DLA
Dienstleistung
Lebensmittel
Analytik GbR

Evaluation-Report
proficiency test

DLA 08/2014

Lactose and Fructose in "lactosefree" food

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Contents

1 Introduction....3
2 Evaluation....3
2.1 Assigned value....3
2.2 Standard deviation....3
2.3 Outliers....3
2.4 Target standard deviation....4
2.4.1 General model (Horwitz / Thompson)....4
2.4.2 Precision experiment....4
2.5 z-Score....5
2.6 z'-Score....5
2.7 Quotient Sx/σ....5
2.8 Standard uncertainty....6
3 Realisation....7
3.1 Test material....7
3.1.1 Homogeneity....8
3.2 Tests....8
3.3 Results and statistic evaluation....8
4 Results....9
4.1 Fructose Sample A in mg/kg....10
4.2 Fructose Sample B in mg/kg....12
4.3 Fructose Sample C in g/100 g....14
4.4 Galactose Sample A in mg/kg....16
4.5 Galactose Sample B in mg/kg....18
4.6 Galactose Sample C in g/100 g....20
4.7 Lactose Sample A in mg/kg....22
4.8 Lactose Sample B in mg/kg....24
4.9 Lactose Sample C in g/100 g....26
5 Documentation....28
5.1 Primary data....28
5.1.1 Fructose....28
Sample A....28
Sample B....29
Sample C....30
5.1.2 Galactose....31
Sample A....31
Sample B....32
Sample C....33
5.1.3 Lactose....34
Sample A....34
Sample B....35
Sample C....36
5.2 Homogeneity....37
5.3 Analytical methods....38
5.3.1 Fructose....38
5.3.2 Lactose and Galactose....39
6 Index of participant laboratories....40
7 Index of literature....41

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 fullfilled 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 materials were two different types of pastry, both stated as „lactosefree" in the declaration. Sample A was toast made on basis of rice. Sample B was crispbread made on basis of corn and rice, to which was added sample C. Sample C was a mixture of cornstarch, infantfood (59% lactose), galactose and fructose.
The compositions of the samples are given in table 1.
App. 1 kg sample A was ground, mixed, heated to 200°C for 10 Minutes, mixed again, packed lightproof in portions to app. 25 g.
The samples C and B were mixed, ground, sieved, mixed again, packed lightproof in portions to approximately 25 g and tested for homogeneity. The portions were numbered chronologically.

Table 1: Composition of samples

Ingredient	**Sample A**	**Sample B**	**Sample C**
Toast with rice Ingredients: Water, Rice 39%, Sorghum 8%, Corn, Lupine 7%, Rice flour 5%, Guar, Seasalt, Yeast. According to declaration 38% Carbohydrates, of which 1 % sugars	100g/100g	-	
Crispbread Ingredients: Corn flour, rice flour, linseed 7%, sesame 7%, lupine flour, corn starch, potato starch, sunflower seed 4%, veg. oil, salt, lecithine, carrot juice, lemon juice, buckweat flour, sugar, yeast, guar, seasalt, baking agent. According to declaration 49,1% Carbohydrates, of which 0,5 % sugars	-	750 g	
Sample C	-	34 g	
Corn starch	-	-	500 g
Infant food (59% lactose)	-	-	80,6 g
Galactose monohydrate	-	-	10,4 g
Fructose	-	-	74,0 g

3.1.1 Homogeneity

Homogeneity was tested by HPLC-analysis as multiple determination of the fructose content of sample B and sample C.
With a standard deviation of the results of rel. 4,5% in sample B and rel. 2,6 % in sample C the samples were considered homogenous.
Results of the tests for homogeneity are given in the documentation.

3.2 Tests

The test samples were sent to every participating laboratory in the 7th week of 2014. The test method was optional. The tests should be finished at 28th march 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 if at least 8 results were submitted. Only lactose in sample A could not be evaluated, the results are shown.
The target standard deviation for fructose, samples A, B and C, and for galactose, sample C was calculated according to Horwitz.
Results for galactose and lactose often showed a higher variation. Hence the target range for galactose, sample A and B, and for lactose, samples B and C, was calculated according to 2.6.
Queried and documented were limits of detection and limits of quantification and the testing method applied.
The submitted results showed no bimodal distribution.
15 out of 17 participants submitted results.

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:

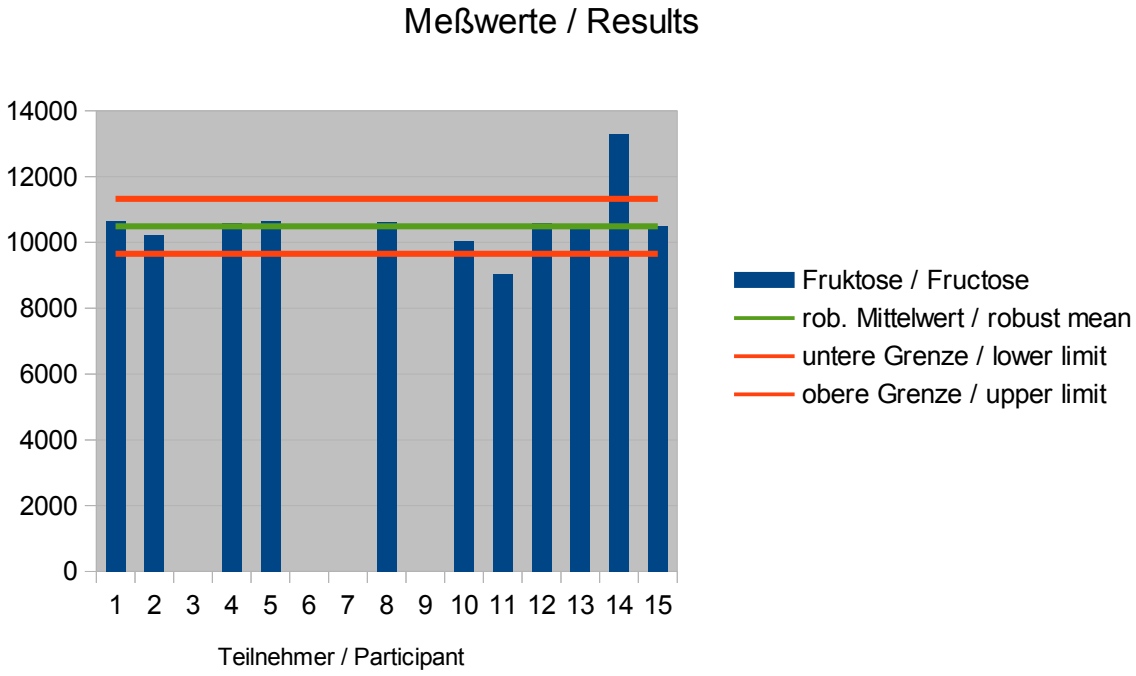
number of the results
number of outliers
mean
median
robust mean (X)
robust standard deviation(S^x)
target standard deviation (σ) or (σ´)
target standard deviation (σ, for information)
lower limit of target range (X – 2σ) or (X – 2σ´)
upper limit of target range (X + 2σ) or (X + 2σ´)
quotient S^x/σ
standard uncertainty u_x
quotient u_X/σ
results in target range.

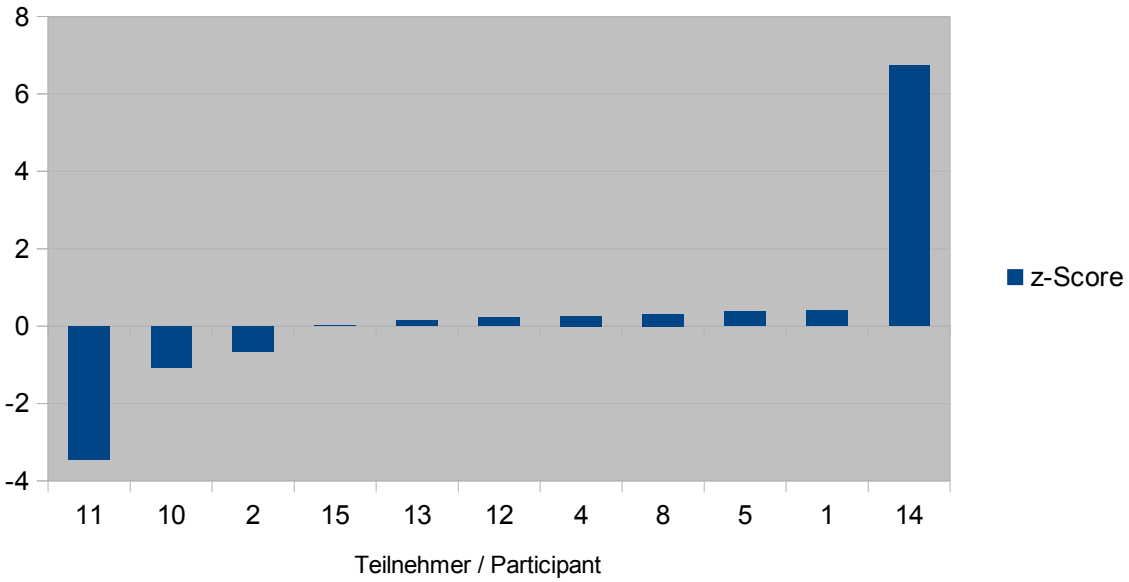
In the lower table -laboratories- the individual results of the participating laboratory are listed:

evaluation number	test result	deviation from assigned value	Z-Score (σ)	remarks

4.1 *Fructose Sample A in mg/kg*

Statistic Data	
number of the results	11
number of outliers	1
mean	10616
median	10583
robust mean (X)	10491
robust standard deviation (S^x)	336
target standard deviation (σ)	417
lower limit of target range	9658
upper limit of target range	11325
quotient S^x/σ	0,8
standard uncertainty U^x	127
quotient U^x/σ	0,3
results in target range	9
percent in target range	82

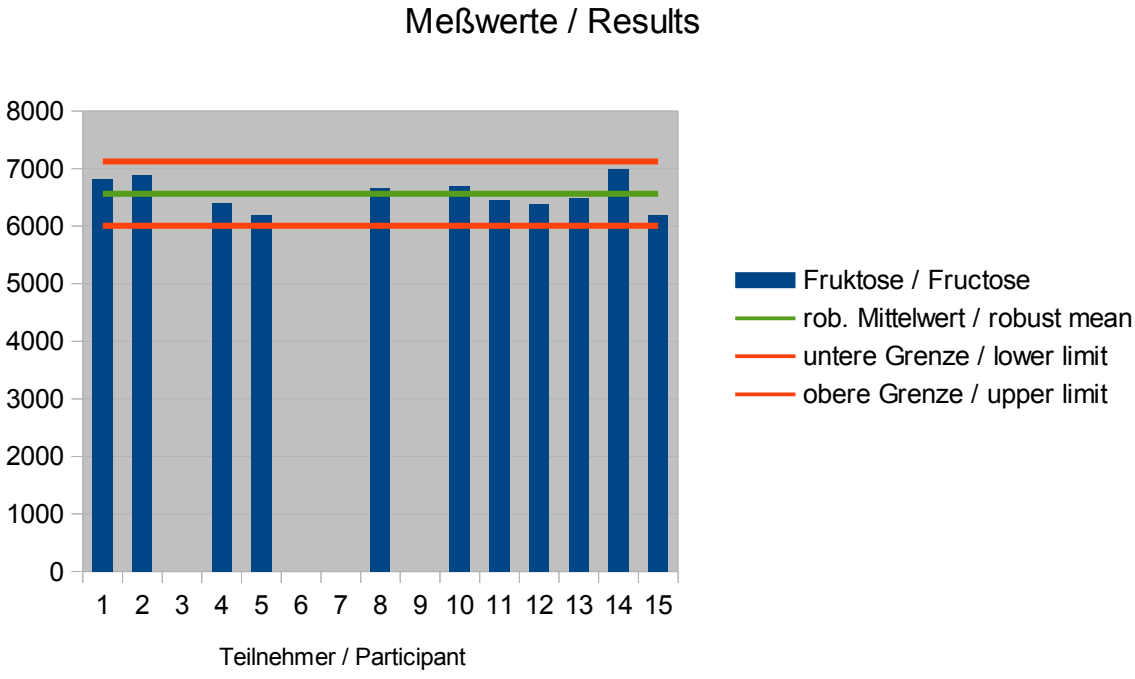


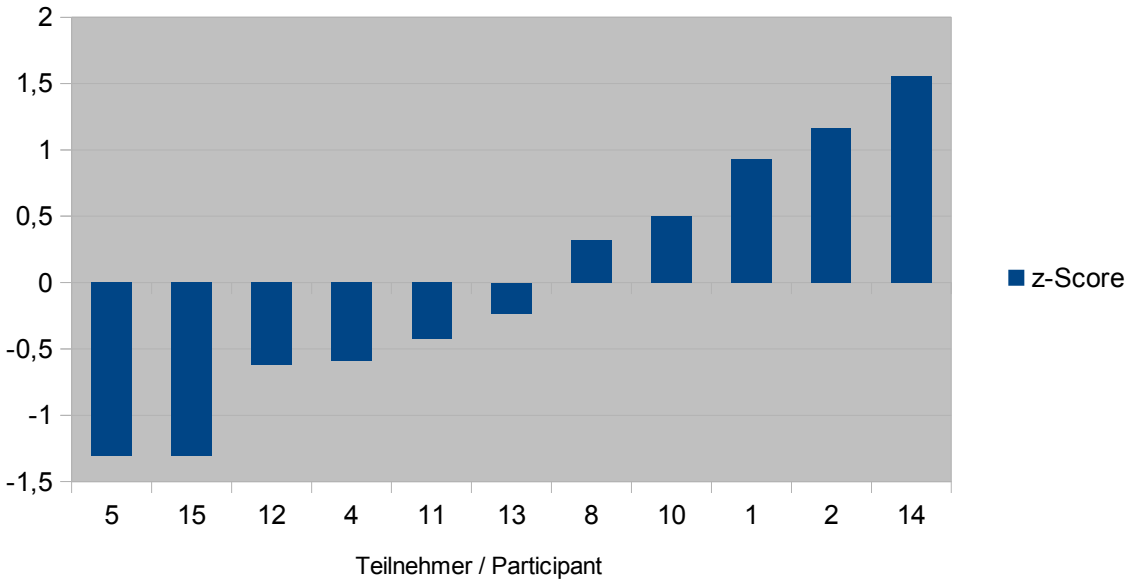


Auswerte nummer / Evaluation number	Fruktose / Fructose	Abweichung / Deviation	z-Score	Hinweis / Remark
1	10659	168	0,4	
2	10220	-271	-0,7	
3				
4	10600	109	0,3	
5	10650	159	0,4	
6				
7				
8	10620	129	0,3	
9				
10	10040	-451	-1,1	
11	9056	-1435	-3,4	
12	10583	92	0,2	
13	10550	59	0,1	
14	13300	2809	6,7	Ausreisser / Outlier
15	10500	9	0,0	

4.2 *Fructose Sample B in mg/kg*

Statistic Data	
number of the results	11
number of outliers	0
mean	6565
median	6500
robust mean (X)	6565
robust standard deviation (S^x)	307
target standard deviation (σ)	280
lower limit of target range	6005
upper limit of target range	7124
quotient S^x/σ	1,1
standard uncertainty U^x	116
quotient U^x/σ	0,4
results in target range	11
percent in target range	100

