient S/σ_{pt}*	*0,85*
Standard uncertainty U(X_{pt})	*0,0637*
Quotient U(X_{pt})/σ_{pt}	*0,33*
Results in the target range	*9*
Percent in the target range	*90%*
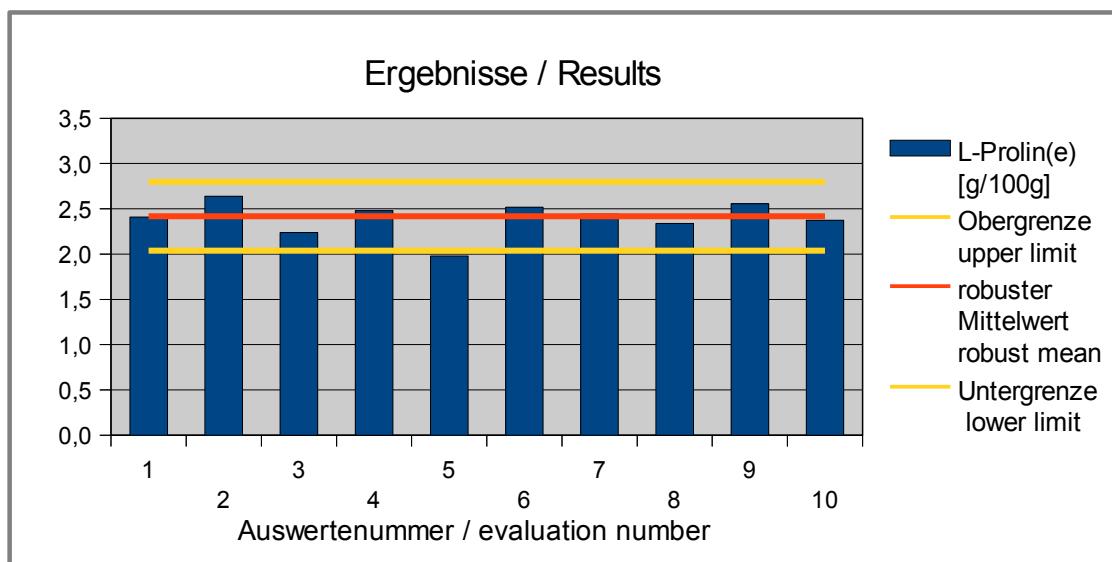
Abb. / Fig. 26: Ergebnisse L-Prolin/ Results L-Proline

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	L-Prolin / L-Proline [g/100g]	Abweichung [g/100g] Deviation [g/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	2,41	−0,0080	0,0	−0,1	
2	2,64	0,2220	1,2	2,6	
3	2,24	−0,1780	−0,9	−2,1	
4	2,48	0,0620	0,3	0,7	
5	1,98	−0,4380	−2,3	−5,2	
6	2,52	0,1020	0,5	1,2	
7	2,44	0,0231	0,1	0,3	
8	2,34	−0,0780	−0,4	−0,9	
9	2,56	0,1400	0,7	1,7	
10	2,38	−0,0430	−0,2	−0,5	
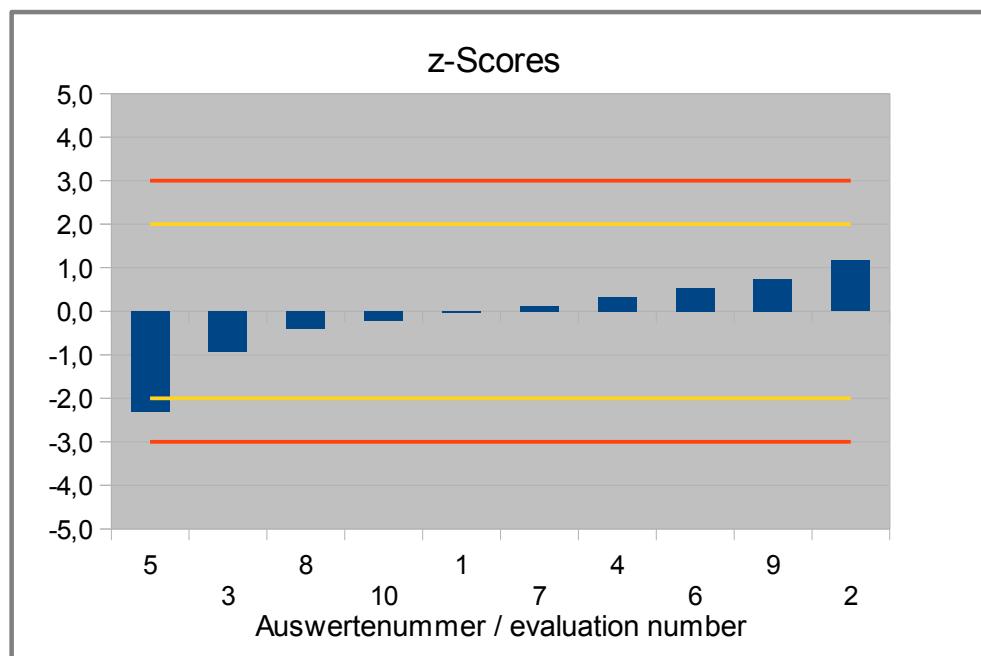
Abb. / Fig. 27:   Z-Scores L-Prolin / L-Proline

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

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	10
Number of outliers	0
Mean	2,24
Median	2,21
Robust Mean (X)	2,23
Robust standard deviation (S*)	0,110
Number with 2 replicates	9
Repeatability SD (S$_r$)	0,0252
Repeatability (CV$_r$)	1,13%
Reproducibility SD (S$_R$)	0,115
Reproducibility (CV$_R$)	5,16%
Target range:	
Target standard deviation σ_{pt}	0,0791
Target standard deviation (for Information)	0,0992
lower limit of target range	2,07
upper limit of target range	2,39
Quotient S*/σ_{pt}	1,39
Standard uncertainty U$_{(Xpt)}$	0,0436
Quotient U$_{(Xpt)}$/σ_{pt}	0,55
Results in the target range	8
Percent in the target range	80%
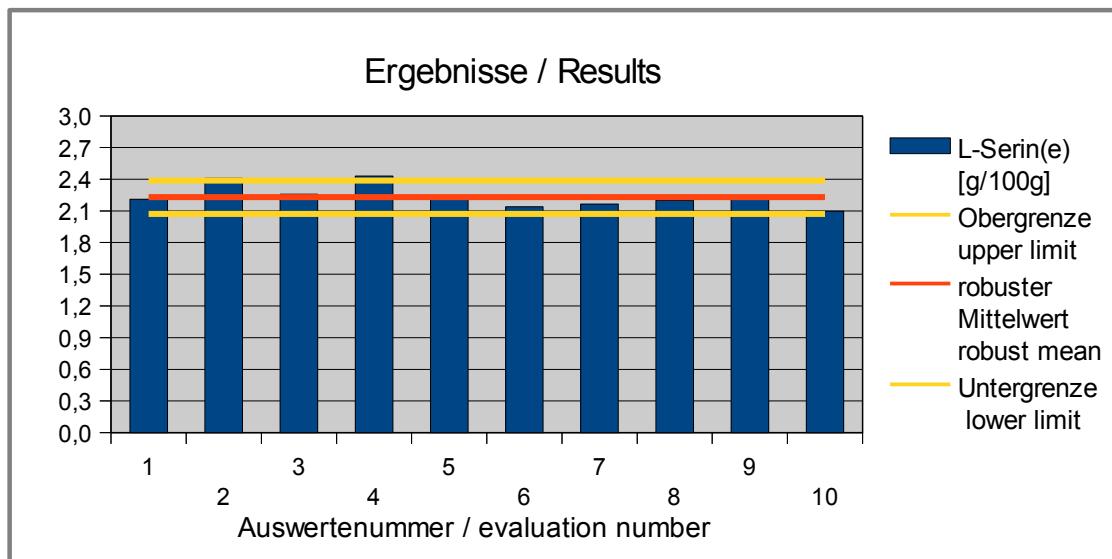
Abb. / Fig. 28: Ergebnisse L-Serin**/** Results L-Serine

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte- nummer Evaluation number	L-Serin / L-Serine [g/100g]	Abweichung [g/100g] Deviation [g/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	2,21	−0,0204	−0,3	−0,2	
2	2,41	0,1796	2,3	1,8	
3	2,26	0,0296	0,4	0,3	
4	2,43	0,1996	2,5	2,0	
5	2,21	−0,0204	−0,3	−0,2	
6	2,14	−0,0904	−1,1	−0,9	
7	2,17	−0,0652	−0,8	−0,7	
8	2,20	−0,0304	−0,4	−0,3	
9	2,24	0,0056	0,1	0,1	
10	2,10	−0,1324	−1,7	−1,3	
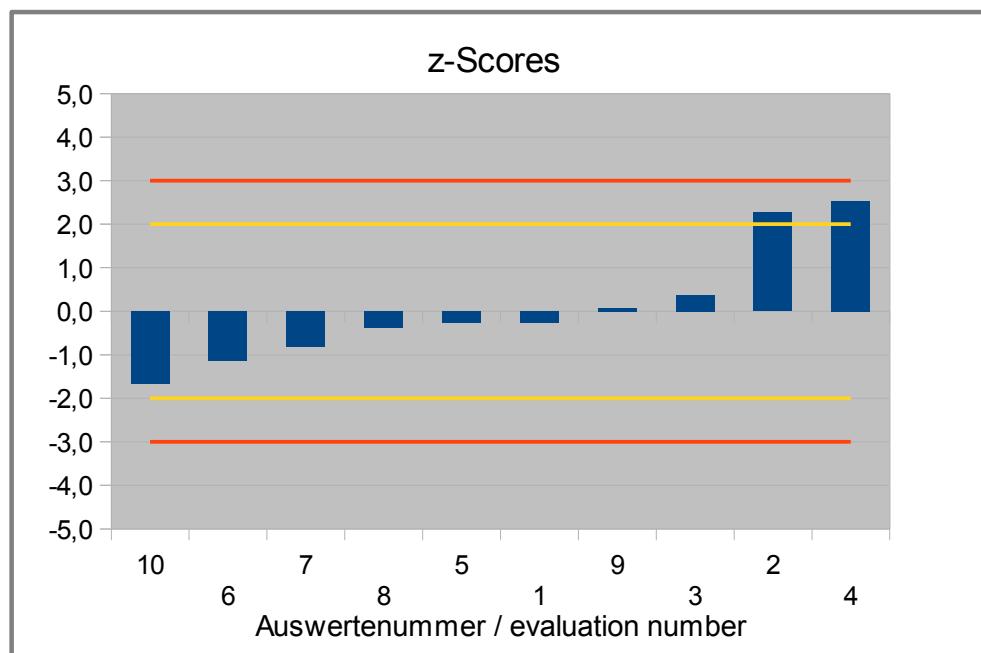
Abb. / Fig. 29:   Z-Scores L-Serin/ L-Serine

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

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	10
Number of outliers	0
Mean	1,59
Median	1,61
Robust Mean (X)	**1,60**
Robust standard deviation (S*)	**0,0914**
Number with 2 replicates	9
Repeatability SD (S_r)	0,0256
Repeatability (CV_r)	1,61%
Reproducibility SD (S_R)	0,106
Reproducibility (CV_R)	6,71%
Target range:	
Target standard deviation σ_{pt}	**0,0900**
Target standard deviation (for Information)	0,0595
lower limit of target range	**1,42**
upper limit of target range	**1,78**
Quotient S*/σ_{pt}	*1,02*
Standard uncertainty U(X_{pt})	*0,0361*
Quotient U(X_{pt})/σ_{pt}	*0,40*
Results in the target range	*9*
Percent in the target range	*90%*
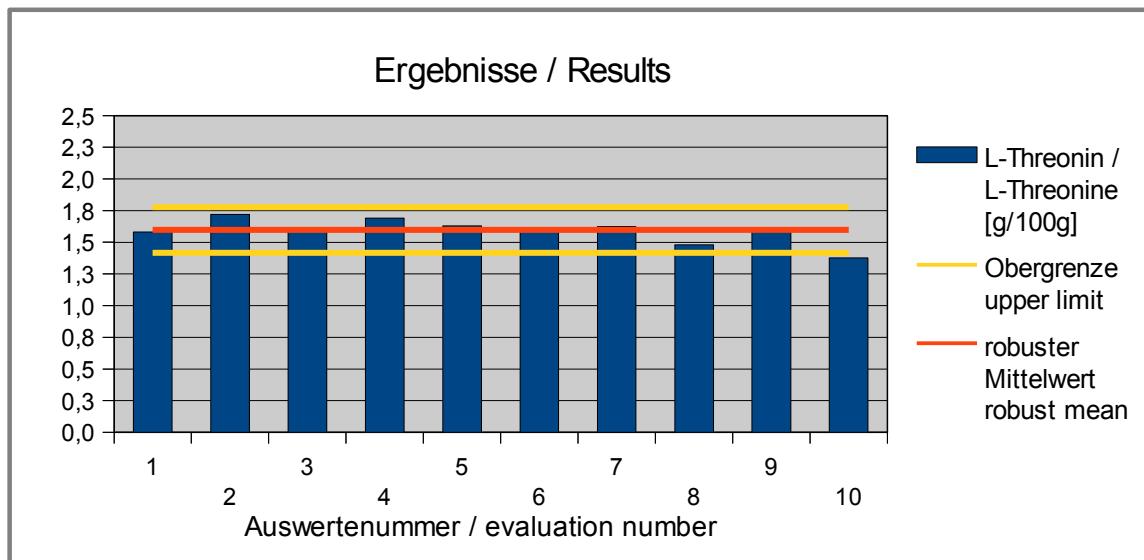
Abb. / Fig. 30: Ergebnisse L-Threonin/ Results L-Threonine

