Evaluation Report
proficiency test

DLA 50/2019

Free Amino Acids and Taurine:

in Infant Food

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

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

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1. Introduction

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

2. Realisation

2.1 Test material

The test material is a common in commerce dietetic food "infant milk powder, for the dietetic treatment of severe food allergies from birth" based on free amino acids with a protein equivalent content <20% from a European supplier.
The raw material was sieved (mesh 600 µm) and homogenized.

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

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

Table 1: Composition of DLA-Samples

Infant Milk Powder
Ingredients: Glucose syrup, vegetable oils , L-arginine, L-aspartate, L-leucine, L-lysine acetate, calcium orthophosphate, potassium citrate, L-glutamine, L-proline, emulsifier (citric acid ester of mono- and diglycerides of fatty acids), L-valine, glycine, L-isoleucine, L-threonine, L-phenylalanine, L-tyrosine, L-serine, L-histidine, L-alanine, sodium chloride, L-cystine, L-tryptophan, magnesium chloride, choline tartrate, L-methionine, magnesium-L-aspartate, calcium citrate, inositol, vitamin C, taurine, iron-II-sulfate, zinc sulfate, L-carnitine, nucleotides (sodium salts of uridine-, cytidine-, inosine-, adenosine-, guanosine-5-monophosphate), niacin, pantothenic acid, antioxidants (L-ascorbyl palmitate, alpha-tocopherol, extracts high in tocopherol), vitamin E, copper sulfate, vitamin B1, vitamin B6, vitamin B2, manganese II-sulfate, vitamin A, potassium iodide, folic acid, chromium chloride, vitamin K, sodium selenite, sodium molybdate, biotin, vitamin D, vitamin B12 Nutrients per 100 g: Fat 25 g, carbohydrates 53 g thereof sugar 4,7 g, fiber 0 g, protein 13 g, salt 0,5 g

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

2.1.1 Homogeneity

The calculation of the **repeatability standard deviation S_r of the participants** was used as an indicator of homogeneity. For 12 analytes it is < 5% (2,5% - 4,7%), for another 7 analytes in the range of 6,5% - 9,4% and for alanine at approx. 15% (s. Tab.2). Therefore the repeatability standard deviations are mostly similar to precision data of the referring standardized methods (e.g. ASU §64, s. 3.6.2) (see Tab. 3) [18-20]. The repeatability standard deviations of the participants' results are given in the tables of statistic data (see 4.1 to 4.21).

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

Parameter	CV_r	Parameter	CV_r
L-Alanine	14,9 %	L-Lysine	8,5 %
L-Arginine	3,3 %	L-Methionine	4,3 %
L-Aspartic acid	3,7 %	L-Phenylalanine	7,5 %
L-Cysteine	–	L-Proline	4,1 %
L-Cystine	6,5 %	L-Serine	4,3 %
L-Glutamine	4,7 %	L-Threonine	4,2 %
L-Glutamic acid	–	L-Tryptophan	2,6 %
Glycine	9,4 %	L-Tyrosine	2,5 %
L-Histidine	3,0 %	L-Valine	7,6 %
L-Isoleucine	3,5 %		
L-Leucine	3,1 %	Taurine	3,7 %

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

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

2.1.2 Stability

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

The experience with various DLA test materials showed good storage stability with respect to the durability of the sample (spoilage) and the content of the PT parameters for comparable food matrices and water activity (a_W value <0,5).
The a_W value of the EP samples was approx. 0,20 (23,6°C). The stability of the sample material was thus ensured during the investigation period under the specified storage conditions.

2.2 Sample shipment and information to the test

Two portions of test material were sent to every participating laboratory in the 36th week of 2019. The testing method was optional. The tests should be finished at 18th October 2019 the latest.

With the cover letter along with the sample shipment the following in-formation was given to participants:

The *two* portions contain *identical samples* of a *infant food (balanced diet)* with the parameters of free amino acids and taurine in the matrix of *powder for preparation of infant milk*. The protein equivalent content is < 20%. The analysis method is optional.

Please note the attached information on the proficiency test.
(see documentation, section 5.4 Information on the PT)

2.3 Submission of results

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

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

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

All 22 participants submitted their results in time.

3. Evaluation

3.1 Consensus value from participants (assigned value)

The robust mean of the submitted results was used as assigned value (X_{pt}) („consensus value from participants") providing a normal distribution. The calculation was done according to algorithm A as described in annex C of ISO 13528 [3]. If there are < 12 quantitative results and an increased difference between robust mean and median, the median may be used as the assigned value (criterion: Δ median - rob. mean > 0,3 σ_{pt}) [3].

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

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

The statistical evaluation is carried out for all the parameters for a minimum of 7 values are present, in justified cases, an evaluation may also be carried out from 5 results onwards.

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

3.2 Robust standard deviation

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

3.3 Repeatability standard deviation

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

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

The relative repeatability standard deviation as a percentage of the mean value is indicated as coefficient of variation CV_r in the table of 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 CV_R in percent of the mean is given as variation coefficient in the statistical data of participant for each parameter. The significance of CV_R is further explained in section 3.9.

3.5 Exclusion of results and outliers

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

All results should be given at least with 2 significant digits. Specifying 3 significant digits is usually sufficient.

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

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

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

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

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

In the present PT for valuation of all following parameters the target standard deviation according to the <u>general model of Horwitz</u> was applied (see 3.6.1):
<u>L-Alanine, L-Glutamine, L-Serine, L-Tryptophan and Taurine.</u>

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

<u>Additionally</u> for <u>L-Cystine, L-Glutamine and Glycine</u> the standard uncertainty was considered by evaluation using z'-scores (see 3.6.8).

Due to the inconsistency and the small number of results L-Glutamic Acid was not evaluated using z-scores.

There were no quantitative results for cysteine.

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 relative repeatability standard deviations (RSD_r) and relative reproducibility standard deviation (RSD_R) given in Table 4 were determined in ring tests using the indicated methods.
The resulting target standard deviations σ_{pt}, which were identified there, were used to evaluate the results and to provide additional information for the statistical data.

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

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

[1] *used in evaluation or given for information (s. chapter 4)*
The given values from ASU 49.07-1 are mean values of samples I and II (exceptions: Methionine sample I, Phenylalanine sample II only)

3.6.3 Value by perception

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

For the present evaluation the target standard deviation according to 3.6.1 was regarded suitable.
Table 5 shows selected statistic data of participants results of present PT compared to PT results of previous years.

3.7 z-Score

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

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

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

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

The valid z-Score for each parameter is indicated as z-Score (σ_{pt}). The value indicated as z-Score (Info) only obtains a informative character. The both z-Scores were calculated with the different target standard deviations in accordance with 3.6.

3.7.1 Warning and action signals

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

An error or cause analysis can be carried out by checking the analysis process including understanding and implementation of the measurement by the staff, details of the measurement procedure, calibration of equipment and composition of reagents, transmission error or an error in the calcu-lation, in the trueness and precision and use of reference material. If necessary, the problems must be addressed through appropriate corrective action [3].

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

Table 5: Characteristics of the present PT (on grey) in comparison to previous PTs since 2015, free amino acids in dietetic foods (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	Diet. food	0,460	0,0369	8,02	1,8	DLA 43/2015
L-Alanine	Diet. food	1,95	0,170	8,75	1,9[1]	DLA 47/2017
L-Alanine	Diet. food	0,595	0,0340	5,71	1,3	DLA 50/2019
L-Arginine	Diet. food	0,749	0,0616	8,22	0,61	DLA 43/2015
L-Arginine	Diet. food	1,42	0,222	15,6	1,1	DLA 47/2017
L-Arginin	Diet. food	1,00	0,0731	7,27	0,54	DLA 50/2019
L-Aspartic acid	Diet. food	0,740	0,0332	4,49	1,1	DLA 43/2015
L-Aspartic acid	Diet. food	3,86	0,470	12,2	1,8[1]	DLA 47/2017
L-Aspartic acid	Diet. food	0,951	0,0663	6,97	1,2	DLA 50/2019
L-Cysteine L-Cystine	Diet. food	**	–	–	–	DLA 43/2015
L-Cysteine L-Cystine	Diet. food	**	–	–	–	DLA 47/2017
L-Cysteine	Diet. food	**	–	–	–	DLA 50/2019
L-Cystine	Diet. food	0,376	0,219	58,4	2,1[1]	DLA 50/2019
L-Glutamine	Diet. food	1,32	0,179	13,5	1,7[1]	DLA 50/2019
L-Glut-amic acid	Diet. food	**	–	–	–	DLA 43/2015
L-Glut-amic acid	Diet. food	1,48	0,177	12,0	1,8	DLA 47/2017
L-Glut-amic acid	Diet. food	**	–	–	–	DLA 50/2019
Glycine	Diet. food	0,688	0,0964	14,0	1,9[1]	DLA 43/2015
Glycine	Diet. food	0,755	0,0863	11,4	1,8[1]	DLA 47/2017
Glycine	Diet. food	0,920	0,107	11,6	1,9[1]	DLA 50/2019
L-Histidine	Diet. food	0,435	0,0354	8,14	0,51	DLA 43/2015
L-Histidine	Diet. food	1,66	0,404	24,4	1,5	DLA 47/2017
L-Histidine	Diet. food	0,590	0,106	17,9	1,1	DLA 50/2019
L-Isoleucine	Diet. food	0,686	0,0246	3,59	0,85	DLA 43/2015
L-Isoleucine	Diet. food	3,76	0,381	10,1	1,8[1]	DLA 47/2017
L-Isoleucine	Diet. food	0,917	0,0730	7,95	1,7	DLA 50/2019
L-Leucine	Diet. food	1,17	0,0237	2,03	0,52	DLA 43/2015
L-Leucine	Diet. food	6,56	0,532	8,12	1,9	DLA 47/2017
L-Leucine	Diet. food	1,55	0,100	6,47	1,5	DLA 50/2019
L-Lysine	Diet. food	0,797	0,0666	8,36	0,66	DLA 43/2015
L-Lysine	Diet. food	4,01	0,435	10,9	0,85	DLA 47/2017
L-Lysine	Diet. food	1,07	0,124	11,6	0,91	DLA 50/2019

continuation table 5:

Parameter	Matrix (Pulver)	rob. Mittelwert [g/100g]	rob. SD (S*) [g/100g]	rel. SD (VK$_{S*}$) [%]	Quotient S*/σ$_{pt}$	DLA-Bericht
L-Methionine	Diet. food	0,179	0,0164	9,16	1,8	DLA 43/2015
L-Methionine	Diet. food	1,17	0,108	9,21	1,4	DLA 47/2017
L-Methionine	Diet. food	0,252	0,0152	6,03	0,91	DLA 50/2019
L-Phenylalanine	Diet. food	0,515	0,0273	5,30	1,8	DLA 43/2015
L-Phenylalanine	Diet. food	1,89	0,157	8,28	1,4	DLA 47/2017
L-Phenylalanine	Diet. food	0,698	0,0621	8,90	1,5	DLA 50/2019
L-Proline	Diet. food	0,837	0,0915	10,9	1,9	DLA 43/2015
L-Proline	Diet. food	2,23	0,263	11,8	2,0	DLA 47/2017
L-Proline	Diet. food	1,16	0,112	9,65	1,6	DLA 50/2019
L-Serine	Diet. food	0,500	0,0594	11,9	1,7	DLA 43/2015
L-Serine	Diet. food	1,80	0,256	14,2	1,6[1]	DLA 47/2017
L-Serine	Diet. food	0,693	0,0311	4,49	1,1	DLA 50/2019
L-Threonine	Diet. food	0,608	0,0666	11,0	1,9	DLA 43/2015
L-Threonine	Diet. food	2,77	0,236	8,52	1,4	DLA 47/2017
L-Threonine	Diet. food	0,771	0,0498	4,46	1,1	DLA 50/2019
L-Tryptophan	Diet. food	**	–	–	–	DLA 43/2015
L-Tryptophan	Diet. food	2,75	0,199	7,25	0,37	DLA 47/2017
L-Tryptophan	Diet. food	0,305	0,0177	5,80	1,2	DLA 50/2019
L-Tyrosine	Diet. food	0,524	0,0213	4,06	0,92	DLA 43/2015
L-Tyrosine	Diet. food	1,18	0,0878	7,47	1,0	DLA 47/2017
L-Tyrosine	Diet. food	0,684	0,0445	6,51	0,89	DLA 50/2019
L-Valine	Diet. food	0,762	0,0320	4,20	1,0	DLA 43/2015
L-Valine	Diet. food	3,04	0,327	10,7	1,8[1]	DLA 47/2017
L-Valine	Diet. food	0,983	0,0950	9,66	2,0	DLA 50/2019
Taurine	Diet. food	0,0317	0,00378	12,0	1,8	DLA 50/2019

[1] *with target standard deviation σpt'*
** *no statistical evaluation (< 7 results)*

3.8 z'-Score
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The z'-score can be used for the valuation of the results of the participants, in cases the standard uncertainty has to be considered (s. 3.11). The z'-score represents the relation of the deviation of the result (x) of the participant from the respective consensus value (X) to the square root of quadrat sum of the target standard deviation (σ_{pt}) and the standard uncertainty (Ux_{pt}) [3].

The calculation is performed by:

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

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

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

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

For warning and action signals see 3.7.1.

3.9 Reproducibility cofficient of variation (CV_R)
_____

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

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

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

3.10 Quotient S*/σpt

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

3.11 Standard uncertainty of the assigned value

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

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

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

The traceability of the assigned value is ensured on the basis of the consensus value as a robust mean of the participant results.

4. Results

Comments to the distribution of the results:

The kernel density plots showed for all parameters nearly a symmetrical distribution of results (figures see documentation 5.3). Partly slight shoulders and separated smaller peaks can be seen, which are due to individual results, outliers or values outside the target range.

Comments to the statistic data:

There were 7 results for L-glutamic acid, which showed a high variability. Therefore no statistical evaluation was carried out. L-glutamic acid was not added to the samples.
There were 5 results for L-glutamine. Due to the small number of results, the significance of the statistical evaluation can be limited. However, the statistical characteristics allow an assessment.

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

For all parameters except for L-cystine, L-glutamine and glycine the distribution of results showed a normal variability. The quotients S^*/σ_{pt} were in the range of 0,54 to 2,0 (see table 4).

For L-cystine, L-glutamine and glycine the distributions of results showed an increased variability with quotients above 2,0. Therefore these parameters were evaluated considering the standard uncertainties by z´-scores. The quotients S^*/σ_{pt}' were between 1,7 to 2,1 then (see table 4).

For information, the target standard deviation according to the characteristics of a precision experiment (ASU §64 method) or according to the general model of Horwitz was also given.

The robust standard deviation and the repeatability and reproducibility standard deviations were in the expected range of usual values for the used determination methods (s. 3.6.2). For L-cystine the standard deviation and the reproducibility standard deviation were increased, while the repeatability standard deviation was in the normal range.

The comparability of results is given.

64% to 100% of results were in the respective target range.

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

In the first table the characteristics are listed:

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

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

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

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

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

4.1 L-Alanine in g/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	18
Number of outliers	0
Mean	0,593
Median	0,598
Robust Mean (X_{pt})	**0,595**
Robust standard deviation (S*)	**0,0340**
Number with 2 replicates	18
Repeatability SD (S_r)	0,0882
Repeatability (CV_r)	14,9%
Reproducibility SD (S_R)	–
Reproducibility (CV_R)	–
Target range:	
Target standard deviation σ_{pt}	**0,0257**
Target standard deviation (for Information)	0,0233
lower limit of target range	**0,543**
upper limit of target range	**0,646**
Quotient S*/σ_{pt}	*1,3*
Standard uncertainty $U_{(Xpt)}$	*0,0100*
Results in the target range	*16*
Percent in the target range	*89%*
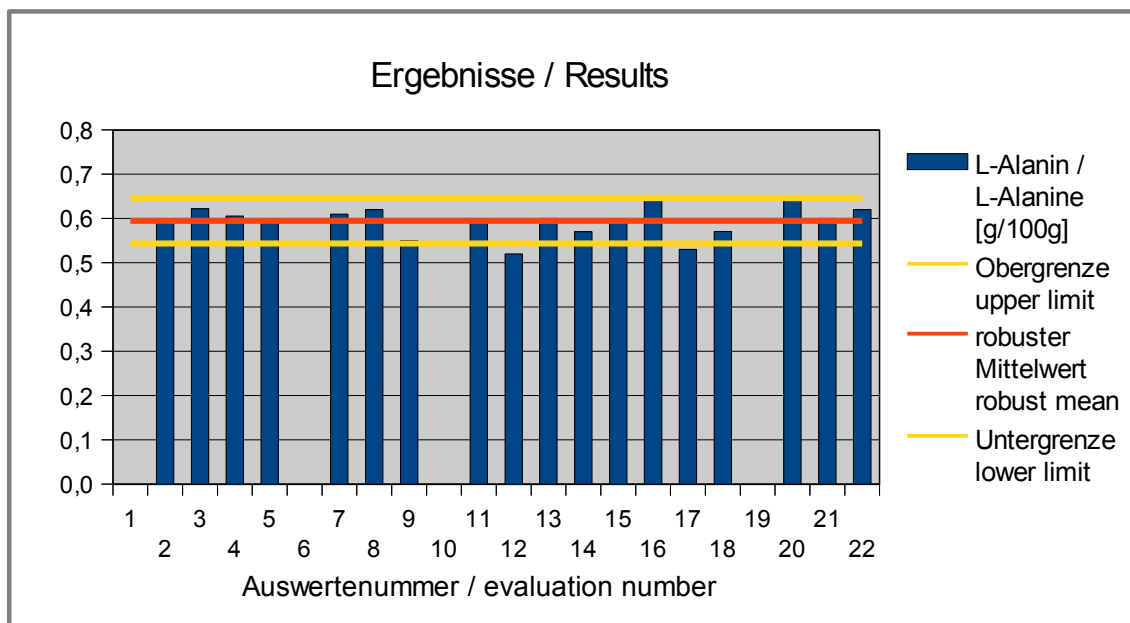
Abb. / Fig. 1: Ergebnisse L-Alanin **/** Results L-Alanine

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					
2	0,590	−0,0048	−0,19	−0,20	
3	0,622	0,0272	1,1	1,2	
4	0,606	0,0107	0,42	0,46	
5	0,590	−0,0048	−0,19	−0,20	
6					
7	0,610	0,0152	0,59	0,65	
8	0,620	0,0252	0,98	1,1	
9	0,550	−0,0448	−1,7	−1,9	
10					
11	0,595	0,0002	0,01	0,01	
12	0,520	−0,0748	−2,9	−3,2	
13	0,600	0,0052	0,20	0,23	
14	0,570	−0,0248	−0,96	−1,1	
15	0,592	−0,0028	−0,11	−0,12	
16	0,640	0,0452	1,8	1,9	
17	0,530	−0,0648	−2,5	−2,8	
18	0,571 *	−0,0243	−0,94	−1,04	
19					
20	0,643	0,0482	1,9	2,1	
21	0,600 *	0,0052	0,20	0,23	
22	0,620	0,0252	0,98	1,1	

* Mean calculated by DLA
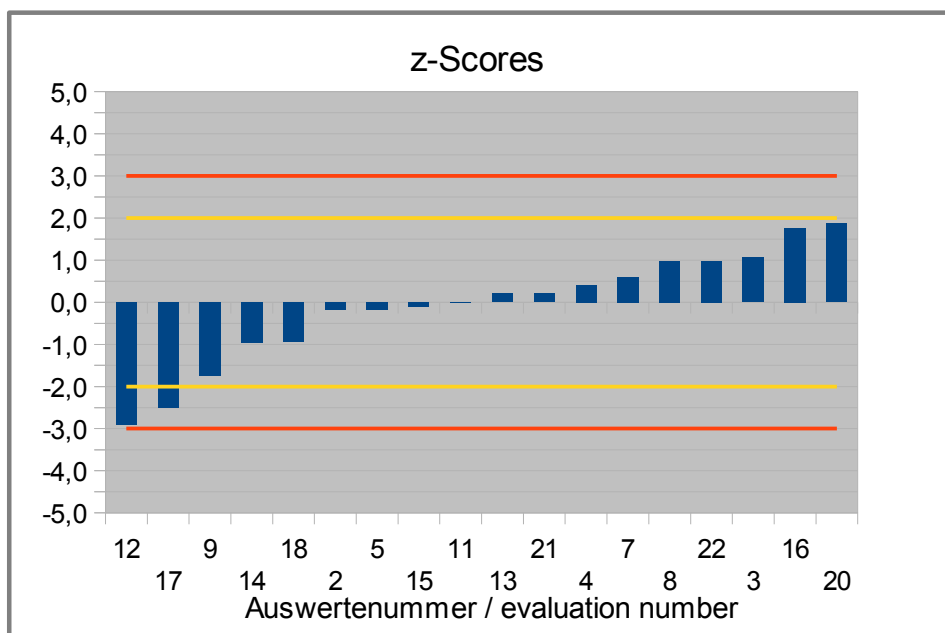
Abb. / Fig. 2:   z-Scores L-Alanin/ L-Alanine

4.2 L-Arginine in g/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	18
Number of outliers	0
Mean	1,00
Median	1,01
Robust Mean (X_{pt})	**1,00**
Robust standard deviation (S*)	**0,0731**
Number with 2 replicates	18
Repeatability SD (S_r)	0,0333
Repeatability (CV_r)	3,34%
Reproducibility SD (S_R)	0,0926
Reproducibility (CV_R)	9,28%
Target range:	
Target standard deviation σ_{pt}	**0,137**
Target standard deviation (for Information)	0,0402
lower limit of target range	**0,732**
upper limit of target range	**1,28**
Quotient S*/σ_{pt}	*0,54*
Standard uncertainty U(X_{pt})	*0,0215*
Results in the target range	*18*
Percent in the target range	*100%*
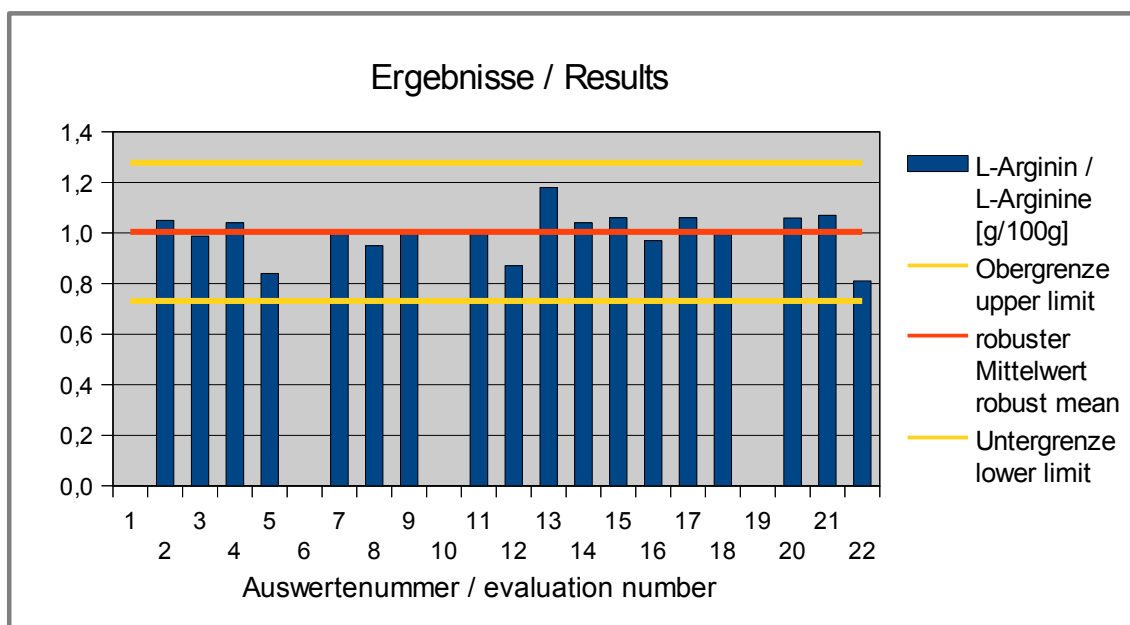
Abb. / Fig. 3: Ergebnisse L-Arginin / Results L-Arginine

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	1,05	0,0452	0,33	1,1	
3	0,988	−0,0168	−0,12	−0,42	
4	1,04	0,0352	0,26	0,88	
5	0,840	−0,1648	−1,2	−4,1	
6					
7	1,01	0,0052	0,04	0,13	
8	0,950	−0,0548	−0,40	−1,4	
9	1,00	−0,0048	−0,04	−0,12	
10					
11	0,995	−0,0098	−0,07	−0,25	
12	0,870	−0,1348	−0,99	−3,4	
13	1,18	0,1752	1,3	4,4	
14	1,04	0,0352	0,26	0,88	
15	1,06	0,0552	0,40	1,4	
16	0,970	−0,0348	−0,26	−0,87	
17	1,06	0,0552	0,40	1,4	
18	0,995 *	−0,0098	−0,07	−0,25	
19					
20	1,06	0,0542	0,40	1,3	
21	1,07 *	0,0652	0,48	1,6	
22	0,810	−0,1948	−1,4	−4,9	

* Mean calculated by DLA
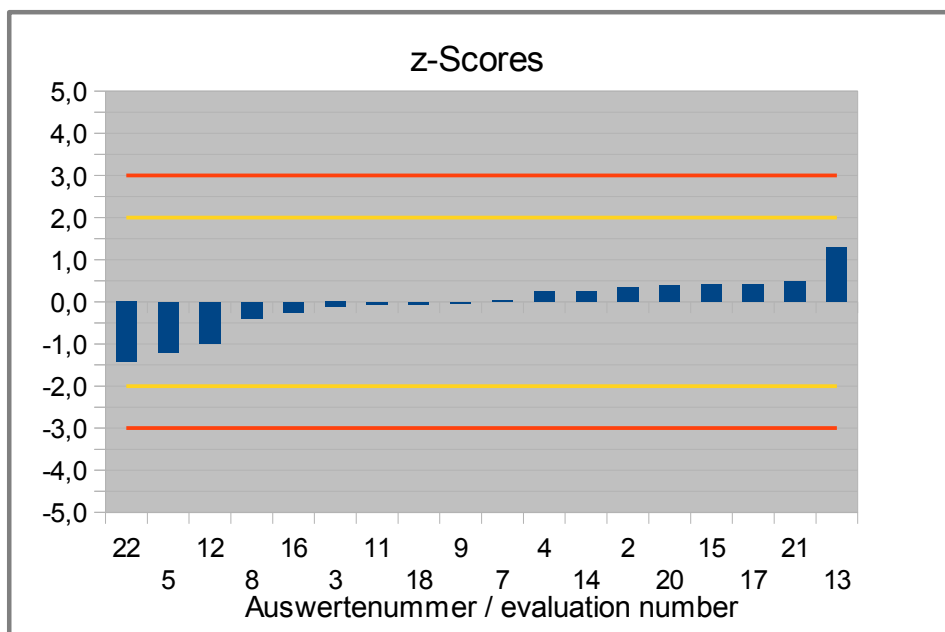
Abb. / Fig. 4:   z-Scores L-Arginin / L-Arginine