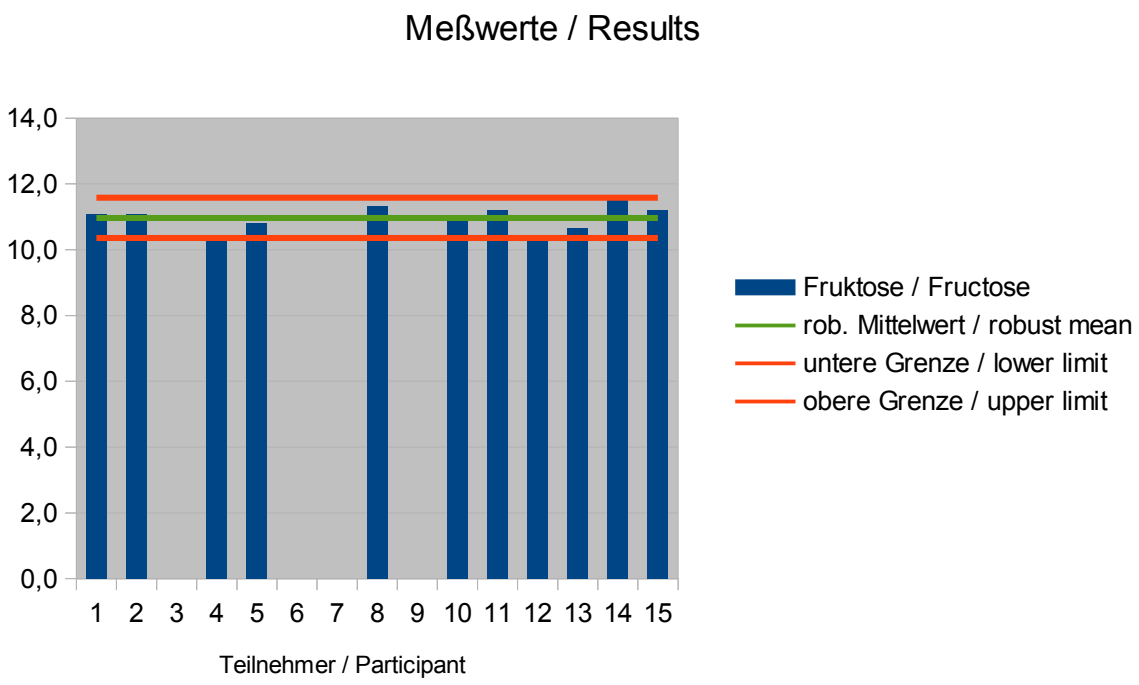


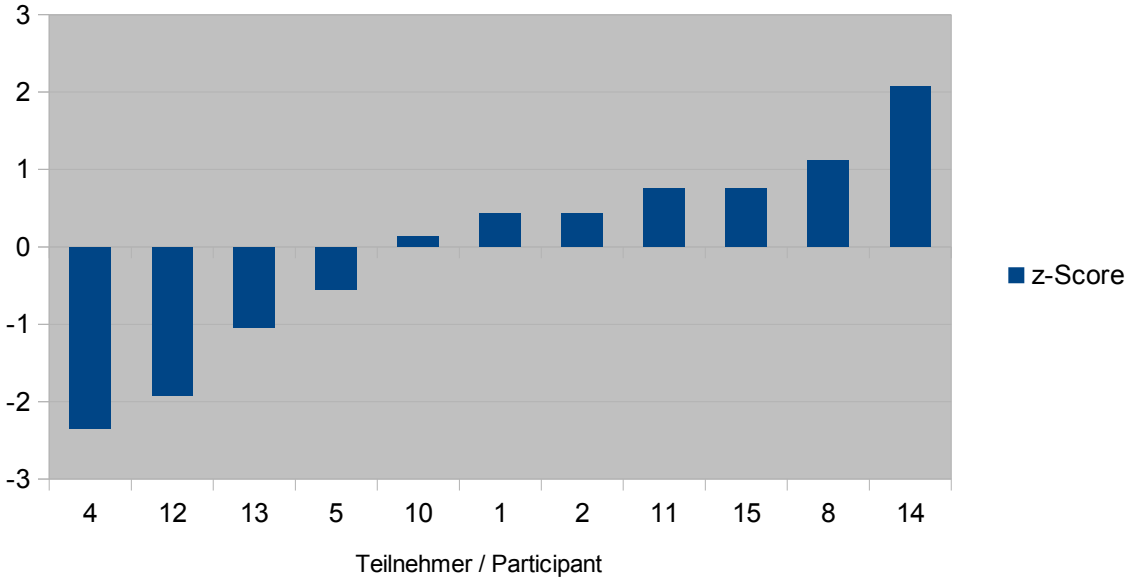


Auswerte nummer / Evaluation number	Fruktose / Fructose	Abweichung / Deviation	z-Score	Hinweis / Remark
1	6825	260	0,9	
2	6890	325	1,2	
3				
4	6400	-165	-0,6	
5	6200	-365	-1,3	
6				
7				
8	6655	90	0,3	
9				
10	6705	140	0,5	
11	6447	-118	-0,4	
12	6392	-173	-0,6	
13	6500	-65	-0,2	
14	7000	435	1,6	
15	6200	-365	-1,3	

4.3 *Fructose Sample C in g/100 g*

Statistic Data	
number of the results	11
number of outliers	0
mean	11,0
median	11,1
robust mean (X)	11,0
robust standard deviation (S^x)	0,45
target standard deviation (σ)	0,31
lower limit of target range	10,4
upper limit of target range	11,6
quotient S^x/σ	1,5
standard uncertainty U^x	0,17
quotient U^x/σ	0,6
results in target range	9
percent in target range	82

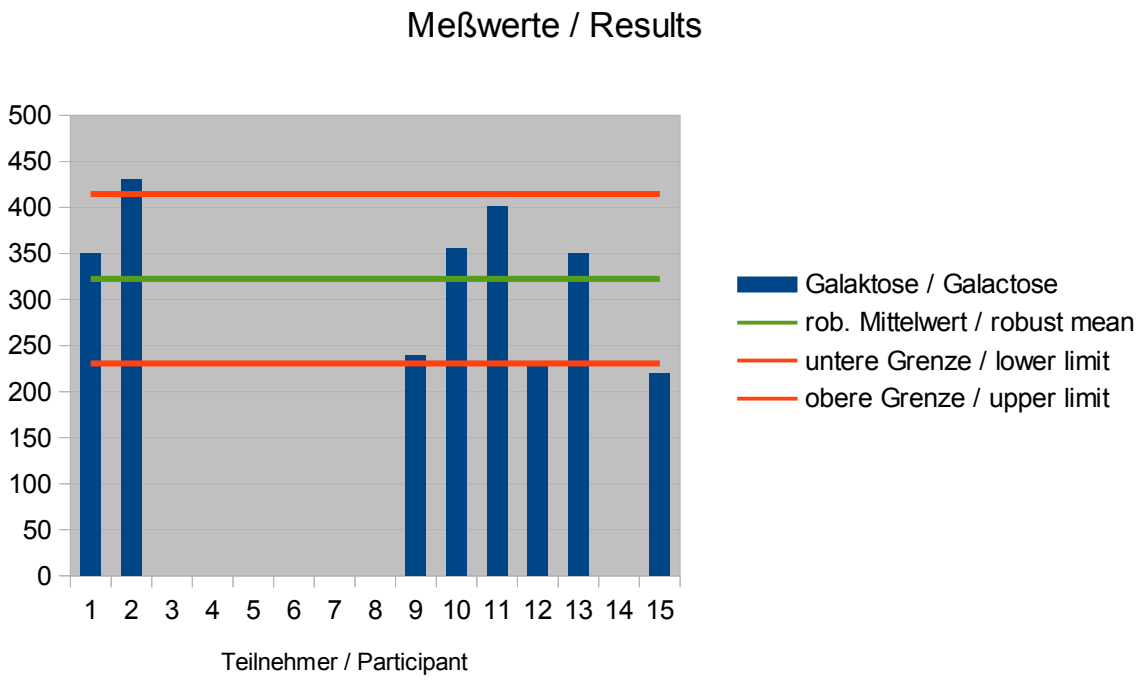


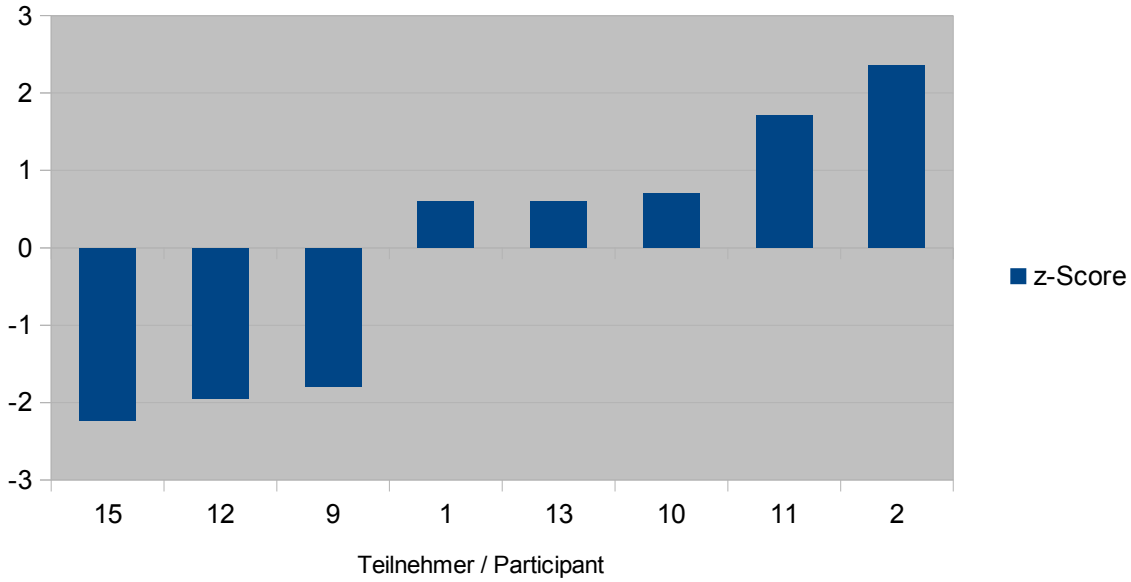


Auswerte nummer / Evaluation number	Fruktose / Fructose	Abweichung / Deviation	z-Score	Hinweis / Remark
1	11,1	0,13	0,4	
2	11,1	0,13	0,4	
3				
4	10,25	-0,72	-2,3	
5	10,8	-0,17	-0,5	
6				
7				
8	11,31	0,34	1,1	
9				
10	11,01	0,04	0,1	
11	11,2	0,23	0,8	
12	10,38	-0,59	-1,9	
13	10,65	-0,32	-1,0	
14	11,6	0,63	2,1	
15	11,2	0,23	0,8	

