DLA
Dienstleistung
Lebensmittel
Analytik GbR

Evaluation-Report
proficiency test

DLA 32/2014

Total amino acids

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Content

1 Introduction

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

2 Evaluation

2.1 Assigned value

Because the analysed material was no certified reference material the robust mean of the submitted results was used as assigned value X. The distribution of submitted results showed no hint for bimodal distribution or other reasons for a higher variability.

2.2 Standard deviation

For comparison to the target standard deviation a robust standard deviation (Sx) was calculated.

2.3 Outliers

Statistical outliers were determined by Mandel´s-H-Statistic (95% significance). Detected outliers were stated for information only, when z-score was < -2 or > 2.

2.4 Target standard deviation

The target standard deviation of the assigned value is determined according to the following methods.

2.4.1 General model (Horwitz / Thompson)

The relative target standard deviation in % of the assigned value was derived from following equation (Horwitz)

$$\sigma_{(\%)} = 2^{(1-0,5\log X)} .$$

From the result the target standard deviation was calculated

$$\sigma = X * \sigma_{(\%)} / 100.$$

For analytes with a content below 120 µg/kg after the evaluation of a lot of mycotoxin- proficiency testing schemes after 1997 it was suggested for the target standard deviation a steady value of 22 % (Thompson), analogical:

$$\sigma = 0,22\ C / mr;$$

with σ = Target standard deviation for contents < 120 µg/kg
C = assigned content, expressed as a dimensionless mass ratio
mr = dimensionless mass ratio.

2.4.2 Precision experiment

Using the reproducibility standard deviation σ_R and the repeatability standard deviation σ_r of a precision experiment the between-laboratories standard deviation (σ_L) can be calculated :

$$\sigma_L = \sqrt{(\sigma_R^2 - \sigma_r^2)} .$$

And then, using the number of replicate measurements n, each participant is to perform, the standard deviation for proficiency assessment is calculated :

$$\sigma = \sqrt{(\sigma_L^2 + (\sigma_r^2 / n))} .$$

If available, the precision data from official methods for each parameter are used to calculate the target standard deviation.

2.5 *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) the result (x) of the participant is deviating from the assigned value (X).

Participants' z-scores are derived as:

$$z = (x - X) / \sigma \quad ;$$

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

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

2.6 *z'-Score*

The z'-Score can be used to assess the results of the participants in case the standard uncertainty must be considered (s. 2.8).

The calculation is carried out as follows (3)

$$z' = (x - X) / \sqrt{\hat{\sigma}^2 + u_X^2}$$

For the following evaluation $\sqrt{\hat{\sigma}^2 + u_X^2}$ is defined as $\hat{\sigma}'$, the target standard deviation considering the standard uncertainty of the results.

The requirements for the analytical performance are considered as fulfilled then, if

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

2.7 *Quotient S^x/σ*

Following the Horrat-value the results of a proficiency-test (PT) can be considered convincing, if the quotient of robust standard deviation and target standard deviation 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.

2.8 Standard uncertainty

The assigned value X has a standard uncertainty u_X that depends on the analytical method, differences between the analytical methods used, the test material, the number of participant laboratories and perhaps on other factors. The standard uncertainty u_X for this PT is calculated as follows

$$u_X = 1{,}25 * S^x / \sqrt{(p)} \quad .$$

If $u_X \leq 0{,}3 * \sigma$ the standard uncertainty of the assigned value needs not be included in the interpretation of the results of the PT. The quotient u_X / σ is given in the evaluation.

3 Realisation

3.1 Test material

Test material was a sports product on basis of whey protein. The protein content was adjusted to app. 50% with addition of glucose. App. 600g of the material were mixed and put in portions of app. 10 gram. The portions where numbered chronologically.

3.1.1 *Homogeneity*

The calculation of the repeatability standard deviation of the participants for alanine and histidine was used as an indicator of homogeneity. The results are 1,9% and 3,7% and in the same magnitude as the repeatability standard deviation of the German official method. The repeatability standard deviation of the participants is given in the documentation.
In the documentation the portion numbers are graphically assigned to the results of alanine and histidine. There is no trend recognizable in the results which could suggest inhomogeneity.

3.2 Tests

Two test samples were sent to every participating laboratory in the 40th week of 2014. The test method was optional. The tests should be finished at 14.11.2014.

3.3 Results and statistic evaluation

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

The statistical evaluation was carried out according to 2.4.2 of this report if at least 7 results were submitted, which were:
The targe standard deviation,σ, for each amino acid was calculated from ASU L 49.07-2.
For information the target standard deviation according to 2.4.1, „Horwitz", is stated additionally.
Cysteine and Cystine were evaluated as sum of both.
Phenylalanin was evaluated with the precision data from ASU L 49.07-1, for Tryptophan the precision data from ASU L 49.07-3 were used.

For Taurine only two results were submitted, which are documented.
Queried and documented were further results and the testing method applied.
13 out of 14 participants submitted results, two of them after consultation within two weeks after deadline.

4 Results

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

In the upper table the characteristics are listed:

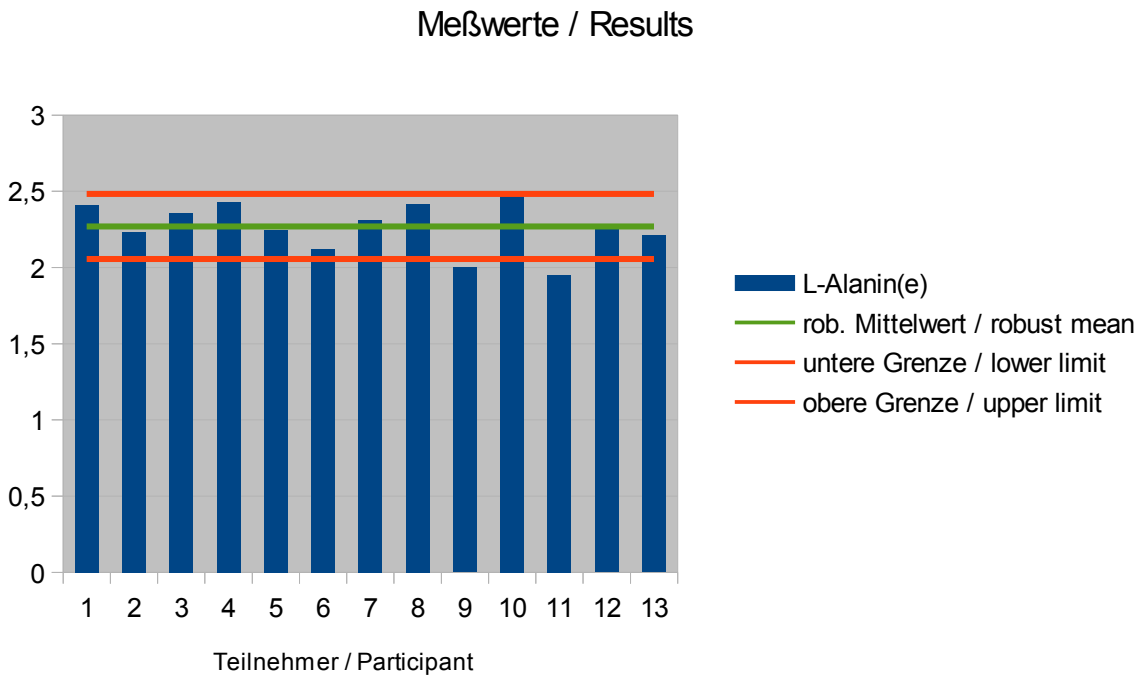
Number of the results
Number of outliers
Mean
Median
Robust mean (X)
Robust standard deviation(S^x)
Target standard deviation (σ´)
Target standard deviation Horwitz (for information)
Lower limit of target range (X – 2σ´)
Upper limit of target range (X + 2σ´)
Quotient S^x/σ
Standard uncertainty u_x
Quotient u_X/σ
Results in target range.

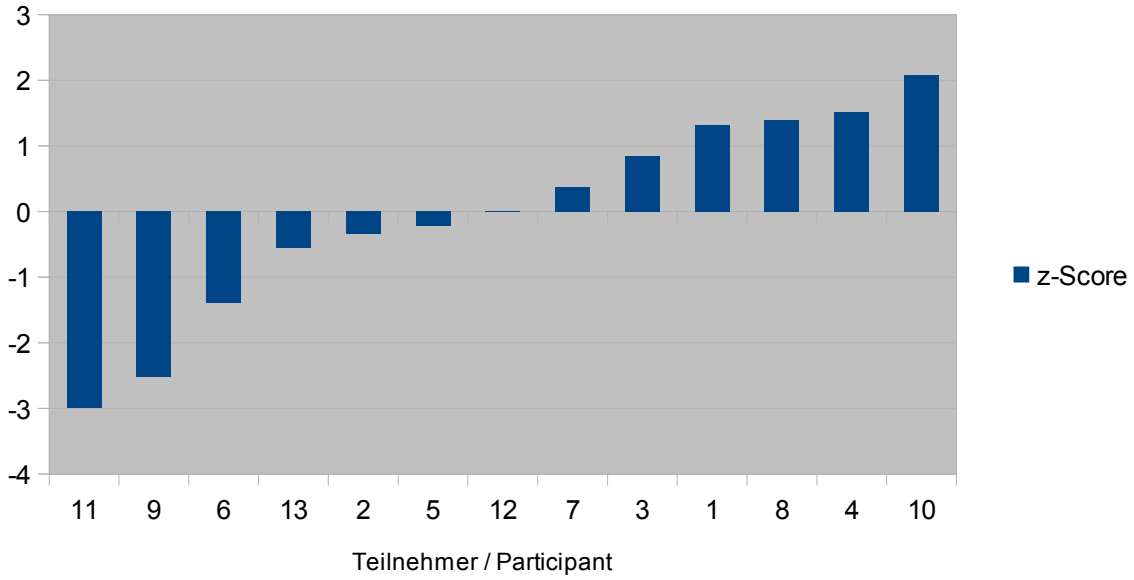
In the lower table -laboratories- the individual results of the participating laboratory are listed:

Evaluation number	test result	deviation from assigned value	Z-Score (σ)	Z-Score (Horwitz) for information	remarks

4.1 Alanin(e) in g/100 g

Statistic Data	
Number of the results	13
Number of outliers	1
Mean	2,26
Median	2,27
Robust mean (X)	2,27
Robust standard deviation (S^x)	0,179
Target standard deviation (sigma)	0,107
Target standard deviation (Horwitz) for information	0,080
Lower limit of target range	2,06
Upper limit of target range	2,48
Quotient S^x/σ	1,7
Standard uncertainty U^x	0,062
Quotient U^x/σ	0,6
Results in target range	10
Percent in target range	77

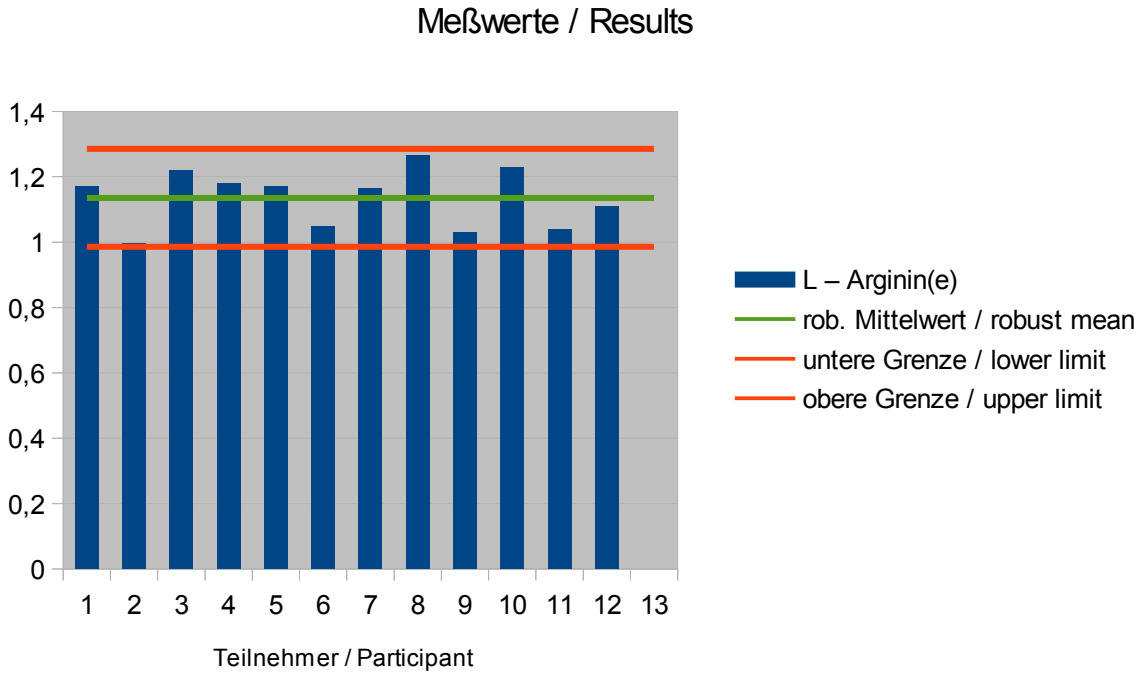


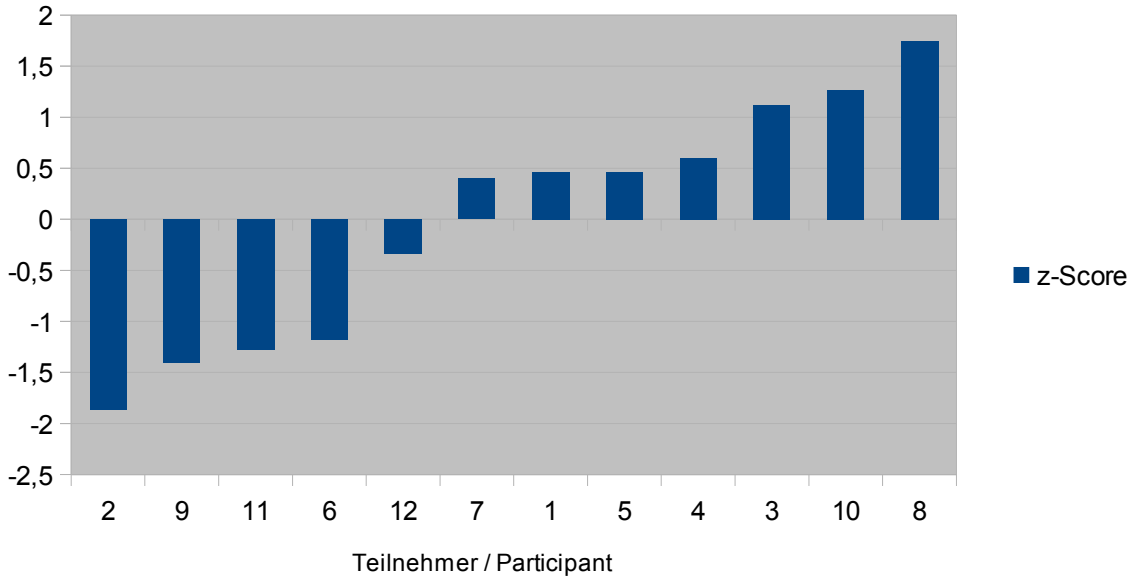


Auswerte nummer / Evaluation number	L-Alanin(e)	Abweichung / Deviation	z-Score	z-Score (Horwitz) zur info	Hinweis / Remark
1	2,41	0,14	1,3	1,8	
2	2,23	-0,04	-0,3	-0,5	
3	2,36	0,09	0,8	1,1	
4	2,43	0,16	1,5	2,0	
5	2,25	-0,02	-0,2	-0,3	
6	2,12	-0,15	-1,4	-1,9	
7	2,31	0,04	0,4	0,5	
8	2,42	0,15	1,4	1,9	
9	2	-0,27	-2,5	-3,4	
10	2,49	0,22	2,1	2,8	
11	1,95	-0,32	-3,0	-4,0	Ausreisser / Outlier
12	2,27	0	0,0	0,0	
13	2,21	-0,06	-0,6	-0,7	

4.2 Arginin(e) in g/100 g

Statistic Data	
Number of the results	12
Number of outliers	0
Mean	1,14
Median	1,17
Robust mean (X)	1,14
Robust standard deviation (S^x)	0,101
Target standard deviation (sigma)	0,075
Target standard deviation (Horwitz) for information	0,045
Lower limit of target range	0,99
Upper limit of target range	1,28
Quotient S^x/σ	1,3
Standard uncertainty U^x	0,036
Quotient U^x/σ	0,5
Results in target range	12
Percent in target range	100

