Proficiency Tests
DLA
food
cosmetics
consumer goods
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Evaluation Report
proficiency test

DLA 18/2017

Lactose and Fructose:

in “lactose free” food (bread baking mixture)

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

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Unteraufträge *Subcontractors*	Die Prüfung der Gehalte, Homogenität und Stabilität von EP-Parametern wird von DLA im Unterauftrag vergeben. The analysis of the content, homogeneity and stability of PT-parameters are subcontracted by DLA.
Vertraulichkeit *Confidentiality*	Die Teilnehmerergebnisse sind im EP-Bericht in anonymisierter Form mit Auswertenummern benannt. Daten einzelner Teilnehmer werden ausschließlich nach vorheriger Zustimmung des Teilnehmers an Dritte weitergegeben. Participant result are named anonymously with evaluation numbers in the PT report. Data of individual participants will be passed on to third parties only with prior consent of the participant.

Inhalt / Content

1. Introduction

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

2. Realisation

2.1 Test material

Two PT-samples for the detection of lactose/galactose and fructose with contents in the range of mg/100g and one spiking level sample with a simple matrix were provided for analysis. To one of the PT-samples (spiked sample) and the spiking level sample the EP-paramaters lactose and fructose were added in similar concentrations. The results of the spiking level sample should give the possibility of a comparison with the spiked sample in respect to the detectability of the paramaters 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 samples A and B was the same (see table 1). After homogenization of the basic mixture the spiked sample B was produced as follows:

The spiking materials lactose and fructose were grounded, sieved (mesh 400 µm) and added to an aliquot of the basic mixture. The resulting mixture was homogenized anew. Afterwards basic mixture was added in 3 further steps, followed by an mechanical homogenization until the total amount was reached.
The spiking level sample was produced using the mentioned spiking materials by stepwise addition of potato powder/potato flour and homogenization of the resulting mixture. Afterwards the total amount was sieved by a centrifugal mill (mesh 500 µm).

Afterwards the samples A, B and spiking level sample were portioned to approximately 25 g into metallised PET film bags and chronologically numbered.

The composition of the PT samples and the spiking level sample is given in table 1.

Table 1: Composition of DLA-Samples

Ingredients	**Sample A**	**Sample B**	**Spiking Level Sample**
Baking Mixture White Bread Ingredients: Wheat flour, salt, dried yeast, glucose, sugar, emulsifier: E 481, malt extract (barley, rye), flour treatment agents: ascorbic acid Nutrients per 100 g: protein 12 g, carbohydrates 69 g, sugar 0,5 g, fat 1,0 g, dietary fibre 4,3 g, salt 1,9 g	100 g/100g	99,6 g/100g	-
Potato Powder Ingredients: Potato, E471, E304, E223, E100	-	-	80,7 g/100g
Potato Flour	-	-	19,0 g/100g
Lactose	-	82,0 mg/100g	78,6 mg/100g
Fructose	-	310 mg/100g **	185 mg/100g

*Ingredient contents according to gravimetric mixture
** Sample B contains additionally fructose from the bread baking mixture

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

2.1.1 Homogeneity

The **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 A, B and the spiking level sample showed a probability of 13%, 83% and 88%. 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 1,8, 0,90 and 0,80 respectively. The results of microtracer analysis are given in the documentation.

The calculation of the **repeatability standard deviations S_r of the participants** can not used as an indicator of homogeneity in the present PT, as only one results per participant was submitted.

Furthermore, the homogeneity was characterized by the **trend line function of participants' results for chronological bottled single samples**. The maximum deviations from the mean value of the trend line was 40% of the target standard deviation for sample B (lactose) and 26% for the spiking level sample (lactose) (s. 5.2 homogeneity) and can therefore be regarded as normal and low, respectively, because instead of two symmetrically distributed samples only one sample was analyzed by the participants during this PT.

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

2.1.2 Stability

The experience with various DLA reference materials showed good storage stability with respect to the durability of the sample (spoilage) and the content of the PT parameters fructose, lactose and galactose for comparable food matrices and water activity (a_W value <0,5). The stability of sample material is therefore given during the investigation period under consideration of given storage conditions.

2.2 Sample shipment and information to the test

One portion of each test material (sample BA, sample B and spiking level sample) was sent to every participating laboratory in the 17th week of 2017. The testing method was optional. The tests should be finished at 9st June 2017 the latest.

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

*There are **two different samples A and B** possibly containing the parameters lactose/galactose and fructose in the range relevant for labeling (of lactose) of mg/100g in the matrix of bread baking mixture. One of these samples and the "spiking level sample" were prepared adding lactose and fructose. The **"spiking level sample"** contains the parameters in a simple matrix in **similar amounts.***

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

2.3 Submission of results

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

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

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

18 out of 20 registered participants submitted the results in time. One participant submitted results delayed after consultation with DLA and another participant submitted no results.

3. Evaluation

3.1 Consensus value from participants (assigned value)

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

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

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

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

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

3.2 Robust standard deviation

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

3.3 Repeatability standard deviation

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

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

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

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 in percent of the mean is given as variation coefficient VK_R in the statistical data in the results, if single results of participants were submitted. The meaning is explained in section 3.9.

3.5 Exclusion of results and outliers

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

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

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

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

3.6 Target standard deviation (for proficiency assessment)

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

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

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

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

For the evaluation of the parameters fructose and lactose the target standard deviation according to the general model of Horwitz was applied (see 3.6.1). Additionally the standard uncertainty was considered for these PT-parameters and the results were evaluated by z´-score.

Due to the number of < 7 results for galactose, no statistical evaluation by z-score was performed.

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 Precision experiment

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

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

The relative repeatability standard deviations (RSD_r) and relative reproducibility standard deviation (RSD_R) given in Table 2 were determined in ring tests using the indicated methods.
The resulting target standard deviations σ_{pt}, which were identified there, were used to evaluate the results and to provide additional information for the statistical data.

Table 2: Relative repeatability standard deviations (RSD_r) and relative reproducibility standard deviation (RSD_R) according to selected evaluations of tests for precision and the resulting target standard deviation σ_{pt} [16-21]

Parameter	**Matrix**	**Mean** [g/100g]	**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	Baby 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] *in the evaluation (s. section 4) given values for information, lactose is given 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 3.6.1 considering the standard uncertainty (s. 3.6.8) was regarded suitable.

3.7 z-Score

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

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

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

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

The valid z-score is indicated as z-score (σ_{pt}) in the evaluation. The as z-score (info) designated value only obtains an informative character. The both z-scores were calculated with different target standard deviations described in 3.6.

3.7.1 Warning and action signals

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

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

Table 3: Characteristics of the present PT (on blue-grey) in comparison to previous PTs since 2015 (SD = standard deviation, CV = coefficient of variation)

Parameter	**Matrix**	**robust Mean** [mg/100g]	**rob. SD (S*)** [mg/100g]	**rel. SD (VK_{S*}) [%]**	**Quotient S^*/σ_{pt}**	**DLA-report**
Fructose	Cookies	288	119	41,3	(9,0)*	DLA 8/2013 (Sample B)
Fructose	Crisp-bread	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)**
Fructose	Bread baking mixture	999	287	28,7	2,3	DLA 18/2017 (Sample B)
Lactose	Cookies	142	37,1	26,1	(4,9)*	DLA 8/2013 (Sample A)
Lactose	Crisp-bread	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)
Lactose	Bread baking mixture	77,7	10,5	13,5	1,9	DLA 18/2017 (Sample B)

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

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

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

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

For warning and action signals see 3.7.1.

3.9 Reproducibility coefficient of variation (CV)

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

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

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

3.10 Quotient S*/σ_{pt}

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

3.11 Standard uncertainty of the assigned value

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

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

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

The Quotient $U_{(Xpt)}/\sigma_{pt}$ is reported in the characteristics of the test.

3.12 Recovery rates: Spiking

For the results of the spiking level 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 (16-21).
For lactose results of the spiking level 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.

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

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

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

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

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

4.1 Fructose

4.1.1 Fructose Sample A (in mg/100g)

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	9
Number of outliers	0
Mean	762
Median	802
Robust Mean (X)	**762**
Robust standard deviation (S*)	**354**
Target range:	
Target standard deviation σ_{pt}´	**151**
Target standard deviation (for Information)	17,8
lower limit of target range	**460**
upper limit of target range	**1060**
Quotient S/σ_{pt}´*	*2,3*
Standard uncertainty $U_{(Xpt)}$	*148*
Quotient $U_{(Xpt)}/\sigma_{pt}$´	*1,0*
Results in the target range	*6*
Percent in the target range	*67%*

Comments:

The target standard deviation was calculated according to the model of Horwitz (s. 3.6.1). Additionally the target standard deviation using data from precision experiments (ASU §64 L 48.02.07-1) is given for information.

The distribution of results showed an increased variability. Valuation was done considering the standard uncertainty by z'-score. The quotient S/σ_{pt}´ was at 2,3. 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 1,0 and thus slightly increased too. The comparability of results is limited.*

67% of results were in the target range.

Sample A was not spiked with fructose. The analysed is explained by the content of the ingredients of the basic mixture (s. p. 6).

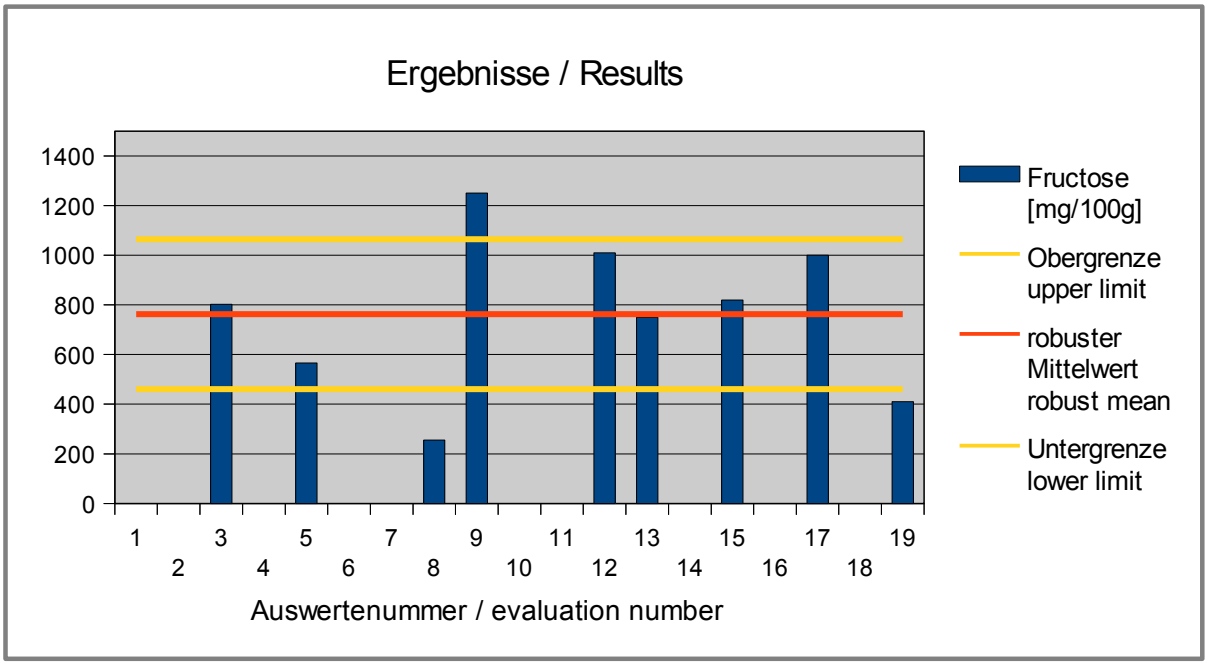

Abb. / Fig. 1: Ergebnisse Fructose / Results Fructose

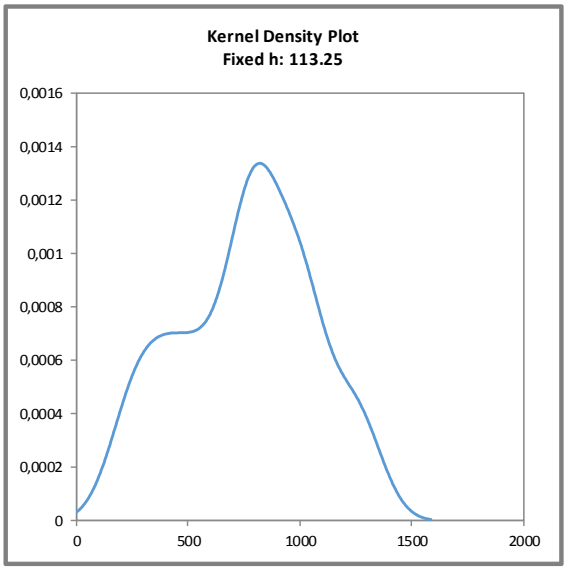

Abb. / Fig. 2:
Kerndichte-Schätzung der Ergebnisse
(mit h = 0,75 x σ_{pt} von X_{pt})

Kernel density plot of results
(with h = 0,75 x σ_{pt} of X_{pt})

Comment:
The kernel density shows almost a normal distribution of results with a shoulder, due to two participant results below the target range.