4.3 L-Aspartic Acid in g/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	19
Number of outliers	0
Mean	0,947
Median	0,962
Robust Mean (X_{pt})	**0,951**
Robust standard deviation (S*)	**0,0663**
Number with 2 replicates	19
Repeatability SD (S_r)	0,0350
Repeatability (CV_r)	3,70%
Reproducibility SD (S_R)	0,0732
Reproducibility (CV_R)	7,74%
Target range:	
Target standard deviation σ_{pt}	**0,0535**
Target standard deviation (for Information)	0,0383
lower limit of target range	**0,844**
upper limit of target range	**1,06**
Quotient S*/σ_{pt}	*1,2*
Standard uncertainty $U_{(Xpt)}$	*0,0190*
Quotient $U_{(Xpt)}/\sigma_{pt}$	*0,35*
Results in the target range	*17*
Percent in the target range	*89%*
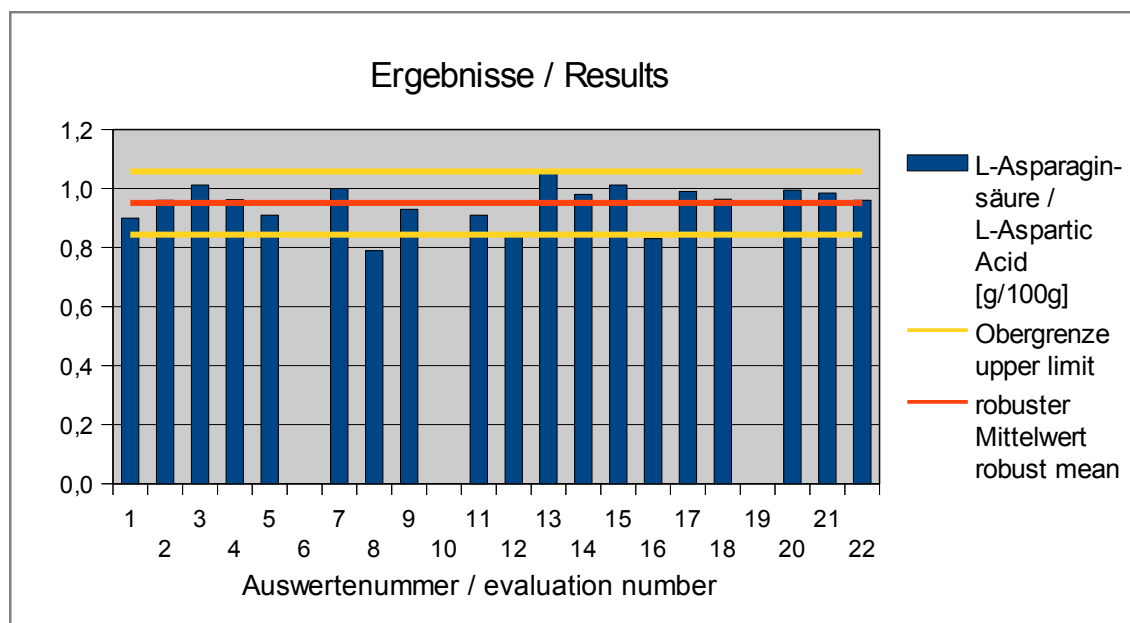
Abb. / Fig. 5: Ergebnisse L-Asparaginsäure **/** Results L-Aspartic acid

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	0,900	–0,0513	–0,96	–1,3	
2	0,960	0,0087	0,16	0,23	
3	1,01	0,0607	1,1	1,6	
4	0,962	0,0107	0,20	0,28	
5	0,910	–0,0413	–0,77	–1,1	
6					
7	1,00	0,0487	0,91	1,3	
8	0,790	–0,1613	–3,0	–4,2	
9	0,930	–0,0213	–0,40	–0,56	
10					
11	0,910	–0,0413	–0,77	–1,1	
12	0,850	–0,1013	–1,9	–2,6	
13	1,06	0,1037	1,9	2,7	
14	0,980	0,0287	0,54	0,75	
15	1,012	0,0607	1,1	1,6	
16	0,830	–0,1213	–2,3	–3,2	
17	0,990	0,0387	0,72	1,0	
18	0,964　　　*	0,0122	0,23	0,32	
19					
20	0,995	0,0432	0,81	1,1	
21	0,985　　　*	0,0337	0,63	0,88	
22	0,960	0,0087	0,16	0,23	

* Mean calculated by DLA
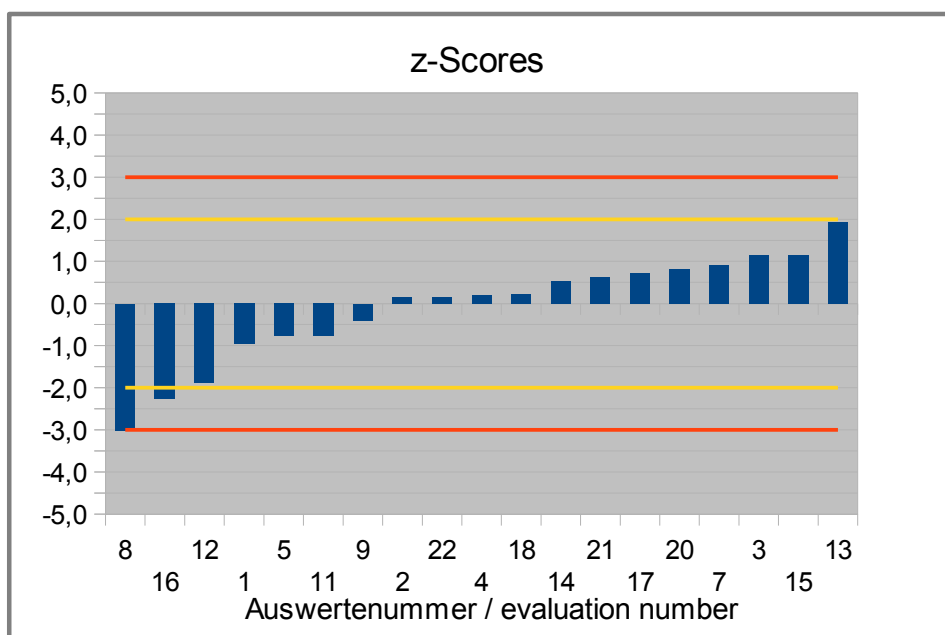
Abb. / Fig. 6:　　z-Scores L-Asparaginsäure **/** L-Aspartic acid

4.4 L-Cysteine in g/100g

Note: No quantitative results were submitted.

Auswerte-nummer Evaluation number	L-Cystein / L-Cysteine [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					
9					
10					
11	< 0,02				
12					
13					
14					
15					
16					
17					
18					
19					
20					
21					
22					

4.5 L-Cystine in g/100g

Vergleichsuntersuchung / Proficiency Test

| Statistic Data	
Number of results	11
Number of outliers	0
Mean	0,400
Median	0,391
Robust Mean (X_{pt})	**0,376**
Robust standard deviation (S*)	**0,219**
Number with 2 replicates	11
Repeatability SD (S_r)	0,0260
Repeatability (CV_r)	6,54%
Reproducibility SD (S_R)	0,241
Reproducibility (CV_R)	60,5%
Target range:	
Target standard deviation σ_{pt}	**0,106**
Target standard deviation (for Information)	0,0174
lower limit of target range	**0,165**
upper limit of target range	**0,588**
Quotient S*/σ_{pt}	*2,1*
Standard uncertainty $U_{(Xpt)}$	*0,0827*
Results in the target range	7
Percent in the target range	64%
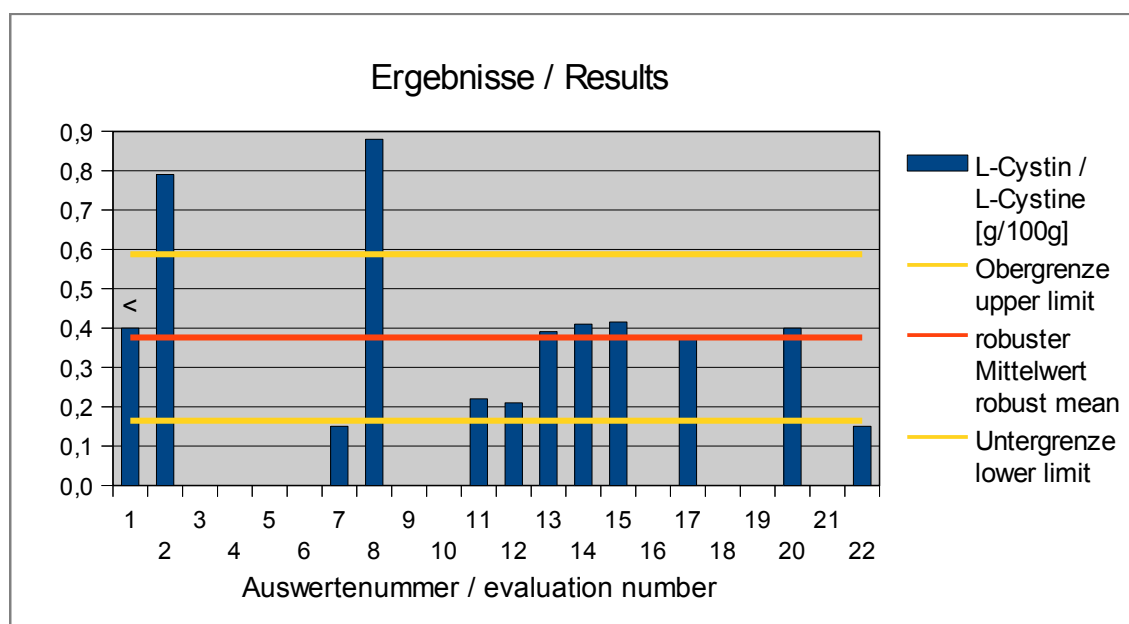
Abb. / Fig. 7: Ergebnisse L-Cystin **/** Results L-Cystine

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	L-Cystin / L-Cystine [g/100g]	Abweichung [g/100g] Deviation [g/100g]	z'-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	< 0,40				
2	0,790	0,414	3,9	24	
3					
4					
5					
6					
7	0,150	−0,226	−2,1	−13	
8	0,880	0,504	4,8	29	
9					
10					
11	0,220	−0,156	−1,5	−9,0	
12	0,210	−0,166	−1,6	−9,5	
13	0,391	0,015	0,14	0,86	
14	0,410	0,034	0,32	1,9	
15	0,415	0,039	0,37	2,2	
16					
17	0,380	0,004	0,04	0,23	
18					
19					
20	0,400	0,024	0,23	1,4	
21					
22	0,150	−0,226	−2,1	−13	
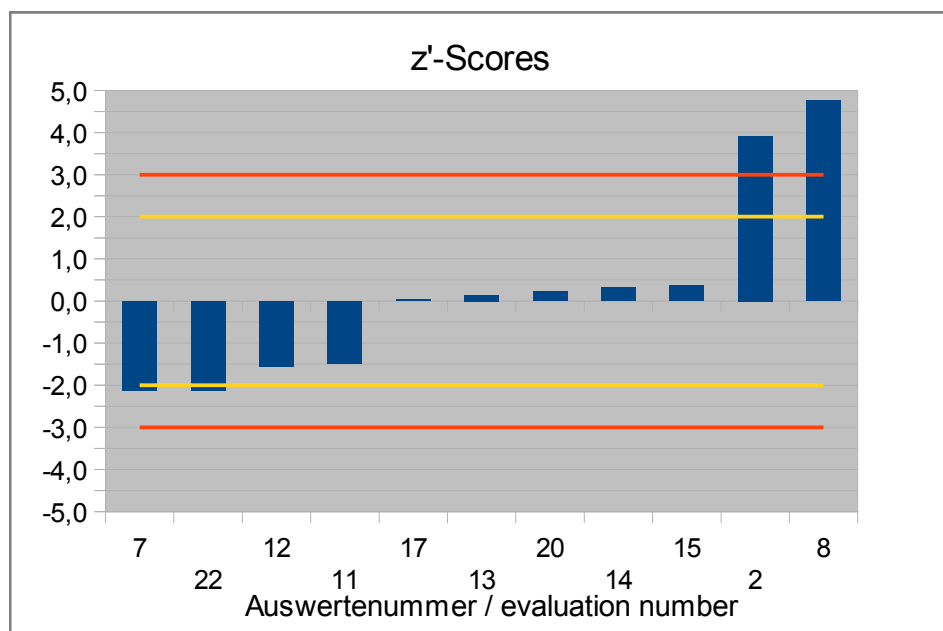
Abb. / Fig. 8:   z'-Scores L-Cystin / L-Cystine

4.6 L-Glutamic Acid in g/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	7
Number of outliers	
Mean	0,394
Median	0,146
Robust Mean (X_{pt})	**0,460**
Robust standard deviation (S*)	**0,617**
Number with 2 replicates	
Repeatability SD (S$_r$)	
Repeatability (CV$_r$)	
Reproducibility SD (S$_R$)	
Reproducibility (CV$_R$)	
Target range:	
Target standard deviation σ_{pt}	
Target standard deviation (for Information)	
lower limit of target range	
upper limit of target range	
Quotient S*/σ_{pt}	
Standard uncertainty U(X_{pt})	
Results in the target range	
Percent in the target range	
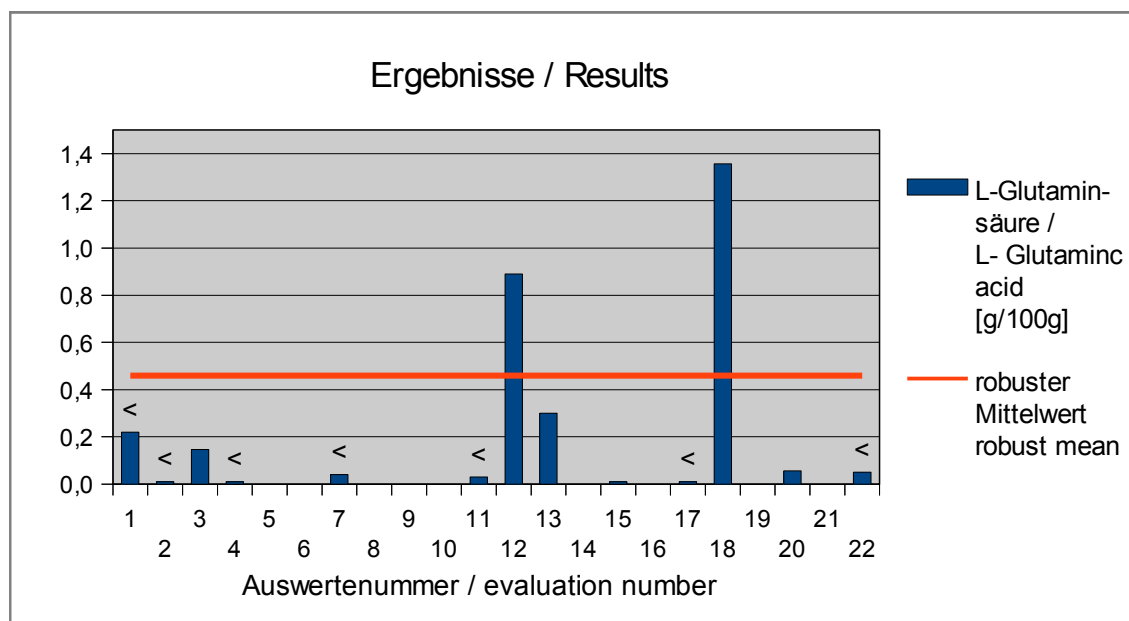
Abb. / Fig. 9: Ergebnisse L-Glutaminsäure **/** Results L-Glutamic acid

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	L-Glutamin-säure / L- Glutaminc acid [g/100g]	Abweichung [g/100g] Deviation [g/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	< 0,22				
2	< 0,01				
3	0,146	−0,314			
4	< 0,01				
5					
6					
7	< 0,04				
8					
9					
10					
11	< 0,03				
12	0,890	0,430			
13	0,300	−0,160			
14					
15	0,010	−0,450			
16					
17	< 0,01				
18	1,36 *	0,897			
19					
20	0,056	−0,404			
21					
22	< 0,05				

* Mean calculated by DLA

4.7 Glycine in g/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	19
Number of outliers	–
Mean	0,912
Median	0,940
Robust Mean (X_{pt})	**0,920**
Robust standard deviation (S*)	**0,107**
Number with 2 replicates	18
Repeatability SD (S_r)	0,0870
Repeatability (CV_r)	9,35%
Reproducibility SD (S_R)	0,112
Reproducibility (CV_R)	12,1%
Target range:	
Target standard deviation σ_{pt}	**0,0564**
Target standard deviation (for Information)	0,0373
lower limit of target range	**0,807**
upper limit of target range	**1,03**
Quotient S*/σ_{pt}	*1,9*
Standard uncertainty U(X_{pt})	*0,0307*
Results in the target range	*14*
Percent in the target range	*74%*
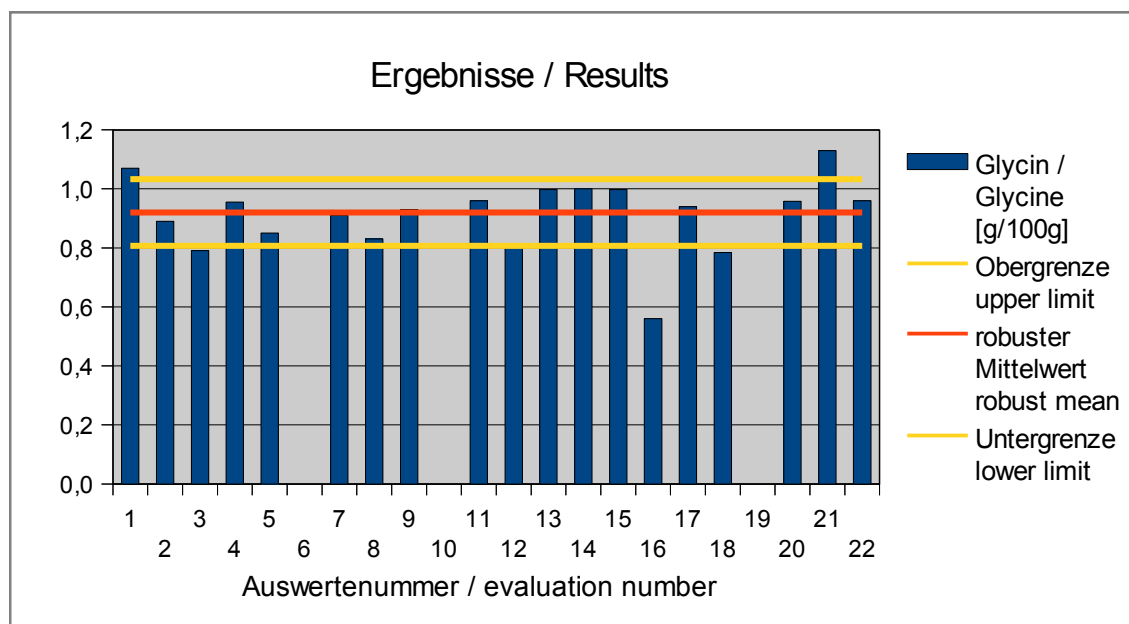
Abb. / Fig. 10: Ergebnisse Glycin / Results Glycine

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Glycin / Glycine [g/100g]	Abweichung [g/100g] Deviation [g/100g]	z'-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	1,07	0,150	2,7	4,0	
2	0,890	−0,030	−0,53	−0,80	
3	0,791	−0,129	−2,3	−3,5	
4	0,956	0,036	0,63	0,96	
5	0,850	−0,070	−1,2	−1,9	
6					
7	0,910	−0,010	−0,17	−0,26	
8	0,830	−0,090	−1,6	−2,4	
9	0,930	0,010	0,18	0,28	
10					
11	0,960	0,040	0,71	1,1	
12	0,810	−0,110	−1,9	−2,9	
13	0,998	0,078	1,4	2,1	
14	1,00	0,080	1,4	2,2	
15	0,998	0,078	1,4	2,1	
16	0,560	−0,360	−6,4	−9,7	
17	0,940	0,020	0,36	0,55	
18	0,784 *	−0,136	−2,4	−3,6	
19					
20	0,958	0,038	0,68	1,0	
21	1,13 *	0,210	3,7	5,6	
22	0,960	0,040	0,71	1,1	

* Mean calculated by DLA
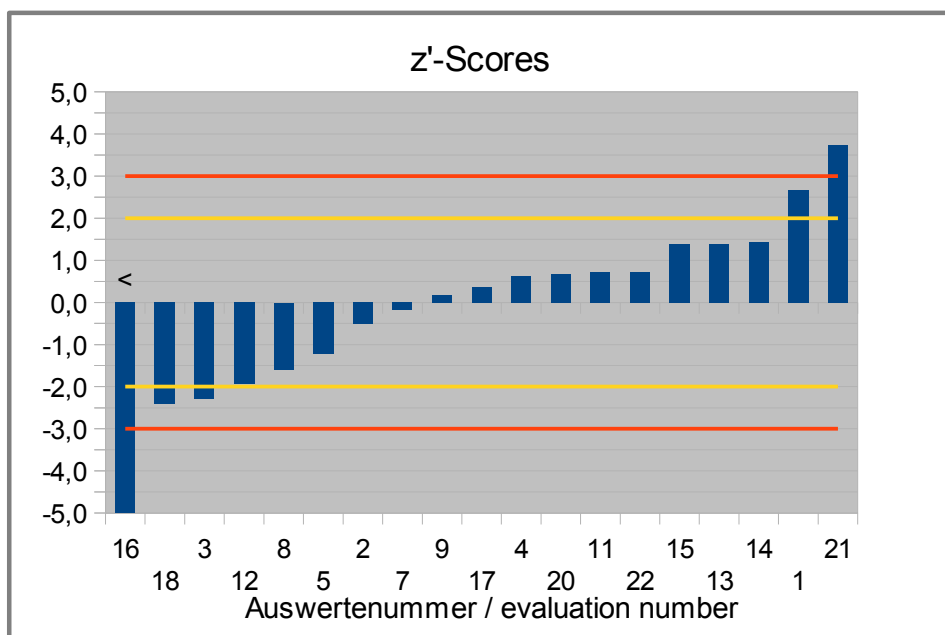
Abb. / Fig. 11:   z'-Scores Glycin / Glycine

4.8 L-Histidine in g/100g

Vergleichsuntersuchung / Proficiency Test

| Statistic Data	
Number of results	19
Number of outliers	-
Mean	0,749
Median	0,580
Robust Mean (X_{pt})	**0,590**
Robust standard deviation (S*)	**0,106**
Number with 2 replicates	15
Repeatability SD (S_r)	0,0172
Repeatability (CV_r)	3,02%
Reproducibility SD (S_R)	0,0611
Reproducibility (CV_R)	10,8%
Target range:	
Target standard deviation σ_{pt}	**0,0947**
Target standard deviation (for Information)	0,0255
lower limit of target range	**0,400**
upper limit of target range	**0,779**
Quotient S*/σ_{pt}	*1,1*
Standard uncertainty U(X_{pt})	*0,0303*
Results in the target range	*15*
Percent in the target range	*79%*
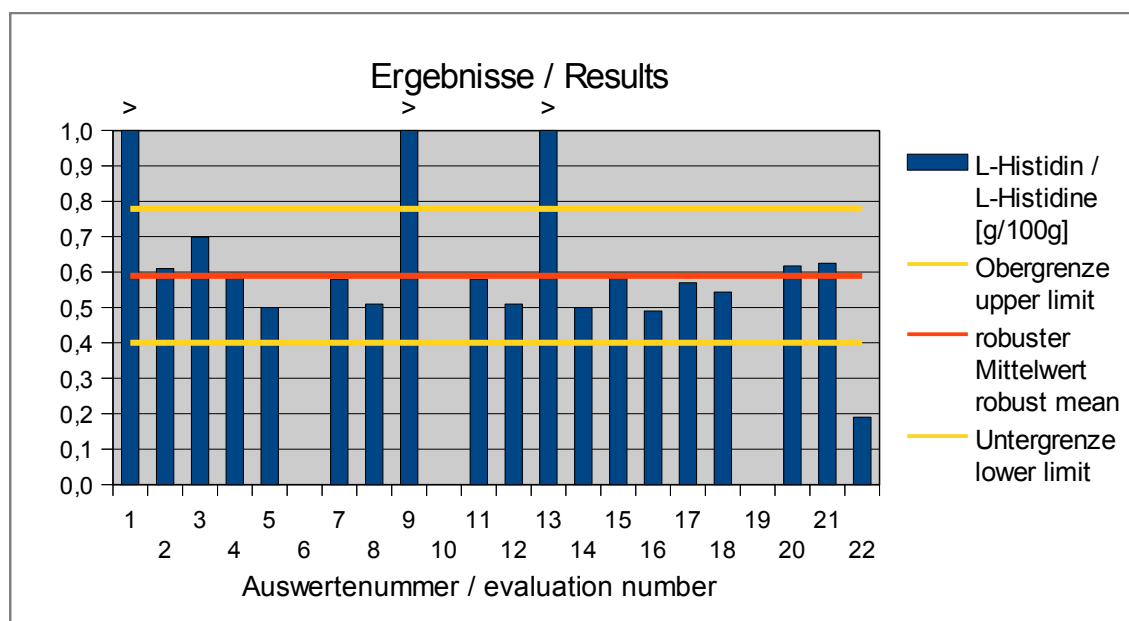
Abb. / Fig. 12: Ergebnisse L-Histidin **/** Results L-Histidine

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	2,30	1,710	18	67	
2	0,610	0,020	0,22	0,80	
3	0,699	0,109	1,2	4,3	
4	0,596	0,006	0,07	0,25	
5	0,500	−0,090	−0,95	−3,5	
6					
7	0,580	−0,010	−0,10	−0,37	
8	0,510	−0,080	−0,84	−3,1	
9	1,93	1,340	14	52	
10					
11	0,580	−0,010	−0,10	−0,37	
12	0,510	−0,080	−0,84	−3,1	
13	1,29	0,700	7,4	27	
14	0,500	−0,090	−0,95	−3,5	
15	0,596	0,006	0,07	0,25	
16	0,490	−0,100	−1,1	−3,9	
17	0,570	−0,020	−0,21	−0,77	
18	0,544 *	−0,046	−0,49	−1,8	
19					
20	0,617	0,027	0,29	1,1	
21	0,625 *	0,035	0,37	1,4	
22	0,190	−0,400	−4,2	−16	