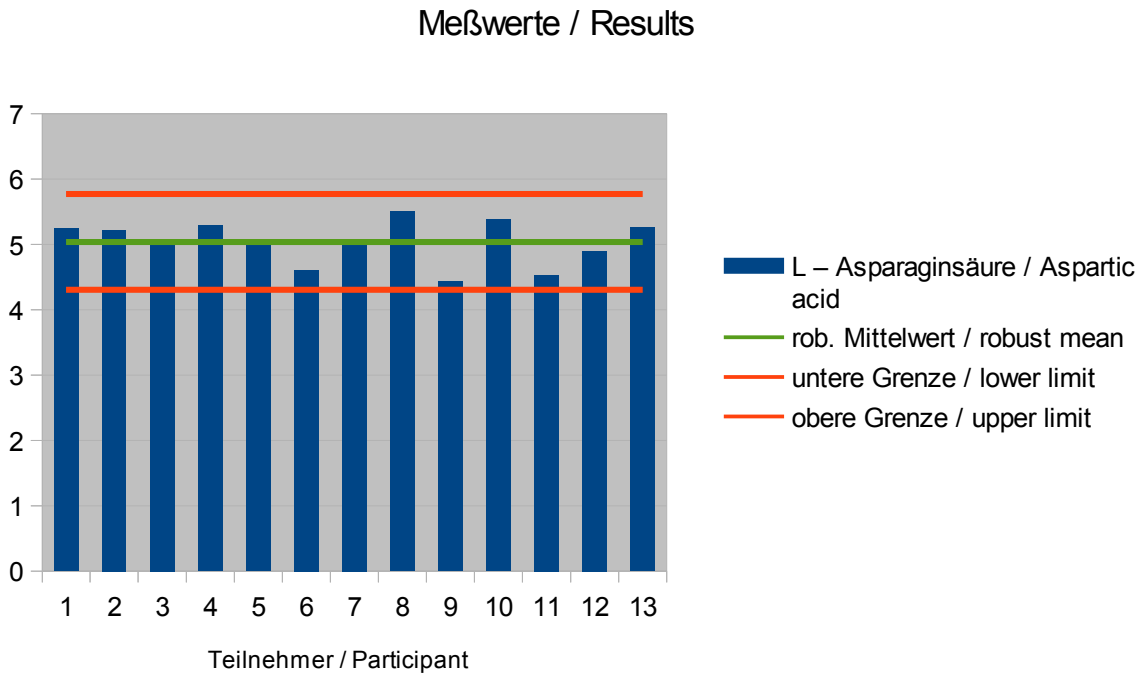


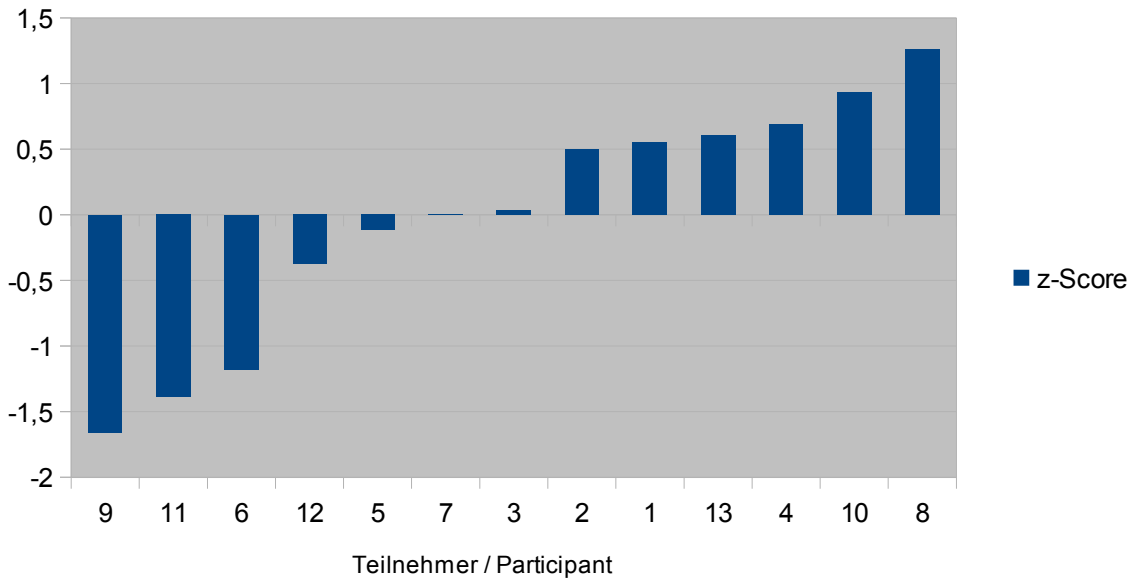


Auswerte nummer / Evaluation number	L - Arginin(e)	Abweichung / Deviation	z-Score	z-Score (Horwitz) zur info	Hinweis / Remark
1	1,17	0,03	0,5	0,8	
2	1	-0,14	-1,9	-3,1	
3	1,22	0,08	1,1	1,9	
4	1,18	0,04	0,6	1,0	
5	1,17	0,03	0,5	0,8	
6	1,05	-0,09	-1,2	-2,0	
7	1,17	0,03	0,4	0,7	
8	1,27	0,13	1,7	2,9	
9	1,03	-0,11	-1,4	-2,4	
10	1,23	0,09	1,3	2,1	
11	1,04	-0,1	-1,3	-2,1	
12	1,11	-0,03	-0,3	-0,6	
13					

4.3 Asparaginsäure (Aspartic acid) in g/100 g

Statistic Data	
Number of the results	13
Number of outliers	0
Mean	5,03
Median	5,05
Robust mean (X)	5,04
Robust standard deviation (S^x)	0,372
Target standard deviation (sigma)	0,366
Target standard deviation (Horwitz) for information	0,158
Lower limit of target range	4,31
Upper limit of target range	5,77
Quotient S^x/σ	1,0
Standard uncertainty U^x	0,129
Quotient U^x/σ	0,4
Results in target range	13
Percent in target range	100

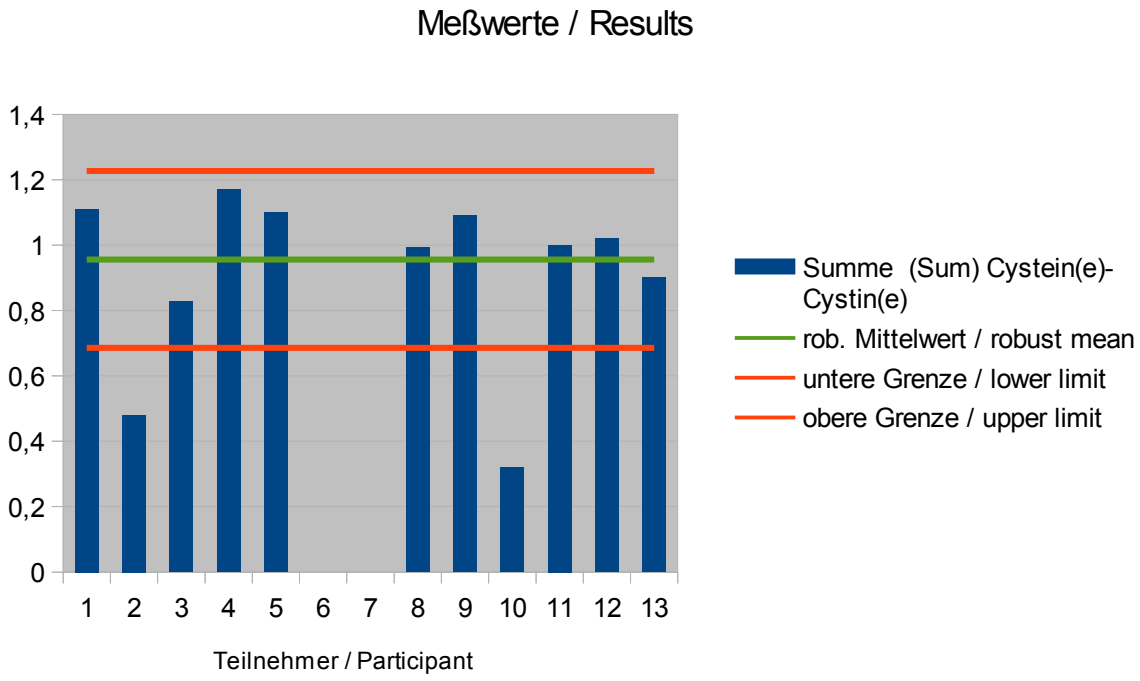


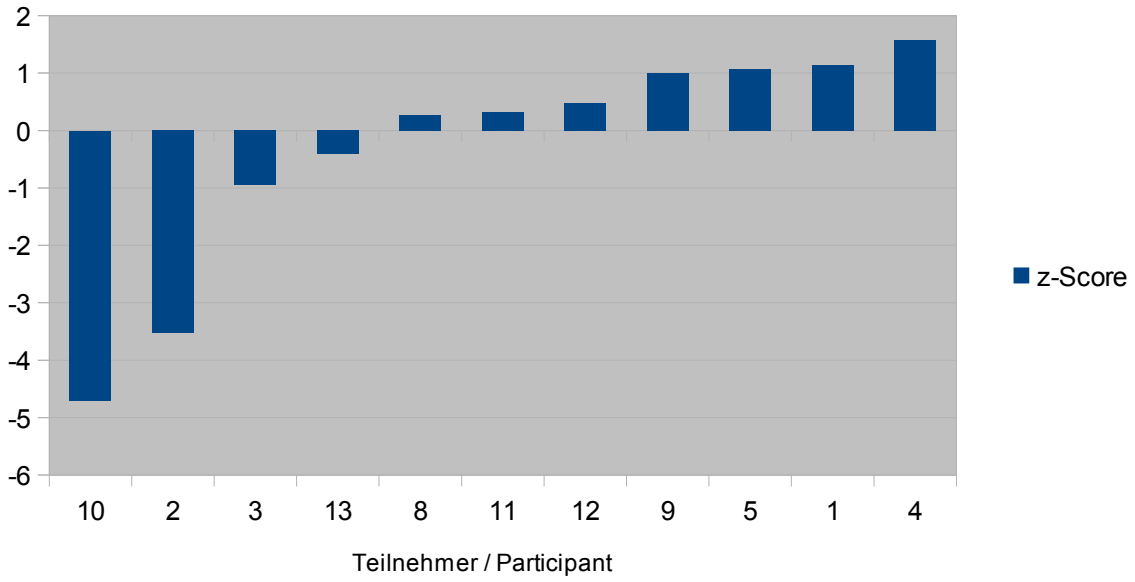


Auswerte nummer / Evaluation number	L - Asparaginsäure / Aspartic acid	Abweichung / Deviation	z-Score	z-Score (Horwitz) zur info	Hinweis / Remark
1	5,24	0,2	0,6	1,3	
2	5,22	0,18	0,5	1,2	
3	5,05	0,01	0,0	0,1	
4	5,29	0,25	0,7	1,6	
5	5	-0,04	-0,1	-0,3	
6	4,61	-0,43	-1,2	-2,7	
7	5,04	0	0,0	0,0	
8	5,5	0,46	1,3	2,9	
9	4,43	-0,61	-1,7	-3,8	
10	5,38	0,34	0,9	2,2	
11	4,53	-0,51	-1,4	-3,2	
12	4,9	-0,14	-0,4	-0,9	
13	5,26	0,22	0,6	1,4	

4.4 Summe (Sum) Cystein(e) / Cystin(e) in g/100 g

Statistic Data	
Number of the results	11
Number of outliers	1
Mean	0,910
Median	1,000
Robust mean (X)	0,956
Robust standard deviation (S^x)	0,203
Target standard deviation (sigma)	0,135
Target standard deviation (Horwitz) for information	0,039
Lower limit of target range	0,686
Upper limit of target range	1,226
Quotient S^x/σ	1,5
Standard uncertainty U^x	0,076
Quotient U^x/σ	0,6
Results in target range	9
Percent in target range	82

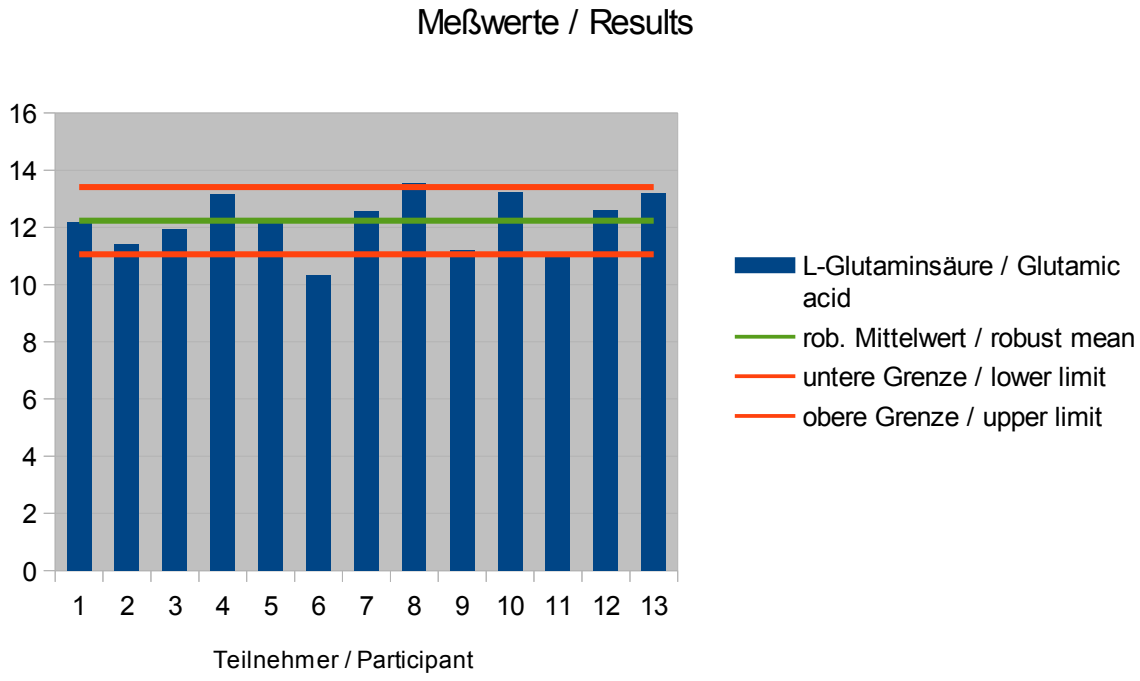


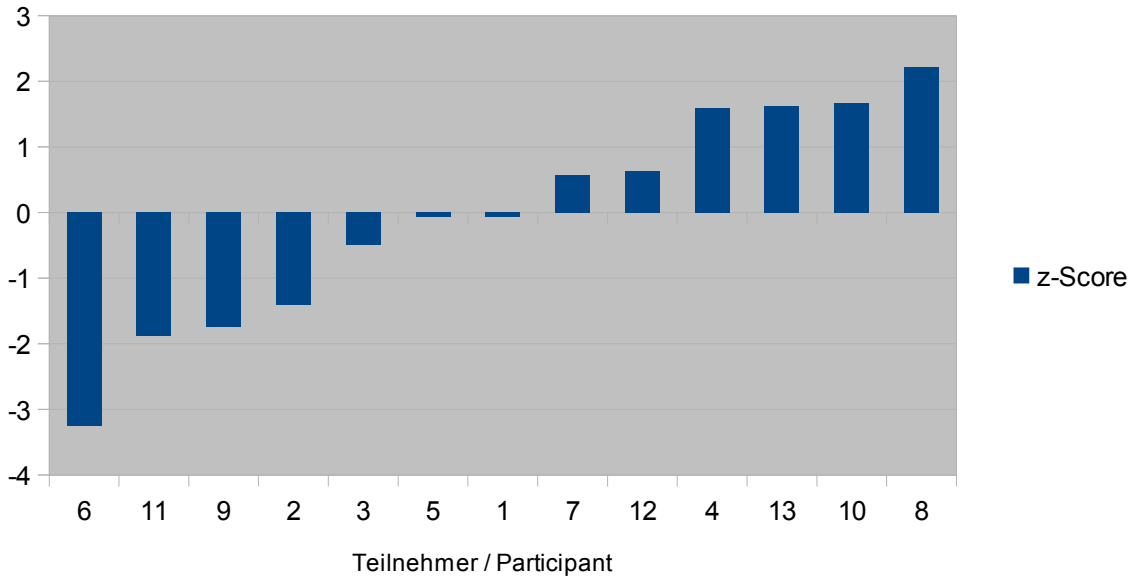


Auswerte nummer / Evaluation number	Summe (Sum) Cystein(e)-Cystin(e)	Abweichung / Deviation	z-Score	z-Score (Horwitz) zur info	Hinweis / Remark
1	1,11	0,15	1,1	4,0	
2	0,48	-0,48	-3,5	-12,4	
3	0,83	-0,13	-0,9	-3,3	
4	1,17	0,21	1,6	5,6	
5	1,1	0,14	1,1	3,7	
6					
7					
8	0,99	0,04	0,3	1,0	
9	1,09	0,13	1,0	3,5	
10	0,32	-0,64	-4,7	-16,5	Ausreisser / Outlier
11	1	0,04	0,3	1,1	
12	1,02	0,06	0,5	1,7	
13	0,9	-0,06	-0,4	-1,5	