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Fructose [mg/100g]	Abweichung [mg/100g] Deviation [mg/100g]	z'-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1					
2					
3	802	39,6	0,26	2,2	
4					
5	566	-196	-1,3	-11	
6					
7					
8	255	-507	-3,4	-29	
9	1250	488	3,2	27	
10					
11					
12	1010	248	1,6	14	
13	750	-12,4	-0,08	-0,7	
14					
15	819	56,6	0,37	3,2	
16					
17	1000	238	1,6	13	
18					
19	410	-352	-2,3	-20	

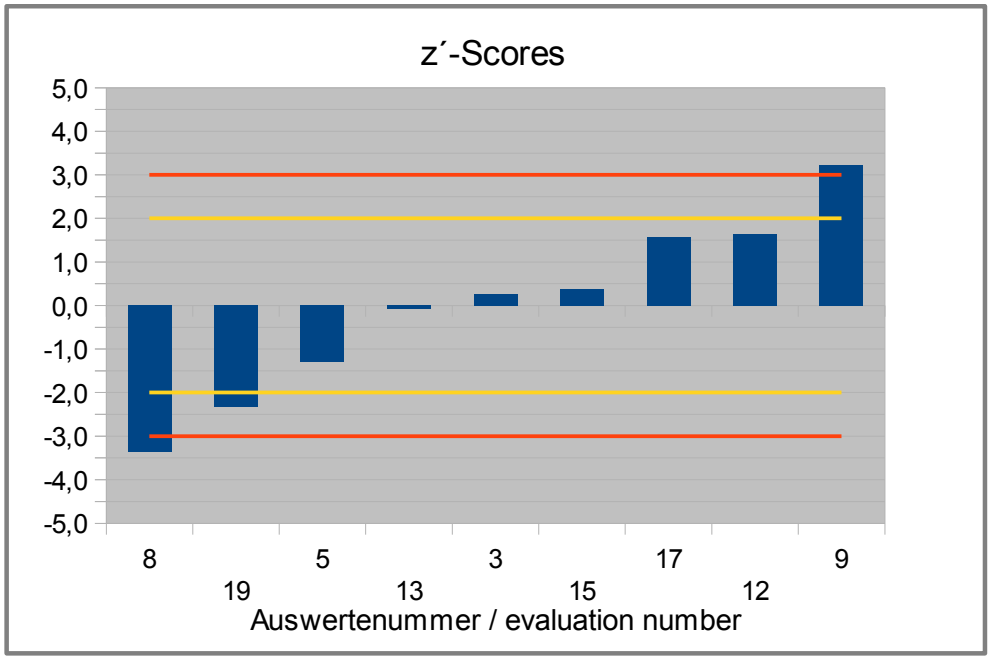

Abb. / Fig. 3: Z´-Scores Fructose (Probe A / Sample A)

4.1.2 Fructose Sample B (in mg/100g)

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	9
Number of outliers	0
Mean	991
Median	1030
Robust Mean (X)	**999**
Robust standard deviation (S*)	**287**
Target range:	
Target standard deviation σ_{pt}´	**126**
Target standard deviation (for Information)	23,3
lower limit of target range	**747**
upper limit of target range	**1250**
Quotient S/σ_{pt}´*	*2,3*
Standard uncertainty $U_{(Xpt)}$	*120*
Quotient $U_{(Xpt)}/\sigma_{pt}$´	*0,95*
Results in the target range	*5*
Percent in the target range	*56%*

Comments:

The target standard deviation was calculated according to the model of Horwitz (s. 3.6.1). Additionally the target standard deviation using data from precision experiments (ASU §64 L 48.02.07-1) is given for information.

The distribution of results showed an increased variability. Valuation was done considering the standard uncertainty by z'-score. The quotient S^*/σ_{pt}´ was at 2,3. 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 0,95 and thus slightly increased too. The comparability of results is limited.

56% of results were in the target range.

The difference between the robust mean of participant results for sample A and sample B (= 237 mg/kg) were at 76 % of the addition level from fructose to sample B (s. p. 6).

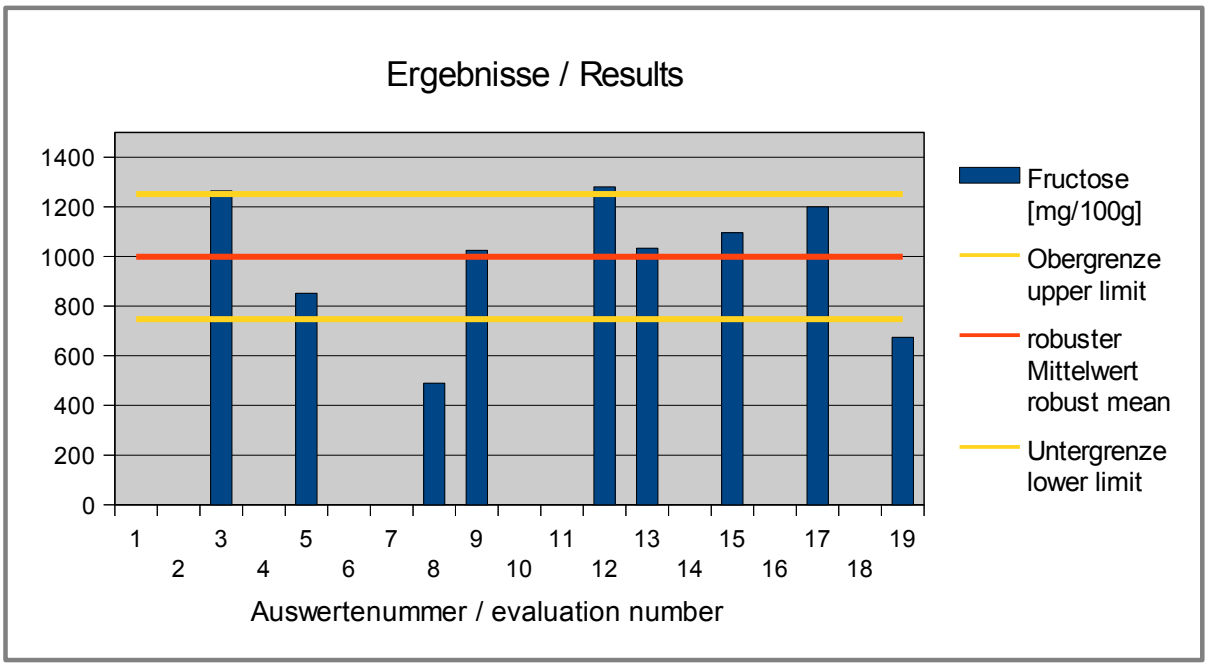

Abb. / Fig. 4: Ergebnisse Fructose / Results Fructose

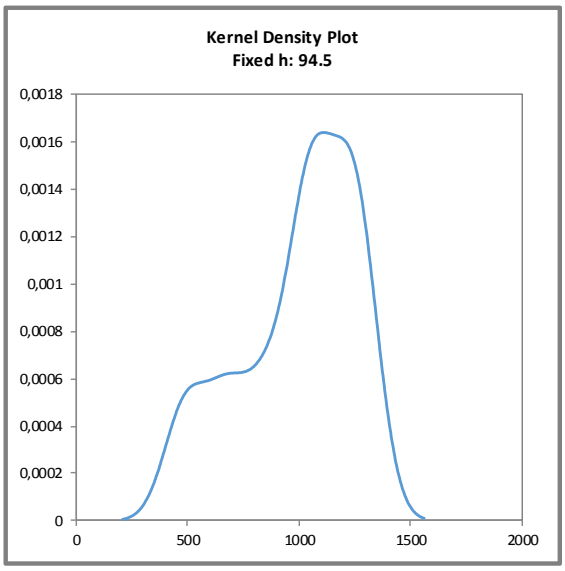

Abb. / Fig. 5:
Kerndichte-Schätzung der Ergebnisse
(mit h = 0,75 x σ_{pt} von X_{pt})

Kernel density plot of results
(with h = 0,75 x σ_{pt} of X_{pt})

Comment:
The kernel density shows almost a normal distribution of results with a shoulder, due to two participant results below the target range.

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Fructose [mg/100g]	Abweichung [mg/100g] Deviation [mg/100g]	z'-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1					
2					
3	1264	265	2,1	11	
4					
5	852	-147	-1,2	-6,3	
6					
7					
8	490	-509	-4,0	-22	
9	1025	25,7	0,20	1,1	
10					
11					
12	1280	281	2,2	12	
13	1033	33,7	0,27	1,4	
14					
15	1096	96,7	0,77	4,1	
16					
17	1200	201	1,6	8,6	
18					
19	675	-324	-2,6	-14	

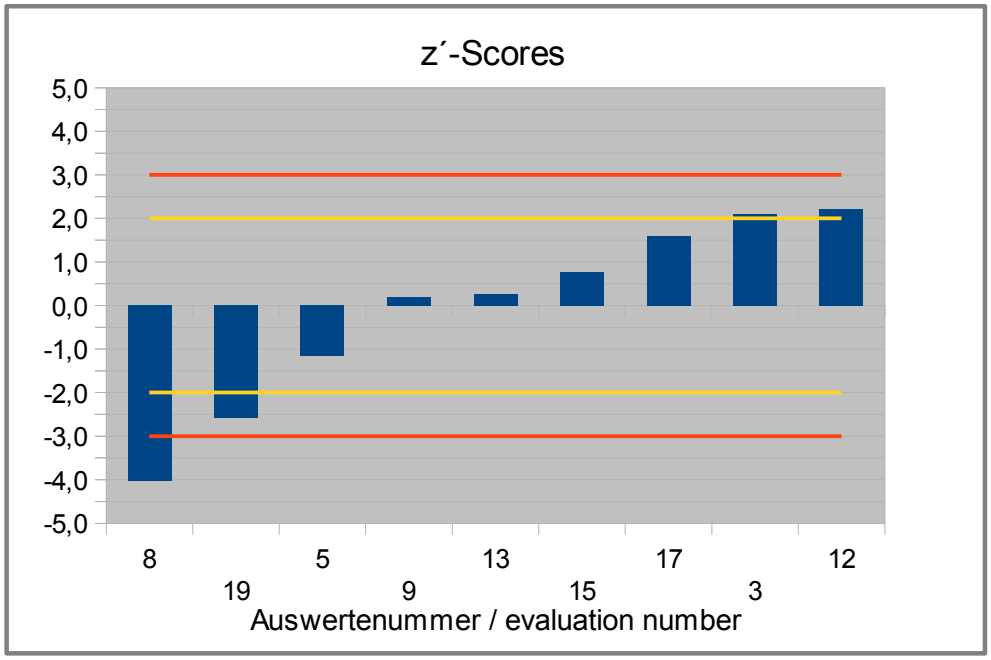

Abb. / Fig. 6: Z´-Scores Fructose (Probe B / Sample B)

4.1.3 Fructose Spiking Level Sample (in mg/100g)

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	8
Number of outliers	0
Mean	197
Median	213
Robust Mean (X)	**197**
Robust standard deviation (S*)	**51,9**
Target range:	
Target standard deviation σ_{pt}´	**25,0**
Target standard deviation (for Information)	4,59
lower limit of target range	**147**
upper limit of target range	**247**
Quotient S/σ_{pt}´*	*2,1*
Standard uncertainty $U_{(Xpt)}$	*22,9*
Quotient $U_{(Xpt)}/\sigma_{pt}$´	*0,92*
Results in the target range	*6*
Percent in the target range	*75%*

Comments:

The target standard deviation was calculated according to the model of Horwitz (s. 3.6.1). Additionally the target standard deviation using data from precision experiments (ASU §64 L 48.02.07-1) is given for information.

The distribution of results showed an increased variability. Valuation was done considering the standard uncertainty by z'-score. The quotient S^*/σ_{pt}´ was at 2,1. 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 0,92 and thus slightly increased too. The comparability of results is limited.

75% of results were in the target range.

The robust mean of participant results was 106 % of the addition level of fructose to the spiking level sample (s. p. 6).