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	L-Threonin / L-Threonine [g/100g]	Abweichung [g/100g] Deviation [g/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	1,58	−0,0175	−0,2	−0,3	
2	1,72	0,1225	1,4	2,1	
3	1,61	0,0125	0,1	0,2	
4	1,69	0,0925	1,0	1,6	
5	1,63	0,0325	0,4	0,5	
6	1,60	0,0025	0,0	0,0	
7	1,62	0,0263	0,3	0,4	
8	1,48	−0,1175	−1,3	−2,0	
9	1,58	−0,0175	−0,2	−0,3	
10	1,38	−0,2195	−2,4	−3,7	
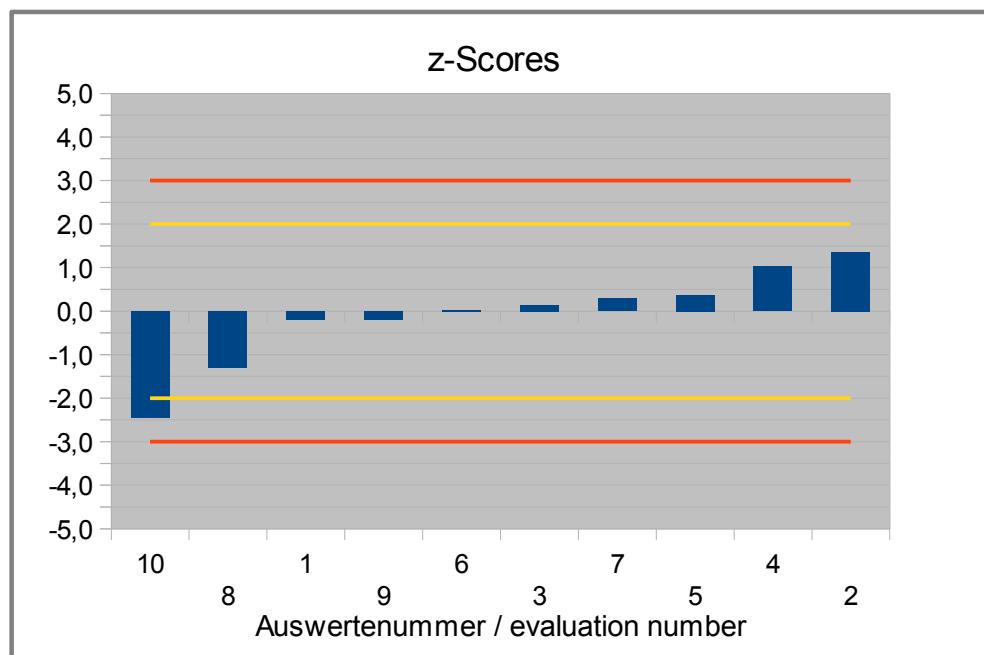
Abb. / Fig. 31:   Z-Scores L-Threonin / L-Threonine

4.17 L-Tryptophan in g/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	7
Number of outliers	0
Mean	0,570
Median	0,570
Robust Mean (X)	**0,563**
Robust standard deviation (S*)	**0,0590**
Number with 2 replicates	6
Repeatability SD (S_r)	0,0315
Repeatability (CV_r)	5,56%
Reproducibility SD (S_R)	0,0764
Reproducibility (CV_R)	13,5%
Target range:	
Target standard deviation σ_{pt}	**0,0395**
Target standard deviation (for Information)	0,0245
lower limit of target range	**0,48**
upper limit of target range	**0,64**
Quotient S*/σ_{pt}	*1,49*
Standard uncertainty U(Xpt)	*0,0279*
Quotient U(Xpt)/σ_{pt}	*0,71*
Results in the target range	*6*
Percent in the target range	*86%*
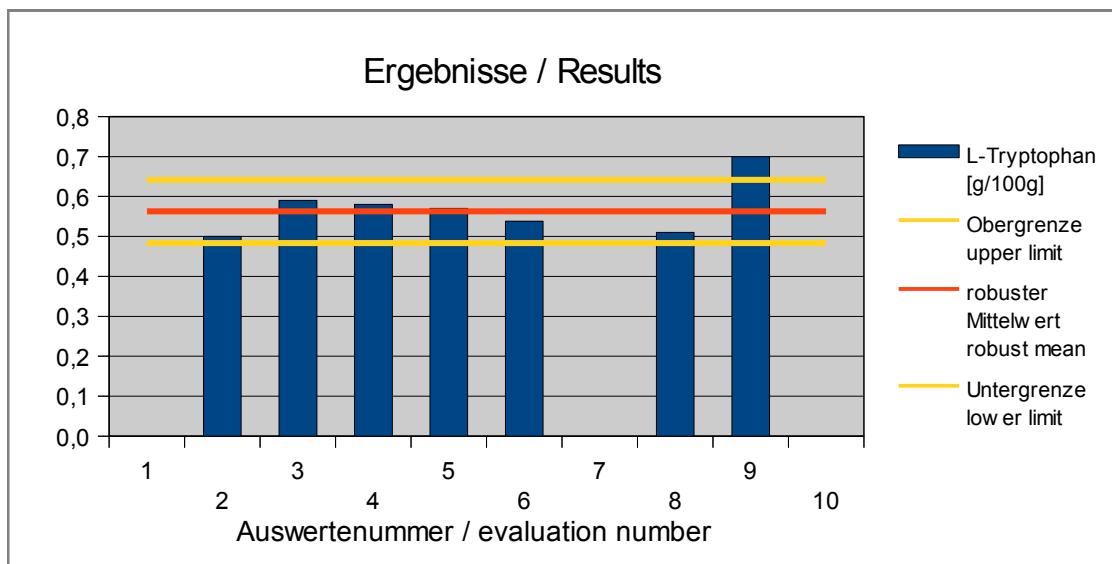
Abb. / Fig. 32: Ergebnisse L-Tryptophan **/** Results L-Tryptophan

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	L-Tryptophan [g/100g]	Abweichung [g/100g] Deviation [g/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1					
2	0,500	-0,0627	-1,6	-2,6	
3	0,590	0,0273	0,7	1,1	
4	0,580	0,0173	0,4	0,7	
5	0,570	0,0073	0,2	0,3	
6	0,538	-0,0247	-0,6	-1,0	
7					
8	0,510	-0,0527	-1,3	-2,1	
9	0,700	0,1373	3,5	5,6	
10					
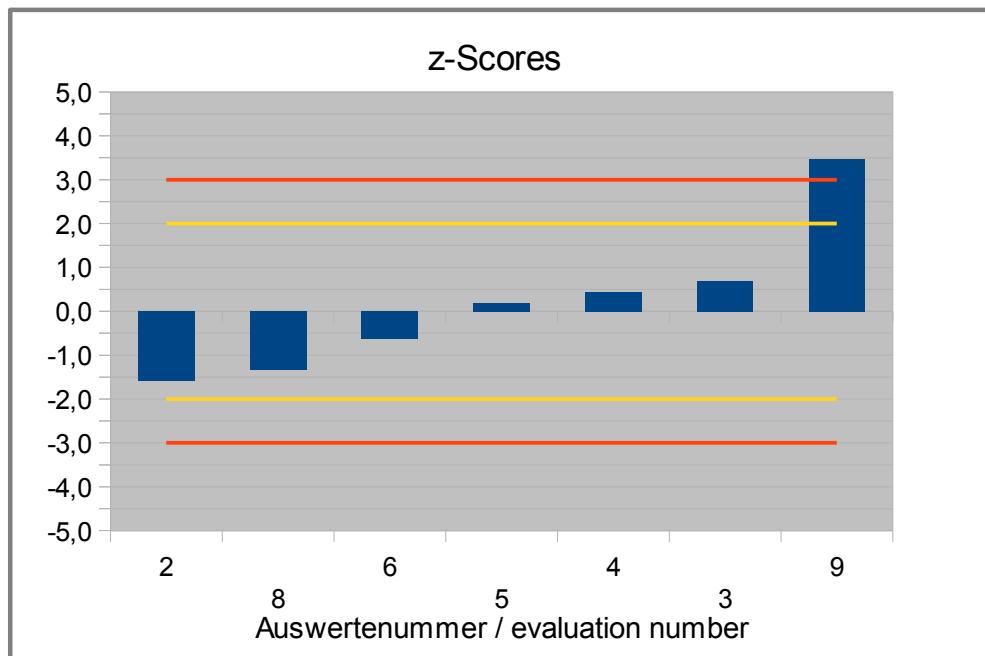
Abb. / Fig. 33:   Z-Scores L-Tryptophan

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

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	9
Number of outliers	0
Mean	1,55
Median	1,61
Robust Mean (X)	**1,56**
Robust standard deviation (S*)	**0,166**
Number with 2 replicates	8
Repeatability SD (S$_r$)	0,0254
Repeatability (CV$_r$)	1,66%
Reproducibility SD (S$_R$)	0,154
Reproducibility (CV$_R$)	10,0%
Target range:	
Target standard deviation σ_{pt}	**0,103**
Target standard deviation (for Information)	0,0583
lower limit of target range	**1,35**
upper limit of target range	**1,77**
Quotient S*/σ_{pt}	*1,61*
Standard uncertainty U$_{(Xpt)}$	*0,0693*
Quotient U$_{(Xpt)}$/σ_{pt}	*0,67*
Results in the target range	*8*
Percent in the target range	*89%*
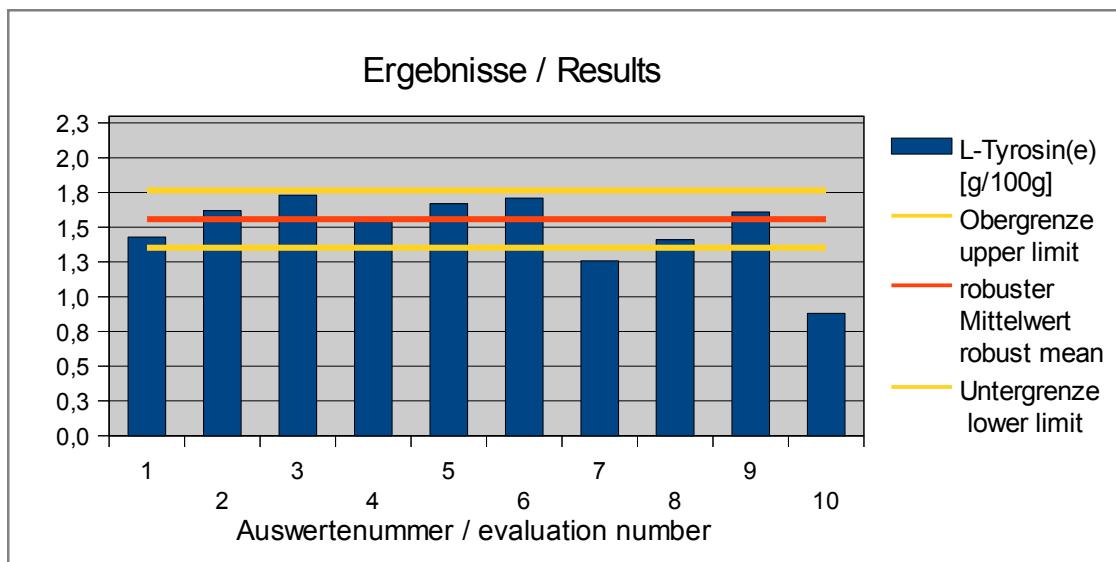
**Abb. / Fig. 34:** Ergebnisse L-Tyrosin**/** Results L-Tyrosine