4.5 Glutaminsäure (Glutamic acid) in g/100 g

Statistic Data	
Number of the results	13
Number of outliers	1
Mean	12,2
Median	12,2
Robust mean (X)	12,2
Robust standard deviation (S^x)	1,05
Target standard deviation (sigma)	0,588
Target standard deviation (Horwitz) for information	0,336
Lower limit of target range	11,1
Upper limit of target range	13,4
Quotient S^x/σ	1,8
Standard uncertainty U^x	0,363
Quotient U^x/σ	0,6
Results in target range	11
Percent in target range	85

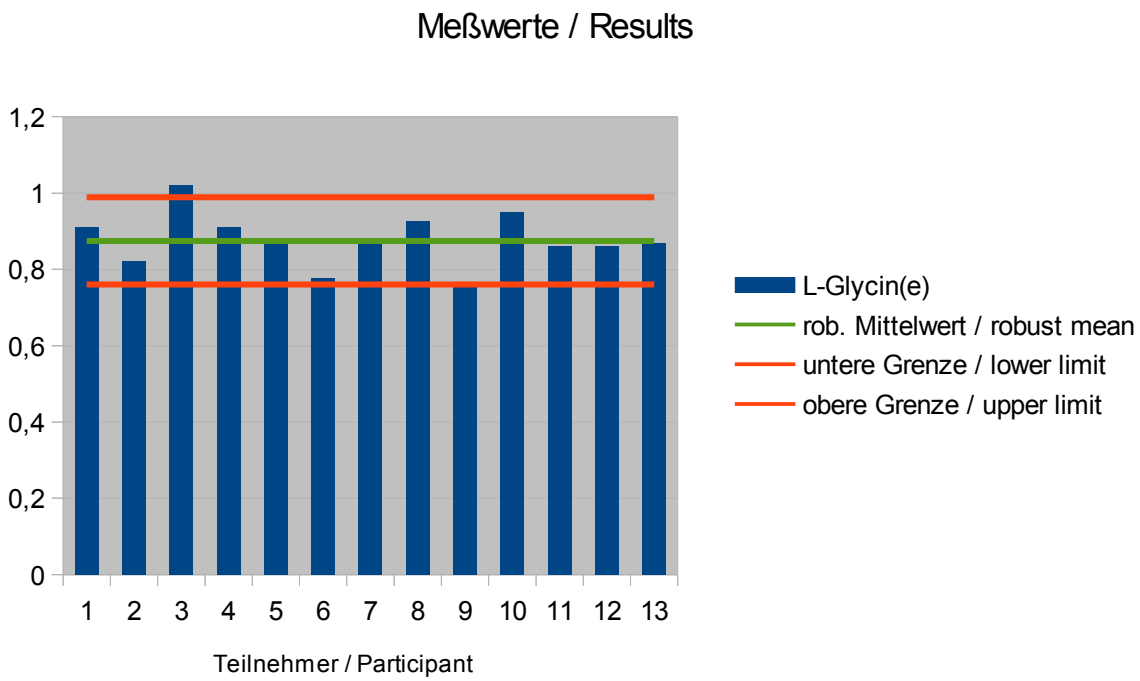


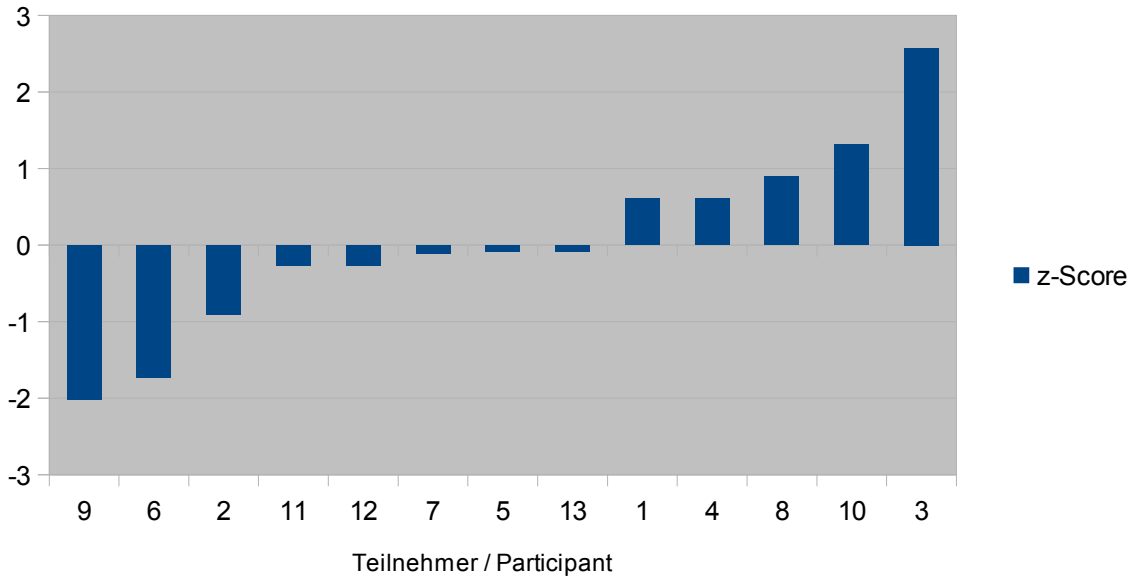


Auswerte nummer / Evaluation number	L-Glutaminsäure / Glutamic acid	Abweichung / Deviation	z-Score	z-Score (Horwitz) zur info	Hinweis / Remark
1	12,2	-0,03	-0,1	-0,1	
2	11,41	-0,83	-1,4	-2,5	
3	11,94	-0,29	-0,5	-0,9	
4	13,17	0,94	1,6	2,8	
5	12,2	-0,03	-0,1	-0,1	
6	10,32	-1,91	-3,3	-5,7	Ausreisser / Outlier
7	12,57	0,34	0,6	1,0	
8	13,54	1,3	2,2	3,9	
9	11,21	-1,02	-1,7	-3,1	
10	13,22	0,99	1,7	2,9	
11	11,13	-1,1	-1,9	-3,3	
12	12,61	0,38	0,6	1,1	
13	13,19	0,96	1,6	2,8	

4.6 Glycin(e) in g/100 g

Statistic Data	
Number of the results	13
Number of outliers	1
Mean	0,877
Median	0,870
Robust mean (X)	0,875
Robust standard deviation (S^x)	0,070
Target standard deviation (sigma)	0,057
Target standard deviation (Horwitz) for information	0,036
Lower limit of target range	0,761
Upper limit of target range	0,989
Quotient S^x/σ	1,2
Standard uncertainty U^x	0,024
Quotient U^x/σ	0,4
Results in target range	12
Percent in target range	92

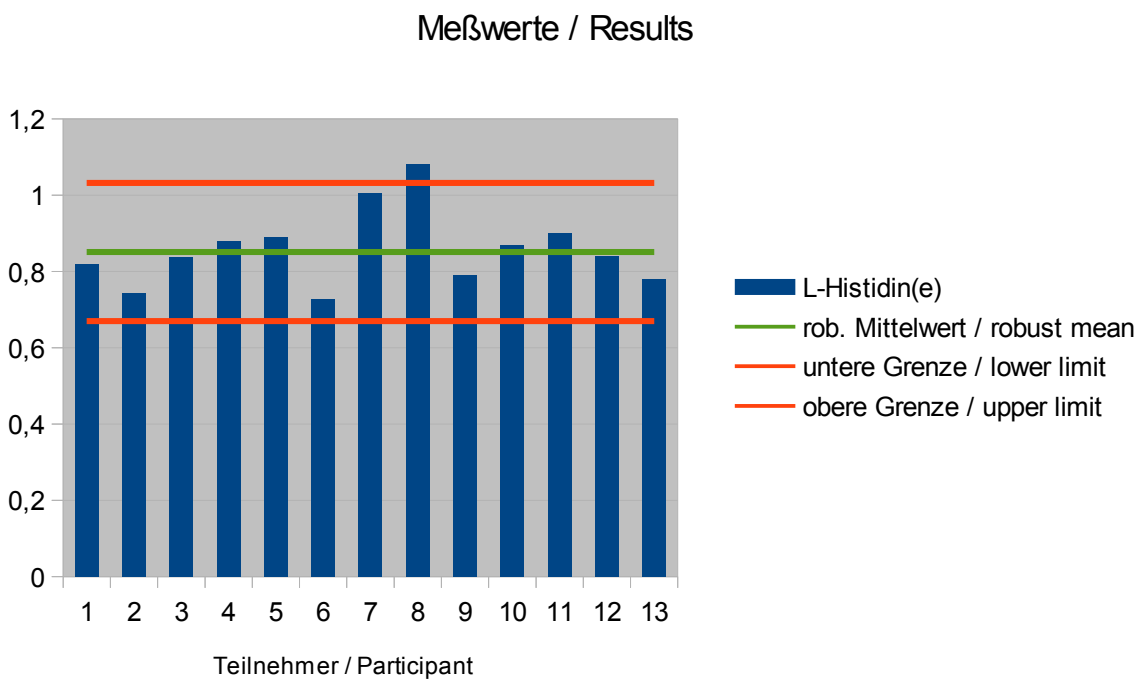


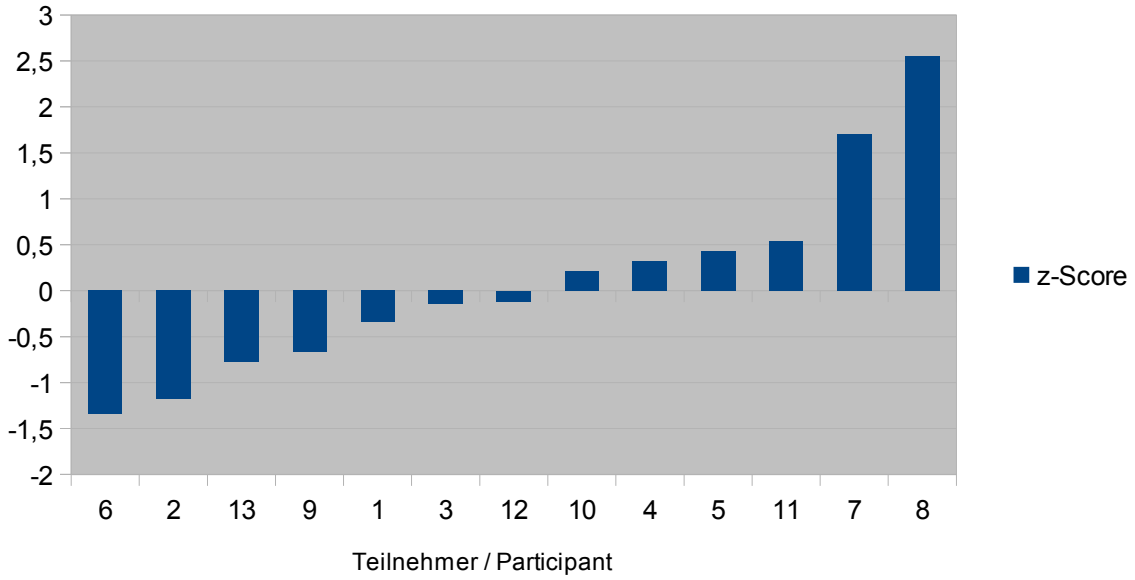


Auswerte nummer / Evaluation number	L-Glycin(e)	Abweichung / Deviation	z-Score	z-Score (Horwitz) zur info	Hinweis / Remark
1	0,91	0,04	0,6	1,0	
2	0,82	-0,05	-0,9	-1,5	
3	1,02	0,15	2,6	4,1	Ausreisser / Outlier
4	0,91	0,04	0,6	1,0	
5	0,87	0	-0,1	-0,1	
6	0,78	-0,1	-1,7	-2,8	
7	0,87	-0,01	-0,1	-0,2	
8	0,93	0,05	0,9	1,4	
9	0,76	-0,11	-2,0	-3,2	
10	0,95	0,08	1,3	2,1	
11	0,86	-0,01	-0,3	-0,4	
12	0,86	-0,01	-0,3	-0,4	
13	0,87	0	-0,1	-0,1	

4.7 Histidin(e) in g/100 g

Statistic Data	
Number of the results	13
Number of outliers	1
Mean	0,859
Median	0,840
Robust mean (X)	0,851
Robust standard deviation (S^x)	0,0926
Target standard deviation (sigma)	0,0906
Target standard deviation (Horwitz) for information	0,0349
Lower limit of target range	0,670
Upper limit of target range	1,032
Quotient S^x/σ	1,0
Standard uncertainty U^x	0,0321
Quotient U^x/σ	0,4
Results in target range	12
Percent in target range	92