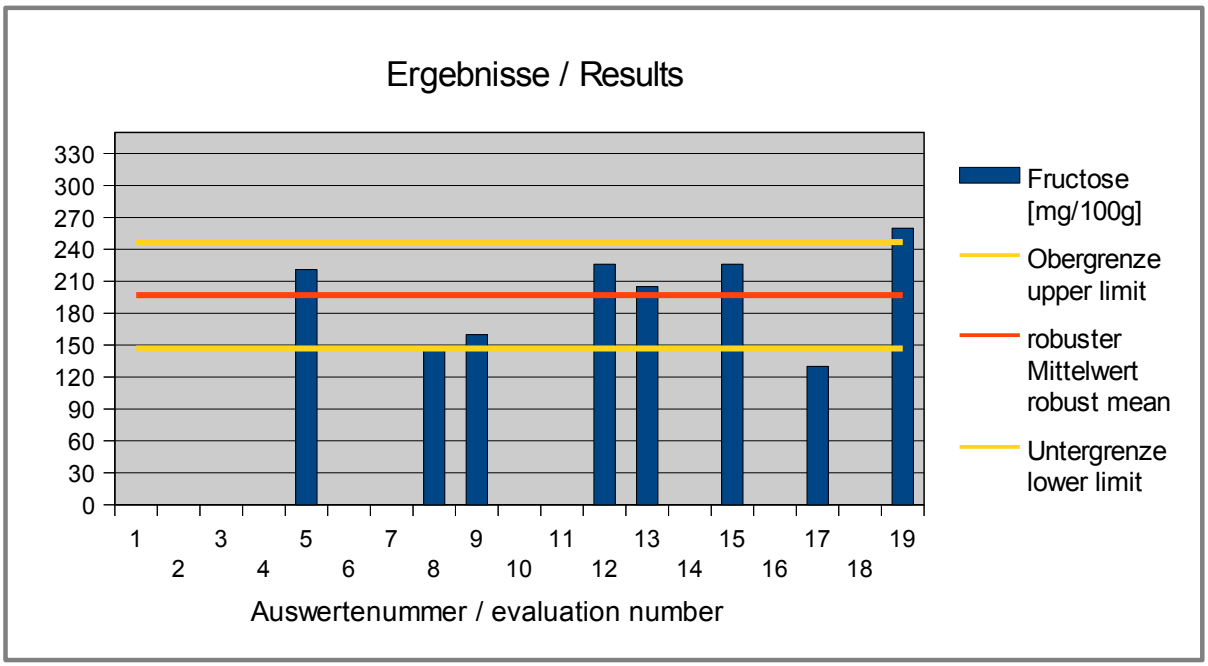

Abb. / Fig. 7: Ergebnisse Fructose / Results Fructose

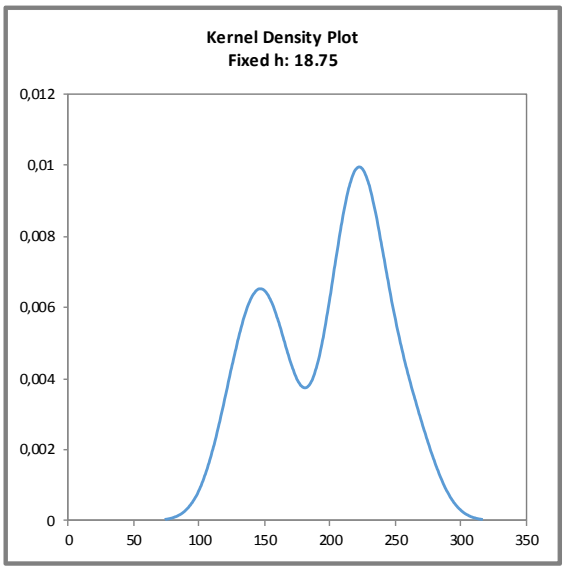

Abb. / Fig. 8:
Kerndichte-Schätzung der Ergebnisse
(mit h = 0,75 x σ_{pt} von X_{pt})

Kernel density plot of results
(with h = 0,75 x σ_{pt} of X_{pt})

Comment:
The kernel density shows a distribution with two peaks, which are not related to the applied methods. The lower peak is due to three lower results.

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Fructose [mg/100g]	Abweichung [mg/100g] Deviation [mg/100g]	z'-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1					
2					
3					
4					
5	221	24,1	1,0	5,3	
6					
7					
8	147	-49,9	-2,0	-11	
9	160	-36,9	-1,5	-8,0	
10					
11					
12	226	29,1	1,2	6,3	
13	205	8,13	0,32	1,8	
14					
15	226	29,1	1,2	6,3	
16					
17	130	-66,9	-2,7	-15	
18					
19	260	63,1	2,5	14	
20					

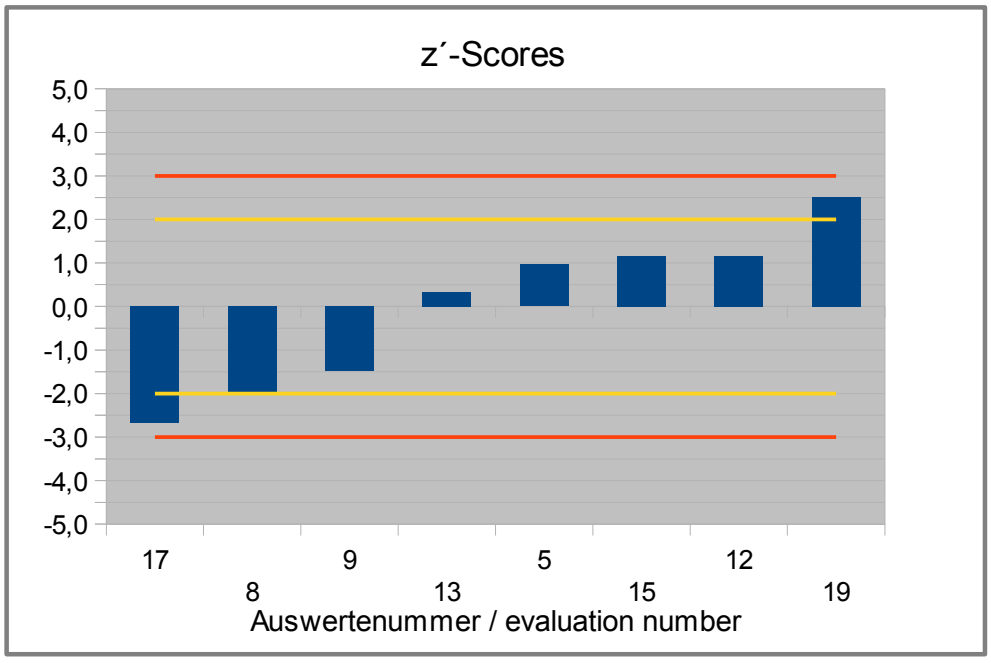

Abb. / Fig. 9: Z´-Scores Fructose

4.2 Lactose

4.2.1 Qualitative Evaluation 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	positive	11,7	positive	87,5	1/1 (100%)	
2	negative	<2	positive	68,8	1/1 (100%)	
3	positive	<10	positive	64,0	1/1 (100%)	
4	positive	113	positive	169	1/1 (100%)	
5	negative	n.n.	positive	78,6	1/1 (100%)	
6	positive	2128	positive	< 300	1/1 (100%)	Sample A + B reversed?
7	negative	<10	positive	77,0	1/1 (100%)	
8	negative	n.d.	positive	66,0	1/1 (100%)	
9	negative	<2,0	positive	73,0	1/1 (100%)	
10	negative	<100	positive	126	1/1 (100%)	
11	negative	0	positive	89,6	1/1 (100%)	
12	negative	< 4	positive	74,0	1/1 (100%)	
13	negative	<10	positive	76,0	1/1 (100%)	
14	positive	< LOQ	positive	73,0	1/1 (100%)	
15	negative	< LOQ	positive	76,0	1/1 (100%)	
16	positive	< 10	positive	87,0	1/1 (100%)	
17	negative		positive	76,0	1/1 (100%)	
18	negative	<10	positive	80,0	1/1 (100%)	
19	negative	<10	positive	65,0	1/1 (100%)	

	Sample A		Sample B	
Number positive	6		19	
Number negative	13		0	
Percent positive	32		100	
Percent negative	68		0	
Consensus value	none		positive	

Comments:
For sample B 100% positive results were obtained. A consensus value for sample A was not obtained. & out of 19 participants submitted a positive result for sample A. Three of these results were located underneath the regarding limit of determination and two of the results above 100 mg/100g.

4.2.2 Lactose Sample B (in mg/100g)

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	18
Number of outliers	2
Mean	83,7
Median	76,0
Robust Mean (X)	**77,7**
Robust standard deviation (S*)	**10,5**
Target range:	
Target standard deviation σ_{pt}´	**5,51**
Target standard deviation (for Information)	6,10
lower limit of target range	**66,7**
upper limit of target range	**88,7**
Quotient S/σ_{pt}´*	*1,9*
Standard uncertainty $U_{(Xpt)}$	*3,09*
Quotient $U_{(Xpt)}/\sigma_{pt}$´	*0,56*
Results in the target range	*12*
Percent in the target range	*67%*

Comments:

The target standard deviation was calculated according to the model of Horwitz (s. 3.6.1). Additionally the target standard deviation using data from precision experiments (ASU §64 L 01.00-17) is given for information.

The distribution of results showed an increased variability. Valuation was done considering the standard uncertainty by z'-score. The quotient S^*/σ_{pt}´ was under 2,0. The robust standard deviation was in the range of previous PTs (see 3.6.3). The quotient $U_{(Xpt)}/\sigma_{pt}$´ was 0,56 and thus slightly increased. The comparability of results is given.

67% of results were in the target range.

The robust mean of participant results was 95 % of the addition level of lactose to sample B (s. p. 6).

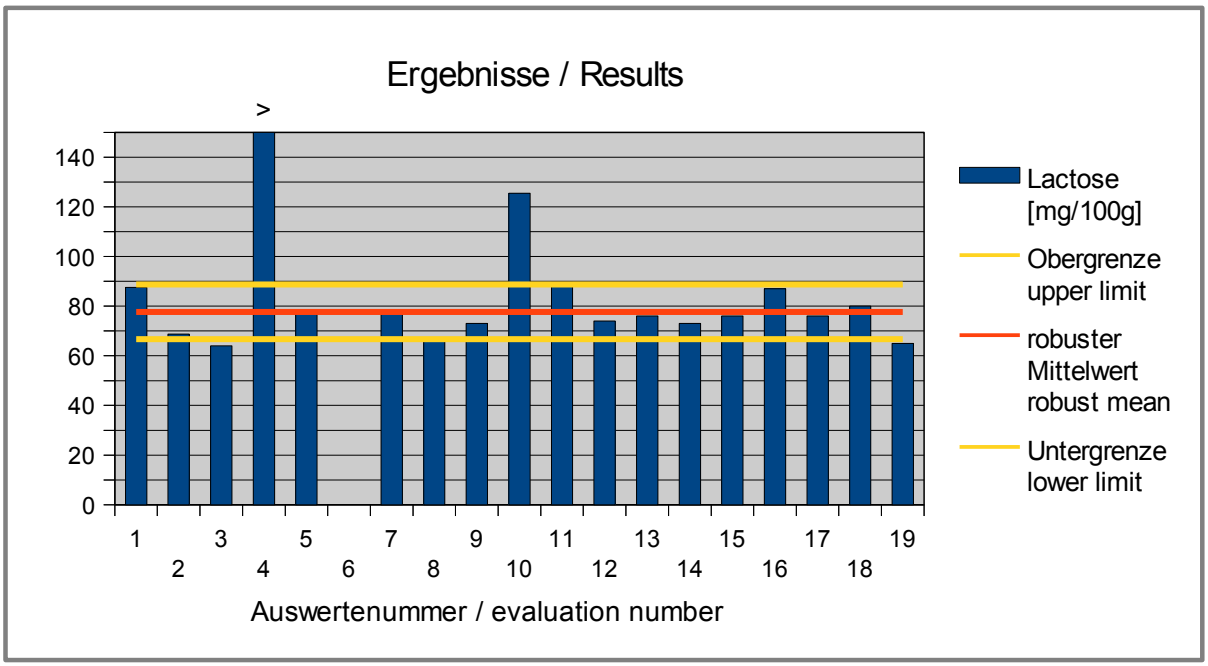

Abb. / Fig. 10: Ergebnisse Lactose / Results Lactose

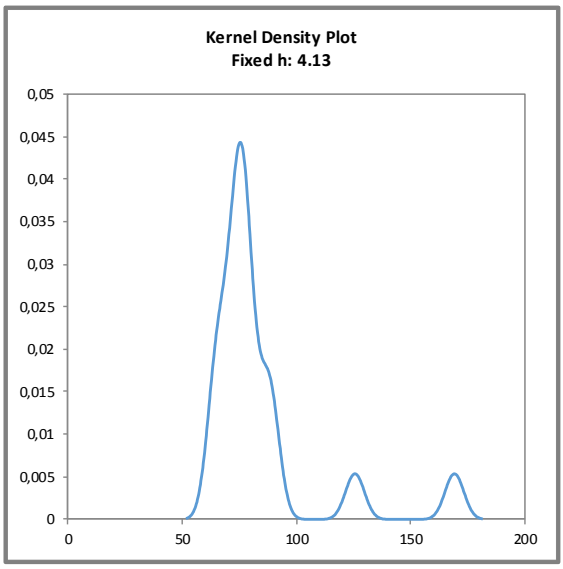

Abb. / Fig. 11:
Kerndichte-Schätzung der Ergebnisse
(mit h = 0,75 x σ_{pt} von X_{pt})

Kernel density plot of results
(with h = 0,75 x σ_{pt} of X_{pt})

Comment:
The kernel density shows a nearly normal distribution of results with two small side peak, due to two outliers.

