DLA
Dienstleistung
Lebensmittel
Analytik GbR

Evaluation Report
proficiency test

DLA 14/2016

Lactose and Fructose

in "lactose free" food (bread baking mixture)

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Inhalt / Content

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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

Two PT-samples for the detection of lactose/galactose and fructose and one spiking material sample were provided for analysis. The spiking material sample contains the respective analytes in the range of 1-10 % and was added to the spiked PT-sample. The results of the spiking material sample should give the possibility of a comparison with the spiked sample in respect to the detectability of the allergens with and without the influence of matrix and / or food processing.

The test material consists of a common in commerce "lactose free" bread baking mixture. The basic composition of both sample A and sample B was the same (see table 1). The spiking material, which contains lactose and fructose, was added to sample B. The sugars were ground and sieved before (mesh 400 µm). The composition of the spiking material sample and the amounts of analytes in sample B are given in table 2.
Before homogenization microtracer particles were added in order to check the accuracy of mixing. After homogenization aliquots were taken for microtracer analysis (s. 2.1.1).
Afterwards the samples were portioned to approximately 25 g into metallised PET film bags and chronologically numbered.

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Table 1: Composition of DLA-Sample

Ingredients	**Sample A**	**Sample B**
Baking Mixture, Farmhouse Bread Ingredients: Wheat flour 75%, rye flour 20%, iodized salt, yeast, glucose, barley malt, flour treatment agent: ascorbic acid	100 g/100g	96,5 g/100g
Spiking Material Sample	-	3,49 g/100g

Table 2: Added amounts of lactose and fructose

Ingredients	**Spiking material sample**	**Sample B**
Potato flour	88,6 %	3,09 %
Lactose	4,21 %	147 mg/100g
Fructose	7,16 %	250 mg/100g *

* Sample B contains additional fructose from the matrix of the baking mixture

2.1.1 Homogeneity

The **mixture homogeneity before bottling** was examined 8-fold by **microtracer analysis**. It is a standardized method that is part of the international GMP certification system for feed [14].
Before mixing dye coated iron particles of µm size are added to the sample and the number of particles is determined after homogenization in taken aliquots. The evaluation of the mixture homogeneity is based on the Poisson distribution using the chi-square test. A probability of ≥ 5 % is equivalent to a good homogeneous mixture and of ≥ 25% to an excellent mixture [14, 15].
The microtracer analysis of the present PT samples (spiking material sample and sample B) showed probabilities of 99% and 89%, respectively. Additionally particle number results were converted into concentrations, statistically evaluated according to normal distribution and compared to the standard deviation according to Horwitz. This gave HorRat values of 0,5 and 0,9, respectively. The results of microtracer analysis are given in the documentation.

The **homogeneity of bottled numbered DLA-samples** was checked by 5fold determination of lactose by enzyme UV-test (ASU §64 L 48.01-4). The repeatability standard deviations are 0,56 % and 1,2% and are in the range of the repeatability standard deviation of e.g. the German official method ASU §64 L 48.02-7 for glucose and fructose in rusk. The results of the homogeneity test are given in the documentation.

The calculation of the **repeatability standard deviation S_r of the participants** could not be calculated, because only single results were requested from participants.

Furthermore, the homogeneity was characterized by the **trend line function of participants' results for chronological bottled single samples**. The

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maximum deviations from the mean value of the trend lines were in the range of 30% of the target standard deviations $\sigma_{pt'}$ (s. 5.2 homogeneity) for spiking material sample, sample A and sample B and can therefore be regarded as low.

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

2.2 Sample shipment and information to the test

Two portions of test material were sent to every participating laboratory in the 15th week of 2016. The testing method was optional. The tests should be finished at 27th May 2016 the latest.

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

Notes on test:
There are two different test samples "lactose free" bread baking mixture (sample A and sample B) possibly containing lactose, galactose and/or fructose. Additionally a „Spiking Material Sample" is provided which was used for the spiking of the positive sample (A or B). It contains 3-15% of lactose and fructose. It should be analysed like a normal sample (eventually diluted).
The homogeneity of the material was tested. Every suitable method for detection or determination of the analytes may be applied. In general we recommend to homogenize a representative sample amount before analysis according to good laboratory practice, especially in case of low sample weights.

2.3 Submission of results

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

The finally calculated concentrations of the parameter were used for the statistical evaluation.

Queried and documented were single results, recovery and the used testing methods.

From 21 participants one participant submitted no results and another submitted the results delayed in consultation with DLA. All other participants submitted the result in time.

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3. Evaluation

3.1 Consensus value from participants (assigned value)

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

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

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

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

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

3.2 Robust standard deviation

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

3.3 Repeatability standard deviation

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

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

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

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from participants are available.

3.4 Reproducibility standard deviation

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

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

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

The relative reproducibility standard deviation as a percentage of the mean value is indicated as coefficient of variation CV_R in the table of statistical characteristics in the results section in case single results from participants are availble. Its meaning is explained in more detail in 3.9.

3.5 Exclusion of results and outliers

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

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

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

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3.6 Target standard deviation (for proficiency assessment)

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

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

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

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

For the valuation of lactose in the spiking material sample as well as fructose and galactose the target standard deviation according to the general model of Horwitz was applied (see 3.4.2).

For the valuation of lactose in sample B the target standard deviation according to a precision experiment was applied (3.4.2, official German ASU §64 method: 17), because in this case a broader target range in comparison to the general model according to Horwitz (z'-score included) was achieved.

Additionally for all analytes the standard uncertainty was considered by valuating with z'-scores (see 3.6).

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3.6.1 General model (Horwitz)

Based on statistical characteristics obtained in numerous PTs for different parameters and methods Horwitz has derived a general model for estimating the reproducibility standard deviation σ_R [6]. Later the model was modified by Thompson for certain concentration ranges [10]. The reproducibility standard deviation σ_R can be applied as the relative target standard deviation σ_{pt} in % of the assigned values and calculated according to the following equations [3]. For this the assigned value X_{pt} is used for the concentration c.

Equations	**Range of concentrations**	**corresponds to**
$\sigma_R = 0{,}22c$	$c < 1{,}2 \times 10^{-7}$	< 120 µg/kg
$\sigma_R = 0{,}02c^{0{,}8495}$	$1{,}2 \times 10^{-7} \leq c \leq 0{,}138$	≥ 120 µg/kg
$\sigma_R = 0{,}01c^{0{,}5}$	$c > 0{,}138$	> 13,8 g/100g

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

3.6.2 Value by precision experiment

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

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

The values given in Table 2 relative repeatability standard deviation (RSD_r) and relative reproducibility standard deviation (RSD_R) were determined in collaborative trials using the specified methods.
The in the table indicated resulting target standard deviations σ_{pt} were used for evaluation of the results.
For information the target standard deviations according to Horwitz are given additionally.

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Table 2: Relative repeatability standard deviations (RSD_r) and relative reproducibility standard deviations (RSD_R) from precision experiments and resulting target standard deviations σ_{pt} [16-21]

Parameter	**Matrix**	**Mean values**	**RSD_r**	**RSD_R**	**σ_{pt}**	**Method / Literature**
Fructose	rusk	7,0%	1,59%	2,59%	2,59%[1]	ASU §64 L 48.02.07-1
Lactose	infant food	28,7%	1,66%	3,33%	3,33%	ASU §64 L 48.02.07-1
Lactose	"lactose free" skimmed milk	0,13%	20 %	30 %	30 %	ASU §64 L 01.00-17
Lactose	"lactose free" milk (3 samples)	0,0282% 0,0804% 0,1257%	6,74% 1,71% 6,25%	10,86% 3,95% 7,33%	10,86%[1] 3,95% 7,33%[1]	ASU §64 L 01.00-90
Lactose	milk	4,55%	0,48%	1,01%	1,01%	ISO 22662
Lactose	cream	3,04%	0,66%	4,41%	4,41%	ISO 22662
Lactose	milk powder	44,5%	0,30%	2,36%	2,36%	ISO 22662

[1] *used in evaluation (s. chapter 4), for lactose as mean (9,10%)*

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 3.6.2 was regarded suitable.

The participants' results of the present PT are within the usual range of the results of previous years. Table 4 gives an overview of the data of each PT sample spiked with fructose or lactose. The coefficients of variation are between about 5 and 40%. Lactose was always evaluated with an extended target range (since 2014 by z'-score).

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Table 4: Characteristics of the present PT (on dark gray) of fructose and lactose in comparison to previous PTs since 2013 (SD = standard deviation, CV = coefficient of variation)

Parameter	**Matrix**	**robust Mean [mg/100g]**	**robust SD (S*) [mg/100g]**	**rel. SD (CV_{S*}) [%]**	**Quotient $S^*/\sigma_{pt'}$**	**DLA-Report**
Fructose	cookies	288	119	41,3	(9,0)*	DLA 8/2013 (sample B)
Fructose	rusk	657	30,7	4,7	1,1*	DLA 8/2014 (sample B)
Fructose	cookies	1130	122	10,8	1,7	DLA 9/2015 (sample B)
Fructose	bread baking mixture	880 660	105 187	11,9 28,3	1,6 2,1	DLA 14/2016 (sample B)**
Lactose	cookies	142	37,1	26,1	(4,9)*	DLA 8/2013 (sample A)
Lactose	rusk	269	56,6	21,1	2,5	DLA 8/2014 (sample B)
Lactose	cookies	116	37,3	32,2	2,8	DLA 9/2015 (sample B)
Lactose	bread baking mixture	154	26,7	17,3	1,6	DLA 14/2016 (sample B)

** with target standard deviation σ_{pt}*
*** enzyme methods (1st line) and other methods (2nd line)*

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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

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

3.7.1 Warning and action signals

In accordance with the norm ISO 13528 it is recommended that a result that gives rise to a *z*-score above 3,0 or below −3,0, shall be considered to give an "action signal" [3]. Likewise, a *z*-score above 2,0 or below −2,0 shall be considered to give a "warning signal". A single "action signal", or "warning signal" in two successive PT-rounds, shall be taken as evidence that an anomaly has occurred which requires investigation. For example a fault isolation or a root cause analysis through the examination of transmission error or an error in the calculation, in the trueness and precision must be performed and if necessary appropriate corrective measures should be applied [3].

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

3.8 z'-Score

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

The calculation is performed by:

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

If carried out an evaluation of the results by means of z 'score, we have

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defined below the expression in the denominator as a target standard deviation σ_{pt}'.

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

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

For warning and action signals see 3.7.1.

3.9 Reproducibility coefficient of variation (CK_R)

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

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

3.11 Standard uncertainty

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

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

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

The quotient $U(X_{pt})/\sigma_{pt}$ is reported in the characteristics of the test.

3.12 Recovery rates: Spiking

For the results of the spiking material sample and the spiked sample recovery rates were calculated by DLA with respect to the known content of added lactose. The related values of added lactose are given in 2.1 test material in table 2. As a range of acceptance RA for valuating participant's results the range of 85 - 115% for the recovery rates were deduced from published methods (15-19).
For lactose results of the spiking material sample and the spiked sample recovery rates were calculated with respect to the known added content of lactose. The recovery rates were given for information only. No statistical evaluation was done. The recovery rates should exclusively give an estimation of the matrix- and/or processing influences.

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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 first table the characteristics are listed:

Statistic Data
Number of results
Number of outliers
Mean
Median
Robust mean(X_{pt})
Robust standard deviation (S^*)
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})
Quotient U(X_{pt})/σ_{pt} or U(X_{pt})/σ_{pt}'
Number of results in the target range
Percent in the target range

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

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

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

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4.1 Fructose

4.1.1 Fructose Sample A (in mg/100g)

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	13
Number of outliers	1
Mean	669
Median	551
Robust Mean (X)	**578**
Robust standard deviation (S*)	**205**
Target range:	
Target standard deviation *σpt'*	**75,5**
Target standard deviation (for Information)	15,0
lower limit of target range	**427**
upper limit of target range	**729**
Quotient S/$\sigma_{pt'}$*	*2,7*
Standard uncertainty $U_{(Xpt)}$	*71,2*
Quotient $U_{(Xpt)}/\sigma_{pt'}$	*0,94*
Results in the target range	*8*
Percent in the target range	*62%*

Comments to the statistic data:

The target standard deviation was calculated according to the model of Horwitz.

The distribution of results showed an increased variability. Valuation was done considering the standard uncertainty by z'-score. The quotient $S^*/\sigma_{pt'}$ was above 2,0. The robust standard deviation was in the range of previous PTs (see 3.6.3), but higher than values of established, standardized methods (see 3.6.2). The quotient $U_{(Xpt)}/\sigma_{pt'}$ was 0,94 and increased too. The comparability of results is limited.

62% of results were in the target range.