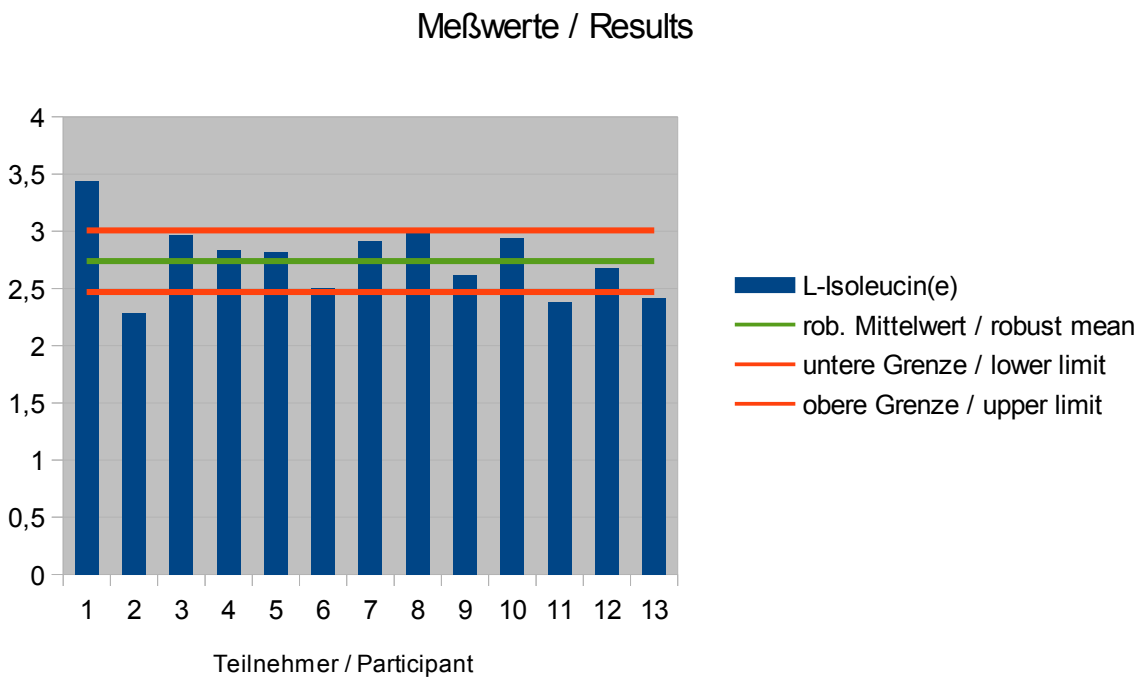


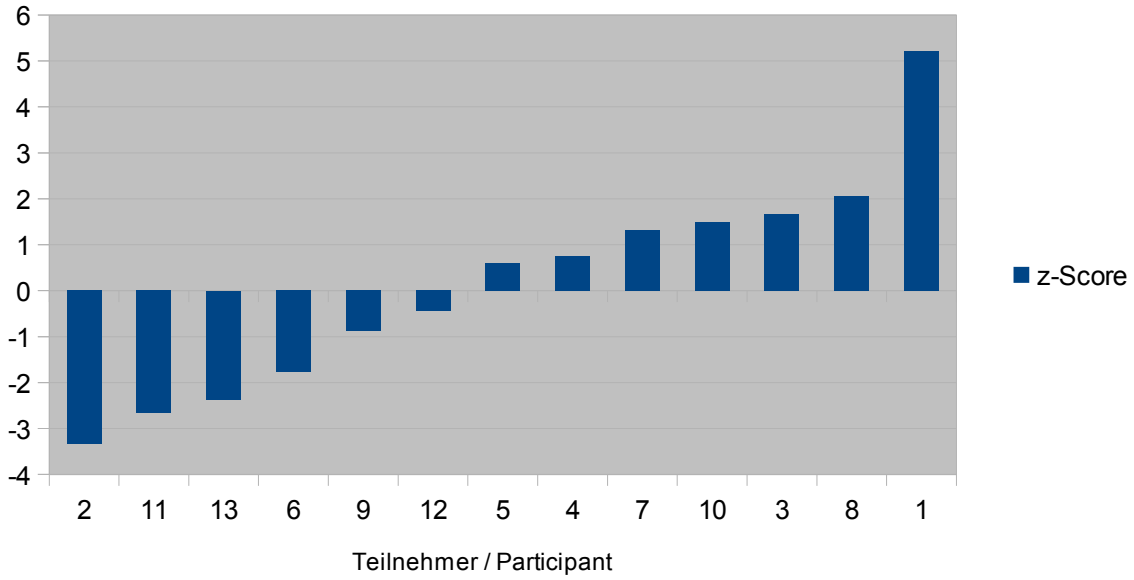


Auswerte nummer / Evaluation number	L-Histidin(e)	Abweichung / Deviation	z-Score	z-Score (Horwitz) zur info	Hinweis / Remark
1	0,82	-0,03	-0,3	-0,9	
2	0,74	-0,11	-1,2	-3,1	
3	0,84	-0,01	-0,1	-0,4	
4	0,88	0,03	0,3	0,8	
5	0,89	0,04	0,4	1,1	
6	0,73	-0,12	-1,3	-3,5	
7	1,01	0,15	1,7	4,4	
8	1,08	0,23	2,6	6,6	Ausreisser / Outlier
9	0,79	-0,06	-0,7	-1,7	
10	0,87	0,02	0,2	0,6	
11	0,9	0,05	0,5	1,4	
12	0,84	-0,01	-0,1	-0,3	
13	0,78	-0,07	-0,8	-2,0	

4.8 Isoleucin(e) in g/100 g

Statistic Data	
Number of the results	13
Number of outliers	1
Mean	2,76
Median	2,82
Robust mean (X)	2,74
Robust standard deviation (S^x)	0,320
Target standard deviation (sigma)	0,135
Target standard deviation (Horwitz) for information	0,094
Lower limit of target range	2,47
Upper limit of target range	3,01
Quotient S^x/σ	2,4
Standard uncertainty U^x	0,111
Quotient U^x/σ	0,8
Results in target range	8
Percent in target range	62

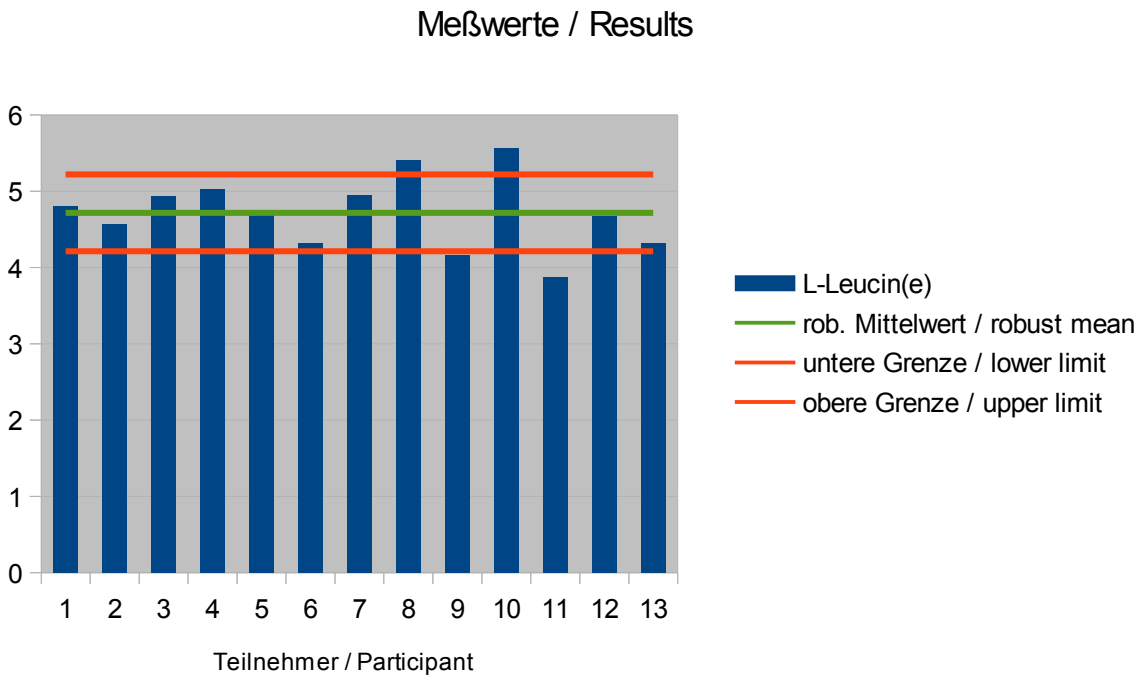


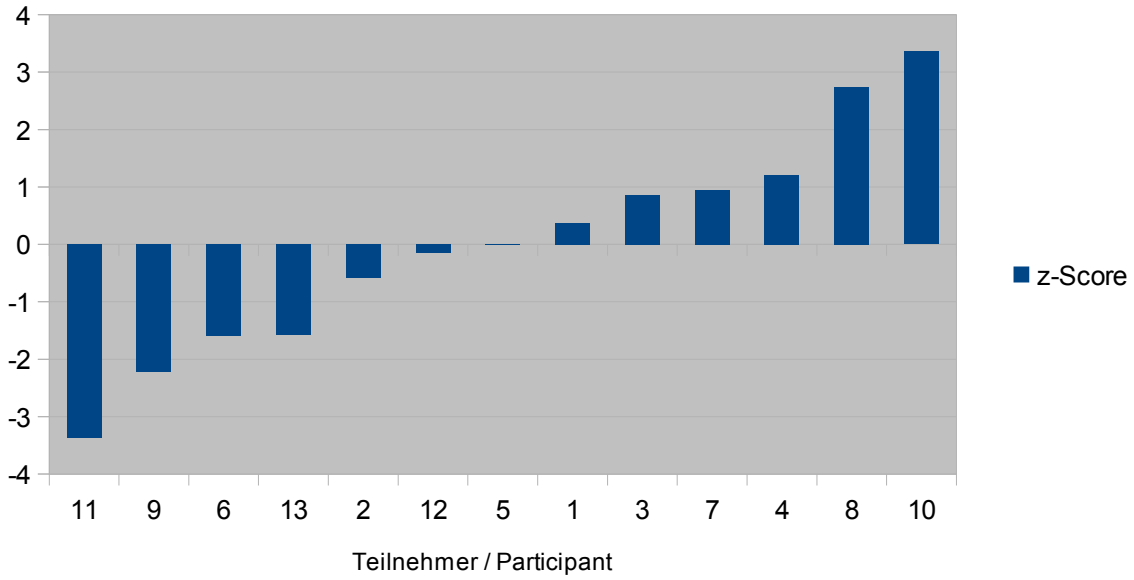


Auswerte nummer / Evaluation number	L-Isoleucin(e)	Abweichung / Deviation	z-Score	z-Score (Horwitz) zur info	Hinweis / Remark
1	3,44	0,7	5,2	7,4	Ausreisser / Outlier
2	2,29	-0,45	-3,3	-4,8	
3	2,96	0,22	1,7	2,4	
4	2,84	0,1	0,7	1,1	
5	2,82	0,08	0,6	0,9	
6	2,5	-0,24	-1,8	-2,5	
7	2,92	0,18	1,3	1,9	
8	3,02	0,28	2,1	2,9	
9	2,62	-0,12	-0,9	-1,3	
10	2,94	0,2	1,5	2,1	
11	2,38	-0,36	-2,7	-3,8	
12	2,68	-0,06	-0,4	-0,6	
13	2,42	-0,32	-2,4	-3,4	

4.9 Leucin(e) in g/100 g

Statistic Data	
Number of the results	13
Number of outliers	0
Mean	4,72
Median	4,72
Robust mean (X)	4,72
Robust standard deviation (S^x)	0,524
Target standard deviation (sigma)	0,251
Target standard deviation (Horwitz) for information	0,149
Lower limit of target range	4,22
Upper limit of target range	5,22
Quotient S^x/σ	2,1
Standard uncertainty U^x	0,182
Quotient U^x/σ	0,7
Results in target range	9
Percent in target range	69

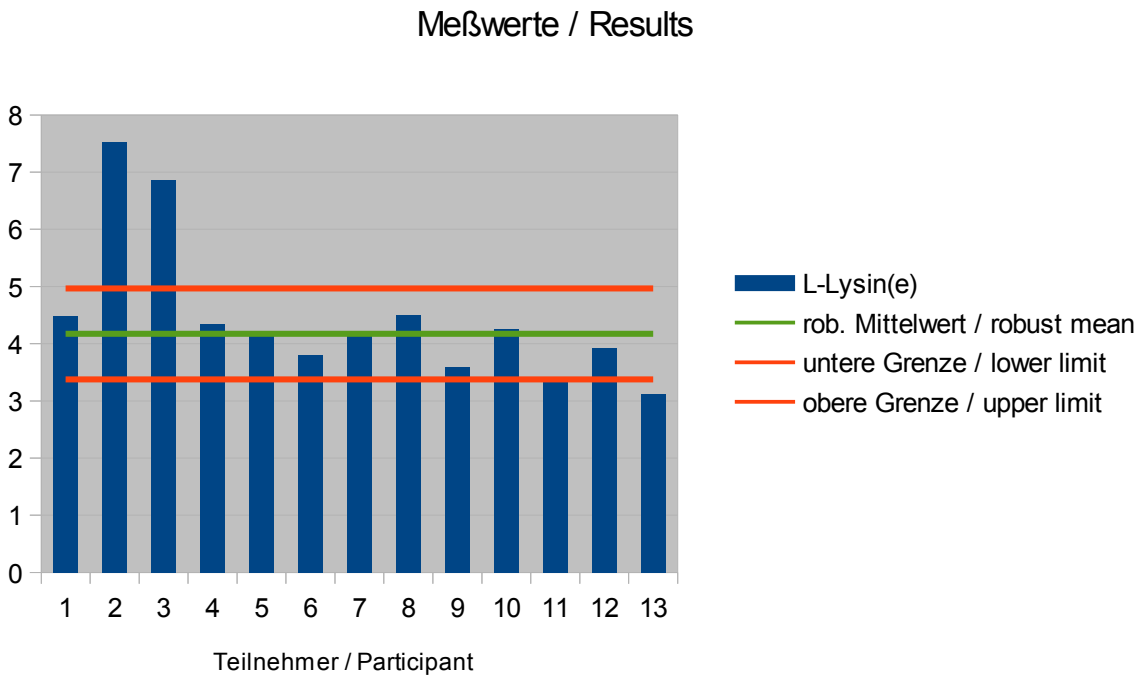


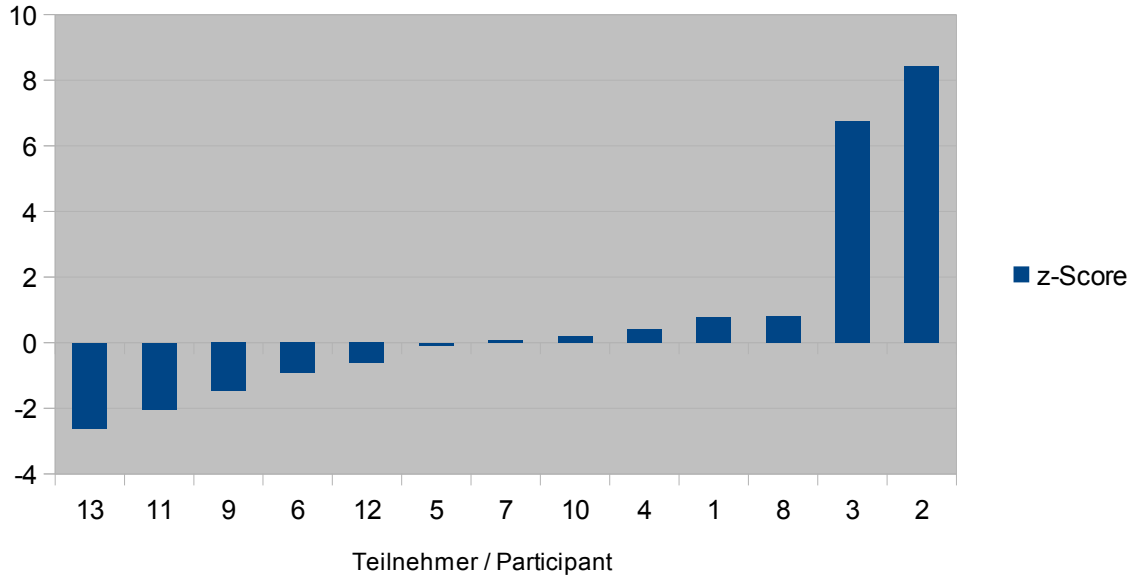


Auswerte nummer / Evaluation number	L-Leucin(e)	Abweichung / Deviation	z-Score	z-Score (Horwitz) zur info	Hinweis / Remark
1	4,81	0,09	0,4	0,6	
2	4,57	-0,15	-0,6	-1,0	
3	4,93	0,22	0,9	1,5	
4	5,02	0,3	1,2	2,0	
5	4,72	0	0,0	0,0	
6	4,32	-0,4	-1,6	-2,7	
7	4,95	0,24	0,9	1,6	
8	5,4	0,69	2,7	4,6	
9	4,16	-0,56	-2,2	-3,7	
10	5,56	0,84	3,4	5,6	
11	3,87	-0,85	-3,4	-5,7	
12	4,68	-0,04	-0,1	-0,2	
13	4,32	-0,4	-1,6	-2,7	

4.10 Lysin(e) in g/100 g

Statistic Data	
Number of the results	13
Number of outliers	2
Mean	4,47
Median	4,21
Robust mean (X)	4,17
Robust standard deviation (S^x)	0,721
Target standard deviation (sigma)	0,399
Target standard deviation (Horwitz) for information	0,135
Lower limit of target range	3,37
Upper limit of target range	4,97
Quotient S^x/σ	1,8
Standard uncertainty U^x	0,250
Quotient U^x/σ	0,6
Results in target range	10
Percent in target range	77