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Lactose [mg/100g]	Abweichung [mg/100g] Deviation [mg/100g]	z'-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	87,5	9,81	1,8	1,6	
2	68,8	-8,89	-1,6	-1,5	
3	64,0	-13,7	-2,5	-2,2	
4	169	91,3	16,6	15,0	Ausreisser / Outlier
5	78,6	0,913	0,2	0,1	
6					
7	77,0	-0,687	-0,1	-0,1	
8	66,0	-11,7	-2,1	-1,9	
9	73,0	-4,69	-0,8	-0,8	
10	126	47,8	8,7	7,8	Ausreisser / Outlier
11	89,6	11,9	2,2	2,0	
12	74,0	-3,69	-0,7	-0,6	
13	76,0	-1,69	-0,3	-0,3	
14	73,0	-4,69	-0,8	-0,8	
15	76,0	-1,69	-0,3	-0,3	
16	87,0	9,31	1,7	1,5	
17	76,0	-1,69	-0,3	-0,3	
18	80,0	2,31	0,4	0,4	
19	65,0	-12,7	-2,3	-2,1	

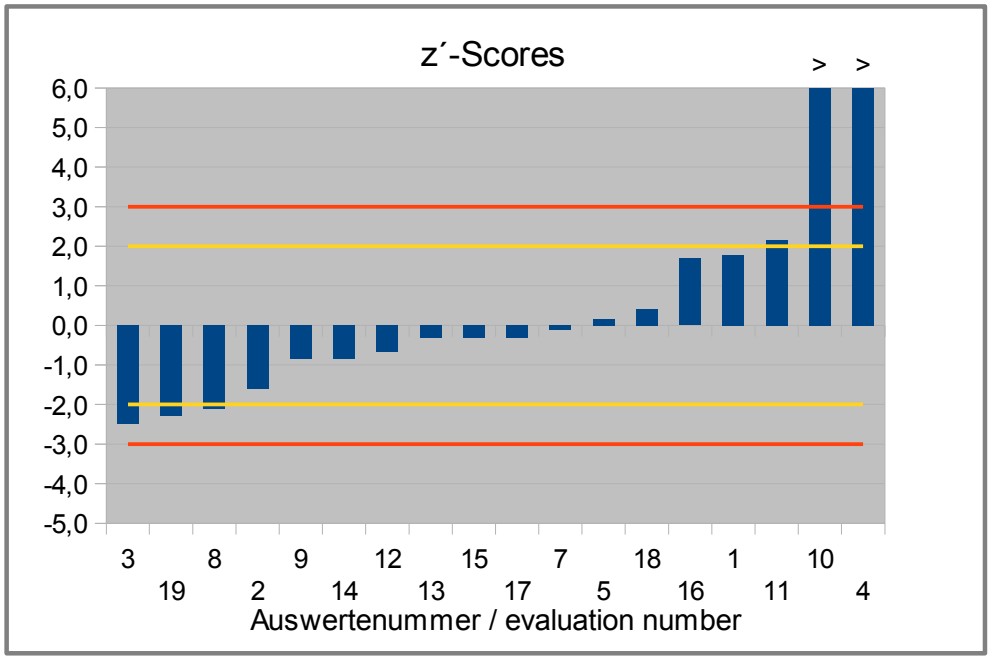

Abb. / Fig. 12: Z´-Scores Lactose (Probe B / Sample B)

4.2.3 Lactose Spiking Level Sample (in mg/100g)

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	17
Number of outliers	3
Mean	81,4
Median	71,1
Robust Mean (X)	**73,9**
Robust standard deviation (S*)	**13,0**
Target range:	
Target standard deviation σ_{pt}	**5,88**
Target standard deviation (for Information)	5,80
lower limit of target range	**62,1**
upper limit of target range	**85,6**
Quotient S/σ_{pt}*	*2,2*
Standard uncertainty $U_{(Xpt)}$	*3,93*
Quotient $U_{(Xpt)}/\sigma_{pt}$	*0,67*
Results in the target range	*11*
Percent in the target range	*65%*

Comments:

The target standard deviation was calculated according to the model of Horwitz (s. 3.6.1). Additionally the target standard deviation using data from precision experiments (ASU §64 L 01.00-17) is given for information.

The distribution of results showed an increased variability. Valuation was done considering the standard uncertainty by z'-score. The quotient S^*/σ_{pt}´ was at 2,2. The robust standard deviation was in the range of previous PTs (see 3.6.3). The quotient $U_{(Xpt)}/\sigma_{pt}$´ was 0,67 and thus slightly increased too. The comparability of results is limited.

66% of results were in the target range. One result (evaluation number 6) was excluded prior to evaluation.

The robust mean of participant results was 94 % of the addition level of lactose to the spiking level sample (s. p. 6).

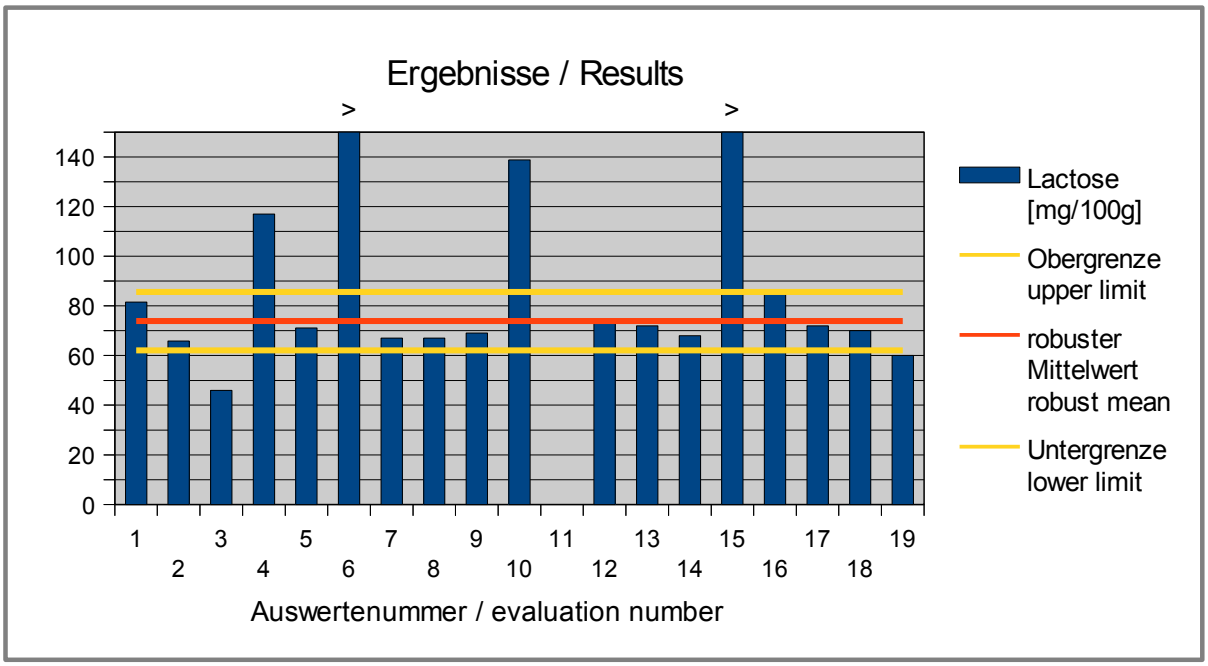

Abb. / Fig. 13: Ergebnisse Lactose / Results Lactose

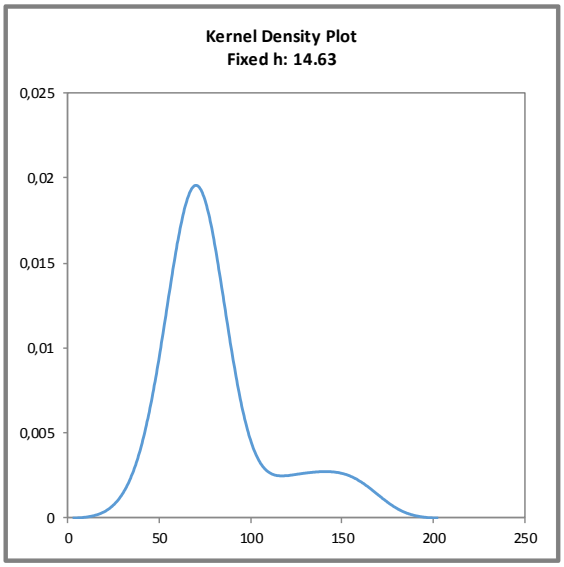

Abb. / Fig. 14:
Kerndichte-Schätzung der Ergebnisse
(mit h = 0,75 x σ_{pt} von X_{pt})

Kernel density plot of results
(with h = 0,75 x σ_{pt} of X_{pt})

Comment:
The kernel density shows a normal distribution of results with a side peak, due to the three outliers above the target range.

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Lactose [mg/100g]	Abweichung [mg/100g] Deviation [mg/100g]	z'-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	81,5	7,62	1,3	1,3	
2	65,9	-8,0	-1,4	-1,4	
3	46,0	-27,9	-4,7	-4,8	
4	117	43,1	7,3	7,4	Ausreisser / Outlier
5	71,1	-2,78	-0,5	-0,5	
6	5350				Ergebnis ausgeschlossen / Result excluded
7	67,0	-6,88	-1,2	-1,2	
8	67,0	-6,88	-1,2	-1,2	
9	69,1	-4,78	-0,8	-0,8	
10	139	65,0	11,0	11,2	Ausreisser / Outlier
11					
12	73,0	-0,88	-0,1	-0,2	
13	72,0	-1,88	-0,3	-0,3	
14	68,0	-5,88	-1,0	-1,0	
15	159	85,1	14,5	14,7	Ausreisser / Outlier
16	86,0	12,1	2,1	2,1	
17	72,0	-1,88	-0,3	-0,3	
18	70,0	-3,88	-0,7	-0,7	
19	60,0	-13,9	-2,4	-2,4	

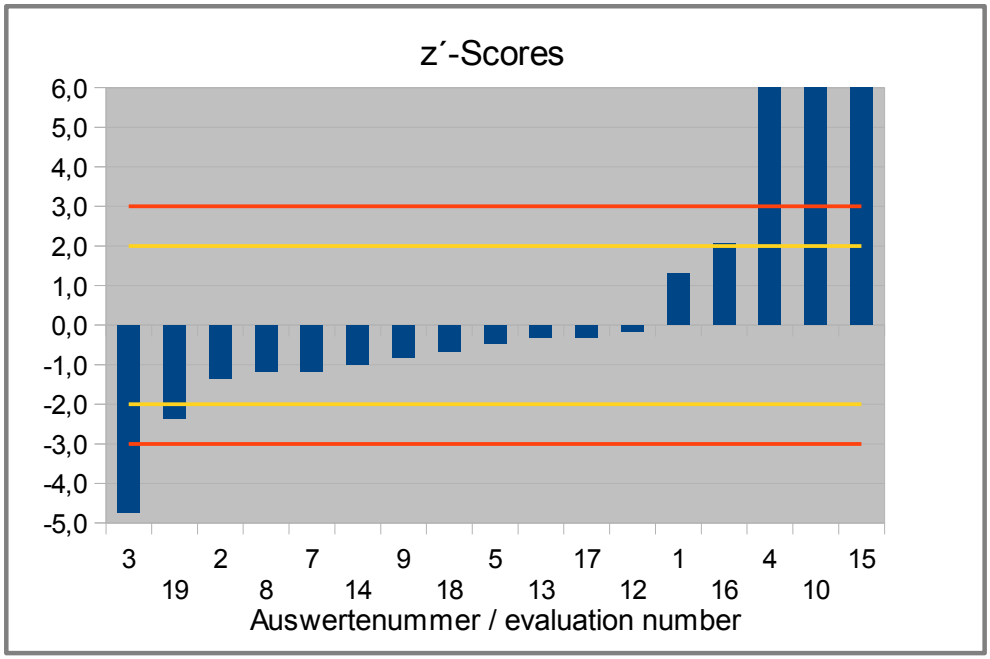

Abb. / Fig. 15: Z´-Scores Lactose

4.2.4 Recovery Rates for Lactose

Hereafter the recovery rates of the participants' results with respect to the level of addition (page 6, table 1) were calculated by DLA and given for information only.

Spiking Level Sample and Sample B

Evaluation number	Spiking Level Sample	Recovery rate*	Sample B	Recovery rate*	Remarks
	[mg/kg]	[%]	[mg/kg]	[%]	
1	81,5	**104**	87,5	**107**	
2	65,9	84	68,8	84	
3	46,0	59	64,0	78	
4	117	149	169	206	
5	71,1	**90**	78,6	**96**	
6					
7	67,0	**85**	77,0	**94**	
8	67,0	**85**	66,0	80	
9	69,1	**88**	73,0	**89**	
10	139	177	126	153	
11			89,6	**109**	
12	73,0	**93**	74,0	**90**	
13	72,0	**92**	76,0	**93**	
14	68,0	**87**	73,0	**89**	
15	159	202	76,0	**93**	
16	86,0	**109**	87,0	**106**	
17	72,0	**92**	76,0	**93**	
18	70,0	**89**	80,0	**98**	
19	60,0	76	65,0	79	

RA**	85-115 %	RA**	85-115 %
Number in RA	**11**	Number in RA	**12**
Number in RA	**65**	Number in RA	**67**

* Recovery rate 100% relative size: lactose, s. page 5

** Range of acceptance 3.12 (s. page 15)

Comments:

For the spiking level sample 65% of the participants obtained a recovery rate within the range of 85-115%. For the spiked sample B produced with the spiking level sample 67% of the recovery rates were in this range.