4.4 *Galactose Sample A in mg/kg*

Statistic Data	
number of the results	8
number of outliers	0
mean	323
median	350
robust mean (X)	323
robust standard deviation (S^x)	91,8
target standard deviation extended (σ´)	46,0
target standard deviation (σ, for information)	21,6
lower limit of target range	231
upper limit of target range	414
quotient S^x/σ	4,2
standard uncertainty U^x	40,5
quotient U^x/σ	1,9
results in target range	6
percent in target range	75

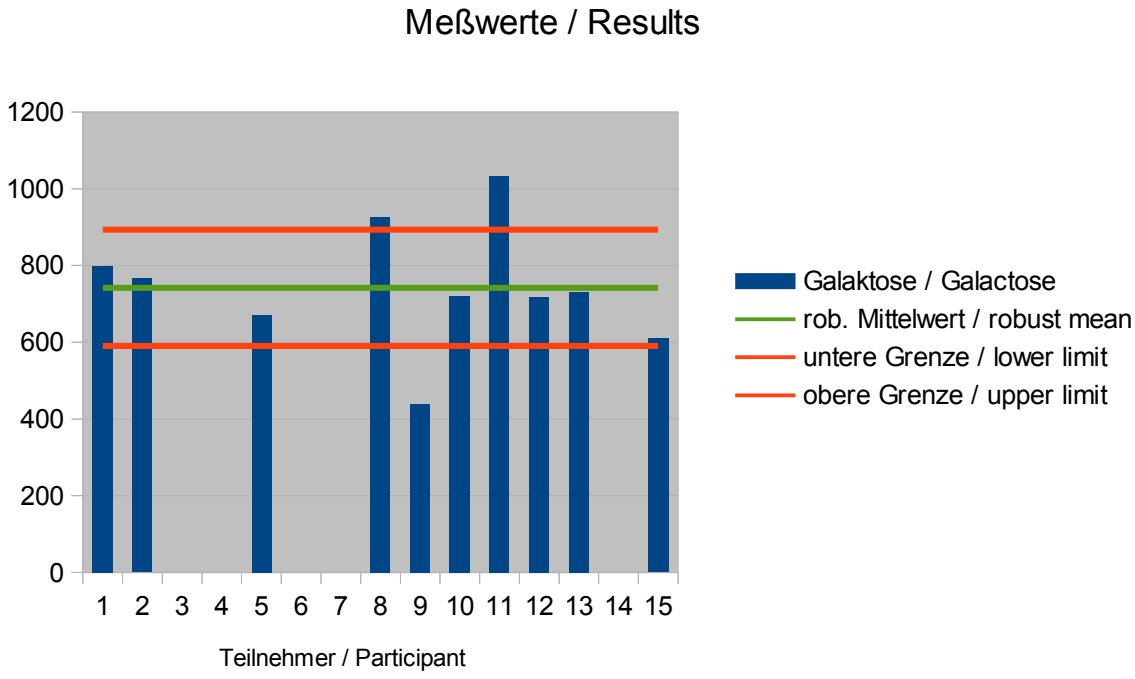


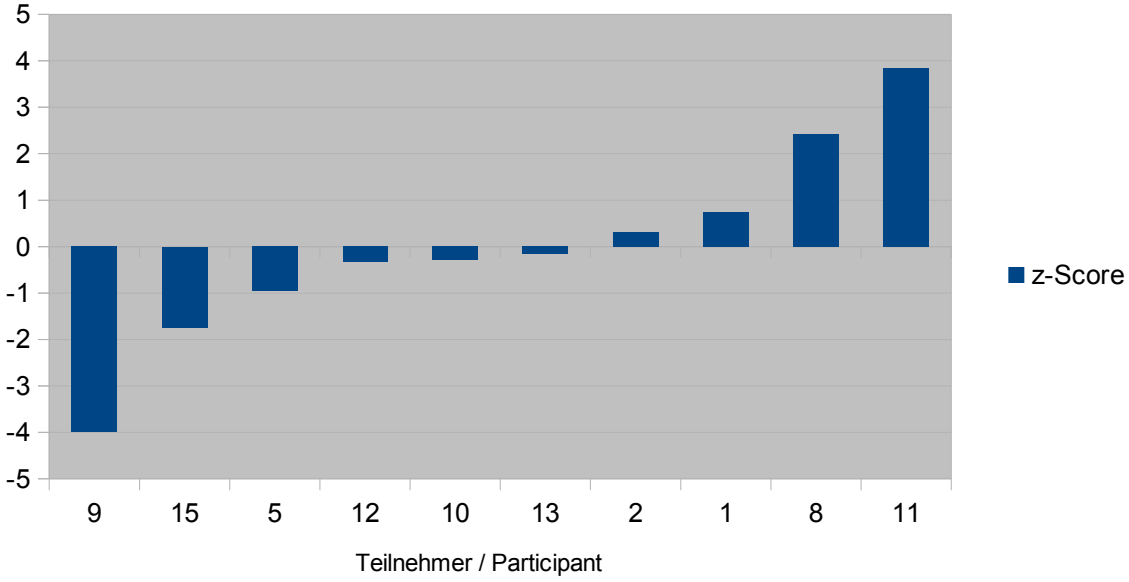


Auswerte nummer / Evaluation number	Galaktose / Galactose	Abweichung / Deviation	z-Score	Hinweis / Remark
1	350	27,5	0,6	
2	431	108,5	2,4	
3				
4				
5				
6				
7				
8	< 60	> 263	< -5,7	
9	240	-82,5	-1,8	
10	355	32,5	0,7	
11	401	78,5	1,7	
12	233	-89,5	-1,9	
13	350	27,5	0,6	
14				
15	220	-102,5	-2,2	

4.5 *Galactose Sample B in mg/kg*

Statistic Data	
number of the results	10
number of outliers	2
mean	741
median	725
robust mean (X)	742
robust standard deviation (S^x)	156
target standard deviation extended (σ´)	75,7
target standard deviation (σ, for information)	43,9
lower limit of target range	591
upper limit of target range	893
quotient S^x/σ	3,6
standard uncertainty U^x	61,7
quotient U^x/σ	1,4
results in target range	7
percent in target range	70