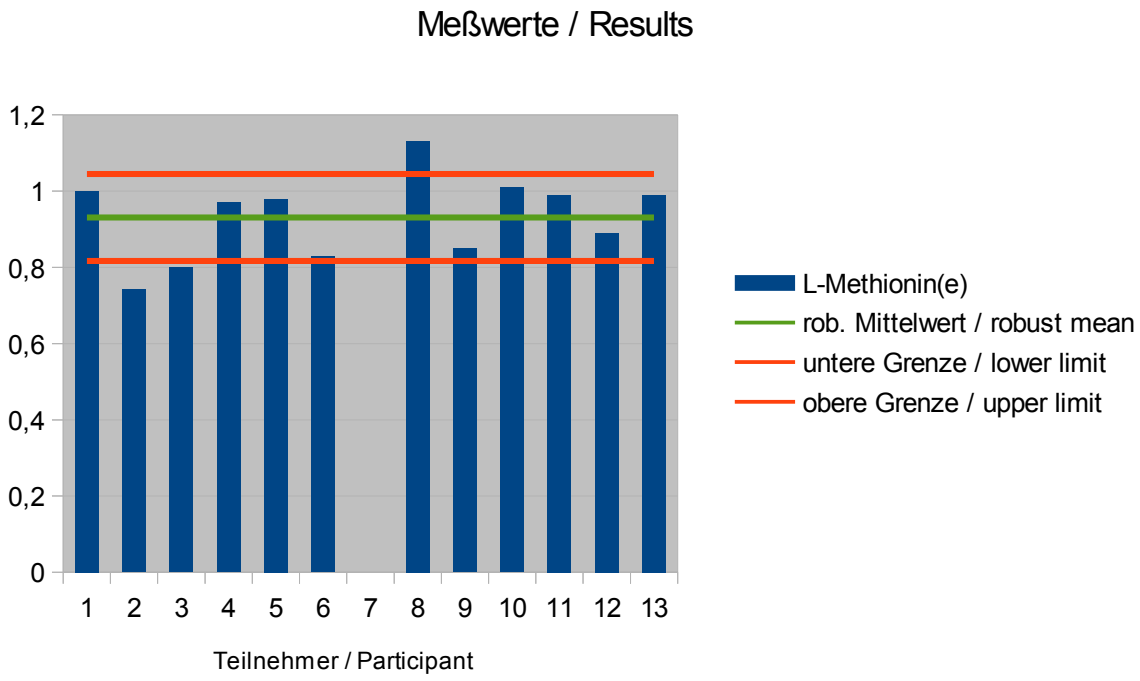


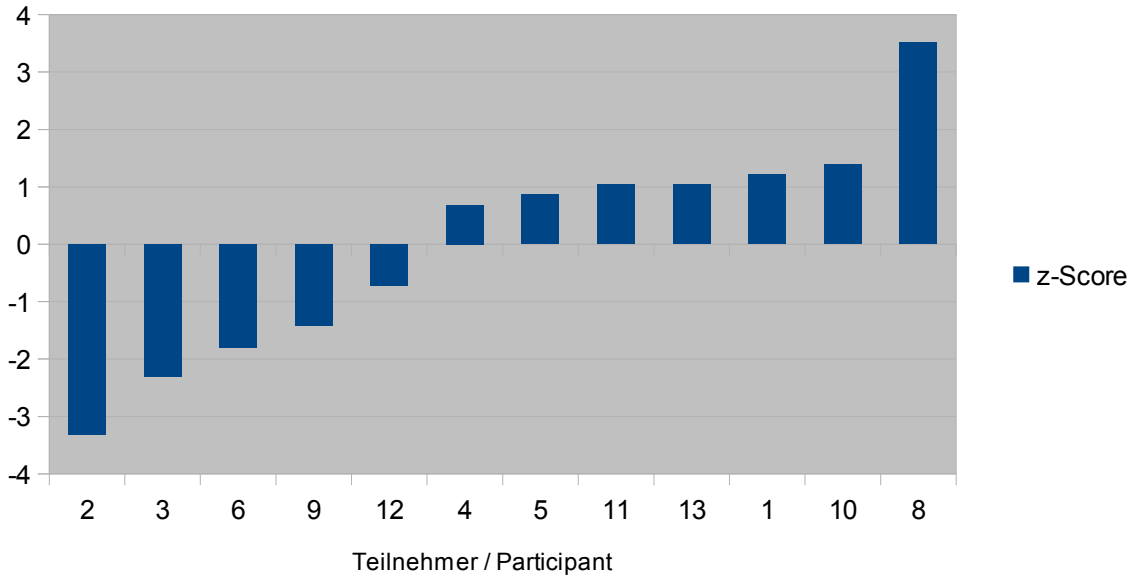


Auswerte nummer / Evaluation number	L-Lysin(e)	Abweichung / Deviation	z-Score	z-Score (Horwitz) zur info	Hinweis / Remark
1	4,48	0,31	0,8	2,3	
2	7,53	3,36	8,4	25,0	Ausreisser / Outlier
3	6,86	2,69	6,7	20,0	Ausreisser / Outlier
4	4,34	0,17	0,4	1,3	
5	4,14	-0,03	-0,1	-0,2	
6	3,81	-0,36	-0,9	-2,7	
7	4,21	0,03	0,1	0,2	
8	4,5	0,32	0,8	2,4	
9	3,59	-0,58	-1,5	-4,3	
10	4,25	0,08	0,2	0,6	
11	3,36	-0,81	-2,0	-6,0	
12	3,93	-0,24	-0,6	-1,8	
13	3,13	-1,04	-2,6	-7,7	

4.11 Methionin(e) in g/100 g

Statistic Data	
Number of the results	12
Number of outliers	0
Mean	0,932
Median	0,975
Robust mean (X)	0,931
Robust standard deviation (S^x)	0,119
Target standard deviation (sigma)	0,057
Target standard deviation (Horwitz) for information	0,038
Lower limit of target range	0,817
Upper limit of target range	1,045
Quotient S^x/σ	2,1
Standard uncertainty U^x	0,043
Quotient U^x/σ	0,8
Results in target range	9
Percent in target range	75

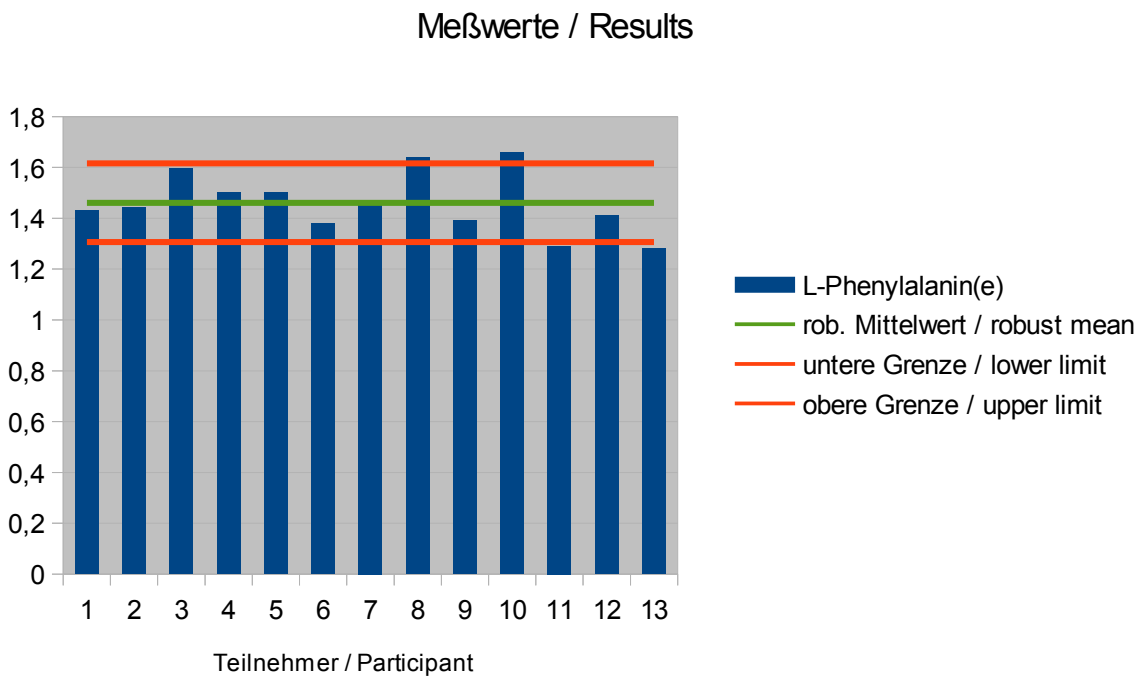


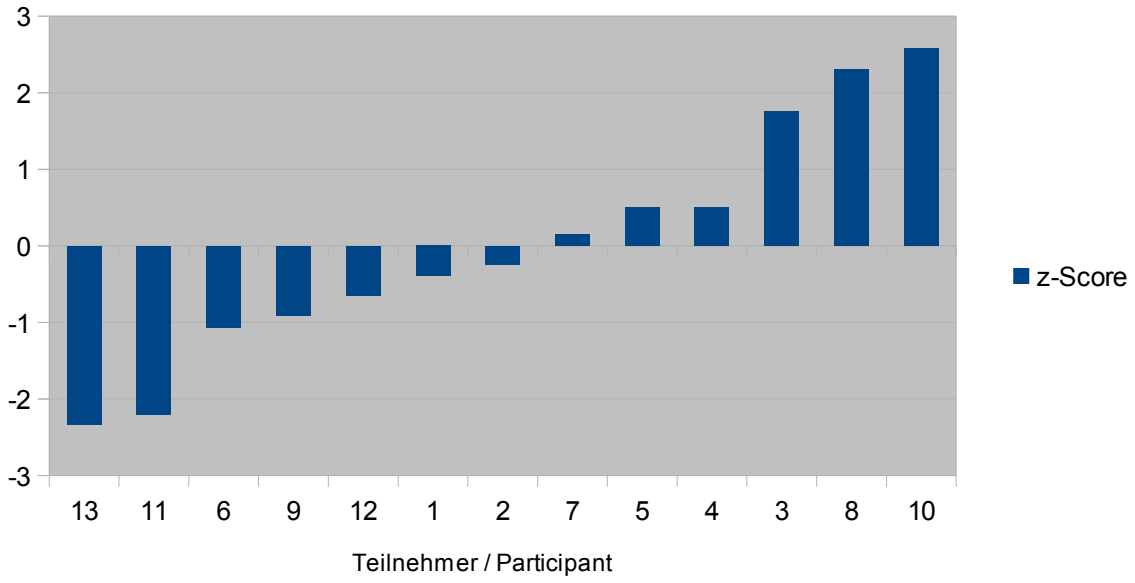


Auswerte nummer / Evaluation number	L-Methionin(e)	Abweichung / Deviation	z-Score	z-Score (Horwitz) zur info	Hinweis / Remark
1	1	0,07	1,2	1,8	
2	0,74	-0,19	-3,3	-5,0	
3	0,8	-0,13	-2,3	-3,5	
4	0,97	0,04	0,7	1,0	
5	0,98	0,05	0,9	1,3	
6	0,83	-0,1	-1,8	-2,7	
7					
8	1,13	0,2	3,5	5,3	
9	0,85	-0,08	-1,4	-2,1	
10	1,01	0,08	1,4	2,1	
11	0,99	0,06	1,0	1,6	
12	0,89	-0,04	-0,7	-1,1	
13	0,99	0,06	1,0	1,6	

4.12 Phenylalanin(e) in g/100 g

Statistic Data	
Number of the results	13
Number of outliers	0
Mean	1,46
Median	1,44
Robust mean (X)	1,46
Robust standard deviation (S^x)	0,135
Target standard deviation (sigma)	0,0775
Target standard deviation (Horwitz) for information	0,0552
Lower limit of target range	1,31
Upper limit of target range	1,62
Quotient S^x/σ	1,7
Standard uncertainty U^x	0,0469
Quotient U^x/σ	0,6
Results in target range	9
Percent in target range	69

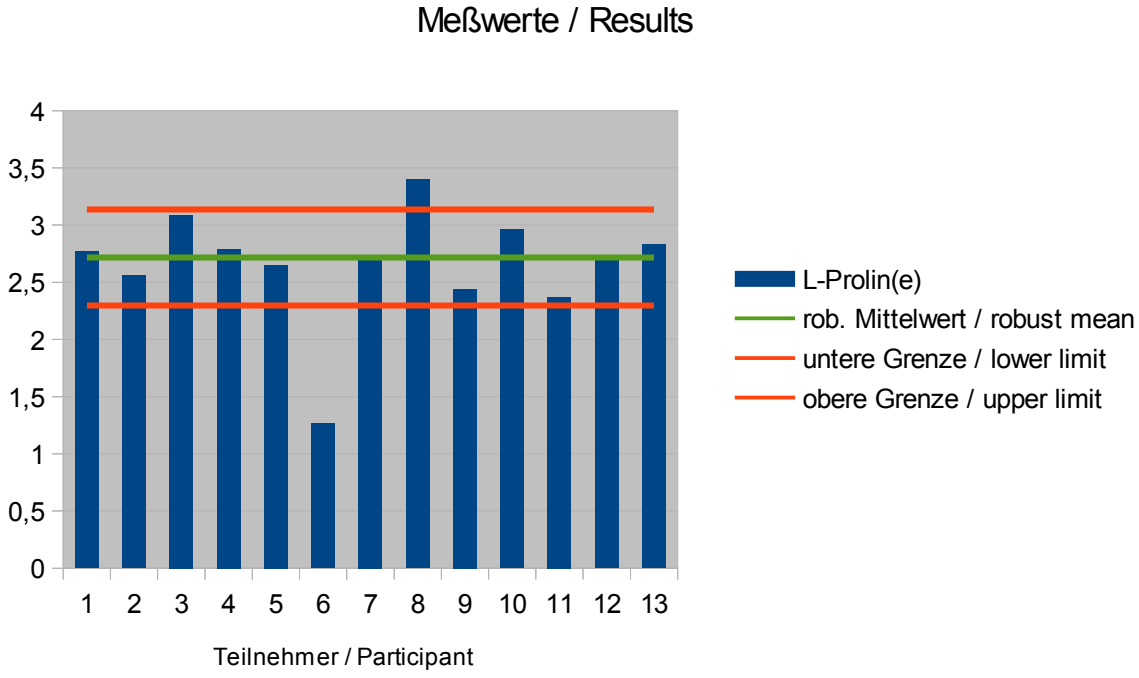


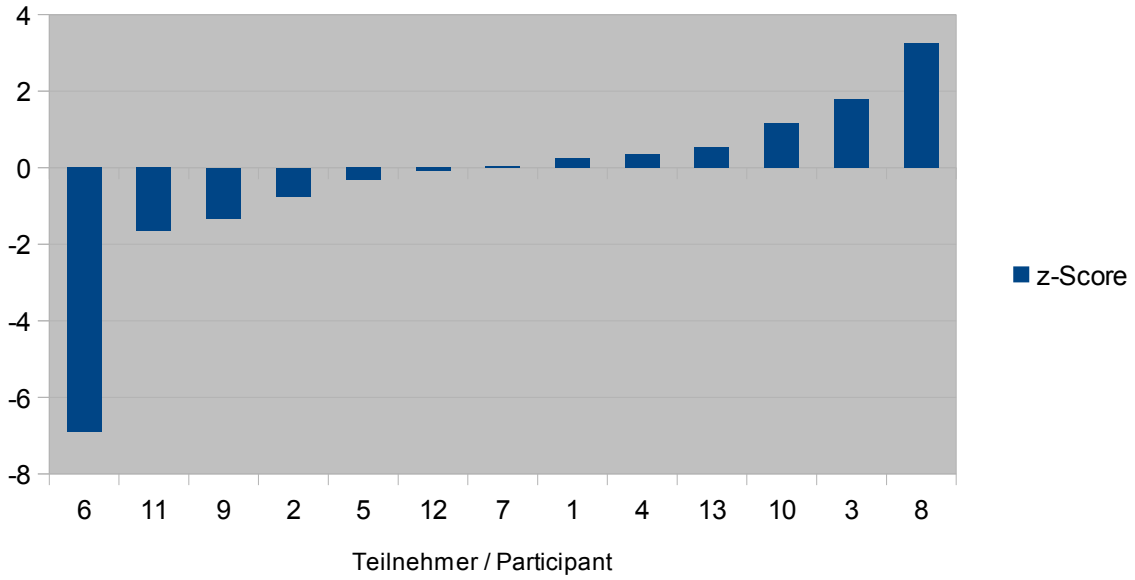


Auswerte nummer / Evaluation number	L-Phenylalanin(e)	Abweichung / Deviation	z-Score	z-Score (Horwitz) zur info	Hinweis / Remark
1	1,43	-0,03	-0,4	-0,6	
2	1,44	-0,02	-0,2	-0,3	
3	1,6	0,14	1,8	2,5	
4	1,5	0,04	0,5	0,7	
5	1,5	0,04	0,5	0,7	
6	1,38	-0,08	-1,1	-1,5	
7	1,47	0,01	0,1	0,2	
8	1,64	0,18	2,3	3,2	
9	1,39	-0,07	-0,9	-1,3	
10	1,66	0,2	2,6	3,6	
11	1,29	-0,17	-2,2	-3,1	
12	1,41	-0,05	-0,7	-0,9	
13	1,28	-0,18	-2,3	-3,3	