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer / Evaluation number	L-Tyrosin / L-Tyrosine [g/100g]	Abweichung [g/100g] Deviation [g/100g]	z-Score (σpt)	z-Score (Info)	Hinweis Remark
1	1,43	−0,1287	−1,2	−2,2	
2	1,62	0,0613	0,6	1,1	
3	1,73	0,1713	1,7	2,9	
4	1,54	−0,0187	−0,2	−0,3	
5	1,67	0,1113	1,1	1,9	
6	1,71	0,1513	1,5	2,6	
7	1,26	−0,3001	−2,9	−5,1	
8	1,41	−0,1487	−1,4	−2,5	
9	1,61	0,0503	0,5	0,9	
10	0,88	−0,6400			Ausreisser ausgeschlossen* / Outlier excluded*

* Einfluss auf robuste Statistik / * influence on robust statistics
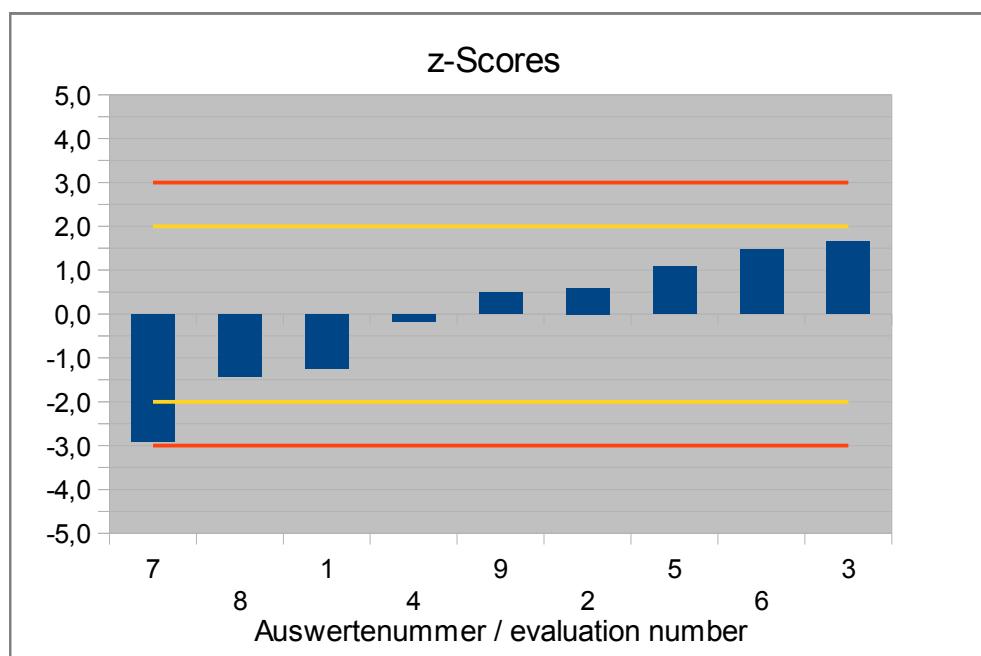
Abb. / Fig. 35:   Z-Scores L-Tyrosin/ L-Tyrosine

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

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	10
Number of outliers	0
Mean	2,06
Median	2,09
Robust Mean (X)	**2,06**
Robust standard deviation (S*)	**0,197**
Number with 2 replicates	9
Repeatability SD (S$_r$)	0,134
Repeatability (CV$_r$)	6,71%
Reproducibility SD (S$_R$)	0,164
Reproducibility (CV$_R$)	8,21%
Target range:	
Target standard deviation σ_{pt}	**0,121**
Target standard deviation (for Information)	0,0738
lower limit of target range	**1,82**
upper limit of target range	**2,30**
Quotient S*/σ_{pt}	*1,63*
Standard uncertainty U$_{(Xpt)}$	*0,0779*
Quotient U$_{(Xpt)}$/σ_{pt}	*0,64*
Results in the target range	*9*
Percent in the target range	*90%*
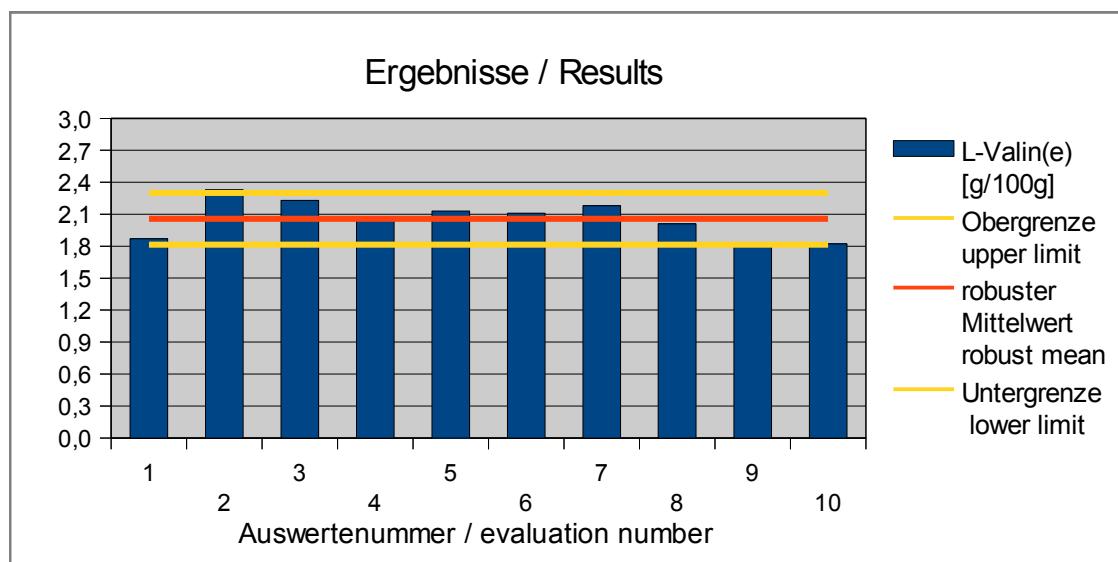
Abb. / Fig. 36: Ergebnisse L-Valin**/** Results L-Valine

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	L-Valin / L-Valine [g/100g]	Abweichung [g/100g] Deviation [g/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	1,87	−0,1872	−1,5	−2,5	
2	2,33	0,2728	2,3	3,7	
3	2,23	0,1728	1,4	2,3	
4	2,06	0,0028	0,0	0,0	
5	2,13	0,0728	0,6	1,0	
6	2,11	0,0528	0,4	0,7	
7	2,18	0,1232	1,0	1,7	
8	2,01	−0,0472	−0,4	−0,6	
9	1,83	−0,2282	−1,9	−3,1	
10	1,82	−0,2342	−1,9	−3,2	
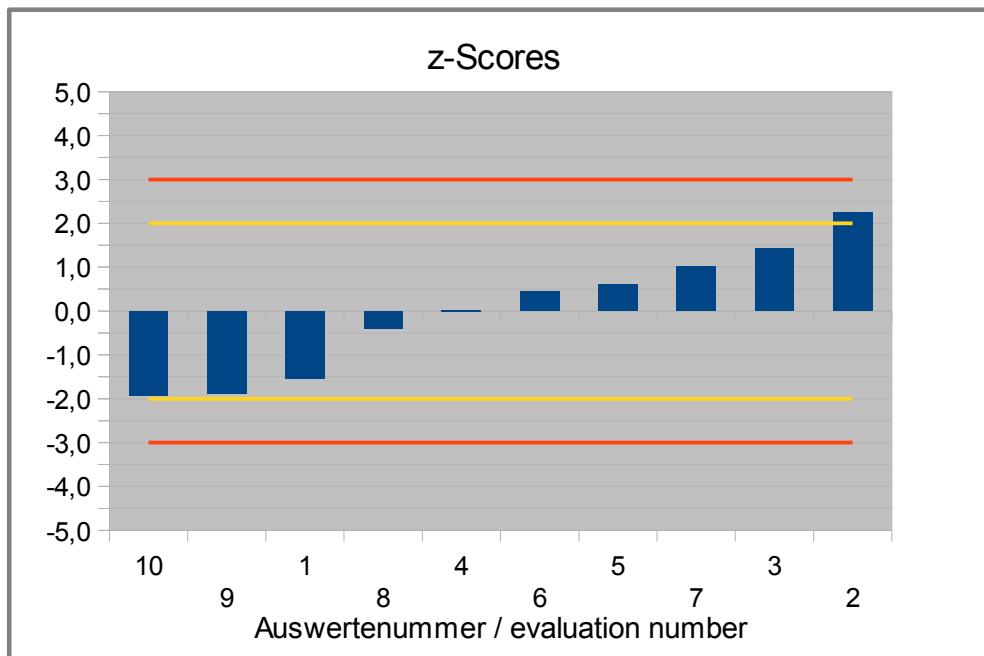
Abb. / Fig. 37:   Z-Scores L-Valin/ L-Valine

5. Documentation

5.1 Details by participants
5.1.1 Primary data

Parameter Analyte	Teilnehmer Participant	Einheit Unit	Proben-Nr.1 Sample No.1	Proben-Nr.2 Sample No.2	Datum d. Analyse Date of analysis	Ergebnis (Mittel) Result (Mean)	Ergebnis 1 Result 1	Ergebnis 2 Result 2	inkl. WF incl. RR	Wiederfindungsrate [%] Recovery rate [%]	WF mit gleicher Matrix bestimmt RR determined with identic matrix
L-Alanin(e)	1	g/100g	29	57	26.09.	1,66	1,69	1,63	no		
	2	g/100g	24	48		1,77	1,76	1,77	no		
	3	g/100g	5	46		1,82	1,82	1,82	no		
	4	g/100g	17	59	04.10.2016	1,69	1,69	1,68	N/A	N/A	N/A
	5	g/100g	19	31	26.09.2016	1,66	1,64	1,682	no	x	no
	6	g/100g	16	54	26.09.2016	1,7	1,7	1,7	no		
	7	g/100g	8	43	01.10.2016	1,728	1,741	1,715	no		
	8	g/100g	2	34	19.10.2016	1,63	1,657	1,600	no		
	9	g/100g	10	36	07.10.2016	1,687	1,712	1,662	no		
	10	g/100g	25	60	20.09.	1,659	1,674	1,644	no		
L-Arginin(e)	1	g/100g	29	57	26.09.	2,67	2,71	2,63	no		
	2	g/100g	24	48		3,02	3,00	3,04	no		
	3	g/100g	5	46		2,88	2,88	2,88	no		
	4	g/100g	17	59	04.10.2016	2,79	2,79	2,78	N/A	N/A	N/A
	5	g/100g	19	31	26.09.2016	2,60	2,577	2,633	no	x	no
	6	g/100g	16	54	26.09.2016	2,87	2,93	2,8	no		
	7	g/100g	8	43	01.10.2016	2,710	2,725	2,696	no		
	8	g/100g	2	34	19.10.2016	2,48	2,444	2,522	no		
	9	g/100g	10	36	07.10.2016	2,818	2,846	2,789	no		
	10	g/100g	25	60	20.09.	2,349	2,311	2,387	no		
L-Asparaginsäure L-Aspartic acid	1	g/100g	29	57	26.09.	4,50	4,64	4,35	no		
	2	g/100g	24	48		4,83	4,80	4,86	no		
	3	g/100g	5	46		4,59	4,59	4,59	no		
	4	g/100g	17	59	04.10.2016	4,48	4,49	4,47	N/A	N/A	N/A
	5	g/100g	19	31	26.09.2016	3,92	3,876	3,961	no	x	no
	6	g/100g	16	54	26.09.2016	4,53	4,54	4,51	no		
	7	g/100g	8	43	01.10.2016	4,664	4,708	4,620	no		
	8	g/100g	2	34	19.10.2016	4,47	4,444	4,490	no		
	9	g/100g	10	36	07.10.2016	4,586	4,665	4,507	no		
	10	g/100g	25	60	20.09.	3,547	3,581	3,513	no		
L-Cystein(e)	1	g/100g	29	57							
	2	g/100g	24	48							
	3	g/100g	5	46							
	4	g/100g	17	59	04.10.2016	N/A	N/A	N/A	N/A	N/A	N/A
	5	g/100g	19	31	x	x	x	x	x	x	x
	6	g/100g	16	54							
	7	g/100g	8	43	01.10.2016				no		
	8	g/100g	2	34	19.10.2016	0,52	0,539	0,499	no		
	9	g/100g	10	36	07.10.2016	0,443	0,462	0,424	no		
	10	g/100g	25	60							