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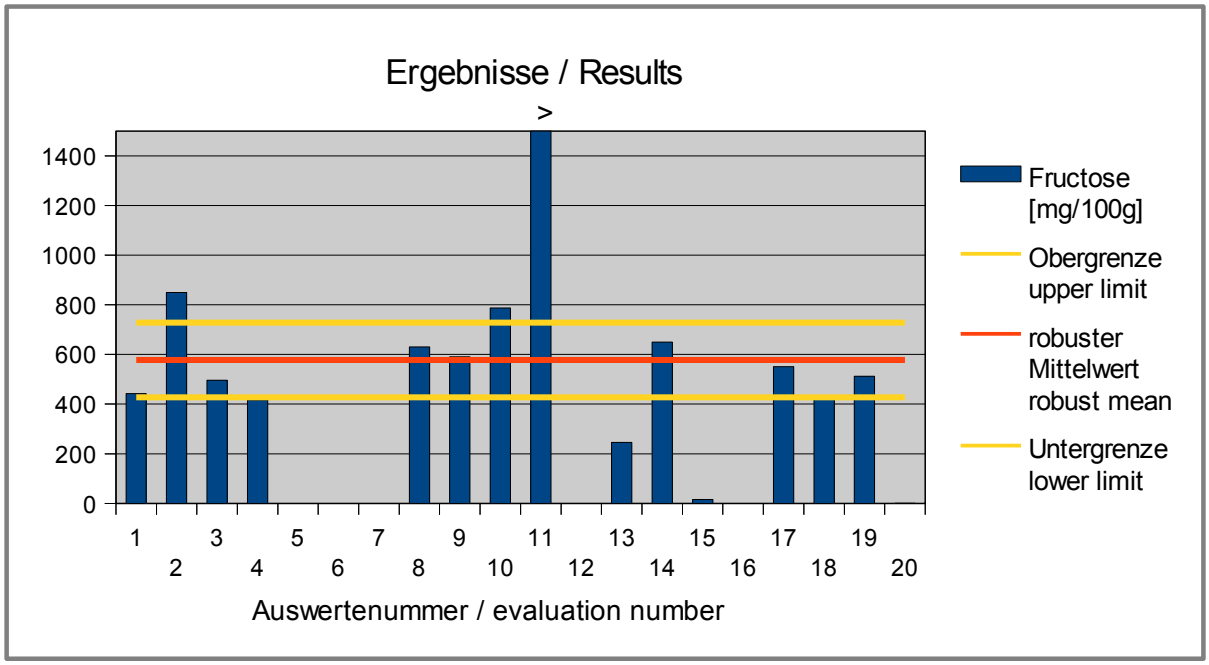

Abb. 1: Ergebnisse Fructose
Fig. 1: Results total fructose

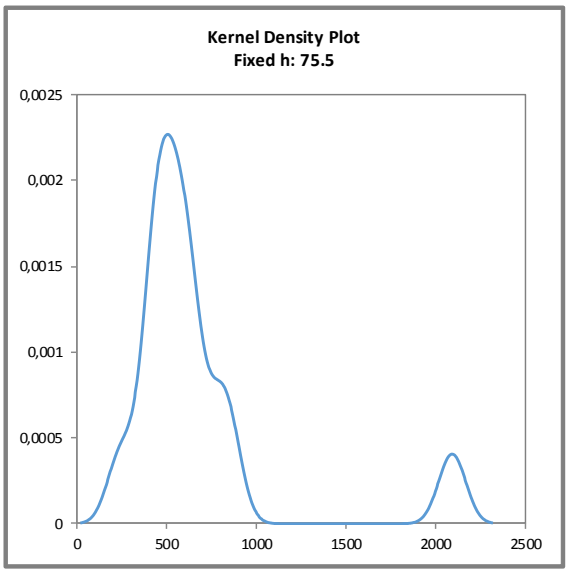

Abb. 2: Kerndichte-Schätzung der Ergebnisse für Fructose Probe A
(mit h = σ_{pt} von X_{pt})

Fig. 2: Kernel density plot of fructose results sample A
(with h = σ_{pt} von X_{pt})

Comments:
The kernel density estimation shows nearly a normal distribution with two shoulders at approximately 250 mg/100g and 800 mg/100g. The peak at approximately 2000 mg/100g is caused by an outlier (s. fig. 2).

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Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Fructose [mg/100g]	Abweichung [mg/100g] Deviation [mg/100g]	z'-Score ($\sigma_{pt'}$)	z-Score (Info)	Hinweis Remark
1	442	-136	-1,8	-9,1	
2	850	272	3,6	18,2	
3	496	-82	-1,1	-5,5	
4	417	-161	-2,1	-10,7	
8	630	52	0,7	3,5	
9	590,7	13	0,2	0,9	
10	787	209	2,8	14,0	
11	2090	1512	20,0	101,1	Ausreisser / Outlier
13	246,1	-332	-4,4	-22,2	
14	650	72	1,0	4,8	
15	15,8 *				
17	551	-27	-0,4	-1,8	
18	430	-148	-2,0	-9,9	
19	512,05	-66	-0,9	-4,4	
20	0,8 *				

* data excluded

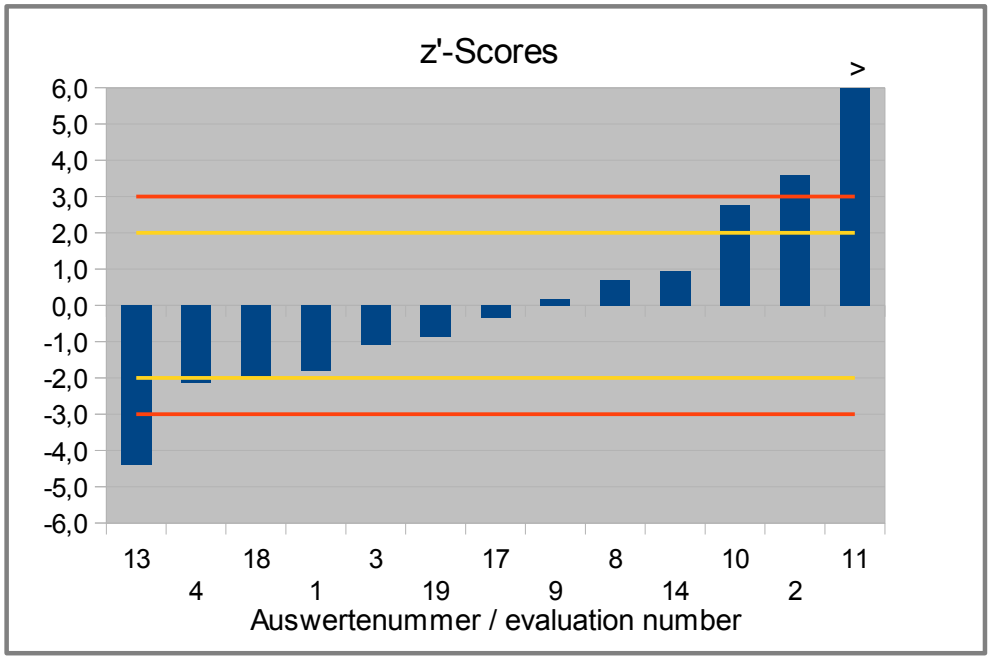

Abb. 3: Z'-Scores Fructose
Fig. 3: Z'-Scores total fructose

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4.1.2 Fructose Sample B (in mg/100g)

Vergleichsuntersuchung / Proficiency Test

Statistic Data	All Meth.	Enzyme Meth.	Other
Number of results	14	6	8
Number of outliers	1	1	0
Mean	903	1210	670
Median	789	870	649
Robust Mean (X)	**768**	**880**	**660**
Robust standard deviation (S*)	**200**	**105**	**187**
Target range:			
Target standard deviation *σpt'*		**64,5**	**87,1**
Target standard deviation (for Information)		22,8	17,1
lower limit of target range		**751**	**486**
upper limit of target range		**1010**	**834**
Quotient S/$\sigma_{pt'}$*		*1,6*	*2,1*
Standard uncertainty $U_{(Xpt)}$		*53,5*	*82,5*
Quotient $U_{(Xpt)}/\sigma_{pt'}$		*0,83*	*0,95*
Results in the target range		*5*	*6*
Percent in the target range		*83%*	*75%*

Comments to the statistic data:

The results of all methods showed an increased variability with two summits of distribution (see fig. 5 kernel density estimation). For this reason evaluation was not done across the methods, but separately for enzymatic methods and other methods.

The target standard deviation was calculated according to the model of Horwitz.

Valuations were done considering the standard uncertainty by z'-score. The quotients $S^*/\sigma_{pt'}$ were 1,6 and 2,1, respectively. The robust standard deviations were in the range of previous PTs (see 3.6.3), but higher than values of established, standardized methods (see 3.6.2). The quotients $U_{(Xpt)}/\sigma_{pt'}$ were 0,83 and 0,95 and increased too. The comparability of results across the methods is limited.

83% and 75% of results were in the target range, respectively.

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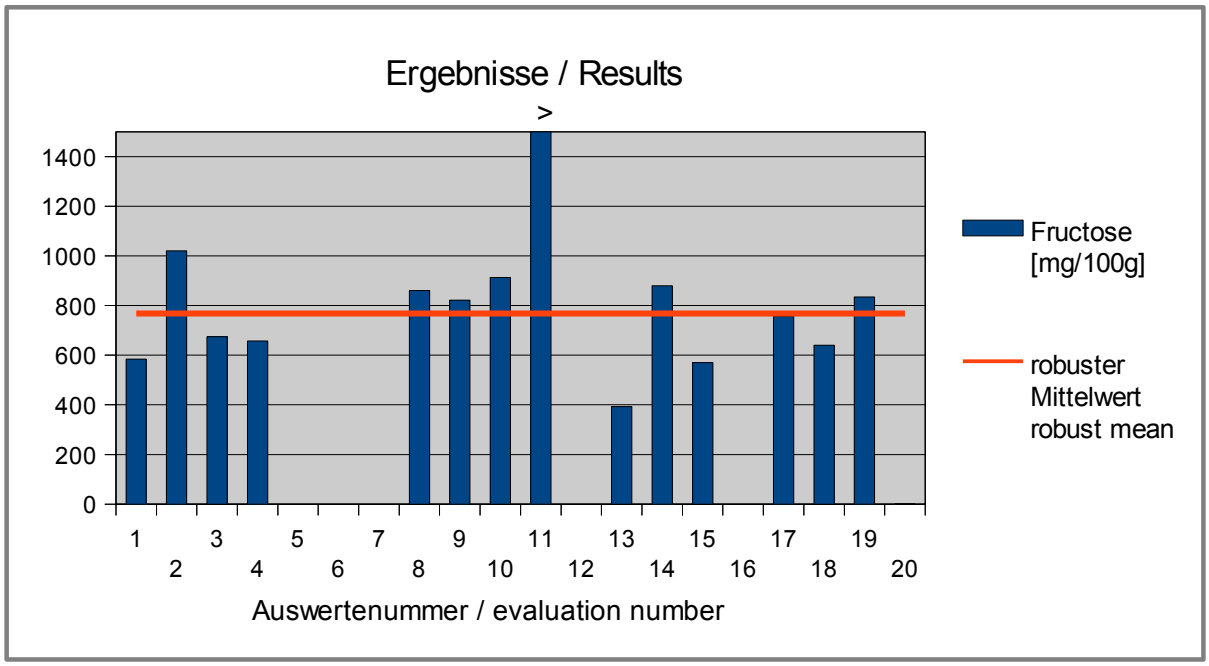

Abb. 4: Ergebnisse Fructose - alle Methoden
Fig. 4: Results total fructose - all methods

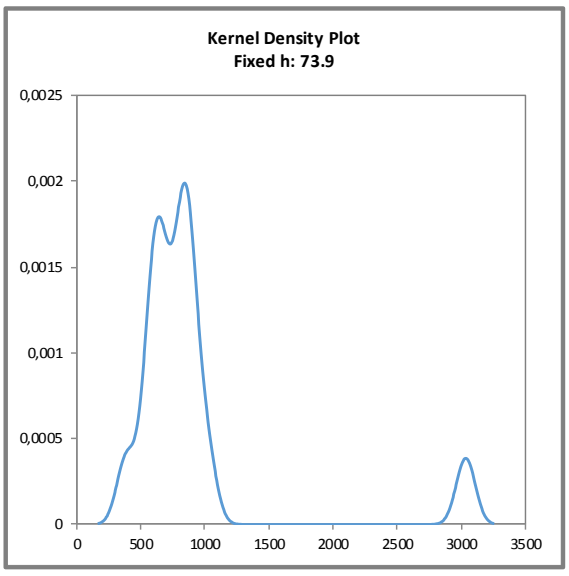

Abb. 5: Kerndichte-Schätzung der Ergebnisse aller Methoden für Fructose Probe B (mit h = σ_{pt} von X_{pt})

Fig. 5: Kernel density plot of fructose results of all methods sample B (with h = σ_{pt} von X_{pt})

Comments:
The kernel density estimation of all methods shows two summits of distribution and a shoulder at approximately 400 mg/100g. The peak at approximately 3000 mg/100g is caused by an outlier (s. fig. 5).