* Mean calculated by DLA
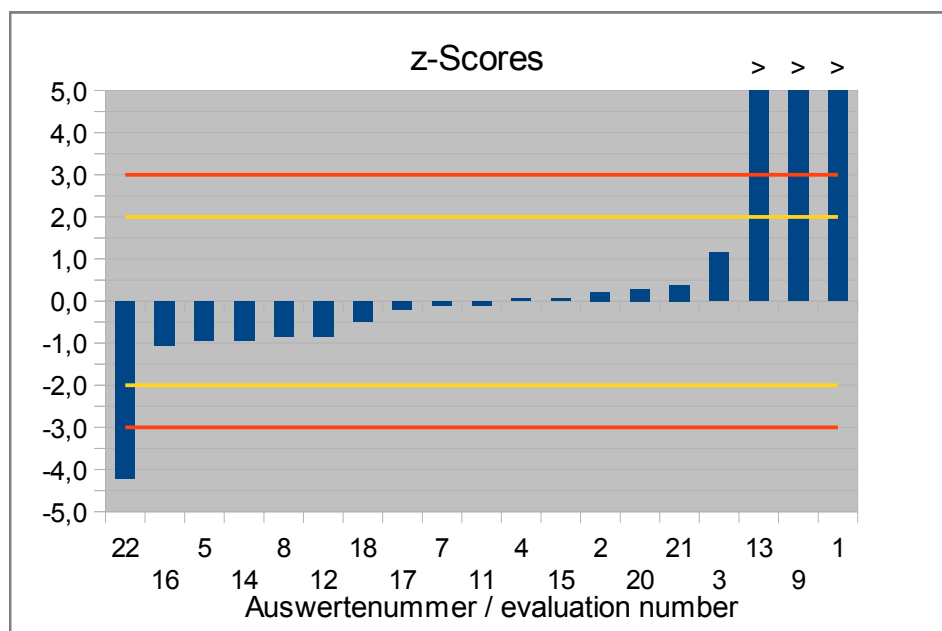
Abb. / Fig. 13:   z-Scores L-Histidin / L-Histidine

4.9 L-Isoleucine in g/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	20
Number of outliers	–
Mean	0,931
Median	0,910
Robust Mean (X_{pt})	**0,917**
Robust standard deviation (S*)	**0,0730**
Number with 2 replicates	18
Repeatability SD (S_r)	0,0320
Repeatability (CV_r)	3,54%
Reproducibility SD (S_R)	0,0762
Reproducibility (CV_R)	8,45%
Target range:	
Target standard deviation σ_{pt}	**0,0419**
Target standard deviation (for Information)	0,0372
lower limit of target range	**0,834**
upper limit of target range	**1,00**
Quotient S*/σ_{pt}	*1,7*
Standard uncertainty $U_{(Xpt)}$	*0,0204*
Results in the target range	*16*
Percent in the target range	*80%*
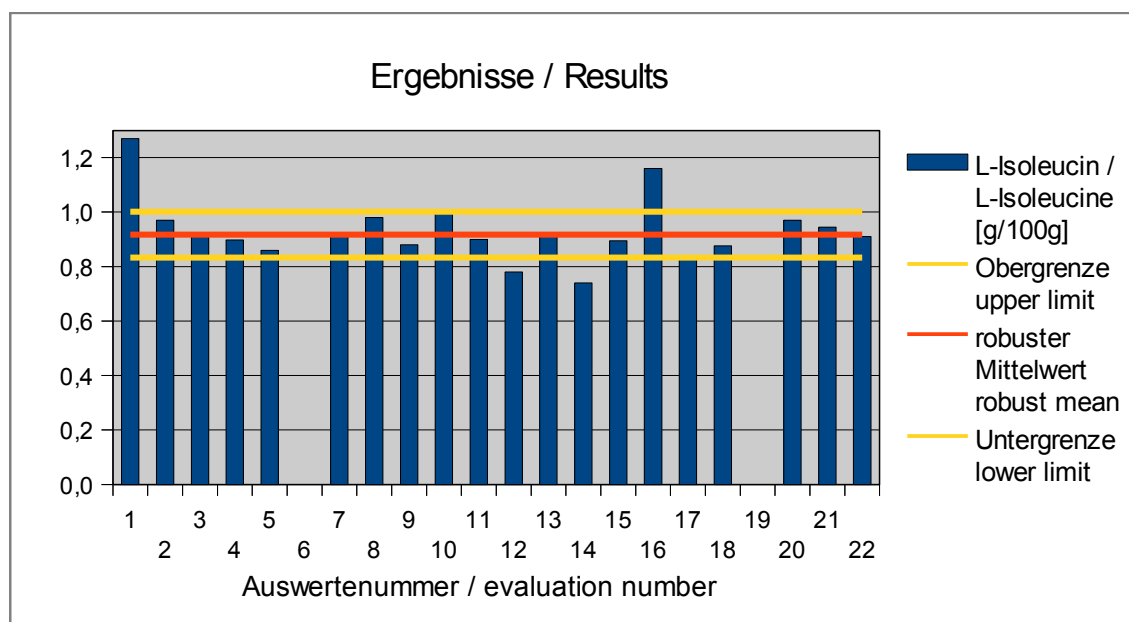
Abb. / Fig. 14: Ergebnisse L-Isoleucin **/** Results L-Isoleucine

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	1,27	0,353	8,4	9,5	
2	0,970	0,053	1,3	1,4	
3	0,924	0,007	0,16	0,18	
4	0,898	−0,020	−0,47	−0,53	
5	0,860	−0,057	−1,4	−1,5	
6					
7	0,910	−0,007	−0,17	−0,20	
8	0,980	0,063	1,5	1,7	
9	0,880	−0,037	−0,89	−1,0	
10	1,00	0,083	2,0	2,2	
11	0,900	−0,017	−0,41	−0,47	
12	0,780	−0,137	−3,3	−3,7	
13	0,919	0,002	0,04	0,05	
14	0,740	−0,177	−4,2	−4,8	
15	0,895	−0,022	−0,53	−0,60	
16	1,16	0,243	5,8	6,5	
17	0,840	−0,077	−1,8	−2,1	
18	0,877 *	−0,041	−0,97	−1,1	
19					
20	0,970	0,053	1,3	1,4	
21	0,945 *	0,028	0,66	0,74	
22	0,910	−0,007	−0,17	−0,20	

* Mean calculated by DLA
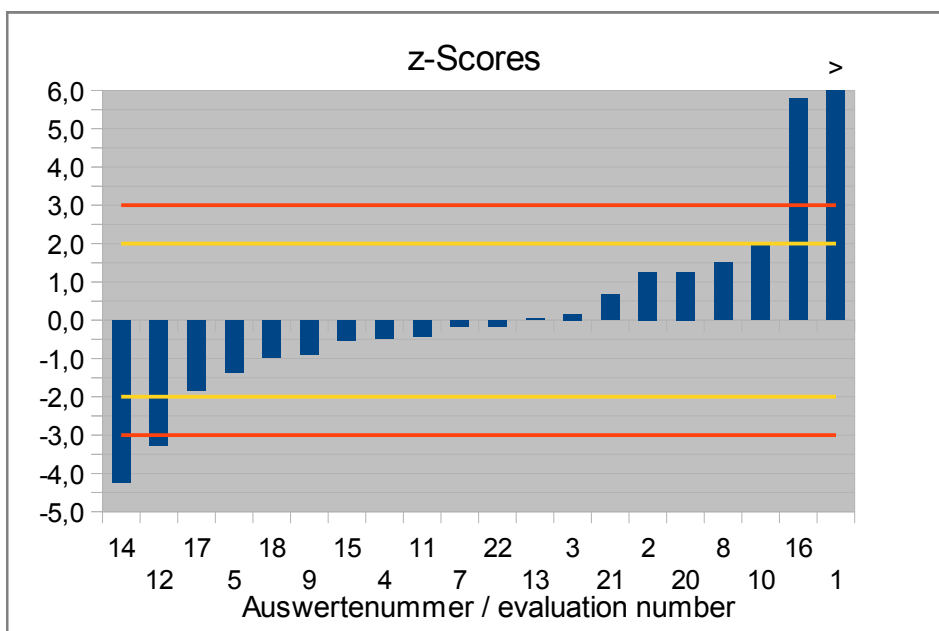
Abb. / Fig. 15:   z-Scores L-Isoleucin/ L-Isoleucine

4.10 L-Leucine in g/100g

Vergleichsuntersuchung / Proficiency Test

| Statistic Data	
Number of results	20
Number of outliers	–
Mean	1,54
Median	1,54
Robust Mean (X_{pt})	**1,55**
Robust standard deviation (S*)	**0,100**
Number with 2 replicates	19
Repeatability SD (S_r)	0,0485
Repeatability (CV_r)	3,11%
Reproducibility SD (S_R)	0,113
Reproducibility (CV_R)	7,26%
Target range:	
Target standard deviation σ_{pt}	**0,0668**
Target standard deviation (for Information)	0,0579
lower limit of target range	**1,41**
upper limit of target range	**1,68**
Quotient S*/σ_{pt}	*1,5*
Standard uncertainty U(X_{pt})	*0,0280*
Results in the target range	*16*
Percent in the target range	*80%*
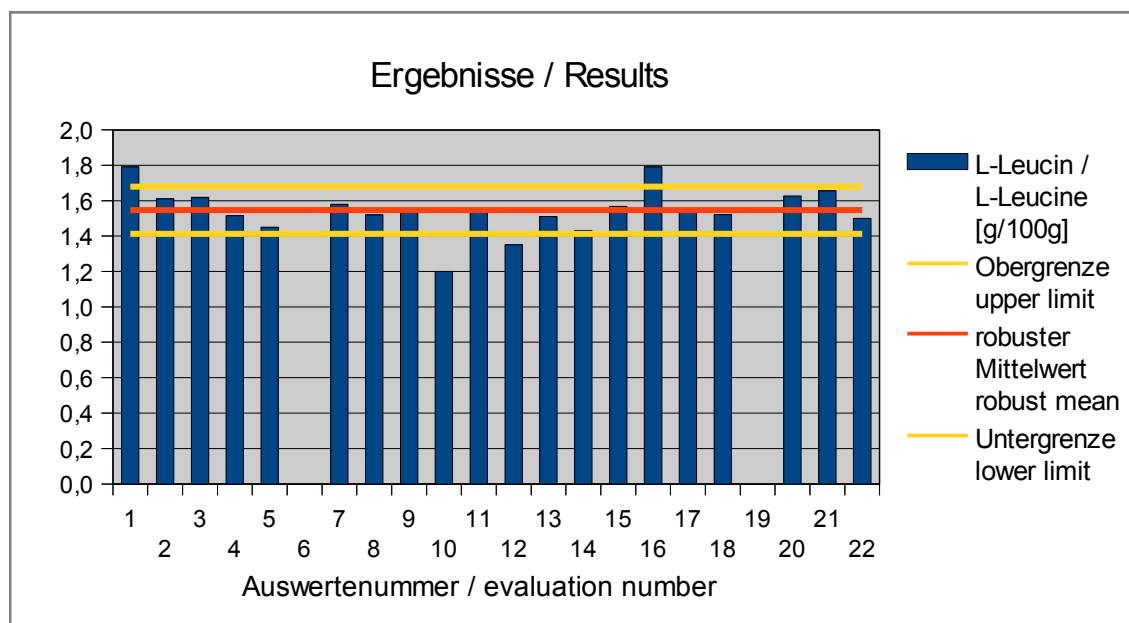
Abb. / Fig. 16: Ergebnisse L-Leucin / Results L-Leucine

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	1,79	0,244	3,7	4,2	
2	1,61	0,064	0,95	1,1	
3	1,62	0,073	1,1	1,3	
4	1,52	−0,031	−0,47	−0,54	
5	1,45	−0,096	−1,4	−1,7	
6					
7	1,58	0,034	0,51	0,58	
8	1,52	−0,026	−0,39	−0,45	
9	1,55	0,004	0,06	0,06	
10	1,20	−0,346	−5,2	−6,0	
11	1,55	−0,001	−0,02	−0,02	
12	1,35	−0,196	−2,9	−3,4	
13	1,51	−0,036	−0,54	−0,63	
14	1,43	−0,116	−1,7	−2,0	
15	1,57	0,022	0,33	0,37	
16	1,79	0,244	3,7	4,2	
17	1,54	−0,006	−0,09	−0,11	
18	1,52　　*	−0,025	−0,37	−0,43	
19					
20	1,63	0,081	1,2	1,4	
21	1,66　　*	0,109	1,6	1,9	
22	1,50	−0,046	−0,69	−0,80	

　　　　* Mean calculated by DLA
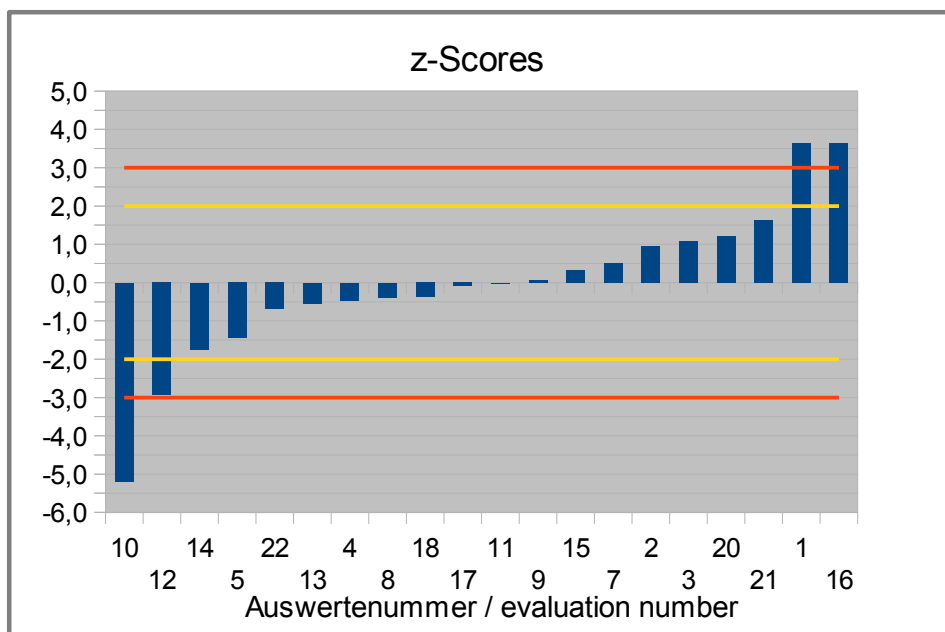
Abb. / Fig. 17:　　z-Scores L-Leucin / L-Leucine

4.11 L-Lysine in g/100g

Vergleichsuntersuchung / Proficiency Test

| Statistic Data	
Number of results	19
Number of outliers	0
Mean	1,07
Median	1,07
Robust Mean (X_{pt})	**1,07**
Robust standard deviation (S*)	**0,124**
Number with 2 replicates	19
Repeatability SD (S_r)	0,0902
Repeatability (CV_r)	8,45%
Reproducibility SD (S_R)	0,126
Reproducibility (CV_R)	11,8%
Target range:	
Target standard deviation σ_{pt}	**0,136**
Target standard deviation (for Information)	0,0423
lower limit of target range	**0,796**
upper limit of target range	**1,34**
Quotient S*/σ_{pt}	*0,91*
Standard uncertainty U$_{(Xpt)}$	*0,0355*
Results in the target range	*19*
Percent in the target range	*100%*
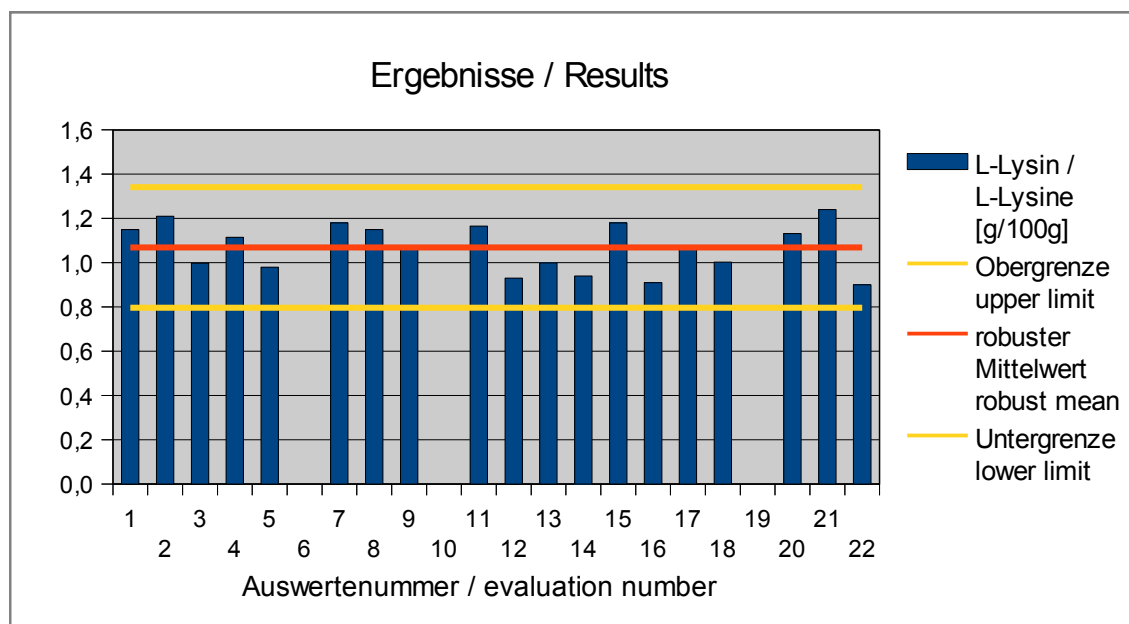
Abb. / Fig. 18: Ergebnisse L-Lysin / Results L-Lysine

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	1,15	0,081	0,60	1,9	
2	1,21	0,141	1,0	3,3	
3	1,00	−0,072	−0,53	−1,7	
4	1,12	0,046	0,34	1,1	
5	0,980	−0,089	−0,65	−2,1	
6					
7	1,18	0,111	0,82	2,6	
8	1,15	0,081	0,60	1,9	
9	1,06	−0,009	−0,07	−0,21	
10					
11	1,17	0,096	0,71	2,3	
12	0,930	−0,139	−1,0	−3,3	
13	1,00	−0,070	−0,51	−1,7	
14	0,940	−0,129	−0,95	−3,0	
15	1,18	0,111	0,82	2,6	
16	0,910	−0,159	−1,2	−3,8	
17	1,07	0,001	0,01	0,03	
18	1,00 *	−0,067	−0,49	−1,6	
19					
20	1,13	0,062	0,46	1,5	
21	1,24 *	0,171	1,3	4,0	
22	0,900	−0,169	−1,2	−4,0	

* Mean calculated by DLA
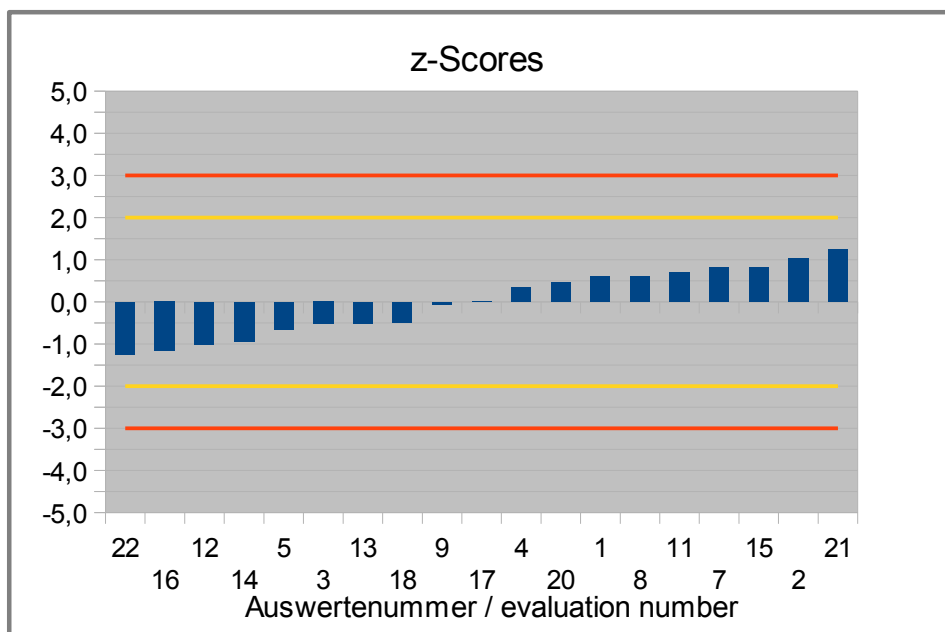
Abb. / Fig. 19:   z-Scores L-Lysin / L-Lysine

4.12 L-Methionine in g/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	19
Number of outliers	–
Mean	0,255
Median	0,251
Robust Mean (X_{pt})	**0,252**
Robust standard deviation (S*)	**0,0152**
Number with 2 replicates	18
Repeatability SD (S_r)	0,0107
Repeatability (CV_r)	4,28%
Reproducibility SD (S_R)	0,0151
Reproducibility (CV_R)	6,05%
Target range:	
Target standard deviation σ_{pt}	**0,0167**
Target standard deviation (for Information)	0,0124
lower limit of target range	**0,219**
upper limit of target range	**0,286**
Quotient S*/σ_{pt}	*0,91*
Standard uncertainty U(X_{pt})	*0,00436*
Results in the target range	*18*
Percent in the target range	*95%*
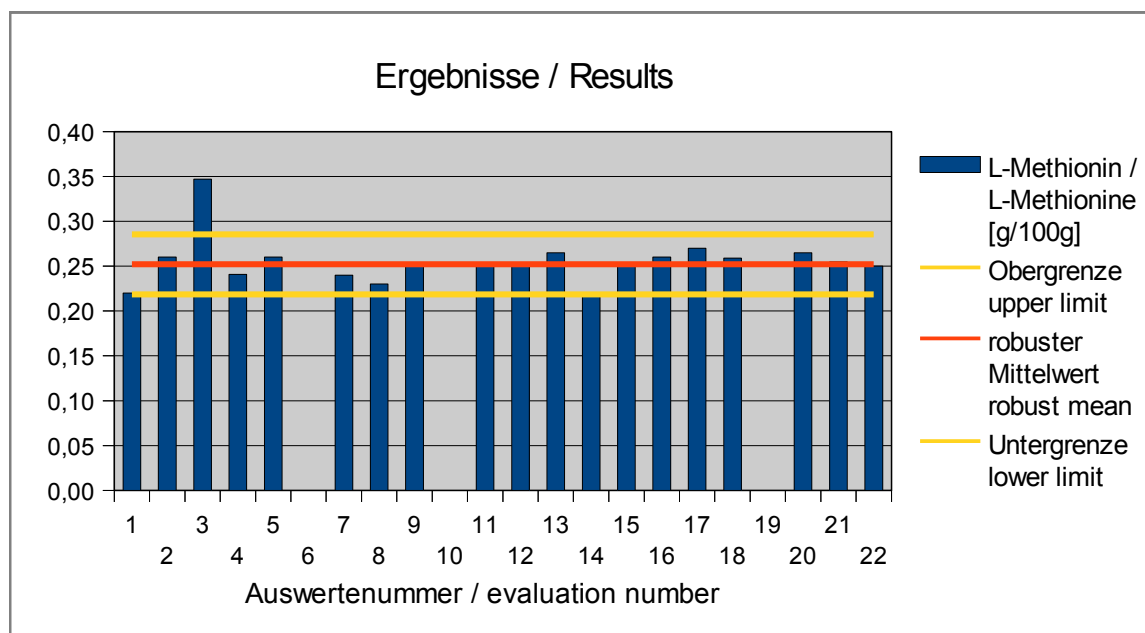
Abb. / Fig. 20: Ergebnisse L-Methionin **/** Results L-Methionine

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	L-Methionin / L-Methionine [g/100g]		Abweichung [g/100g] Deviation [g/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	0,220		−0,0321	−1,9	−2,6	
2	0,260		0,0079	0,47	0,64	
3	0,347		0,0949	5,7	7,7	
4	0,241		−0,0111	−0,66	−0,89	
5	0,260		0,0079	0,47	0,64	
6						
7	0,240		−0,0121	−0,72	−0,97	
8	0,230		−0,0221	−1,3	−1,8	
9	0,250		−0,0021	−0,12	−0,17	
10						
11	0,250		−0,0021	−0,12	−0,17	
12	0,250		−0,0021	−0,12	−0,17	
13	0,265		0,0129	0,77	1,0	
14	0,220		−0,0321	−1,9	−2,6	
15	0,251		−0,0011	−0,06	−0,09	
16	0,260		0,0079	0,47	0,64	
17	0,270		0,0179	1,1	1,4	
18	0,259	*	0,0069	0,41	0,56	
19						
20	0,265		0,0129	0,77	1,0	
21	0,255	*	0,0029	0,17	0,24	
22	0,250		−0,0021	−0,12	−0,17	

* Mean calculated by DLA
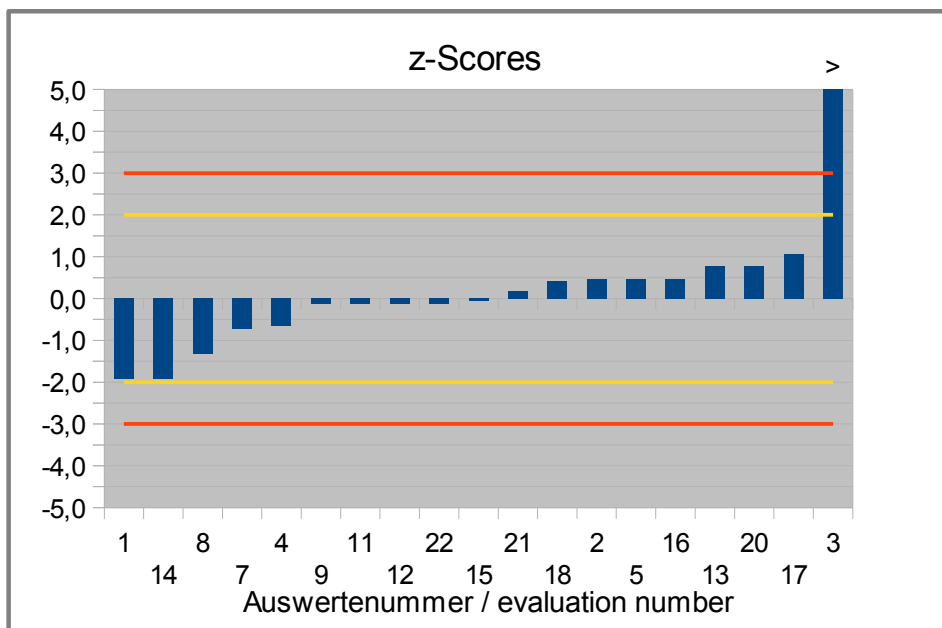
Abb. / Fig. 21:   z-Scores L-Methionin / L-Methionine