Parameter Analyte	Teilnehmer Participant	Einheit Unit	Proben-Nr.1 Sample No.1	Proben-Nr.2 Sample No.2	Datum d. Analyse Date of analysis	Ergebnis (Mittel) Result (Mean)	Ergebnis 1 Result 1	Ergebnis 2 Result 2	inkl. WF incl. RR	Wiederfindungsrate [%] Recovery rate [%]	WF mit gleicher Matrix bestimmt RR determined with identic matrix
L-Cystine	1	g/100g	29	57	04.10.	0,41	0,40	0,41	no		
L-Cystine	2	g/100g	24	48		0,71	0,70	0,72	no		
L-Cystine	3	g/100g	5	46		0,52	0,52	0,52	no		
L-Cystine	4	g/100g	17	59	04.10.2016	0,46	0,46	0,45	N/A	N/A	N/A
L-Cystine	5	g/100g	19	31	26.09.2016	0,46	0,452	0,475	no	x	no
L-Cystine	6	g/100g	16	54	17.10.2016	0,485	0,483	0,486	no		
L-Cystine	7	g/100g	8	43	01.10.2016				no		
L-Cystine	8	g/100g	2	34	19.10.2016						
L-Cystine	9	g/100g	10	36	07.10.2016	0,590	0,604	0,576	no		
L-Cystine	10	g/100g	25	60	20.09.	0,260	0,27	0,25	no		
L-Glutaminc acid	1	g/100g	29	57	26.09.	8,09	8,33	7,84	no		
L-Glutaminc acid	2	g/100g	24	48		9,02	9,08	9,10	no		
L-Glutaminc acid	3	g/100g	5	46		8,67	8,67	8,67	no		
L-Glutaminc acid	4	g/100g	17	59	04.10.2016	7,98	8	7,95	N/A	N/A	N/A
L-Glutaminc acid	5	g/100g	19	31	26.09.2016	7,69	7,653	7,733	no	x	no
L-Glutaminc acid	6	g/100g	16	54	26.09.2016	8,18	8,19	8,16	no		
L-Glutaminc acid	7	g/100g	8	43	01.10.2016	8,768	8,807	8,728	no		
L-Glutaminc acid	8	g/100g	2	34	19.10.2016	8,62	8,598	8,637	no		
L-Glutaminc acid	9	g/100g	10	36	07.10.2016	8,061	8,191	7,930	no		
L-Glutaminc acid	10	g/100g	25	60	20.09.	7,859	7,942	7,775	no		
Glycine	1	g/100g	29	57	26.09.	1,49	1,51	1,46	no		
Glycine	2	g/100g	24	48		1,66	1,65	1,66	no		
Glycine	3	g/100g	5	46		1,60	1,6	1,6	no		
Glycine	4	g/100g	17	59	04.10.2016	1,57	1,57	1,56	N/A	N/A	N/A
Glycine	5	g/100g	19	31	26.09.2016	1,57	1,568	1,579	no	x	no
Glycine	6	g/100g	16	54	26.09.2016	1,56	1,57	1,55	no		
Glycine	7	g/100g	8	43	01.10.2016	1,573	1,585	1,560	no		
Glycine	8	g/100g	2	34	19.10.2016	1,40	1,405	1,391	no		
Glycine	9	g/100g	10	36	07.10.2016	1,544	1,565	1,522	no		
Glycine	10	g/100g	25	60	20.09.	1,407	1,429	1,384	no		
L-Histidine	1	g/100g	29	57	26.09.	1,01	1,03	0,98	no		
L-Histidine	2	g/100g	24	48		1,21	1,20	1,21	no		
L-Histidine	4	g/100g	17	59	04.10.2016	1,03	1,03	1,02	N/A	N/A	N/A
L-Histidine	3	g/100g	5	46		1,22	1,22	1,22	no		
L-Histidine	5	g/100g	19	31	26.09.2016	1,19	1,176	1,206	no	x	no
L-Histidine	6	g/100g	16	54	26.09.2016	1,08	1,09	1,07	no		
L-Histidine	7	g/100g	8	43	01.10.2016	1,057	1,071	1,042	no		
L-Histidine	8	g/100g	2	34	19.10.2016	1,04	1,028	1,041	no		
L-Histidine	9	g/100g	10	36	07.10.2016	1,110	1,141	1,080	no		
L-Histidine	10	g/100g	25	60	20.09.	1,029	1,03	1,027	no		

Parameter / Analyte	Teilnehmer / Participant	Einheit / Unit	Proben-Nr.1 / Sample No.1	Proben-Nr.2 / Sample No.2	Datum d. Analyse / Date of analysis	Ergebnis (Mittel) / Result (Mean)	Ergebnis 1 / Result 1	Ergebnis 2 / Result 2	inkl. WF / incl. RR	Wiederfindungsrate [%] / Recovery rate [%]	WF mit gleicher Matrix bestimmt / RR determined with identic matrix
L-Isoleucine	1	g/100g	29	57	26.09.	1,79	1,83	1,75	no		
L-Isoleucine	2	g/100g	24	48		2,08	2,07	2,08	no		
L-Isoleucine	3	g/100g	5	46		1,96	1,96	1,96	no		
L-Isoleucine	4	g/100g	17	59	04.10.2016	1,97	1,98	1,96	N/A	N/A	N/A
L-Isoleucine	5	g/100g	19	31	26.09.2016	2,11	2,099	2,113	no	x	no
L-Isoleucine	6	g/100g	16	54	26.09.2016	2,09	2,11	2,06	no		
L-Isoleucine	7	g/100g	8	43	01.10.2016	2,044	2,073	2,014	no		
L-Isoleucine	8	g/100g	2	34	19.10.2016	1,93	1,938	1,916	no		
L-Isoleucine	9	g/100g	10	36	07.10.2016	1,747	1,766	1,727	no		
L-Isoleucine	10	g/100g	25	60	20.09.	1,361	1,405	1,317	no		
L-Leucine	1	g/100g	29	57	26.09.	3,30	3,35	3,24	no		
L-Leucine	2	g/100g	24	48		3,72	3,69	3,70	no		
L-Leucine	3	g/100g	5	46		3,63	3,63	3,63	no		
L-Leucine	4	g/100g	17	59	04.10.2016	3,35	3,36	3,33	N/A	N/A	N/A
L-Leucine	5	g/100g	19	31	26.09.2016	3,81	3,778	3,832	no	x	no
L-Leucine	6	g/100g	16	54	26.09.2016	3,5	3,53	3,47	no		
L-Leucine	7	g/100g	8	43	01.10.2016	3,433	3,474	3,393	no		
L-Leucine	8	g/100g	2	34	19.10.2016	3,39	3,431	3,339	no		
L-Leucine	9	g/100g	10	36	07.10.2016	3,307	3,353	3,261	no		
L-Leucine	10	g/100g	25	60	20.09.	3,032	3,08	2,984	no		
L-Lysine	1	g/100g	29	57	26.09.	2,59	2,63	2,54	no		
L-Lysine	2	g/100g	24	48		2,86	2,85	2,87	no		
L-Lysine	3	g/100g	5	46		2,81	2,81	2,81	no		
L-Lysine	4	g/100g	17	59	04.10.2016	2,89	2,9	2,88	N/A	N/A	N/A
L-Lysine	5	g/100g	19	31	26.09.2016	2,76	2,699	2,813	no	x	no
L-Lysine	6	g/100g	16	54	26.09.2016	2,84	2,82	2,85	no		
L-Lysine	7	g/100g	8	43	01.10.2016	2,704	2,733	2,676	no		
L-Lysine	8	g/100g	2	34	19.10.2016	2,55	2,564	2,532	no		
L-Lysine	9	g/100g	10	36	07.10.2016	2,608	2,656	2,559	no		
L-Lysine	10	g/100g	25	60	20.09.	2,458	2,481	2,435	no		
L-Methionine	1	g/100g	29	57	26.09.	0,59	0,60	0,58	no		
L-Methionine	2	g/100g	24	48		0,60	0,60	0,59	no		
L-Methionine	3	g/100g	5	46		0,65	0,65	0,65	no		
L-Methionine	4	g/100g	17	59	04.10.2016	0,58	0,57	0,58	N/A	N/A	N/A
L-Methionine	5	g/100g	19	31	26.09.2016	0,56	0,549	0,569	no	x	no
L-Methionine	6	g/100g	16	54	17.10.2016	0,648	0,629	0,667	no		
L-Methionine	7	g/100g	8	43	01.10.2016	0,524	0,527	0,521	no		
L-Methionine	8	g/100g	2	34	19.10.2016	0,41	0,412	0,400	no		
L-Methionine	9	g/100g	10	36	07.10.2016	0,559	0,566	0,551	no		
L-Methionine	10	g/100g	25	60	20.09.	0,528	0,563	0,492	no		

Parameter / Analyte	Teilnehmer / Participant	Einheit / Unit	Proben-Nr.1 / Sample No.1	Proben-Nr.2 / Sample No.2	Datum d. Analyse / Date of analysis	Ergebnis (Mittel) / Result (Mean)	Ergebnis 1 / Result 1	Ergebnis 2 / Result 2	inkl. WF incl. RR	Wiederfindungsrate [%] / Recovery rate [%]	WF mit gleicher Matrix bestimmt / RR determined with identic matrix
L-Phenylalanine	1	g/100g	29	57	26.09.	2,03	2,07	1,98	no		
L-Phenylalanine	2	g/100g	24	48		2,31	2,29	2,32	no		
L-Phenylalanine	3	g/100g	5	46		2,12	2,12	2,12	no		
L-Phenylalanine	4	g/100g	17	59	04.10.2016	2,09	2,09	2,08	N/A	N/A	N/A
L-Phenylalanine	5	g/100g	19	31	26.09.2016	2,49	2,462	2,513	no	x	no
L-Phenylalanine	6	g/100g	16	54	26.09.2016	2,21	2,22	2,2	no		
L-Phenylalanine	7	g/100g	8	43	01.10.2016	2,155	2,182	2,127	no		
L-Phenylalanine	8	g/100g	2	34	19.10.2016	2,16	2,159	2,160	no		
L-Phenylalanine	9	g/100g	10	36	07.10.2016	2,083	2,107	2,059	no		
L-Phenylalanine	10	g/100g	25	60	20.09.	1,862	1,897	1,827	no		
L-Proline	1	g/100g	29	57	26.09.	2,41	2,46	2,36	no		
L-Proline	2	g/100g	24	48		2,64	2,60	2,67	no		
L-Proline	3	g/100g	5	46		2,24	2,24	2,24	no		
L-Proline	4	g/100g	17	59	04.10.2016	2,48	2,45	2,51	N/A	N/A	N/A
L-Proline	5	g/100g	19	31	26.09.2016	1,98	2,002	1,965	no	x	no
L-Proline	6	g/100g	16	54	26.09.2016	2,52	2,52	2,51	no		
L-Proline	7	g/100g	8	43	01.10.2016	2,441	2,474	2,408	no		
L-Proline	8	g/100g	2	34	19.10.2016	2,34	2,389	2,289	no		
L-Proline	9	g/100g	10	36	07.10.2016	2,558	2,516	2,599	no		
L-Proline	10	g/100g	25	60	20.09.	2,375	2,298	2,451	no		
L-Serine	1	g/100g	29	57	26.09.	2,21	2,22	2,20	no		
L-Serine	2	g/100g	24	48		2,41	2,39	2,43	no		
L-Serine	3	g/100g	5	46		2,26	2,26	2,26	no		
L-Serine	4	g/100g	17	59	04.10.2016	2,43	2,44	2,42	N/A	N/A	N/A
L-Serine	5	g/100g	19	31	26.09.2016	2,21	2,199	2,215	no	x	no
L-Serine	6	g/100g	16	54	26.09.2016	2,14	2,13	2,15	no		
L-Serine	7	g/100g	8	43	01.10.2016	2,165	2,175	2,155	no		
L-Serine	8	g/100g	2	34	19.10.2016	2,20	2,197	2,209	no		
L-Serine	9	g/100g	10	36	07.10.2016	2,236	2,279	2,194	no		
L-Serine	10	g/100g	25	60	20.09.	2,098	2,11	2,086	no		
L-Threonine	1	g/100g	29	57	26.09.	1,58	1,61	1,55	no		
L-Threonine	2	g/100g	24	48		1,72	1,71	1,73	no		
L-Threonine	3	g/100g	5	46		1,61	1,61	1,61	no		
L-Threonine	4	g/100g	17	59	04.10.2016	1,69	1,7	1,68	N/A	N/A	N/A
L-Threonine	5	g/100g	19	31	26.09.2016	1,63	1,616	1,649	no	x	no
L-Threonine	6	g/100g	16	54	26.09.2016	1,6	1,59	1,6	no		
L-Threonine	7	g/100g	8	43	01.10.2016	1,624	1,633	1,615	no		
L-Threonine	8	g/100g	2	34	19.10.2016	1,48	1,475	1,477	no		
L-Threonine	9	g/100g	10	36	07.10.2016	1,580	1,618	1,542	no		
L-Threonine	10	g/100g	25	60	20.09.	1,378	1,381	1,374	no		

