Evaluation Report
proficiency test

DLA ptAI01 (2021)

Lactose and Fructose

in “lactose free” Food - Cereal Pap Powder (Infant Food)

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1st Correction 01/09/2021:
The following errors were corrected in the present evaluation:

For participants 9 and 17, the qualitative results for sample A (lactose negative) were added on page 25.
On page 25 the units of quantitative results in the table heading were incorrect, they were corrected to “mg/100g”.
For participant 9, the calculation of the recovery rate including the z-score for sample B was added on page 32. The z-Score was added on page 41.
In the evaluation of fructose (spiking level sample), an incorrect value was used for participant 6 by mistake. The correctly transmitted result has been added to Figures 5 and 6 (p. 23) and to the tables on pages 24 and 44.

The corrections do not affect the evaluations of other participant results.

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

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EP-Nummer *PT-Number*	DLA ptAI01 (2021)
EP-Koordinator *PT-Coordinator*	Alexandra Scharf MSc.
Status des EP-Bericht *Status of PT-Report*	Abschlussbericht / Final report (1. September 2021) 1. Korrektur / 1st Correction Gültig ist die jeweils letzte Version/Korrektur des Berichts. Sie ersetzt alle vorangegangenen Versionen. Only the latest version/correction of the report is valid. It replaces all preceding versions.
EP-Bericht Freigabe *PT-Report Authorization*	Dr. Matthias Besler-Scharf (Technischer Leiter / Technical Manager) *- gezeichnet / signed M. Besler-Scharf* Alexandra Scharf MSc. (QM-Beauftragte / Quality Manager) *- gezeichnet / signed A. Scharf* Datum / Date: 1. September 2021
Unteraufträge *Subcontractors*	Im Rahmen dieser Eignungsprüfung wurden nachstehende Leistungen im Unterauftrag vergeben: Keine As part of the present proficency test the following services were subcontracted: none
Vertraulichkeit *Confidentiality*	Die Teilnehmerergebnisse sind im EP-Bericht in anonymisierter Form mit Auswertenummern benannt. Daten einzelner Teilnehmer werden ausschließlich nach vorheriger Zustimmung des Teilnehmers an Dritte weitergegeben. Participant result are named anonymously with evaluation numbers in the PT report. Data of individual participants will be passed on to third parties only with prior consent of the participant.

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1. Introduction

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

2. Realisation

2.1 Test material

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 is a mixture of common in commerce infant food products "cereal pap" for children with additional maize flour. The basic composition of both samples A and B was the same (see table 1).
After crushing and sieving by means of an impact mill (mesh 1,5 mm) the basic mixture was homogenized.
Afterwards the **spiked sample B** was produced as follows:
The spiking materials lactose and fructose were sieved by means of a centrifugal mill (mesh 250 µm), added to an aliquot of the basic mixture and the mixture was homogenized. Subsequently, the basic mixture was again added in additional steps and homogenized in each case until the total quantity had been reached.
For the **spiking level sample**, the spiking materials above mentioned were added during a multi-stage addition of potato powder (mesh 500 µm) and homogenized at each stage.

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

The composition of the PT samples is shown in Table 1.

Table 1: Composition of DLA-Samples

Ingredients	**Sample A**	**Sample B**	**Spiking Level Sample**
Organic-Cereal-Pap Mixture, infant pap after 4th month Ingredients: *whole grain oat flour, whole grain millet flour, rice semolina, maize flour, thiamine* Nutrients per 100g: Fat 4,3 g, carbohydrates 73 g, sugar 0,7 g, fiber 6,2 g, protein 11 g	100 g/100 g	99,5 g/100g	-
Potato powder Ingredients: Potatoes, E471, E304, E223, E100	-	-	99,5 g/100 g
Lactose*	-	180 mg/100g	176 mg/100g
Fructose*	-	34,5 mg/100g	333 mg/100g

*All contents according to gravimetric 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 B and the spiking level sample showed a probability of 75% and 92%. Additionally particle number results were converted into concentrations, statistically evaluated according to normal distribution and compared to the standard deviation according to Horwitz. For the assessment HorRat values between 0,3 and 1,3 are to be accepted under repeat conditions (measurements within the laboratory) [17].
This gave a HorRat value of 0,98 and 0,84 respectively. The results of microtracer analysis are given in the documentation.

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

2.1.2 Stability

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

The experience with various DLA test materials showed good storage stability with respect to the durability of the sample (spoilage) and the content of the PT parameters for comparable food matrices and water activity (a_W value <0,5).
The a_W value of the EP samples was approx. 0,24 (20°C) and 0,34 (18°). The stability of the sample material was thus ensured during the investigation period under the specified storage conditions.

2.2 Sample shipment and information to the test

The portions of test materials sample A, B and spiking level sample were sent to every participating laboratory in the 16th week of 2021. The testing method was optional. The tests should be finished at 18th June 2021 the latest (extended).

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 labelling (of lactose) of mg/100g in the **matrix** of **Cereal Pap Powder** (lactose-free). 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**. The spiking level sample should be analysed like a regular sample.*

Please note the attached information on the proficiency test.
(see documentation, section 5.3 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).
Queried and documented were the indicated results and details of the test methods like specificity, test kit manufacturer and hints about the procedure.
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 19 registered participants submitted at least one result. One 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]. If there are < 12 quantitative results and an increased difference between robust mean and median, the median may be used as the assigned value (criterion: Δ median - rob. mean > 0,3 σ_{pt}) [3].
The condition is that the majority of the participants' results show a normal distribution or are distributed unimodal and symmetrically. To this end, an examination of the distribution is carried out, inter alia, using the kernel density estimate [3, 12].
In case there are indications for sources of higher variability such as a bimodal distribution of results, a cause analysis is performed. Frequently different analytical methods may cause an anomaly in results' distribution. If this is the case, separate evaluations with own assigned values (X_{pti}) are made whenever possible.
The evaluation is usually carried out starting from 7 results, in justified cases a valuation is also allowed from 5 results.
The actual measurement results will be drafted. Individual results, which are outside the specified measurement range of the participating laboratory (for example with the result > 25 mg/kg or < 2,5 mg/kg) or the indicating "0" will not be considered for the statistic evaluation [3].

3.2 Robust standard deviation

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

3.3 Repeatability standard deviation

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

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

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

3.4 Reproducibility standard deviation

The reproducibility standard deviation S_R represents a inter-laboratory estimate of the standard deviation for the determination of each parameter on the bases of (outlier free) individual participant results. It takes into account both the repeatability standard deviation S_r and the within-laboratory standard deviation S_S. Reproducibility standard deviations of PTs 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 given as the coefficient of variation CV_R in the statistical characteristics in the results section, provided that the individual results of the participants are available, and the meaning is explained in more detail under 3.9.

3.5 Exclusion of results and outliers

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

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

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

3.6 Target standard deviation (for proficiency assessment)

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

If an acceptable quotient S^*/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.

In the present PT for evaluation of the results of the parameter fructose the target standard deviation according to the general model of Horwitz was applied (see 3.6.1).

For the parameters lactose and galactose the target standard deviation from the evaluation of a precision experiment (see 3.6.2) was used (ASU §64 Method: L 01.00-90, [19]).

Additionally for the evaluation of fructose (samples A, B and spiking level sample) and galactose (spiking level sample) the standard uncertainty was considered and the results were evaluated by z´-score (see 3.8).

3.6.1 General model (Horwitz)

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

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

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

3.6.2 Value by precision experiment

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

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

The relative repeatability standard deviations (RSD_r) and relative reproducibility standard deviations (RSD_R) given in table 2 were obtained in precision experiments by 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} [18-23]

Parameter	**Matrix**	**Mean** [g/100g]	**RSD_r**	**RSD_R**	**σ_{pt}**	**Method / Literature**
Fructose	Rusk	7,0%	1,59%	2,59%	2,33%[1]	ASU §64 L 48.02.07-1
Lactose	Baby food	28,7%	1,66%	3,33%	3,12%	ASU §64 L 48.02.07-1
Lactose	"lactose free" skimmed Milk	0,13%	20%	30%	26,5%	ASU §64 L 01.00-17
Lactose	"lactose free" Milk (3 samples)	0,0282% 0,0804% 0,1257%	6,74% 1,71% 6,25%	10,9% 3,95% 7,33%	9,76%[1] 3,76% 5,85%[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] *values used or given for information in the evaluation (s. section 4), for lactose and galactose calculated from means of the standard deviations (7,85%)*

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].

In the present PT, the target standard deviations of 3.6.1. and 3.6.2 were considered suitable.

Table 3 shows selected statistic data of participants results of the present PT compared to PT results of previous years.

Table 3: Characteristics of the present PT (on grey) in comparison to previous PTs since 2016 (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	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)
Fructose	Cereal pap powder	544	41,3	7,6	1,7	DLA 18/2018 (Sample A)
Fructose	Cake baking mixture	525	38,1	7,3	1,6	DLA 18/2019 (Sample B)
Fructose	Cookies	2390	506	21,2	2,5*	DLA ptAI01 2020 (Probe B)
Fructose	Cereal pap powder	102	38,7	37,9	2,0*	DLA ptAI01 2021 (Probe 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)
Lactose	Cereal pap powder	289	29,2	10,1	1,3	DLA 18/2018 (Sample A)
Lactose	Cake baking mixture	104	13,1	12,6	1,6	DLA 18/2019 (Sample B)
Lactose	Cookies	209	35,2	16,8	1,9*	DLA ptAI01 2020 (Probe B)
Lactose	Cereal pap powder	172	15,1	8,8	1,1	DLA ptAI01 2021 (Probe B)

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

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 z-score valid for the proficiency test is called z-score (σ_{pt}) in the evaluation, while the value called z-score (info) is purely informative. The two z scores are calculated with the different target standard deviations according to 3.6.

3.7.1 Warning and action signals

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

An error or cause analysis can be carried out by checking the analysis process including understanding and implementation of the measurement by the staff, details of the measurement procedure, calibration of equipment and composition of reagents, transmission or calculation errors, trueness and precision and use of reference material. 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. 3.11). The z'-score represents the relation of the deviation of the result (x_i) of the participant from the respective consensus value (X) to the square root of quadrat sum of the target standard deviation (σ_{pt}) and the standard uncertainty (Ux_{pt}) [3].

The calculation is performed by:

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

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

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

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

For warning and action signals see 3.7.1.

3.9 Reproducibility coefficient of variation (CV)

The variation coefficient (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 CV_R 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 levels or the performance evaluation of the participating laboratories possibly can not be done [3].

3.10 Quotient S*/σpt

Following the HorRat-value the results of a proficiency-test 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 and traceability

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

3.12 Recovery rates: Spiking

For the lactose 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 1. 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 [18-23]. The calculation of the associated z-scores was carried out according to 3.5 with the target standard deviation of 7,5%

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^x)
Target range:
Target standard deviation σ_{pt} or σ_{pt}'
Target standard deviation for information
lower limit of target range ($X_{pt} - 2\sigma_{pt}$) or ($X_{pt} - 2\sigma_{pt}$') *
upper limit of target range ($X_{pt} + 2\sigma_{pt}$) or ($X_{pt} + 2\sigma_{pt}$´) *
Quotient S/σ_{pt} or S*/σ_{pt}'*
Standard uncertainty U(X_{pt})
Number of results in the target range
Percent in the target range

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

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

Auswerte-nummer / **Evaluation number**	**Parameter [Einheit / Unit]**	**Abweichung** / **Deviation**	**z-Score σpt**	**z-Score** (Info)	**Hinweis** / **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	6
Number of outliers	0
Mean	65,7
Median	61,7
Robust Mean (X_{pt})	**65,7**
Robust standard deviation (S*)	**20,6**
Target range:	
Target standard deviation σ_{pt}'	**11,2**
Target standard deviation (for Information)	1,53
lower limit of target range	**43,2**
upper limit of target range	**88,1**
Quotient S/σ_{pt}'*	*1,8*
Standard uncertainty $U_{(Xpt)}$	*10*
Results in the target range	*4*
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, [22]) is given for information (s. 3.6.2).