4.13 Prolin(e) in g/100 g

Statistic Data	
Number of the results	13
Number of outliers	1
Mean	2,66
Median	2,73
Robust mean (X)	2,72
Robust standard deviation (S^x)	0,304
Target standard deviation (sigma)	0,210
Target standard deviation (Horwitz) for information	0,0935
Lower limit of target range	2,3
Upper limit of target range	3,14
Quotient S^x/σ	1,4
Standard uncertainty U^x	0,105
Quotient U^x/σ	0,5
Results in target range	11
Percent in target range	85

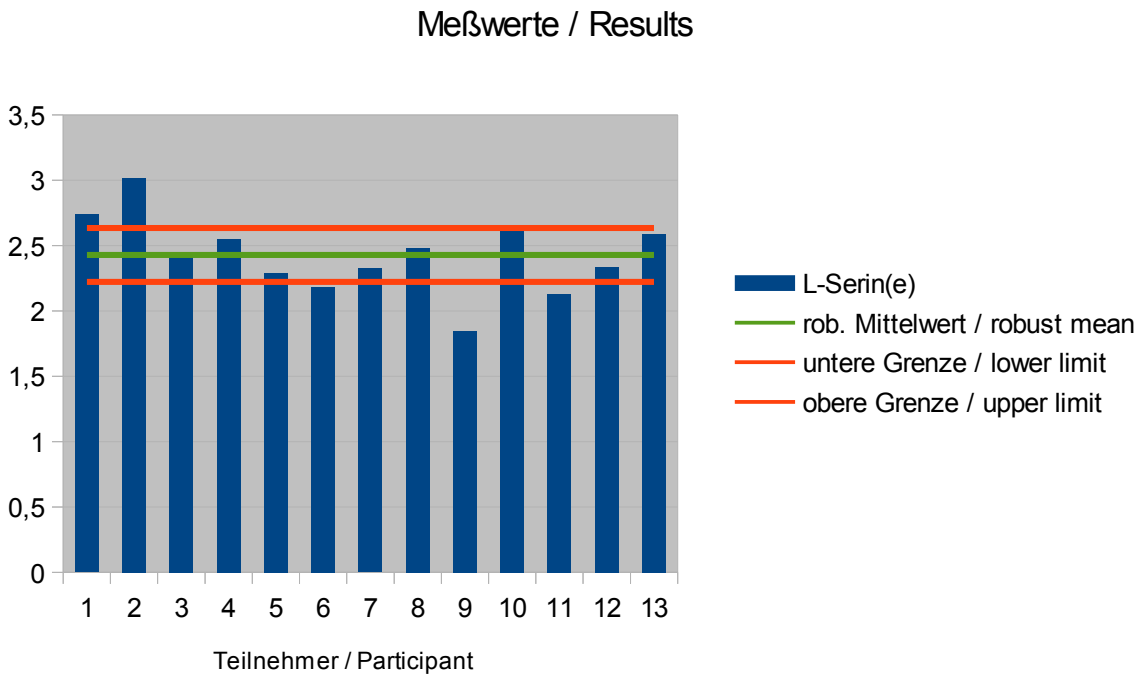


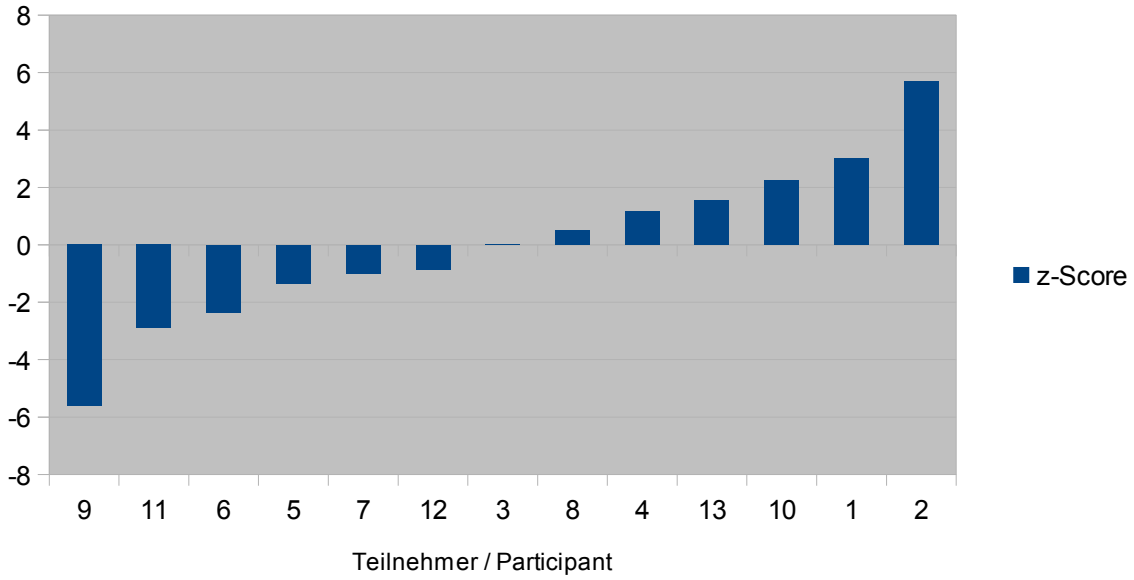


Auswerte nummer / Evaluation number	L-Prolin(e)	Abweichung / Deviation	z-Score	z-Score (Horwitz) zur info	Hinweis / Remark
1	2,77	0,05	0,3	0,6	
2	2,56	-0,16	-0,8	-1,7	
3	3,09	0,37	1,8	4,0	
4	2,79	0,07	0,3	0,8	
5	2,65	-0,07	-0,3	-0,7	
6	1,27	-1,45	-6,9	-15,5	Ausreisser / Outlier
7	2,73	0,01	0,1	0,1	
8	3,4	0,68	3,2	7,3	
9	2,44	-0,28	-1,3	-3,0	
10	2,96	0,24	1,2	2,6	
11	2,37	-0,35	-1,7	-3,7	
12	2,7	-0,02	-0,1	-0,2	
13	2,83	0,11	0,5	1,2	

4.14 Serin(e) in g/100 g

Statistic Data	
Number of the results	13
Number of outliers	2
Mean	2,43
Median	2,43
Robust mean (X)	2,43
Robust standard deviation (S^x)	0,280
Target standard deviation (sigma)	0,103
Target standard deviation (Horwitz) for information	0,085
Lower limit of target range	2,22
Upper limit of target range	2,64
Quotient S^x/σ	2,7
Standard uncertainty U^x	0,097
Quotient U^x/σ	0,9
Results in target range	7
Percent in target range	54

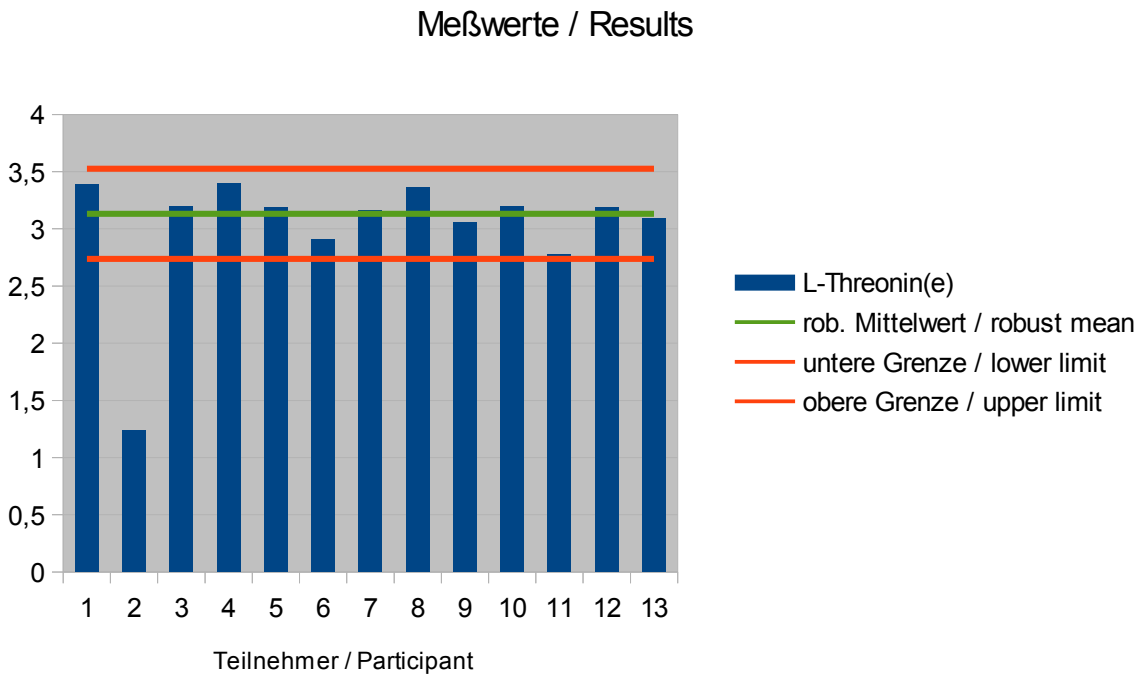


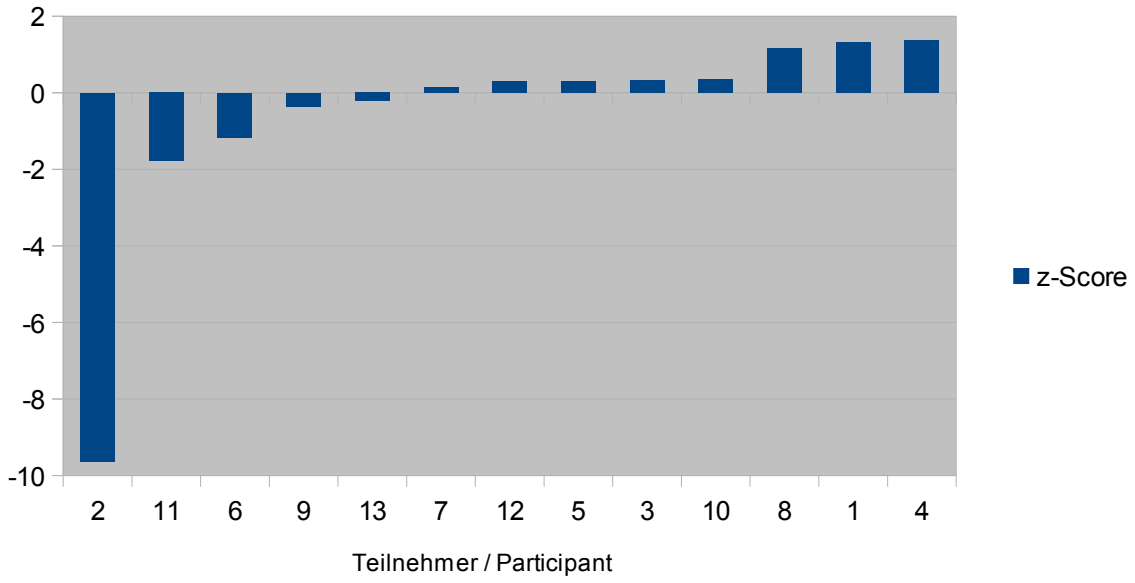


Auswerte nummer / Evaluation number	L-Serin(e)	Abweichung / Deviation	z-Score	z-Score (Horwitz) zur info	Hinweis / Remark
1	2,74	0,31	3,0	3,7	
2	3,02	0,59	5,7	6,9	Ausreisser / Outlier
3	2,43	0	0,0	0,0	
4	2,55	0,12	1,2	1,4	
5	2,29	-0,14	-1,4	-1,6	
6	2,19	-0,24	-2,3	-2,8	
7	2,33	-0,1	-1,0	-1,2	
8	2,48	0,05	0,5	0,6	
9	1,85	-0,58	-5,6	-6,8	Ausreisser / Outlier
10	2,66	0,23	2,2	2,7	
11	2,13	-0,3	-2,9	-3,5	
12	2,34	-0,09	-0,9	-1,1	
13	2,59	0,16	1,6	1,9	

4.15 Threonin(e) in g/100 g

Statistic Data	
Number of the results	13
Number of outliers	1
Mean	3,01
Median	3,19
Robust mean (X)	3,13
Robust standard deviation (S^x)	0,235
Target standard deviation (sigma)	0,197
Target standard deviation (Horwitz) for information	0,105
Lower limit of target range	2,74
Upper limit of target range	3,52
Quotient S^x/σ	1,2
Standard uncertainty U^x	0,082
Quotient U^x/σ	0,4
Results in target range	12
Percent in target range	92

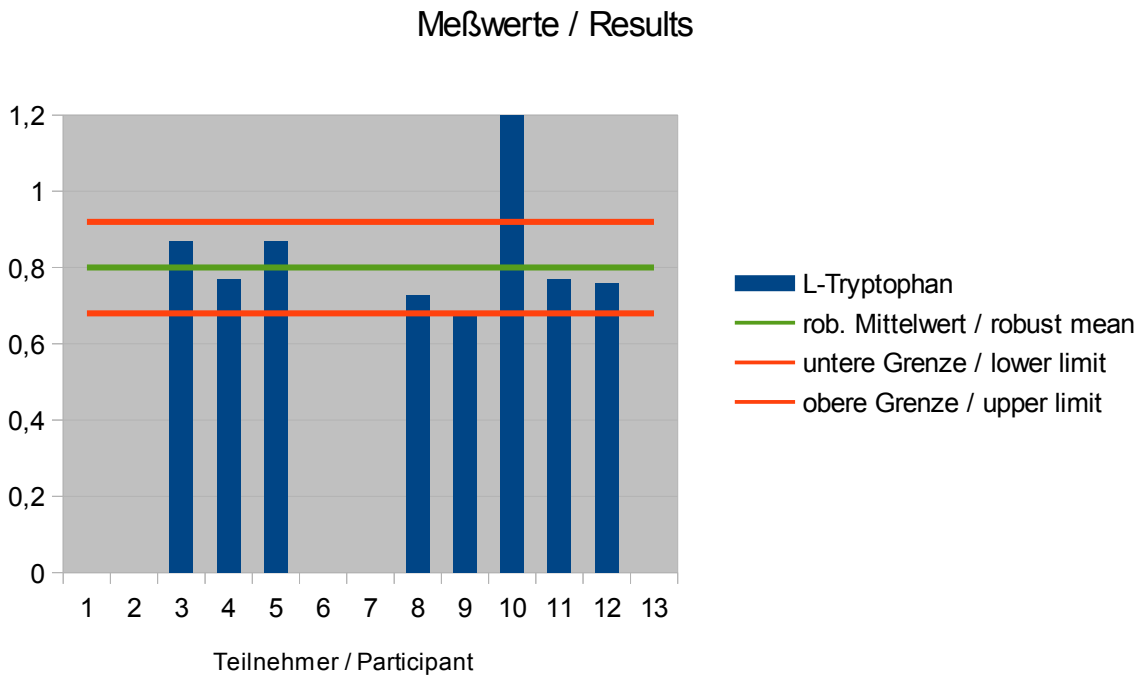


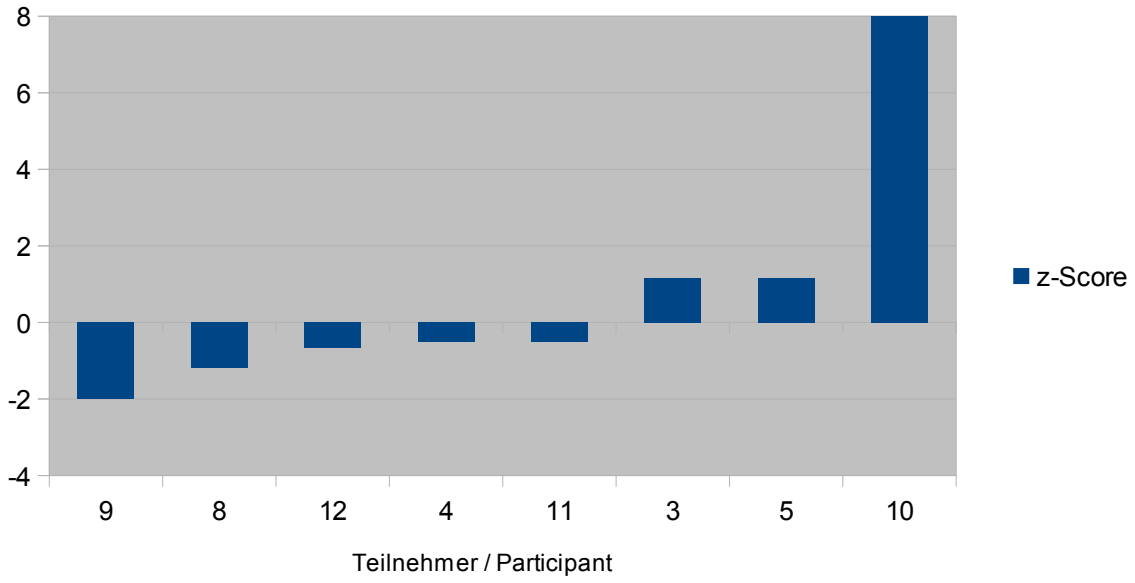


Auswerte nummer / Evaluation number	L-Threonin(e)	Abweichung / Deviation	z-Score	z-Score (Horwitz) zur info	Hinweis / Remark
1	3,39	0,26	1,3	2,5	
2	1,23	-1,9	-9,6	-18,0	Ausreisser / Outlier
3	3,2	0,06	0,3	0,6	
4	3,4	0,27	1,4	2,6	
5	3,19	0,06	0,3	0,6	
6	2,9	-0,23	-1,2	-2,2	
7	3,16	0,03	0,2	0,3	
8	3,36	0,23	1,2	2,2	
9	3,06	-0,07	-0,4	-0,7	
10	3,2	0,07	0,4	0,7	
11	2,78	-0,35	-1,8	-3,3	
12	3,19	0,06	0,3	0,6	
13	3,09	-0,04	-0,2	-0,4	