Parameter Analyte	Teilnehmer Participant	Einheit Unit	Proben-Nr.1 Sample No.1	Proben-Nr.2 Sample No.2	Datum d. Analyse Date of analysis	Ergebnis (Mittel) Result (Mean)	Ergebnis 1 Result 1	Ergebnis 2 Result 2	inkl. WF incl. RR	Wiederfindungsrate [%] Recovery rate [%]	WF mit gleicher Matrix bestimmt RR determined with identic matrix
L-Tryptophan	1	g/100g	29	57							
L-Tryptophan	2	g/100g	24	48		0,5	0,49	0,50	no		
L-Tryptophan	3	g/100g	5	46		0,59	0,59	0,59	no		
L-Tryptophan	4	g/100g	17	59	04.10.2016	0,58	0,58	0,58	N/A	N/A	N/A
L-Tryptophan	5	g/100g	19	31	29.09.2016	0,57	0,573	0,564	no	x	no
L-Tryptophan	6	g/100g	16	54	12.10.2016	0,538	0,534	0,541	no		
L-Tryptophan	7	g/100g	8	43	01.10.2016				no		
L-Tryptophan	8	g/100g	2	34	19.10.2016	0,51	0,513	0,515	no		
L-Tryptophan	9	g/100g	10	36	07.10.2016	0,700	0,754	0,646	no		
L-Tryptophan	10	g/100g	25	60							
L-Tyrosine	1	g/100g	29	57	26.09.	1,43	1,46	1,39	no		
L-Tyrosine	2	g/100g	24	48		1,62	1,60	1,63	no		
L-Tyrosine	3	g/100g	5	46		1,73	1,73	1,73	no		
L-Tyrosine	4	g/100g	17	59	04.10.2016	1,54	1,54	1,53	N/A	N/A	N/A
L-Tyrosine	5	g/100g	19	31	26.09.2016	1,67	1,649	1,697	no	x	no
L-Tyrosine	6	g/100g	16	54	26.09.2016	1,71	1,71	1,7	no		
L-Tyrosine	7	g/100g	8	43	01.10.2016	1,259	1,247	1,271	no		
L-Tyrosine	8	g/100g	2	34	19.10.2016	1,41	1,420	1,406	no		
L-Tyrosine	9	g/100g	10	36	07.10.2016	1,609	1,626	1,591	no		
L-Tyrosine	10	g/100g	25	60	20.09.	0,880	0,829	0,93	no		
L-Valine	1	g/100g	29	57	26.09.	1,87	1,90	1,83	no		
L-Valine	2	2,33	24	48		2,33	2,32	1,76	no		
L-Valine	3	g/100g	5	46		2,23	2,23	2,23	no		
L-Valine	4	g/100g	17	59	04.10.2016	2,06	2,07	2,05	N/A	N/A	N/A
L-Valine	5	g/100g	19	31	26.09.2016	2,13	2,122	2,136	no	x	no
L-Valine	6	g/100g	16	54	26.09.2016	2,11	2,13	2,08	no		
L-Valine	7	g/100g	8	43	01.10.2016	2,180	2,196	2,165	no		
L-Valine	8	g/100g	2	34	19.10.2016	2,01	2,011	1,998	no		
L-Valine	9	g/100g	10	36	07.10.2016	1,829	1,856	1,803	no		
L-Valine	10	g/100g	25	60	20.09.	1,823	1,82	1,825	no		

5.1.2 Analytical methods

Parameter	Teilnehmer	Methodenbeschreibung	Homogen-isierung	Einwaage	Aufschluss-methode	Derivatisierung	Referenzmaterial	Kalibrierverfahren	Methode ist akkreditiert	sonstige Hinweise
Analyte	Participant	Method descripton	Homogeni-zation	Sample weight	Hydrolization	Derivatisation	Reference material	Calibration	Method accredited	Further remarks
L-Alanine	1	Amino acid analyzer (post column de-rivatisation)		appr. 100 mg/50 ml, for Cystine appr. 200 mg/50 ml	Hydrolysis with HCl (c = 6 mol/l)	Ninhydrin staining	Standard solution for physiolo-gial liquids from Sykam, Aspar-tic acid from Fluka	external standard	yes	
L-Alanine	2								yes	
L-Alanine	3	VO (EG) 152/2009 Annex III F	mixing	0,25 g	acidic hydrolysis	-		one point calibration with internal standard norleucine	yes	
L-Alanine	4	AM/V/206	N/A	0.5g	7h 115°C	yes	Internal Reference Material, Cer-tified refernce Material	yes	Yes	The above Arginine result should be considered as approximation if the amino acid Taurine is present within this sample
L-Alanine	5	§64 LFGB L 49.07-2		0,5g	HCL, 6 mol/l	Ninhydrin	VDLUFA-Enq.	blank method, external standard	yes	sample material agglutinated strongly during oxidation, therefore loss of cystine and methionine could not be excluded
L-Alanine	6	Determination of amino acids after acidic hydrolysis (24 h, 6 N HCl, 110 °C) of sample material by amino acid analyzer (SOP AS 04).	none	0,1 g	saure Hydrolyse (24 h, 6 N HCl, 110 °C)	post column deriva-tisation by ninhydrin	Amino Acids Mix Solution, Sig-ma-Aldrich 79248	one point calibration	yes	
L-Alanine	7	Total amino acids after acidic hydroly-sis by amino acid analyzer (post co-lumn derivatisation)	mortal	500 mg	acidic hydrolysis by 6 M HCl, 24 h at 110°C	Ninhydrin	milk protein	External standard, one point calibration	yes	
L-Alanine	8	Determination by amino acid analyzer (according to ASU L 49.07-1 and L49.07-2)		0,5 g	Hydrolysis, Oxida-tion			external; 5-point calibra-tion	no	value of L-Cysteine corresponds to sum of L-Cysteine and L-Cystinen
L-Alanine	9	VDLUFA III 4.11.1	overhead shaking	0,25g	Oxidation/ Hydro-lysis	post column deriva-tisation by ninhydrin		linear	yes	
L-Alanine	10	ASU §64 LFGB L49.07-2/ mod.		0,2g		post column deriva-tisation by ninhydrin		internal standard	yes	

Parameter	Teilnehmer	Methodenbeschreibung	Homogen-isierung	Einwaage	Aufschluss-me-thode	Derivatisierung	Referenzmaterial	Kalibrierverfahren	Methode ist akkreditiert	sonstige Hinweise
Analyte	Participant	Method descripton	Homogeni-zation	Sample weight	Hydrolization	Derivatisation	Reference material	Calibration	Method ac-credited	Further remarks
L-Arginine	1	Amino acid analyzer (post column de-rivatisation)								
L-Arginine	2									
L-Arginine	3	VO (EG) 152/2009 Annex III F								
L-Arginine	4	AM/V/206								
L-Arginine	5	§64 LFGB L 49.07-2								
L-Arginine	6	Determination of amino acids after acidic hydrolysis (24 h, 6 N HCl, 110 °C) of sample material by amino acid analyzer (SOP AS 04).								
L-Arginine	7	Total amino acids after acidic hydroly-sis by amino acid analyzer (post co-lumn derivatisation)								
L-Arginine	8	Determination by amino acid analyzer (according to ASU L 49.07-1 and L49.07-2)								
L-Arginine	9	VDLUFA III 4.11.1								
L-Arginine	10	ASU §64 LFGB L49.07-2/ mod.		0,2g		post column deriva-tisation by ninhydrin		internal standard	yes	
L-Aspartic acid	1	Amino acid analyzer (post column de-rivatisation)								
L-Aspartic acid	2									
L-Aspartic acid	3	VO (EG) 152/2009 Annex III F								
L-Aspartic acid	4	AM/V/206								
L-Aspartic acid	5	§64 LFGB L 49.07-2								
L-Aspartic acid	6	Determination of amino acids after acidic hydrolysis (24 h, 6 N HCl, 110 °C) of sample material by amino acid analyzer (SOP AS 04).								Sum Asp + Asn
L-Aspartic acid	7	Total amino acids after acidic hydroly-sis by amino acid analyzer (post co-lumn derivatisation)								
L-Aspartic acid	8	Determination by amino acid analyzer (according to ASU L 49.07-1 and L49.07-2)								
L-Aspartic acid	9	VDLUFA III 4.11.1								
L-Aspartic acid	10	ASU §64 LFGB L49.07-2/ mod.		0,2g		post column deriva-tisation by ninhydrin		internal standard	yes	

Parameter	Teilnehmer	Methodenbeschreibung	Homogen-isierung	Einwaage	Aufschluss-me-thode	Derivatisierung	Referenzmaterial	Kalibrierverfahren	Methode ist akkreditiert	sonstige Hinweise
Analyte	Participant	Method descripton	Homogeni-zation	Sample weight	Hydrolization	Derivatisation	Reference material	Calibration	Method ac-credited	Further remarks
L-Cysteine	1	Amino acid analyzer (post column de-rivatisation)								
L-Cysteine	2									
L-Cysteine	3	VO (EG) 152/2009 Annex III F								
L-Cysteine	4	AM/V/206								
L-Cysteine	5	§64 LFGB L 49.07-2								
L-Cysteine	6	Determination of amino acids after acidic hydrolysis (24 h, 6 N HCl, 110 °C) of sample material by amino acid analyzer (SOP AS 04).								
L-Cysteine	7	Total amino acids after acidic hydroly-sis by amino acid analyzer (post co-lumn derivatisation)								
L-Cysteine	8	Determination by amino acid analyzer (according to ASU L 49.07-1 and L49.07-2)								
L-Cysteine	9	VDLUFA III 4.11.1								
L-Cysteine	10	ASU §64 LFGB L49.07-2/ mod.		0,2g		post column deriva-tisation by ninhydrin		internal standard	yes	
L-Cystine	1	Amino acid analyzer (post column de-rivatisation)								
L-Cystine	2									
L-Cystine	3	VO (EG) 152/2009 Annex III F								
L-Cystine	4	AM/V/206								
L-Cystine	5	§64 LFGB L 49.07-2								
L-Cystine	6	Determination of Cystine and Methio-nine after per-formic acid treatment of sample material (15h) followed by 24 h hydrolysis with 6 N HCl at 110 °C by amino acid analyzator (SOP AS 05).	none	0,2 g	see method des-cription	post column deriva-tisation by ninhydrin	single reference	one point calibration	yes	Sum Cystein + Cystin
L-Cystine	7	Total amino acids after acidic hydroly-sis by amino acid analyzer (post co-lumn derivatisation)								
L-Cystine	8	Determination by amino acid analyzer (according to ASU L 49.07-1 and L49.07-2)								
L-Cystine	9	VDLUFA III 4.11.1								
L-Cystine	10	ASU §64 LFGB L49.07-2/ mod.		0,2g		post column deriva-tisation by ninhydrin		internal standard	yes	