4.13 L-Phenylalanine in g/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	18
Number of outliers	0
Mean	0,700
Median	0,695
Robust Mean (X_{pt})	**0,698**
Robust standard deviation (S*)	**0,0621**
Number with 2 replicates	18
Repeatability SD (S_r)	0,0523
Repeatability (CV_r)	7,48%
Reproducibility SD (S_R)	0,0770
Reproducibility (CV_R)	11,0%
Target range:	
Target standard deviation σ_{pt}	**0,0419**
Target standard deviation (for Information)	0,0295
lower limit of target range	**0,614**
upper limit of target range	**0,781**
Quotient S*/σ_{pt}	*1,5*
Standard uncertainty U(X_{pt})	*0,0183*
Results in the target range	*15*
Percent in the target range	*83%*
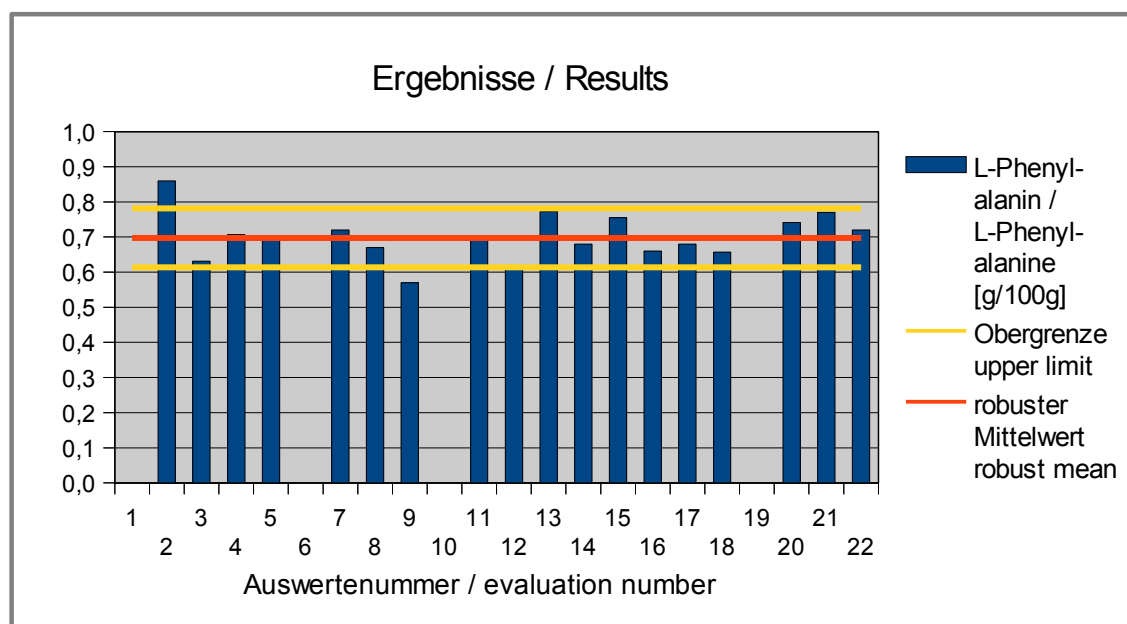
Abb. / Fig. 22: Ergebnisse L-Phenylalanin **/** Results L-Phenylalanine

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer / Evaluation number	L-Phenyl-alanin/ L-Phenyl-alanine [g/100g]	Abweichung [g/100g] / Deviation [g/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis / Remark
1					
2	0,860	0,1624	3,9	5,5	
3	0,631	−0,0666	−1,6	−2,3	
4	0,706	0,0084	0,20	0,28	
5	0,700	0,0024	0,06	0,08	
6					
7	0,720	0,0224	0,53	0,76	
8	0,670	−0,0276	−0,66	−0,94	
9	0,570	−0,1276	−3,0	−4,3	
10					
11	0,690	−0,0076	−0,18	−0,26	
12	0,610	−0,0876	−2,1	−3,0	
13	0,772	0,0744	1,8	2,5	
14	0,680	−0,0176	−0,42	−0,60	
15	0,755	0,0574	1,4	1,9	
16	0,660	−0,0376	−0,90	−1,3	
17	0,680	−0,0176	−0,42	−0,60	
18	0,657 *	−0,0406	−0,97	−1,4	
19					
20	0,741	0,0434	1,0	1,5	
21	0,770 *	0,0724	1,7	2,5	
22	0,720	0,0224	0,53	0,76	

* Mean calculated by DLA
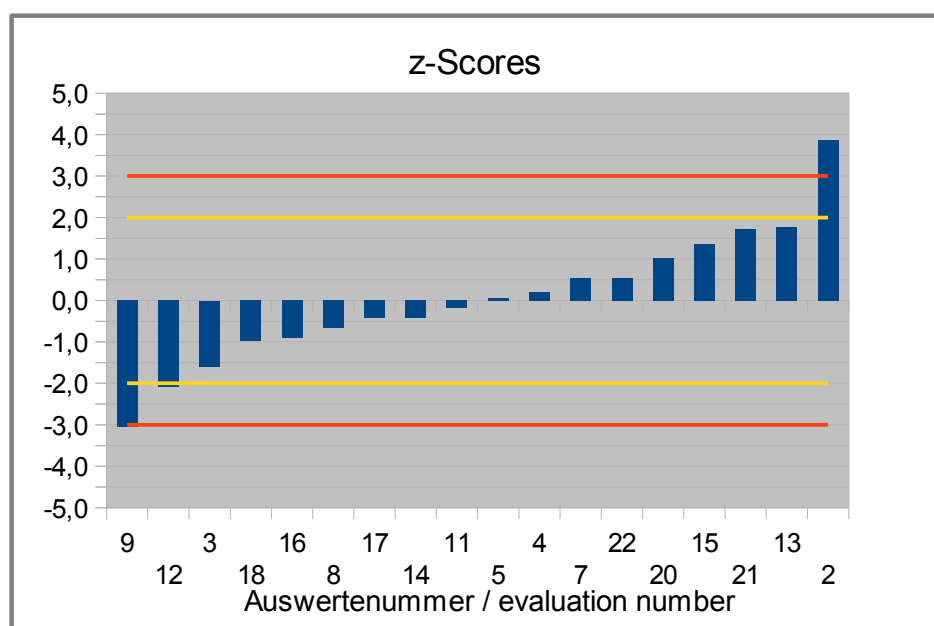
Abb. / Fig. 23:   z-Scores L-Phenylalanin / L-Phenylalanine

4.14 L-Proline in g/100g

Vergleichsuntersuchung / Proficiency Test

| Statistic Data	
Number of results	19
Number of outliers	0
Mean	1,15
Median	1,14
Robust Mean (X_{pt})	**1,16**
Robust standard deviation (S*)	**0,112**
Number with 2 replicates	19
Repeatability SD (S_r)	0,0468
Repeatability (CV_r)	4,06%
Reproducibility SD (S_R)	0,121
Reproducibility (CV_R)	10,5%
Target range:	
Target standard deviation σ_{pt}	**0,0702**
Target standard deviation (for Information)	0,0454
lower limit of target range	**1,02**
upper limit of target range	**1,30**
Quotient S*/σ_{pt}	*1,6*
Standard uncertainty $U_{(Xpt)}$	*0,0321*
Results in the target range	*14*
Percent in the target range	*74%*

Abb. / Fig. 24: 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	1,12	−0,040	−0,58	−0,89	
2	1,09	−0,070	−1,0	−1,6	
3	1,31	0,149	2,1	3,3	
4	1,14	−0,025	−0,36	−0,56	
5	0,85	−0,310	−4,4	−6,8	
6					
7	1,12	−0,040	−0,58	−0,89	
8	1,31	0,150	2,1	3,3	
9	1,08	−0,080	−1,1	−1,8	
10					
11	1,12	−0,040	−0,58	−0,89	
12	1,10	−0,060	−0,86	−1,3	
13	1,16	0,000	−0,01	−0,01	
14	1,16	0,000	−0,01	−0,01	
15	1,15	−0,010	−0,15	−0,23	
16	1,34	0,180	2,6	4,0	
17	1,08	−0,080	−1,1	−1,8	
18	1,04 *	−0,118	−1,7	−2,6	
19					
20	1,21	0,048	0,68	1,0	
21	1,22 *	0,055	0,78	1,2	
22	1,33	0,170	2,4	3,7	

* Mean calculated by DLA
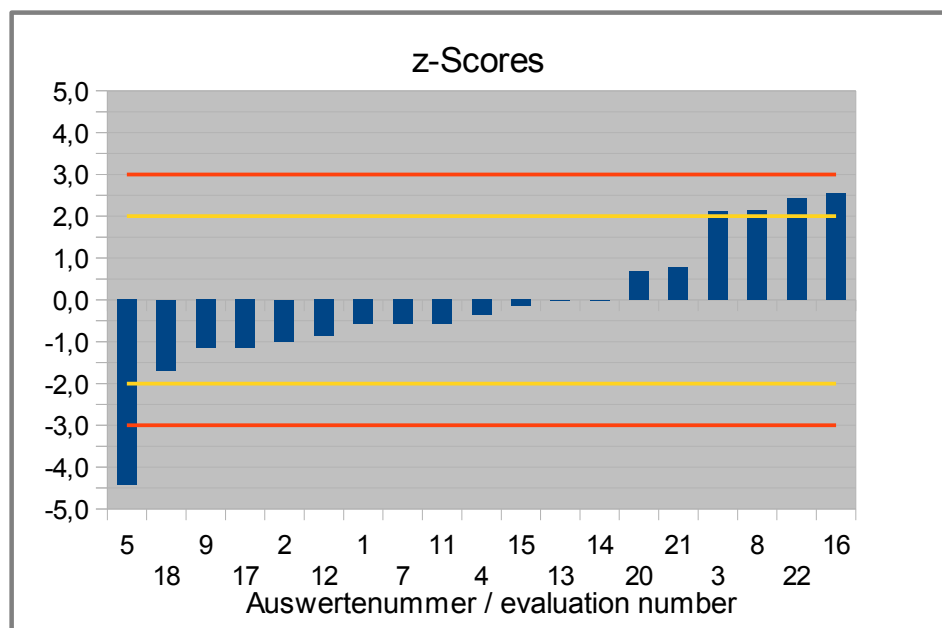
Abb. / Fig. 25:   z-Scores L-Prolin / L-Proline

4.15 L-Serine in g/100g

Vergleichsuntersuchung / Proficiency Test

| Statistic Data	
Number of results	19
Number of outliers	–
Mean	0,736
Median	0,695
Robust Mean (X_{pt})	**0,693**
Robust standard deviation (S*)	**0,0311**
Number with 2 replicates	17
Repeatability SD (S_r)	0,0297
Repeatability (CV_r)	4,28%
Reproducibility SD (S_R)	0,033
Reproducibility (CV_R)	4,73%
Target range:	
Target standard deviation σ_{pt}	**0,0293**
Target standard deviation (for Information)	0,0513
lower limit of target range	**0,634**
upper limit of target range	**0,751**
Quotient S*/σ_{pt}	*1,1*
Standard uncertainty U$_{(Xpt)}$	*0,00892*
Results in the target range	*17*
Percent in the target range	*89%*
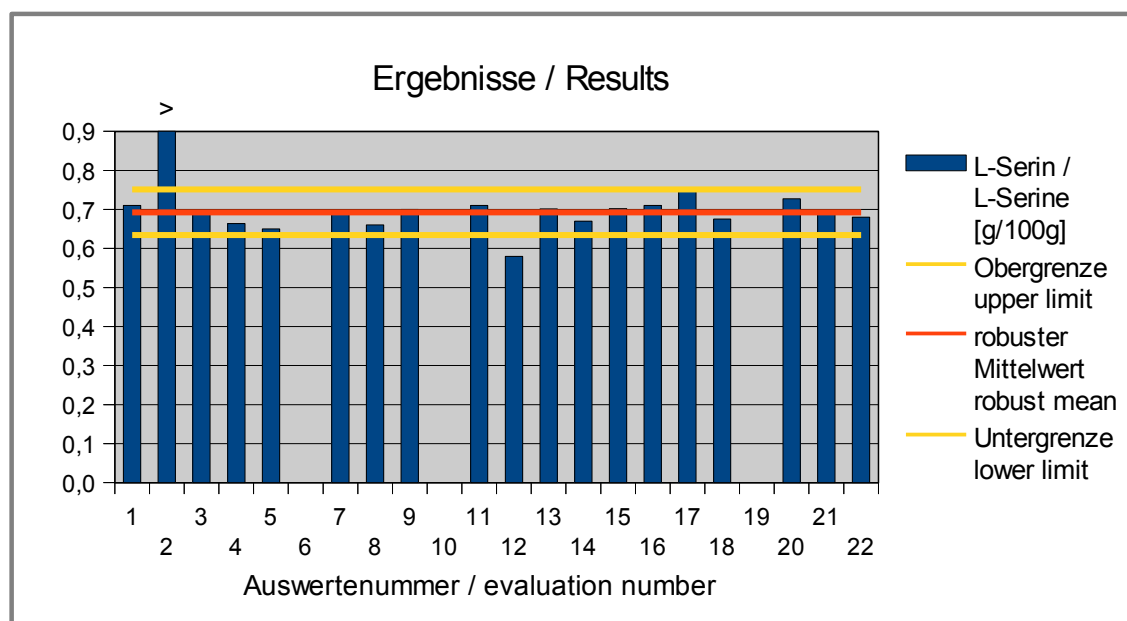
Abb. / Fig. 26: Ergebnisse L-Serin / Results L-Serine

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	0,710	0,0173	0,59	0,34	
2	1,61	0,9173	31	18	
3	0,693	0,0003	0,01	0,01	
4	0,664	−0,0292	−1,0	−0,57	
5	0,650	−0,0427	−1,5	−0,83	
6					
7	0,690	−0,0027	−0,09	−0,05	
8	0,660	−0,0327	−1,1	−0,64	
9	0,700	0,0073	0,25	0,14	
10					
11	0,710	0,0173	0,59	0,34	
12	0,580	−0,1127	−3,8	−2,2	
13	0,701	0,0083	0,28	0,16	
14	0,670	−0,0227	−0,78	−0,44	
15	0,702	0,0093	0,32	0,18	
16	0,710	0,0173	0,59	0,34	
17	0,750	0,0573	2,0	1,1	
18	0,676 *	−0,0172	−0,59	−0,34	
19					
20	0,727	0,0343	1,2	0,67	
21	0,695 *	0,0023	0,08	0,04	
22	0,680	−0,0127	−0,43	−0,25	

* Mean calculated by DLA
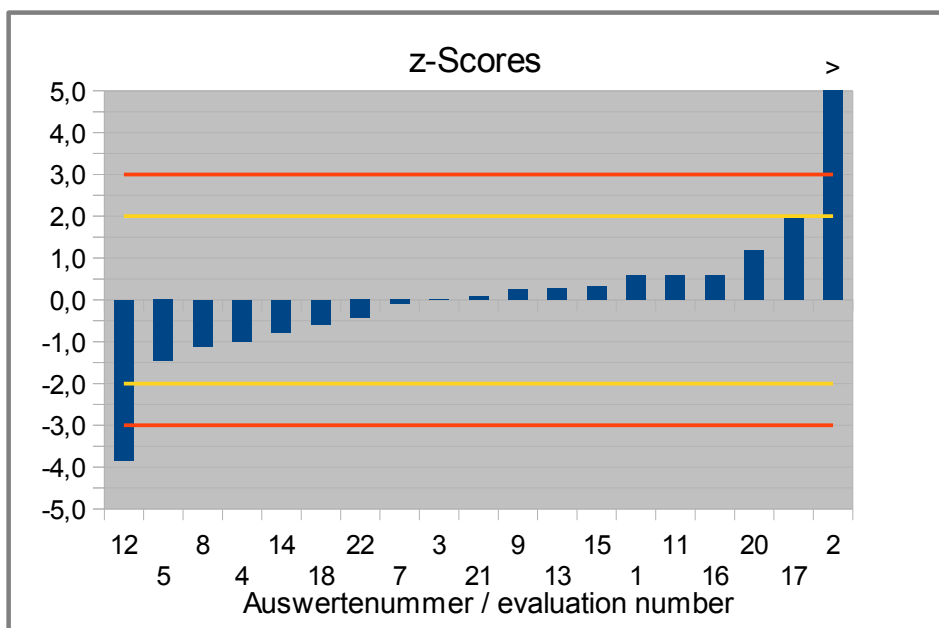
Abb. / Fig. 27:   z-Scores L-Serin / L-Serine

4.16 L-Threonine in g/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	18
Number of outliers	–
Mean	0,796
Median	0,769
Robust Mean (X_{pt})	**0,771**
Robust standard deviation (S*)	**0,0498**
Number with 2 replicates	16
Repeatability SD (S_r)	0,0323
Repeatability (CV_r)	4,24%
Reproducibility SD (S_R)	0,0460
Reproducibility (CV_R)	6,05%
Target range:	
Target standard deviation σ_{pt}	**0,0460**
Target standard deviation (for Information)	0,0321
lower limit of target range	**0,679**
upper limit of target range	**0,863**
Quotient S*/σ_{pt}	*1,1*
Standard uncertainty $U_{(Xpt)}$	*0,0147*
Results in the target range	*16*
Percent in the target range	*89%*
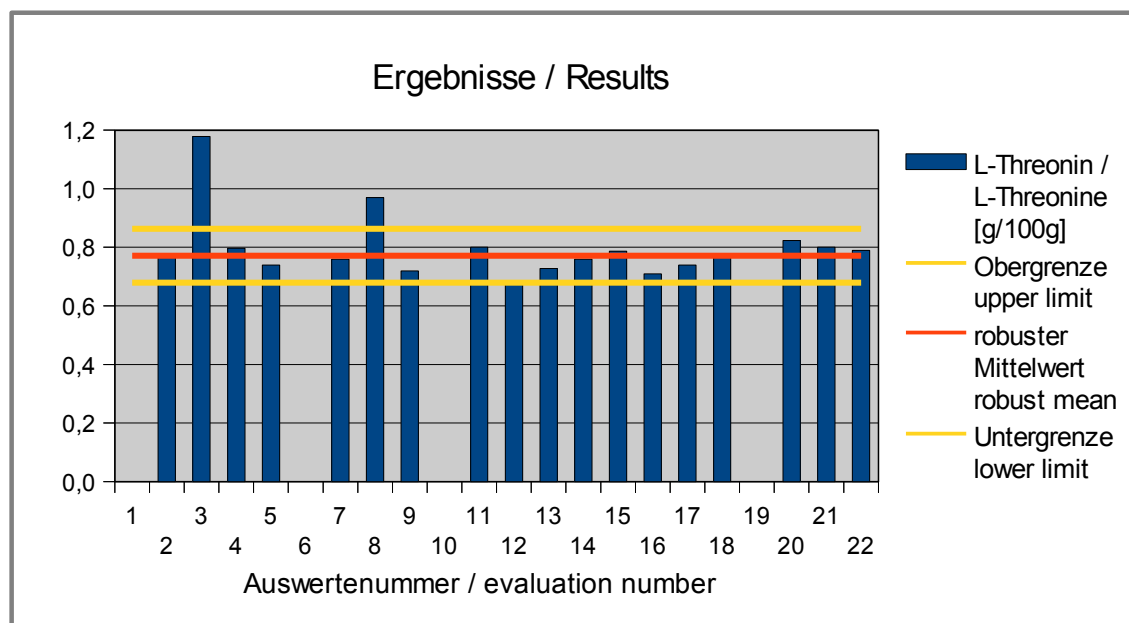
Abb. / Fig. 28: Ergebnisse L-Threonin **/** Results L-Treonine

Ergebnisse der Teilnehmer:
Results of Participants:

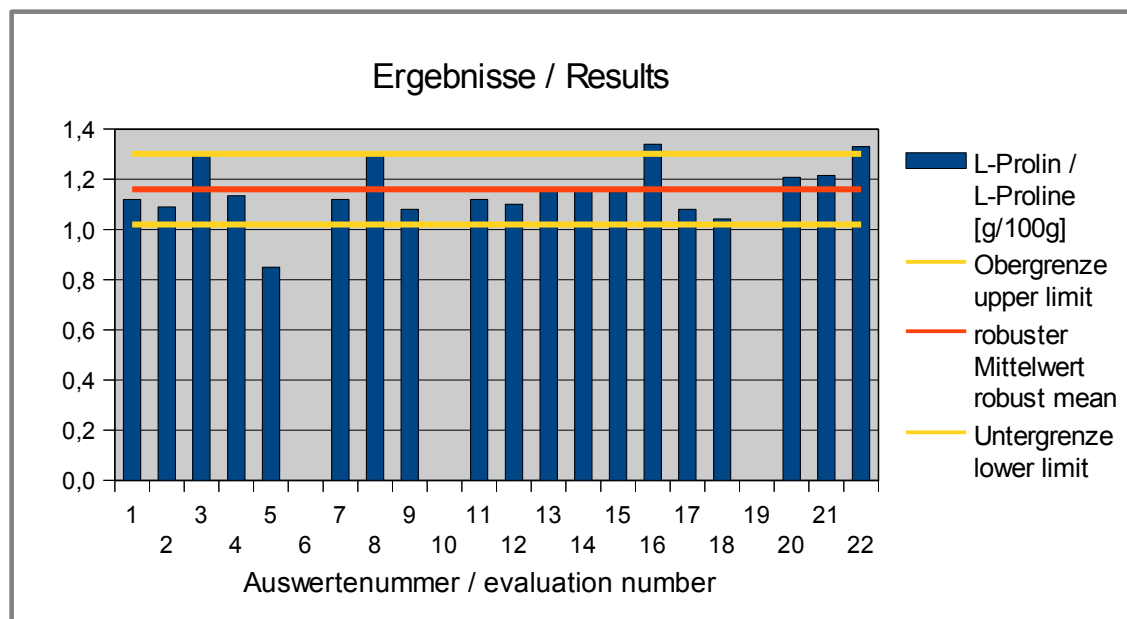| Auswerte-nummer Evaluation number	L-Threonin / L-Threonine [g/100g]	Abweichung [g/100g] Deviation [g/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1					
2	0,770	−0,0012	−0,03	−0,04	
3	1,18	0,4068	8,9	13	
4	0,797	0,0258	0,56	0,80	
5	0,740	−0,0312	−0,68	−0,97	
6					
7	0,760	−0,0112	−0,24	−0,35	
8	0,970	0,1988	4,3	6,2	
9	0,720	−0,0512	−1,1	−1,6	
10					
11	0,800	0,0288	0,63	0,90	
12	0,680	−0,0912	−2,0	−2,8	
13	0,728	−0,0432	−0,94	−1,3	
14	0,760	−0,0112	−0,24	−0,35	
15	0,787	0,0158	0,34	0,49	
16	0,710	−0,0612	−1,3	−1,9	
17	0,740	−0,0312	−0,68	−0,97	
18	0,768 *	−0,0032	−0,07	−0,10	
19					
20	0,823	0,0518	1,1	1,6	
21	0,800 *	0,0288	0,63	0,90	
22	0,790	0,0188	0,41	0,59	

* Mean calculated by DLA
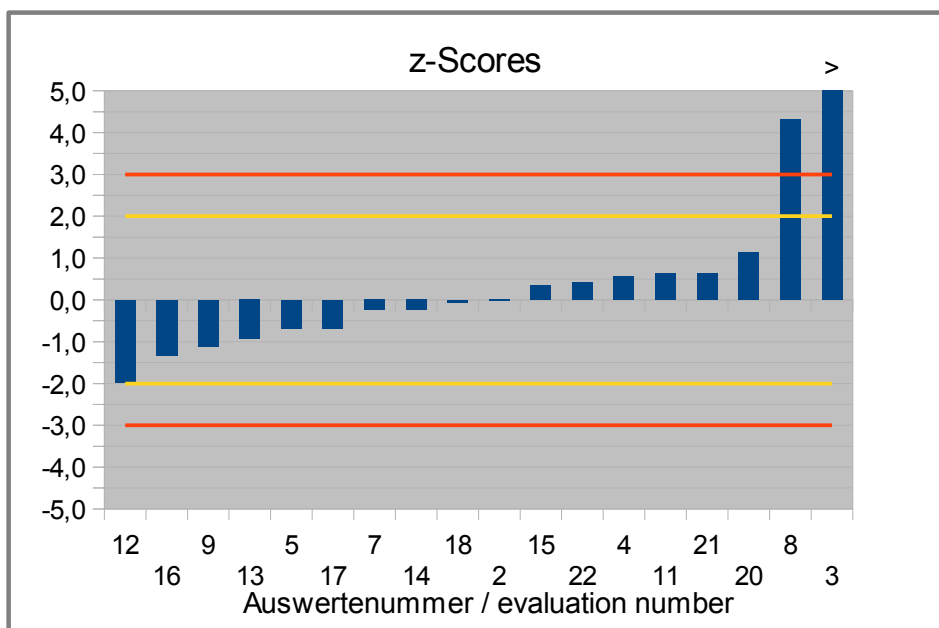
Abb. / Fig. 29:   z-Scores L-Threonin / L-Threonine