The distribution of results showed an increased variability. The quotient S^*/σ_{pt} was above 2. Therefore the valuation was done by z'-scores considering the standard uncertainty. The quotient S^*/σ_{pt}' was then 1,8. The robust standard deviation was in the range of previous PTs (see 3.6.3). The comparability of results is given.

67% of results were in the target range.

Fructose was not added to sample A, the fructose content comes from the ingredients of the basic matrix (s. p. 5).

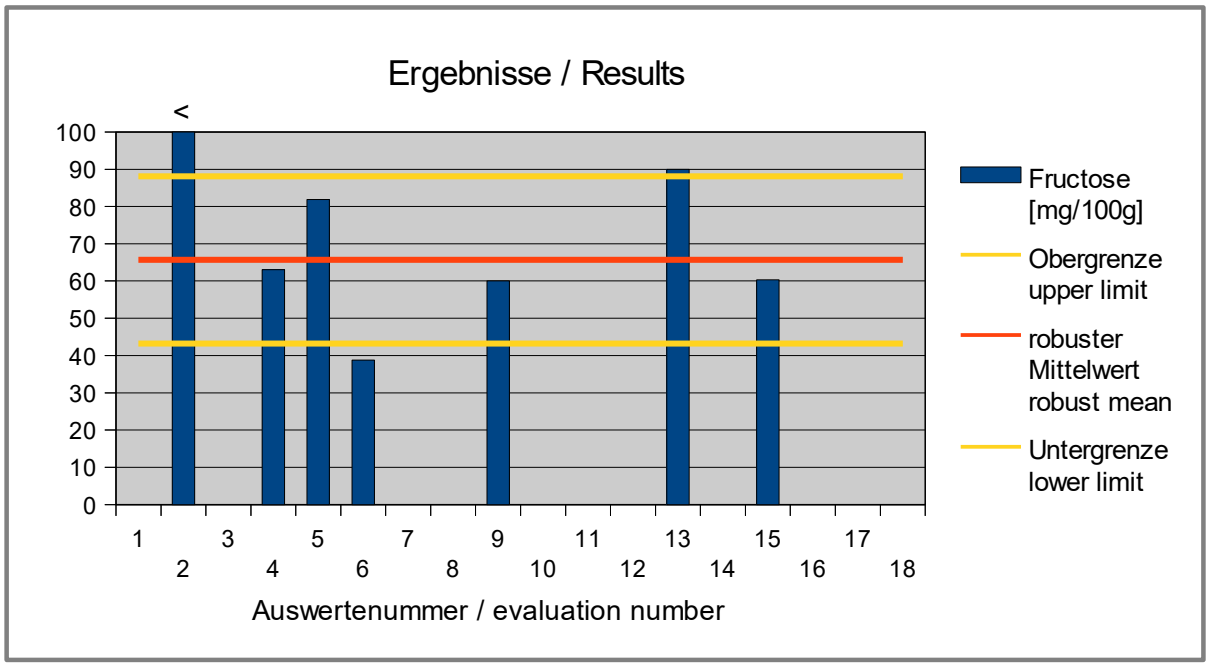

Abb. / Fig. 1: Ergebnisse Fructose Probe A/ Results fructose sample A

Note:
Due to the low number of < 8 results a kernel density estimation could not be carried out.

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	< 100				
3					
4	63	-2,7	-0,24	-1,7	
5	82	16,2	1,4	11	
6	39	-26,9	-2,4	-18	
7					
8					
9	60	-5,7	-0,51	-3,7	
10	0				Ergebnis ausgeschlossen / Result excluded
11					
12					
13	90	24,3	2,2	16	
14					
15	60	-5,4	-0,48	-3,5	
16					
17					
18	< LOD				

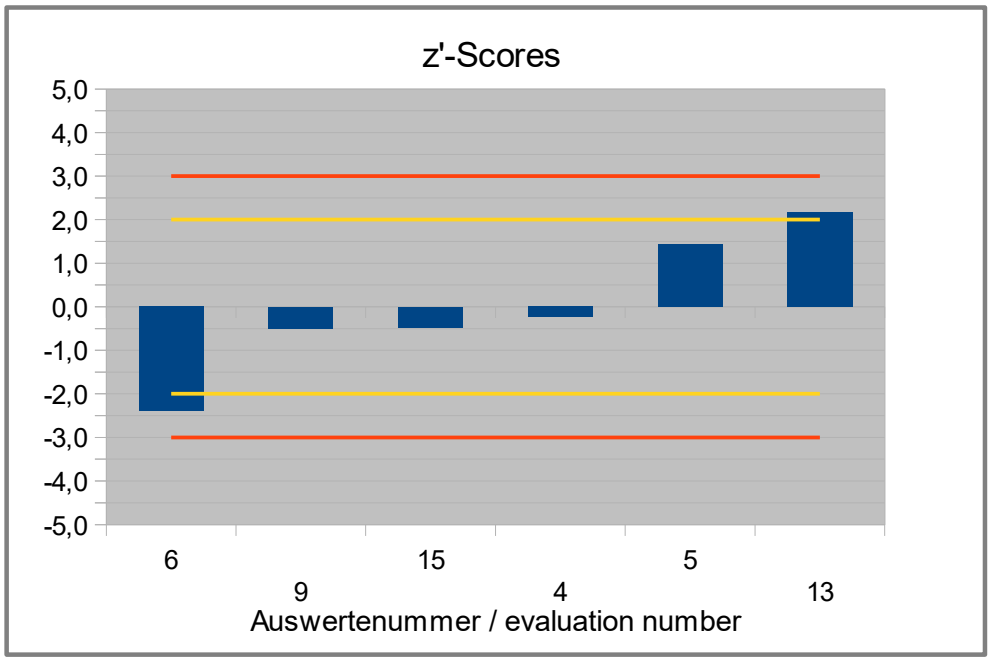

Abb. / Fig. 2: z'-Scores Fructose Probe A / fructose sample A

4.1.2 Fructose Sample B (in mg/100g)

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	7
Number of outliers	-
Mean	116
Median	105
Robust mean (X_{pt})	**102**
Robust standard deviation (S*)	**38,7**
Target range:	
Target standard deviation $\sigma_{pt'}$	**19,2**
Target standard deviation (for Information)	2,38
lower limit of target range	**63,5**
upper limit of target range	**140**
Quotient S/$\sigma_{pt'}$*	*2,0*
Standard uncertainty $U_{(Xpt)}$	*18*
Results in the target range	*5*
Percent in the target range	*71%*

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, [22]) is given for information (s. 3.6.2).

The distribution of results showed an increased variability. The quotient S^*/σ_{pt} was well above 2. Therefore the valuation was done by z'-scores considering the standard uncertainty. The quotient $S^*/\sigma_{pt'}$ was then 2,0. The robust standard deviation was in the range of previous PTs (see 3.6.3). The comparability of results is given.

71% of results were in the target range.

The difference of the robust means of the participants' results for sample B and sample A (36,3 mg/100g) was at 105% of the spiking level of fructose to the sample B (s. p. 5).

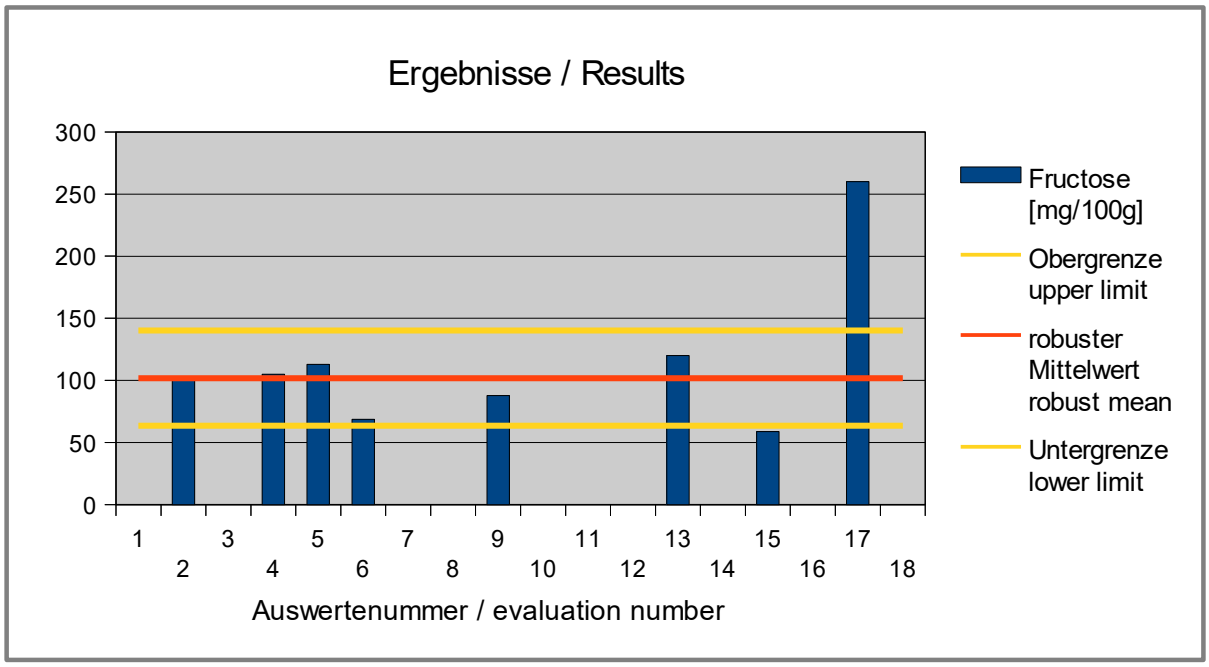

Abb. / Fig. 3: Ergebnisse Fructose Probe B/ Results fructose sample B

Note:
Due to the low number of < 8 results a kernel density estimation could not be carried out.

