**Evaluation Report**
proficiency test

**DLA 18/2019**

# Lactose and Fructose:

## in “lactose free” Food - Cake Bake Mix

***DLA - Proficiency Tests GmbH***
*Kalte Weide 21*
*24641 Sievershütten/Germany*

*proficiency-testing@dla-lvu.de* *www.dla-lvu.de*

*Coordinator of this PT:*
*Dr. Matthias Besler-Scharf / Alexandra Scharf*

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

| | |
|---|---|
| *EP-Anbieter*<br>*PT-Provider* | **DLA - Proficiency Tests GmbH**<br>Kalte Weide 21, 24641 Sievershütten, Germany<br><br>Geschäftsführer/CEO: Dr. Matthias Besler-Scharf<br>Stellv. Leitung/Deputy Lead: Alexandra Scharf MSc.<br><br>Tel. ++49-(0)4532-9183358<br>Mob. ++49(0)171-1954375<br>Fax. ++49(0)4102-9944976<br>eMail. proficiency-testing@dla-lvu.de |
| *EP-Nummer*<br>*PT-Number* | DLA 18/2019 |
| *EP-Koordinator*<br>*PT-Coordinator* | Dr. Matthias Besler-Scharf |
| *Status des EP-Bericht*<br>*Status of PT-Report* | Abschlussbericht / Final report (22 July 2019)<br><br>Gültig ist die jeweils letzte Version/Korrektur des Berichts. Sie ersetzt alle vorangegangenen Versionen.<br>Only the latest version/correction of the report is valid. It replaces all preceding versions. |
| *EP-Bericht Freigabe*<br>*PT-Report Authorization* | Dr. Matthias Besler-Scharf (Technischer Leiter / Technical Manager)<br>*- gezeichnet / signed M. Besler-Scharf*<br>Alexandra Scharf MSc. (QM-Beauftragte / Quality Manager)<br>*- gezeichnet / signed A. Scharf*<br>Datum / Date: 22 July 2019 |
| *Unteraufträge*<br>*Subcontractors* | Falls im Rahmen der Eignungsprüfung eine Prüfung der Gehalte, Homogenität und Stabilität von EP-Parametern durchgeführt wurde, hat DLA diese im Unterauftrag vergeben.<br>In case the analysis of the content, homogeneity and stability of PT-parameters was part of the proficiency test, the determinations were subcontracted by DLA. |
| *Vertraulichkeit*<br>*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.<br>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 is a common in commerce baking mix "marble cake" (labeled as lactose-free). The basic composition of both samples A and B was the same (see table 1).
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 3 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 and B were portioned to approximately 25 g, the spiking level sample 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** |
|---|---|---|---|
| Marble cake, baking mix with cocoa mixture<br>Ingredients: BAKING MIXTURE (93%): wheat flour, sugar, starch, baking powder (acidifier: diphosphates, raising agent: sodium bicarbonate), emulsifier: E475, flavoring, salt. COCOA MIXTURE (6.7%): sugar, low-fat cocoa powder (40%)<br>Nutrients per 100g: Fat 1.9 g, carbohydrates 80 g, fiber 2.6 g, protein 4.6 g, salt 0.6 g | 100 g/100g | 99,4 g/100g | - |
| Potato powder<br>Ingredients:<br>Potatoes, E471, E304, E223, E100 | | - | 99,3 g/100g |
| Lactose* | | 115 mg/100g | 111 mg/100g |
| Fructose* | | 495 mg/100g | 551 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 100% 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. 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 0,27 and 0,72 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. 0,43 (23,6°C). 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, and B were sent to every participating laboratory in the 14th week of 2019. The testing method was optional. The tests should be finished at 17th May 2019 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 **cake baking mix**. 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.

21 out of 22 registered participants submitted the results in time. 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 $\sigma_{pt}$) [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 $\sigma_{pt}$ (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 $\sigma_{pt}$ (= standard deviation for proficiency assessment) can be determined according to the following methods.

If an acceptable quotient $S^*/\sigma_{pt}$ 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 parameter lactose the target standard deviation from 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 in the spiking level sample the standard uncertainty was considered and the results were evaluated by z´-score (see 3.8).***

***Due to the low number of < 7 the results of galactose were not evaluated by means of z-scores.***

### *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 $\sigma_R$ [6]. Later the model was modified by Thompson for certain concentration ranges [10]. The reproducibility standard deviation $\sigma_R$ can be applied as the relative target standard deviation $\sigma_{pt}$ 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 $\sigma_R$ and the repeatability standard deviation $\sigma_r$ of a precision experiment (collaborative trial or proficiency test) the target standard deviation $\sigma_{pt}$ 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 $\sigma_{pt}$, 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 $\sigma_{pt}$ [18-23]

| **Parameter** | **Matrix** | **Mean** [g/100g] | **$RSD_r$** | **$RSD_R$** | **$\sigma_{pt}$** | **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%<br>0,0804%<br>0,1257% | 6,74%<br>1,71%<br>6,25% | 10,9%<br>3,95%<br>7,33% | 9,76%[1]<br>3,76%<br>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 calculated from means of the standard deviations (7,805%)*

*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 present PT compared to PT results of previous years.