Parameter	Teilnehmer	Methodenbeschreibung	Homogen-isierung	Einwaage	Aufschluss-me-thode	Derivatisierung	Referenzmaterial	Kalibrierverfahren	Methode ist akkreditiert	sonstige Hinweise
Analyte	Participant	Method descripton	Homogeni-zation	Sample weight	Hydrolization	Derivatisation	Reference material	Calibration	Method ac-credited	Further remarks
L-Glutamic acid	1	Amino acid analyzer (post column de-								
L-Glutamic acid	2									
L-Glutamic acid	3	VO (EG) 152/2009 Annex III F								
L-Glutamic acid	4	AM/V/206								
L-Glutamic acid	5	§64 LFGB L 49.07-2								
L-Glutamic acid	6	Determination of amino acids after acidic hydrolysis (24 h, 6 N HCl, 110 °C) of sample material by amino acid analyzer (SOP AS 04).								Sum Glu + Gln
L-Glutamic acid	7	Total amino acids after acidic hydroly-sis by amino acid analyzer (post co-lumn derivatisation)								
L-Glutamic acid	8	Determination by amino acid analyzer (according to ASU L 49.07-1 and L49.07-2)								
L-Glutamic acid	9	VDLUFA III 4.11.1								
L-Glutamic acid	10	ASU §64 LFGB L49.07-2/ mod.		0,2g		post column deriva-tisation by ninhydrin		internal standard	yes	
Glycine	1	Amino acid analyzer (post column de-rivatisation)								
Glycine	2									
Glycine	3	VO (EG) 152/2009 Annex III F								
Glycine	4	AM/V/206								
Glycine	5	§64 LFGB L 49.07-2								
Glycine	6	Determination of amino acids after acidic hydrolysis (24 h, 6 N HCl, 110 °C) of sample material by amino acid analyzer (SOP AS 04).								
Glycine	7	Total amino acids after acidic hydroly-sis by amino acid analyzer (post co-lumn derivatisation)								
Glycine	8	Determination by amino acid analyzer (according to ASU L 49.07-1 and L49.07-2)								
Glycine	9	VDLUFA III 4.11.1								
Glycine	10	ASU §64 LFGB L49.07-2/ mod.		0,2g		post column deriva-tisation by ninhydrin		internal standard	yes	

Parameter Analyte	Teilnehmer Participant	Methodenbeschreibung Method descripton	Homogen- isierung Homogeni- zation	Einwaage Sample weight	Aufschluss-me- thode Hydrolization	Derivatisierung Derivatisation	Referenzmaterial Reference material	Kalibrierverfahren Calibration	Methode ist akkreditiert Method ac- credited	sonstige Hinweise Further remarks
L-Histidine	1	Amino acid analyzer (post column de-rivatisation)								
L-Histidine	2									
L-Histidine	3	VO (EG) 152/2009 Annex III F								
L-Histidine	4	AM/V/206								
L-Histidine	5	§64 LFGB L 49.07-2								
L-Histidine	6	Determination of amino acids after acidic hydrolysis (24 h, 6 N HCl, 110 °C) of sample material by amino acid analyzer (SOP AS 04).								
L-Histidine	7	Total amino acids after acidic hydroly-sis by amino acid analyzer (post co-lumn derivatisation)								
L-Histidine	8	Determination by amino acid analyzer (according to ASU L 49.07-1 and L49.07-2)								
L-Histidine	9	VDLUFA III 4.11.1								
L-Histidine	10	ASU §64 LFGB L49.07-2/ mod.		0,2g		post column deriva-tisation by ninhydrin		internal standard	yes	
L-Isoleucine	1	Amino acid analyzer (post column de-rivatisation)								
L-Isoleucine	2									
L-Isoleucine	3	VO (EG) 152/2009 Annex III F								
L-Isoleucine	4	AM/V/206								
L-Isoleucine	5	§64 LFGB L 49.07-2								
L-Isoleucine	6	Determination of amino acids after acidic hydrolysis (24 h, 6 N HCl, 110 °C) of sample material by amino acid analyzer (SOP AS 04).								
L-Isoleucine	7	Total amino acids after acidic hydroly-sis by amino acid analyzer (post co-lumn derivatisation)								
L-Isoleucine	8	Determination by amino acid analyzer (according to ASU L 49.07-1 and L49.07-2)								
L-Isoleucine	9	VDLUFA III 4.11.1								
L-Isoleucine	10	ASU §64 LFGB L49.07-2/ mod.		0,2g		post column deriva-tisation by ninhydrin		internal standard	yes	

Parameter	Teilnehmer	Methodenbeschreibung	Homogen-isierung	Einwaage	Aufschluss-me-thode	Derivatisierung	Referenzmaterial	Kalibrierverfahren	Methode ist akkreditiert	sonstige Hinweise
Analyte	Participant	Method descripton	Homogeni-zation	Sample weight	Hydrolization	Derivatisation	Reference material	Calibration	Method ac-credited	Further remarks
L-Leucine	1	Amino acid analyzer (post column de-rivatisation)								
L-Leucine	2									
L-Leucine	3	VO (EG) 152/2009 Annex III F								
L-Leucine	4	AMV/206								
L-Leucine	5	§64 LFGB L 49.07-2								
L-Leucine	6	Determination of amino acids after acidic hydrolysis (24 h, 6 N HCl, 110 °C) of sample material by amino acid analyzer (SOP AS 04).								
L-Leucine	7	Total amino acids after acidic hydroly-sis by amino acid analyzer (post co-lumn derivatisation)								
L-Leucine	8	Determination by amino acid analyzer (according to ASU L 49.07-1 and L49.07-2)								
L-Leucine	9	VDLUFA III 4.11.1								
L-Leucine	10	ASU §64 LFGB L49.07-2/ mod.		0,2g		post column deriva-tisation by ninhydrin		internal standard	yes	
L-Lysine	1	Amino acid analyzer (post column de-rivatisation)								
L-Lysine	2									
L-Lysine	3	VO (EG) 152/2009 Annex III F								
L-Lysine	4	AMV/206								
L-Lysine	5	§64 LFGB L 49.07-2								
L-Lysine	6	Determination of amino acids after acidic hydrolysis (24 h, 6 N HCl, 110 °C) of sample material by amino acid analyzer (SOP AS 04).								
L-Lysine	7	Total amino acids after acidic hydroly-sis by amino acid analyzer (post co-lumn derivatisation)								
L-Lysine	8	Determination by amino acid analyzer (according to ASU L 49.07-1 and L49.07-2)								
L-Lysine	9	VDLUFA III 4.11.1								
L-Lysine	10	ASU §64 LFGB L49.07-2/ mod.		0,2g		post column deriva-tisation by ninhydrin		internal standard	yes	

Parameter	Teilnehmer	Methodenbeschreibung	Homogen-isierung	Einwaage	Aufschluss-me-thode	Derivatisierung	Referenzmaterial	Kalibrierverfahren	Methode ist akkreditiert	sonstige Hinweise
Analyte	Participant	Method descripton	Homogeni-zation	Sample weight	Hydrolization	Derivatisation	Reference material	Calibration	Method ac-credited	Further remarks
L-Methionine	1	Amino acid analyzer (post column de-rivatisation)								
L-Methionine	2									
L-Methionine	3	VO (EG) 152/2009 Annex III F								
L-Methionine	4	AM/V/206								
L-Methionine	5	§64 LFGB L 49.07-2								
L-Methionine	6	Determination of Cystine and Methio-nine after per-formic acid treatment of sample material (15h) followed by 24 h hydrolysis with 6 N HCl at 110 °C by amino acid analyzator (SOP AS 05).	none	0,2 g	see method des-cription	post column deriva-tisation by ninhydrin	single reference	one point calibration	yes	
L-Methionine	7	Total amino acids after acidic hydroly-sis by amino acid analyzer (post co-lumn derivatisation)								
L-Methionine	8	Determination by amino acid analyzer								
L-Methionine	9	VDLUFA III 4.11.1								
L-Methionine	10	ASU §64 LFGB L49.07-2/ mod.		0,2g		post column deriva-tisation by ninhydrin		internal standard	yes	
L-Phenylalanine	1	Amino acid analyzer (post column de-rivatisation)								
L-Phenylalanine	2									
L-Phenylalanine	3	VO (EG) 152/2009 Annex III F								
L-Phenylalanine	4	AM/V/206								
L-Phenylalanine	5	§64 LFGB L 49.07-2								
L-Phenylalanine	6	Determination of amino acids after acidic hydrolysis (24 h, 6 N HCl, 110 °C) of sample material by amino acid analyzer (SOP AS 04).								
L-Phenylalanine	7	Total amino acids after acidic hydroly-sis by amino acid analyzer (post co-lumn derivatisation)								
L-Phenylalanine	8	Determination by amino acid analyzer (according to ASU L 49.07-1 and L49.07-2)								
L-Phenylalanine	9	VDLUFA III 4.11.1								
L-Phenylalanine	10	ASU §64 LFGB L49.07-2/ mod.		0,2g		post column deriva-tisation by ninhydrin		internal standard	yes	

Parameter	Teilnehmer	Methodenbeschreibung	Homogen-isierung	Einwaage	Aufschluss-me-thode	Derivatisierung	Referenzmaterial	Kalibrierverfahren	Methode ist akkreditiert	sonstige Hinweise
Analyte	Participant	Method descripton	Homogeni-zation	Sample weight	Hydrolization	Derivatisation	Reference material	Calibration	Method ac-credited	Further remarks
L-Proline	1	Amino acid analyzer (post column de-rivatisation)								
L-Proline	2									
L-Proline	3	VO (EG) 152/2009 Annex III F								
L-Proline	4	AM/V/206								
L-Proline	5	§64 LFGB L 49.07-2								
L-Proline	6	Determination of amino acids after acidic hydrolysis (24 h, 6 N HCl, 110 °C) of sample material by amino acid analyzer (SOP AS 04).								
L-Proline	7	Total amino acids after acidic hydroly-sis by amino acid analyzer (post co-lumn derivatisation)								
L-Proline	8	Determination by amino acid analyzer								
L-Proline	9	VDLUFA III 4.11.1								
L-Proline	10	ASU §64 LFGB L49.07-2/ mod.		0,2g		post column deriva-tisation by ninhydrin		internal standard	yes	
L-Serine	1	Amino acid analyzer (post column de-rivatisation)								
L-Serine	2									
L-Serine	3	VO (EG) 152/2009 Annex III F								
L-Serine	4	AM/V/206								
L-Serine	5	§64 LFGB L 49.07-2								
L-Serine	6	Determination of amino acids after acidic hydrolysis (24 h, 6 N HCl, 110 °C) of sample material by amino acid analyzer (SOP AS 04).								
L-Serine	7	Total amino acids after acidic hydroly-sis by amino acid analyzer (post co-lumn derivatisation)								
L-Serine	8	Determination by amino acid analyzer (according to ASU L 49.07-1 and L49.07-2)								
L-Serine	9	VDLUFA III 4.11.1								
L-Serine	10	ASU §64 LFGB L49.07-2/ mod.		0,2g		post column deriva-tisation by ninhydrin		internal standard	yes	

Parameter	Teilnehmer	Methodenbeschreibung	Homogen-isierung	Einwaage	Aufschluss-me-thode	Derivatisierung	Referenzmaterial	Kalibrierverfahren	Methode ist akkreditiert	sonstige Hinweise
Analyte	Participant	Method descripton	Homogeni-zation	Sample weight	Hydrolization	Derivatisation	Reference material	Calibration	Method ac-credited	Further remarks
L-Threonine	1	Amino acid analyzer (post column de-rivatisation)								
L-Threonine	2									
L-Threonine	3	VO (EG) 152/2009 Annex III F								
L-Threonine	4	AM/V/206								
L-Threonine	5	§64 LFGB L 49.07-2								
L-Threonine	6	Determination of amino acids after acidic hydrolysis (24 h, 6 N HCl, 110 °C) of sample material by amino acid analyzer (SOP AS 04).								
L-Threonine	7	Total amino acids after acidic hydroly-sis by amino acid analyzer (post co-lumn derivatisation)								
L-Threonine	8	Determination by amino acid analyzer (according to ASU L 49.07-1 and L49.07-2)								
L-Threonine	9	VDLUFA III 4.11.1								
L-Threonine	10	ASU §64 LFGB L49.07-2/ mod.		0,2g		post column deriva-tisation by ninhydrin		internal standard	yes	
L-Tryptophan	1	Amino acid analyzer (post column de-rivatisation)								
L-Tryptophan	2				4.2 N NaOH					
L-Tryptophan	3	VO (EG) 152/2009 Annex III G	mixing	0,25 g	alkalinie hydroly-sis	-		one point calibration with internal standard α-Methyl-Tryptophan	yes	
L-Tryptophan	4	AM/V/228	N/A	0.5g	7h 115°C	no	Internal and certified reference		yes	-
L-Tryptophan	5	VDLUFA III 4.11.2		0,5g	LiOH, 2 mol/l	x	VDLUFA-Enq.	blank method	yes	
L-Tryptophan	6	Tryptophan determined after alkaline hydrolysis (20 h, 5.6 N NaOH, 110 °C) of sample material by amino acid ana-lyzer (SOP AS 03).	none	0,3 g	alkaline hydrolysis (20 h, 5.6 N NaOH, 110 °C)	post column deriva-tisation by ninhydrin	single reference	one point calibration	yes	
L-Tryptophan	7	Total amino acids after acidic hydroly-sis by amino acid analyzer (post co-lumn derivatisation)								
L-Tryptophan	8	HPLC-Fluorescence		0,5 g	hydrolization			external; 5-point calibra-tion	yes	
L-Tryptophan	9	VDLUFA III 4.11.2	overhead shaking	0,5g	hydrolization	none		linear	no	
L-Tryptophan	10	ASU §64 LFGB L49.07-2/ mod.		0,2g		post column deriva-tisation by ninhydrin		internal standard	yes	