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	< 100				
3					
4	105	3,2	0,17	1,3	
5	113	11,0	0,57	4,6	
6	68,6	-33,2	-1,7	-14	
7					
8					
9	87,7	-14,1	-0,74	-5,9	
10	0				Ergebnis ausgeschlossen / Result excluded
11					
12					
13	120	18,2	0,95	7,7	
14					
15	58,7	-43,1	-2,2	-18,1	
16					
17	260	158	8,2	66,6	
18	< LOD				

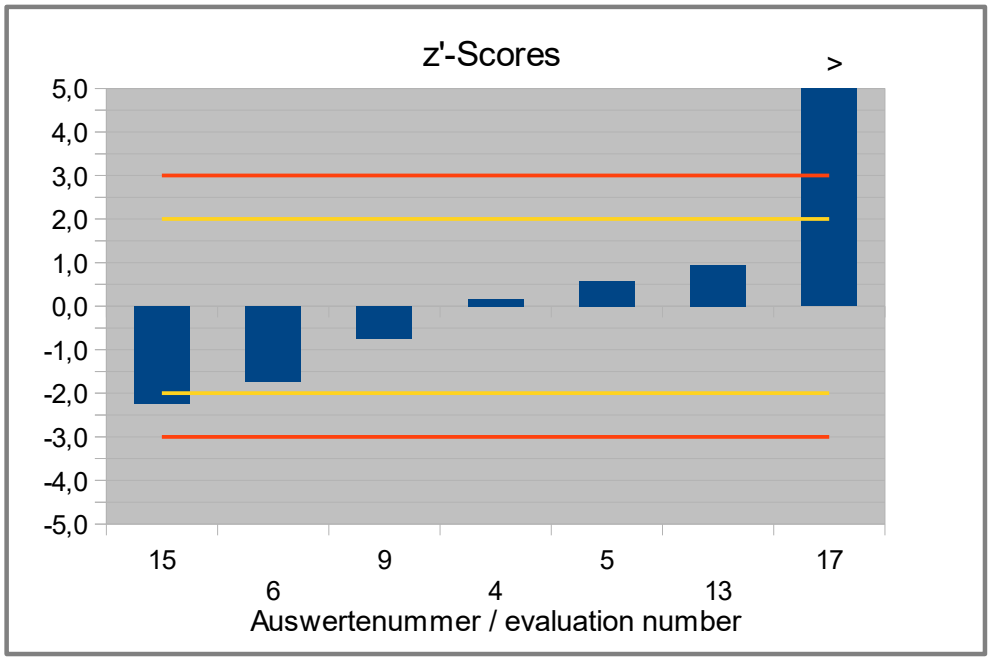

Abb. / Fig. 4: z-Scores Fructose Probe B / fructose sample B

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

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results°	8
Number of outliers	1
Mean	556
Median	579
Robust Mean (x_{pt})	**556**
Robust standard deviation (S*)	**45,8**
Target range:	
Target standard deviation σ_{pt}'	**31,6**
Target standard deviation (for Information)	13,0
lower limit of target range	**493**
upper limit of target range	**620**
Quotient S/σ_{pt}'*	*1,4*
Standard uncertainty $U_{(Xpt)}$	*20*
Results in the target range	*8*
Percent in the target range	*100%*

° *without outlier (result no. 6)*

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, [22]) is given for information (s. 3.6.2).

The distribution of results showed a slightly increased variability. Therefore the valuation was done by z'-scores considering the standard uncertainty. The quotient S/σ_{pt}' was then 1,4. The robust standard deviation was in the range of previous PTs (see 3.6.3). The comparability of results is given.*

100% of results were in the target range.

The robust mean of participant results was 167 % of the spiking level of fructose to the spiking level sample (s. p. 5). It should be noted that the potato powder matrix can also contain fructose.

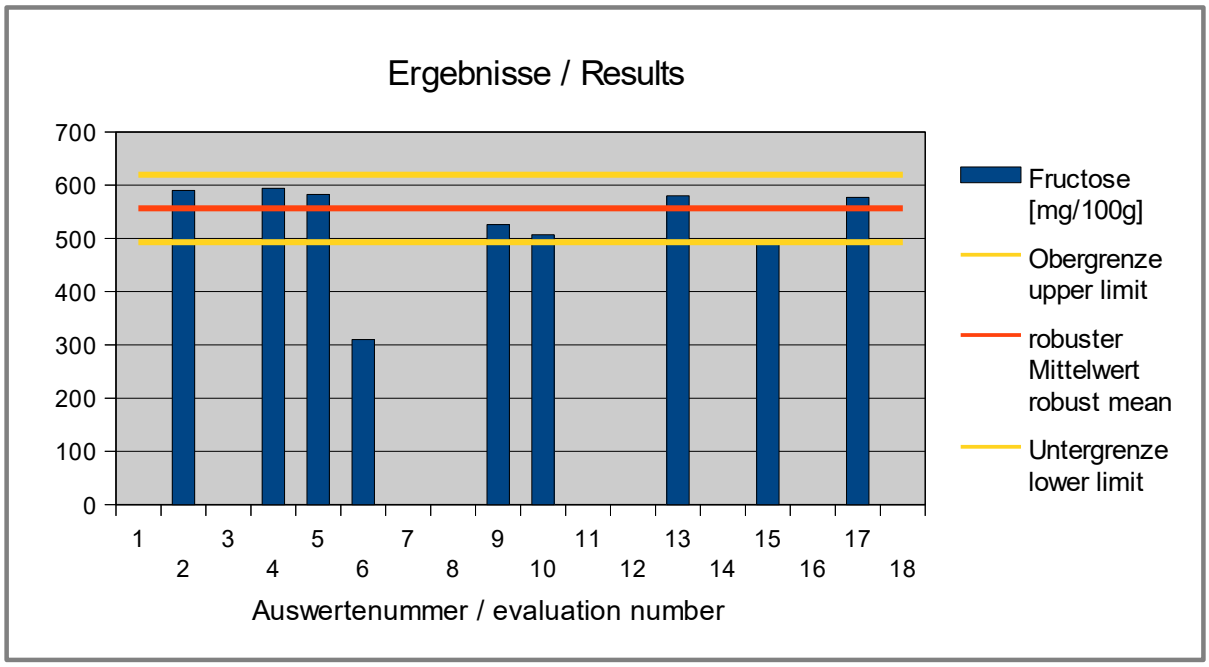

Abb. / Fig. 5: Ergebnisse Fructose Dotierungsniveauprobe / Results Fructose spiking level sample

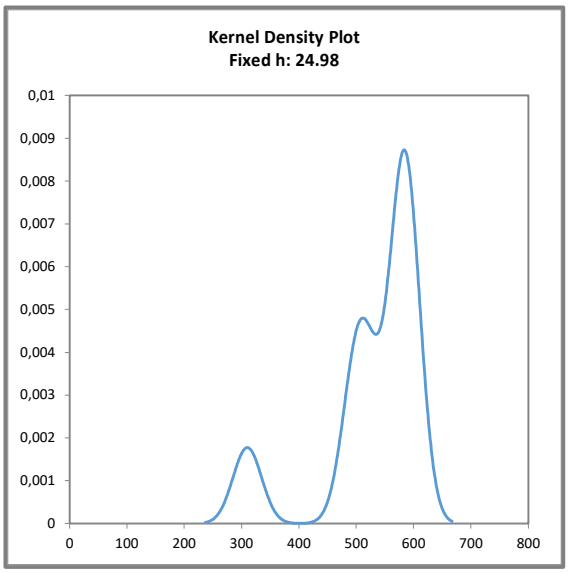

Abb. / Fig. 6:
Kerndichte-Schätzung der Ergebnisse (mit h = 0,75 x σ_{pt} von X_{pt})

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

Comment:
The kernel density shows almost a symmetrical distribution of results with a clear shoulder at approx. 500 mg/100g and an additional peak at 310 mg/100g due to a result outside 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	590	34	1,1	2,6	
3					
4	594	37,6	1,2	2,9	
5	583	26,3	0,83	2,0	
6	310				Ausreißer ausgeschlossen / Outlier excluded
7					
8					
9	526	-30,4	-0,96	-2,3	
10	507	-49,4	-1,6	-3,8	
11					
12					
13	580	23,6	0,75	1,8	
14					
15	495	-61,9	-2,0	-4,8	
16					
17	577	21	0,65	1,6	
18					

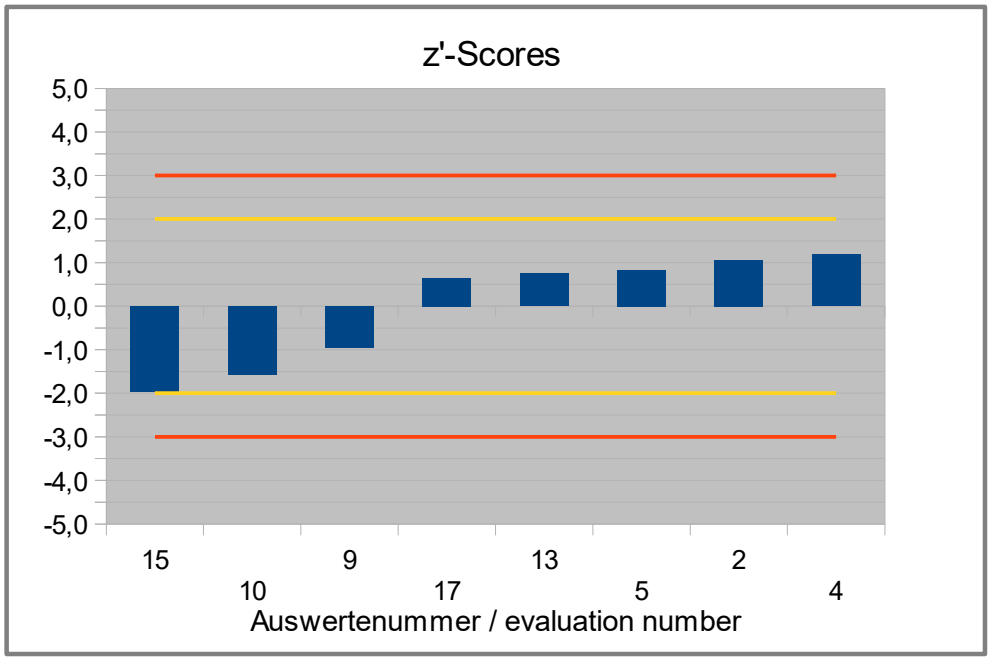

Abb. / Fig. 7: z'-Scores Fructose Dotierungsniveauprobe / fructose spiking level sample

4.2 Lactose

4.2.1 Qualitative Evaluation Sample A and Sample B

Evaluation number	Sample A	Sample A	Sample B	Sample B	Qualitative Valuation	Remarks
	pos/neg	[mg/100g]	pos/neg	[mg/100g]	Agreement with consensus value	
1	negative	< 2	positive	168	2/2 (100%)	
2	negative	< 10	positive	270	2/2 (100%)	
3	negative	< 10	positive	185	2/2 (100%)	
4	negative	< 2.5	positive	169	2/2 (100%)	
5	negative	< 10,0	positive	171	2/2 (100%)	
6	negative	< 6	positive	174	2/2 (100%)	
7	negative	< 3	positive	170	2/2 (100%)	
8	negative	< 100	positive	153	2/2 (100%)	
9	negative		positive	162	2/2 (100%)	
10	negative	0	positive	180	2/2 (100%)	
11	negative	0	positive	20	2/2 (100%)	
12	negative	< 5	positive	190	2/2 (100%)	
13	negative	< 50	positive	170	2/2 (100%)	
14	negative	< 30	positive	191	2/2 (100%)	
15	negative	0	positive	173	2/2 (100%)	
16	negative	< 3.64	positive	170	2/2 (100%)	
17	negative		positive	180	2/2 (100%)	
18	negative	< LOD	positive	136	2/2 (100%)	

	Sample A		Sample B	
Number positive	0		18	
Number negative	18		0	
Percent positive	0		100	
Percent negative	100		0	
Consensus value	negative		positive	

Comments:
The consensus values are in qualitative agreement with the spiking of sample B.

4.2.2 Lactose Sample B (in mg/100g)

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	18
Number of outliers	-
Mean	168
Median	170
Robust Mean (X_{pt})	**172**
Robust standard deviation (S*)	**15,1**
Target range:	
Target standard deviation σ_{pt}	**13,5**
Target standard deviation (for Information)	8,98
lower limit of target range	**145**
upper limit of target range	**199**
Quotient S/σ_{pt}*	*1,1*
Standard uncertainty $U_{(Xpt)}$	*4,45*
Results in the target range	*15*
Percent in the target range	*83%*

Comments:

The target standard deviation was calculated using data from a precision experiment (ASU §64 L 01.00-90, [19]*)(3.6.2). Additionally the target standard deviation according to the model of Horwitz (s. 3.6.1) is given for information.*

The distribution of results showed a normal variability. The quotient S^*/σ_{pt} was well below 2,0. The robust standard deviation was in the range of previous PTs (see 3.6.3). The comparability of results is given.