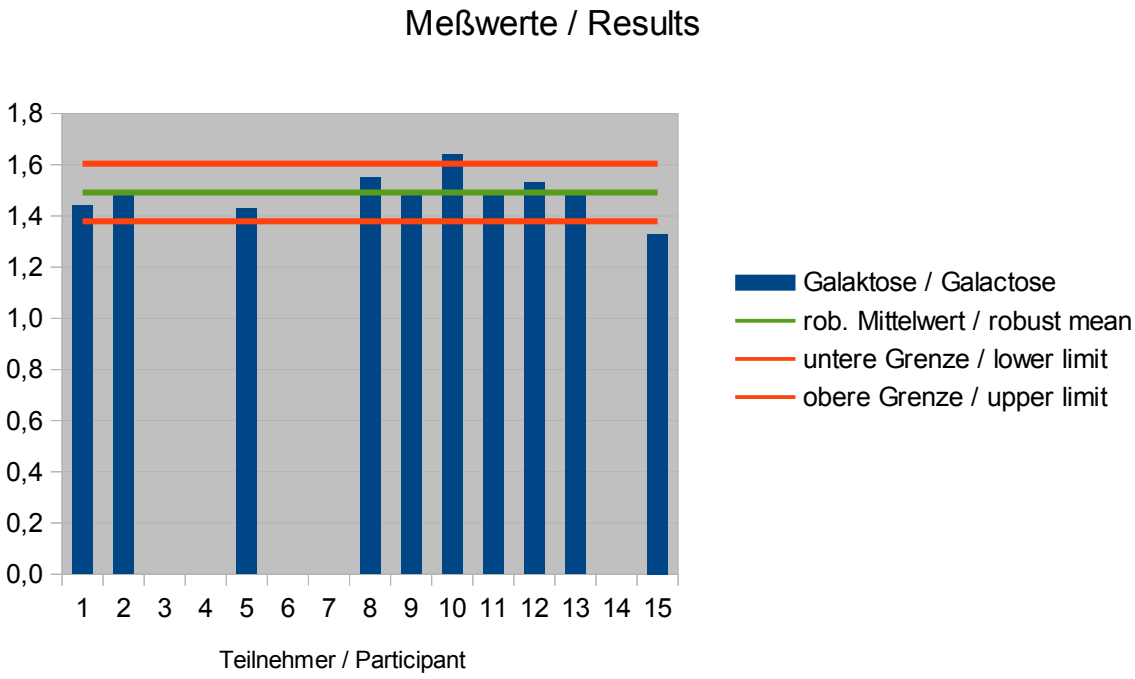


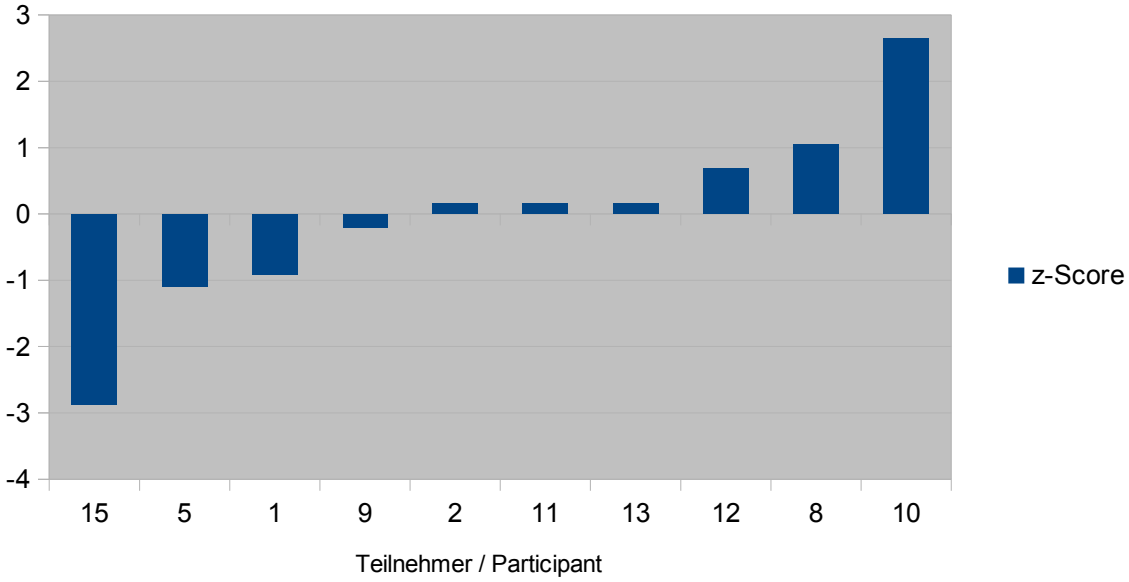


Auswerte nummer / Evaluation number	Galaktose / Galactose	Abweichung / Deviation	z-Score	Hinweis / Remark
1	798	56	0,7	
2	766	24	0,3	
3				
4				
5	670	-72	-1,0	
6				
7				
8	925	183	2,4	
9	440	-302	-4,0	Ausreisser / Outlier
10	720	-22	-0,3	
11	1033	291	3,8	Ausreisser / Outlier
12	717	-25	-0,3	
13	730	-12	-0,2	
14				
15	610	-132	-1,7	

4.6 *Galactose Sample C in g/100 g*

Statistic Data	
number of the results	10
number of outliers	2
mean	1,49
median	1,50
robust mean (X)	1,49
robust standard deviation (S^x)	0,07
target standard deviation (σ)	0,06
lower limit of target range	1,38
upper limit of target range	1,60
quotient S^x/σ	1,2
standard uncertainty U^x	0,03
quotient U^x/σ	0,5
results in target range	8
percent in target range	80

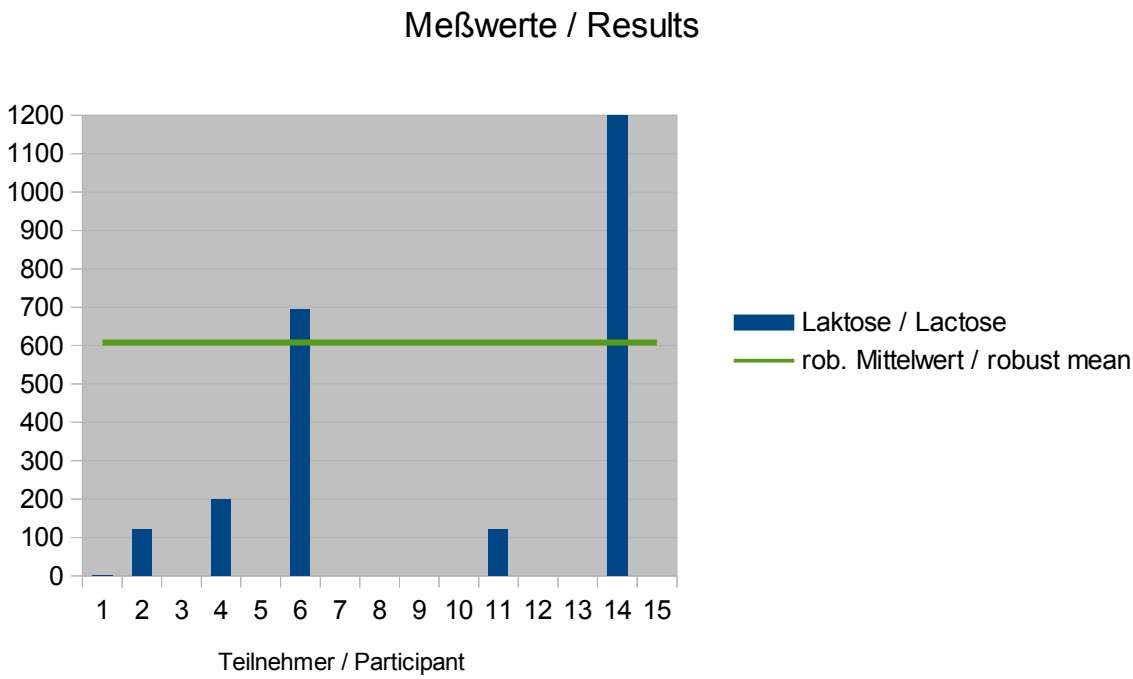




Auswerte nummer / Evaluation number	Galaktose / Galactose	Abweichung / Deviation	z-Score	Hinweis / Remark
1	1,44	-0,05	-0,9	
2	1,5	0,01	0,2	
3				
4				
5	1,43	-0,06	-1,1	
6				
7				
8	1,55	0,06	1,0	
9	1,48	-0,01	-0,2	
10	1,64	0,15	2,6	
11	1,5	0,01	0,2	
12	1,53	0,04	0,7	
13	1,5	0,01	0,2	
14				
15	1,33	-0,16	-2,9	