Parameter	Teilnehmer	Methodenbeschreibung	Homogen-isierung	Einwaage	Aufschluss-me-thode	Derivatisierung	Referenzmaterial	Kalibrierverfahren	Methode ist akkreditiert	sonstige Hinweise
Analyte	Participant	Method descripton	Homogeni-zation	Sample weight	Hydrolization	Derivatisation	Reference material	Calibration	Method ac-credited	Further remarks
L-Tyrosine	1	Amino acid analyzer (post column de-rivatisation)								
L-Tyrosine	2									
L-Tyrosine	3	VO (EG) 152/2009 Annex III F								
L-Tyrosine	4	AM/V/206								
L-Tyrosine	5	§64 LFGB L 49.07-2								
L-Tyrosine	6	Determination of amino acids after acidic hydrolysis (24 h, 6 N HCl, 110 °C) of sample material by amino acid analyzer (SOP AS 04).								
L-Tyrosine	7	Total amino acids after acidic hydroly-sis by amino acid analyzer (post co-lumn derivatisation)								
L-Tyrosine	8	Determination by amino acid analyzer (according to ASU L 49.07-1 and L49.07-2)								
L-Tyrosine	9	VDLUFA III 4.11.1								
L-Tyrosine	10	ASU §64 LFGB L49.07-2/ mod.		0,2g		post column deriva-tisation by ninhydrin		internal standard	yes	
L-Valine	1	Amino acid analyzer (post column de-rivatisation)								
L-Valine	2									
L-Valine	3	VO (EG) 152/2009 Annex III F								
L-Valine	4	AM/V/206								
L-Valine	5	§64 LFGB L 49.07-2								
L-Valine	6	Determination of amino acids after acidic hydrolysis (24 h, 6 N HCl, 110 °C) of sample material by amino acid analyzer (SOP AS 04).								
L-Valine	7	Total amino acids after acidic hydroly-sis by amino acid analyzer (post co-lumn derivatisation)								
L-Valine	8	Determination by amino acid analyzer (according to ASU L 49.07-1 and L49.07-2)								
L-Valine	9	VDLUFA III 4.11.1								
L-Valine	10	ASU §64 LFGB L49.07-2/ mod.		0,2g		post column deriva-tisation by ninhydrin		internal standard	yes	

5.2 Homogeneity

5.2.1 Mixture homogeneity before bottling

Microtracer Homogeneity Test
DLA 59-2016

Weight whole sample	1,51	kg
Microtracer	FSS-rot lake	
Particle size	75 – 300	µm
Weight per particle	2,0	µg
Addition of tracer	16,9	mg/kg

Result of analysis

Sample	Weight [g]	Particle number	Particles [mg/kg]
1	5,24	57	21,8
2	5,07	50	19,7
3	4,99	46	18,4
4	5,09	44	17,3
5	5,15	40	15,5
6	5,13	53	20,7
7	5,13	46	17,9
8	5,06	56	22,1

Poisson distribution		
Number of samples	8	
Degree of freedom	7	
Mean	49,0	Particles
Standard deviation	5,86	Particles
χ^2 (CHI-Quadrat)	4,90	
Probability	**67**	%
Recovery rate	113	%

Normal distribution		
Number of samples	8	
Mean	19,2	mg/kg
Standard deviation	2,29	mg/kg
rel. Standard deviaton	12,0	%
Horwitz standard deviation	10,3	%
HorRat-value	**1,2**	
Recovery rate	113	%

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-Alanine		
Target standard deviation σ_{pt}	0,0627	g/100 g
Sample numbers	2 - 60	
Total numbers of samples	18	
Slope	-0,00173	
Trend line range	1,702 - 1,734	g/100 g
Deviation trend line	1,718 ± 0,0156	g/100 g
Percent of σpt	24,8 %	
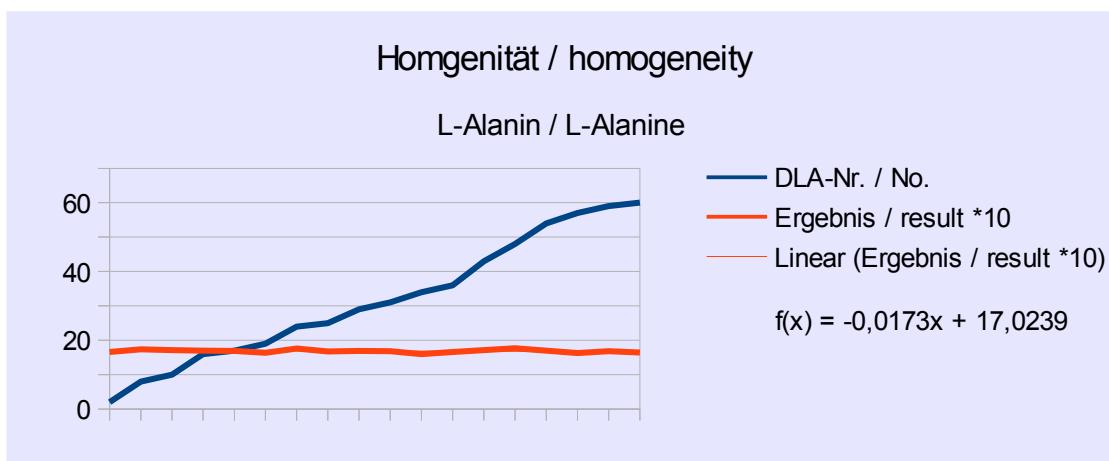
Abb./Fig. 38:
Trendfunktion Probennummern vs. Ergebnisse (*10 dargestellt)
trend line function sample number vs. results (*10 shown)

L-Methionine	without participant 8 (2 + 34)		
Target standard deviation σ_{pt}	0,0382		g/100 g
Sample numbers	2 – 60		
Total numbers of samples	16		
Slope	-0,000269		
Trend line range	0,5701 – 0,5744		g/100 g
Deviation trend line	0,5723 ± 0,0022		g/100 g
Percent of σpt	5,6 %		

Homgenität / homogeneity

L-Methionin / L-Methionine

Legend:
- DLA-Nr. / No.
- Ergebnis / result *100
- Linear (Ergebnis / result *100)

$f(x) = -0{,}0269x + 57{,}4438$
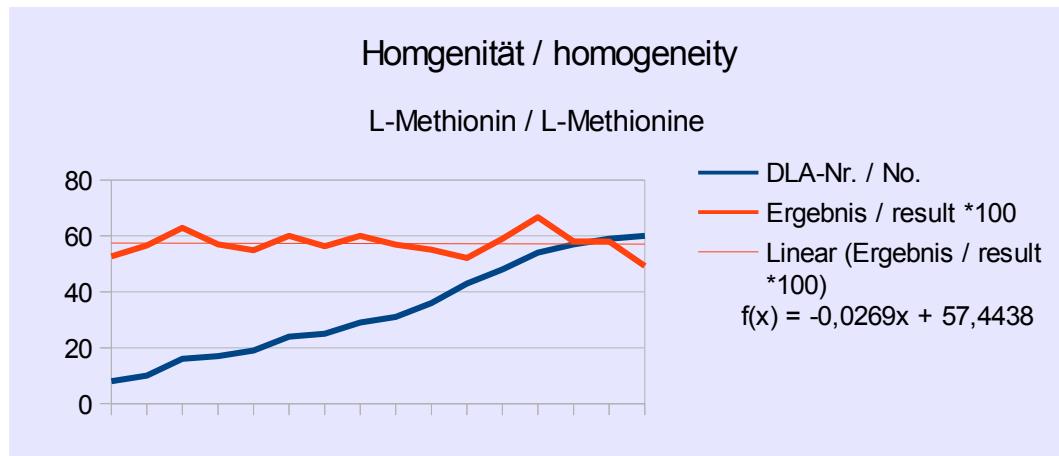
Abb./Fig. 39:
Trendfunktion Probennummern vs. Ergebnisse (*100 dargestellt)
trend line function sample number vs. results (*100 shown)

5.3 Kernel Density Plots of Results

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

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

L-Asparaginsäure/L-Aspartic acid

Kernel Density Plot
Fixed h: .1086

L-Alanin/L-Alanine
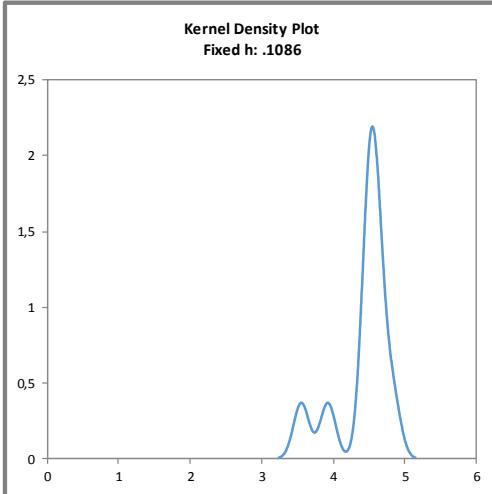
Kernel Density Plot
Fixed h: .04703
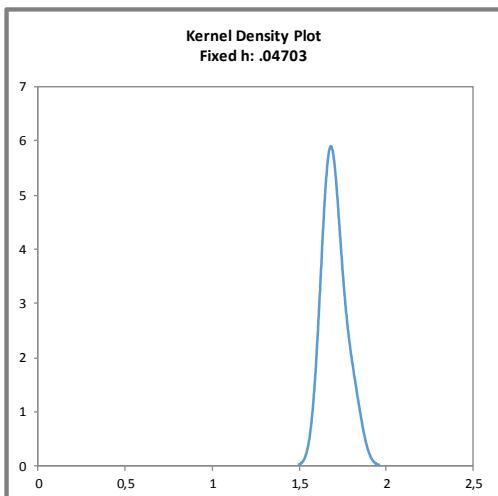
L-Cystein/L-Cysteine

<8 Ergebnisse

<8 Results

L-Arginin/L-Arginine
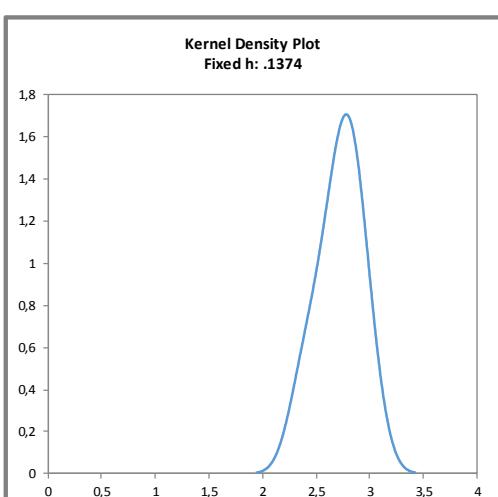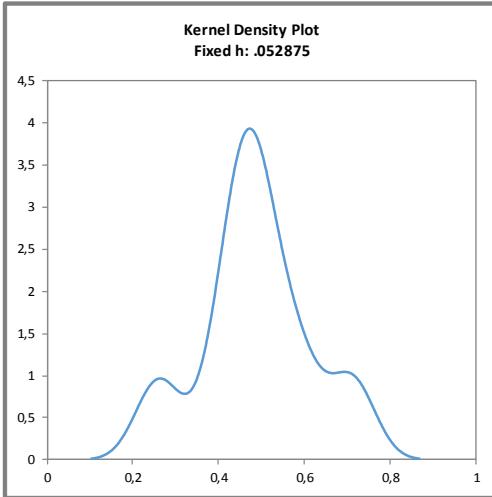
Kernel Density Plot
Fixed h: .1374