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	3
Number of outliers	1
Mean	145
Median	9,00
Robust Mean (X)	**23,2**
Robust standard deviation (S*)	
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	

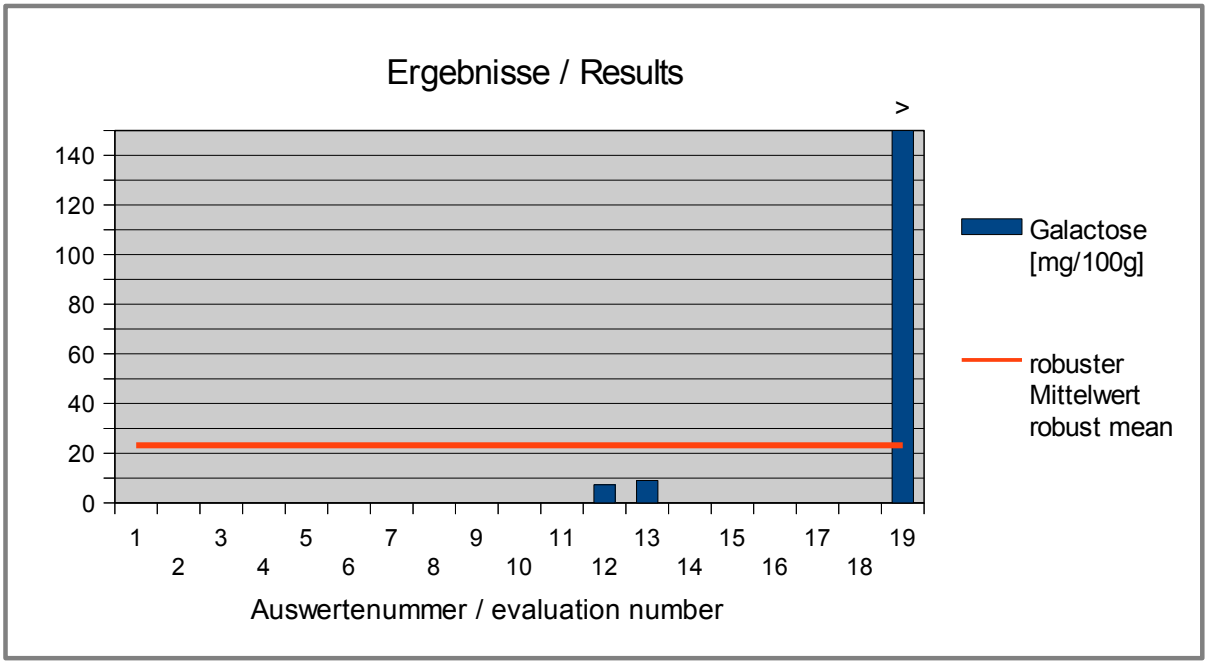

Abb. / Fig. 16: Ergebnisse Galactose / Results Galactose

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
1					
2					
3					
4					
5					
6					
7					
8					
9	<500				
10					
11					
12	7,30	-15,9			
13	9,00	-14,2			
14					
15	<LOQ				
16	< 10				
17	-				
18					
19	420	397			

4.3.2 Galactose Sample B (in mg/100g)

Vergleichsuntersuchung / Proficiency Test

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

Statistic Data	
Number of results	4
Number of outliers	1
Mean	100
Median	9,65
Robust Mean (X)	**13,9**
Robust standard deviation (S*)	
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	

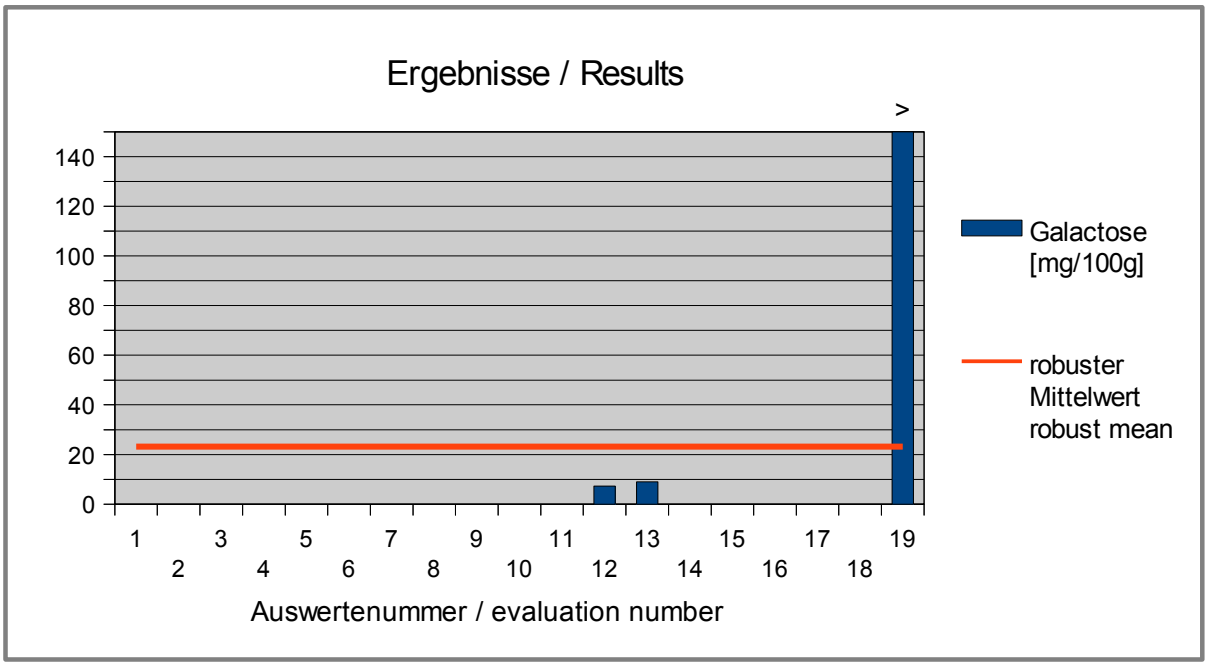

Abb. / Fig. 17: Ergebnisse Galactose / Results Galactose

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
1					
2					
3					
4					
5					
6					
7					
8					
9	<500				
10					
11					
12	8,30	-5,60			
13	11,0	-2,90			
14					
15	<LOQ				
16	< 10				
17	6,00	-7,90			
18					
19	375	361			

4.3.3 Galactose Spiking Level Sample (in mg/100g)

Vergleichsuntersuchung / Proficiency Test

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

Statistic Data	
Number of results	2
Number of outliers	0
Mean	37,9
Median	37,9
Robust Mean (X)	**37,9**
Robust standard deviation (S*)	
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	

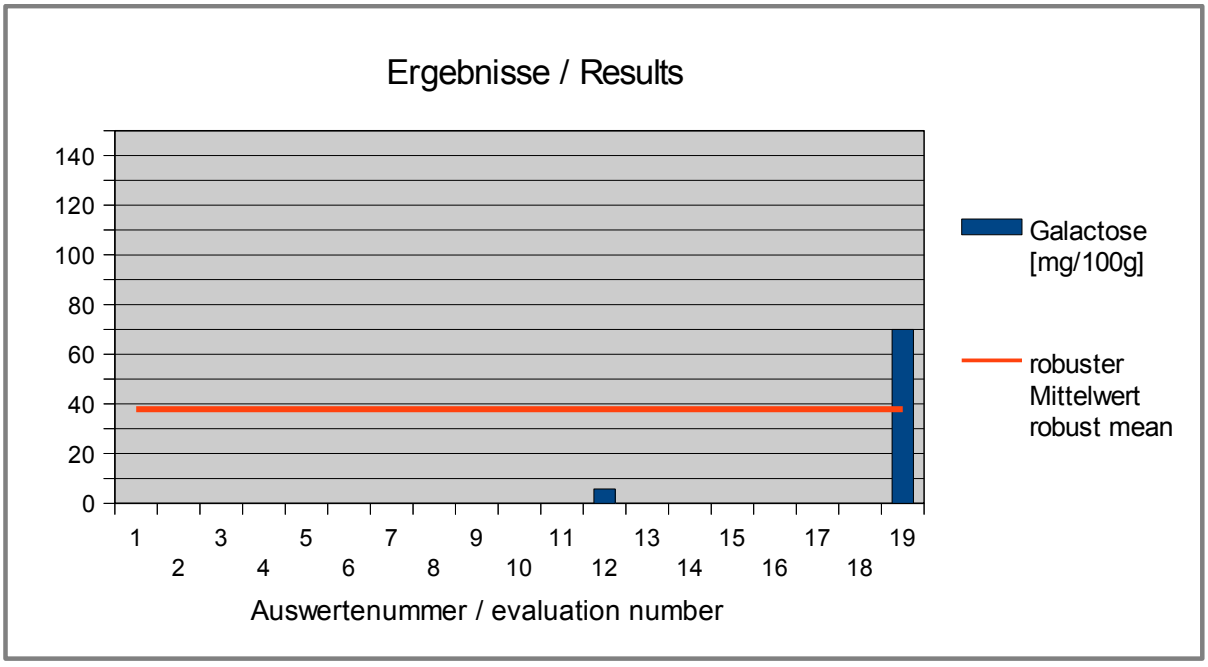

Abb. / Fig. 18: Ergebnisse Galactose / Results Galactose

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
1					
2					
3					
4					
5					
6					
7					
8					
9	<500				
10					
11					
12	5,80	-32,1			
13	<10				
14					
15	<LOQ				
16	< 20				
17					
18					
19	70,0	32,1			

5. Documentation

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

5.1 Details by the participants

5.1.1 Primary Data

Fructose Sample A

Parameter	Teilnehmer	Einheit	Proben-Nr.	Datum d. Analyse	Nach-weisbar	NWG	BG	Ergebnis	Inkl. WF	Wiederfin-dungsrate [%]
Analyte	Participant	Unit	Sample-No.	Date of ana-lysis	Detecta-ble	LOD	LOQ	Result	Incl. RR	Recovery rate [%]
Fructose	1	mg/100g	?							
	2	mg/100g	24							
	3	mg/100g	23		802	yes	100		no	
	4	mg/100g								
	5	mg/100g	49	42893	566	yes	2,7	9,3	no	omitted
	6	mg/100g								
	7	mg/100g	18-2017							
	8	mg/100g	35	09.+15.05.	255	yes	3	10	no	
	9	mg/100g	50	42870	1250	yes	-	50	no	-
	10	mg/100g	SAMPLE A							
	11	mg/100g	22							
	12	mg/100g	55		1010	yes		100	no	-
	13	mg/100g	38/18	31.5.	750	yes		100		
	14	mg/100g				no				
	15	mg/100g		08.05.	819	yes	10	30	no	
	16	mg/100g	31							
	17	mg/100g	57	02.05-15.05	1000	yes	110	120	no	-
	18	mg/100g	12							
	19	mg/100g	19	07. Jun	410	yes	5	10	-	-

Fructose Sample B

Parameter	Teilnehmer	Einheit	Proben-Nr.	Datum d. Analyse	Nach-weisbar	NWG	BG	Ergebnis	Inkl. WF	Wiederfin-dungsrate [%]
Analyte	Participant	Unit	Sample-No.	Date of ana-lysis	Detecta-ble	LOD	LOQ	Result	Incl. RR	Recovery rate [%]
Fructose	1	mg/100g	?							
	2	mg/100g	59							
	3	mg/100g	56		1264	yes	100		no	
	4	mg/100g								
	5	mg/100g	33	42893	852	yes	2,7	9,3	no	omitted
	6	mg/100g								
	7	mg/100g	18-2017							
	8	mg/100g	5	09.+15.05.	490	yes	3	10	no	
	9	mg/100g	4	42870	1025	yes	-	50	no	-
	10	mg/100g	SAMPLE B							
	11	mg/100g	12							
	12	mg/100g	61		1280	yes		100	no	-
	13	mg/100g	66/13	31.5.	1033	yes		100		
	14	mg/100g				no				
	15	mg/100g		08.05.	1096	yes	10	30	no	
	16	mg/100g	65							
	17	mg/100g	20	02.05-15.05	1200	yes	110	120	no	-
	18	mg/100g	2							
	19	mg/100g	70	07. Jun	675	yes	5	10	-	-

Fructose Spiking Level Sample

Parameter	Teilnehmer	Einheit	Proben-Nr.	Datum d. Analyse	Nach-weisbar	NWG	BG	Ergebnis	Inkl. WF	Wiederfin-dungsrate [%]
Analyte	**Participant**	**Unit**	**Sample-No.**	**Date of ana-lysis**	**Detecta-ble**	**LOD**	**LOQ**	**Result**	**Incl. RR**	**Recovery rate [%]**
Fructose	1	mg/100g	?							
	2	mg/100g	53							
	3	mg/100g	2							
	4	mg/100g								
	5	mg/100g	26	42893	221	yes	2,7	9,3	no	omitted
	6	mg/100g								
	7	mg/100g	18-2017							
	8	mg/100g	9	29.05.	147	yes	3	10	no	
	9	mg/100g	31	15. Mai	160	yes	-	50	no	-
	10	mg/100g	SPIKING LEVEL SAMPLE							
	11	mg/100g								
	12	mg/100g	37		226	yes		100	no	-
	13	mg/100g	43/08	31.5.	205	yes		100		
	14	mg/100g				no				
	15	mg/100g	18/2017	08.05.	226	yes	10	30	no	
	16	mg/100g	15							
	17	mg/100g	60	02.05-15.05	130	yes	110	120	no	-
	18	mg/100g	71							
	19	mg/100g	22	07. Jun	260	yes	5	10	-	-

Lactose Sample A

Parameter	Teilnehmer	Einheit	Proben-Nr.	Datum d. Analyse	Nach-weisbar	NWG	BG	Ergebnis	Inkl. WF	Wiederfin-dungsrate [%]
Analyte	Participant	Unit	Sample-No.	Date of ana-lysis	Detecta-ble	LOD	LOQ	Result	Incl. RR	Recovery rate [%]
Lactose	1	mg/100g	?	42870	11,7	yes	0,6 mg/100g	1,8 mg/100g	no	101,7
	2	mg/100g	24	42895	<2		2	5		
	3	mg/100g	23		<10	yes	10		no	
	4	mg/100g		42893	113	yes	3	5	no	
	5	mg/100g	49	42893	n.n.	no	4,1	14,8	no	omitted
	6	mg/100g		42884	2128,1	yes	300	300	no	
	7	mg/100g	18-2017	42885	<10	no				
	8	mg/100g	35	09.+15.05.	n.n.	no	3	10	no	
	9	mg/100g	50	42883	<2,0	no	-	2	no	-
	10	mg/100g	SAMPLE A	42894	<100	no		<100		
	11	mg/100g	22	42880	0	no		2	yes	99
	12	mg/100g	55	3.5.17 and 18.5.17	< 4	no		4	yes	102 to 101
	13	mg/100g	38/18	19.5.	<10	no		10		
	14	mg/100g		42881	< LQ	yes	0,5	1	no	98,9
	15	mg/100g		08.05.	<LOQ	no	10	30	no	
	16	mg/100g	31	31/05	< 10	yes	2	10	no	81
	17	mg/100g	57	02.05-15.05		no	7	10	no	-
	18	mg/100g	12	24.05./31.5.2017	<10	no	10	20	no	100,48
	19	mg/100g	19	07. Jun	<10	no	5	10	-	-