4.7 *Lactose Sample A in mg/kg*

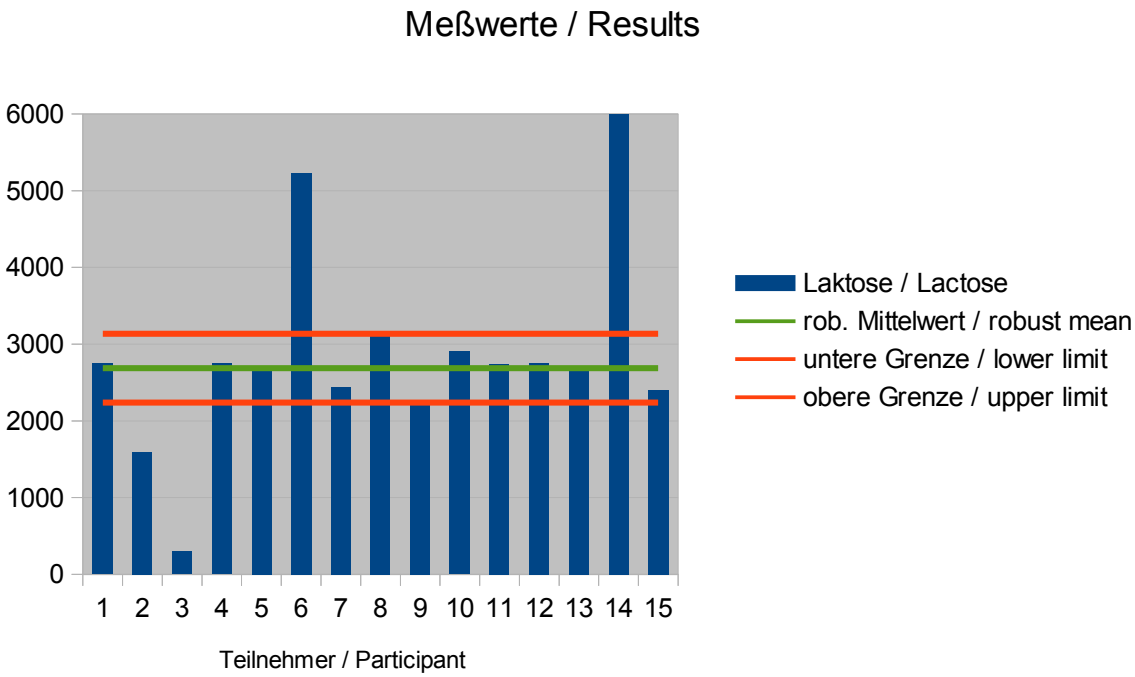
Statistic Data	
number of the results	6
number of outliers	Nicht berechnet/not calculated
mean	1756
median	161
robust mean (X)	608
robust standard deviation (S^x)	704
target standard deviation (σ)	Nicht berechnet/not calculated
target standard deviation (for information)	Nicht berechnet/not calculated
lower limit of target range	Nicht berechnet/not calculated
upper limit of target range	Nicht berechnet/not calculated
quotient S^x/σ	Nicht berechnet/not calculated
standard uncertainty U^x	Nicht berechnet/not calculated
quotient U^x/σ	Nicht berechnet/not calculated
results in target range	Nicht berechnet/not calculated
percent in target range	Nicht berechnet/not calculated

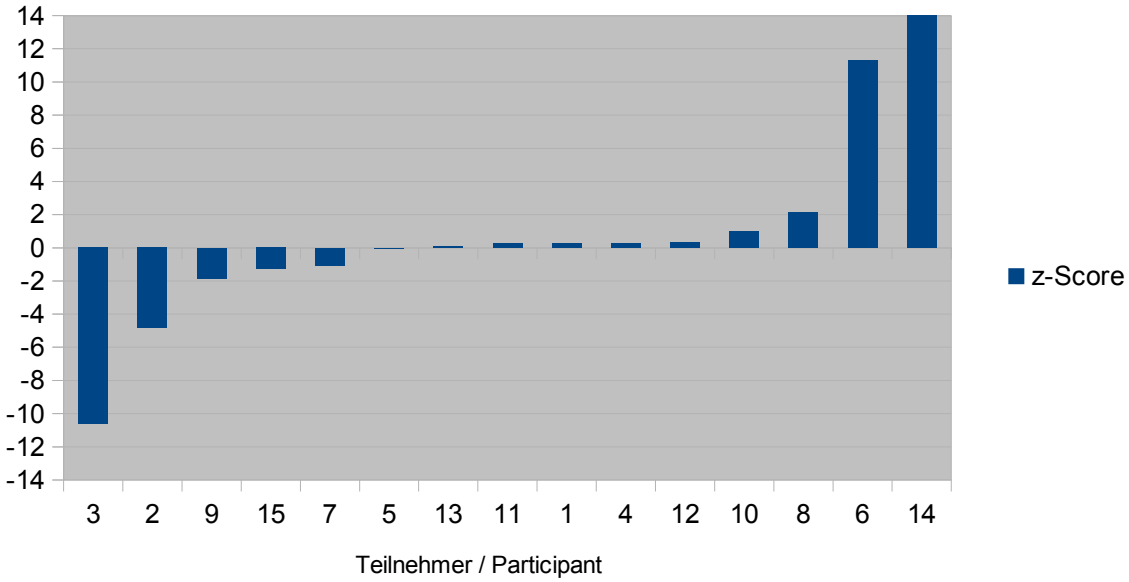


Auswerte nummer / Evaluation number	Laktose / Lactose	Abweichung / Deviation	z-Score	Hinweis / Remark
1				
2	121	-487		
3				
4	200	-408		
5				
6	694	86		
7				
8				
9				
10				
11	121	-487		
12				
13				
14	9400	8792		
15				

4.8 *Lactose Sample B in mg/kg*

Statistic Data	
number of the results	15
number of outliers	1
mean	3079
median	2741
robust mean (X)	2688
robust standard deviation (S^x)	566
target standard deviation extended (σ´)	225
target standard deviation (σ, for information)	131
lower limit of target range	2238
upper limit of target range	3137
quotient S^x/σ	4,3
standard uncertainty U^x	183
quotient U^x/σ	1,4
results in target range	10
percent in target range	67