4.17 L-Tryptophan in g/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	15
Number of outliers	0
Mean	0,305
Median	0,302
Robust Mean (X_{pt})	**0,305**
Robust standard deviation (S*)	**0,0177**
Number with 2 replicates	15
Repeatability SD (S$_r$)	0,00799
Repeatability (CV$_r$)	2,63%
Reproducibility SD (S$_R$)	0,0163
Reproducibility (CV$_R$)	5,36%
Target range:	
Target standard deviation σ_{pt}	**0,0146**
Target standard deviation (for Information)	0,0603
lower limit of target range	**0,276**
upper limit of target range	**0,334**
Quotient S*/σ_{pt}	*1,2*
Standard uncertainty U(X_{pt})	*0,00570*
Results in the target range	*15*
Percent in the target range	*100%*
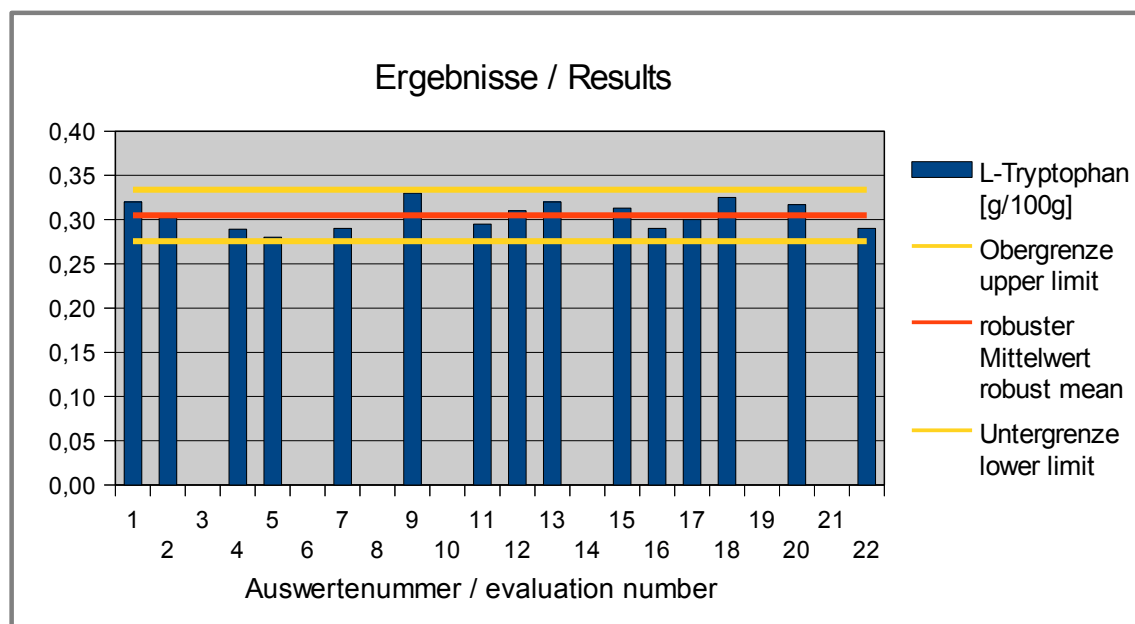
Abb. / Fig. 30: Ergebnisse L-Tryptophan **/** Results L-Tryptophan

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	0,320	0,0153	1,0	0,25	
2	0,302	−0,0027	−0,19	−0,05	
3					
4	0,289	−0,0157	−1,1	−0,26	
5	0,280	−0,0247	−1,7	−0,41	
6					
7	0,290	−0,0147	−1,0	−0,24	
8					
9	0,330	0,0253	1,7	0,42	
10					
11	0,295	−0,0097	−0,67	−0,16	
12	0,310	0,0053	0,36	0,09	
13	0,320	0,0153	1,0	0,25	
14					
15	0,313	0,0083	0,57	0,14	
16	0,290	−0,0147	−1,0	−0,24	
17	0,300	−0,0047	−0,32	−0,08	
18	0,325 *	0,0203	1,4	0,34	
19					
20	0,317	0,0123	0,84	0,20	
21					
22	0,290	−0,0147	−1,0	−0,24	

* Mean calculated by DLA
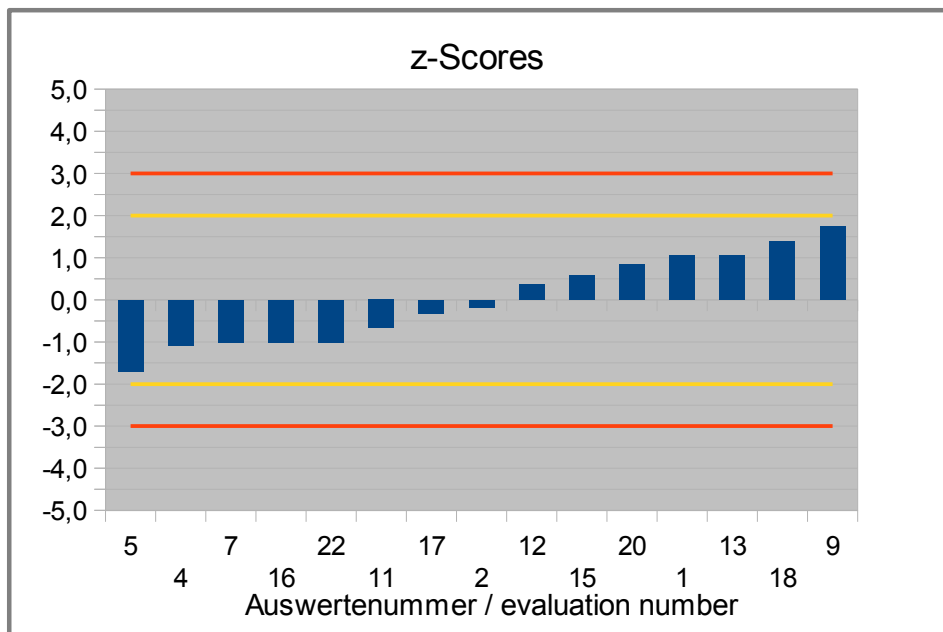
Abb. / Fig. 31:   z-Scores   L-Tryptophan

4.18 L-Tyrosine in g/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	20
Number of outliers	-
Mean	0,691
Median	0,685
Robust Mean (X_{pt})	**0,684**
Robust standard deviation (S*)	**0,0445**
Number with 2 replicates	17
Repeatability SD (S_r)	0,0165
Repeatability (CV_r)	2,45%
Reproducibility SD (S_R)	0,0441
Reproducibility (CV_R)	6,53%
Target range:	
Target standard deviation σ_{pt}	**0,0502**
Target standard deviation (for Information)	0,0290
lower limit of target range	**0,584**
upper limit of target range	**0,784**
Quotient S*/σ_{pt}	*0,89*
Standard uncertainty $U_{(Xpt)}$	*0,0124*
Results in the target range	*16*
Percent in the target range	*80%*
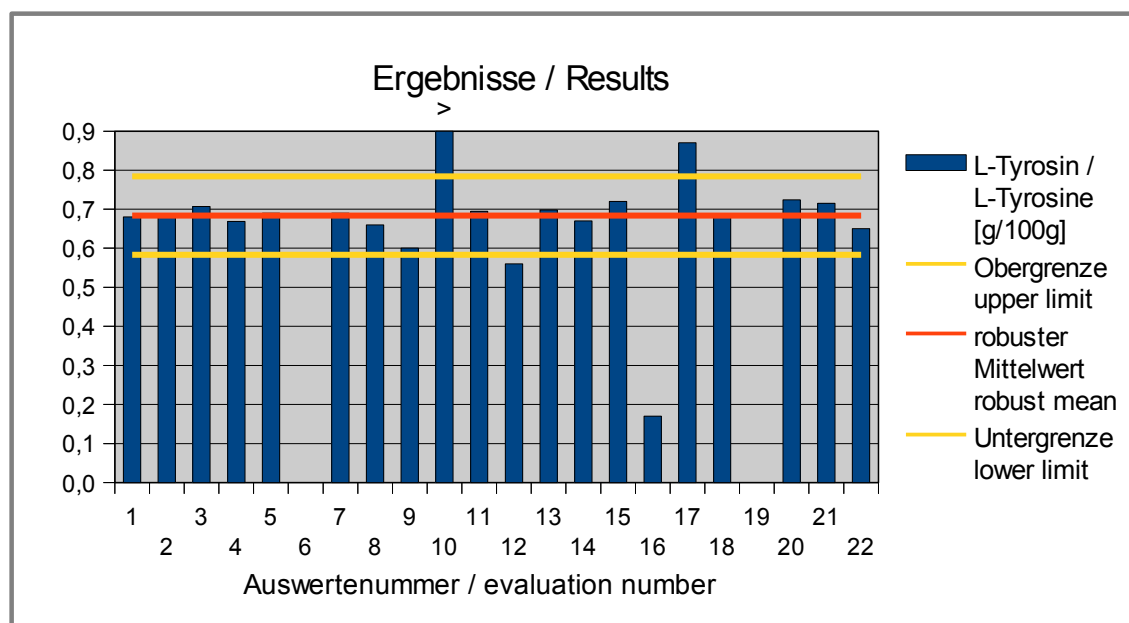
Abb. / Fig. 32: Ergebnisse L-Tyrosin **/** Results L-Tyrosine

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer / Evaluation number	L-Tyrosin / L-Tyrosine [g/100g]	Abweichung [g/100g] Deviation [g/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis / Remark
1	0,680	−0,0040	−0,08	−0,14	
2	0,680	−0,0040	−0,08	−0,14	
3	0,707	0,0230	0,46	0,79	
4	0,669	−0,0155	−0,31	−0,53	
5	0,690	0,0060	0,12	0,21	
6					
7	0,690	0,0060	0,12	0,21	
8	0,660	−0,0240	−0,48	−0,83	
9	0,600	−0,0840	−1,7	−2,9	
10	1,30	0,6160	12	21	
11	0,695	0,0110	0,22	0,38	
12	0,560	−0,1240	−2,5	−4,3	
13	0,698	0,0140	0,28	0,48	
14	0,670	−0,0140	−0,28	−0,48	
15	0,720	0,0360	0,72	1,2	
16	0,170	−0,5140	−10	−18	
17	0,870	0,1860	3,7	6,4	
18	0,679 *	−0,0050	−0,10	−0,17	
19					
20	0,724	0,0400	0,80	1,4	
21	0,715 *	0,0310	0,62	1,1	
22	0,650	−0,0340	−0,68	−1,2	

* Mean calculated by DLA
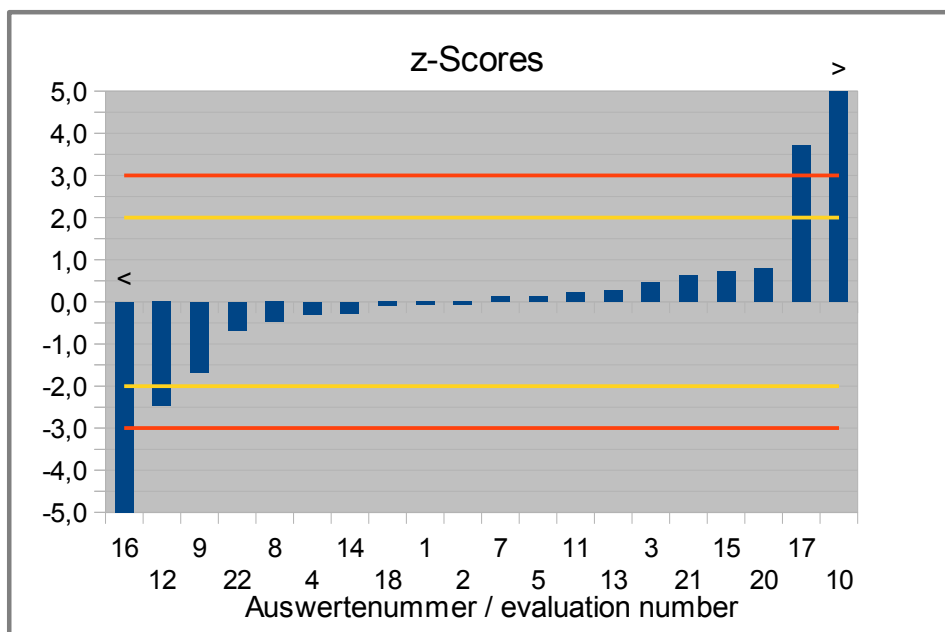
Abb. / Fig. 33:   z-Scores L-Tyrosin / L-Tyrosine

4.19 L-Valine in g/100g

Vergleichsuntersuchung / Proficiency Test

| Statistic Data	
Number of results	19
Number of outliers	0
Mean	0,983
Median	0,988
Robust Mean (X_{pt})	**0,983**
Robust standard deviation (S*)	**0,0950**
Number with 2 replicates	19
Repeatability SD (S_r)	0,0743
Repeatability (CV_r)	7,55%
Reproducibility SD (S_R)	0,125
Reproducibility (CV_R)	12,7%
Target range:	
Target standard deviation σ_{pt}	**0,048**
Target standard deviation (for Information)	0,0394
lower limit of target range	**0,888**
upper limit of target range	**1,08**
Quotient S*/σ_{pt}	*2,0*
Standard uncertainty $U_{(Xpt)}$	*0,0272*
Results in the target range	*13*
Percent in the target range	*68%*
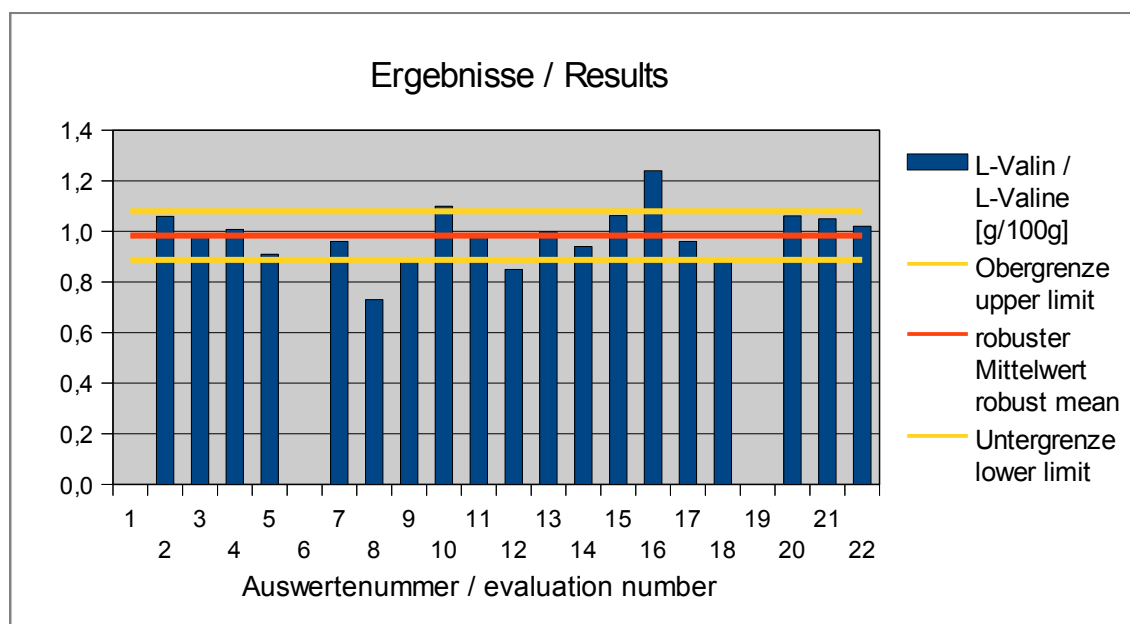
Abb. / Fig. 34: Ergebnisse L-Valin / Results L-Valine

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					
2	1,06	0,0767	1,6	1,9	
3	0,988	0,0047	0,10	0,12	
4	1,01	0,0247	0,52	0,63	
5	0,910	−0,0733	−1,5	−1,9	
6					
7	0,960	−0,0233	−0,49	−0,59	
8	0,730	−0,2533	−5,3	−6,4	
9	0,880	−0,1033	−2,16	−2,6	
10	1,10	0,1167	2,4	3,0	
11	0,985	0,0017	0,04	0,04	
12	0,850	−0,1333	−2,8	−3,4	
13	0,998	0,0147	0,31	0,37	
14	0,940	−0,0433	−0,91	−1,1	
15	1,06	0,0797	1,7	2,0	
16	1,24	0,2567	5,4	6,5	
17	0,960	−0,0233	−0,49	−0,59	
18	0,882 *	−0,1013	−2,1	−2,6	
19					
20	1,06	0,0787	1,6	2,0	
21	1,05 *	0,0667	1,4	1,7	
22	1,020	0,0367	0,77	0,93	

* Mean calculated by DLA
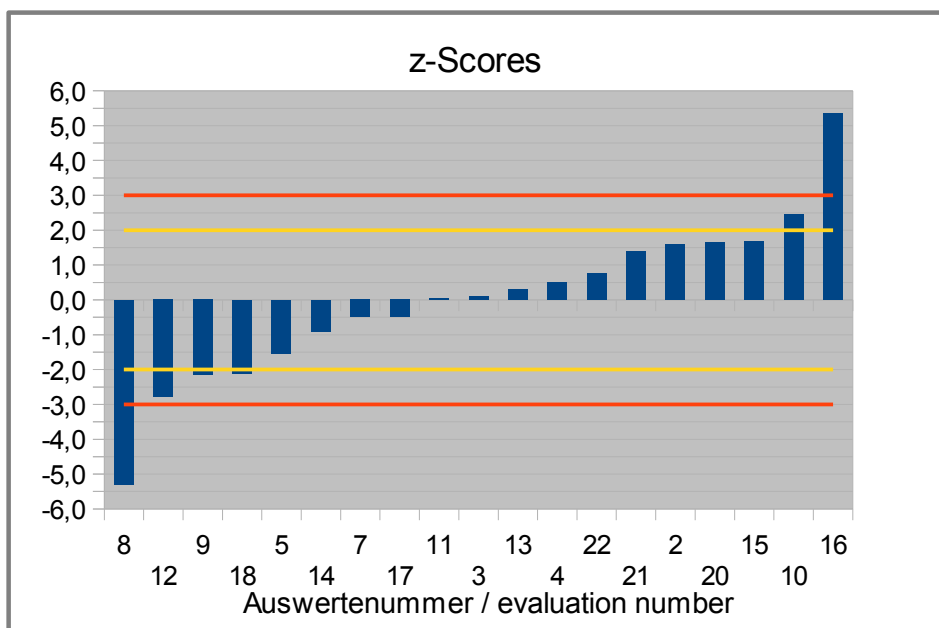
Abb. / Fig. 35:   z-Scores L-Valin / L-Valine

4.20 Taurine in g/100g

Vergleichsuntersuchung / Proficiency Test

| Statistic Data	
Number of results	13
Number of outliers	–
Mean	0,0329
Median	0,0300
Robust Mean (X_{pt})	**0,0317**
Robust standard deviation (S*)	**0,00378**
Number with 2 replicates	12
Repeatability SD (S_r)	0,00117
Repeatability (CV_r)	3,73%
Reproducibility SD (S_R)	0,00360
Reproducibility (CV_R)	11,5%
Target range:	
Target standard deviation σ_{pt}	**0,00213**
lower limit of target range	**0,0274**
upper limit of target range	**0,0359**
Quotient S*/σ_{pt}	*1,8*
Standard uncertainty U(X_{pt})	*0,00131*
Results in the target range	*10*
Percent in the target range	*77%*
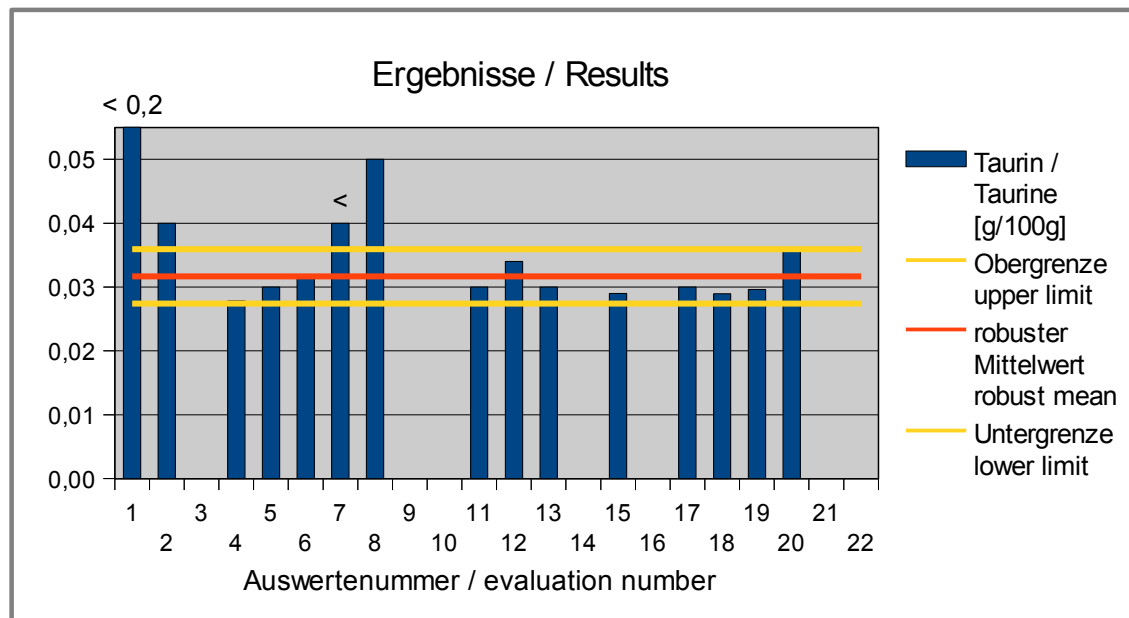
Abb. / Fig. 36: Ergebnisse Taurin **/** Results Taurine

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Taurin / Taurine [g/100g]	Abweichung [g/100g] Deviation [g/100g]	z-Score (σ_{pt})	Hinweis Remark
1	< 0,19			
2	0,0400	0,00833	3,9	
3				
4	0,0278	−0,00387	−1,8	
5	0,0300	−0,00167	−0,79	
6	0,0317	0,00003	0,01	
7	< 0,04			
8	0,0500	0,01833	8,6	
9				
10				
11	0,0300	−0,00167	−0,79	
12	0,0340	0,00233	1,1	
13	0,0300	−0,00167	−0,79	
14				
15	0,0290	−0,00267	−1,3	
16				
17	0,0300	−0,00167	−0,79	
18	0,0290 *	−0,00272	−1,3	
19	0,0296	−0,00207	−0,97	
20	0,0360	0,00433	2,0	
21				
22				

* Mean calculated by DLA
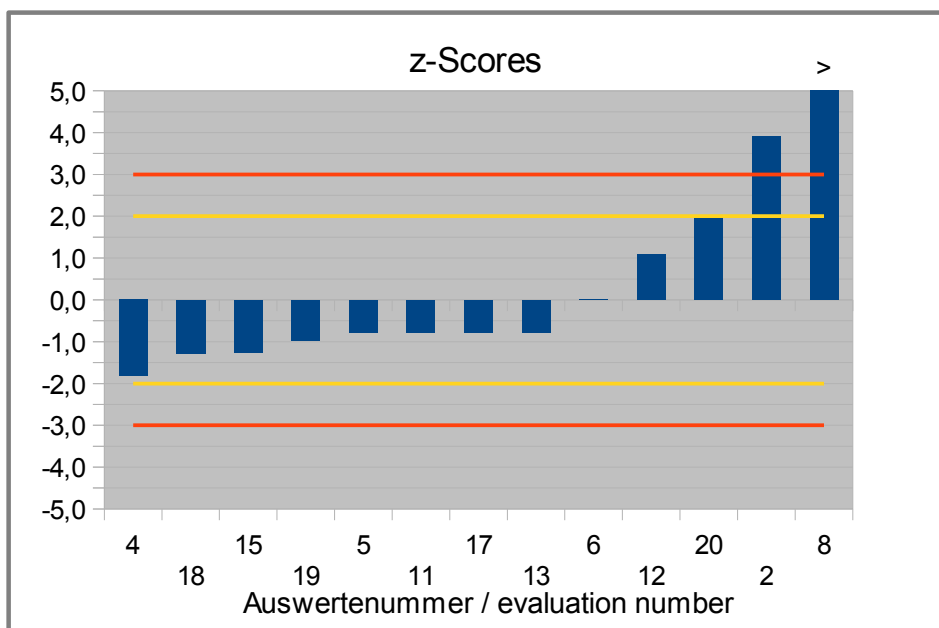
Abb. / Fig. 37: z-Scores Taurin / Taurine

4.21 Further Parameter: L-Glutamine in g/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	6
Number of outliers	0
Mean	1,32
Median	1,28
Robust Mean (X_{pt})	**1,32**
Robust standard deviation (S*)	**0,179**
Number with 2 replicates	6
Repeatability SD (S_r)	0,0617
Repeatability (CV_r)	4,67%
Reproducibility SD (S_R)	0,163
Reproducibility (CV_R)	12,3%
Target range:	
Target standard deviation σ_{pt}	**0,104**
lower limit of target range	**1,11**
upper limit of target range	**1,53**
Quotient S*/σ_{pt}	*1,7*
Standard uncertainty U$_{(Xpt)}$	*0,0912*
Results in the target range	*5*
Percent in the target range	*83%*
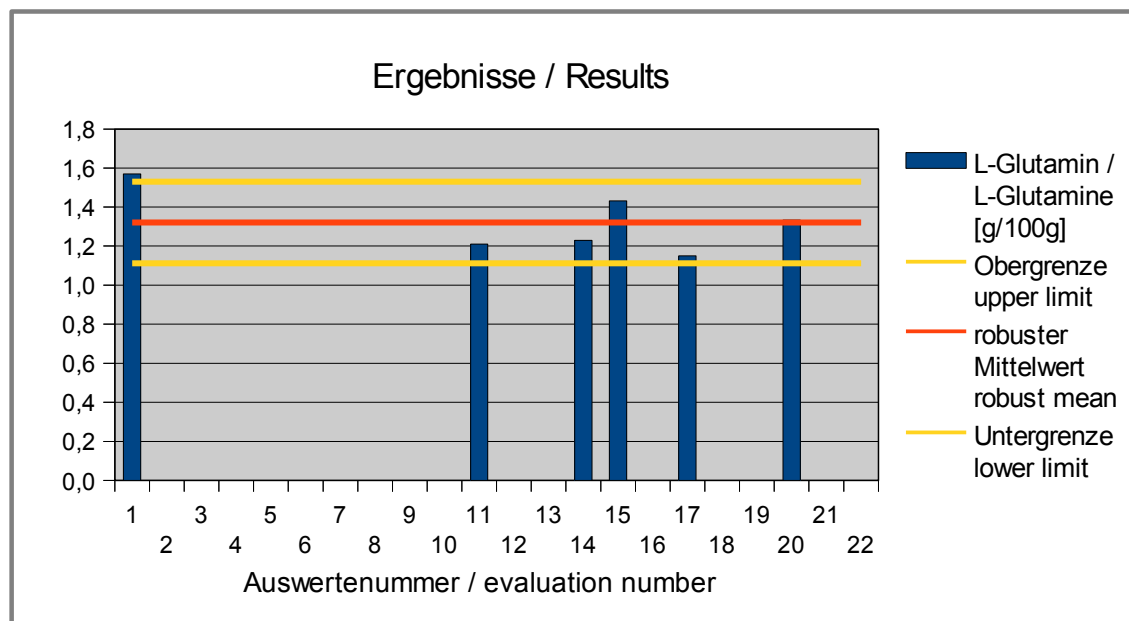
Abb. / Fig. 38: Ergebnisse L-Glutamin **/** Results L-Glutamine