85% of results were in the target range.

The robust mean of participant results was 96 % of the spiking level of fructose to sample B (s. p. 5).

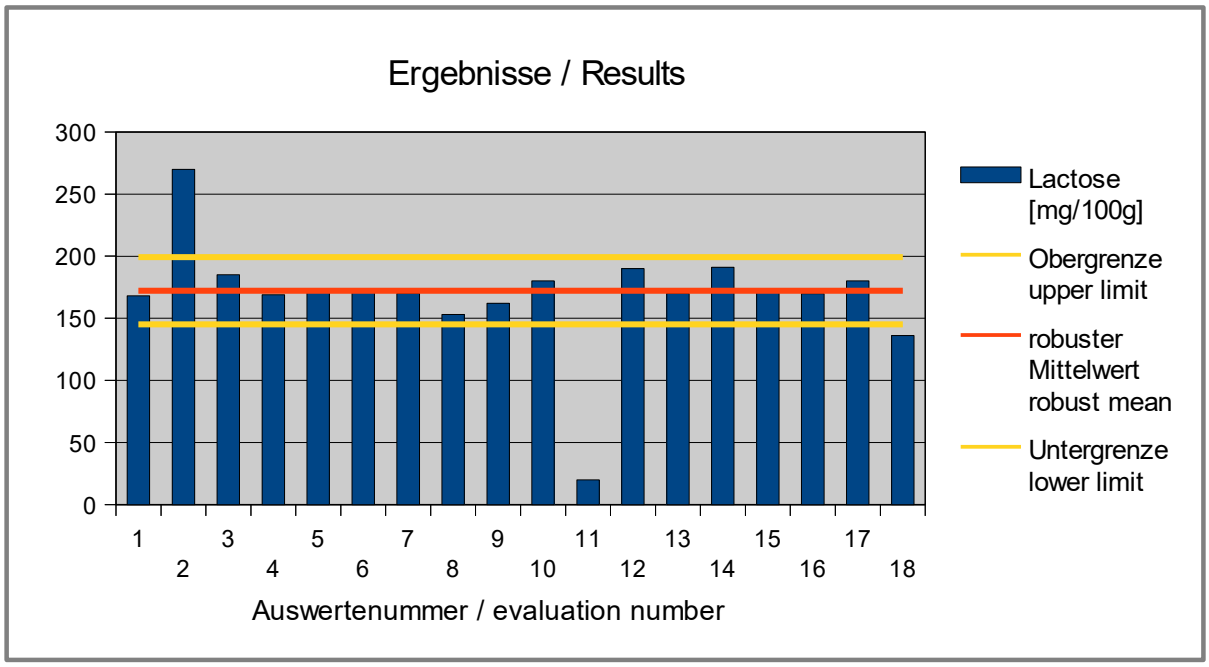

Abb. / Fig. 8: Ergebnisse Lactose Probe B / Results lactose sample B

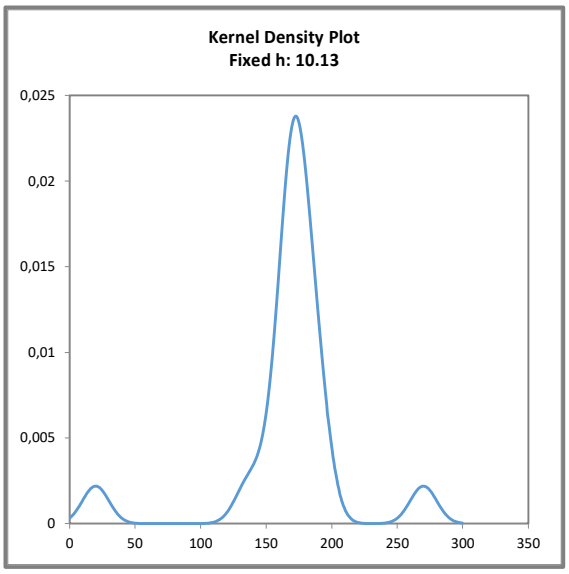

Abb. / Fig. 9:
Kerndichte-Schätzung der Ergebnisse
(mit h = 0,75 x σ_{pt} von X_{pt})

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

Comment:
The kernel density shows almost a symmetrical distribution of results with two additional small peaks due to two results outside 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	168	-4,2	-0,31	-0,47	
2	270	97,8	7,2	11	
3	185	12,8	0,95	1,4	
4	169	-3,2	-0,24	-0,35	
5	171	-1,4	-0,10	-0,15	
6	174	1,8	0,13	0,20	
7	170	-2,2	-0,16	-0,24	
8	153	-19,2	-1,4	-2,1	
9	162	-10,2	-0,75	-1,1	
10	180	7,8	0,58	0,87	
11	20	-152	-11	-17	
12	190	17,8	1,3	2,0	
13	170	-2,2	-0,16	-0,24	
14	191	18,8	1,4	2,1	
15	173	0,6	0,05	0,07	
16	170	-2,4	-0,18	-0,27	
17	180	7,8	0,58	0,87	
18	136	-36,2	-2,7	-4,0	

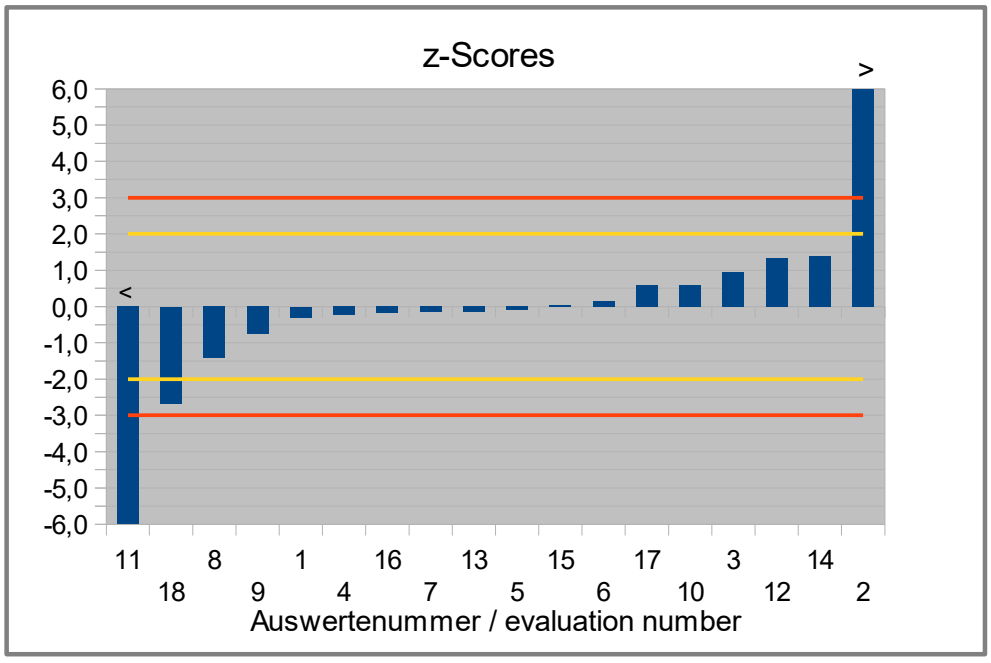

Abb. / Fig. 10: z-Scores Lactose Probe B / lactose sample B

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

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	17
Number of outliers	-
Mean	180
Median	165
Robust Mean (x_{pt})	**163**
Robust standard deviation (S*)	**14,7**
Target range:	
Target standard deviation σ_{pt}	**12,8**
Target standard deviation (for Information)	8,6
lower limit of target range	**138**
upper limit of target range	**189**
Quotient S/σ_{pt}*	*1,1*
Standard uncertainty $U_{(Xpt)}$	*4,46*
Results in the target range	*15*
Percent in the target range	*88%*

Comments:

The target standard deviation was calculated using data from a precision experiment (ASU §64 L 01.00-90, [19]*)(3.6.2). Additionally the target standard deviation according to the model of Horwitz (s. 3.6.1) is given for information.*

The distribution of results showed a normal variability. The quotient S^*/σ_{pt} was below 2,0. The robust standard deviation was in the range of previous PTs (see 3.6.3). The comparability of results is given.

88% of results were in the target range.

The robust mean of participant results was 93 % of the spiking level of lactose to the spiking level sample (s. p. 5).

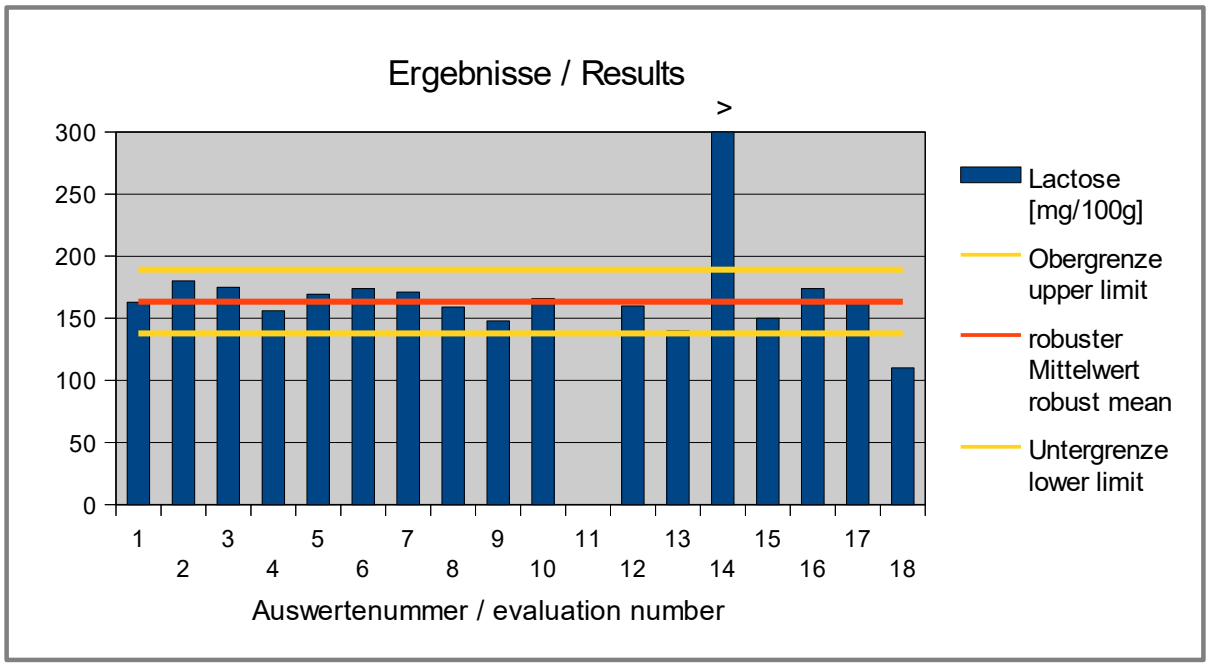

Abb. / Fig. 11: Ergebnisse Lactose Dotierungsniveauprobe / Results lactose spiking level sample