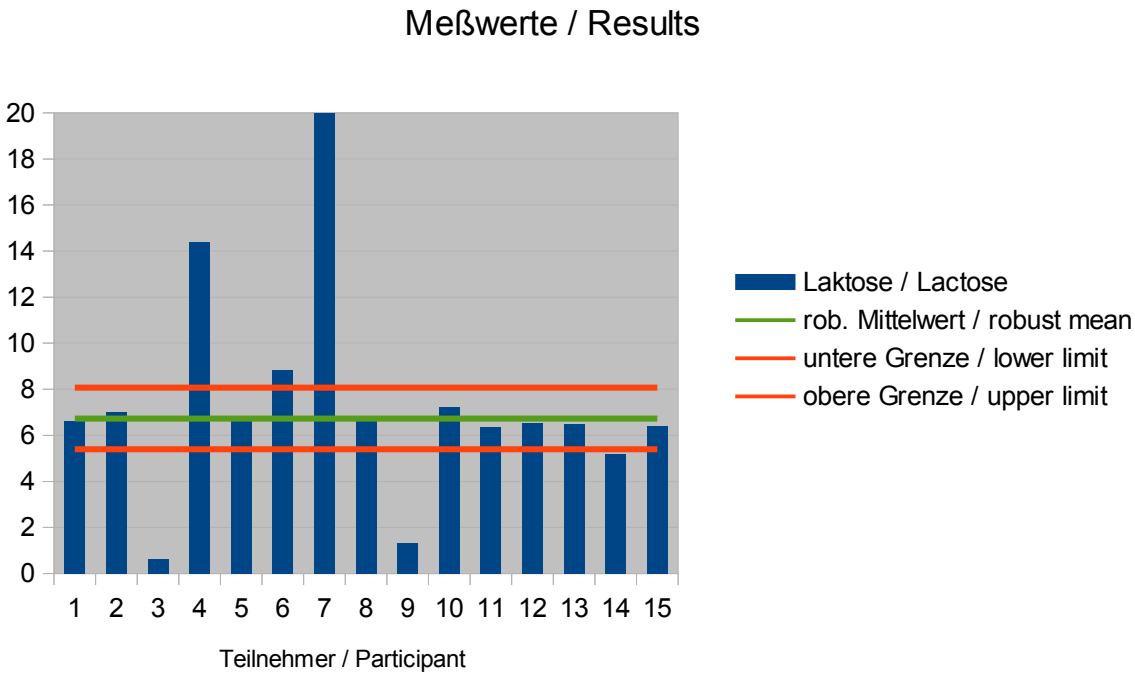


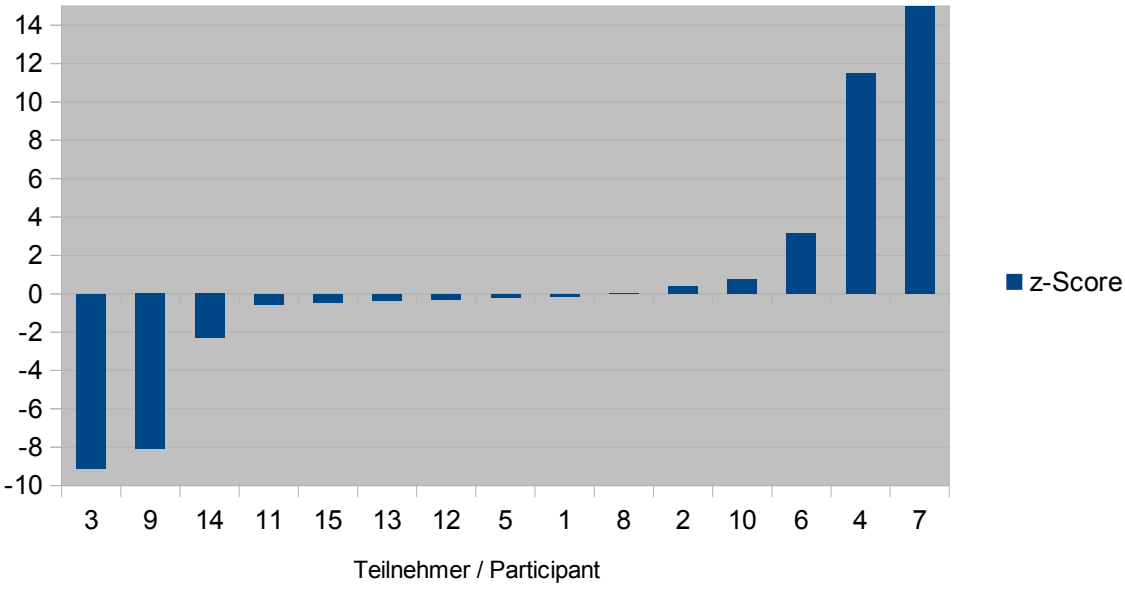


Auswerte nummer / Evaluation number	Laktose / Lactose	Abweichung / Deviation	z-Score	Hinweis / Remark
1	2750	62	0,3	
2	1600	-1088	-4,8	
3	300	-2388	-10,6	
4	2750	62	0,3	
5	2680	-8	0,0	
6	5228	2540	11,3	
7	2444	-244	-1,1	
8	3165	477	2,1	
9	2270	-418	-1,9	
10	2905	217	1,0	
11	2741	53	0,2	
12	2758	70	0,3	
13	2700	12	0,1	
14	9500	6812	30,3	Ausreisser / Outlier
15	2400	-288	-1,3	

4.9 *Lactose Sample C in g/100 g*

Statistic Data	
number of the results	15
number of outliers	1
mean	2990
median	6,60
robust mean (X)	6,73
robust standard deviation (S^x)	1,97
target standard deviation extended (σ´)	0,67
target standard deviation (σ, for information)	0,20
lower limit of target range	5,40
upper limit of target range	8,07
quotient S^x/σ	9,8
standard uncertainty U^x	0,64
quotient U^x/σ	3,1
results in target range	9
percent in target range	60





Auswerte nummer / Evaluation number	Laktose / Lactose	Abweichung / Deviation	z-Score	Hinweis / Remark
1	6,64	-0,09	-0,1	
2	7	0,27	0,4	
3	0,65	-6,08	-9,1	
4	14,4	7,67	11,5	
5	6,6	-0,13	-0,2	
6	8,82	2,09	3,1	
7	44760	44753,27	67031,8	Ausreisser (Einheit?) / Outlier (Unit?)
8	6,72	-0,01	0,0	
9	1,33	-5,4	-8,1	
10	7,25	0,52	0,8	
11	6,37	-0,36	-0,5	
12	6,53	-0,2	-0,3	
13	6,5	-0,23	-0,3	
14	5,19	-1,54	-2,3	
15	6,42	-0,31	-0,5	

5 Documentation

5.1 *Primary data*

5.1.1 Fructose

Sample A

Teilnehmer / Participant	Einheit / Unit	Nachweisbar / Detectable	Nachweisgrenze / Limit of Detection	Bestimmungsgrenze / Limit of quantification	Endergebnis / Final result	Wiederfindungsrate / Recovery	Wiederfindung mit gleicher Matrix / Recovery in the same matrix
		yes / no				in %	yes / no
1	mg/kg	yes			10659		
2	mg/kg	yes		1000	10220		
3	mg/kg						
4	mg/kg	yes			10600		
5	mg/kg	yes	100	500	10650	95	yes
6	mg/kg						
7	mg/kg						
8	mg/kg	yes	10	30	10620	n.c.	n.c.
9	mg/kg	not performed					
10	mg/kg	yes		1000	10040		
11	mg/kg	yes	170	510	9056	99,3	no
12	mg/kg	yes	30	100	10583		no
13	mg/kg	yes			10550		
14	mg/kg	yes			13300		
15	mg/kg	yes	30	100	10500		no

Sample B

Teilnehmer / Participant	Einheit / Unit	Nachweisbar / Detectable	Nachweisgrenze / Limit of Detection	Bestimmungsgrenze / Limit of quantification	Endergebnis / Final result	Wiederfindungsrate / Recovery	Wiederfindung mit gleicher Matrix / Recovery in the same matrix
		yes / no				in %	yes / no
1	mg/kg	yes			6825		
2	mg/kg	yes		1000	6890		
3	mg/kg						
4	mg/kg	yes			6400		
5	mg/kg	yes	50	250	6200	105	yes
6	mg/kg						
7	mg/kg						
8	mg/kg	yes	10	30	6655	n.c.	n.c.
9	mg/kg	not performed					
10	mg/kg	yes		1000	6705		
11	mg/kg	yes	170	510	6447	99,3	no
12	mg/kg	yes	30	100	6392		no
13	mg/kg	yes			6500		
14	mg/kg	yes			7000		
15	mg/kg	yes	30	100	6200		

Sample C

Teilnehmer / Participant	Einheit / Unit	Nachweisbar / Detectable	Nachweisgrenze / Limit of Detection	Bestimmungsgrenze / Limit of quantification	Endergebnis / Final result	Wiederfindungsrate / Recovery	Wiederfindung mit gleicher Matrix / Recovery in the same matrix
		yes / no				in %	yes / no
1	%	yes			11,1		
2	%	yes		0,1	11,1		
3	%						
4	%	yes			10,25		
5	%	yes	0,005%	0,025%	10,8	106	yes
6	%						
7	%						
8	%	yes	10	30	11,31	n.c.	n.c.
9	%	not performed					
10	%	yes		0,1	11,01		
11	%	yes			11,2	99,3	no
12	%	yes	30	100	10,38		no
13	%	yes			10,65		
14	%	yes			11,6		
15	%	yes	0	0,01	11,2		no

5.1.2 Galactose

Sample A

Teilnehmer / Participant	Einheit / Unit	Nachweisbar / Detectable	Nachweisgrenze / Limit of Detection	Bestimmungsgrenze / Limit of quantification	Endergebnis / Final result	Wiederfindungsrate / Recovery	Wiederfindung mit gleicher Matrix / Recovery in the same matrix
		yes / no				in %	yes / no
1	mg/kg	yes			350		
2	mg/kg	yes		20	431		
3	mg/kg						
4	mg/kg						
5	mg/kg	no	100	500	<500	-	-
6	mg/kg						
7	mg/kg						
8	mg/kg	yes	20	60	<BG	n.c.	n.c.
9	mg/kg	yes		0.008 %	0.024 %		
10	mg/kg	yes		100	355		
11	mg/kg	yes	45	135	401	98,1	no
12	mg/kg	yes	10	30	233		no
13	mg/kg	yes			350		
14	mg/kg						
15	mg/kg	yes	30	100	220		no

Sample B

Teilnehmer / Participant	Einheit / Unit	Nachweisbar / Detectable	Nachweisgrenze / Limit of Detection	Bestimmungsgrenze / Limit of quantification	Endergebnis / Final result	Wiederfindungsrate / Recovery	Wiederfindung mit gleicher Matrix / Recovery in the same matrix
		yes / no				in %	yes / no
1	mg/kg	yes			798		
2	mg/kg	yes		20	766		
3	mg/kg						
4	mg/kg						
5	mg/kg	yes	50	250	670	134	yes
6	mg/kg						
7	mg/kg						
8	mg/kg	yes	20	60	925	n.c.	n.c.
9	mg/kg	yes		0.008 %	0.044 %		
10	mg/kg	yes		100	720		
11	mg/kg	yes	45	135	1033	98,1	no
12	mg/kg	yes	10	30	717		no
13	mg/kg	yes			730		
14	mg/kg						
15	mg/kg	yes	30	100	610		