4.16 Tryptophan in g/100 g

Statistic Data	
Number of the results	8
Number of outliers	1
Mean	1,57
Median	0,77
Robust mean (X)	0,80
Robust standard deviation (S^x)	0,101
Target standard deviation (sigma)	0,060
Target standard deviation (Horwitz) for information	0,033
Lower limit of target range	0,680
Upper limit of target range	0,920
Quotient S^x/σ	1,7
Standard uncertainty U^x	0,045
Quotient U^x/σ	0,7
Results in target range	7
Percent in target range	88

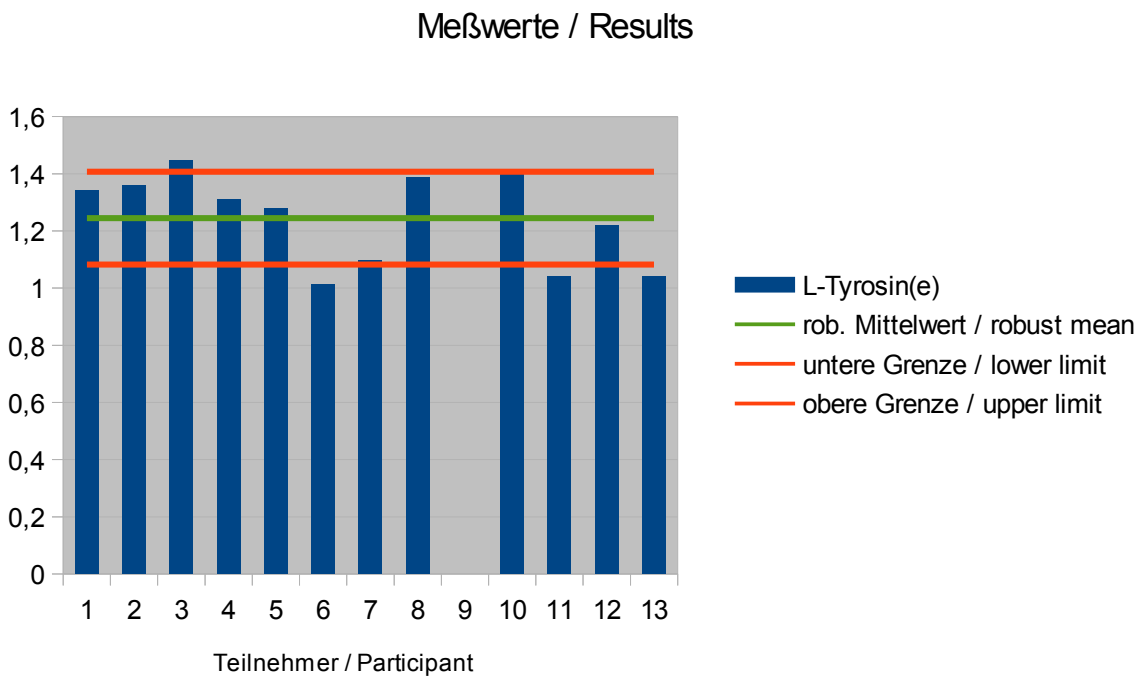


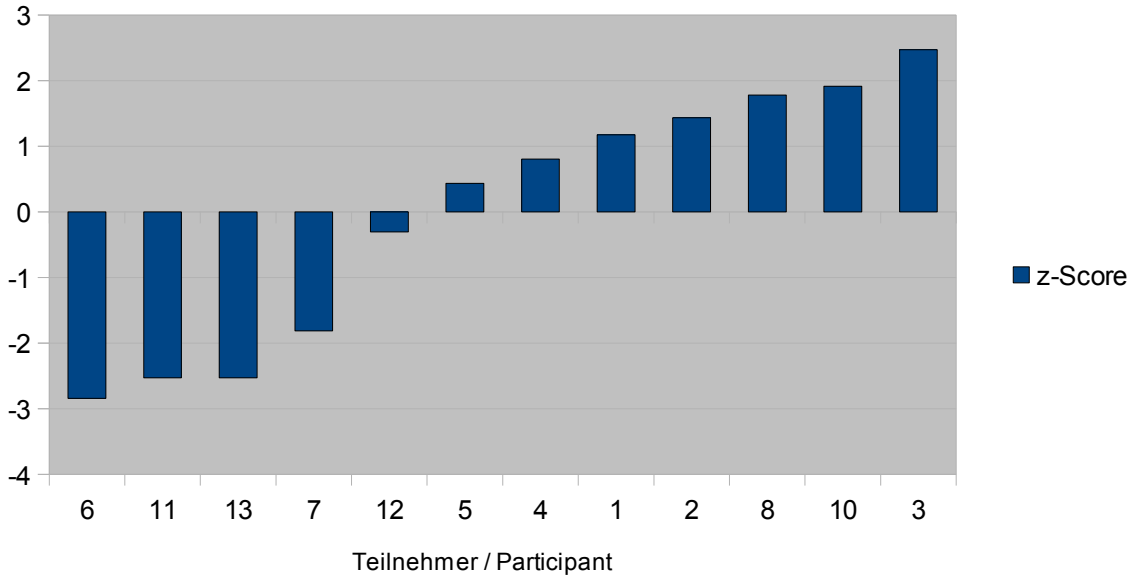


Auswerte nummer / Evaluation number	L-Tryptophan	Abweichung / Deviation	z-Score	z-Score (Horwitz) zur info	Hinweis / Remark
1					
2					
3	0,87	0,07	1,2	2,1	
4	0,77	-0,03	-0,5	-0,9	
5	0,87	0,07	1,2	2,1	
6					
7					
8	0,73	-0,07	-1,2	-2,2	
9	0,68	-0,12	-2,0	-3,6	
10	7,09	6,29	104,7	190,1	Ausreisser / Outlier
11	0,77	-0,03	-0,5	-0,9	
12	0,76	-0,04	-0,7	-1,2	
13					

4.17 Tyrosin in g/100 g

Statistic Data	
Number of the results	12
Number of outliers	0
Mean	1,24
Median	1,30
Robust mean (X)	1,24
Robust standard deviation (S^x)	0,178
Target standard deviation (sigma)	0,081
Target standard deviation (Horwitz) for information	0,048
Lower limit of target range	1,08
Upper limit of target range	1,41
Quotient S^x/σ	2,2
Standard uncertainty U^x	0,064
Quotient U^x/σ	0,8
Results in target range	8
Percent in target range	67

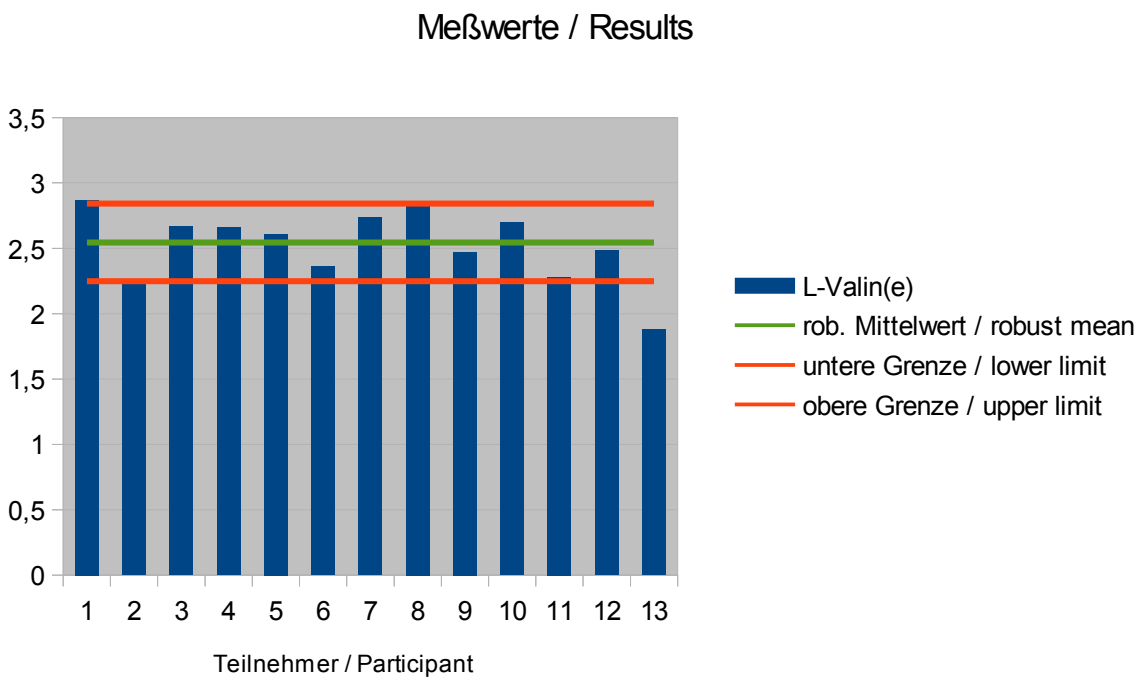


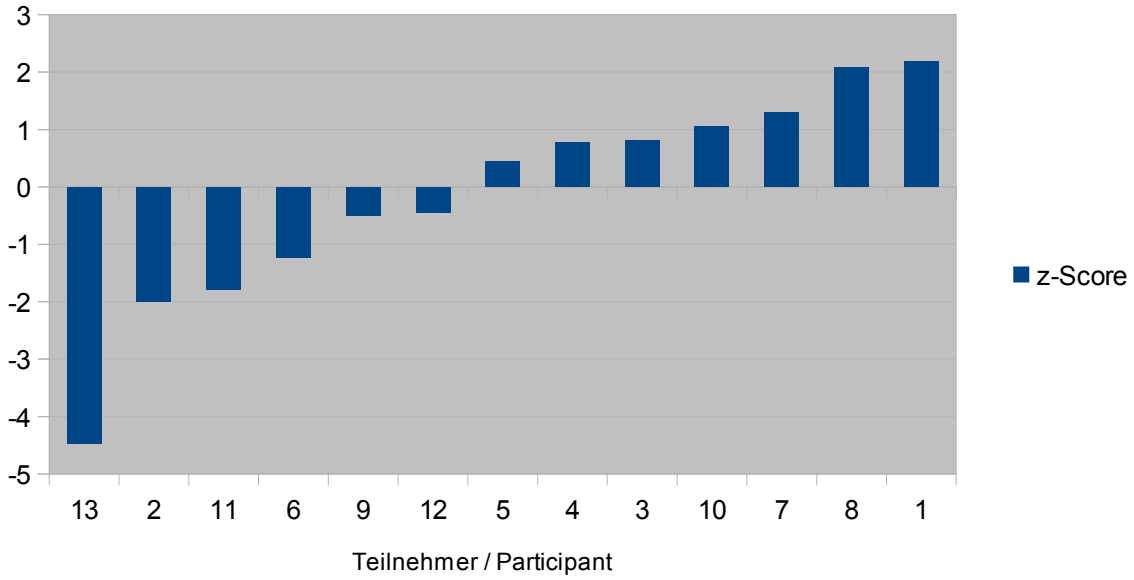


Auswerte nummer / Evaluation number	L-Tyrosin(e)	Abweichung / Deviation	z-Score	z-Score (Horwitz) zur info	Hinweis / Remark
1	1,34	0,1	1,2	2,0	
2	1,36	0,12	1,4	2,4	
3	1,45	0,2	2,5	4,2	
4	1,31	0,07	0,8	1,4	
5	1,28	0,04	0,4	0,7	
6	1,01	-0,23	-2,8	-4,8	
7	1,1	-0,15	-1,8	-3,0	
8	1,39	0,14	1,8	3,0	
9					
10	1,4	0,16	1,9	3,2	
11	1,04	-0,2	-2,5	-4,3	
12	1,22	-0,02	-0,3	-0,5	
13	1,04	-0,2	-2,5	-4,3	

4.18 Valin(e) in g/100 g

Statistic Data	
Number of the results	13
Number of outliers	1
Mean	2,52
Median	2,61
Robust mean (X)	2,54
Robust standard deviation (S^x)	0,265
Target standard deviation (sigma)	0,149
Target standard deviation (Horwitz) for information	0,088
Lower limit of target range	2,25
Upper limit of target range	2,84
Quotient S^x/σ	1,8
Standard uncertainty U^x	0,092
Quotient U^x/σ	0,6
Results in target range	10
Percent in target range	77





Auswerte nummer / Evaluation number	L-Valin(e)	Abweichung / Deviation	z-Score	z-Score (Horwitz) zur info	Hinweis / Remark
1	2,87	0,33	2,2	3,7	
2	2,25	-0,3	-2,0	-3,4	
3	2,67	0,12	0,8	1,4	
4	2,66	0,12	0,8	1,3	
5	2,61	0,07	0,4	0,7	
6	2,36	-0,18	-1,2	-2,1	
7	2,74	0,19	1,3	2,2	
8	2,85	0,31	2,1	3,5	
9	2,47	-0,07	-0,5	-0,8	
10	2,7	0,16	1,0	1,8	
11	2,28	-0,26	-1,8	-3,0	
12	2,48	-0,06	-0,4	-0,7	
13	1,88	-0,66	-4,5	-7,5	Ausreisser / Outlier

5 Documentation

5.1 *Primary data*

5.1.1 *Amino acids in g/100g*

Teilnehmer / Participant	DLA-Nr Probe A / Sample A	DLA-Nr Probe B / Sample B	Alanin(e)	Ergebnis / Result A	Ergebnis / Result B	Arginin(e)	Ergebnis / Result A	Ergebnis / Result B
1	33	51	2,41	2,39	2,42	1,17	1,17	1,17
2	17	50	2,233	2,277	2,188	0,996	0,949	1,042
3	20	40	2,359	2,356	2,361	1,219	1,239	1,199
4	5	30	2,43	2,44	2,42	1,18	1,18	1,18
5	6	38	2,25	2,22	2,27	1,17	1,15	1,19
6 *	12	37	21199	21150	21247	10474	10312	10635
7	29	47	2,309	2,299	2,32	1,165	1,148	1,182
8	13	25	2,418	2,43	2,406	1,266	1,273	1,258
9	10	39	2	2,01	1,99	1,03	1,04	1,03
10			2,49	2,48	2,50	1,23	1,25	1,20
11	48	11	1,95	1,95	1,94	1,04	1,02	1,05
12	8	36	2,27	2,22	2,32	1,11	0,94	1,28
13	18	54	2,21	2,209	2,209			