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Ergebnisse der Teilnehmer - enzymatische Methoden:
Results of Participants - Enzyme Methods:

Auswerte-nummer Evaluation number	Fructose [mg/100g]	Abweichung [mg/100g] Deviation [mg/100g]	z'-Score ($\sigma_{pt'}$)	z-Score (Info)	Hinweis Remark
8	860	-20	-0,3	-0,9	
10	913	33	0,5	1,4	
11	3030	2150	33,4	94,3	Ausreisser / Outlier
14	880	0	0,0	0,0	
17	757	-123	-1,9	-5,4	
19	834,6	-45	-0,7	-2,0	

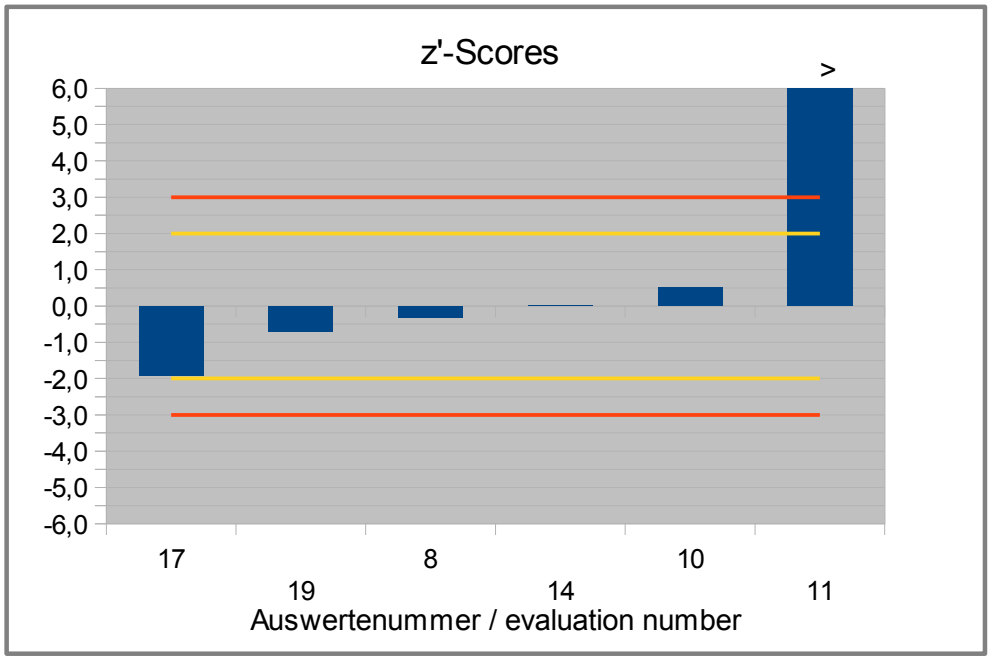

Abb. 6: Z'-Scores Fructose - enzymatische Methoden
Fig. 6: Z'-Scores total fructose - enzyme methods

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Ergebnisse der Teilnehmer - andere Methoden (IC, HPLC, GC):
Results of Participants - other Methods (IC, HPLC, GC):

Auswerte-nummer Evaluation number	Fructose [mg/100g]	Abweichung [mg/100g] Deviation [mg/100g]	z'-Score ($\sigma_{pt'}$)	z-Score (Info)	Hinweis Remark
1	584	-76	-0,9	-4,4	
2	1020	360	4,1	21,1	
3	675	15	0,2	0,9	
4	657	-3	0,0	-0,2	
9	821,9	162	1,9	9,5	
13	392,6	-267	-3,1	-15,6	
15	570	-90	-1,0	-5,3	
18	640	-20	-0,2	-1,2	
20	1,03 *				

* vorab ausgeschlossen

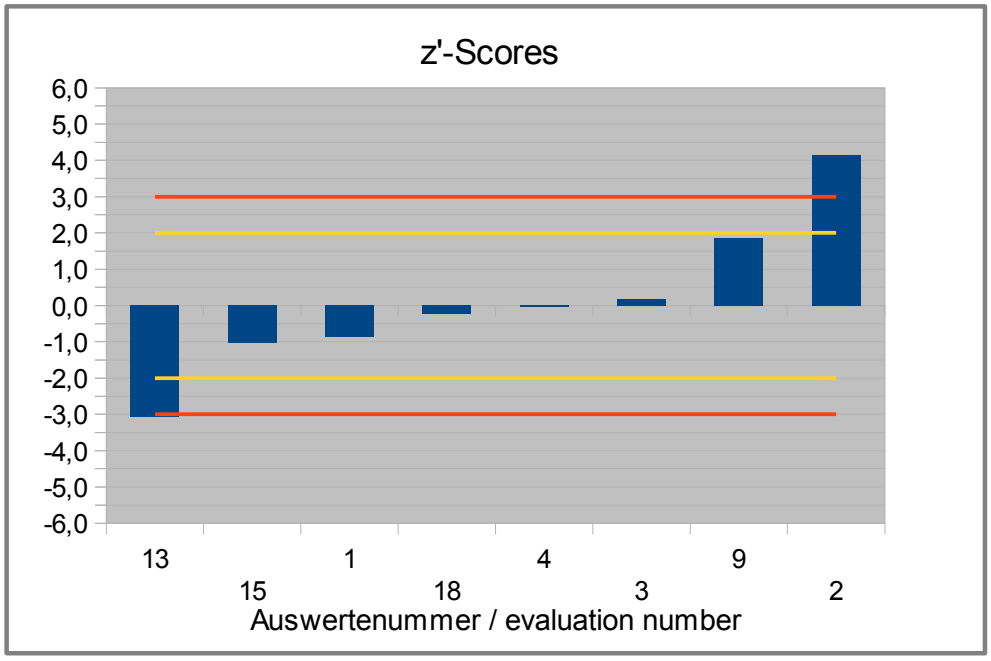

Abb. 7: Z'-Scores Fructose - andere Methoden
Fig. 7: Z'-Scores total fructose - other methods

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4.1.3 Fructose Spiking Material Sample (in g/100g)

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	15
Number of outliers	1
Mean	6,94
Median	7,22
Robust Mean (X)	**7,16**
Robust standard deviation (S*)	**0,788**
Target range:	
Target standard deviation *σpt'*	**0,332**
Target standard deviation (for Information)	0,167
lower limit of target range	**6,50**
upper limit of target range	**7,82**
Quotient S/$\sigma_{pt'}$*	*2,4*
Standard uncertainty $U_{(Xpt)}$	*0,254*
Quotient $U_{(Xpt)}/\sigma_{pt'}$	*0,77*
Results in the target range	*10*
Percent in the target range	*67%*

Comments to the statistic data:

The target standard deviation was calculated according to the model of Horwitz.

The distribution of results showed an increased variability. Valuation was done considering the standard uncertainty by z'-score. The quotient $S^*/\sigma_{pt'}$ was above 2,0. The robust standard deviation was in the range of previous PTs (see 3.6.3), but higher than values of established, standardized methods (see 3.6.2). The quotient $U_{(Xpt)}/\sigma_{pt'}$ was 0,77 and increased too. The comparability of results is limited.

67% of results were in the target range.

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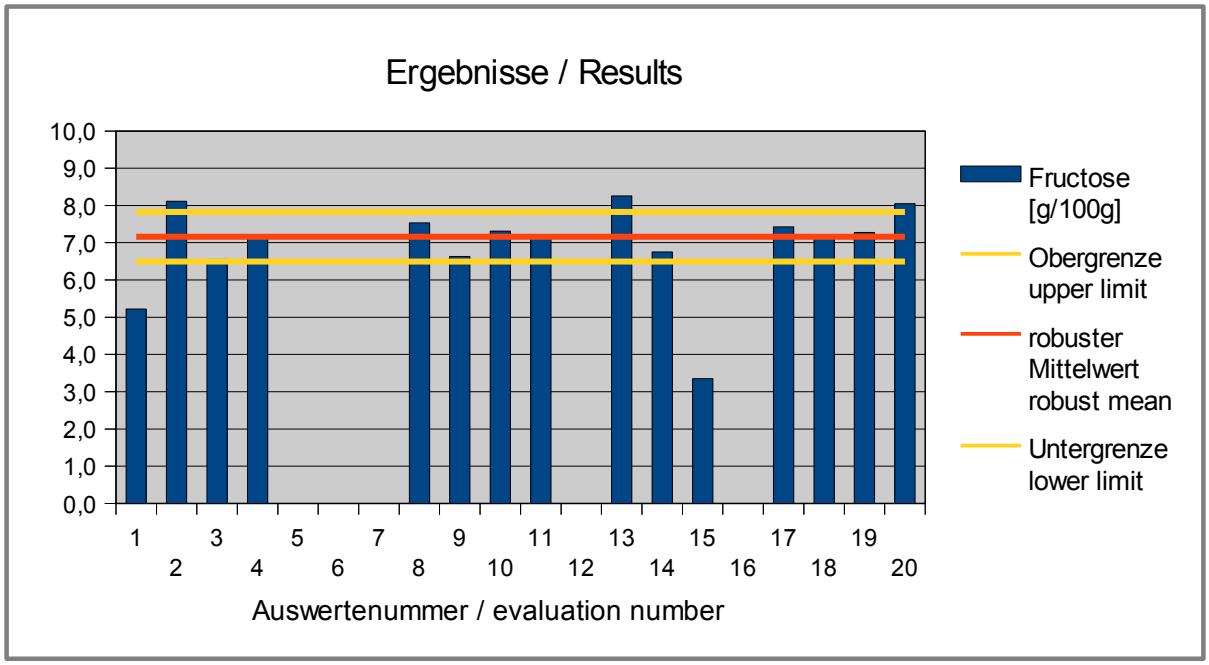

Abb. 8: Ergebnisse Fructose
Fig. 8: Results total fructose

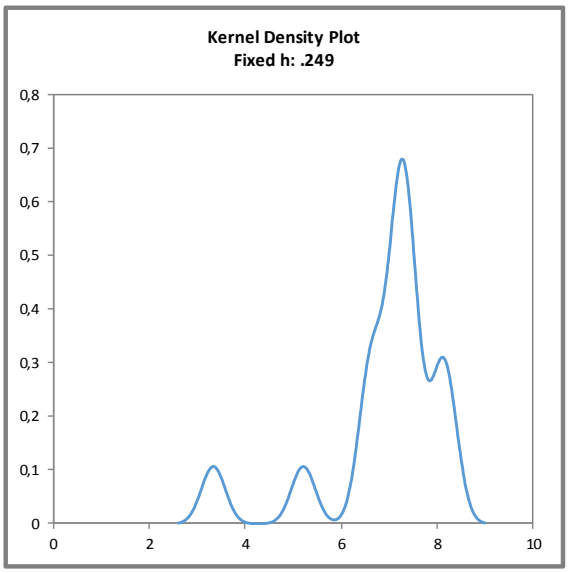

Abb. 9: Kerndichte-Schätzung der Ergebnisse für Fructose Dotierungsmaterialprobe (mit h = 0,75 x *σpt* von Xpt)

Fig. 9: Kernel density plot of fructose results spiking material spiking sample (with h = 0,75 x *σpt* von Xpt)

Comments:
The kernel density estimation shows nearly a normal distribution with two shoulders at approximately 6,6 g/100g and 8,1 g/100g. The two peaks at approximately 3,4 and 5,2 g/100g are caused by single values (s. fig. 9).

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Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Fructose [g/100g]	Abweichung [g/100g] Deviation [g/100g]	z'-Score ($\sigma_{pt'}$)	z-Score (Info)	Hinweis Remark
1	5,22	-1,941	-5,8	-11,6	
2	8,11	0,949	2,9	5,7	
3	6,55	-0,611	-1,8	-3,7	
4	7,132 *	-0,029	-0,1	-0,2	
8	7,53	0,369	1,1	2,2	
9	6,63	-0,531	-1,6	-3,2	
10	7,31	0,149	0,4	0,9	
11	7,21	0,049	0,1	0,3	
13	8,26	1,099	3,3	6,6	
14	6,75	-0,411	-1,2	-2,5	
15	3,35	-3,811	-11,5	-22,8	Ausreisser / Outlier
17	7,429	0,268	0,8	1,6	
18	7,22	0,059	0,2	0,4	
19	7,2765 *	0,115	0,3	0,7	
20	8,05	0,889	2,7	5,3	

* Einheiten von DLA korrigiert

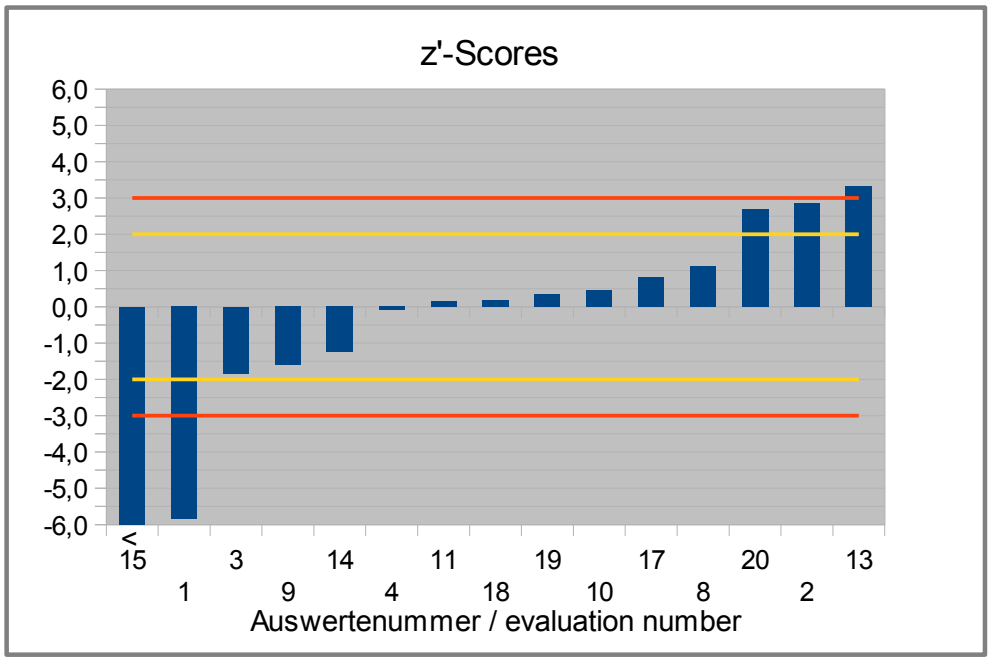

Abb. 10: Z'-Scores Fructose
Fig. 10: Z'-Scores total fructose

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4.2 Lactose

4.2.1 Qualitative Valuation Sample A and Sample B

Vergleichsuntersuchung / Proficiency Test

Evaluation number	Sample A	Sample A	Sample B	Sample B	Qualitative Valuation	Remarks
	pos/neg	[mg/kg]	pos/neg	[mg/kg]	Agreement with Consensus Value	
1	negative	n.n.	positive	116	1/1 (100%)	
2	positive	2510	positive	2800	1/1 (100%)	
3	negative	<	positive	148,2	1/1 (100%)	
4	negative	-	positive	149	1/1 (100%)	
5	negative	< 10	positive	174	1/1 (100%)	
6	negative	n.d.	positive	146	1/1 (100%)	
7	positive	1,6	positive	242,8	1/1 (100%)	
8	negative	<10	positive	146	1/1 (100%)	
9	negative	< 10	positive	158,5	1/1 (100%)	
10	negative	n.d.	positive	145	1/1 (100%)	
11	positive	20	positive	430	1/1 (100%)	
12	positive	22	positive	159	1/1 (100%)	
13	negative	<10	positive	126,6	1/1 (100%)	
14	negative	<4	positive	134	1/1 (100%)	
15	positive	18,6	positive	232	1/1 (100%)	
16	negative	<3.55	positive	9,77	1/1 (100%)	
17	negative	<LOQ	positive	138	1/1 (100%)	
18	negative	<5	positive	150	1/1 (100%)	
20	negative	<0,01	positive	0,12	1/1 (100%)	