Lactose Sample B

Parameter	Teilnehmer	Einheit	Proben-Nr.	Datum d. Analyse	Nach-weisbar	NWG	BG	Ergebnis	Inkl. WF	Wiederfin-dungsrate [%]
Analyte	Participant	Unit	Sample-No.	Date of ana-lysis	Detecta-ble	LOD	LOQ	Result	Incl. RR	Recovery rate [%]
Lactose	1	mg/100g	?	42870	87,5	yes	0,6 mg/100g	1,8 mg/100g	no	101,7
	2	mg/100g	59	42895	68,8		2	5		
	3	mg/100g	56		64	yes	10		no	
	4	mg/100g		42893	169	yes	3	5	no	
	5	mg/100g	33	42893	78,6	yes	4,1	14,8	no	omitted
	6	mg/100g		42884	<300	yes	300	300	no	
	7	mg/100g	18-2017	42885	77	yes				
	8	mg/100g	5	29.05.	66	yes	3	10	no	
	9	mg/100g	4	42883	73	yes	-	2	no	-
	10	mg/100g	SAMPLE B	42894	125,51	yes		<100		
	11	mg/100g	12	42880	89,6	yes		2	yes	99
	12	mg/100g	61	3.5.17 and 18.5.17	74	yes		4	yes	102 to 101
	13	mg/100g	66/13	19.5.	76	yes		10		
	14	mg/100g		42881	73	yes	0,5	1	no	
	15	mg/100g		08.05.	76	yes	10	30	no	
	16	mg/100g	65	31/05	87	yes	2	10	no	125
	17	mg/100g	20	02.05-15.05	76	yes	7	10	no	-
	18	mg/100g	2	24.05./31.5.2017	80	yes	10	20	no	100,48
	19	mg/100g	70	07. Jun	65	yes	5	10	-	-

Lactose Spiking Level Sample

Parameter	Teilnehmer	Einheit	Proben-Nr.	Datum d. Analyse	Nach-weisbar	NWG	BG	Ergebnis	Inkl. WF	Wiederfin-dungsrate [%]
Analyte	**Participant**	**Unit**	**Sample-No.**	**Date of ana-lysis**	**Detecta-ble**	**LOD**	**LOQ**	**Result**	**Incl. RR**	**Recovery rate [%]**
Lactose	1	mg/100g	?	42870	81,5	yes	0,6 mg/100g	1,8 mg/100g	no	101,7
	2	mg/100g	53	42895	65,9					
	3	mg/100g	2		46	yes	10		no	
	4	mg/100g		42893	117	yes	3	5	no	
	5	mg/100g	26	42893	71,1	yes	4,1	14,8	no	omitted
	6	mg/100g		42884	5349,9	yes	300	300	no	
	7	mg/100g	18-2017	42885	67	yes				
	8	mg/100g	9	29.05.	67	yes	3	10	no	
	9	mg/100g	31	42883	69,1	yes	-	2	no	-
	10	mg/100g	SPIKING LEVEL SAMPLE	42894	138,83	yes		<100		
	11	mg/100g								
	12	mg/100g	37	3.5.17 and 18.5.17	73	yes		4	yes	102 to 101
	13	mg/100g	43/08	19.5.	72	yes		10		
	14	mg/100g		42881	68	yes	0,5	1	no	
	15	mg/100g	18/2017	06.06.	159	yes	10	30	no	
	16	mg/100g	15	31/05	86	yes	4	20	no	103
	17	mg/100g	60	02.05-15.05	72	yes	7	10	no	-
	18	mg/100g	71	24.05./31.5.2017	70	yes	10	20	no	100,48
	19	mg/100g	22	07. Jun	60	yes	5	10	-	-

Galactose Sample A

Parameter	Teilnehmer	Einheit	Proben-Nr.	Datum d. Analyse	Nach-weisbar	NWG	BG	Ergebnis	Inkl. WF	Wiederfin-dungsrate [%]
Analyte	Participant	Unit	Sample-No.	Date of ana-lysis	Detecta-ble	LOD	LOQ	Result	Incl. RR	Recovery rate [%]
Galactose	1	mg/100g	?							
	2	mg/100g	24							
	3	mg/100g	23							
	4	mg/100g								
	5	mg/100g	49							
	6	mg/100g								
	7	mg/100g	18-2017							
	8	mg/100g	35							
	9	mg/100g	50	42870	<500	no	-	500	no	-
	10	mg/100g	SAMPLE A							
	11	mg/100g	22							
	12	mg/100g	55		7,3	yes		2	yes	-
	13	mg/100g	38/18	19.5.	9	yes		10		
	14	mg/100g				no				
	15	mg/100g		08.05.	<LOQ	no	20	60	no	
	16	mg/100g	31	31/05	< 10	yes	2	10	no	
	17	mg/100g	57	02.05-15.05	-					-
	18	mg/100g	12							
	19	mg/100g	19	07. Jun	420	yes	5	10	-	-

Galactose Sample B

Parameter	Teilnehmer	Einheit	Proben-Nr.	Datum d. Analyse	Nach-weisbar	NWG	BG	Ergebnis	Inkl. WF	Wiederfin-dungsrate [%]
Analyte	Participant	Unit	Sample-No.	Date of ana-lysis	Detecta-ble	LOD	LOQ	Result	Incl. RR	Recovery rate [%]
Galactose	1	mg/100g	?							
	2	mg/100g	59							
	3	mg/100g	56							
	4	mg/100g								
	5	mg/100g	33							
	6	mg/100g								
	7	mg/100g	18-2017							
	8	mg/100g	5							
	9	mg/100g	4	42870	<500	no	-	500	no	-
	10	mg/100g	SAMPLE B							
	11	mg/100g	12							
	12	mg/100g	61		8,3	yes		2	yes	-
	13	mg/100g	66/13	19.5.	11	yes		10		
	14	mg/100g				no				
	15	mg/100g		08.05.	<LOQ	no	20	60	no	
	16	mg/100g	65	31/05	< 10	yes	2	10	no	
	17	mg/100g	20	02.05-15.05	6	yes	4	5	no	-
	18	mg/100g	2							
	19	mg/100g	70	07. Jun	375	yes	5	10	-	-

Galactose Spiking Level Sample

Parameter	Teilnehmer	Einheit	Proben-Nr.	Datum d. Analyse	Nach-weisbar	NWG	BG	Ergebnis	Inkl. WF	Wiederfin-dungsrate [%]
Analyte	Participant	Unit	Sample-No.	Date of ana-lysis	Detecta-ble	LOD	LOQ	Result	Incl. RR	Recovery rate [%]
Galactose	1	mg/100g	?							
	2	mg/100g	53							
	3	mg/100g	2							
	4	mg/100g								
	5	mg/100g	26							
	6	mg/100g								
	7	mg/100g	18-2017							
	8	mg/100g	9							
	9	mg/100g	31	42870	<500	no	-	500	no	-
	10	mg/100g	SPIKING LEVEL SAMPLE							
	11	mg/100g								
	12	mg/100g	37		5,8	yes		2	yes	-
	13	mg/100g	43/08	19.5.	<10	no		10		
	14	mg/100g				no				
	15	mg/100g	18/2017	06.06.	<LOQ	no	20	60	no	
	16	mg/100g	15	31/05	< 20	yes	4	20	no	
	17	mg/100g	60	02.05-15.05		no	4	5	no	-
	18	mg/100g	71							
	19	mg/100g	22	07. Jun	70	yes	5	10	-	-

5.1.2 Analytical Methods

Fructose Sample A

Parameter	Teil-nehmer	Methodenangabe wie in Prüfbericht/ Norm /Literatur	Probenvorbereitung	Messmethode	Kalibrierung/ Referenzmate-rial	Wiederfin-dung mit glei-cher Matrix	Methode ak-kreditiert	Sonstige Hinweise
Analyte	**Partici-pant**	**Method description as in test report / norm / literature**	**Sample preparation**	**Measuring method**	**Calibration / Re-ference materi-al**	**Recovery with same matrix**	**Method accredi-ted**	**Further remarks**
Fructose Probe A / Sample A	1							
	2							
	3			enzymatic			yes	
	4							
	5	Determination from lactose in lactose free products via ion-chromatography	1,0 g sample in weight, Carrez-treatment, 100 ml total volume, filtration	IC with amperometric Detection	External one point calibration	cancelled	no	
	6							
	7							
	8	Ion-chromatography	Prodution of aqueous extract, 70°C	IC with Dialysis, pulsed amperometric Detector	Sigma Aldrich, F0127-100G		yes	Limit of detection and limit of determination analyzed via matrixfree calibration solution
	9	HPAE-PAD - internal method PNTA0179			external calib. curve and internal RM	no	yes	
	10							
	11							
	12	enzymatic					yes	
	13	Thermo Fisher Scientific 984302, Spectrophotometry					yes	
	14							
	15	Enzymatic	Homogenization, aqueous extraction, Carrez-treatment, filtration		Standards from Enzym-Kit r-biopharm	no	yes	Sample A and B were significant inhomogenous
	16							
	17	RI detector, Zorbax Carbohydrate column	extraction, protein precipitation, filtration	HPLC	milkshake powder	no	yes	
	18							
	19	HPIEC-PAD	Ultra sonic-Extraction, 60°C	-	external	no	yes	-

Fructose Sample B

Parameter	Teil-nehmer	Methodenangabe wie in Prüfbericht/ Norm /Literatur	Probenvorbereitung	Messmethode	Kalibrierung/ Referenzmate-rial	Wiederfin-dung mit glei-cher Matrix	Methode ak-kreditiert	Sonstige Hinweise
Analyte	**Partici-pant**	**Method description as in test report / norm / literature**	**Sample preparation**	**Measuring method**	**Calibration / Re-ference materi-al**	**Recovery with same matrix**	**Method accredi-ted**	**Further remarks**
Fructose Probe B / Sample B	1							
	2							
	3			enzymatic			yes	
	4							
	5	Determination from lactose in lactose free products via ion-chromatography	1,0 g sample in w eight, Carrez-treatment, 100 ml total volume, filtration	IC w ith amperometric Detection	External one point calibration	cancelled	no	
	6							
	7							
	8	Ion-chromatography	Prodution of aqueous extract, 70°C	IC w ith Dialysis, pulsed amperometric Detector	Sigma Aldrich, F0127-100G		yes	Limit of detection and limit of determination analyzed via matrixfree calibration solution
	9	HPAE-PAD - internal method PNTA0179			external calib. curve and internal RM	no	yes	
	10							
	11							
	12	enzymatic					yes	
	13	Thermo Fisher Scientific 984302, Spectrophotometry					yes	
	14							
	15	Enzymatic	Homogenization, aqueous extraction, Carrez-treatment, filtration		Standards from Enzym-Kit r-biopharm	no	yes	Sample A and B w ere significant inhomogenous
	16							
	17	RI detector, Zorbax Carbohydrate column	extraction, protein precipitation, filtration	HPLC	milkshake pow der	no	yes	
	18							
	19	HPIEC-PAD	Ultra sonic-Extraction, 60°C	-	external	no	yes	-

Fructose Spiking Level Sample

Parameter	Teil-nehmer	Methodenangabe wie in Prüfbericht/ Norm /Literatur	Probenvorbereitung	Messmethode	Kalibrierung/ Referenzmate-rial	Wiederfin-dung mit glei-cher Matrix	Methode ak-kreditiert	Sonstige Hinweise
Analyte	**Partici-pant**	**Method description as in test report / norm / literature**	**Sample preparation**	**Measuring method**	**Calibration / Re-ference materi-al**	**Recovery with same matrix**	**Method accredi-ted**	**Further remarks**
Fructose Dotierungs niveauprob e / Spiking Level Sample	1							
	2							
	3			enzymatic			yes	
	4							
	5	Determination from lactose in lactose free products via ion-chromatography	1,0 g sample in w eight, Carrez-treatment, 100 ml total volume, filtration	IC w ith amperometric Detection	External one point calibration	cancelled	no	
	6							
	7							
	8	Ion-chromatography	Prodution of aqueous extract, 70°C	IC w ith Dialysis, pulsed amperometric Detector	Sigma Aldrich, F0127-100G		yes	Limit of detection and limit of determination analyzed via matrixfree calibration solution
	9	HPAE-PAD - internal method PNTA0179			external calib. curve and internal RM	no	yes	
	10							
	11							
	12	enzymatic					yes	
	13	Thermo Fisher Scientific 984302, Spectrophotometry					yes	
	14							
	15	Enzymatic	Homogenization, aqueous extraction, Carrez-treatment, filtration		Standards from Enzym-Kit r-biopharm	no	yes	Unusual small amount for a spiking level sample
	16							
	17	RI detector, Zorbax Carbohydrate column	extraction, protein precipitation, filtration	HPLC	milkshake pow der	no	yes	
	18							
	19	HPIEC-PAD	Ultra sonic-Extraction, 60°C	-	external	no	yes	-