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer / Evaluation number	L-Glutamin / L-Glutamine [g/100g]	Abweichung [g/100g] / Deviation [g/100g]	z'-Score (σ_{pt})	Hinweis / Remark
1	1,57	0,249	2,4	
2				
3				
4				
5				
6				
7				
8				
9				
10				
11	1,21	−0,111	−1,1	
12				
13				
14	1,23	−0,091	−0,87	
15	1,43	0,111	1,1	
16				
17	1,15	−0,171	−1,6	
18				
19				
20	1,33	0,012	0,1	
21				
22				

Abb. / Fig. 39:   z'-Scores L-Glutamin / L-Glutamine

5. Documentation

5.1 Details by the participants

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

5.1.1 Primary Data

Parameter	Participant	Unit	Sample I DLA No.	Sample II DLA No.	Date of analysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantification	Incl. RR	Recovery rate
					day/month					yes/no	in %
L-Alanin/ L-Alanine	1	g/100g	31	49	27.09.19					no	
	2	g/100g	12	68	01+02/10	0,59	0,52	0,67		yes	97-99%
	3	g/100g	15	65	16.10.	0,622	0,653	0,591	0,006	no	
	4	g/100g	37	43	15.10.19	0,6055	0,586	0,625	0,01	no	
	5	g/100g	7	73	24.09.	0,59	0,61	0,57	0,001	no	
	6	g/100g	24	56							
	7	g/100g	3	77	15.10.19	0,61	0,62	0,6	< 0.04	no	97
	8	g/100g	34	46	11.10.19	0,62	0,84	0,39		no	
	9	g/100g	36	44	15.10.19	0,55	0,57	0,52	0,09	no	
	10	g/100g	64	16						no	
	11	g/100g	10	70	17.09.19	0,595	0,56	0,63	0,01	no	
	12	g/100g	14	66	03.10.19	0,52	0,54	0,49	0,034	no	
	13	g/100g	35	45	26.10.19	0,6	0,685	0,515			
	14	g/100g	22	58	27.9.	0,57	0,58	0,57		no	
	15	g/100g	30	50	div.	0,592	0,589	0,596	0,01	yes	98
	16	g/100g	5	75	20.09.19	0,64	0,66	0,62	0,00001		
	17	g/100g	28	52	19.09.19	0,53	0,52	0,54	0,01	no	-
	18	g/100g	26	54	02.10.19		0,568	0,573	<0,01	no	
	19	g/100g	13	67							
	20	g/100g	19	61	13.09.19	0,643	0,619	0,667	0,00535	no	
	21	g/100g	23	57			0,63	0,57			
	22	g/100g	38	42	23.09.19	0,62	0,63	0,6	0,05	no	-

Parameter	Participant	Unit	Sample I DLA No.	Sample II DLA No.	Date of analysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantification	Incl. RR	Recovery rate
					day/month					yes/no	in %
L-Arginin/ L-Arginine	1	g/100g	31	49							
	2	g/100g	12	68	01+02/10	1,05	1,05	1,05		yes	97-99%
	3	g/100g	15	65		0,988	0,972	1,004	0,013		
	4	g/100g	37	43	15.10.19	1,04	1,05	1,03	0,01	no	
	5	g/100g	7	73	24.09.	0,84	0,85	0,83	0,001	no	
	6	g/100g	24	56							
	7	g/100g	3	77	15.10.19	1,01	0,97	1,04	< 0.04	no	104
	8	g/100g	34	46	11.10.19	0,95	0,98	0,92		no	
	9	g/100g	36	44	15.10.19	1	1,03	0,97	0,09	no	
	10	g/100g	64	16							
	11	g/100g	10	70	17.09.19	0,995	1	0,99	0,02	no	
	12	g/100g	14	66	03.10.19	0,87	0,84	0,89	0,091	no	
	13	g/100g	35	45	26.10.19	1,18	1,14	1,22			
	14	g/100g	22	58		1,04	1,05	1,02			
	15	g/100g	30	50	div.	1,06	1,051	1,069	0,01	yes	98
	16	g/100g	5	75	20.09.19	0,97	1,02	0,93	0,00001		
	17	g/100g	28	52	19.09.19	1,06	1,03	1,09	0,01	no	-
	18	g/100g	26	54	02.10.19		1	0,99	<0,01	no	
	19	g/100g	13	67							
	20	g/100g	19	61	13.09.19	1,059	1,059	1,058	0,01915	no	
	21	g/100g	23	57			1,08	1,06			
	22	g/100g	38	42	23.09.19	0,81	0,84	0,78	0,05	no	-

Parameter	Participant	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana-lysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifi-cation	Incl. RR	Recovery rate
					day/month					yes/no	in %
	1	g/100g	31	49		0,9	0,91	0,88			
	2	g/100g	12	68	01+02/10	0,96	0,96	0,97		yes	97-99%
	3	g/100g	15	65		1,012	1,005	1,018	0,009		
	4	g/100g	37	43	15.10.19	0,962	0,957	0,967	0,01	no	
	5	g/100g	7	73	24.09.	0,91	0,92	0,89	0,001	no	
	6	g/100g	24	56							
	7	g/100g	3	77	15.10.19	1	0,95	1,05	< 0.04	no	92
	8	g/100g	34	46	11.10.19	0,79	0,83	0,75		no	
	9	g/100g	36	44	15.10.19	0,93	0,95	0,9	0,13	no	
	10	g/100g	64	16							
L-Asparaginsäure/ L-Aspartic acid	11	g/100g	10	70	17.09.19	0,91	0,92	0,9	0,01	no	
	12	g/100g	14	66	03.10.19	0,85	0,81	0,89	0,05	no	
	13	g/100g	35	45	26.10.19	1,055	1,01	1,1			
	14	g/100g	22	58		0,98	0,98	0,97			
	15	g/100g	30	50	div.	1,012	0,986	1,039	0,01	yes	99
	16	g/100g	5	75	20.09.19	0,83	0,86	0,79	0,00001		
	17	g/100g	28	52	19.09.19	0,99	0,97	1,01	0,01	no	-
	18	g/100g	26	54	02.10.19		0,959	0,968	<0,01	no	
	19	g/100g	13	67							
	20	g/100g	19	61	13.09.19	0,9945	0,993	0,996	0,01295	no	
	21	g/100g	23	57			0,98	0,99			
	22	g/100g	38	42	23.09.19	0,96	0,97	0,94	0,05	no	-

Parameter	Participant	Unit	Sample I DLA No.	Sample II DLA No.	Date of analysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantification	Incl. RR	Recovery rate
					day/month					yes/no	in %
L-Cystein/ L-Cysteine	1	g/100g	31	49							
	2	g/100g	12	68						yes	97-99%
	3	g/100g	15	65							
	4	g/100g	37	43	15.10.19	\	\	\	0,01	no	
	5	g/100g	7	73	24.09.					no	
	6	g/100g	24	56							
	7	g/100g	3	77	15.10.19	not analysed	-	-			
	8	g/100g	34	46	11.10.19					no	
	9	g/100g	36	44	15.10.19				0,07	no	
	10	g/100g	64	16							
	11	g/100g	10	70	17.09.19	< 0,02	< 0,02	< 0,02	0,02	no	
	12	g/100g	14	66							
	13	g/100g	35	45							
	14	g/100g	22	58							
	15	g/100g	30	50							
	16	g/100g	5	75							
	17	g/100g	28	52	26.09.19				0,1	no	-
	18	g/100g	26	54							
	19	g/100g	13	67							
	20	g/100g	19	61	N/A	N/A	N/A	N/A	N/A	N/A	N/A
	21	g/100g	23	57							
	22	g/100g	38	42	23.09.19	-	-	-	0,05	no	-

Parameter	Participant	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana-lysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifi-cation	Incl. RR	Recovery rate
					day/month					yes/no	in %
L-Cystin/ L-Cystine	1	g/100g	31	49		< 0,40	< 0,40	< 0,40	0,4		
	2	g/100g	12	68	01+02/10	0,79	0,79	0,79		yes	97-99%
	3	g/100g	15	65							
	4	g/100g	37	43	15.10.19	\	\	\	0,01	no	
	5	g/100g	7	73	24.09.					no	
	6	g/100g	24	56							
	7	g/100g	3	77	15.10.19	0,15	0,15	0,14	< 0.04	no	100
	8	g/100g	34	46	11.10.19	0,88	0,83	0,92		no	
	9	g/100g	36	44							
	10	g/100g	64	16							
	11	g/100g	10	70	17.09.19	0,22	0,19	0,25	0,02	no	
	12	g/100g	14	66	08.10.19	0,21	0,21	0,2	0,006	no	
	13	g/100g	35	45	26.10.19	0,391	0,396	0,386			
	14	g/100g	22	58		0,41	0,42	0,4			
	15	g/100g	30	50	div.	0,415	0,426	0,403	0,01	yes	90
	16	g/100g	5	75							
	17	g/100g	28	52	19.09.19	0,38	0,4	0,36	0,01	no	-
	18	g/100g	26	54							
	19	g/100g	13	67							
	20	g/100g	19	61	13.09.19	0,4	0,41	0,39	0,031	no	
	21	g/100g	23	57							
	22	g/100g	38	42	23.09.19	0,15	0,15	0,15	0,05	no	-

Parameter	Participant	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana-lysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifi-cation	Incl. RR	Recovery rate
					day/month					yes/no	in %
L-Glutaminsäure/ L-Glutamic acid	1	g/100g	31	49		< 0,22	< 0,22	< 0,22	0,22		
	2	g/100g	12	68	01+02/10	< 0,01	< 0,01	< 0,01		yes	97-99%
	3	g/100g	15	65		0,146	0,146	0,145	0,01		
	4	g/100g	37	43	15.10.19	<LQ	<LQ	<LQ	0,01	no	
	5	g/100g	7	73	24.09.	0	0	0	0,004	no	
	6	g/100g	24	56							
	7	g/100g	3	77	15.10.19	< 0.04	< 0.04	< 0.04	< 0.04	no	93
	8	g/100g	34	46	11.10.19				1 mg/100g		
	9	g/100g	36	44	15.10.19				0,09	no	
	10	g/100g	64	16						no	
	11	g/100g	10	70	17.09.19	< 0,03	< 0,03	< 0,03	0,03	no	
	12	g/100g	14	66	03.10.19	0,89	0,99	0,8	0,065	no	
	13	g/100g	35	45	26.10.19	0,3	0,305	0,294			
	14	g/100g	22	58							
	15	g/100g	30	50	div.	0,01	0,01	0,011	0,01	yes	98
	16	g/100g	5	75							
	17	g/100g	28	52	19.09.19	< 0,01	< 0,01	< 0,01	0,01	no	-
	18	g/100g	26	54	02.10.19		1,317	1,396	<0,01	no	
	19	g/100g	13	67							
	20	g/100g	19	61	13.09.19	0,056	0,052	0,06	0,00765	no	
	21	g/100g	23	57							
	22	g/100g	38	42	23.09.19	<0,05	<0,05	<0,05	0,05	no	-

Parameter	Participant	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana-lysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifi-cation	Incl. RR	Recovery rate
					day/month					yes/no	in %
Glycin / Glycine	1	g/100g	31	49		1,07	1,09	1,04			
	2	g/100g	12	68	01+02/10	0,89	0,84	0,93		yes	97-99%
	3	g/100g	15	65		0,791	0,847	0,735	0,006		
	4	g/100g	37	43	15.10.19	0,9555	0,983	0,928	0,01	no	
	5	g/100g	7	73	24.09.	0,85	0,9	0,8	0,001	no	
	6	g/100g	24	56							
	7	g/100g	3	77	15.10.19	0,91	0,87	0,96	< 0.04	no	105
	8	g/100g	34	46	11.10.19	0,83	0,78	0,88		no	
	9	g/100g	36	44	15.10.19	0,93	0,95	0,9	0,04	no	
	10	g/100g	64	16							
	11	g/100g	10	70	17.09.19	0,96	0,93	0,99	0,01	no	
	12	g/100g	14	66	03.10.19	0,81	0,8	0,81	0,033	no	
	13	g/100g	35	45	26.10.19	0,998	0,826	1,17			
	14	g/100g	22	58		1	1,09	0,91			
	15	g/100g	30	50	div.	0,998	0,993	1,003	0,01	yes	98
	16	g/100g	5	75	20.09.19	0,56	0,59	0,53	0,00001		
	17	g/100g	28	52	19.09.19	0,94	0,85	1,03	0,01	no	-
	18	g/100g	26	54	02.10.19		0,792	0,776	<0,01	no	
	19	g/100g	13	67							
	20	g/100g	19	61	13.09.19	0,958	0,941	0,974	0,00885	no	
	21	g/100g	23	57			1,05	1,21			
	22	g/100g	38	42	23.09.19	0,96	0,94	0,98	0,05	no	-

Parameter	Participant	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana-lysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifi-cation	Incl. RR	Recovery rate
					day/month					yes/no	in %
	1	g/100g	31	49		2,3	2,31	2,28			
	2	g/100g	12	68	01+02/10	0,61	0,61	0,61		yes	97-99%
	3	g/100g	15	65		0,699	0,704	0,693	0,009		
	4	g/100g	37	43	15.10.19	0,596	0,598	0,594	0,01	no	
	5	g/100g	7	73	24.09.	0,5	0,51	0,49	0,002	no	
	6	g/100g	24	56							
	7	g/100g	3	77	15.10.19	0,58	0,59	0,57	< 0.04	no	103
	8	g/100g	34	46	11.10.19	0,51	0,52	0,49		no	
	9	g/100g	36	44	15.10.19	1,93	1,96	1,9	0,16	no	
	10	g/100g	64	16							
L-Histidin/	11	g/100g	10	70	17.09.19	0,58	0,56	0,6	0,02	no	
L-Histidine	12	g/100g	14	66	03.10.19	0,51	0,51	0,5	0,09	no	
	13	g/100g	35	45	26.10.19	1,29	1,29	1,29			
	14	g/100g	22	58		0,5	0,52	0,47			
	15	g/100g	30	50	div.	0,596	0,612	0,581	0,01	yes	100
	16	g/100g	5	75	20.09.19	0,49	0,48	0,51	0,00001		
	17	g/100g	28	52	19.09.19	0,57	0,57	0,57	0,01	no	-
	18	g/100g	26	54	02.10.19		0,558	0,529	<0,01	no	
	19	g/100g	13	67							
	20	g/100g	19	61	13.09.19	0,617	0,618	0,616	0,01425	no	
	21	g/100g	23	57			0,62	0,63			
	22	g/100g	38	42	23.09.19	0,19	0,2	0,17	0,05	no	-

Parameter	Participant	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana-lysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifi-cation	Incl. RR	Recovery rate
					day/month					yes/no	in %
	1	g/100g	31	49		1,27	1,18	1,35			
	2	g/100g	12	68	01+02/10	0,97	0,97	0,97		yes	97-99%
	3	g/100g	15	65		0,924	0,904	0,944	0,009		
	4	g/100g	37	43	15.10.19	0,8975	0,889	0,906	0,01	no	
	5	g/100g	7	73	24.09.	0,86	0,88	0,84	0,002	no	
	6	g/100g	24	56							
	7	g/100g	3	77	15.10.19	0,91	0,88	0,94	< 0.04	no	100
	8	g/100g	34	46	11.10.19	0,98	0,98	0,98		no	
	9	g/100g	36	44	15.10.19	0,88	0,9	0,85	0,07	no	
	10	g/100g	64	16		1	1,1	1	0,001		
L-Isoleucin/ L-Isoleucine	11	g/100g	10	70	17.09.19	0,9	0,9	0,9	0,01	no	
	12	g/100g	14	66	03.10.19	0,78	0,76	0,79	0,062	no	
	13	g/100g	35	45	26.10.19	0,919	0,943	0,895			
	14	g/100g	22	58		0,74	0,77	0,71			
	15	g/100g	30	50	div.	0,895	0,902	0,887	0,01	yes	94
	16	g/100g	5	75	20.09.19	1,16	1,19	1,13	0,00001		
	17	g/100g	28	52	19.09.19	0,84	0,81	0,86	0,01	no	-
	18	g/100g	26	54	02.10.19		0,903	0,85	<0,01	no	
	19	g/100g	13	67							
	20	g/100g	19	61	13.09.19	0,97	0,987	0,953	0,01685	no	
	21	g/100g	23	57			0,95	0,94			
	22	g/100g	38	42	23.09.19	0,91	0,94	0,88	0,05	no	-

Parameter	Participant	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana-lysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifi-cation	Incl. RR	Recovery rate
					day/month					yes/no	in %
L-Leucin/ L-Leucine	1	g/100g	31	49		1,79	1,71	1,87			
	2	g/100g	12	68	01+02/10	1,61	1,61	1,6		yes	97-99%
	3	g/100g	15	65		1,619	1,67	1,567	0,01		
	4	g/100g	37	43	15.10.19	1,515	1,49	1,54	0,01	no	
	5	g/100g	7	73	24.09.	1,45	1,5	1,41	0,002	no	
	6	g/100g	24	56							
	7	g/100g	3	77	15.10.19	1,58	1,52	1,63	< 0.04	no	104
	8	g/100g	34	46	11.10.19	1,52	1,55	1,48		no	
	9	g/100g	36	44	15.10.19	1,55	1,58	1,52	0,07	no	
	10	g/100g	64	16		1,2	1,3	1,2	0,001		
	11	g/100g	10	70	17.09.19	1,545	1,54	1,55	0,01	no	
	12	g/100g	14	66	03.10.19	1,35	1,36	1,34	0,067	no	
	13	g/100g	35	45	26.10.19	1,51	1,53	1,49			
	14	g/100g	22	58		1,43	1,47	1,39			
	15	g/100g	30	50	div.	1,568	1,582	1,554	0,01	yes	96
	16	g/100g	5	75	20.09.19	1,79	1,78	1,8	0,00001		
	17	g/100g	28	52	19.09.19	1,54	1,53	1,54	0,01	no	-
	18	g/100g	26	54	02.10.19		1,534	1,509	<0,01	no	
	19	g/100g	13	67							
	20	g/100g	19	61	13.09.19	1,627	1,655	1,599	0,0108	no	
	21	g/100g	23	57			1,62	1,69			
	22	g/100g	38	42	23.09.19	1,5	1,53	1,47	0,05	no	-

Parameter	Participant	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana-lysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifi-cation	Incl. RR	Recovery rate
					day/month					yes/no	in %
L-Lysin/ L-Lysine	1	g/100g	31	49		1,15	1,23	1,07			
	2	g/100g	12	68	01+02/10	1,21	1,09	1,32		yes	97-99%
	3	g/100g	15	65		0,997	0,95	1,043	0,06		
	4	g/100g	37	43	15.10.19	1,115	1,1	1,13	0,01	no	
	5	g/100g	7	73	24.09.	0,98	1,01	0,94	0,001	no	
	6	g/100g	24	56							
	7	g/100g	3	77	15.10.19	1,18	1,12	1,24	< 0.04	no	97
	8	g/100g	34	46	11.10.19	1,15	1,09	1,21		no	
	9	g/100g	36	44	15.10.19	1,06	1,1	1,02	0,15	no	
	10	g/100g	64	16							
	11	g/100g	10	70	17.09.19	1,165	1,14	1,19	0,02	no	
	12	g/100g	14	66	03.10.19	0,93	0,84	1,02	0,076	no	
	13	g/100g	35	45	26.10.19	0,999	0,898	1,1			
	14	g/100g	22	58		0,94	0,95	0,93			
	15	g/100g	30	50	div.	1,18	1,163	1,197	0,01	yes	100
	16	g/100g	5	75	20.09.19	0,91	0,85	0,97	0,00001		
	17	g/100g	28	52	19.09.19	1,07	1,07	1,06	0,01	no	-
	18	g/100g	26	54	02.10.19		1,137	0,867	<0,01	no	
	19	g/100g	13	67							
	20	g/100g	19	61	13.09.19	1,131	1,129	1,133	0,0139	no	
	21	g/100g	23	57			1,19	1,29			
	22	g/100g	38	42	23.09.19	0,9	0,94	0,86	0,05	no	-

Parameter	Participant	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana-lysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifi-cation	Incl. RR	Recovery rate
					day/month					yes/no	in %
L-Methionin/ L-Methionine	1	g/100g	31	49		0,22	0,21	0,24			
	2	g/100g	12	68	01+02/10	0,26	0,26	0,26		yes	97-99%
	3	g/100g	15	65		0,347	0,378	0,315	0,03		
	4	g/100g	37	43	15.10.19	0,241	0,241	0,241	0,01	no	
	5	g/100g	7	73	24.09.	0,26	0,25	0,27	0,001	no	
	6	g/100g	24	56							
	7	g/100g	3	77	15.10.19	0,24	0,24	0,25	< 0.04	no	99
	8	g/100g	34	46	11.10.19	0,23	0,23	0,23		no	
	9	g/100g	36	44	15.10.19	0,25	0,25	0,25	0,07	no	
	10	g/100g	64	16							
	11	g/100g	10	70	17.09.19	0,25	0,24	0,26	0,02	no	
	12	g/100g	14	66	08.10.19	0,25	0,25	0,25	0,009	no	
	13	g/100g	35	45	26.10.19	0,265	0,254	0,276			
	14	g/100g	22	58		0,22	0,23	0,22			
	15	g/100g	30	50	div.	0,251	0,253	0,248	0,01	yes	95
	16	g/100g	5	75	20.09.19	0,26	0,27	0,24	0,00001		
	17	g/100g	28	52	19.09.19	0,27	0,27	0,27	0,01	no	-
	18	g/100g	26	54	02.10.19		0,268	0,25	<0,01	no	
	19	g/100g	13	67							
	20	g/100g	19	61	13.09.19	0,265	0,265	0,264	0,0098	no	
	21	g/100g	23	57			0,25	0,26			
	22	g/100g	38	42	23.09.19	0,25	0,24	0,26	0,05	no	-

Parameter	Participant	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana-lysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifi-cation	Incl. RR	Recovery rate
					day/month					yes/no	in %
L-Phenylalanin/ L-Phenylalanine	1	g/100g	31	49							
	2	g/100g	12	68	01+02/10	0,86	0,93	0,79		yes	97-99%
	3	g/100g	15	65		0,631	0,672	0,59	0,01		
	4	g/100g	37	43	15.10.19	0,706	0,693	0,719	0,01	no	
	5	g/100g	7	73	24.09.	0,7	0,7	0,7	0,002	no	
	6	g/100g	24	56							
	7	g/100g	3	77	15.10.19	0,72	0,74	0,71	< 0.04	no	101
	8	g/100g	34	46	11.10.19	0,67	0,71	0,63		no	
	9	g/100g	36	44	15.10.19	0,57	0,57	0,57	0,17	no	
	10	g/100g	64	16							
	11	g/100g	10	70	17.09.19	0,69	0,7	0,68	0,02	no	
	12	g/100g	14	66	03.10.19	0,61	0,63	0,58	0,079	no	
	13	g/100g	35	45	26.10.19	0,772	0,761	0,782			
	14	g/100g	22	58		0,68	0,68	0,67			
	15	g/100g	30	50	div.	0,755	0,741	0,768	0,01	yes	95
	16	g/100g	5	75	20.09.19	0,66	0,6	0,71	0,00001		
	17	g/100g	28	52	19.09.19	0,68	0,78	0,58	0,01	no	-
	18	g/100g	26	54	02.10.19		0,618	0,696	<0,01	no	
	19	g/100g	13	67							
	20	g/100g	19	61	13.09.19	0,741	0,758	0,724	0,0159	no	
	21	g/100g	23	57			0,76	0,78			
	22	g/100g	38	42	23.09.19	0,72	0,72	0,71	0,05	no	-

Parameter	Participant	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana-lysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifi-cation	Incl. RR	Recovery rate
					day/month					yes/no	in %
L-Prolin/ L-Proline	1	g/100g	31	49		1,12	1,14	1,11			
	2	g/100g	12	68	01+02/10	1,09	1,03	1,15		yes	97-99%
	3	g/100g	15	65		1,309	1,259	1,359	0,01		
	4	g/100g	37	43	15.10.19	1,135	1,14	1,13	0,01	no	
	5	g/100g	7	73	24.09.	0,85	0,83	0,87	0,003	no	
	6	g/100g	24	56							
	7	g/100g	3	77	15.10.19	1,12	1,11	1,14	< 0.04	no	103
	8	g/100g	34	46	11.10.19	1,31	1,37	1,24		no	
	9	g/100g	36	44	15.10.19	1,08	1,07	1,08	0,06	no	
	10	g/100g	64	16							
	11	g/100g	10	70	17.09.19	1,12	1,17	1,07	0,01	no	
	12	g/100g	14	66	03.10.19	1,1	1,09	1,11	0,045	no	
	13	g/100g	35	45	26.10.19	1,16	1,17	1,15			
	14	g/100g	22	58		1,16	1,16	1,16			
	15	g/100g	30	50	div.	1,15	1,137	1,164	0,01	yes	95
	16	g/100g	5	75	20.09.19	1,34	1,35	1,33	0,00001		
	17	g/100g	28	52	19.09.19	1,08	1,11	1,05	0,01	no	-
	18	g/100g	26	54	02.10.19		0,989	1,095	<0,01	no	
	19	g/100g	13	67							
	20	g/100g	19	61	13.09.19	1,208	1,167	1,249	0,0086	no	
	21	g/100g	23	57			1,25	1,18			
	22	g/100g	38	42	23.09.19	1,33	1,33	1,32	0,05	no	-