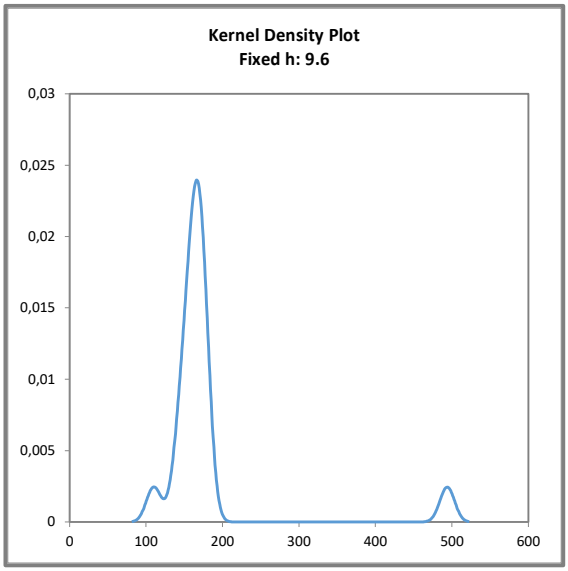

Abb. / Fig. 12:
Kerndichte-Schätzung der Ergebnisse (mit h = 0,75 x σ_{pt} von X_{pt})

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

Comment:
The kernel density shows nearly a symmetrical distribution of results with two small side peaks, due to two results outside 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	163	-0,4	-0,03	-0,05	
2	180	16,6	1,3	1,9	
3	175	11,6	0,90	1,3	
4	156	-7,4	-0,58	-0,87	
5	169	5,9	0,46	0,68	
6	174	10,6	0,82	1,2	
7	171	7,6	0,59	0,88	
8	159	-4,4	-0,34	-0,52	
9	148	-15,4	-1,2	-1,8	
10	166	2,6	0,20	0,30	
11					
12	160	-3,4	-0,27	-0,40	
13	140	-23,4	-1,8	-2,7	
14	494	331	26	39	
15	150	-13,4	-1,0	-1,6	
16	174	10,3	0,81	1,2	
17	165	1,6	0,12	0,18	
18	110	-53,4	-4,2	-6,2	

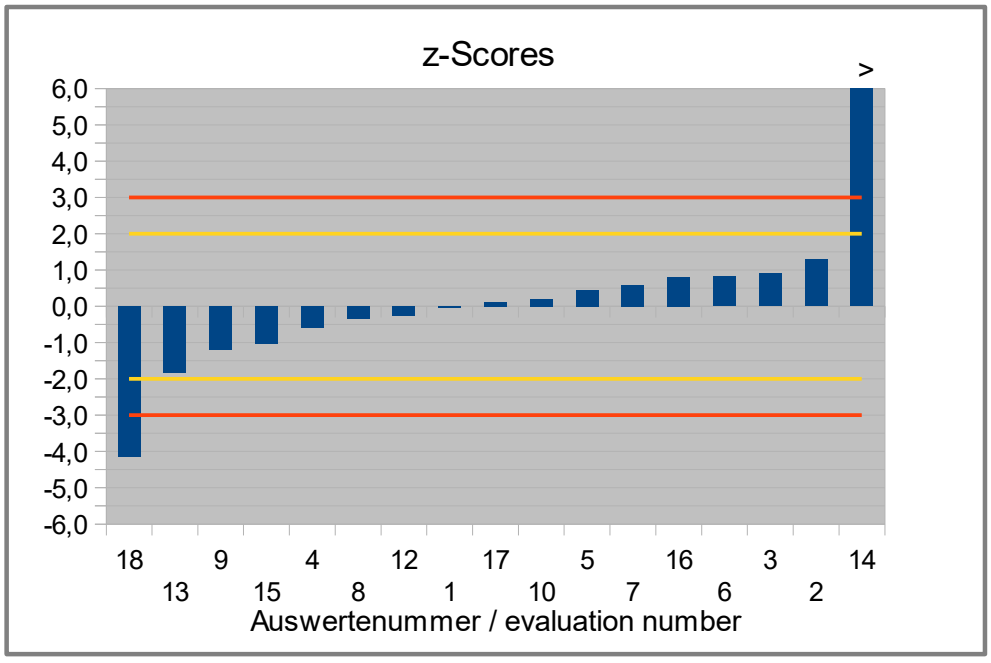

Abb. / Fig. 13: z-Scores Lactose Dotierungsniveauprobe / lactose spiking level sample

4.2.4 Recovery Rates for Lactose

Hereafter the recovery rates of the participants' results with respect to the level of addition (page 5, table 1) were calculated by DLA and given for information only. The related z-scores are based on the target standard deviation of 7,5%.

Spiking Level Sample and Sample B

Evaluation number	Spiking Level Sample	Recovery rate*		Sample B	Recovery rate*		Remarks
	[mg/kg]	[%]	[Z_{RR}]	[mg/kg]	[%]	[Z_{RR}]	
1	163	**93**	-0,98	168	**93**	-0,89	
2	180	**102**	0,30	270	150	6,7	
3	175	**99**	-0,08	185	**103**	0,37	
4	156	**89**	-1,5	169	**94**	-0,81	
5	169	**96**	-0,51	171	**95**	-0,68	
6	174	**99**	-0,15	174	**97**	-0,44	
7	171	**97**	-0,38	170	**94**	-0,74	
8	159	**90**	-1,3	153	**85**	-2,0	
9	148	**84**	-2,1	162	**90**	-1,3	
10	166	**94**	-0,76	180	**100**	0,00	
11				20	11	-12	
12	160	**91**	-1,2	190	**106**	0,74	
13	140	**80**	-2,7	170	**94**	-0,74	
14	494	281	24	191	**106**	0,81	
15	150	**85**	-2,0	173	**96**	-0,53	
16	174	**99**	-0,17	170	**94**	-0,76	
17	165	**94**	-0,83	180	**100**	0,00	
18	110	63	-5,0	136	**76**	-3,3	

RA**	85-115 %	RA**	85-115 %
Number in RA	**13**	Number in RA	**17**
Percent in RA	**62**	Percent in RA	**74**

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

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

Comments:

For the spiking level sample 62% (13) of the participants obtained a recovery rate within the range of 85-115%. For the spiked food matrix sample B 73% (16) 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 and variability of results no statistical evaluation was done.

Statistic Data	
Number of results	3
Number of outliers	
Mean	29,2
Median	7,70
Robust Mean (X_{pt})	**29,2**
Robust standard deviation (S*)	**44,0**
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 $U_{(Xpt)}$	
Results in the target range	
Percent in the target range	

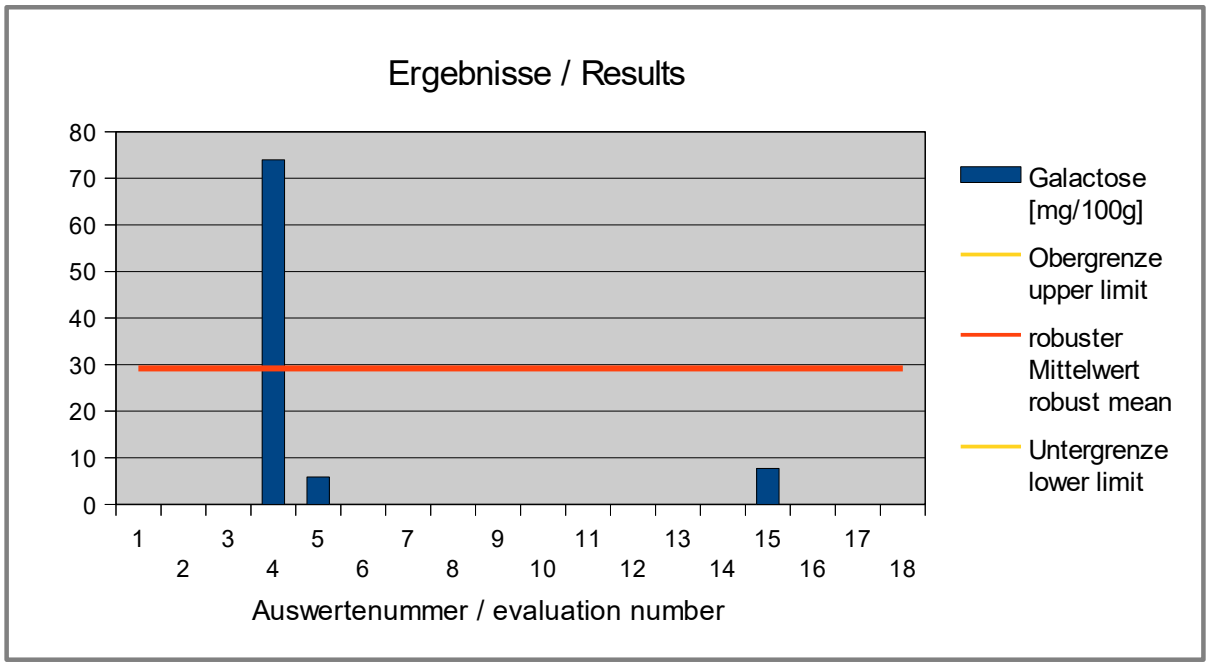

Abb. / Fig. 14: Ergebnisse Galactose Probe A / Results galactose sample A

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	< 100				
3	< 10				
4	74,0	44,8			
5	5,9	-23,3			
6	< 10				
7					
8	< 100				
9					
10	0				
11					
12					
13	< 50				
14	< 30				
15	7,7	-21,5			
16					
17					
18					

4.3.2 Galactose Sample B (in mg/100g)

Vergleichsuntersuchung / Proficiency Test

Due to the low number of results and variability of results no statistical evaluation was done.

Kenndaten	
Anzahl der Messergebnisse	3
Anzahl der Ausreißer	0
Mittelwert	28,3
Median	6,30
Robuster Mittelwert (X_{pt})	**28,3**
Robuste Standardabweichung (S*)	**43,9**
Zielkenndaten:	
Zielstandardabweichung σ_{pt}'	
Zielstandardabweichung (zur Information)	
Untere Grenze des Zielbereichs	
Obere Grenze des Zielbereichs	
Quotient S/σ_{pt}'*	
Standardunsicherheit $U_{(Xpt)}$	
Ergebnisse im Zielbereich	
Prozent im Zielbereich	