L-Cystin/L-Cystine

Kernel Density Plot
Fixed h: .052875

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

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

L-Histidin/L-Histidine
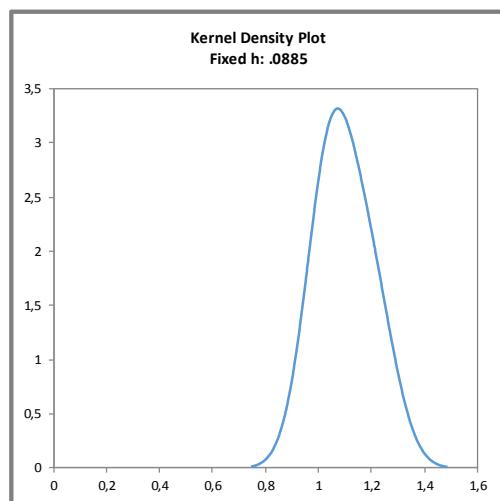
L-Glutaminsäure/L-Glutamic acid
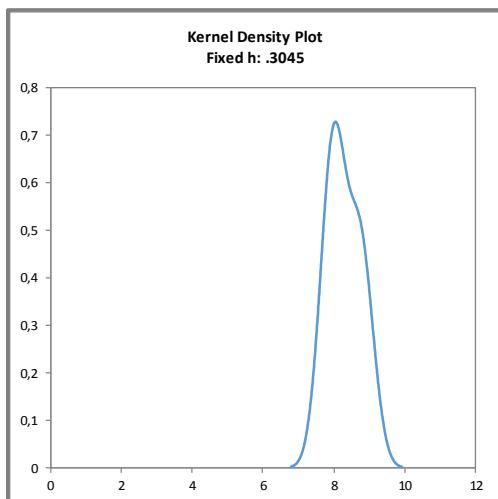
L-Isoleucin/L-Isoleucine
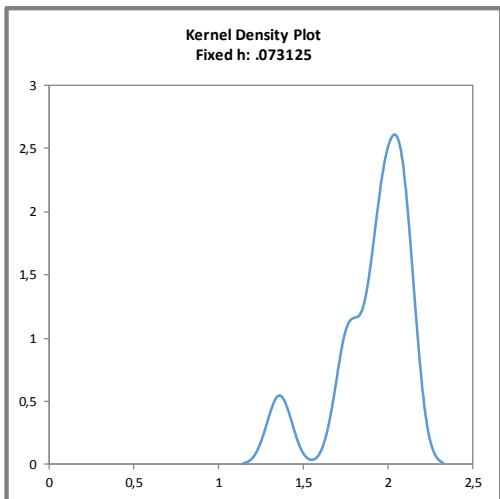
Glycin/Glycine
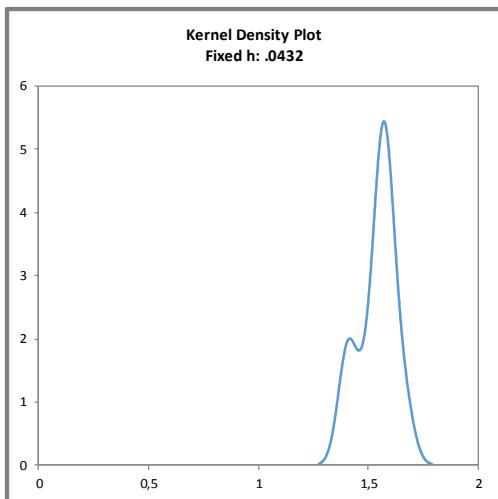
L-Leucin/L-Leucine
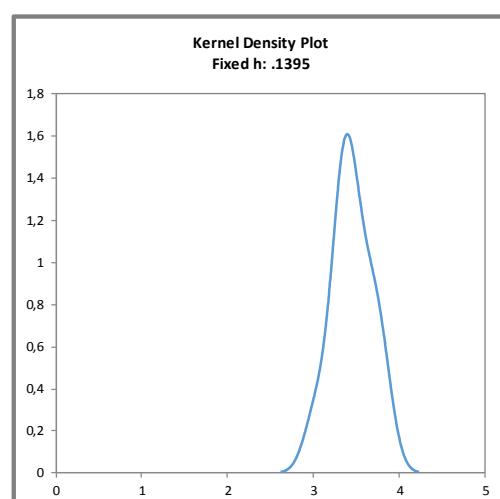
Abbildungen:
Kerndichte-Schätzungen
der Teilnehmerergebnisse
(mit h = 0,75 x σ_{pt} von Xpt)

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

L-Phenylalanin/L-Phenylalanine
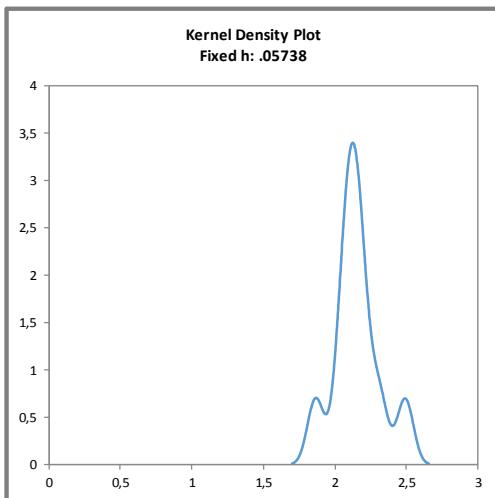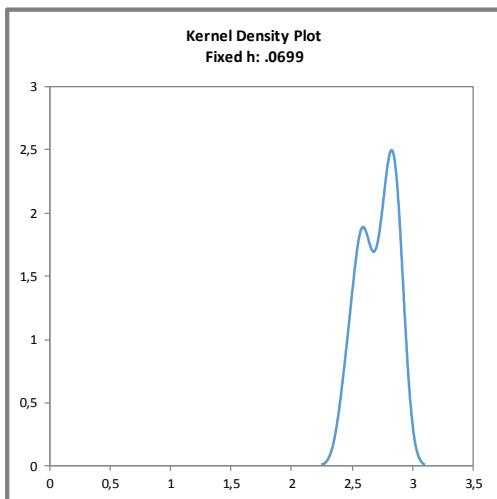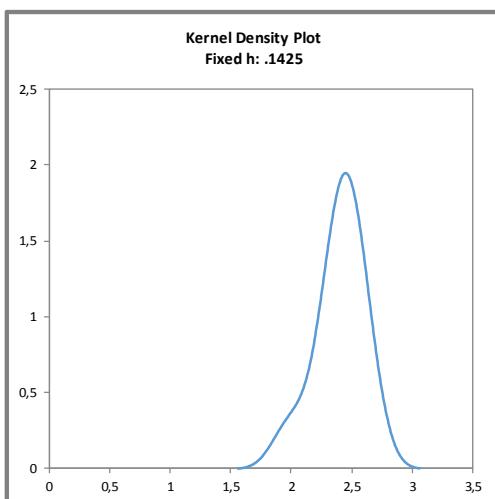
Kernel Density Plot
Fixed h: .05738

L-Lysin/L-Lysine

Kernel Density Plot
Fixed h: .0699

L-Prolin/L-Proline

Kernel Density Plot
Fixed h: .1425

L-Methionin/L-Methionine

Kernel Density Plot
Fixed h: .02865

L-Serin/L-Serine

Kernel Density Plot
Fixed h: .05933
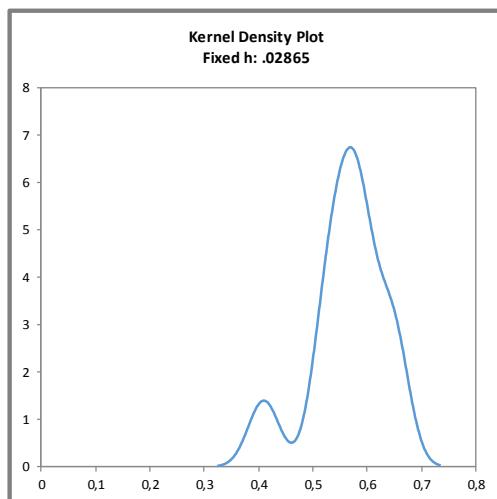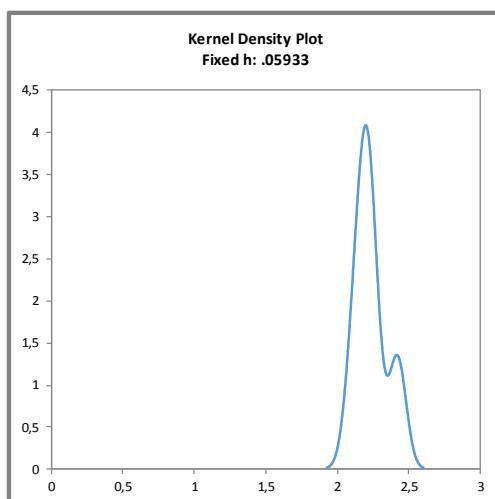
Abbildungen:
Kerndichte-Schätzungen
der Teilnehmerergebnisse
(mit h = 0,75 x σ_{pt} von X_{pt})

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

L-Tyrosin/L-Tyrosine

Kernel Density Plot
Fixed h: .07725

L-Threonin/L-Threonine

Kernel Density Plot
Fixed h: .04463

L-Valin/L-Valine

Kernel Density Plot
Fixed h: .09075
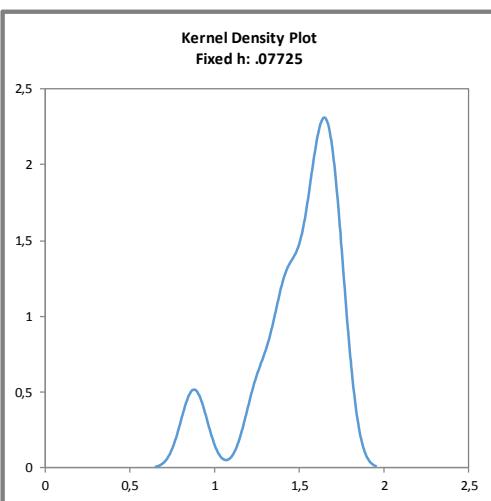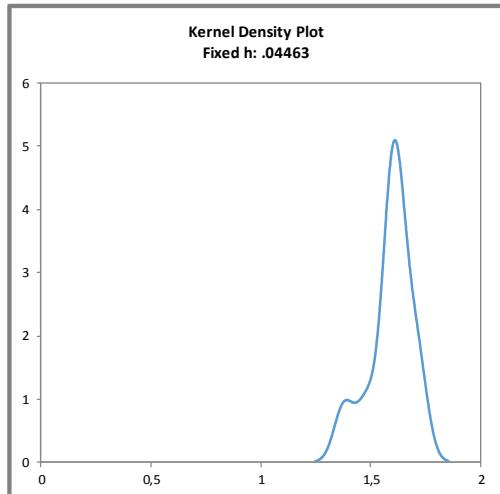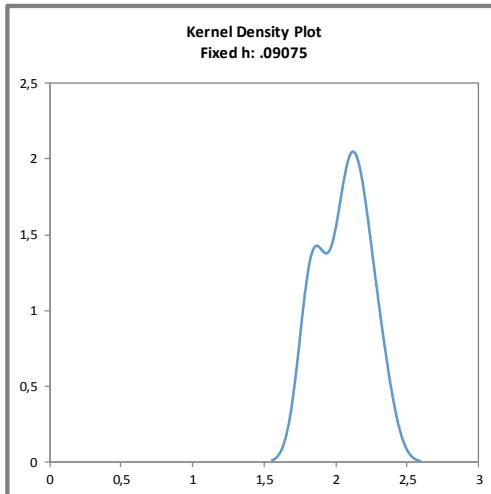
L-Tryptophan/L-Tryptophan

< 8 Ergebnisse
< 8 Results

6. Index of participant laboratories in alphabetical order

Teilnehmer / Participant	Ort / Town	Land / Country
		GREAT BRITAIN
		THAILAND
		Germany
		Germany
		Germany
		Germany
		Germany
		Germany
		Germany
		Germany

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

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

7. Index of references

1. DIN EN ISO/IEC 17025:2005; Allgemeine Anforderungen an die Kompetenz von Prüf- und Kalibrierlaboratorien / General requirements for the competence of testing and calibration laboratories
2. DIN EN ISO/IEC 17043:2010; Konformitätsbewertung – Allgemeine Anforderungen an Eignungsprüfungen / Conformity assessment – General requirements for proficiency testing
3. ISO 13528:2015 & DIN ISO 13528:2009; Statistische Verfahren für Eignungsprüfungen durch Ringversuche / Statistical methods for use in proficiency testing by interlaboratory comparisons
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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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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. ASU §64 LFGB L 49.07-1 Bestimmung der Aminosäuren in Aminosäurengemischen (1985) [Determination of amino acids in amino acid mixtures]
17. 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]
18. 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]
19. 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