	Sample A		Sample B		
Number positive	5		19		
Number negative	14		0		
Percent positive	26		100		
Percent negative	74		0		
Consensus	none		positive		

Comments:

There were 100% positive results for sample B. For sample A no consensus value of > 75% was obtained. For sample A all results except one were < 25 mg/100g and thereof 9 results < 10 mg/100g. 5 out of 19 participants stated lactose was detectable in sample A.

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4.2.2 Lactose Sample B (in mg/100g)

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	16
Number of outliers	2
Mean	175
Median	149
Robust Mean (X)	**154**
Robust standard deviation (S*)	**26,7**
Target range:	
Target standard deviation σ_{pt}'	**16,3**
Target standard deviation (for Information)	8,18
lower limit of target range	**122**
upper limit of target range	**187**
Quotient S/σ_{pt}'*	*1,6*
Standard uncertainty $U_{(Xpt)}$	*8,33*
Quotient $U_{(Xpt)}/\sigma_{pt}$'	*0,51*
Results in the target range	*12*
Percent in the target range	*75%*

Comments to the statistic data:

The target standard deviation was calculated according to 3.4.2 value by precision experiment (ASU §64 L 01.00-90).

The distribution of results showed an increased variability. Valuation was done considering the standard uncertainty by z'-score. The quotient S*/σ_{pt}' was below 2,0 then. The robust standard deviation was in the range of previous PTs (see 3.6.3), but higher than values of established, standardized methods (see 3.6.2). The quotient $U_{(Xpt)}/\sigma_{pt}$' was 0,51 and increased. The comparability of results is given using the z'-score valuation.

75% of results were in the target range.

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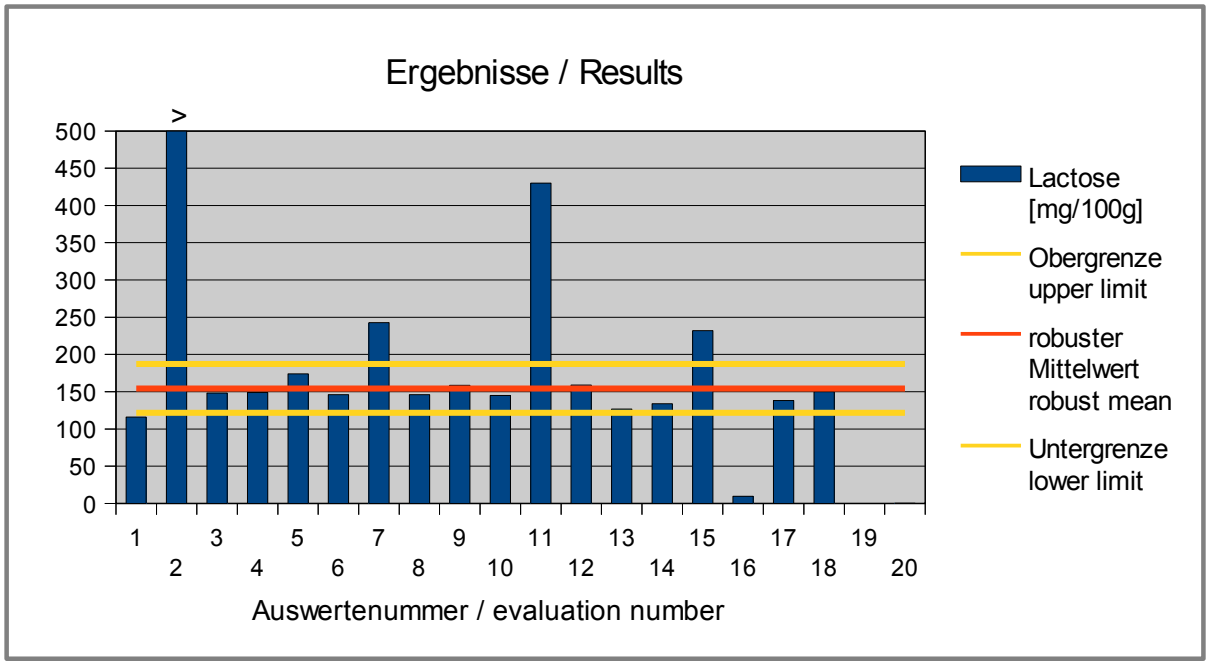

Abb. 11: Ergebnisse Lactose
Fig. 11: Results total lactose

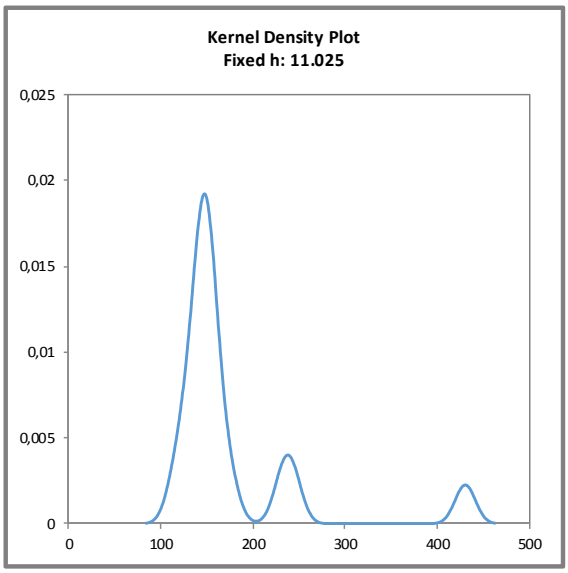

Abb. 12: Kerndichte-Schätzung der Ergebnisse für Lactose Probe B
(mit h = 0,75 x *σpt* von Xpt; *σpt* = 14,7)

Fig. 12: Kernel density plot of lactose results sample B (with h = 0,75 x *σpt* von Xpt; *σpt* = 14,7)

Comments:
The kernel density estimation shows a normal distribution with two peaks at approximately 240 mg/100g und 430 mg/100g caused by three single values including two outliers (s. fig. 12).

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Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Lactose [mg/100g]	Abweichung [mg/100g] Deviation [mg/100g]	z'-Score ($\sigma_{pt'}$)	z-Score (Info)	Hinweis Remark
1	116	-38,45	-2,4	-4,7	
2	2800 *				
3	148,2	-6,25	-0,4	-0,8	
4	149	-5,45	-0,3	-0,7	
5	174	19,55	1,2	2,4	
6	146	-8,45	-0,5	-1,0	
7	242,8	88,35	5,4	10,8	Ausreisser / Outlier
8	146	-8,45	-0,5	-1,0	
9	158,5	4,05	0,2	0,5	
10	145	-9,45	-0,6	-1,2	
11	430	275,55	16,9	33,7	Ausreisser / Outlier
12	159	4,55	0,3	0,6	
13	126,6	-27,85	-1,7	-3,4	
14	134	-20,45	-1,3	-2,5	
15	232	77,55	4,7	9,5	
16	9,77 *				
17	138	-16,45	-1,0	-2,0	
18	150	-4,45	-0,3	-0,5	
20	0,12 *				

* data excluded

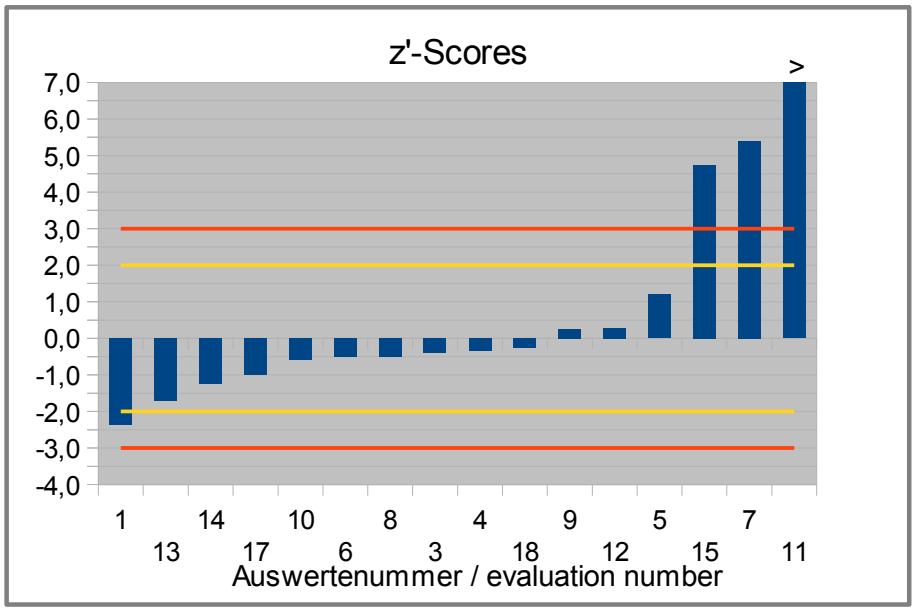

Abb. 13: Z'-Scores Lactose
Fig. 13: Z'-Scores total lactose

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4.2.3 Lactose Spiking Material Sample (in g/100g)

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	16
Number of outliers	0
Mean	3,96
Median	4,03
Robust Mean (X)	**3,98**
Robust standard deviation (S*)	**0,354**
Target range:	
Target standard deviation σ_{pt}'	**0,170**
Target standard deviation (for Information)	0,362
lower limit of target range	**3,64**
upper limit of target range	**4,32**
Quotient S/σ_{pt}'*	*2,1*
Standard uncertainty $U_{(Xpt)}$	*0,111*
Quotient $U_{(Xpt)}/\sigma_{pt}$'	*0,65*
Results in the target range	*12*
Percent in the target range	*75%*

Comments to the statistic data:

The target standard deviation was calculated according to the model of Horwitz.

The distribution of results showed an increased variability. Valuation was done considering the standard uncertainty by z'-score. The quotient S*/σ_{pt}' was 2,1 then. The robust standard deviation was in the range of previous PTs (see 3.6.3), but higher than values of established, standardized methods (see 3.6.2). The quotient $U_{(Xpt)}/\sigma_{pt}$' was 0,65 and increased. The comparability of results is fair using the z'-score evaluation.

75% of results were in the target range.