Parameter	Participant	Unit	Sample I DLA No.	Sample II DLA No.	Date of analysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantification	Incl. RR	Recovery rate
					day/month					yes/no	in %
L-Serin/ L-Serine	1	g/100g	31	49		0,71	0,69	0,73			
	2	g/100g	12	68	01+02/10	1,61	1,61	1,61		yes	97-99%
	3	g/100g	15	65		0,693	0,678	0,708	0,007		
	4	g/100g	37	43	15.10.19	0,6635	0,651	0,676	0,01	no	
	5	g/100g	7	73	24.09.	0,65	0,67	0,63	0,001	no	
	6	g/100g	24	56							
	7	g/100g	3	77	15.10.19	0,69	0,69	0,69	< 0.04	no	100
	8	g/100g	34	46	11.10.19	0,66	0,71	0,61		no	
	9	g/100g	36	44	15.10.19	0,7	0,73	0,67	0,05	no	
	10	g/100g	64	16							
	11	g/100g	10	70	17.09.19	0,71	0,71	0,71	0,01	no	
	12	g/100g	14	66	03.10.19	0,58	0,57	0,58	0,043	no	
	13	g/100g	35	45	26.10.19	0,701	0,677	0,724			
	14	g/100g	22	58		0,67	0,66	0,67			
	15	g/100g	30	50	div.	0,702	0,717	0,687	0,01	yes	97
	16	g/100g	5	75	20.09.19	0,71	0,69	0,73	0,00001		
	17	g/100g	28	52	19.09.19	0,75	0,73	0,76	0,01	no	-
	18	g/100g	26	54	02.10.19		0,671	0,68	<0,01	no	
	19	g/100g	13	67							
	20	g/100g	19	61	13.09.19	0,727	0,751	0,702	0,0061	no	
	21	g/100g	23	57			0,71	0,68			
	22	g/100g	38	42	23.09.19	0,68	0,7	0,65	0,05	no	-

Parameter	Participant	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana-lysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifi-cation	Incl. RR	Recovery rate
					day/month					yes/no	in %
L-Threonin/ L-Threonine	1	g/100g	31	49							
	2	g/100g	12	68	01+02/10	0,77	0,76	0,78		yes	97-99%
	3	g/100g	15	65		1,178	1,198	1,157	0,055		
	4	g/100g	37	43	15.10.19	0,797	0,795	0,799	0,01	no	
	5	g/100g	7	73	24.09.	0,74	0,79	0,7	0,002	no	
	6	g/100g	24	56							
	7	g/100g	3	77	15.10.19	0,76	0,75	0,77	< 0.04	no	100
	8	g/100g	34	46	11.10.19	0,97	1,13	0,8		no	
	9	g/100g	36	44	15.10.19	0,72	0,72	0,71	0,06	no	
	10	g/100g	64	16							
	11	g/100g	10	70	17.09.19	0,8	0,8	0,8	0,01	no	
	12	g/100g	14	66	03.10.19	0,68	0,68	0,67	0,048	no	
	13	g/100g	35	45	26.10.19	0,728	0,774	0,681			
	14	g/100g	22	58		0,76	0,8	0,72			
	15	g/100g	30	50	div.	0,787	0,811	0,763	0,01	yes	97
	16	g/100g	5	75	20.09.19	0,71	0,72	0,7	0,00001		
	17	g/100g	28	52	19.09.19	0,74	0,76	0,71	0,01	no	-
	18	g/100g	26	54	02.10.19		0,793	0,743	<0,01	no	
	19	g/100g	13	67							
	20	g/100g	19	61	13.09.19	0,823	0,81	0,835	0,0098	no	
	21	g/100g	23	57			0,79	0,81			
	22	g/100g	38	42	23.09.19	0,79	0,8	0,78	0,05	no	-

Parameter	Participant	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana-lysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifi-cation	Incl. RR	Recovery rate
					day/month					yes/no	in %
	1	g/100g	31	49		0,32	0,31	0,32			
	2	g/100g	12	68	10.10.19	0,302	0,298	0,306		yes	
	3	g/100g	15	65							
	4	g/100g	37	43	15.10.19	0,289	0,286	0,292	0,01	no	
	5	g/100g	7	73	24.09.	0,28	0,27	0,29	0,004	no	
	6	g/100g	24	56							
	7	g/100g	3	77	15.10.19	0,29	0,29	0,3	< 0.04	no	100
	8	g/100g	34	46	11.10.19				1 mg/100g		
	9	g/100g	36	44	15.10.19	0,33	0,34	0,32	0,1	no	
	10	g/100g	64	16							
L-Tryptophan	11	g/100g	10	70	17.09.19	0,295	0,3	0,29	0,02	no	
	12	g/100g	14	66	08.10.19	0,31	0,3	0,31	0,023	no	
	13	g/100g	35	45	25.10.19	0,32	0,32	0,32			
	14	g/100g	22	58							
	15	g/100g	30	50	div.	0,313	0,318	0,308	0,01	yes	94
	16	g/100g	5	75	20.09.19	0,29	0,3	0,28	0,00001		
	17	g/100g	28	52	19.09.19	0,3	0,3	0,3	0,01	no	-
	18	g/100g	26	54	02.10.19		0,327	0,323	<0,01	no	
	19	g/100g	13	67							
	20	g/100g	19	61	13.09.19	0,317	0,316	0,317	0,00945	no	
	21	g/100g	23	57							
	22	g/100g	38	42	23.09.19	0,29	0,29	0,28	0,05	no	-

Parameter	Participant	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana-lysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifi-cation	Incl. RR	Recovery rate
					day/month					yes/no	in %
	1	g/100g	31	49		0,68	0,67	0,68			
	2	g/100g	12	68	01+02/10	0,68	0,7	0,66		yes	97-99%
	3	g/100g	15	65		0,707	0,703	0,71	0,02		
	4	g/100g	37	43	15.10.19	0,6685	0,664	0,673	0,01	no	
	5	g/100g	7	73	24.09.	0,69	0,67	0,71	0,001	no	
	6	g/100g	24	56							
	7	g/100g	3	77	15.10.19	0,69	0,67	0,71	< 0.04	no	101
	8	g/100g	34	46	11.10.19	0,66	0,66	0,66		no	
	9	g/100g	36	44	15.10.19	0,6	0,63	0,58	0,09	no	
	10	g/100g	64	16		1,3	1,1	1,4	0,001		
L-Tyrosin/ L-Tyrosine	11	g/100g	10	70	17.09.19	0,695	0,7	0,69	0,02	no	
	12	g/100g	14	66	03.10.19	0,56	0,56	0,55	0,088	no	
	13	g/100g	35	45	26.10.19	0,698	0,704	0,692			
	14	g/100g	22	58		0,67	0,68	0,67			
	15	g/100g	30	50	div.	0,72	0,727	0,714	0,01	yes	95
	16	g/100g	5	75	20.09.19	0,17	0,17	0,17	0,00001		
	17	g/100g	28	52	19.09.19	0,87	0,86	0,87	0,01	no	-
	18	g/100g	26	54	02.10.19		0,684	0,674	<0,01	no	
	19	g/100g	13	67							
	20	g/100g	19	61	13.09.19	0,724	0,728	0,72	0,0129	no	
	21	g/100g	23	57			0,71	0,72			
	22	g/100g	38	42	23.09.19	0,65	0,66	0,63	0,05	no	-

Parameter	Participant	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana-lysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifi-cation	Incl. RR	Recovery rate
					day/month					yes/no	in %
L-Valin/ L-Valine	1	g/100g	31	49							
	2	g/100g	12	68	01+02/10	1,06	1,07	1,05		yes	97-99%
	3	g/100g	15	65		0,988	1,026	0,949	0,008		
	4	g/100g	37	43	15.10.19	1,008	0,976	1,04	0,01	no	
	5	g/100g	7	73	24.09.	0,91	0,95	0,87	0,001	no	
	6	g/100g	24	56							
	7	g/100g	3	77	15.10.19	0,96	0,97	0,96	< 0.04	no	101
	8	g/100g	34	46	11.10.19	0,73	0,72	0,73		no	
	9	g/100g	36	44	15.10.19	0,88	0,89	0,87	0,06	no	
	10	g/100g	64	16		1,1	1,1	1,2	0,001		
	11	g/100g	10	70	17.09.19	0,985	1,02	0,95	0,01	no	
	12	g/100g	14	66	03.10.19	0,85	0,78	0,91	0,057	no	
	13	g/100g	35	45	26.10.19	0,998	1,06	0,936			
	14	g/100g	22	58		0,94	0,94	0,94			
	15	g/100g	30	50	div.	1,063	1,026	1,1	0,01	yes	97
	16	g/100g	5	75	20.09.19	1,24	1,35	1,12	0,00001		
	17	g/100g	28	52	19.09.19	0,96	0,88	1,03	0,01	no	-
	18	g/100g	26	54	02.10.19		0,995	0,769	<0,01	no	
	19	g/100g	13	67							
	20	g/100g	19	61	13.09.19	1,062	1,033	1,09	0,0103	no	
	21	g/100g	23	57			1	1,1			
	22	g/100g	38	42	23.09.19	1,02	1,01	1,02	0,05	no	-

Parameter	Participant	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana-lysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifi-cation	Incl. RR	Recovery rate
					day/month					yes/no	in %
Taurin/ Taurine	1	g/100g	31	49		< 0,19	< 0,19	< 0,19	0,19		
	2	g/100g	12	68	01+02/10	0,04	0,04	0,04		yes	97-99%
	3	g/100g	15	65							
	4	g/100g	37	43	15.10.19	0,0278	0,0267	0,0289	0,01	no	
	5	g/100g	7	73	24.09.	0,03	0,03	0,03	0,002	no	
	6	g/100g	24	56	19.09.19	0,0317	0,0298	0,0335	0,00005	no	
	7	g/100g	3	77	15.10.19	< 0.04	< 0.04	< 0.04	< 0.04	no	103
	8	g/100g	34	46	11.10.19	0,05	0,05	0,05		no	
	9	g/100g	36	44							
	10	g/100g	64	16							
	11	g/100g	10	70	17.09.19	0,03	0,03	0,03	0,01	no	
	12	g/100g	14	66	08.10.19	0,034	0,035	0,032	0,006	no	
	13	g/100g	35	45	14.10.19	0,03	0,03	0,03			
	14	g/100g	22	58							
	15	g/100g	30	50	div.	0,029	0,03	0,028	0,01	yes	100
	16	g/100g	5	75							
	17	g/100g	28	52	19.09.19	0,03	0,03	0,03	0,01	no	-
	18	g/100g	26	54	02.10.19		0,0295	0,0284	<0,01	no	
	19	g/100g	13	67	19.09.19	0,0296	0,0298	0,0294		no	
	20	g/100g	19	61	13.09.19	0,036	0,036	0,036	0,00985	no	
	21	g/100g	23	57							
	22	g/100g	38	42							

Parameter	Participant	Unit	Sample I DLA No.	Sample II DLA No.	Date of ana-lysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifi-cation	Incl. RR	Recovery rate
					day/month					yes/no	in %
L-Glutamin/ L-Glutamine	1	g/100g	31	49		1,57	1,49	1,65			
	2	g/100g	12	68							
	3	g/100g	15	65							
	4	g/100g	37	43							
	5	g/100g	7	73							
	6	g/100g	24	56							
	7	g/100g	3	77							
	8	g/100g	34	46							
	9	g/100g	36	44							
	10	g/100g	64	16							
	11	g/100g	10	70	17.09.19	1,21	1,23	1,19	0,01	no	
	12	g/100g	14	66							
	13	g/100g	35	45							
	14	g/100g	22	58		1,23	1,28	1,19			
	15	g/100g	30	50	div.	1,432	1,473	1,39	0,01	yes	98
	16	g/100g	5	75							
	17	g/100g	28	52	19.09.19	1,15	1,16	1,14	0,01	no	-
	18	g/100g	26	54							
	19	g/100g	13	67							
	20	g/100g	19	61	13.09.19	1,333	1,361	1,305	0,01365	no	
	21	g/100g	23	57							
	22	g/100g	38	42							

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

Parameter	Partici- pant	Method specification, as in test report / standard / literature *	Notes to sample preparation*	Notes to analytical method*	Calibration and reference mate- rial	Recovery with same ma- trix	Method accr. ISO / IEC 17025	Further remarks
						yes / no	yes / no	
L-Alanin/ L-Alanine	1	HPAEC-IPAD		HPAEC-IPAD	external Standard, AAS 18 Sigma and single substances		yes	
	2	DIN EN ISO 13903:2005-09				yes	yes	
	3	§64 LFGB L 49.07-2	extraction in 0,1M HCl	derivatization Ninhydrin	DLA-59 2016	no	yes	
	4	Method Code. 07(S212) - Title: Free Aminoacid determination		HPLC-FL			YES	
	5	determination of free amino acids by HPLC with after column derivatization	aqueous extract after protein precipitation with 5-Sulfosalicylic acid solution	Ion exchange chromatography with after column derivatization (Ninhydrin)	External Standard, one point calibration, sausage		yes	
	6							
	7	Z1220	extraction with HCl 0.01 M	UPLC with UV-Detektion	1-point calibration	yes	yes	
	8	in house method	dilution in a solvent	HPLC	calibration with one point	no	no	
	9	Precolumn derivatization of amino acids with AQC and detection by HPLC-PDA	None	LOQ in calibration curve is 5 pmol / ul	Standard AA (Waters, Ref WAT088122)		No	We are in the process of accreditation
	10	§64 LFGB L 07.00-59 (modifed)	aqueous solution, precipitation with Carrez,	HPLC, Fluorescence		no	yes	
	11	03-31-MAA-M-AMINOSRE, 2019-03, IC	Approx. 1g of sample in 60 ml bidest water for 15 min at 40°C in shaking water bath extracted. After cooling 10 ml Sulfosalicylic acid added and for 30 min cooled at +4°C. Afterwards the beaker with bi-dest water filled ad 100 ml and centrifugated. Su-pernatant with sample dilution buffer (company Sykam) diluted.	amino acid analyser (after column derivatization)	amino acid standard company Sykam	no	yes	
	12	determination of free amino acids by amino acid analyser			internal Standard		yes	
	13						Yes	
	14	total amino acids (analog regulation (EC) No. 152/2009)	free AA (Point 5.2 of regulation (EC) No. 152/2009)		int. Std., 1-point calibration, feed		yes	
	15	SOP M 3123, LC-MS/MS		LC-MS/MS after derivatization		no	yes	
	16	EZ:faast Kit (Phenomenex)	acidic extraction	LC-MS/MS	Multi-point calibration, internal Std.		yes	
	17	determination of free amino acids with amino acid analyser (in-house method AS 02, Version 05).	samples crushed in mortar	According to Ph. Eur. 2.2.56, Method 1, after column derivatization with Ninhydrin	amino acid mix solution 79248 (Sigma-Aldrich), single reference substances		yes	
	18	ASU L 49.07-1:1985-05, mod.		IC-UV	yes		yes	
	19							
	20	AOAC (2000) 999.13 Ch4 p17	free amino acid analysis	QC0601 Internal Method	single point	no	yes	
	21	§64 LFGB 49.07-1						
	22	LC/FLD - internal method PNTQ1339			external calib. curve and internal RM	no	no	

Parameter	Partici-pant	Method specification, as in test report / standard / literature *	Notes to sample preparation*	Notes to analytical method*	Calibration and reference material	Recovery with same matrix	Method accr. ISO / IEC 17025	Further remarks
						yes / no	yes / no	
	1							
	2	DIN EN ISO 13903:2005-09				yes	yes	
	3							
	4						YES	
	5						yes	
	6							
	7	Z1220	dilution		Waters-Standard	yes	yes	
	8							
	9			LOQ in calibration curve is 2,5 pmol / ul			No	We are in the process of accreditation
L-Arginin/ L-Arginine	10							
	11				Arginine hydrochloride from Merck (QRK) and Sigma-Aldrich (Kal)	no	yes	
	12				internal Standard		yes	
	13						Yes	
	14							
	15							
	16							
	17						yes	
	18	ASU L 49.07-1:1985-05, mod.		IC-UV	yes		yes	
	19							
	20				calibration and	no	yes	
	21							
	22	LC/FLD - internal method PNTQ1339			external calib. curve and internal RM	no	no	

Parameter	Partici-pant	Method specification, as in test report / standard / literature *	Notes to sample preparation*	Notes to analytical method*	Calibration and reference mate-rial	Recovery with same ma-trix	Method accr. ISO / IEC 17025	Further remarks
						yes / no	yes / no	
	1							
	2	DIN EN ISO 13903:2005-09				yes	yes	
	3							
	4						YES	
	5						yes	
	6							
	7	Z1220	Filtration		+Tau+Trp	yes	yes	
	8							
	9			LOQ in calibration curve is 5 pmol / ul			No	We are in the process of accreditation
L-Asparagin-säure/ L-Aspartic acid	10							
	11				L-Aspartic acid from Sigma-Aldrich (QRK) and Roth (Kal)	no	yes	
	12				internal Standard		yes	
	13						Yes	
	14							
	15							
	16							
	17						yes	
	18	ASU L 49.07-1:1985-05, mod.		IC-UV	yes		yes	
	19							
	20				internal QC used.	no	yes	
	21							
	22	LC/FLD - internal method PNTQ1339			external calib. curve and internal RM	no	no	

Parameter	Partici-pant	Method specification, as in test report / standard / literature *	Notes to sample preparation*	Notes to analytical method*	Calibration and reference material	Recovery with same matrix	Method accr. ISO / IEC 17025	Further remarks
						yes / no	yes / no	
L-Cystein/ L-Cysteine	1							
	2	DIN EN ISO 13903:2005-09				yes	yes	
	3							
	4						YES	
	5						yes	
	6							
	7	Z1220	ACCQ Tag Derivatisation Kit			yes	yes	
	8							
	9			LOQ in calibration curve is 2,5 pmol / ul			No	
	10							
	11				Aminosäurestandard Fa. Sykam	no	yes	
	12							
	13							
	14							
	15							
	16							
	17	Free L-Cysteine determined by amino acid analyser (in-house method AS 11, Version 03).	samples crushed in mortar, stabilized with sodium sulfite		single reference substances		yes	
	18							
	19							
	20	N/A	N/A	N/A	N/A	N/A	N/A	Unable to analyse
	21							
	22	LC/FLD - internal method PNTQ1339			external calib. curve and internal RM	no	no	

Parameter	Partici-pant	Method specification, as in test report / standard / literature *	Notes to sample preparation*	Notes to analytical method*	Calibration and reference mate-rial	Recovery with same ma-trix	Method accr. ISO / IEC 17025	Further remarks
						yes / no	yes / no	
L-Cystin/ L-Cystine	1							
	2	DIN EN ISO 13903:2005-09				yes	yes	
	3							
	4						YES	
	5						yes	
	6							
	7	Z1220	from Waters			yes	yes	
	8							
	9							
	10							
	11				Amino acid standard from Sykam	no	yes	
	12				internal Standard		yes	
	13						Yes	
	14							
	15							
	16							
	17	determination of free amino acids by amino acid analyser (in-house method AS 02, Version 05).	samples crushed in mortar		amino acid mix solution 79248 (Sigma-Aldrich), single reference substances		yes	
	18							
	19							
	20					no	yes	
	21							
	22	LC/FLD - internal method PNTQ1339			external calib. curve and internal RM	no	no	

Parameter	Partici-pant	Method specification, as in test report / standard / literature *	Notes to sample preparation*	Notes to analytical method*	Calibration and reference material	Recovery with same matrix	Method accr. ISO / IEC 17025	Further remarks
						yes / no	yes / no	
L-Glutaminsäure, Glycin, L-Isoleucin, L-Leucin, L-Serin, L-Threonin, L-Valin/ **L-Glutamic acid, Glycine, L-Isoleucine, L-Leucine, L-Serine, L-Threonine, L-Valine**	1							
	2	DIN EN ISO 13903:2005-09				yes	yes	
	3							
	4						YES	
	5						yes	
	6							
	7	Z1220				yes	yes	
	8							
	9			LOQ in calibration curve is 2,5 pmol / ul			No	We are in the process of accreditation
	10							
	11				Amino acid standard from Sykam internal Standard	no	yes	
	12						yes	
	13						Yes	
	14							
	15							
	16							
	17						yes	
	18	ASU L 49.07-1:1985-05, mod.		IC-UV	yes		yes	
	19							
	20					no	yes	
	21							
	22	LC/FLD - internal method PNTQ1339			external calib. curve and internal RM	no	no	

Parameter	Partici-pant	Method specification, as in test report / standard / literature *	Notes to sample preparation*	Notes to analytical method*	Calibration and reference mate-rial	Recovery with same ma-trix	Method accr. ISO / IEC 17025	Further remarks
						yes / no	yes / no	
L-Histidin, L-Lysin, L-Phenylalanin/ **L-Histidine, L-Lysine, L-Phenylalanine**	1							
	2	DIN EN ISO 13903:2005-09				yes	yes	
	3							
	4						YES	
	5						yes	
	6							
	7	Z1220				yes	yes	
	8							
	9			LOQ in calibration curve is 5 pmol / ul			No	We are in the process of accreditation
	10							
	11				Amino acid standard from Sykam	no	yes	
	12				internal Standard		yes	
	13						Yes	
	14							
	15							
	16							
	17						yes	
	18	ASU L 49.07-1:1985-05, mod.		IC-UV	yes		yes	
	19							
	20					no	yes	
	21							
	22	LC/FLD - internal method PNTQ1339			external calib. curve and internal RM	no	no	

Parameter	Partici-pant	Method specification, as in test report / standard / literature *	Notes to sample preparation*	Notes to analytical method*	Calibration and reference material	Recovery with same matrix	Method accr. ISO / IEC 17025	Further remarks
						yes / no	yes / no	
L-Prolin/ L-Proline	1							
	2	DIN EN ISO 13903:2005-09				yes	yes	
	3							
	4						YES	
	5						yes	
	6							
	7	Z1220				yes	yes	
	8							
	9			LOQ in calibration curve is 2,5 pmol / ul			No	We are in the process of accreditation
	10							
	11				L-Proline from Sigma-Aldrich (QRK & Kal, different lots)	no	yes	
	12				internal Standard		yes	
	13						Yes	
	14							
	15							
	16							
	17						yes	
	18	ASU L 49.07-1:1985-05, mod.		IC-UV	yes		yes	
	19							
	20					no	yes	
	21							
	22	LC/FLD - internal method PNTQ1339			external calib. curve and internal RM	no	no	

Parameter	Partici-pant	Method specification, as in test report / standard / literature *	Notes to sample preparation*	Notes to analytical method*	Calibration and reference mate-rial	Recovery with same ma-trix	Method accr. ISO / IEC 17025	Further remarks
						yes / no	yes / no	
L-Tryptophan	1							
	2	PA 804 :2017-03						
	3							
	4						YES	
	5						yes	
	6							
	7	Z1220				yes	yes	
	8							
	9			LOQ in calibration curve is 2,5 pmol / ul			No	We are in the process of accreditation
	10							
	11				Amino acid standard from Sykam	no	yes	
	12				internal Standard		yes	
	13						Yes	
	14							
	15							
	16							
	17						yes	
	18	ASU L 49.07-1:1985-05, mod.		IC-UV	yes		yes	
	19							
	20					no	yes	
	21							
	22	LC/FLD - internal method PNTQ1339			external calib. curve and internal RM	no	no	

Parameter	Partici-pant	Method specification, as in test report / standard / literature *	Notes to sample preparation*	Notes to analytical method*	Calibration and reference material	Recovery with same matrix	Method accr. ISO / IEC 17025	Further remarks
						yes / no	yes / no	
	1							
	2	DIN EN ISO 13903:2005-09				yes	yes	
	3							
	4						YES	
	5						yes	
	6							
	7	Z1220				yes	yes	
	8							
	9			LOQ in calibration curve is 2,5 pmol / ul			No	
	10							
L-Tyrosin/ L-Tyrosine	11				Amino acid standard from Sykam	no	yes	
	12				internal Standard		yes	
	13						Yes	
	14							
	15							
	16							
	17						yes	
	18	ASU L 49.07-1:1985-05, mod.		IC-UV	yes		yes	
	19							
	20					no	yes	
	21							
	22	LC/FLD - internal method PNTQ1339			external calib. curve and internal RM	no	no	

Parameter	Partici-pant	Method specification, as in test report / standard / literature *	Notes to sample preparation*	Notes to analytical method*	Calibration and reference material	Recovery with same matrix	Method accr. ISO / IEC 17025	Further remarks
						yes / no	yes / no	
Taurin/ Taurine	1							
	2	DIN EN ISO 13903:2005-09				yes	yes	
	3							
	4						YES	
	5						yes	
	6	in-house method, SOP M857, HPLC-UV		HPLC-UV	external; Ref. material available	yes	yes	
	7	Z1220				yes	yes	
	8							
	9							
	10							
	11				Amino acid standard from Sykam	no	yes	
	12				internal Standard		yes	
	13						No	
	14							
	15							
	16							
	17						yes	
	18	in house method		IC-UV	yes		yes	
	19	PV-322-TauCar					yes	
	20					no	yes	
	21							
	22							

Parameter	Partici-pant	Method specification, as in test report / standard / literature *	Notes to sample preparation*	Notes to analytical method*	Calibration and reference material	Recovery with same ma-trix	Method accr. ISO / IEC 17025	Further remarks
						yes / no	yes / no	
L-Glutamin/ L-Glutamine	1							
	2							
	3							
	4							
	5							
	6							
	7							
	8							
	9							
	10							
	11				Glutamine from Merck (QRK) and Sigma-Aldrich (Kal)	no	yes	
	12							
	13							
	14							
	15							
	16							
	17						yes	
	18							
	19							
	20	AOAC (2000) 999.13 Ch4 p17						
	21							
	22							

5.2 Homogeneity

5.2.1 Trend line function of the participants results

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

Homgenität / homogeneity

L-Isoleucin / L-Isoleucine

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

$f(x) = 0{,}0175x + 92{,}9235$
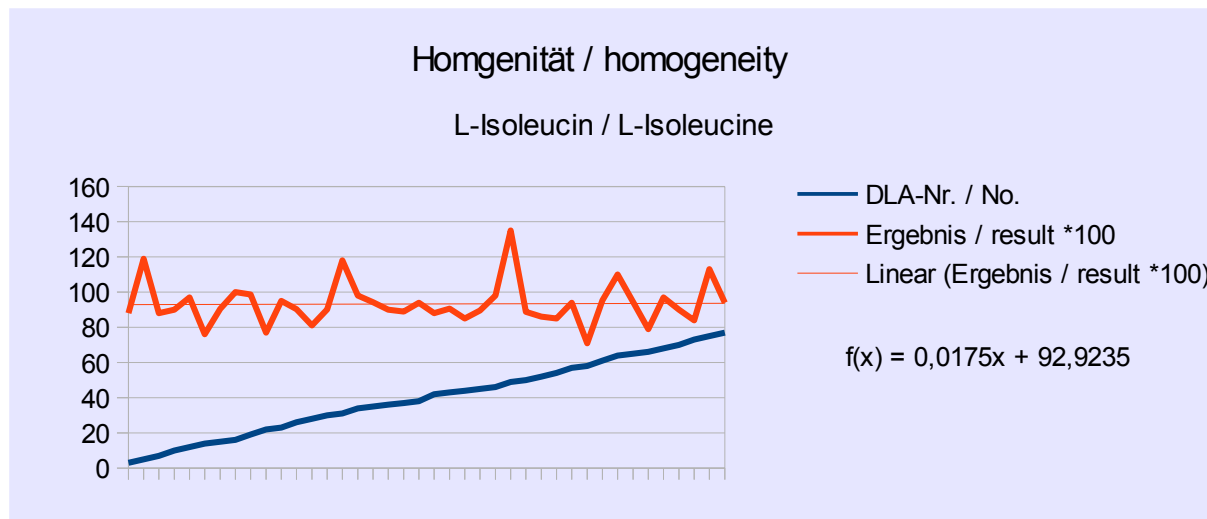
Abb./Fig. 40:
Trendfunktion L-Isoleucin Probennummern vs. Ergebnisse (1*100 dargestellt)
trend line function L-isoleucine sample number vs. results (1*100 shown)