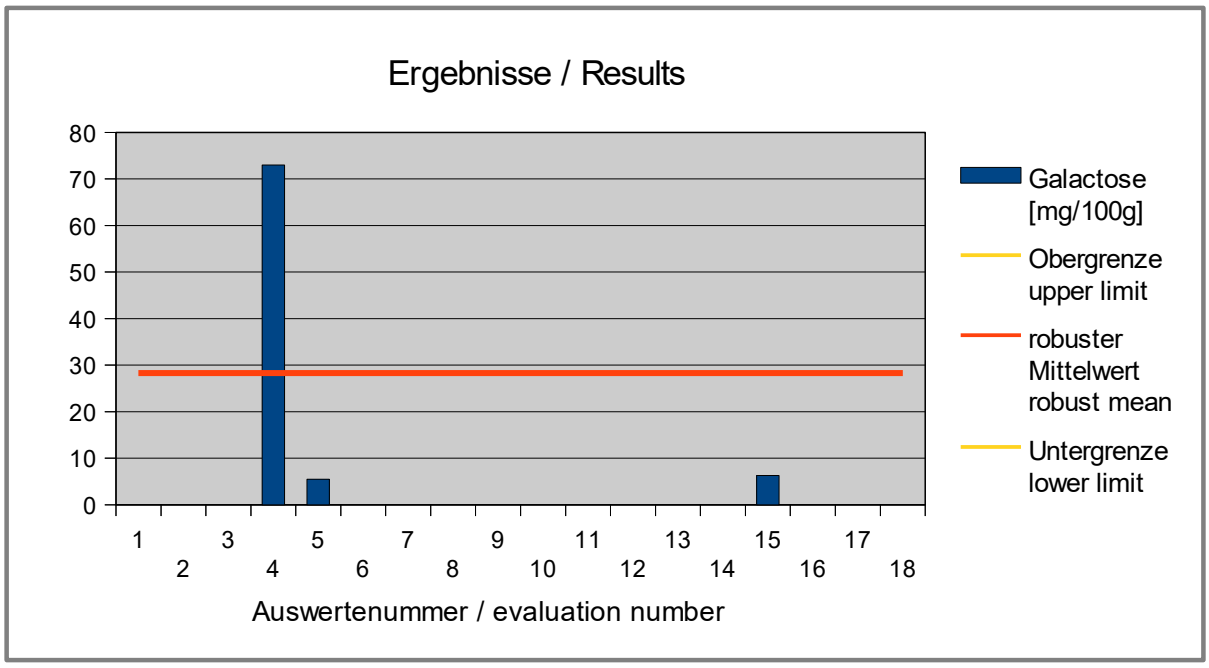

Abb. / Fig. 15: Ergebnisse Galactose Probe B / Results galactose sample B

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	< 100				
3	< 10				
4	73,0	44,7			
5	5,5	-22,8			
6	< 10				
7					
8	< 100				
9					
10	0				
11					
12					
13	< 50				
14	< 30				
15	6,3	-22,0			
16					
17					
18					

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

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	5
Number of outliers	0
Mean	12,4
Median	12,0
Robust Mean (X_{pt})	**12,4**
Robust standard deviation (S*)	**3,43**
Target range:	
Target standard deviation $\sigma_{pt'}$	**2,15**
Target standard deviation (for Information)	0,96
lower limit of target range	**8,12**
upper limit of target range	**16,7**
Quotient S/$\sigma_{pt'}$*	*1,6*
Standard uncertainty $U_{(Xpt)}$	*1,92*
Results in the target range	*5*
Percent in the target range	*100%*

Comments:

The target standard deviation was calculated using data from a precision experiment (ASU §64 L 01.00-90, [19]*)(3.6.2). Additionally the target standard deviation according to the model of Horwitz (s. 3.6.1) is given for information.*

The distribution of results showed a slightly increased variability. Therefore the valuation was done by z'-scores considering the standard uncertainty. The quotient S/$\sigma_{pt'}$ was then 1,6. The robust standard deviation was in the range of previous PTs (see 3.6.3). The comparability of results is given.*

100% of results were in the target range.

No galactose was added to the samples, so a recovery rate cannot be given (s. p. 5).

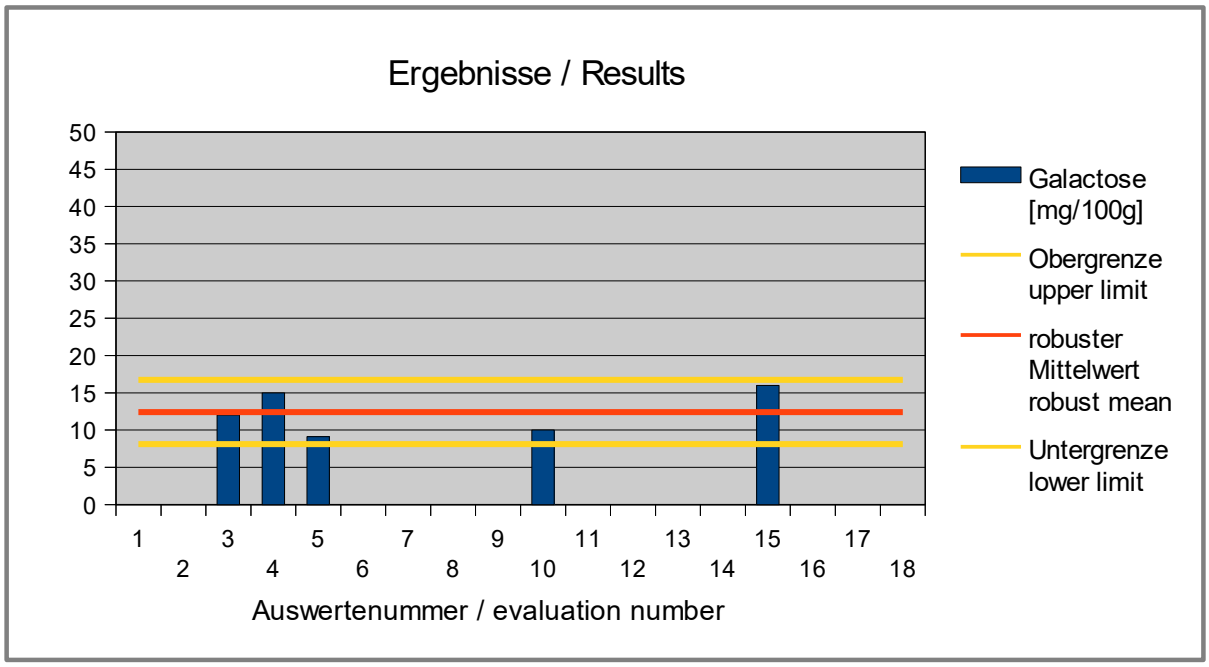

Abb. / Fig. 16: Ergebnisse Galactose Dotierungsniveauprobe / Results Fructose spiking level sample

Note:
Due to the low number of < 8 results a kernel density estimation could not be carried out.

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	< 100				
3	12,0	-0,4	-0,20	-0,44	
4	15,0	2,6	1,2	2,7	
5	9,10	-3,3	-1,5	-3,5	
6	< 10				
7					
8	< 100				
9					
10	10,0	-2,4	-1,1	-2,5	
11					
12					
13	< 50				
14	< 30				
15	16,0	3,6	1,7	3,7	
16					
17					
18					

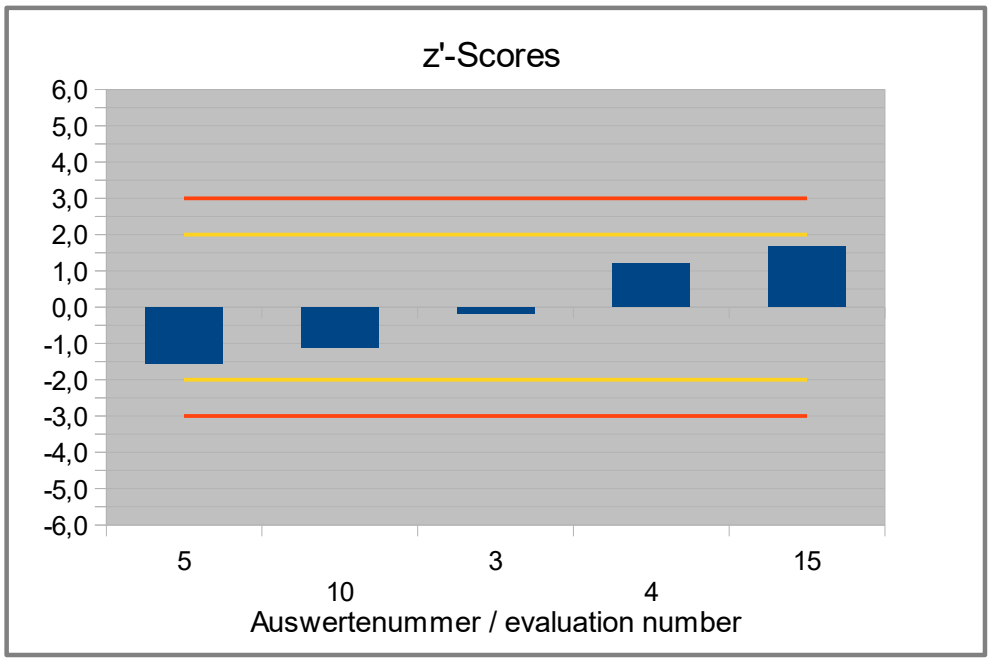

Abb. / Fig. 17: z-Scores Galactose Dotierungsniveauprobe / galactose spiking level sample

4.4 Participant z-Scores: overview table

Z-Scores for the assigned values from participants results (consensus values)

Evaluation number	Fructose			Lactose		Galactose
	Sample A°	Sample B°	Spiking Level Sample°	Sample B	Spiking Level Sample	Spiking Level Sample°
1				-0,31	-0,03	
2			1,1	7,2	1,3	
3				1,0	0,90	-0,20
4	-0,24	0,17	1,2	-0,24	-0,58	1,2
5	1,4	0,57	0,83	-0,10	0,46	-1,5
6	-2,4	-1,7		0,13	0,82	
7				-0,16	0,59	
8				-1,4	-0,34	
9	-0,51	-0,74	-1,0	-0,75	-1,2	
10			-1,6	0,58	0,20	-1,1
11				-11		
12				1,3	-0,27	
13	2,2	0,90	0,75	-0,16	-1,8	
14				1,4	26	
15	-0,48	-2,2	-2,0	0,05	-1,0	1,7
16				-0,18	0,81	
17		8,2	0,65	0,58	0,12	
18				-2,7	-4,2	

° z'-Score

Bewertung des z-Scores / valuation of z-score (DIN ISO 13528:2009-01):
-2 ≤ z-score ≤ 2 erfolgreich / successful (in green)
-2 > z-score > 2 „Warnsignal" / warning signal (in yellow)
-3 > z-score > 3 „Eingriffssignal" / action signal (in red)

Z-Scores for the assigned values from spiking level (recovery rates)

Evaluation number	Lactose	
	Sample B	Spiking Level Sample
1	-0,89	-0,98
2	6,7	0,30
3	0,37	-0,08
4	-0,81	-1,5
5	-0,68	-0,51
6	-0,44	-0,15
7	-0,74	-0,38
8	-2,0	-1,3
9	-1,3	-2,1
10	0,00	-0,76
11	-12	
12	0,74	-1,2
13	-0,74	-2,7
14	0,81	24
15	-0,53	-2,0
16	-0,76	-0,17
17	0,00	-0,83
18	-3,3	-5,0

Bewertung des z-Scores / valuation of z-score (DIN ISO 13528:2009-01):
-2 ≤ z-score ≤ 2 erfolgreich / successful (in green)
-2 > z-score > 2 „Warnsignal" / warning signal (in yellow)
-3 > z-score > 3 „Eingriffssignal" / action signal (in red)

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	Participant	Unit	Date of analysis	Final result	Detectable	LOD	LOQ	Incl. RR	Recovery rate [%]
Sample A			Day /Month	mg/100g	yes / no	mg/100g	mg/100g		
Fructose	1	mg/100g							
	2	mg/100g	10.05.21	<100	Yes	10	100	No	N/A
	3	mg/100g							
	4	mg/100g	27.05.21	63	Yes	10	10	No	NA
	5	mg/100g	10.05.21	81,9	yes	5	10	no	
	6	mg/100g	26.05.21	38,8	yes	1	10	no	
	7	mg/100g							
	8	mg/100g		n.t.					
	9	mg/100g	31.05.21	60		3	10	no	
	10	mg/100g	17.05.21	0	no	5	5	no	
	11	mg/100g							
	12	mg/100g							
	13	mg/100g	18.05.21	90	yes		50		
	14	mg/100g							
	15	mg/100g	09.06.21	60,3	yes	not determined	not determined	no	not determined
	16	mg/100g							
	17	mg/100g	14.06.21		no	30	100	no	
	18	mg/100g	08.06.21	< Limit of detection	no	100	300	no	