Lactose Sample A

Parameter	Teil-nehmer	Methodenangabe wie in Prüfbericht/ Norm /Literatur	Probenvorbereitung	Messmethode	Kalibrierung/ Referenzmaterial	Wiederfindung mit gleicher Matrix	Methode akkreditiert	Sonstige Hinweise
Analyte	**Participant**	**Method description as in test report / norm / literature**	**Sample preparation**	**Measuring method**	**Calibration / Reference material**	**Recovery with same matrix**	**Method accredited**	**Further remarks**
Lactose Probe A /Sample A	1	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.	-	DS81 REF012 internal reference sample	no	no	-
	2					no		
	3			enzymatic			yes	
	4	enzimatic kit (spectrophotometer)		MPI25C		no	no	
	5	Determination from lactose in lactose free products via ion-chromatography	1,0 g sample in weight, Carrez-treatment, 100 ml total volume, filtration	IC with amperometric Detection	External calibration, Lactose as certified reference material Sigma Nr. 61339 BioUltra 99,5 %	cancelled	no	
	6	Enzyme	50'c Water Extraction and clean up	Megazyme Lactose/D-Galactose Kit	Internal Control	No	No	
	7	UV-Test r-biopharm 10176303035	as per kit instruction	as per kit instruction			yes	
	8	Ion-chromatography	Prodution of aqueous extract, 70°C	IC with Dialysis, pulsed amperometric Detector	Sigma Aldrich, 61339-25G		yes	Limit of detection and limit of determination analyzed via matrixfree calibration solution
	9	LC-MS/MS - internal method PNTA0189			external calib. curve and internal RM	no	no	
	10	HPLC-IR	1 g/ 20 mL				NO	
	11	HPLC-MS		recovery calculated by C13-Lactose internal standard	Anhydrous lactose (Fluka)	yes	yes	
	12	enzymatic				yes	yes	
	13	r-biopharm Test-Combination 10 176 303 035, Spektrophotometry					yes	
	14	Internal method (07(S189) rev0 2015)		LCMS	Sigma-Aldrich lot. BCBM2068V	yes	yes	
	15	Enzymatic	Homogenization, aqueous extraction, Carrez-treatment, filtration		Standards from Enzym-Kit r-biopharm	no	yes	Sample A and B were significat inhomogenous
	16	Enzymatic	5,0 g / 100 ml; 70 °C 15 Min., Carrez-treatment			yes	no	
	17	R-Biopharm enzymatic kits: glucose remover, lactose/d-glucose, lactose/d-galactose	extraction, protein precipitation, filtration	Spectrophotometry	1g/l lactose+25 g/l glucose	no	no	is not accr. for that matrix
	18	enzymatic, ASU L 01.00-17		Lactose/Galactose	Enzyme Fast Romer Kit	no	no	
	19	HPIEC-PAD	Ultra sonic-Extraction, 60°C	-	external	no	yes	-

Lactose Sample B

Parameter	Teil-nehmer	Methodenangabe wie in Prüfbericht/ Norm /Literatur	Probenvorbereitung	Messmethode	Kalibrierung/ Refe-renzmaterial	Wiederfindung mit gleicher Matrix	Methode ak-kreditiert	Sonstige Hinweise
Analyte	**Partici-pant**	**Method description as in test report / norm / literature**	**Sample preparation**	**Measuring method**	**Calibration / Refe-rence material**	**Recovery with same matrix**	**Method accre-dited**	**Further remarks**
Lactose Probe B /Sample B	1	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 pow dered beverages for aroma (PBA), and has been adapted and validated to enable the quantification of lactose in lactose-free infant formulae	Bring the w hole laboratory sample (original container) to room temperature and homogenise it by mixing. Take the test portion for analysis from the homogeneous test sample.	-	DS81 REF012 internal reference sample	no	no	-
	2					no		
	3			enzymatic			yes	
	4	enzimatic kit (spectrophotometer)		MPI25C		no	no	
	5	Determination from lactose in lactose free products via ion-chromatography	1,0 g sample in w eight, Carrez-treatment, 100 ml total volume, filtration	IC w ith amperometric Detection	External calibration, Lactose as certified reference material Sigma Nr. 61339 BioUltra 99,5 %	cancelled	no	
	6	Enzyme	50'c Water Extraction and clean up	Megazyme Lactose/D-Galactose Kit	Internal Control	No	No	
	7	UV-Test r-biopharm 10176303035	as per kit instruction	as per kit instruction			yes	
	8	Ion-chromatography	Prodution of aqueous extract, 70°C	IC w ith Dialysis, pulsed amperometric Detector	Sigma Aldrich, 61339-25G		yes	Limit of detection and limit of determination analyzed via matrixfree calibration solution
	9	LC-MS/MS - internal method PNTA0189			external calib. curve and internal RM	no	no	
	10	HPLC-IR	1 g/ 20 mL				NO	
	11	HPLC-MS		recovery calculated by C13-Lactose internal standard	Anhydrous lactose (Fluka)	yes	yes	
	12	enzymatic				yes	yes	
	13	r-biopharm Test-Combination 10 176 303 035, Spektrophotometry					yes	
	14	Internal method (07(S189) rev0 2015)		LCMS	Sigma-Aldrich lot. BCBM2068V	yes	yes	
	15	Enzymatic	Homogenization, aqueous extraction, Carrez-treatment, filtration		Standards from Enzym-Kit r-biopharm	no	yes	Sample A and B w ere significat inhomogenous
	16	Enzymatic	5,0 g / 100 ml; 70 °C 15 Min., Carrez-treatment			yes	no	
	17	R-Biopharm enzymatic kits: glucose remover, lactose/d-glucose, lactose/d-galactose	extraction, protein precipitation, filtration	Spectrophotometry	1g/l lactose+25 g/l glucose	no	no	is not accr. for that matrix
	18	enzymatic, ASU L 01.00-17		Lactose/Galactose	Enzyme Fast Romer Kit	no	no	
	19	HPIEC-PAD	Ultra sonic-Extraction, 60°C	-	external	no	yes	-

Lactose Spiking Level Sample

Parameter	Teil-nehmer	Methodenangabe wie in Prüfbericht/ Norm /Literatur	Probenvorbereitung	Messmethode	Kalibrierung/ Referenzmaterial	Wiederfindung mit gleicher Matrix	Methode akkreditiert	Sonstige Hinweise
Analyte	**Participant**	**Method description as in test report / norm / literature**	**Sample preparation**	**Measuring method**	**Calibration / Reference material**	**Recovery with same matrix**	**Method accredited**	**Further remarks**
Lactose Dotierungsniveauprobe / Spiking Level Sample	1	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.	-	DS81 REF012 internal reference sample	no	no	-
	2					no		
	3			enzymatic			yes	
	4	enzimatic kit (spectrophotometer)		MPI25C		no	no	
	5	Determination from lactose in lactose free products via ion-chromatography	1,0 g sample in weight, Carrez-treatment, 100 ml total volume, filtration	IC with amperometric Detection	External calibration, Lactose as certified reference material Sigma Nr. 61339 BioUltra 99,5 %	entfällt	no	
	6	Enzyme	50'c Water Extraction and clean up	Megazyme Lactose/D-Galactose Kit	Internal Control	no	no	
	7	UV-Test r-biopharm 10176303035	as per kit instruction	as per kit instruction			yes	
	8	Ion-chromatography	Prodution of aqueous extract, 70°C	IC with Dialysis, pulsed amperometric Detector	Sigma Aldrich, 61339-25G		yes	Limit of detection and limit of determination analyzed via matrixfree calibration solution
	9	LC-MS/MS - internal method PNTA0189			external calib. curve and internal RM	no	no	
	10	HPLC-IR	1 g/ 20 mL				no	
	11	HPLC-MS		recovery calculated by C13-Lactose internal standard	Anhydrous lactose (Fluka)	yes	yes	
	12	enzymatic				yes	yes	
	13	r-biopharm Test-Combination 10 176 303 035, Spektrophotometry					yes	
	14	Internal method (07(S189) rev0 2015)		LCMS	Sigma-Aldrich lot. BCBM2068V	no	yes	
	15	Enzymatic	Homogenization, aqueous extraction, Carrez-treatment, filtration		Standards from Enzym-Kit r-biopharm	no	yes	Unusual small amount for a spiking level sample
	16	Enzymatic	2,5 g / 100 ml; 70 °C 15 Min., Carrez-treatment			yes	no	
	17	R-Biopharm enzymatic kits: glucose remover, lactose/d-glucose, lactose/d-galactose	extraction, protein precipitation, filtration	Spectrophotometry	1g/l lactose+25 g/l glucose	no	no	is not accr. for that matrix
	18	enzymatic, ASU L 01.00-17		Lactose/Galactose	Enzyme Fast Romer Kit		no	
	19	HPIEC-PAD	Ultra sonic-Extraction, 60°C	-	external	no	yes	-

Galactose Sample A

Parameter	Teil-nehmer	Methodenangabe wie in Prüfbericht/ Norm /Literatur	Probenvorbereitung	Messmethode	Kalibrierung/ Referenzmate-rial	Wiederfin-dung mit glei-cher Matrix	Methode ak-kreditiert	Sonstige Hinweise
Analyte	Partici-pant	Method description as in test report / norm / literature	Sample preparation	Measuring method	Calibration / Re-ference materi-al	Recovery with same matrix	Method accredi-ted	Further remarks
Galactose Probe A / Sample A	1							
	2							
	3							
	4							
	5							
	6							
	7							
	8							
	9	HPAE-PAD - internal method PNTA0149			external calib. curve and internal RM	no	no	
	10							
	11							
	12	enzymatic				yes	yes	
	13	r-biopharm Test-Combination 10 176 303 035, Spektrophotometry					yes	
	14							
	15	Enzymatic	Homogenization, aqueous extraction, Carrez-treatment, filtration		Standards from Enzym-Kit r-biopharm	no	yes	Sample A and B were significat inhomogenous
	16	Enzymatic	5,0 g / 100 ml; 70 °C 15 Min., Carrez-treatment					
	17	R-Biopharm enzymatic kits: glucose remover, lactose/d-galactose	extraction, protein precipitation, filtration	Spectrophotometry	0,506 g/l lactose	no	no	
	18							
	19	HPIEC-PAD	Ultra sonic-Extraction, 60°C	-	external	no	yes	-

Galactose Sample B

Parameter	Teil-nehmer	Methodenangabe wie in Prüfbericht/ Norm /Literatur	Probenvorbereitung	Messmethode	Kalibrierung/ Referenzmate-rial	Wiederfin-dung mit glei-cher Matrix	Methode ak-kreditiert	Sonstige Hinweise
Analyte	**Partici-pant**	**Method description as in test report / norm / literature**	**Sample preparation**	**Measuring method**	**Calibration / Re-ference materi-al**	**Recovery with same matrix**	**Method accredi-ted**	**Further remarks**
Galactose Probe B / Sample B	1							
	2							
	3							
	4							
	5							
	6							
	7							
	8							
	9	HPAE-PAD - internal method PNTA0149			external calib. curve and internal RM	no	no	
	10							
	11							
	12	enzymatic				yes	yes	
	13	r-biopharm Test-Combination 10 176 303 035, Spektrophotometry					yes	
	14							
	15	Enzymatic	Homogenization, aqueous extraction, Carrez-treatment, filtration		Standards from Enzym-Kit r-biopharm	no	yes	Sample A and B w ere significat inhomogenous
	16	Enzymatic	5,0 g / 100 ml; 70 °C 15 Min., Carrez-treatment					
	17	R-Biopharm enzymatic kits: glucose remover, lactose/d-galactose	extraction, protein precipitation, filtration	Spectrophotometry	0,506 g/l lactose	no	no	
	18							
	19	HPIEC-PAD	Ultra sonic-Extraction, 60°C	-	external	no	yes	-

Galactose Spiking Level Sample

Parameter	Teil-nehmer	Methodenangabe wie in Prüfbericht/ Norm /Literatur	Probenvorbereitung	Messmethode	Kalibrierung/ Referenzmate-rial	Wiederfin-dung mit glei-cher Matrix	Methode ak-kreditiert	Sonstige Hinweise
Analyte	**Partici-pant**	**Method description as in test report / norm / literature**	**Sample preparation**	**Measuring method**	**Calibration / Re-ference materi-al**	**Recovery with same matrix**	**Method accredi-ted**	**Further remarks**
Galactose Dotierungs niveauprob e / Spiking Level Sample	1							
	2							
	3							
	4							
	5							
	6							
	7							
	8							
	9	HPAE-PAD - internal method PNTA0149			external calib. curve and internal RM	no	no	
	10							
	11							
	12	enzymatic				yes	yes	
	13	r-biopharm Test-Combination 10 176 303 035, Spektrophotometry					yes	
	14							
	15	Enzymatic	Homogenization, aqueous extraction, Carrez-treatment, filtration		Standards from Enzym-Kit r-biopharm	no	yes	Unusual small amount for a spiking level sample
	16	Enzymatic	2,5 g / 100 ml; 70 °C 15 Min., Carrez-treatment					
	17	R-Biopharm enzymatic kits: glucose remover, lactose/d-galactose	extraktion, protein fällung, filtration	Spectrophotometry	0,506 g/l lactose	no	no	
	18							
	19	HPIEC-PAD	Ultra sonic-Extraction, 60°C	-	external	no	yes	-