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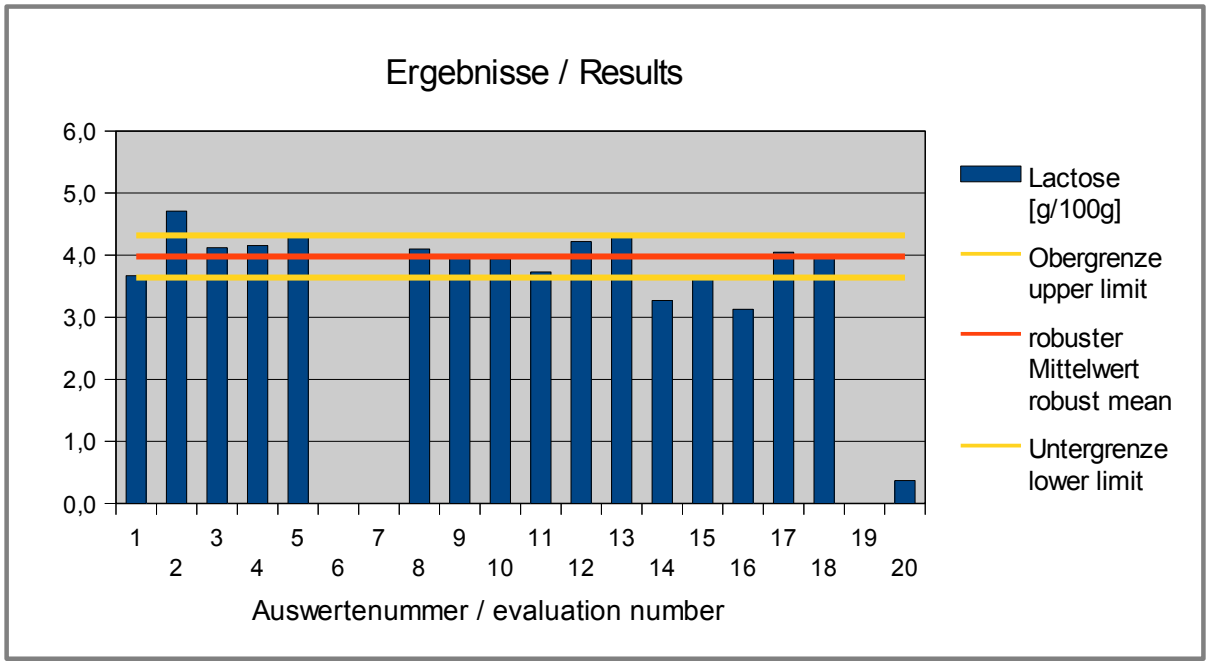

Abb. 14: Ergebnisse Lactose
Fig. 14: Results total Lactose

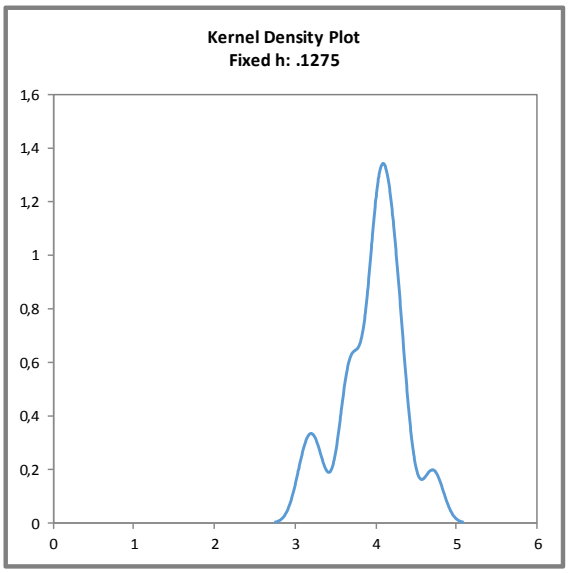

Abb. 15: Kerndichte-Schätzung der Ergebnisse für Lactose Dotierungsmaterialprobe (mit h = 0,75 x σ_{pt} von X_{pt})

Fig. 15: Kernel density plot of lactose results spiking material spiking sample (with h = 0,75 x σ_{pt} von X_{pt})

Comments:
The kernel density estimation shows nearly a normal distribution with a shoulder at approximately 3,7 g/100g and two peaks at approximately 3,2 g/100g and 4,7 g/100g (s. fig. 15).

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Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Lactose [g/100g]	Abweichung [g/100g] Deviation [g/100g]	z'-Score ($\sigma_{pt'}$)	z-Score (Info)	Hinweis Remark
1	3,67	-0,31	-1,8	-0,9	
2	4,71	0,73	4,3	2,0	
3	4,12	0,14	0,8	0,4	
4	4,155	0,18	1,0	0,5	error of units corrected by DLA
5	4,3	0,32	1,9	0,9	
8	4,1	0,12	0,7	0,3	
9	3,98	0,00	0,0	0,0	
10	3,94	-0,04	-0,2	-0,1	
11	3,73	-0,25	-1,5	-0,7	
12	4,22	0,24	1,4	0,7	
13	4,32	0,34	2,0	0,9	
14	3,27	-0,71	-4,2	-2,0	
15	3,65	-0,33	-1,9	-0,9	
16	3,127	-0,85	-5,0	-2,4	
17	4,048	0,07	0,4	0,2	
18	4,01	0,03	0,2	0,1	
20	0,37 *				error of units?

* vorab ausgeschlossen

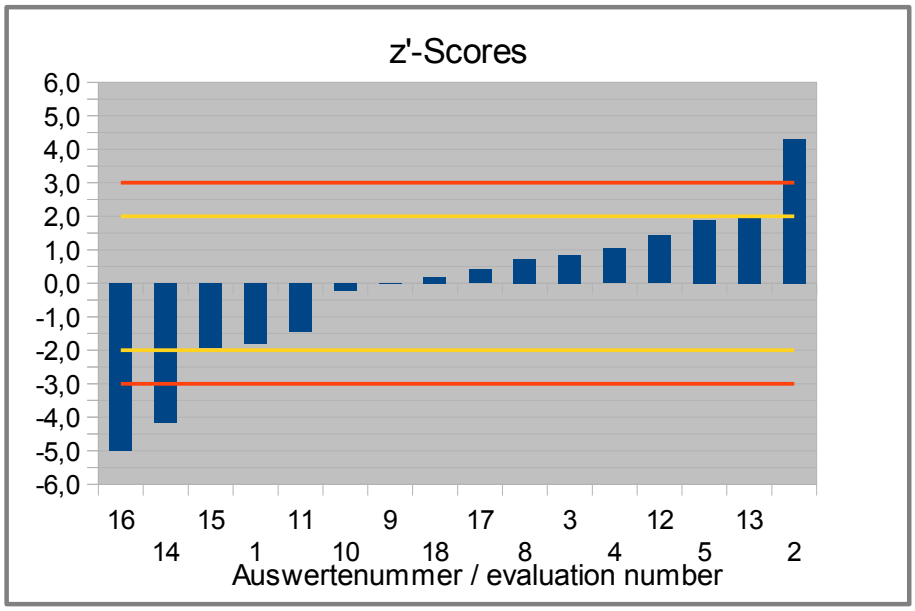

Abb. 16: Z'-Scores Lactose
Fig. 16: Z'-Scores total lactose

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4.2.4 Recovery Rates for Lactose

Hereafter the recovery rates of the participants' results with respect to the level of addition (page 4, table 2) were calculated by DLA and given for information only.

Spiking Material Sample and Sample B

Evaluation number	Spiking material	Recovery rate	Sample B	Recovery rate	Remarks
	[g/100g]	[%]	[mg/100g]	[%]	
1	3,67	**87**	116	79	
2	4,71	**112**	2800	1905	
3	4,12	**98**	148,2	**101**	
4	4,16	**98**	149	**101**	spiking sample: error of units corrected by DLA
5	4,3	**102**	174	118	
6			146	**99**	
7			242,8	165	
8	4,1	**97**	146	**99**	
9	3,98	**95**	158,5	**108**	
10	3,94	**94**	145	**99**	
11	3,73	**89**	430	293	
12	4,22	**100**	159	**108**	
13	4,32	**103**	126,6	**86**	
14	3,27	78	134	**91**	
15	3,65	**87**	232	158	
16	3,13	74	9,77	7	
17	4,05	**96**	138	**94**	
18	4,01	**95**	150	**102**	
20	0,37	9	0,12	0	error of units?

RA*	85-115 %	RA*	85-115 %
Number in RA	**14**	Number in RA	**11**
Percent in RA	**82**	Percent in RA	**58**

* Range of acceptance see 3.9

Comments:

For the spiking material sample 82% of the participants obtained a recovery rate within the range of 85-115%. For the spiked sample B produced with the spiking material sample 58% of the recovery rates were in this range. Considering possible lactose content of the matrix at the limits of determination of the methods (see 4.2.1 sample A), the level of lactose could be appr. 10-25 mg increased. Therefore the recovery rate of the participant 5 for sample B could be assessed acceptable too.

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4.3 Galactose

4.3.1 Galactose Sample A (in mg/100g)

Vergleichsuntersuchung / Proficiency Test

Due to the low number of results <7 no statistical evaluation was done.

Statistic Data	
Number of results	6
Number of outliers	1
Mean	36,0
Median	15,5
Robust Mean (X)	**18,8**
Robust standard deviation (S*)	**18,6**
Target range:	
Target standard deviation σ_{pt}'	
Target standard deviation (for Information)	
lower limit of target range	
upper limit of target range	
Quotient S*/σ_{pt}'	
Standard uncertainty $U_{(Xpt)}$	
Quotient $U_{(Xpt)}/\sigma_{pt}$'	
Results in the target range	
Percent in the target range	

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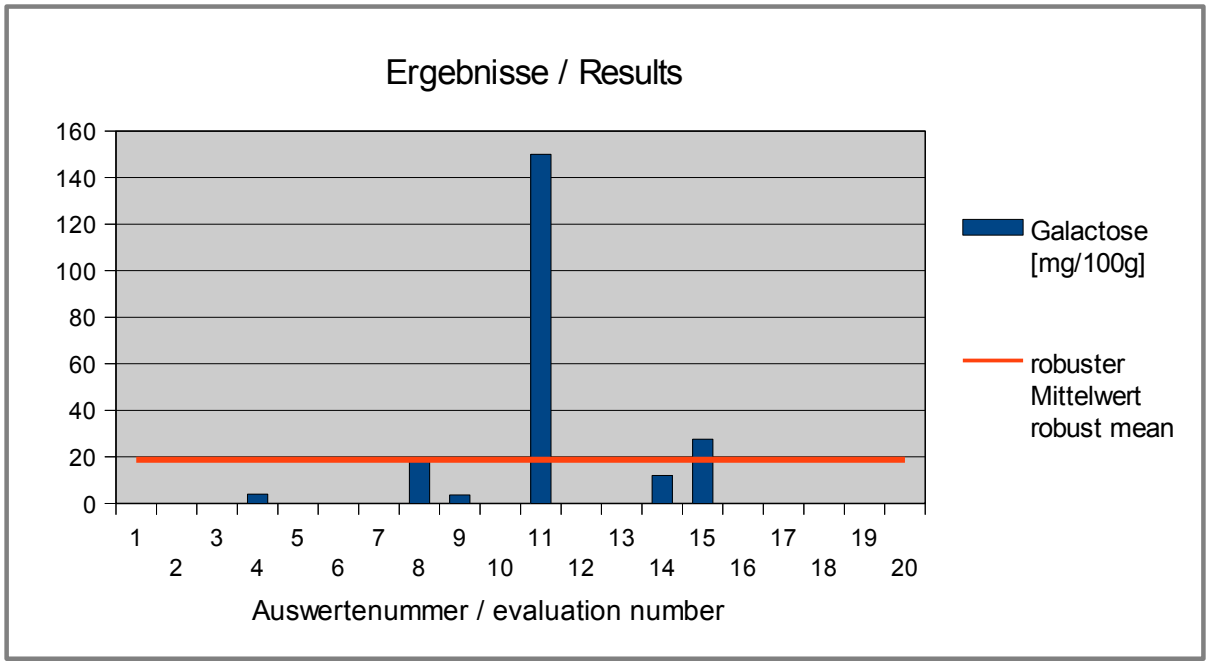

Abb. 17: Ergebnisse Galactose
Fig. 17: Results total galactose

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Galactose [mg/100g]	Abweichung [mg/100g] Deviation [mg/100g]	z'-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
4	4	-14,79			
5	< 10 *				
6	n.a. *				
8	19	0,21			
9	3,6	-15,19			
10	n.a. *				
11	150	131,21			
14	12	-6,79			
15	27,6	8,81			
17	< LOQ *				
18	< 100 *				
20	< 0,01 *				

* data excluded

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4.3.2 Galactose Sample B (in mg/100g)

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	7
Number of outliers	1
Mean	24,3
Median	12,1
Robust Mean (X)	**14,6**
Robust standard deviation (S*)	**11,9**
Target range:	
Target standard deviation σ_{pt}'	**5,71**
Target standard deviation (for Information)	1,10
lower limit of target range	**3,18**
upper limit of target range	**26,0**
Quotient S/σ_{pt}'*	*2,1*
Standard uncertainty $U_{(Xpt)}$	*5,60*
Quotient $U_{(Xpt)}/\sigma_{pt}$'	*0,98*
Results in the target range	*6*
Percent in the target range	*86%*

Comments to the statistic data:

The target standard deviation was calculated according to the model of Horwitz.

The distribution of results showed an increased variability (due to low number of results no kernel density is shown). Valuation was done considering the standard uncertainty by z'-score. The quotient S*/σ_{pt}' was 2,1 then. The robust standard deviation is increased. The quotient $U_{(Xpt)}/\sigma_{pt}$' was 0,65 and increased too. The comparability of results is limited.

86% of results were in the target range.