Sample C

Teilnehmer / Participant	Einheit / Unit	Nachweisbar / Detectable	Nachweisgrenze / Limit of Detection	Bestimmungsgrenze / Limit of quantification	Endergebnis / Final result	Wiederfindungsrate / Recovery	Wiederfindung mit gleicher Matrix / Recovery in the same matrix
		yes / no				in %	yes / no
1	%	yes			1,44		
2	%	yes		0,1	1,5		
3	%						
4	%						
5	%	yes	0,005%	0,025%	1,43	108	yes
6	%						
7	%						
8	%	yes	20	60	1,55	n.c.	n.c.
9	%	yes		0.008 %	1.48 %		
10	%	yes		0,01	1,64		
11	%	yes			1,5	98,1	no
12	%	yes	10	30	1,53		no
13	%	yes			1,5		
14	%						
15	%	yes	0	0,01	1,33		no

5.1.3 Lactose

Sample A

Teilnehmer / Participant	Einheit / Unit	Nachweisbar / Detectable	Nachweisgrenze / Limit of Detection	Bestimmungsgrenze / Limit of quantification	Endergebnis / Final result	Wiederfindungsrate / Recovery	Wiederfindung mit gleicher Matrix / Recovery in the same matrix
		yes / no				in %	yes / no
1	mg/kg	no			<100		
2	mg/kg	yes		40	121		
3	mg/kg	no					
4	mg/kg	yes			200		
5	mg/kg	no	100	500	<500	-	-
6	mg/kg	yes	30	30	694	61,98	61,98
7	mg/kg	no	500	500	<500	90	yes
8	mg/kg	yes	10	30	<LOQ	n.c.	n.c.
9	mg/kg	no		0.008 %		94	no
10	mg/kg	yes		100	<100		
11	mg/kg	yes	30	90	121	95,1	no
12	mg/kg	no	30	100	n.n.		no
13	mg/kg	no			n.n.		
14	mg/kg	yes			9400		
15	mg/kg	no	30	100	<100		no

Sample B

Teilnehmer / Participant	Einheit / Unit	Nachweisbar / Detectable	Nachweisgrenze / Limit of Detection	Bestimmungsgrenze / Limit of quantification	Endergebnis / Final result	Wiederfindungsrate / Recovery	Wiederfindung mit gleicher Matrix / Recovery in the same matrix
		yes / no				in %	yes / no
1	mg/kg	yes			2750		
2	mg/kg	yes		40	1600		
3	mg/kg	yes			300		
4	mg/kg	yes			2750		
5	mg/kg	yes	50	250	2680	110	yes
6	mg/kg	yes	30	30	5228	58,8	58,8
7	mg/kg	yes	500	500	2444	90	yes
8	mg/kg	yes	10	30	3165	n.c.	n.c.
9	mg/kg	yes		0.008 %	0.227 %	96	s.o.
10	mg/kg	yes		100	2905		
11	mg/kg	yes	30	90	2741	95,1	no
12	mg/kg	yes	30	100	2758		no
13	mg/kg	yes			2700		
14	mg/kg	yes			9500		
15	mg/kg	yes	30	100	2400		

Sample C

Teilnehmer / Participant	Einheit / Unit	Nachweisbar / Detectable	Nachweisgrenze / Limit of Detection	Bestimmungsgrenze / Limit of quantification	Endergebnis / Final result	Wiederfindungsrate / Recovery	Wiederfindung mit gleicher Matrix / Recovery in the same matrix
		yes / no				in %	yes / no
1	%	yes			6,64		
2	%	yes		0,1	7,0		
3	%	yes			0,65		
4	%	yes			14,4		
5	%	yes	0,500%	0,025%	6,6	101	yes
6	%	yes	0,03	0,03	8,82%	61,98	61,98
7	%	yes	500	500	44760	90%	yes
8	%	yes	10	30	6,72	n.c.	n.c.
9	%	yes		0.008 %	1.33 %	97	s.o.
10	%	yes		0,01	7,25		
11	%	yes			6,37	95,1	no
12	%	yes	30	100	6,53		no
13	%	yes			6,5		
14	%	yes			5,19		
15	%	yes	0	0,01	6,42		no

5.2 Homogeneity

Homogeneity was tested by 5-fold HPLC-analysis of fructose in samples B and C.
Sample C showed a fructose content of 11,3 % with a standard deviation of relative 2,6%.
Sample B showed a fructose content of 0,64 % with a standard deviation of relative 4,5%.
These results were accepted.

	Fructose	Fructose
	Sample C	Sample B
1	11,54	0,64
2	11,24	0,67
3	10,94	0,64
4	11,64	0,66
5	11,09	0,59
Mean	11,29	0,64
Standard-deviation	0,3	0,03
In %	2,6	4,5

5.3 Analytical methods

5.3.1 *Fructose*

Participant	Method is accredited	Method	Sample preparation	Weight	Extraction	Reference material / Manufacturer	Remark
	Yes / no						
1	yes	Enzymatic acc. Enzytec	Mixing	1-5 g	60 °C Ultrasonic		1
2	yes	Enzymatic					
3							
4	no	HPLC-Light-Scattering			Carrez		
5	no	Determination of fructose after extraction HPAEC-PAD	Mixing, extraction with water, clean up with Chloroforme, filtration, dilution	app. 1g	aqueous (30min at 40 °C)	D-(-)-Fructose/Sigma-Aldrich	
6							
7							
8	yes	Enzymatic	Mixing	1g/10ml	Aqueous extraction	r-biopharm	Sample not homogeneous.
9							
10	yes	Enzymatic, according to kit-manufacturer					
11	Yes / no	Literatur method		1 g/100 mL	Water	Sigma/Aldrich	
12	yes	HPAEC-PAD	Extraction, filtration, dilution	1 g	100 ml demin. Water, 60°C	Merck	
13	yes	48.02.07 § 64 LFGB, modified	60°C water, Carrez I and II	1,208g/0, 909g	Water, 60°C, 15 min magnetic stirer		Sample A, Nr.16
14	no						
15	yes	HPIC-PAD	Extraction with hot water	1 g	60 °C		

5.3.2 *Lactose and Galactose*

Participant	Method is accredited	Method	Sample preparation	Weight	Extraction	Reference material / Manufacturer	Remark
	yes / no						
1	yes	Enzymatic acc. Enzytec	Mixing	1-5 g	60 °C Ultrasonic		1
2	yes	Enzymatic					
3		§ 64 L 07.00-23					
4		Enzymatic			Carrez		
5	no	Determination of lactose after extraction HPAEC-PAD	Mixing, extraction with water, clean up with Chloroforme, filtration, dilution	App. 1g	Aqueous (30 min at 40 °C)	D-(+)-Lactose x H_2O/Sigma-Aldrich	
6	no	Enzym Test (Megazyme K-LACGAR)	Extraction in water and Carrez	1 g	50° C	Former PT	Result with recovery
7	yes	Enzymatic, R-biopharm (roche)	Aqueous extract	2g	70°C 15 Min	Former PT	
8	yes	Enzymatic	Mixing	1g/10ml	Aqueous extraction	r-biopharm	Sample not homogeneous.
9	yes	NMKL Method 155, 2. ed 2006, SLV K2-m229.5 with Testkit Lactose/D-Galactose UV-Test R-biopharm	Sample was shaken and 2.0 g were taken for the analysis.	2.0 g	60ml MQ water and the sample were heated to 70C/15 min. in waterbath. After cooling 5ml Carrez I, 5ml Carrez II and 10ml NaOH, and the extract 30 min. Fill up with wWater to 100ml. To remove fat the sample was centrifuged.	In house Reference aterial from cornflakes	
10	yes	Enzymatic, according to manufacturer					
11	yes / no	Literature method	Carrez	2 g/100 mL	Water 70°C, 15 min	Sigma/Aldrich	
12	yes	HPAEC-PAD	Extraction, filtration, dilution	1 g	100 ml demin. Water, 60°C	Merck	
13	yes	L01.00-17 §64 LFGB, modified	Extraction in water, Carrez I and II	1,151g/0,925g	Water 70°C, 15 min		Sample A, Nr.16
14	yes						
15	yes	HPIC-PAD	Extraction with hot water	1 g	60 °C		

6 Index of participant laboratories

Teilnehmer / Participant	Ort / Location	Land / Country
		GERMANY
		GERMANY
		GERMANY
		GERMANY
		GERMANY
		GERMANY
		GERMANY
		GERMANY
		GERMANY
		GERMANY
		GERMANY
		SWEDEN
		GERMANY
		ENGLAND
		SPAIN
		GERMANY
		NETHERLANDS

[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 Analytical 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 L 17.00-7 Bestimmung von Lactose in Brot einschließlich Kleingebäck aus Brotteigen (1983)
14. ASU §64 LFGB L 48.02.07-1 Bestimmung von Glucose und Fructose in Kinder-Zwieback und Zwiebackmehl (1985).