Fructose Sample B

Parameter	Participant	Unit	Date of analysis	Final result	Detectable	LOD	LOQ	Incl. RR	Recovery rate [%]
Sample B			Day /Month	mg/100g	yes / no	mg/100g	mg/100g		
Fructose	1	mg/100g							
	2	mg/100g	10.05.21	<100	Yes	10	100	No	N/A
	3	mg/100g							
	4	mg/100g	27.05.21	105	Yes	10	10	No	NA
	5	mg/100g	10.05.21	112,8	yes	5	10	no	
	6	mg/100g	26.05.21	68,6	yes	1	10	no	
	7	mg/100g							
	8	mg/100g		n.t.					
	9	mg/100g	31.05.21	87,7		3	10	no	
	10	mg/100g	17.05.21	0	no	5	5	no	
	11	mg/100g							
	12	mg/100g							
	13	mg/100g	18.05.21	120	yes		50		
	14	mg/100g							
	15	mg/100g	10.06.21	58,7	yes	not determined	not determined	no	not determined
	16	mg/100g							
	17	mg/100g	14.06.21	260	yes	30	100	no	
	18	mg/100g	08.06.21	< Limit of quantification	yes	100	300	no	

Fructose Spiking Level Sample

Parameter	Participant	Unit	Date of analysis	Final result	Detectable	LOD	LOQ	Incl. RR	Recovery rate [%]
Spiking Level Sample			Day /Month	mg/100g	yes / no	mg/100g	mg/100g		
Fructose	1	mg/100g							
	2	mg/100g	10.05.21	590	Yes	10	100	No	N/A
	3	mg/100g							
	4	mg/100g	27.05.21	594	Yes	10	10	No	NA
	5	mg/100g	10.05.21	582,7	yes	5	10	no	
	6	mg/100g	26.05.21	310	yes	1	10	no	
	7	mg/100g							
	8	mg/100g		n.t.					
	9	mg/100g	31.05.21	526		3	10	no	
	10	mg/100g	17.05.21	507	yes	5	5	no	
	11	mg/100g							
	12	mg/100g							
	13	mg/100g	18.05.21	580	yes		50		
	14	mg/100g							
	15	mg/100g	10.05.21	494,5	yes	not determined	not determined	no	not determined
	16	mg/100g							
	17	mg/100g	14.06.21	577	yes	30	100	no	
	18	mg/100g		Not analyzed					

Lactose Sample A

Parameter	Participant	Unit	Date of analysis	Final result	Detectable	LOD	LOQ	Incl. RR	Recovery rate [%]
Sample A			Day /Month	mg/100g	yes / no	mg/100g	mg/100g		
Lactose	1	mg/100g	28.04.21	<2	no		2	yes	97
	2	mg/100g	10.05.21	<10	No	1	10	No	N/A
	3	mg/100g	21.05.21	<10	no	5	10	no	
	4	mg/100g	27.05.21	<2.5	No	1	2,5	No	NA
	5	mg/100g	10.05.	< 10,0	no	1	10	no	
	6	mg/100g	26.05.21	< 6	no	1	6	no	104
	7	mg/100g	14.06.21	< 3	no	1	3		
	8	mg/100g	11.06.21	< 100	no	100mg/kg	100mg/kg	no	100,9
	9	mg/100g	31.05.21		no	3	10	no	
	10	mg/100g	10.06.21	0	no	1	2	no	
	11	mg/100g	18.05.21	0	no		10 mg/100g	yes	76,5
	12	mg/100g	04.05.21	<5		-	5	no	
	13	mg/100g	18.05.21	<50	no		50		
	14	mg/100g		< 30	no	15	30	no	
	15	mg/100g	09.06.21	0	no	not determined	not determined	no	not determined
	16	mg/100g	12.05.21	<3.643	No	3,643	3,643	No	N/A
	17	mg/100g	14.06.21		no	30	100	no	
	18	mg/100g	08.06.21	< Limit of detection	no	1,4	5	no	

Lactose Sample B

Parameter	Participant	Unit	Date of analysis	Final result	Detectable	LOD	LOQ	Incl. RR	Recovery rate [%]
Sample B			Day /Month	mg/100g	yes / no	mg/100g	mg/100g		
Lactose	1	mg/100g	14.05.21	168	yes		2	yes	99
	2	mg/100g	10.05.21	270	Yes	1	10	No	N/A
	3	mg/100g	20.05.21	185	yes	5	10	no	
	4	mg/100g	27.05.21	169	Yes	1	2,5	No	NA
	5	mg/100g	10.05.	170,8	yes	1	10	no	
	6	mg/100g	26.05.21	174	yes	1	6	no	104
	7	mg/100g	14.06.21	170	yes	1	3		
	8	mg/100g	11.06.21	153	yes	100mg/kg	100mg/kg	no	99,9
	9	mg/100g	31.05.21	162		3	10	no	
	10	mg/100g	10.06.21	180	yes	1	2	no	
	11	mg/100g	18.05.21	20	yes		10 mg/100g	yes	76,5
	12	mg/100g	04.05.21	190		-	5	no	
	13	mg/100g	18.05.21	170	yes		50		
	14	mg/100g		191	yes	15	30	no	
	15	mg/100g	10.06.21	172,8	yes	not determined	not determined	no	not determined
	16	mg/100g	16.06.21	169,766	Yes	3,643	3,643	No	N/A
	17	mg/100g	14.06.21	180	yes	30	100	no	
	18	mg/100g	08.06.21	136	yes	1,4	5	no	

Lactose Spiking Level Sample

Parameter	Participant	Unit	Date of analysis	Final result	Detectable	LOD	LOQ	Incl. RR	Recovery rate [%]
Spiking Level Sample			Day /Month	mg/100g	yes / no	mg/100g	mg/100g		
Lactose	1	mg/100g	14.05.21	163	yes		2	yes	88
	2	mg/100g	10.05.21	180	Yes	1	10	No	N/A
	3	mg/100g	21.05.21	175	yes	5	10	no	
	4	mg/100g	27.05.21	156	Yes	1	2,5	No	NA
	5	mg/100g	10.05.	169,3	yes	1	10	no	
	6	mg/100g	26.05.21	174	yes	1	6	no	104
	7	mg/100g	14.06.21	171	yes	1	3		
	8	mg/100g	11.06.21	159	yes	100mg/kg	100mg/kg	no	100
	9	mg/100g	31.05.21	148		3	10	no	
	10	mg/100g	10.06.21	166	yes	1	2	no	
	11	mg/100g							
	12	mg/100g	04.05.21	160		-	5	no	
	13	mg/100g	18.05.21	140	yes		50		
	14	mg/100g		494	yes	15	30	no	
	15	mg/100g	10.05.21	150	yes	not determined	not determined	no	not determined
	16	mg/100g	12.05.21	173,773	Yes	3,643	3,643	No	N/A
	17	mg/100g	14.06.21	165	yes	30	100	no	
	18	mg/100g	08.06.21	110	yes	1,4	5	no	

Galactose Sample A

Parameter	Participant	Unit	Date of analysis	Final result	Detectable	LOD	LOQ	Incl. RR	Recovery rate [%]
Sample A			Day /Month	mg/100g	yes / no	mg/100g	mg/100g		
Galactose	1	mg/100g							
	2	mg/100g	10.05.21	<100	No	10	100	No	N/A
	3	mg/100g	20.05.21	<10	yes	5	10	no	
	4	mg/100g	27.05.21	74	Yes	10	10	No	NA
	5	mg/100g	10.05.21	5,9	yes	1	5	no	
	6	mg/100g	26.05.21	< 10	yes	1	10	no	
	7	mg/100g							
	8	mg/100g	11.06.21	< 100	no	100mg/kg	100mg/kg	no	
	9	mg/100g	31.05.21		yes	3	10	no	
	10	mg/100g	17.05.21	0	no	5	5	no	
	11	mg/100g							
	12	mg/100g							
	13	mg/100g	18.05.21	<50	no		50		
	14	mg/100g		< 30	no	15	30	no	
	15	mg/100g	09.06.21	7,7	yes	not determined	not determined	no	not determined
	16	mg/100g							
	17	mg/100g	14.06.21		no	30	100	no	
	18	mg/100g		Not analyzed					

Galactose Sample B

Parameter	Participant	Unit	Date of analysis	Final result	Detectable	LOD	LOQ	Incl. RR	Recovery rate [%]
Sample B			Day /Month	mg/100g	yes / no	mg/100g	mg/100g		
Galactose	1	mg/100g							
	2	mg/100g	10.05.21	<100	No	10	100	No	N/A
	3	mg/100g	19.05.21	<10	yes	5	10	no	
	4	mg/100g	27.05.21	73	Yes	10	10	No	NA
	5	mg/100g	10.05.21	5,5	yes	1	5	no	
	6	mg/100g	26.05.21	< 10	yes	1	10	no	
	7	mg/100g							
	8	mg/100g	11.06.21	< 100	no	100mg/kg	100mg/kg	no	
	9	mg/100g	31.05.21		yes	3	10	no	
	10	mg/100g	17.05.21	0	no	5	5	no	
	11	mg/100g							
	12	mg/100g							
	13	mg/100g	18.05.21	<50	no		50		
	14	mg/100g		< 30	no	15	30	no	
	15	mg/100g	10.06.21	6,3	yes	not determined	not determined	no	not determined
	16	mg/100g							
	17	mg/100g	14.06.21		no	30	100	no	
	18	mg/100g		Not analyzed					

Galactose Spiking Level Sample

Parameter	Participant	Unit	Date of analysis	Final result	Detectable	LOD	LOQ	Incl. RR	Recovery rate [%]
Spiking Level Sample			Day /Month	mg/100g	yes / no	mg/100g	mg/100g		
Galactose	1	mg/100g							
	2	mg/100g	10.05.21	<100	Yes	10	100	No	N/A
	3	mg/100g	20.05.21	12	yes	5	10	no	
	4	mg/100g	27.05.21	15	Yes	10	10	No	NA
	5	mg/100g	10.05.21	9,1	yes	1	5	no	
	6	mg/100g	26.05.21	< 10	yes	1	10	no	
	7	mg/100g							
	8	mg/100g	11.06.21	< 100	no	100mg/kg	100mg/kg	no	
	9	mg/100g	31.05.21		yes	3	10	no	
	10	mg/100g	17.05.21	10	yes	5	5	no	
	11	mg/100g							
	12	mg/100g							
	13	mg/100g	18.05.21	<50	no		50		
	14	mg/100g		< 30	no	15	30	no	
	15	mg/100g	10.05.21	16	yes	not determined	not determined	no	not determined
	16	mg/100g							
	17	mg/100g	14.06.21		no	30	100	no	
	18	mg/100g		Not analyzed					