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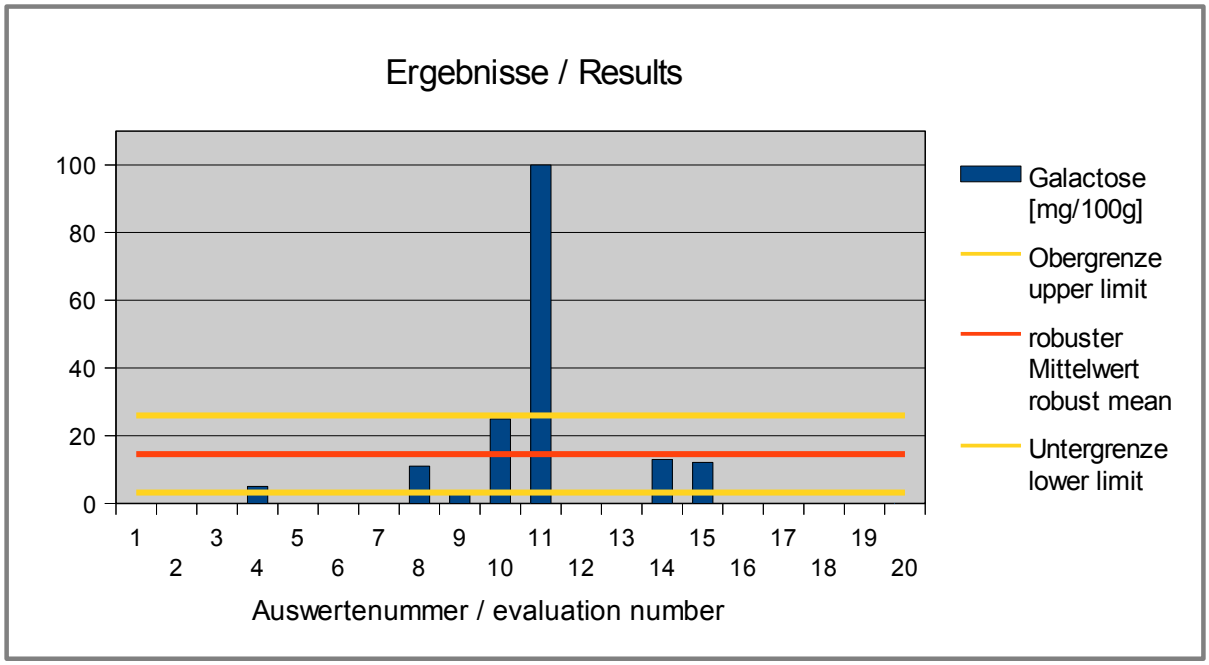

Abb. 18: Ergebnisse Galactose
Fig. 18: Results total galactose

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Galactose [mg/100g]	Abweichung [mg/100g] Deviation [mg/100g]	z'-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
4	5	-9,59	-1,7	-8,7	
5	< 10 *				
6	n.a. *				
8	11	-3,59	-0,6	-3,3	
9	3,7	-10,89	-1,9	-9,9	
10	25	10,41	1,8	9,4	
11	100	85,41	15,0	77,5	Ausreisser / Outlier
14	13	-1,59	-0,3	-1,4	
15	12,1	-2,49	-0,4	-2,3	
17	< LOQ *				
18	< 100 *				
20	0,01 *				error of units?

* data excluded

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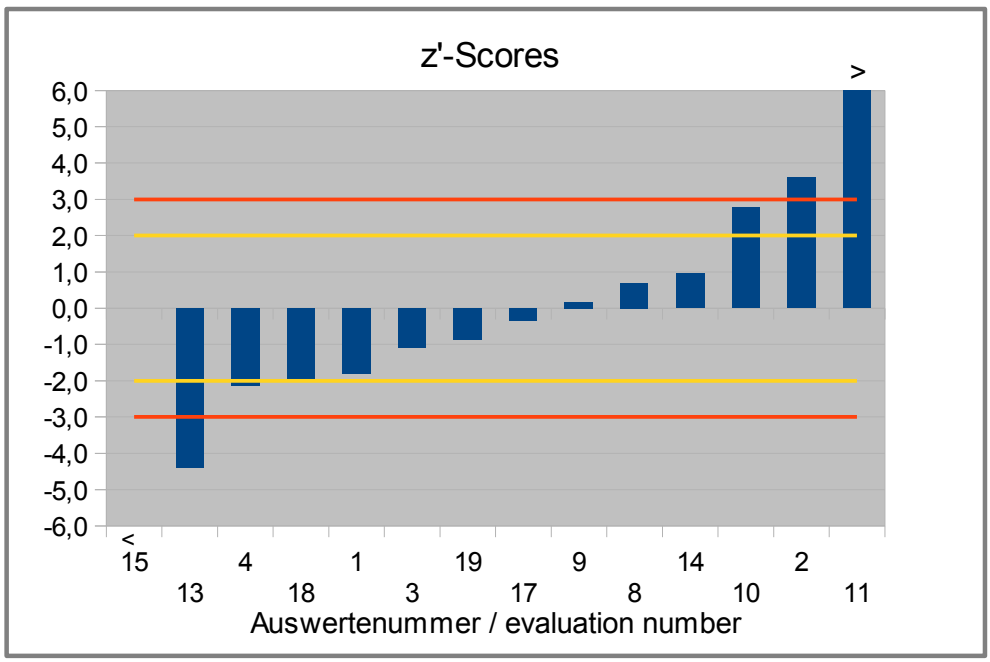

Abb. 19: Z'-Scores Galactose
Fig. 19: Z'-Scores total galactose

4.3.3 Galactose Spiking Material Sample (in g/100g)

Vergleichsuntersuchung / Proficiency Test

There were no positive and quantitative results.
Further details are given in the documentation.

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5. Documentation

5.1 Primary data

Parameter	Teilnehmer	Einheit	Proben-Nr.	Datum d. Analyse	Nach-weisbar	NWG	BG	Ergebnis	Inkl. WF	Wiederfin-dungsrate [%]	WF mit gleicher Matrix
Analyte	**Participant**	**Unit**	**Sample-No.**	**Date of ana-lysis**	**Detecta-ble**	**LOD**	**LOQ**	**Result**	**Incl. RR**	**Recovery rate [%]**	**RR with same Matrix**
Fructose Probe A / Sample A	1	mg/100g	12	9.+23.05.	yes	3	10	442	no		
	2	mg/100g	57		yes			850			
	3	mg/100g	34	17/05	yes		10	496			
	4	mg/100g	2	17/5	yes			417			
	5	mg/100g	25								
	6	mg/100g	21								
	7	mg/100g	36								
	8	mg/100g	24	4.5.	yes			630			
	9	mg/100g	42	21.4.16	yes	3	10	590,7	no		no
	10	mg/100g	32				40	787	no	107	yes
	11	mg/100g	14	13.05.2016	yes			2090	yes	100	no
	12	mg/100g	9	NA							
	13	mg/100g	45				100	246,1	no		
	14	mg/100g	54		yes		100	650	no		
	15	mg/100g	5	27.05.	yes	5	10	15,8	no		
	16	mg/100g	13+51								
	17	mg/100g	11	12.05.16	yes	10	30	551	no		
	18	mg/100g	26	25/04	yes		0	430	no		
	19	mg/100g	30	25/05	yes			512,05		101	
	20	mg/100g	48	24.06.16	yes	0,05	0,1	0,8	no	-	-

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Parameter	Teilnehmer	Einheit	Proben-Nr.	Datum d. Analyse	Nach-weisbar	NWG	BG	Ergebnis	Inkl. WF	Wiederfin-dungsrate [%]	WF mit gleicher Matrix
Analyte	**Participant**	**Unit**	**Sample-No.**	**Date of ana-lysis**	**Detecta-ble**	**LOD**	**LOQ**	**Result**	**Incl. RR**	**Recovery rate [%]**	**RR with same Matrix**
Fructose Probe B / Sample B	1	mg/100g	38	9.+23.05.	yes	3	10	584	no		
	2	mg/100g	22		yes			1020			
	3	mg/100g	52	17/05	yes		10	675			
	4	mg/100g		17/5	yes			657			
	5	mg/100g	51								
	6	mg/100g	57								
	7	mg/100g									
	8	mg/100g	3	4.5.	yes			860			
	9	mg/100g		21.4.16	yes	3	10	821,9	no		no
	10	mg/100g	60				40	913	no		
	11	mg/100g	1	13.05.2016	yes			3030	yes	100	no
	12	mg/100g		NA							
	13	mg/100g	35				100	392,6	no		
	14	mg/100g	18		yes		100	880	no		
	15	mg/100g	27	27.05.	yes	5	10	570	no		
	16	mg/100g									
	17	mg/100g		12.05.16	yes	10	30	757	no		
	18	mg/100g		25/04	yes		0	640	no		
	19	mg/100g		25/05	yes			834,6		101	
	20	mg/100g	11	24.06.16	yes	0,05	0,1	1,03	no	-	-

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Parameter	Teilnehmer	Einheit	Proben-Nr.	Datum d. Analyse	Nach-weisbar	NWG	BG	Ergebnis	Inkl. WF	Wiederfin-dungsrate [%]	WF mit gleicher Matrix
Analyte	**Participant**	**Unit**	**Sample-No.**	**Date of ana-lysis**	**Detecta-ble**	**LOD**	**LOQ**	**Result**	**Incl. RR**	**Recovery rate [%]**	**RR with same Matrix**
Fructose Dotierungs-materialprobe / Spiking Material Sample	1	g/100g	10	9.+23.05.	yes	0	0,01	5,22	no		
	2	g/100g	3		yes			8,11			
	3	g/100g	26	17/05	yes		0,01	6,55			
	4	g/100g		17/5	yes			7132			
	5	g/100g	43								
	6	g/100g									
	7	g/100g									
	8	g/100g	19	4.5.	yes			7,53			
	9	g/100g		21.4.16	yes	3	10	6,63	no		no
	10	g/100g	17				0,4	7,31	no		
	11	g/100g	42	13.05.2016	yes			7,21	yes	100	no
	12	g/100g		NA							
	13	g/100g	16				0,5	8,26	no		
	14	g/100g	36		yes		0,1	6,75	no	-	-
	15	g/100g	7	27.05.	yes	5	10	3,35	no		
	16	g/100g									
	17	g/100g		12.05.16	yes	10	30	7,43	no		
	18	g/100g		25/04	yes		0	7,22	no		
	19	g/100g		25/05	yes			7276,5		101	
	20	g/100g	32	24.06.16	yes	0,05	0,1	8,05	no	-	-

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Parameter	Teilnehmer	Einheit	Proben-Nr.	Datum d. Analyse	Nach-weisbar	NWG	BG	Ergebnis	Inkl. WF	Wiederfin-dungsrate [%]	WF mit gleicher Matrix
Analyte	**Participant**	**Unit**	**Sample-No.**	**Date of ana-lysis**	**Detecta-ble**	**LOD**	**LOQ**	**Result**	**Incl. RR**	**Recovery rate [%]**	**RR with same Matrix**
Lactose Probe A / Sample A	1	mg/100g	12	9.+23.05.	no	3	10	n.n.	no		
	2	mg/100g	57		yes			2510			
	3	mg/100g	34	14/05	no		2	<	yes	0,94	yes
	4	mg/100g	2	17/5	no			-			
	5	mg/100g	25	03/05	yes	2	10	< 10	no	100	yes
	6	mg/100g	21	17.05.2016	no	20 mg/100g	100 mg/100g	not detecta-ble			
	7	mg/100g	36	19/05/2016	yes	2,96mg/ L	20mg/kg	16mg/kg	no		no
	8	mg/100g	24	12.5.	yes			<10			
	9	mg/100g	42	21.4.16	no	3	10	< 10	no		no
	10	mg/100g	32	03.05.2016	no		40	n.d.	no	104	yes
	11	mg/100g	14	13.05.2016	yes			20	yes	100	no
	12	mg/100g	9	06 May	yes	0,6 mg/100g	1,8 mg/100g	22	yes	99,1	no
	13	mg/100g	45				10	<10	no		
	14	mg/100g	54	03.05. und 11.05.2016	yes		4	<4	yes	98	yes
	15	mg/100g	5	27.05.	yes	5	10	18,6	no	96	yes
	16	mg/100g	13+51	27-Apr	no	35.5ppm	35.5ppm	<3.55	no		
	17	mg/100g	11	12.05.16	no	10	30	<LOQ	no		
	18	mg/100g	26	25/04	no		0	<5	no		same matrix was assumed as the spiking material
	19	mg/100g	30								
	20	mg/100g	48	24.06.16	no	0,01	0,01	<0,01	no	-	-

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Parameter	Teilnehmer	Einheit	Proben-Nr.	Datum d. Analyse	Nach-weisbar	NWG	BG	Ergebnis	Inkl. WF	Wiederfin-dungsrate [%]	WF mit gleicher Matrix
Analyte	**Participant**	**Unit**	**Sample-No.**	**Date of ana-lysis**	**Detecta-ble**	**LOD**	**LOQ**	**Result**	**Incl. RR**	**Recovery rate [%]**	**RR with same Matrix**
Lactose Probe B / Sample B	1	mg/100g	38	9.+23.05.	yes	3	10	116	no		
	2	mg/100g	22		yes			2800			
	3	mg/100g	52	17/05	yes		10	148,2			
	4	mg/100g	23	17/5	yes			149			
	5	mg/100g	51	03/05	yes	2	10	174	no	83	yes
	6	mg/100g	57	17.05.2016	yes	20 mg/100g	100 mg/100g	146	no		
	7	mg/100g	29	19/05/2016	yes	2,96mg/L	20mg/kg	2428mg/kg	no		no
	8	mg/100g	3	12.5.	yes			146			
	9	mg/100g	30	21.4.16	yes	3	10	158,5	no		no
	10	mg/100g	60	03.05.2016			40	145	no		
	11	mg/100g	1	13.05.2016	yes			430	yes	100	no
	12	mg/100g	49	06 May	yes	0,6 mg/100g	1,8 mg/100g	159	yes	99,1	no
	13	mg/100g	35				10	126,6	no		
	14	mg/100g	18	03.05. und 11.05.2016	yes		4	134	yes	98	yes
	15	mg/100g	27	27.05.	yes	5	10	232	no	96	yes
	16	mg/100g	5 + 46	27-Apr	yes	35.5ppm	35.5ppm	9,77	no		
	17	mg/100g	44	12.05.16	yes	10	30	138	no		
	18	mg/100g	7	25/04	yes		0	150	no		same matrix was assumed as the spiking material
	19	mg/100g	25								
	20	mg/100g	11	24.06.16	yes	0,01	0,01	0,12	no	-	-