Table 3: Characteristics of the present PT (on grey) in comparison to previous PTs since 2013 (SD = standard deviation, CV = coefficient of variation)

| **Parameter** | **Matrix** | **robust Mean** [mg/100g] | **rob. SD (S*)** [mg/100g] | **rel. SD ($VK_{S*}$) [%]** | **Quotient $S^*/\sigma_{pt}$** | **DLA-report** |
|---|---|---|---|---|---|---|
| Fructose | Bread baking mixture | 880<br>660 | 105<br>187 | 11,9<br>28,3 | 1,6*<br>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) |
| 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) |

** with target standard deviation $\sigma_{pt}$'*
*** 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 ($\sigma_{pt}$) 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 ($\sigma_{pt}$) 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 ($\sigma_{pt}$) 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 $\sigma_{pt}$'.

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*/$\sigma_{pt}$

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 $\sigma_{pt}$ 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 ($u_{(x_{pt})}$) for this PT is calculated as follows [3]:

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

If $u_{(x_{pt})} \leq 0{,}3\ \sigma_{pt}$ the standard uncertainty of the 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 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].
*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 ($S^*$) |
| *Target range:* |
| Target standard deviation $\sigma_{pt}$ or $\sigma_{pt}$' |
| 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*/$\sigma_{pt}$ or S*/$\sigma_{pt}$'* |
| *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 Probe A (in mg/100g)*

**Vergleichsuntersuchung / Proficiency Test**

*Due to the small number of available results (<7), no statistical evaluation was made.*

**Ergebnisse der Teilnehmer:**
**Results of Participants:**

| Auswerte-nummer<br>Evaluation number | Fructose [mg/100g] | Abweichung [mg/100g]<br>Deviation [mg/100g] | z-Score ($\sigma_{pt}$) | z-Score (Info) | Hinweis<br>Remark |
|---|---|---|---|---|---|
| 1 | <10 | | | | |
| 2 | | | | | |
| 3 | <500 | | | | |
| 4 | <100 | | | | |
| 5 | | | | | |
| 6 | <LOD | | | | |
| 7 | | | | | |
| 8 | 15 | | | | |
| 9 | | | | | |
| 10 | | | | | |
| 11 | | | | | |
| 12a | | | | | |
| 12b | | | | | |
| 13 | < 25 | | | | |
| 14a | | | | | |
| 14b | | | | | |
| 15a | <50 | | | | |
| 15b | <100 | | | | |
| 16 | | | | | |
| 17 | | | | | |
| 18 | | | | | |
| 19 | | | | | |
| 20 | 30 | | | | |
| 21 | <100 | | | | |

*Comments:*
*Fructose was not added to sample A. Two participants detected an amount of about 15 and 30 mg/100g in sample A.*

### 4.1.2 Fructose Sample B (in mg/100g)

**Vergleichsuntersuchung / Proficiency Test**

| **Statistic Data** | |
|---|---|
| *Number of results* | 12 |
| *Number of outliers* | - |
| Mean | 534 |
| Median | 525 |
| **Robust Mean (X)** | **525** |
| **Robust standard deviation (S*)** | **38,1** |
| *Target range:* | |
| **Target standard deviation $\sigma_{pt}$** | **23,1** |
| Target standard deviation (for Information) | 12,3 |
| **lower limit of target range** | **479** |
| **upper limit of target range** | **572** |
| *Quotient S*/$\sigma_{pt}$* | *1,6* |
| *Standard uncertainty $U_{(Xpt)}$* | *13,7* |
| *Results in the target range* | *11* |
| *Percent in the target range* | *92%* |

*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 normal variability. The quotient $S^*/\sigma_{pt}$ 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.*

*92% of results were in the target range.*

*The robust mean of the participants' results were at 106% of the spiking level of fructose to the sample B (s. p. 5).*