Homgenität / homogeneity

L-Phenylalanin / L-Phenylalanine

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

$f(x) = -0{,}0916x + 71{,}5614$
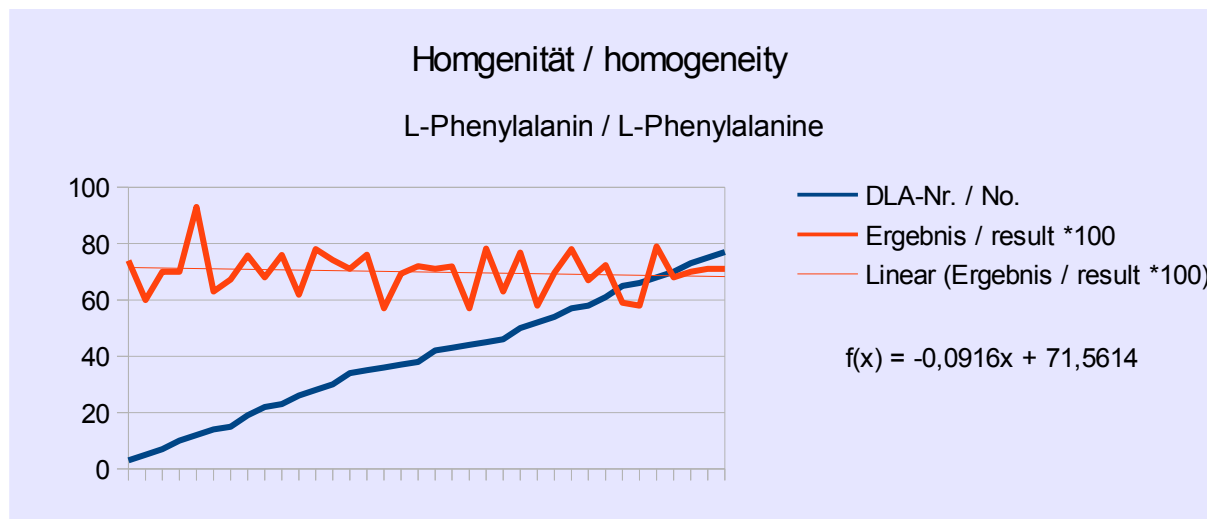
Abb./Fig. 41:
Trendfunktion L-Phenylalanin Probennummern vs. Ergebnisse (1*100 dargestellt)
trend line function L-phenylalanine sample number vs. results (1*100 shown)
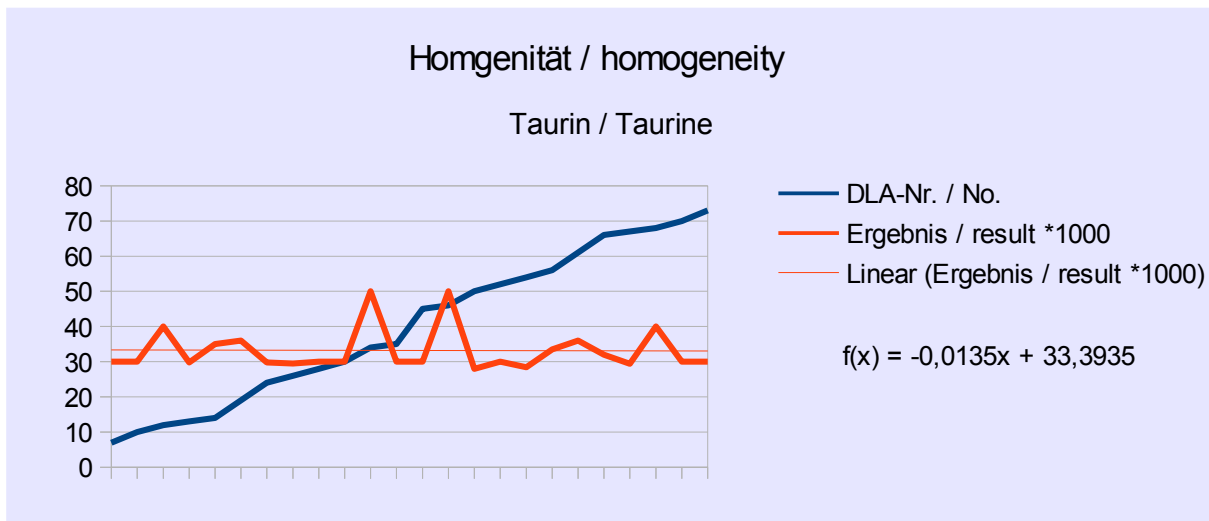
Abb./Fig. 42:
Trendfunktion Taurin Probennummern vs. Ergebnisse (1*1000 dargestellt)
trend line function taurine sample number vs. results (1*1000 shown)

5.3 Kernel Density Plots of Results

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

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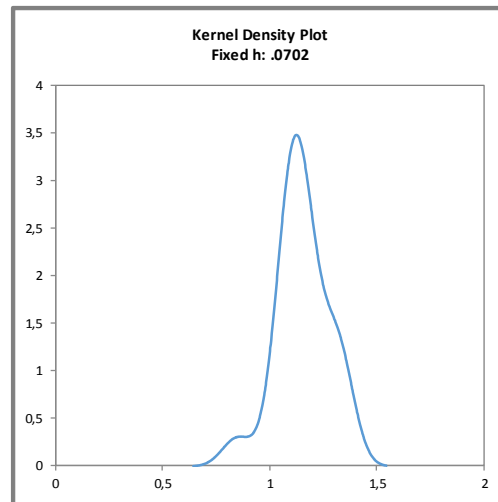of participants' results
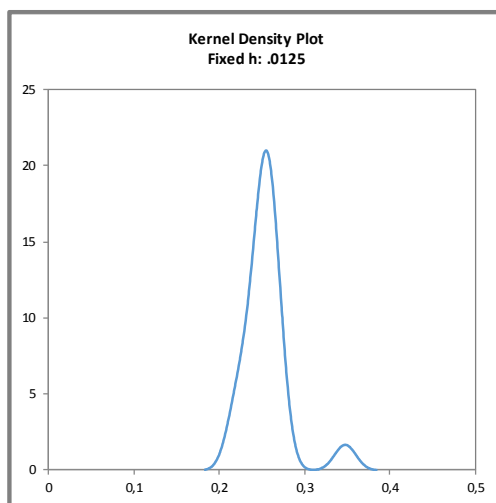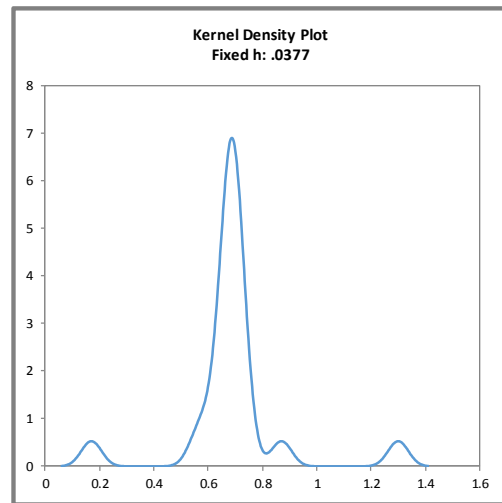(with h = 0,75 x σ_{pt} of X_{pt})

L-Asparaginsäure/L-Aspartic acid

Kernel Density Plot
Fixed h: .0402

L-Alanin/L-Alanine

Kernel Density Plot
Fixed h: .0193
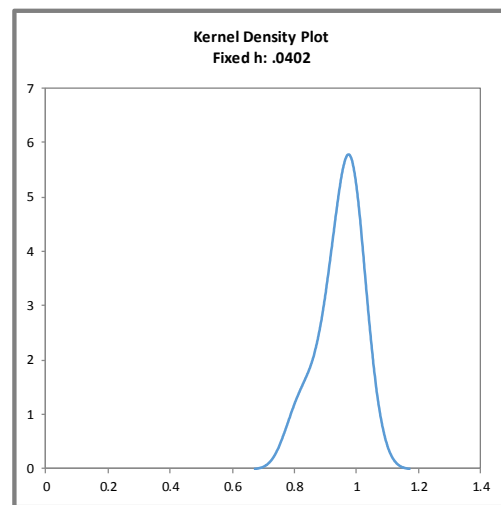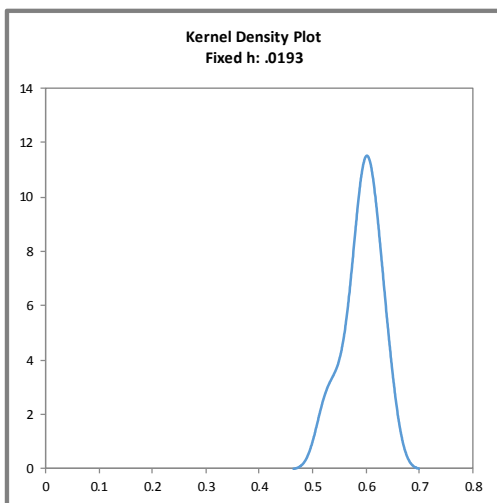
L-Cystein/L-Cysteine

Keine quantitative Ergebnisse

No quantitative results

L-Arginin/L-Arginine
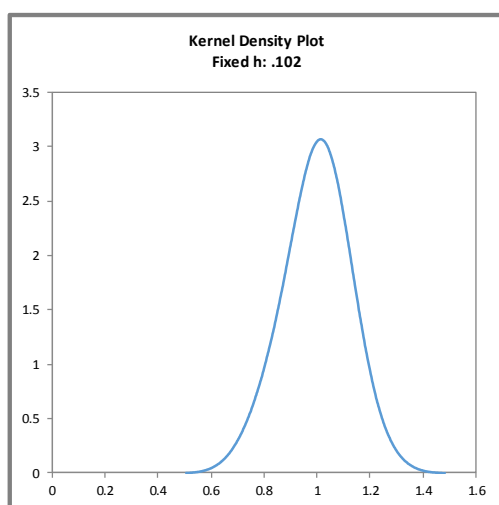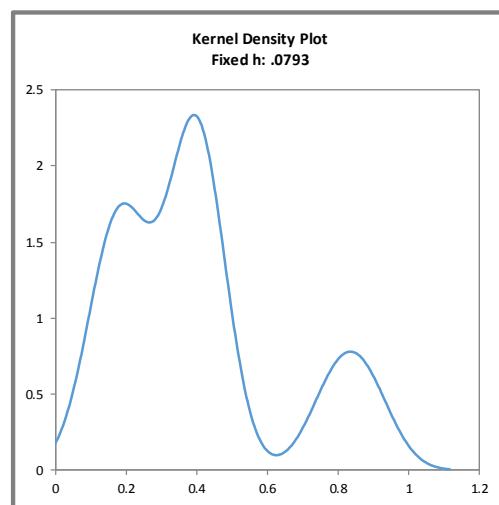
Kernel Density Plot
Fixed h: .102

L-Cystin/L-Cystine

Kernel Density Plot
Fixed h: .0793

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

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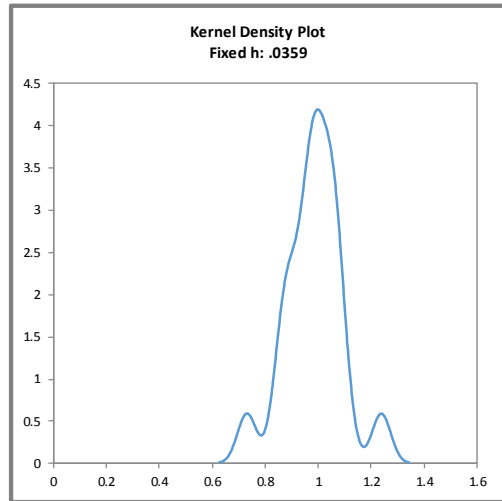of participants' results
(with h = 0,75 x σ_{pt} of X_{pt})
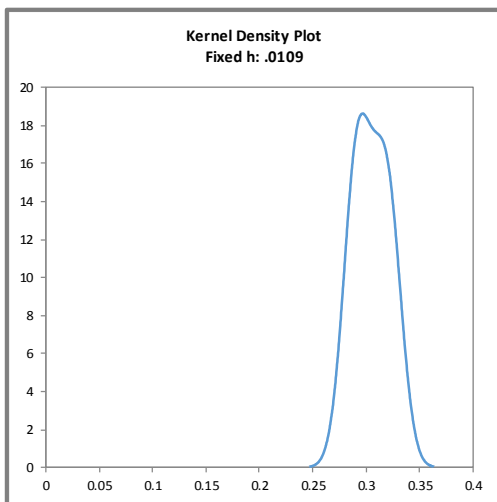
L-Histidin/L-Histidine

Kernel Density Plot
Fixed h: .071
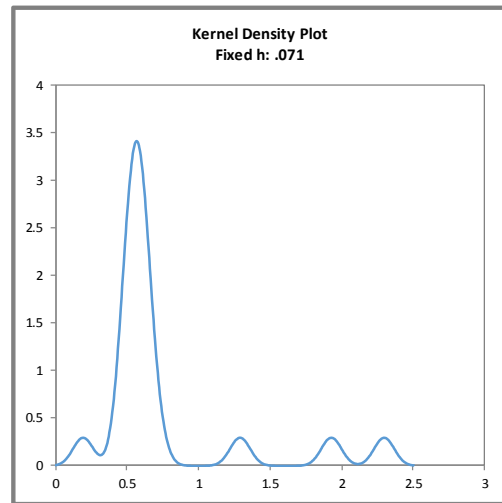
L-Glutaminsäure/L-Glutamic acid

<8 Ergebnisse

<8 Results

L-Isoleucin/L-Isoleucine
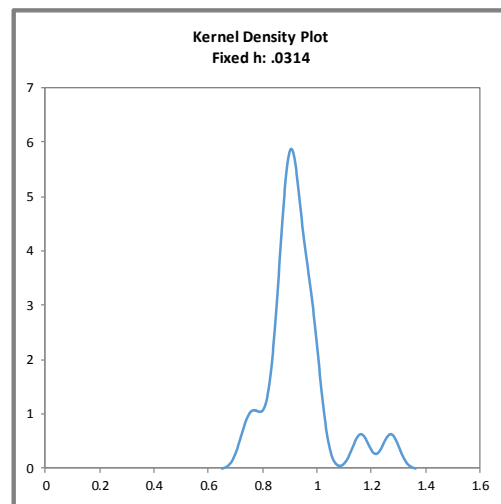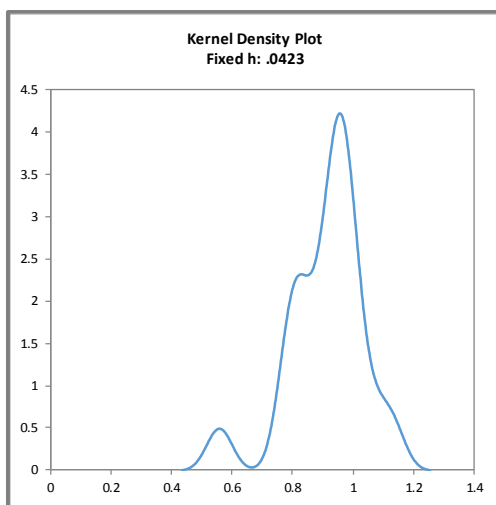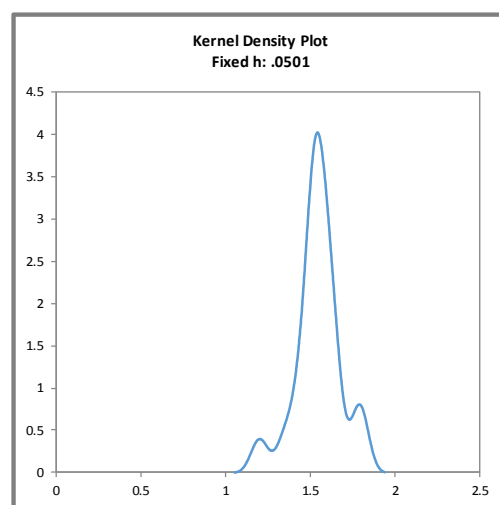
Kernel Density Plot
Fixed h: .0314

Glycin/Glycine

Kernel Density Plot
Fixed h: .0423

L-Leucin/L-Leucine

Kernel Density Plot
Fixed h: .0501

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-Phenylalanin/L-Phenylalanine
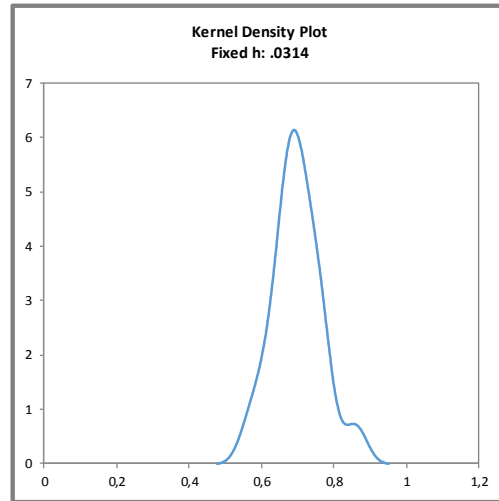
L-Lysin/L-Lysine
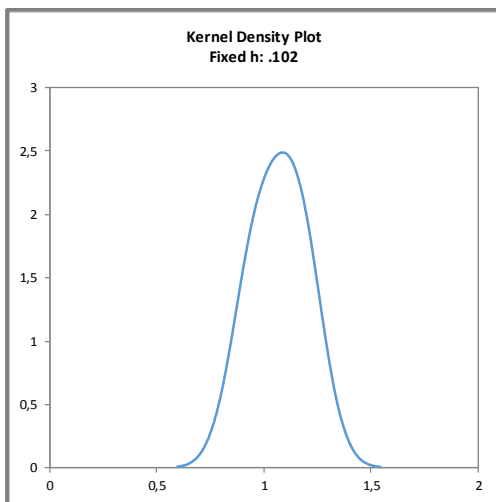
L-Prolin/L-Proline

L-Methionin/L-Methionine

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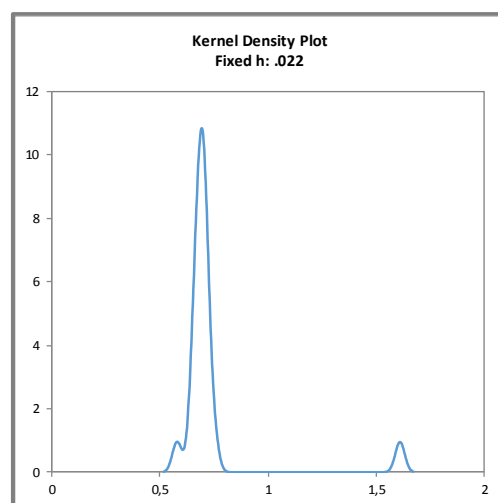
Abbildungen:
Kerndichte-Schätzungen
der Teilnehmerergebnisse
(mit h = 0,75 x σ_{pt} von X_{pt})

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: .0377

L-Threonin/L-Threonine

Kernel Density Plot
Fixed h: .0345

L-Valin/L-Valine

Kernel Density Plot
Fixed h: .0359
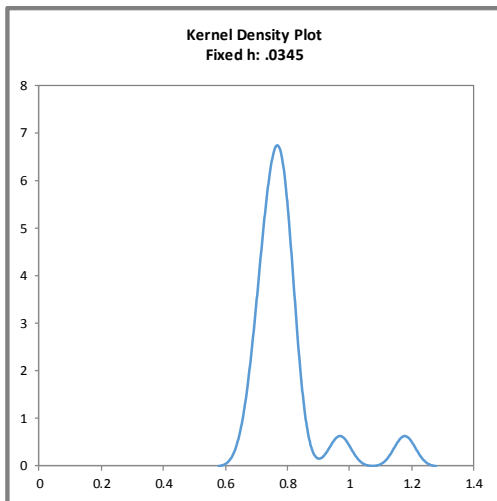
L-Tryptophan/L-Tryptophan

Kernel Density Plot
Fixed h: .0109

Taurin/Taurine

Kernel Density Plot
Fixed h: .0016
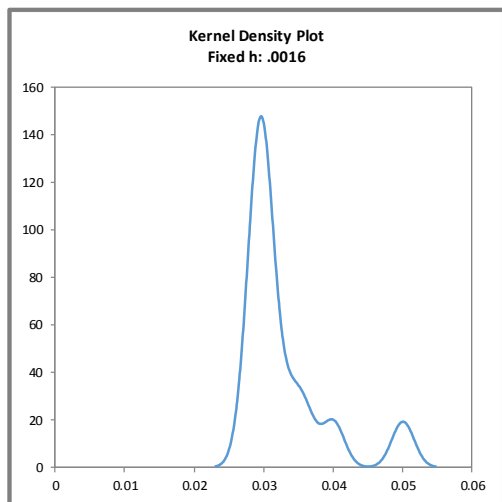
5.4 Information on the Proficiency Test (PT)

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

PT number	**DLA 50-2019**
PT name	**Free Amino Acids and Taurine in Food**
Sample matrix*	**Samples I + II:** Infant food (balanced diet, powder for the preparation of infant milk) / ingredients: Glucose syrup, vegetable oils, amino acids, vitamins, minerals and other food additives; Protein equivalent content <20%
Number of samples and sample amount	2 identical samples I + II: 10 g each.
Storage	Samples I + II: cooled 2 - 10°C (dry and dark)
Intentional use	Laboratory use only (quality control samples)
Parameter	quantitative: diverse free amino acids (see result submission file) and taurine
Methods of analysis	Analytical methods are optional
Notes to analysis	The analysis of PT samples should be performed like a routine laboratory analysis. In general we recommend to homogenize a representative sample amount before analysis according to good laboratory practice, especially in case of low sample weights.
Result sheet	The results for sample I and II as well as the final results calculated as mean of the double determination (samples I and II) should be filled in the result submission file. The recovery rates, if carried out, has to be included in the calculation.
Units	g/100g
Number of significant digits	at least 2
Further information	For information please specify: – Date of analysis – DLA-sample-numbers (for sample I and II) – Limit of detection – Assignment incl. Recovery – Recovery with the same matrix – Method is accredited
Result submission	The result submission file should be sent by e-mail to: **pt@dla-lvu.de**
Deadline	**the latest 18th October 2019**
Evaluation report	The evaluation report is expected to be completed 6 weeks after deadline of result submission and sent as PDF file by e-mail.
Coordinator and contact person of PT	Matthias Besler-Scharf PhD

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

6. Index of participant laboratories in alphabetical order

Teilnehmer / Participant	Ort / Town	Land / Country
		GREAT BRITAIN
		FRANCE
		GREAT BRITAIN
		Germany
		Germany
		Germany
		SPAIN
		Germany
		Germany
		Germany
		SWITZERLAND
		Germany
		Germany
		Germany
		Germany
		Germany
		ITALY
		Germany
		Germany
		Germany
		SPAIN
		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 inter-laboratory comparisons
4. ASU §64 LFGB: Planung und statistische Auswertung von Ringversuchen zur Methodenvalidierung / DIN ISO 5725 series part 1, 2 and 6 Accuracy (trueness and precision) of measurement methods and results
5. Verordnung / Regulation 882/2004/EU; Verordnung über über amtliche Kontrollen zur Überprüfung der Einhaltung des Lebensmittel- und Futtermittelrechts sowie der Bestimmungen über Tiergesundheit und Tierschutz / Regulation on official controls performed to ensure the verification of compliance with feed and food law, animal health and animal welfare rules
6. Evaluation of analytical methods used for regulation of food and drugs; W. Horwitz; Analytical Chemistry, 54, 67-76 (1982)
7. The International Harmonised Protocol for the Proficiency Testing of Ananlytical Laboratories ; J.AOAC Int., 76(4), 926 – 940 (1993)
8. A Horwitz-like funktion describes precision in proficiency test; M. Thompson, P.J. Lowthian; Analyst, 120, 271-272 (1995)
9. Protocol for the design, conduct and interpretation of method performance studies; W. Horwitz; Pure & Applied Chemistry, 67, 331-343 (1995)
10. Recent trends in inter-laboratory precision at ppb and sub-ppb concentrations in relation to fitness for purpose criteria in proficiency testing; M. Thompson; Analyst, 125, 385-386 (2000)
11. The International Harmonised Protocol for the Proficiency Testing of Analytical Chemistry Laboratories; Pure Appl Chem, 78, 145 – 196 (2006)
12. AMC Kernel Density - Representing data distributions with kernel density estimates, amc technical brief, Editor M Thompson, Analytical Methods Committee, AMCTB No 4, Revised March 2006 and Excel Add-in Kernel.xla 1.0e by Royal Society of Chemistry
13. EURACHEM/CITAC Leitfaden, Ermittlung der Messunsicherheit bei analytischen Messungen (2003); Quantifying Uncertainty in Analytical Measurement (1999)
14. GMP+ Feed Certification scheme, Module: Feed Safety Assurance, chapter 5.7 Checking procedure for the process accuracy of compound feed with micro tracers in GMP+ BA2 Control of residues, Version: 1st of January 2015 GMP+ International B.V.
15. MTSE SOP No. 010.01 (2014): Quantitative measurement of mixing uniformity and carry-over in powder mixtures with the rotary detector technique, MTSE Micro Tracers Services Europe GmbH
16. Homogeneity and stability of reference materials; Linsinger et al.; Accred Qual Assur, 6, 20-25 (2001)
17. AOAC Official Methods of Analysis: Guidelines for Standard Method Performance Requirements, Appendix F, p. 2, AOAC Int (2016)

18. ASU §64 LFGB L 49.07-1 Bestimmung der Aminosäuren in Aminosäurengemischen (1985) [Determination of amino acids in amino acid mixtures]
19. 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]
20. 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]
21. 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