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Parameter	Teilnehmer	Einheit	Proben-Nr.	Datum d. Analyse	Nach-weisbar	NWG	BG	Ergebnis	Inkl. WF	Wiederfin-dungsrate [%]	WF mit gleicher Matrix
Analyte	**Participant**	**Unit**	**Sample-No.**	**Date of ana-lysis**	**Detecta-ble**	**LOD**	**LOQ**	**Result**	**Incl. RR**	**Recovery rate** [%]	**RR with same Matrix**
Lactose Dotierungs-materialprobe / Spiking Material Sample	1	g/100g	10	9.+23.05.	yes	0	0,01	3,67	no		
	2	g/100g	3		yes			4,71			
	3	g/100g	26	17/05	yes		0,01	4,12			
	4	g/100g	11	17/5	yes			4155			
	5	g/100g	43	03/05	yes	0,02	0,1	4,3	no		
	6	g/100g	48								
	7	g/100g	40								
	8	g/100g	19	12.5.	yes			4,1			
	9	g/100g	53	21.4.16	yes	3	10	3,98	no		no
	10	g/100g	17	03.05.2016			1,79	3,94	no		
	11	g/100g	42	13.05.2016	yes			3,73	yes	100	no
	12	g/100g	22	06 May	yes	0,6 mg/100g	1,8 mg/100g	4,22	yes	99,1	no
	13	g/100g	16				0,5	4,32	no		
	14	g/100g	36	03.05. und 11.05.2016	yes		0,1	3,27	no	-	-
	15	g/100g	7	27.05.	yes	5	10	3,65	no	96	yes
	16	g/100g	23 + 35	27-Apr	yes	35.5ppm	35.5ppm	3,13	no		
	17	g/100g	29	12.05.16	yes	10	30	4,05	no		
	18	g/100g	14	25/04	yes		0	4,01	yes		not detectable
	19	g/100g	12								
	20	g/100g	32	24.06.16	yes	0,01	0,01	0,37	no	-	-

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Parameter	Teilnehmer	Einheit	Proben-Nr.	Datum d. Analyse	Nach-weisbar	NWG	BG	Ergebnis	Inkl. WF	Wiederfin-dungsrate [%]	WF mit gleicher Matrix
Analyte	**Participant**	**Unit**	**Sample-No.**	**Date of ana-lysis**	**Detecta-ble**	**LOD**	**LOQ**	**Result**	**Incl. RR**	**Recovery rate [%]**	**RR with same Matrix**
Galactose Probe A / Sample A	1	mg/100g	12								
	2	mg/100g	57								
	3	mg/100g	34								
	4	mg/100g	2	17/5	yes			4			
	5	mg/100g	25	03/05	yes	2	10	< 10	no		
	6	mg/100g	21	17.05.2016	no	10 mg/100g	40 mg/100g	not detecta-ble			
	7	mg/100g	36								
	8	mg/100g	24	12.5.	yes			19			
	9	mg/100g	42	21.4.16	yes	1	3	3,6	no		no
	10	mg/100g	32				20	n.d.	no		
	11	mg/100g	14	13.05.2016	yes			150	yes	100	no
	12	mg/100g	9	NA							
	13	mg/100g	45								
	14	mg/100g	54		yes		2	12	yes		yes
	15	mg/100g	5	27.05.	yes	5	10	27,6	no	97	yes
	16	mg/100g	13+51								
	17	mg/100g	11	12.05.16	no	20	60	<LOQ	no		
	18	mg/100g	26	17/05	no		0	<100	yes	104	
	19	mg/100g	30								
	20	mg/100g	48	24.06.16	no	0,01	0,01	<0,01	no	-	-

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Parameter	Teilnehmer	Einheit	Proben-Nr.	Datum d. Analyse	Nach-weisbar	NWG	BG	Ergebnis	Inkl. WF	Wiederfin-dungsrate [%]	WF mit gleicher Matrix
Analyte	**Participant**	**Unit**	**Sample-No.**	**Date of ana-lysis**	**Detecta-ble**	**LOD**	**LOQ**	**Result**	**Incl. RR**	**Recovery rate [%]**	**RR with same Matrix**
Galactose Probe B / Sample B	1	mg/100g	38								
	2	mg/100g	22								
	3	mg/100g	52								
	4	mg/100g	23	17/5	yes			5			
	5	mg/100g	51	03/05	yes	2	10	< 10	no		
	6	mg/100g	57	17.05.2016	no	10 mg/100g	40 mg/100g	not detecta-ble			
	7	mg/100g	29								
	8	mg/100g	3	12.5.	yes			11			
	9	mg/100g	30	21.4.16	yes	1	3	3,7	no		no
	10	mg/100g	60				20	25	no		
	11	mg/100g	1	13.05.2016	yes			100	yes	100	no
	12	mg/100g	49	NA							
	13	mg/100g	35								
	14	mg/100g	18		yes		2	13	yes		yes
	15	mg/100g	27	27.05.	yes	5	10	12,1	no	97	yes
	16	mg/100g	5 + 46								
	17	mg/100g	44	12.05.16	no	20	60	<LOQ	no		
	18	mg/100g	7	17/05	no		0	<100	no		
	19	mg/100g	25								
	20	mg/100g	11	24.06.16	yes	0,01	0,01	0,01	no	-	-

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Parameter	Teilnehmer	Einheit	Proben-Nr.	Datum d. Analyse	Nach-weisbar	NWG	BG	Ergebnis	Inkl. WF	Wiederfin-dungsrate [%]	WF mit gleicher Matrix
Analyte	**Participant**	**Unit**	**Sample-No.**	**Date of ana-lysis**	**Detecta-ble**	**LOD**	**LOQ**	**Result**	**Incl. RR**	**Recovery rate** [%]	**RR with same Matrix**
Galactose Dotierungs-materialprobe / Spiking Material Sample	1	g/100g	10								
	2	g/100g	3								
	3	g/100g	26								
	4	g/100g	11	17/5	no			-			
	5	g/100g	43	03/05	no	0,02	0,1	< 0,1	no		
	6	g/100g	48								
	7	g/100g	40								
	8	g/100g	19	12.5.	no			<0,1			
	9	g/100g	53	21.4.16	no	1	3	< 0,003	no		no
	10	g/100g	17				0,94	n.n.	no		
	11	g/100g	42	13.05.2016	no			<0.08	yes	100	no
	12	g/100g	22	NA							
	13	g/100g	16								
	14	g/100g	36		yes		0,1	<0,1	no	-	-
	15	g/100g	7	27.05.	no	5	10	n.d.	no	97	yes
	16	g/100g	23 + 35								
	17	g/100g	29	12.05.16	no	20	60	<LOQ	no		
	18	g/100g	14	17/05	no		0	<0.1	no		
	19	g/100g	12								
	20	g/100g	32	24.06.16	no	0,01	0,01	<0,01	no	-	-

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5.2 Homogeneity

5.2.1 Mixture homogeneity before bottling

Microtracer Homogeneity Test
DLA 14-2016 Spiking material sample

Weight whole sample	3,00	kg
Microtracer	FSS-rot lake	
Particle size	75 – 300	µm
Weight per particle	2,0	µg
Addition of tracer	11,2	mg/kg

Result of analysis

Sample	Weight [g]	Particle number	Particles [mg/kg]
1	10,06	47	9,3
2	10,19	45	8,8
3	10,24	53	10,4
4	10,19	49	9,6
5	10,18	53	10,4
6	10,16	52	10,2
7	10,21	51	10,0
8	10,04	52	10,4

Poisson distribution		
Number of samples	8	
Degree of freedom	7	
Mean	50,2	Partikel
Standard deviation	2,93	Partikel
χ^2 (CHI-Quadrat)	1,19	
Probability	**99**	%
Recovery rate	89	%

Normal distribution		
Number of samples	8	
Mean	9,9	mg/kg
Standard deviation	0,58	mg/kg
rel. Standard deviaton	5,8	%
Horwitz standard deviation	11,3	%
HorRat-value	**0,5**	
Recovery rate	89	%

Microtracer Homogeneity Test
DLA 14-2016 Sample B

Weight whole sample	4,01	kg
Microtracer	FSS-rot lake	
Particle size	75 – 300	µm
Weight per particle	2,0	µg
Addition of tracer	6,7	mg/kg

Result of analysis

Sample	Weight [g]	Particle number	Particles [mg/kg]
1	10,20	43	8,4
2	10,33	34	6,6
3	10,48	34	6,5
4	10,17	42	8,3
5	10,30	39	7,6
6	10,14	40	7,9
7	10,22	40	7,8
8	10,25	44	8,6

Poisson distribution		
Number of samples	8	
Degree of freedom	7	
Mean	39,5	Partikel
Standard deviation	4,08	Partikel
χ^2 (CHI-Quadrat)	2,94	
Probability	**89**	%
Recovery rate	116	%

Normal distribution		
Number of samples	8	
Mean	7,7	mg/kg
Standard deviation	0,79	mg/kg
rel. Standard deviaton	10,3	%
Horwitz standard deviation	11,8	%
HorRat-value	**0,9**	
Recovery rate	116	%

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5.2.2 Homogeneity of bottled PT-samples

Homogeneity test of lactose by enzyme UV-Test (ASU §64 L 48.01-4):

Spiking material sample (Lactose)

Independant samples	g/100g
1	4,07
2	4,11
3	4,09
4	4,09
5	4,13

Mean	4,10	
Repeatability Standard Deviation	0,0228	0,56%

Sample B (Lactose + Galactose)

Independant samples	mg/100g
1	415
2	417
3	415
4	414
5	426

Mean	417	
Repeatability Standard Deviation	4,93	1,2%

5.2.3 Comparison of sample numbers / test results and trend line

By comparison of the increasing sample numbers and the measurement results, the homogeneity of the chronological bottled PT item can be characterized with the help of the trend line function:

Spiking material sample fructose
Sample numbers: 3 – 53 (without outliers)
Measurement results: 14
Trend line range: 7,19 ± 0,0119 g/100g (= ± 0,036 x σ_{pt}')
Maximum relative deviation to mean: ± 0,17%

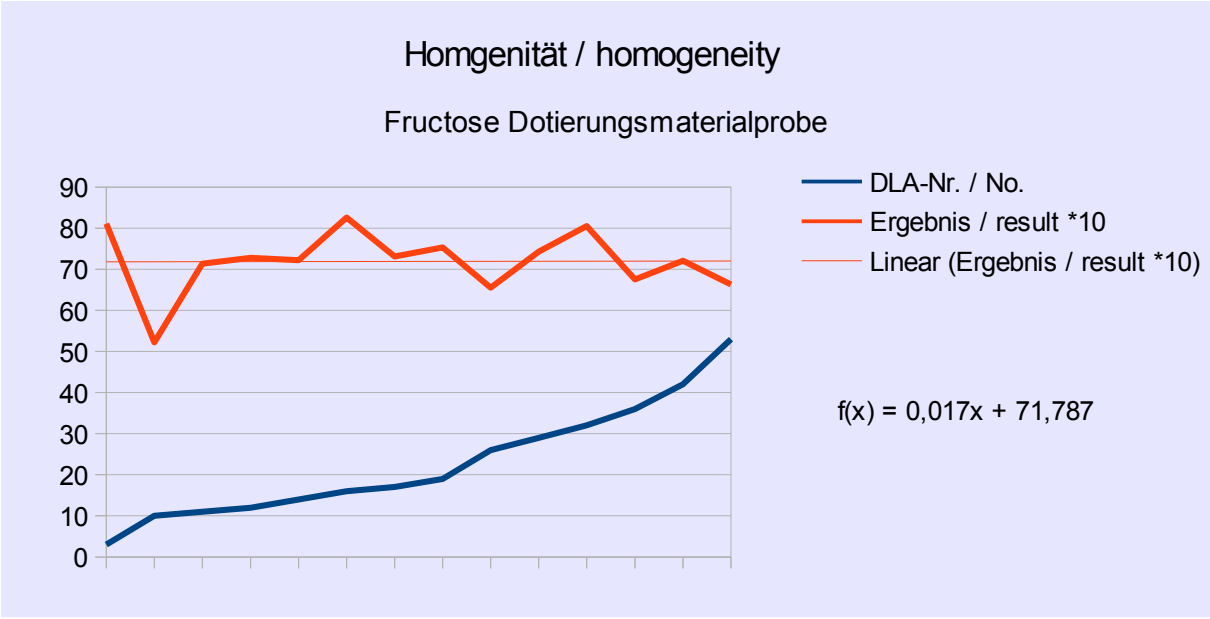

Abb. 20: Trendfunktion Probennummern / Fructose
Ergebnisse (x 10 dargestellt)
Fig. 20: trend line function sample number / fructose
results (x 10 shown)

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Sample A fructose

Sample numbers: 2 – 54 (without outliers and no. 57 z'-score >3)
Measurement results: 21
Trend line range: 521 ± 24,0 mg/100g (= ± 0,32 x σ_{pt}')
Maximum relative deviation to mean: ± 4,61%