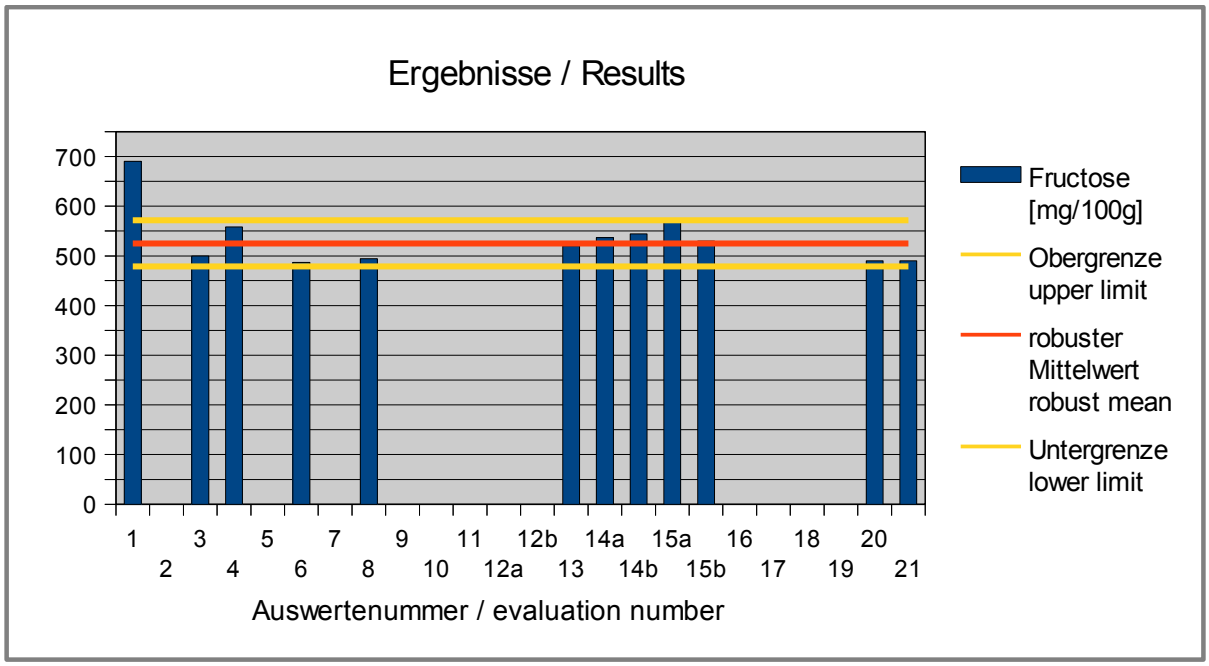


**Abb. / Fig. 1:** Ergebnisse Fructose Probe B/ Results fructose sample B

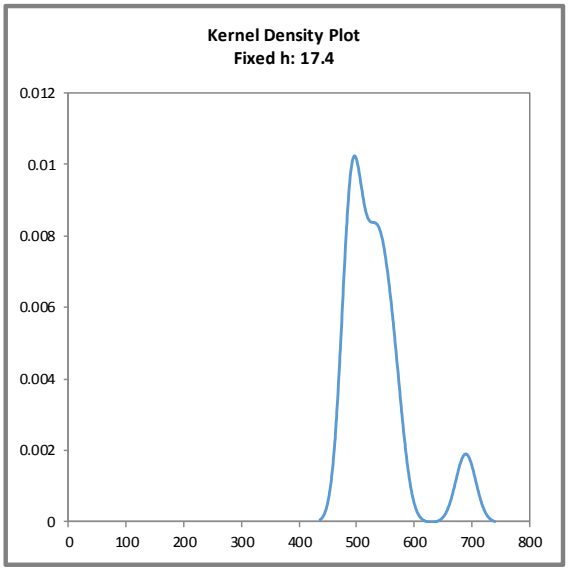


**Abb. / Fig. 2:**
Kerndichte-Schätzung der Ergebnisse
(mit h = 0,75 x $\sigma_{pt}$ von $X_{pt}$)

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

*Comment:*
The kernel density shows almost a symmetrical distribution of results with a shoulder at approx. 550 mg/100g and an additional peak at approx. 700 mg/100g, due to a single result outside the target range.