5.2 Homogeneity

5.2.1 Mixture homogeneity before bottling

Microtracer Homogenitätstest
DLA 18-2017 Sample A

Weight whole sample	1,79	kg
Microtracer	FSS-rot lake	
Particle size	75 – 300	µm
Weight per particle	2,0	µg
Addition of tracer	19,4	mg/kg

Result of analysis

Sample	Weight [g]	Particle number	Particles [mg/kg]
1	5,06	40	15,8
2	5,15	36	14,0
3	5,05	49	19,4
4	5,00	56	22,4
5	5,03	49	19,5
6	4,98	40	16,1
7	5,06	43	17,0
8	5,09	61	24,0

Poisson distribution		
Number of samples	8	
Degree of freedom	7	
Mean	46,8	Particle
Standard deviation	8,69	Particle
χ^2 (CHI-Quadrat)	11,29	
Probability	**13**	%
Recovery rate	95	%

Normal distribution		
Number of samples	8	
Mean	18,5	mg/kg
Standard deviation	3,44	mg/kg
rel. Standard deviaton	18,6	%
Horwitz standard deviation	10,3	%
HorRat-value	**1,8**	
Recovery rate	95	%

Microtracer Homogenitätstest
DLA 18-2017 Sample B

Weight whole sample	2,24	kg
Microtracer	FSS-rot lake	
Particle size	75 – 300	µm
Weight per particle	2,0	µg
Addition of tracer	17,7	mg/kg

Result of analysis

Sample	Weight [g]	Particle number	Particles [mg/kg]
1	5,32	67	25,2
2	4,93	53	21,5
3	5,83	69	23,7
4	5,65	70	24,8
5	4,99	68	27,3
6	5,23	63	24,1
7	5,65	65	23,0
8	5,27	54	20,5

Poisson distribution		
Number of samples	8	
Degree of freedom	7	
Mean	63,6	Particle
Standard deviation	5,71	Particle
χ^2 (CHI-Quadrat)	3,58	
Probability	**83**	%
Recovery rate	134	%

Normal distribution		
Number of samples	8	
Mean	23,7	mg/kg
Standard deviation	2,13	mg/kg
rel. Standard deviaton	8,97	%
Horwitz standard deviation	9,93	%
HorRat-value	**0,90**	
Recovery rate	134	%

Microtracer Homogenitätstest
DLA 18-2017 Spiking Level Sample

Weight whole sample	2,08	kg
Microtracer	FSS-rot lake	
Particle size	75 – 300	µm
Weight per particle	2,0	µg
Addition of tracer	18,1	mg/kg

Result of analysis

Sample	Weight [g]	Particle number	Particles [mg/kg]
1	5,59	62	22,2
2	5,62	72	25,6
3	5,58	69	24,7
4	5,71	72	25,2
5	5,45	77	28,3
6	5,70	65	22,8
7	5,39	62	23,0
8	5,61	68	24,2

Poisson distribution		
Number of samples	8	
Degree of freedom	7	
Mean	68,4	Particle
Standard deviation	5,43	Particle
χ^2 (CHI-Quadrat)	3,02	
Probability	**88**	%
Recovery rate	135	%

Normal distribution		
Number of samples	8	
Mean	24,5	mg/kg
Standard deviation	1,95	mg/kg
rel. Standard deviaton	7,94	%
Horwitz standard deviation	9,89	%
HorRat-value	**0,80**	
Recovery rate	135	%

5.2.2 Comparison of sample numbers / test results and trend line

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

Lactose Sample B				
Target standard deviation σ_{pt}	5,51			mg/100g
Sample numbers	4 – 71			
Total numbers of samples	16			
Slope	-0,276			
Trend line range	78,1	–	73,6	mg/100g
Deviation trend line	75,9	±	2,21	mg/100g
Percent of σpt	40,1	%		

* without results with z-scores > |3,0|

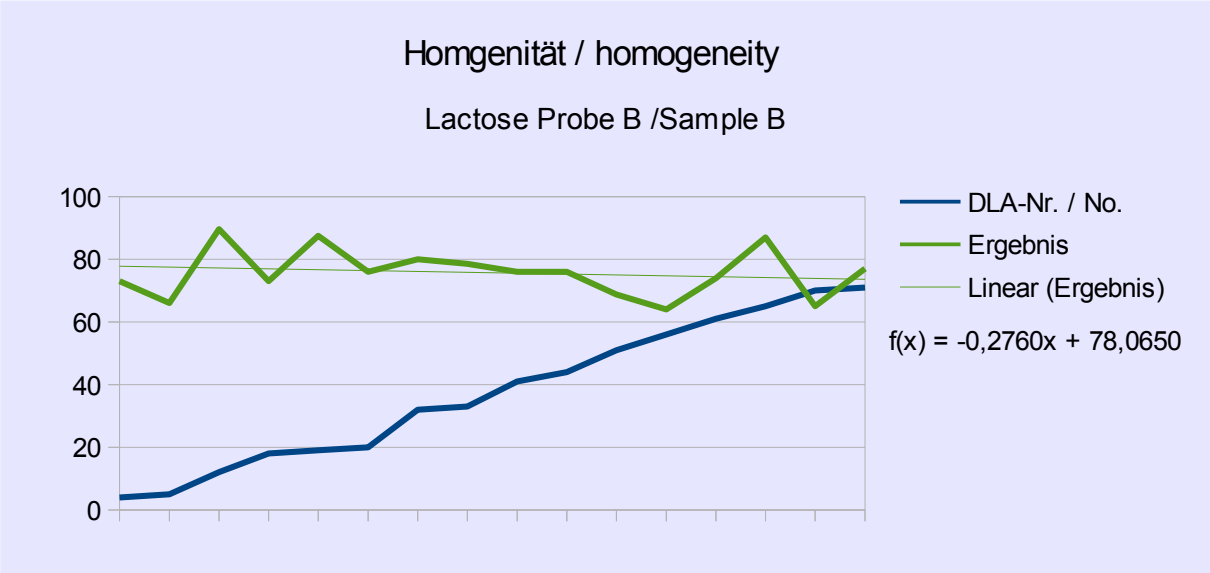

Abb./Fig. 19:
Trendfunktion Probennummern vs. Ergebnisse
trend line function sample number vs. results

Lactose Spiking Level Sample				
Target standard deviation σ_{pt}	5,88			mg/100g
Sample numbers	9 - 71			
Total numbers of samples	13			
Slope	-0,236			
Trend line range	72,6	-	69,6	mg/100g
Deviation trend line	71,1	±	1,53	mg/100g
Percent of σpt	26,1	%		

* without results with z-scores > |3,0|

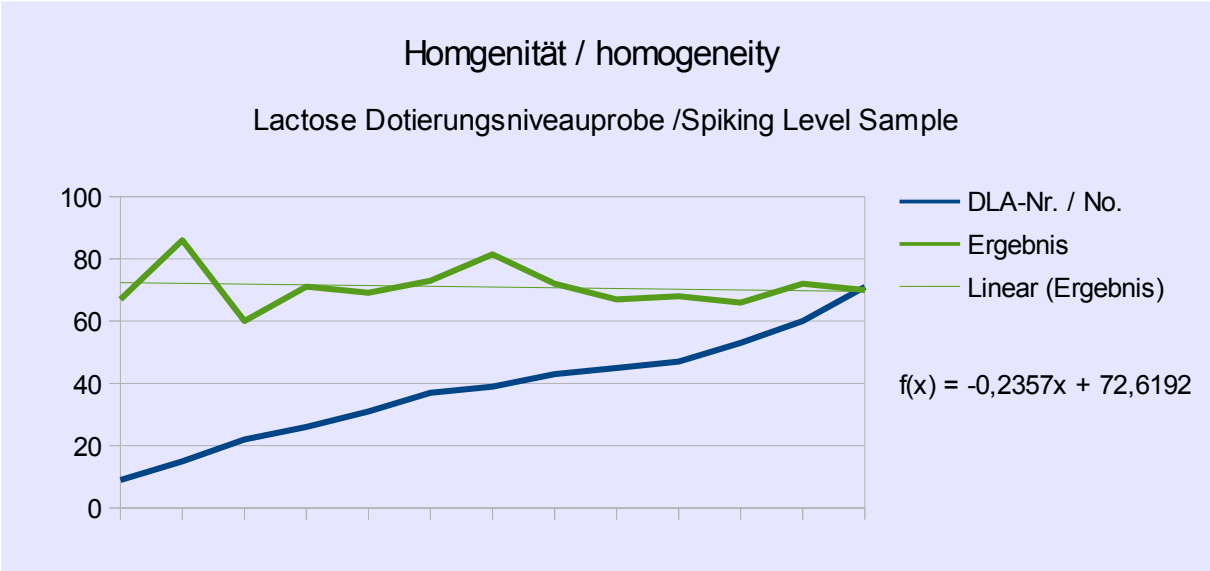

Abb./Fig. 20:
Trendfunktion Probennummern vs. Ergebnisse
trend line function sample number vs. Results

5.3 Information on the Proficiency Test (PT)

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

PT number	***DLA 18-2017***
PT name	***Lactose + Fructose in "lactose-free" food with "Spiking Level Sample“***
*Sample matrix**	***Samples A + B:*** *“Lactose-free” bread baking mixture white bread (powder)/ ingredients: wheat flour, salt, dry yeast, glucose, sugar, emulsifier: 481, malt extract (barley, rye), flour treatment agent: ascorbic acid as well as lactose and fructose foods (one of both samples)* ***Spiking Level Sample:*** *potato powder, potato flour, lactose and fructose*
Number of samples and sample amount	*2 different samples A + B, 25 g each and* *one spiking level sample 25 g*
Storage	*Samples A + B: room temperature (long term 2 - 10°C)* *Spiking Level Sample: room temperature*
Intentional use	*Laboratory use only (quality control samples)*
Parameter	*qualitative + quantitative: Lactose (optional: Galactose) + Fructose* *Proben A + B: Lactose < 500 mg/100g* *Dotierungsniveauprobe: Lactose < 500 mg/100g*
Methods of analysis	*Analytical methods are optional*
Notes to analysis	*The analysis of PT samples should be performed like a routine laboratory analysis.* *In general we recommend to homogenize a representative sample amount before analysis according to good laboratory practice, especially in case of low sample weights. Preferably the total sample amount should be homogenized.*
Result sheet	*One result each should be determined for Samples A and B and the Spiking Level Sample for submission.* *The results should be filled in the result submission file. In case of several determinations the mean.*
Units	*mg/100 g*
Number of significant digits	*at least 2*
Further information	*Further remarks should be provided in the result submission file for information for the PT-provider and participants.*
Result submission	*The result submission file should be sent by e-mail to:* ***pt@dla-lvu.de***
Deadline	***the latest June 09th 2017***
Evaluation report	*The evaluation report is expected to be completed 6 weeks after deadline of result submission and sent as PDF file by e-mail.*
Coordinator and contact person of PT	*Dr. Matthias Besler*

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

6. Index of participant laboratories in alphabetical order

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

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

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

7. Index of references

1. DIN EN ISO/IEC 17025:2005; Allgemeine Anforderungen an die Kompetenz von Prüf- und Kalibrierlaboratorien / General requirements for the competence of testing and calibration laboratories
2. DIN EN ISO/IEC 17043:2010; Konformitätsbewertung – Allgemeine Anforderungen an Eignungsprüfungen / Conformity assessment – General requirements for proficiency testing
3. ISO 13528:2015 & DIN ISO 13528:2009; Statistische Verfahren für Eignungsprüfungen durch Ringversuche / Statistical methods for use in proficiency testing by interlaboratory comparisons
4. ASU §64 LFGB: Planung und statistische Auswertung von Ringversuchen zur Methodenvalidierung / DIN ISO 5725 series part 1, 2 and 6 Accuracy (trueness and precision) of measurement methods and results
5. Verordnung / Regulation 882/2004/EU; Verordnung über über amtliche Kontrollen zur Überprüfung der Einhaltung des Lebensmittel- und Futtermittelrechts sowie der Bestimmungen über Tiergesundheit und Tierschutz / Regulation on official controls performed to ensure the verification of compliance with feed and food law, animal health and animal welfare rules
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16. ASU §64 LFGB L 01.00-17 (2010) / DIN 10344 : Bestimmung des Lactose- und Galactosegehaltes von Milch und Milchprodukten; Enzymatisches Verfahren / Milk and milk products - Determination of lactose and D-galactose content - Enzymatic method
17. ASU §64 LFGB L 01.00-90 Bestimmung des Lactosegehaltes in lactosereduzierter Milch und lactosereduzierten Milchprodukten in Gegenwart von Glucose; Enzymatisches Verfahren (2014) [Milk and milk products - Determination of lactose in lactose-reduced milk products in the presence of glucose - Enzymatic method]
18. ASU §64 LFGB L 17.00-7 Bestimmung von Lactose in Brot einschließlich Kleingebäck aus Brotteigen (1983) [Determination of lactose in bread including small pastries from bread doughs]
19. ASU §64 LFGB L 48.01-4 Bestimmung von Lactose in teiladaptierter Säuglingsnahrung auf Milchbasis (1985) [Determination of lactose in partially-adapted infant milk-based food]
20. ASU §64 LFGB L 48.02.07-1 Bestimmung von Glucose und Fructose in Kinder-Zwieback und Zwiebackmehl (1985) [Determination of glucose and fructose in children's rusk and rusk flour]
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 liquid chromatography (Reference method)