5.1.2 Analytical Methods

Fructose Sample A, Sample B and Spiking Level Sample

Parameter	Participant	Method description as in test report / norm / literature	Sample preparation	Measuring method	Calibration / Reference material	Recovery rate with same matrix	Method accredited ISO/IEC 17025	Further Remarks
						yes/no	yes/no	
Fructose	1							
	2	HPAEC-PAD	N/A	N/A	N/A	N/A	Yes	
	3							
	4	CHROM/344						
	5	PA_A-203:2019-01 (HPAEC/PAD)	aqueous extraction	HPAEC/PAD	external calibration		yes	
	6	IC-PAD	aqueous extraction	IC-PAD	Matrix calibration with wheat flour, reference material DLA 18/2019	no	no	
	7							
	8							
	9	IC-PAD	Dialysis		external calibration		yes	
	10	HPAE-PAD		Method in validation phase	yes and ok		yes	
	11							
	12							
	13							
	14							
	15	no existing method available for this matrix; the sample was processed in the same way as for the determination of carbohydrates in dairy products and vegan milk substitutes	an aliquot was dissolved in water, treated in an ultrasonic bath, then centrifuged, the aqueous phase was additionally filtered off and the filtrate obtained was measured by means of an ion chromatograph with a PAD detector and built-in inline dialysis	for the present matrix no validated method available	calibration linear in the range 0,05-20,0 mg/L per carbohydrate; no reference material available	recovery rate not determined	no	
	16							
	17	Enzymatic	homogenize, aqueous extraction, Carrez clarification, filtration		Standards of Enzyme Kit r-biopharm	no	yes	
	18 *	MP.0002.R2.2020	Extraction with water	IC quantification of sample extract	External std Fructose Merck	no	yes	

* Participant 18: only samples A and B

Lactose Sample A, Sample B and Spiking Level Sample

Parameter	Participant	Method description as in test report / norm / literature	Sample preparation	Measuring method	Calibration / Reference material	Recovery rate with same matrix	Method accredited ISO/IEC 17025	Further Remarks
						yes/no	yes/no	
Lactose	1	HPLC-MS		recovery calculated by C13-Lactose internal standard	Anhydrous lactose (Sigma)	yes	yes	
	2	HPAEC-PAD	N/A	N/A	N/A	N/A	Yes	
	3	r-biopharm Test-Combination 10 176 303 035:2011-06	as per kit instructions				yes	
	4	CHROM/356						
	5	PA_A-203:2019-01 (HPAEC/PAD)	aqueous extraction	HPAEC/PAD	external calibration		yes	
	6	IC-PAD	aqueous extraction	IC-PAD	Matrix calibration with wheat flour, reference material DLA 18/2019	no	no	Recovery rate in real samples (bake mixtures)
	7	Internal method: extraction and analysis by LC-MS/MS					yes	
	8	Lactose/D-Galactose 101763035 rbiopharm	as per kit instructions	as per kit instructions	as per kit instructions	yes	yes	
	9	IC-PAD	Dialysis		external calibration		yes	
	10	PV-448-Lac-HPAE-PAD : 2019-04 (b)			yes and ok		yes	
	11 *	INTERNAL METHOD					yes	
	12	extraction in water and measured by ion chromatography			patron lactosa Merck	yes	no	
	13							
	14	Megazyme K-LACGAR 01/20					yes	
	15	no existing method available for this matrix; the sample was processed in the same way as for the determination of carbohydrates in dairy products and vegan milk substitutes	an aliquot was dissolved in water, treated in an ultrasonic bath, then centrifuged, the aqueous phase was additionally filtered off and the filtrate obtained was measured by means of an ion chromatograph with a PAD detector and built-in inline dialysis	for the present matrix no validated method available	calibration linear in the range 0,05-20,0 mg/L per carbohydrate; no reference material available	recovery rate not determined	no	
	16	Megazyme LOLAC Enzymatic Kit	Water and Carrez Extraction	Spectrophotometer	Internal Quality Control Material	N/A	Yes	
	17	Enzymatic	homogenize, aqueous extraction, Carrez clarification, filtration		Standards of Enzyme Kit r-biopharm	no	yes	
	18	MP.0002.R2.2020	Extraction with water	IC quantification of sample extract	External std Lactose Merck	no	yes	

* Participant 11: only samples A and B

Galactose Sample A, Sample B and Spiking Level Sample

Parameter	Participant	Method description as in test report / norm / literature	Sample preparation	Measuring method	Calibration / Reference material	Recovery rate with same matrix	Method accredited ISO/IEC 17025	Further Remarks
						yes/no	yes/no	
Galactose	1							
	2	HPAEC-PAD	N/A	N/A	N/A	N/A	Yes	
	3	r-biopharm Test-Combination 10 176 303 035:2011-06	as per kit instructions				yes	
	4	CHROM/344						
	5	PA_A-203:2019-01 (HPAEC/PAD)	aqueous extraction	HPAEC/PAD	external calibration		yes	
	6	IC-PAD	aqueous extraction	IC-PAD	Matrix calibration with wheat flour, reference material DLA 18/2019	no	no	
	7							
	8	Lactose/D-Galactose 101763035 rbiopharm	as per kit instructions	as per kit instructions	as per kit instructions			
	9	IC-PAD	Dialysis		externe Kalibrierung		no	
	10	HPAE-PAD		Method in validation phase	yes and ok		yes	
	11							
	12							
	13							
	14	Megazyme K-LACGAR 01/20					yes	
	15	no existing method available for this matrix; the sample was processed in the same way as for the determination of carbohydrates in dairy products and vegan milk substitutes	an aliquot was dissolved in water, treated in an ultrasonic bath, then centrifuged, the aqueous phase was additionally filtered off and the filtrate obtained was measured by means of an ion chromatograph with a PAD detector and built-in inline dialysis	for the present matrix no validated method available	calibration linear in the range 0,05-20,0 mg/L per carbohydrate; no reference material available	recovery rate not determined	no	
	16							
	17	Enzymatic	homogenize, aqueous extraction, Carrez clarification, filtration		Standards of Enzyme Kit r-biopharm	no	yes	
	18							

5.2 Homogeneity

5.2.1 Mixture homogeneity before bottling

Microtracer Homogeneity Test
DLA ptAI01 (2021) Sample B

Weight whole sample	2,56	kg
Microtracer	FSS-rot lake	
Particle size	75 – 300	µm
Weight per particle	2,0	µg
Addition of tracer	20,1	mg/kg

Result of analysis

Sample	Weight [g]	Particle number	Particles [mg/kg]
1	5,00	65	26,0
2	5,01	69	27,5
3	5,00	72	28,8
4	5,03	60	23,9
5	4,97	69	27,8
6	4,98	60	24,1
7	5,01	72	28,7
8	4,98	55	22,1

Poisson distribution		
Number of samples	8	
Degree of freedom	7	
Mean	65,2	Particle
Standard deviation	6,29	Particle
χ^2 (CHI-Quadrat)	4,24	
Probability	**75**	%
Recovery rate	130	%

Normal distribution		
Number of samples	8	
Mean	26,1	mg/kg
Standard deviation	2,52	mg/kg
rel. Standard deviaton	9,63	%
Horwitz standard deviation	9,79	%
HorRat-value	**0,98**	
Recovery rate	130	%

Microtracer Homogeneity Test
DLA ptAI01 (2021) Spiking Level Sample

Weight whole sample	1,84	kg
Microtracer	FSS-rot lake	
Particle size	75 – 300	µm
Weight per particle	2,0	µg
Addition of tracer	20,2	mg/kg

Result of analysis

Sample	Weight [g]	Particle number	Particles [mg/kg]
1	5,01	54	21,6
2	5,03	50	19,9
3	4,97	42	16,9
4	4,97	50	20,1
5	5,00	53	21,2
6	5,03	50	19,9
7	4,97	57	22,9
8	5,01	52	20,8

Poisson distribution		
Number of samples	8	
Degree of freedom	7	
Mean	51,0	Particle
Standard deviation	4,37	Particle
χ^2 (CHI-Quadrat)	2,62	
Probability	**92**	%
Recovery rate	101	%

Normal distribution		
Number of samples	8	
Mean	20,4	mg/kg
Standard deviation	1,75	mg/kg
rel. Standard deviaton	8,57	%
Horwitz standard deviation	10,2	%
HorRat-value	**0,84**	
Recovery rate	101	%

5.3 Information on the Proficiency Test (PT)

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

PT number	***ptAI01 – 2021***
PT name	***Lactose + Fructose in "lactose-free" Food***
Sample matrix (processing)	***Samples A + B: Cereal Pap Powder** / ingredients: whole grain oat flour, whole grain millet flour, rice semolina, maize flour, thiamine and lactose and fructose (one of both samples)* ***Spiking Level Sample:** potato powder, lactose and fructose*
Number of samples and sample amount	*2 different Samples A + B: 25 g each* *+ 1 Spiking Level Sample: 15 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* *Samples A + B: Lactose < 500 mg/100g* *Spiking Level Sample: 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. From **Samples A + B** the **total sample amount** should be **homogenized** each.*
Result sheet	*One result each should be determined for Samples A and B and the Spiking Level Sample.* *The results should be filled in the result submission file.*
Units	*mg/100g*
Number of digits	*at least 2*
Result submission	*The result submission file should be sent by e-mail to:* ***pt@dla-lvu.de***
Last Deadline	***the latest June 18th 2021 (extended)***
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	*Alexandra Scharf MSc.*

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

6. Index of participant laboratories in alphabetical order

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

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

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

7. Index of references

1. DIN EN ISO/IEC 17025:2005; Allgemeine Anforderungen an die Kompetenz von Prüf- und Kalibrierlaboratorien / General requirements for the competence of testing and calibration laboratories
2. DIN EN ISO/IEC 17043:2010; Konformitätsbewertung – Allgemeine Anforderungen an Eignungsprüfungen / Conformity assessment – General requirements for proficiency testing
3. ISO 13528:2015 & DIN ISO 13528:2009; Statistische Verfahren für Eignungsprüfungen durch Ringversuche / Statistical methods for use in proficiency testing by inter-laboratory comparisons
4. ASU §64 LFGB: Planung und statistische Auswertung von Ringversuchen zur Methoden-validierung / DIN ISO 5725 series part 1, 2 and 6 Accuracy (trueness and preci-sion) of measurement methods and results
5. Verordnung / Regulation 882/2004/EU; Verordnung über über amtliche Kontrollen zur Überprüfung der Einhaltung des Lebensmittel- und Futtermittelrechts sowie der Bestimmungen über Tiergesundheit und Tierschutz / Regulation on official controls performed to ensure the verification of compliance with feed and food law, animal health and animal welfare rules
6. Evaluation of analytical methods used for regulation of food and drugs; W. Hor-witz; Analytical Chemistry, 54, 67-76 (1982)
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12. AMC Kernel Density - Representing data distributions with kernel density estim-ates, amc technical brief, Editor M Thompson, Analytical Methods Committee, AMCTB No 4, Revised March 2006 and Excel Add-in Kernel.xla 1.0e by Royal Society of Chemistry
13. EURACHEM/CITAC Leitfaden, Ermittlung der Messunsicherheit bei analytischen Messun-gen (2003); Quantifying Uncertainty in Analytical Measurement (1999)
14. GMP+ Feed Certification scheme, Module: Feed Safety Assurance, chapter 5.7 Check-ing procedure for the process accuracy of compound feed with micro tracers in GMP+ BA2 Control of residues, Version: 1st of January 2015 GMP+ International B.V.
15. MTSE SOP No. 010.01 (2014): Quantitative measurement of mixing uniformity and carry-over in powder mixtures with the rotary detector technique, MTSE Micro Tracers Services Europe GmbH
16. Homogeneity and stability of reference materials; Linsinger et al.; Accred Qual Assur, 6, 20-25 (2001)
17. AOAC Official Methods of Analysis: Guidelines for Standard Method Performance Re-quirements, Appendix F, p. 2, AOAC Int (2016)
18. 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
19. 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]
20. 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]
21. 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]

22. 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]
23. 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)