* Einheit / Unit: mg/kg

Teilnehmer / Participant	Asparaginsäure / Aspartic Acid	Ergebnis / Result A	Ergebnis / Result B	Cystein(e)	Ergebnis / Result A	Ergebnis / Result B	Cystin(e)	Ergebnis / Result A	Ergebnis / Result B
1	5,24	5,20	5,27	N/A	N/A	N/A	1,11	1,11	1,1
2	5,221	4,893	5,549	0,173	0,183	0,164	0,307	0,304	0,311
3	5,05	5,102	4,998	0,828	0,856	0,799			
4	5,29	5,27	5,30	-	-	-	1,17	1,16	1,17
5	5,00	4,96	5,03	1,10	1,08	1,12		s. Cystein	s. Cystein
6 *	46075	45918	46232						
7	5,037	5,035	5,036						
8	5,499	5,541	5,456				0,993	0,993	0,993
9	4,43	4,42	4,44				1,09	1,03	1,15
10	5,38	5,39	5,36	-			0,32	0,33	0,32
11	4,53	4,51	4,54	1	0,98	1,02			
12	4,9	4,82	4,98	1,02	1,04	1,01			
13	5,26	4,924	5,596				0,9	0,884	0,913

* Einheit / Unit: mg/kg

Teilnehmer / Participant	Glutaminsäure / Glutamic Acid	Ergebnis / Result A	Ergebnis / Result B	Glycin(e)	Ergebnis / Result A	Ergebnis / Result B	Histidin(e)	Ergebnis / Result A	Ergebnis / Result B
1	12,2	12,2	12,2	0,91	0,91	0,91	0,82	0,82	0,82
2	11,408	10,655	12,160	0,823	0,824	0,821	0,744	0,720	0,768
3	11,944	12,055	11,833	1,022	1,002	1,042	0,838	0,864	0,811
4	13,17	13,14	13,20	0,91	0,91	0,90	0,88	0,88	0,88
5	12,20	12,08	12,32	0,87	0,86	0,88	0,89	0,89	0,89
6 *	103238	102100	104375	7765	7735	7795	7287	7272	7302
7	12,57	12,54	12,59	0,869	0,863	0,874	1,005	1,006	1,004
8	13,535	13,694	13,376	0,926	0,929	0,922	1,082	1,108	1,056
9	11,21	11,31	11,12	0,76	0,77	0,75	0,79	0,79	0,79
10	13,22	13,42	13,01	0,95	0,96	0,93	0,87	0,86	0,88
11	11,13	11,02	11,23	0,86	0,86	0,86	0,9	0,87	0,94
12	12,61	12,61	12,61	0,86	0,83	0,89	0,84	0,85	0,84
13	13,19	12,778	13,569	0,87	0,869	0,873	0,78	0,77	0,779

* Einheit / Unit: mg/kg

Teilnehmer / Participant	Isoleucin(e)	Ergebnis / Result A	Ergebnis / Result B	Leucin(e)	Ergebnis / Result A	Ergebnis / Result B	Lysin(e)	Ergebnis / Result A	Ergebnis / Result B
1	3,44	3,51	3,37	4,81	4,81	4,81	4,48	4,47	4,48
2	2,290	2,189	2,391	4,571	4,570	4,572	7,530	6,840	8,219
3	2,964	2,972	2,957	4,934	4,972	4,896	6,858	6,843	6,874
4	2,84	2,82	2,85	5,02	5,00	5,03	4,34	4,35	4,33
5	2,82	2,78	2,86	4,72	4,64	4,79	4,14	4,1	4,18
6*	25010	24777	25243	43166	42647	43685	38068	37558	38577
7	2,918	2,915	2,92	4,954	4,948	4,96	4,205	4,202	4,208
8	3,016	3,049	2,982	5,403	5,458	5,348	4,495	4,493	4,496
9	2,62	2,66	2,57	4,16	4,19	4,13	3,59	3,59	3,59
10	2,94	2,88	3,00	5,56	5,44	5,67	4,25	4,17	4,32
11	2,38	2,37	2,38	3,87	3,86	3,88	3,36	3,35	3,37
12	2,68	2,54	2,82	4,68	4,55	4,81	3,93	4,26	3,59
13	2,42	2,358	2,471	4,32	4,373	4,31	3,13	3,291	2,96

* Einheit / Unit: mg/kg

Teilnehmer / Participant	Methionin(e)	Ergebnis / Result A	Ergebnis / Result B	Phenyl alanin(e)	Ergebnis / Result A	Ergebnis / Result B	Prolin(e)	Ergebnis / Result A	Ergebnis / Result B
1	1	1,00	0,99	1,43	1,44	1,42	2,77	2,74	2,79
2	0,742	0,767	0,717	1,442	1,407	1,476	2,559	2,588	2,530
3	0,8	0,806	0,794	1,597	1,609	1,585	3,09	3,183	2,998
4	0,97	0,96	0,97	1,50	1,50	1,49	2,79	2,81	2,77
5	0,98	0,96	0,99	1,50	1,48	1,52	2,65	2,6	2,7
6 *	8285	8255	8315	13783	13748	13817	12669	12663	12674
7				1,472	1,473	1,471	2,728	2,683	2,772
8	1,131	1,14	1,122	1,639	1,648	1,63	3,397	3,247	3,547
9	0,85	0,85	0,85	1,39	1,39	1,4	2,44	2,42	2,45
10	1,01	0,98	1,04	1,66	1,63	1,69	2,96	2,94	2,98
11	0,99	0,96	1,02	1,29	1,3	1,28	2,37	2,42	2,32
12	0,89	0,94	0,84	1,41	1,36	1,45	2,7	2,63	2,77
13	0,99	0,954	1,029	1,28	1,2963	1,262	2,83	2,842	2,826

* Einheit / Unit: mg/kg

Teilnehmer / Participant	Serin(e)	Ergebnis / Result A	Ergebnis / Result B	Threonin(e)	Ergebnis / Result A	Ergebnis / Result B	Tryptophan	Ergebnis / Result A	Ergebnis / Result B
1	2,74	2,72	2,75	3,39	3,39	3,38	Nicht bestimmt / Not tested	Nicht bestimmt / Not tested	Nicht bestimmt / Not tested
2	3,016	2,936	3,095	1,234	1,212	1,256	ND	ND	ND
3	2,43	2,453	2,409	3,195	3,248	3,142	0,87	0,85	0,887
4	2,55	2,55	2,55	3,40	3,41	3,39	0,77	0,77	0,77
5	2,29	2,26	2,32	3,19	3,15	3,22	0,87	0,88	0,868
6*	21872	21742	22002	29018	28787	29248			
7	2,326	2,306	2,346	3,161	3,164	3,158			
8	2,481	2,49	2,471	3,36	3,378	3,341	0,728	0,728	0,727
9	1,85	1,87	1,83	3,06	3,11	3,03	0,68	0,68	0,69
10	2,66	2,65	2,66	3,20	3,15	3,24	7,09	7,21	6,97
11	2,13	2,13	2,13	2,78	2,77	2,78	0,77	0,78	0,77
12	2,34	2,32	2,35	3,19	3,11	3,27	0,76	0,75	0,77
13	2,59	2,496	2,675	3,09	3,004	3,184			

* Einheit / Unit: mg/kg

Teilnehmer / Participant	Tyrosin(e)	Ergebnis / Result A	Ergebnis / Result B	Valin(e)	Ergebnis / Result A	Ergebnis / Result B	Taurin(e)	Ergebnis / Result A	Ergebnis / Result B
1	1,34	1,35	1,33	2,87	2,92	2,82			
2	1,361	1,360	1,361	2,247	2,050	2,443			
3	1,445	1,47	1,42	2,666	2,662	2,671			
4	1,31	1,31	1,30	2,66	2,65	2,67			
5	1,28	1,27	1,28	2,61	2,58	2,64			
6*	10145	9967	10322	23626	23298	23953	2598	2643	2552
7	1,098	1,073	1,123	2,737	2,738	2,737			
8	1,389	1,401	1,376	2,854	2,888	2,819			
9				2,47	2,47	2,46			
10	1,40	1,40	1,40	2,70	2,65	2,74			
11	1,04	1,03	1,04	2,28	2,28	2,28			
12	1,22	1,25	1,2	2,48	2,47	2,48	0,12	0,13	0,11
13	1,04	1,059	1,017	1,88	1,874	1,889			

* Einheit / Unit: mg/kg

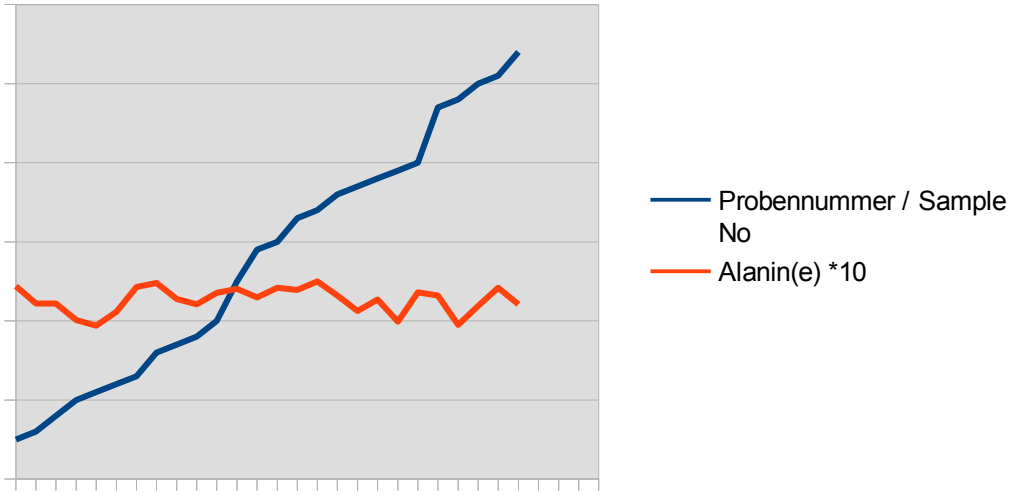
5.2 Homogeneity

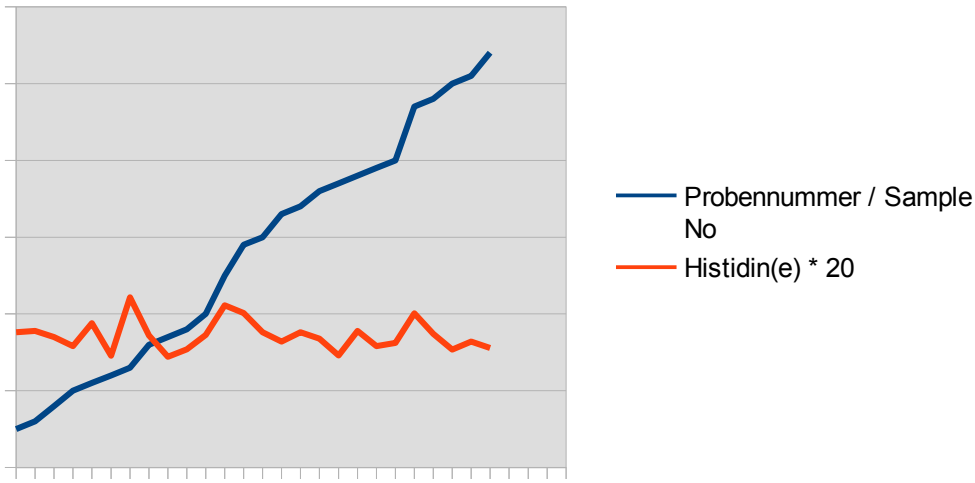
5.2.1 Repeatability standard deviation of duplicate tests of the participants

The repeatability standard deviation was calculated with the data documented in 5.1. It is 1,9% of X (Alanine) in comparison to 2,3 %, which is documented in ASU L 49.07-2.

And it is 3,7 % of X (Histidine) in comparison to 2,7%, which is documented in ASU L 49.07-2.

5.2.2 Comparison of sample number / test result





5.3 Analytical methods

Participant	Method	Homogenei zation	Digestion	Weight	Derivatization	Reference material	Calibration	Method is accredited	Remarks
								yes / no	
1	Amino Acid Analysis	yes	yes	0.5g	yes	yes	yes	yes	N/A
2	yes	yes	yes	0.5 g	yes	no	yes	no	
3	ASU L 49.07-2	yes	110°C dry box 20h	0,5g	Fmoc/OPA	Milkpowder	external with Istd	yes	
4	Amino acids after hydrolysis(analogue VO (EG) 152/2009)		HCl (6 mol/L)	app. 0,3 g		Feed (internal RM)	Int. Std., 1-point-calibration	yes	
5	VO(EG)152/2009, III F		Hydrolysis/oxidation		Ninhydrine			yes	
6	Ion chromatography with post column derivatization		acid hydrolysis		Ninhydrine		intern	yes	
7	Amino acids after acidic hydrolysis with amino acids analyser (post column derivatization)	mortar	acid hydrolysis with 6 M HCl, 24 h at 110°C	200 - 500 mg	Ninhydrine	Milkprotein	External standard, linear regression	yes	
8	§64 LFGB L 49.07-2		Hydrolysis 6 M HCL	0,5 g	Ninhydrine	321 QB Lwk NRW	ext. Standard	yes	
9	Determination with amino acids-analyser after acidic hydrolysis	Labora-tory mill	6 m HCl	20 mg	Ninhydrine	no	internal standard	yes	
10	VOL.72 No.6 1989								
11	HPLC (Fluorescence detection)		Acid hydrolysis, oxidation with performic acid, basic hydrolysis	40 mg	OPA, NBDCl	LVU 2013	ISTD, ESTD	yes	
12	Determination of total amino acids in food with amino acids analyser with ninhydrine analogue § 64 LFGB L49.07-2	none	acid hydrolysis	500 mg	Post column derivatization with ninhydrine	intern	Internal standard multipoint-calibration	yes	
13	Phenomenex EZ: faast		HCl 6 mol/l 16 h at 110°C	1g/100ml	Phenomenex EZ: faast	DLA material	4 point	yes	

6 Index of participant laboratories

Teilnehmer / Participant	Ort / Location	Land / Country
		ENGLAND
		GERMANY
		THAILAND
		GERMANY
		GERMANY
		GERMANY
		TAIWAN
		GERMANY
		GERMANY
		GERMANY
		GERMANY
		GERMANY
		GERMANY
		GERMANY

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

7 Index of literature

1. DIN EN ISO/IEC 17043:2010; Konformitätsbewertung – Allgemeine Anforderungen an Eignungsprüfungen / Conformity assessment – General requirements for proficiency testing
2. Verordnung / Regulation 882/2004/EU; Verordnung über amtliche Kontrollen / Regulation on official controls
3. 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
4. Richtlinie / Directive 1993/99/EU; über zusätzliche Maßnahmen im Bereich der amtlichen Lebensmittelüberwachung / on additional measures concerning the official control of foodstuffs
5. ASU §64 LFGB : Planung und statistische Auswertung von Ringversuchen zur Methodenvalidierung
6. DIN ISO 13528:2009; Statistische Verfahren für Eignungsprüfungen durch Ringversuche
7. The International Harmonised Protocol for the Proficiency Testing of Ananlytical Laboratories ; J.AOAC Int., 76(4), 926 – 940 (1993)
8. The International Harmonised Protocol for the Proficiency Testing of Ananlytical Chemistry Laboratories ; Pure Appl Chem, 78, 145 – 196 (2006)
9. Evaluation of analytical methods used for regulation of food and drugs;W. Horwitz; Analytical Chemistry, 54, 67-76 (1982)
10. A Horwitz-like funktion describes precision in proficiency test; M. Thompson, P.J. Lowthian; Analyst, 120, 271-272 (1995)
11. 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)
12. Protocol for the design, conduct and interpretation of method performance studies; W. Horwitz; Pure & Applied Chemistry, 67, 331-343 (1995)
13. ASU §64 LFGB : L49.07-1; Bestimmung der Aminosäuren in Aminosäurengemischen.
14. ASU §64 LFGB : L49.07-2; Bestimmung der Aminosäuren in diätetischen Lebensmitteln auf Basis von Proteinhydrolysaten
15. ASU §64 LFGB : L49.07-3; Bestimmung des Tryptophangehaltes in diätetischen Lebensmitteln auf Basis von Proteinhydrolysaten
16. VERORDNUNG (EG) Nr. 152/2009 zur Festlegung der Probenahmeverfahren und Analysemethoden für die amtliche Untersuchung von Futtermitteln.

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