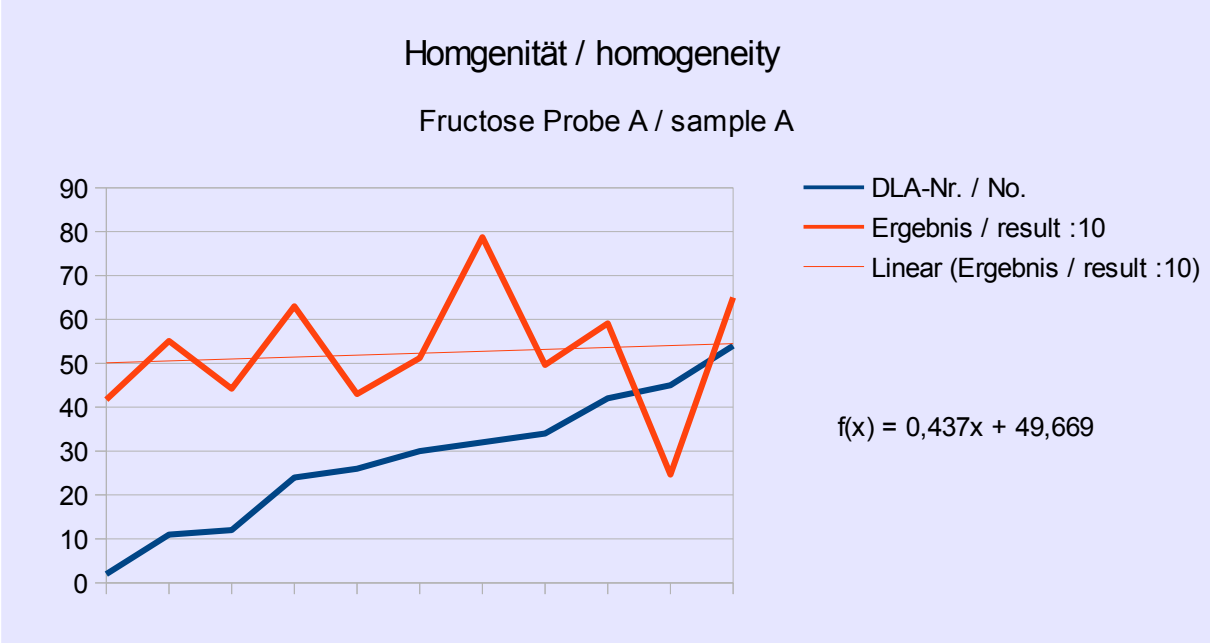

Abb. 21: Trendfunktion Probennummern / Ergebnisse Fructose (Ergebnisse 1/10 dargestellt)
Fig. 21: trend line function sample number / results fructose (results 1/10 shown)

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Sample B Lactose
Sample numbers: 3 - 60
Measurement results: 14
Trend line range: 152 ± 2,94 mg/100g (= ± 0,20 x σ_{pt}')
Maximum relative deviation to mean: ± 1,93%

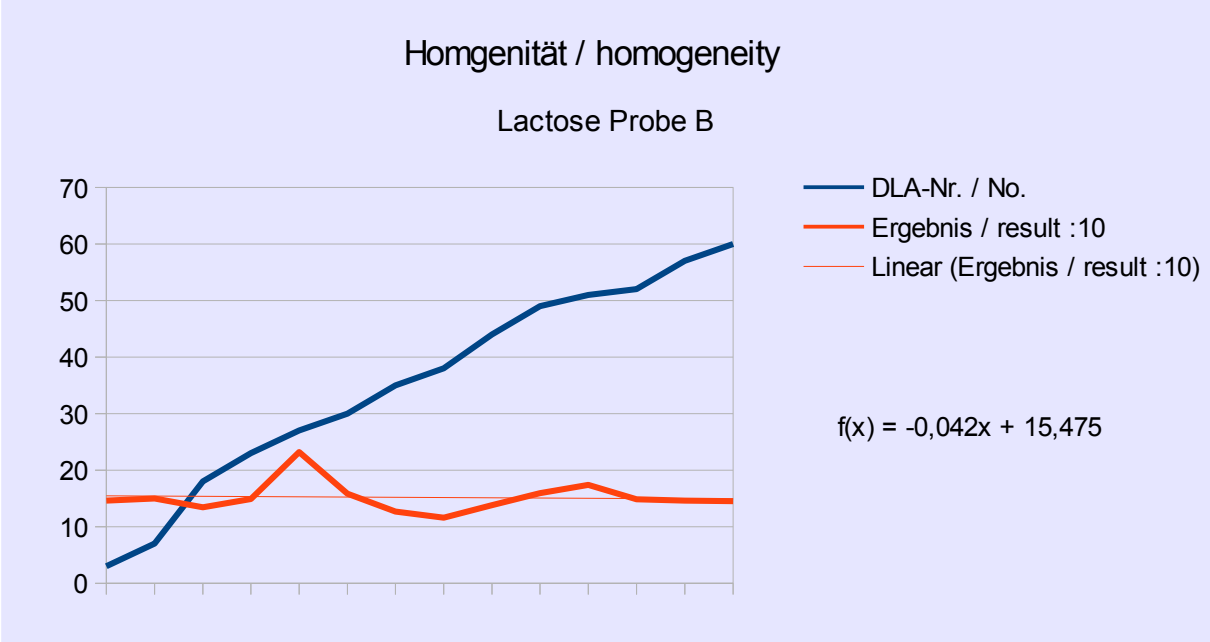

Abb. 22: Trendfunktion Probennummern / Ergebnisse Lactose (Ergebnisse 1/10 dargestellt)
Fig. 22: trend line function sample number / results lactose (results 1/10 shown)

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5.3 Analytical Methods

Details by the participants

Teilnehmer	Analyten	Methode ist akkreditiert	Methodenbeschreibung	Probenvorbereitung	Einwaage	Extraktion	Referenzmaterial	Sonstige Hinweise
Participant	Analytes	Method accredited	Method description	Sample Preparation	Sample weight	Extraction	Referencematerial	Further remarks
1	Fructose	yes	Ion chromatography	preparation of water extract	appr. 1 g	70°C, water	Sigma Aldrich, F0127-100G	
2	Fructose	yes	HPLC					
3	Fructose	yes	IC-PAD	dissolved sample in water	2 g	water	Sigma	
4	Fructose	no	HPLC	dilution and filtration	1g/100ml	25°C, water		
5	Fructose							
6	Fructose							
7	Fructose							
8	Fructose	yes	Spectrophotometry, enzymatically (Thermo Fisher Scientific 984302)					
9	Fructose	yes	HPAEC/PAD	Extraction, Filtration, Dilution	appr. 1 g	100 ml demin. water, 60°C	Merck	
10	Fructose	yes	enzymatically, r-biopharm	water extract ultrasonic bath	5 g	Carrez-precipitation	D (-) Fructose for biochemistry / Merck	
11	Fructose	yes	enzymatically	homogenization	5 g	water	Testkit Enzyme	
12	Fructose							
13	Fructose	no	Ion chromatography-Amperometric pulse detector	Water extraction				
14	Fructose	yes	enzymatically		2 g	water, 60 °C		
15	Fructose	yes	in-house method GC-FID	homogenization	5 g	50 ml water, 10 min ultrasonic, carrez clean-up	Merk	
16	Fructose							
17	Fructose	yes	enzymatically	homogenization	1g/10ml; Dot.probe 3g/100ml	water extraction	r-biopharm	
18	Fructose	yes	HPAEC-PAD	no extra preparation has been done	1g	water, 40 °C	Eurofins	
19	Fructose	yes	Roche art.no.: 10716260035	IFU	0,5g			
20	Fructose	yes	HPIEC-PAD	homogenization	1 g	60°C, 1 h Ultrasonic		

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Teilnehmer	Analyten	Methode ist akkreditiert	Methodenbeschreibung	Probenvorbereitung	Einwaage	Extraktion	Referenzmaterial	Sonstige Hinweise
Participant	Analytes	Method accredited	Method description	Sample Preparation	Sample weight	Extraction	Referencematerial	Further remarks
1	Lactose	yes	Ion chromatography	preparation of water extract	appr. 1 g	70°C, water	Sigma Aldrich, 61339-25G	
2	Lactose	yes	HPLC					
3	Lactose	yes	HPLC-MS	shaking	2 g	formate buffer	Fluka	recovery calculated by C13-Lactose internal standard
4	Lactose	no	HPLC	dilution and filtration	1g/100ml	25°C, water		
5	Lactose	no	enzymatically	5,0 g / 100 ml	5,0 g	70 °C / 15 Min. with Carrez precipitation	DLA 08/2012	
6	Lactose	yes	ASU L 17.00-7 (1983-11); mod.		5 g	water extract 70 °C	chocolate / MUVA Kempten	
7	Lactose	no	by enzymatic method, a modification of AOAC Method 984.15	None	10g	pH 6,5-8,6 and Carrez	R.30.K-LACGAR LACTOSA (INGENASA)	
8	Lactose	yes	Spektrophotometry, enzymatically (r-biopharm Test-Combination 10176303035)					
9	Lactose	yes	HPAEC/PAD	Extraction, Filtration, Dilution	appr. 1 g	100 ml demin. water, 60°C	Merck	
10	Lactose	yes	enzymatically, r-biopharm	water extract ultrasonic bath	5 g	Carrez-precipitation	Lactose-Monohydrat for biochemistry / Merck	
11	Lactose	yes	enzymatically	homogenization	5 g	water	Testkit Enzyme	
12	Lactose	no	Enzymatic method using Boehringer/R-Biopharm Test-Combination kit for the quantitative determination of lactose in any foodstuff. The method has been validated at NRC on powdered beverages for aroma (PBA), and has been adapted and validated to enable the quantification of lactose in lactose-free infant formulae	Bring the whole laboratory sample (original container) to room temperature and homogenise it by mixing. Take the test portion for analysis from the homogeneous test sample.	4,5 g	Addition of 60 mL distilled water and extraction of sugars for 20 min at 70 °C. Addition of Carrez I solution, Carrez II solution and 0.1 M NaOH. Filtration of the solution.	DS81 REF012 internal reference sample	no
13	Lactose	no	Ion chromatography-Amperometric pulse detector	Water extraction				
14	Lactose	yes	enzymatically		5 g	water, 60 °C		
15	Lactose	yes	in-house method GC-FID	homogenization	5 g	50 ml water, 10 min ultrasonic, carrez clean-up	Sigma	
16	Lactose	no	Enzymatic (R-Biopharm)		1g	70'c Water Extraction. Carrez 1 and 2 to clean up		
17	Lactose	yes	enzymatically	homogenization	1g/10ml; Dot.probe 3g/100ml	water extraction	r-biopharm	
18	Lactose	no	HPAEC-PAD	no extra preparation has been done	3g	water, 40 °C	none	
19	Lactose							
20	Lactose	yes	HPIEC-PAD	homogenization	1 g	60°C, 1 h Ultrasonic		

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Teilnehmer Participant	Analyten Analytes	Methode ist akkreditiert Method accredited	Methodenbeschreibung Method description	Probenvorbereitung Sample Preparation	Einwaage Sample weight	Extraktion Extraction	Referenzmaterial Referencematerial	Sonstige Hinweise Further remarks
1	Galactose							
2	Galactose							
3	Galactose							
4	Galactose	no	HPLC	dilution and filtration	1g/100ml	25°C, water		
5	Galactose	no	enzymatically	5,0 g / 100 ml	5,0 g	70 °C / 15 Min. with Carrez precipitation		
6	Galactose	yes	ASU L 17.00-7 (1983-11); mod.		5 g	water extract 70 °C	chocolate / MUVA Kempten	
7	Galactose							
8	Galactose	yes	Spektrophotometry, enzymatically (r-biopharm Test-Combination 10176303035)					
9	Galactose	yes	HPAEC/PAD	Extraction, Filtration, Dilution	appr. 1 g	100 ml demin. water, 60°C	Merck	
10	Galactose	yes	enzymatically, r-biopharm	water extract ultrasonic bath	5 g	Carrez-precipitation	D (+) Galactose for biochemistry / Merck	
11	Galactose	yes	enzymatically	homogenization	5 g	water	Testkit Enzyme	
12	Galactose							
13	Galactose							
14	Galactose	yes	enzymatically		5 g	water, 60 °C		
15	Galactose	yes	in-house method GC-FID	homogenization	5 g	50 ml water, 10 min ultrasonic, carrez clean-up	Fluka	
16	Galactose							
17	Galactose	yes	enzymatically	homogenization	1g/10ml; Dot.probe 3g/100ml	water extraction	r-biopharm	
18	Galactose	no	HPAEC-PAD	no extra preparation has been done	1g	water, 40 °C	Eurofins	
19	Galactose							
20	Galactose	yes	HPIEC-PAD	homogenization	1 g	60°C, 1 h Ultrasonic		

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6. Index of participant laboratories in alphabetical order

Teilnehmer / Participant	Ort / Town	Land / Country
		GREAT BRITAIN
		SPAIN
		ITALY
		Germany
		Germany
		SPAIN
		NETHERLANDS
		Germany
		NETHERLANDS
		Germany
		Germany
		Germany
		Germany
		SWITZERLAND
		Germany
		Germany
		GREAT BRITAIN
		NETHERLANDS
		Germany
		Germany
		Germany

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

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

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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
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21. ISO 22662:2012; Milch und Milchprodukte - Bestimmung des Lactosegehalts mit Hochleistungs-Flüssigchromatographie (Referenzverfahren) / Milk and milk products – Determination of lactose content by high-performance

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liquid chromatography (Reference method)
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