**Ergebnisse der Teilnehmer:**
**Results of Participants:**

| Auswerte-nummer<br>Evaluation number | Fructose [mg/100g] | Abweichung [mg/100g]<br>Deviation [mg/100g] | z-Score ($\sigma_{pt}$) | z-Score (Info) | Hinweis<br>Remark |
|---|---|---|---|---|---|
| 1 | 690 | 164,8 | 7,1 | 13 | |
| 2 | | | | | |
| 3 | 500 | -25,2 | -1,1 | -2,1 | |
| 4 | 558 | 32,8 | 1,4 | 2,7 | |
| 5 | | | | | |
| 6 | 487 | -38,2 | -1,7 | -3,1 | |
| 7 | | | | | |
| 8 | 494 | -30,8 | -1,3 | -2,5 | |
| 9 | | | | | |
| 10 | | | | | |
| 11 | | | | | |
| 12a | | | | | |
| 12b | | | | | |
| 13 | 520 | -5,2 | -0,23 | -0,43 | |
| 14a | 537 | 11,8 | 0,51 | 1,0 | |
| 14b | 544 | 18,8 | 0,81 | 1,5 | |
| 15a | 570 | 44,8 | 1,9 | 3,7 | |
| 15b | 530 | 4,8 | 0,21 | 0,39 | |
| 16 | | | | | |
| 17 | | | | | |
| 18 | | | | | |
| 19 | | | | | |
| 20 | 490 | -35,2 | -1,5 | -2,9 | |
| 21 | 490 | -35,2 | -1,5 | -2,9 | |

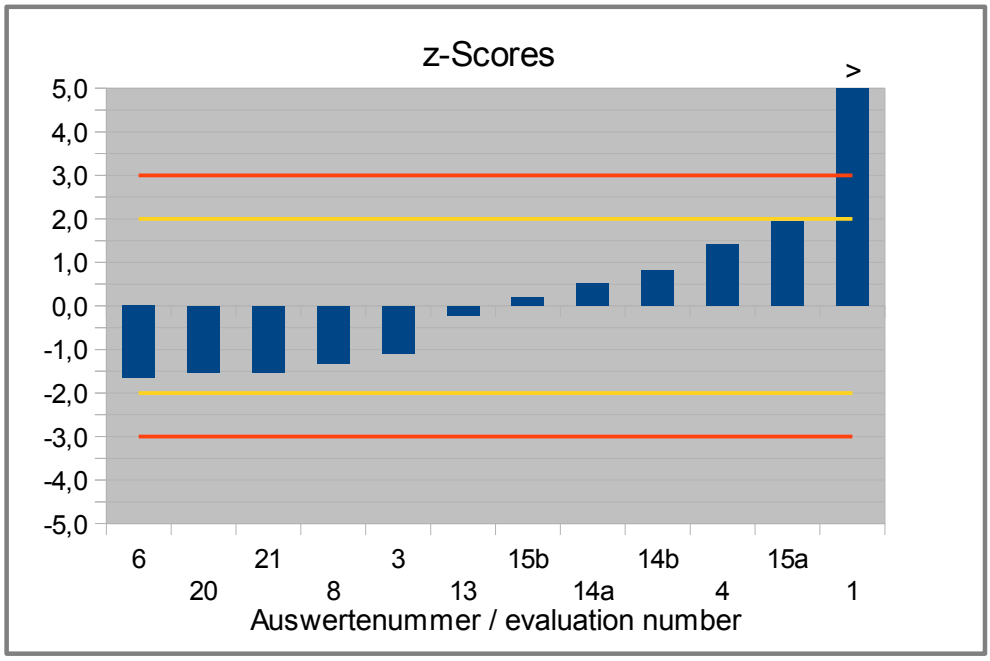


Abb. / Fig. 3: z-Scores Fructose Probe B / fructose sample B

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

**Vergleichsuntersuchung / Proficiency Test**

| **Statistic Data** | |
|---|---|
| *Number of results* | 11 |
| *Number of outliers* | 0 |
| Mean | 571 |
| Median | 550 |
| **Robust Mean (X)** | **566** |
| **Robust standard deviation (S*)** | **64,9** |
| *Target range:* | |
| **Target standard deviation $\sigma_{pt'}$** | **34,7** |
| Target standard deviation (for Information) | 13,2 |
| **lower limit of target range** | **496** |
| **upper limit of target range** | **635** |
| *Quotient S*/$\sigma_{pt'}$* | *1,9* |
| *Standard uncertainty $U_{(Xpt)}$* | *24,5* |
| *Results in the target range* | *7* |
| *Percent in the target range* | *64%* |

*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 with a quotient $S^*/\sigma_{pt}$ above 2,0. Therefore the valuation was done by z'-scores considering the standard uncertainty. The quotient $S^*/\sigma_{pt}$´ was then 1,9. The robust standard deviation was in the range of previous PTs (see 3.6.3). The comparability of results is given.*

*64% of results were in the target range.*

*The robust mean of participant results was 103 % of the spiking level of fructose to the spiking level sample (s. p. 5).*

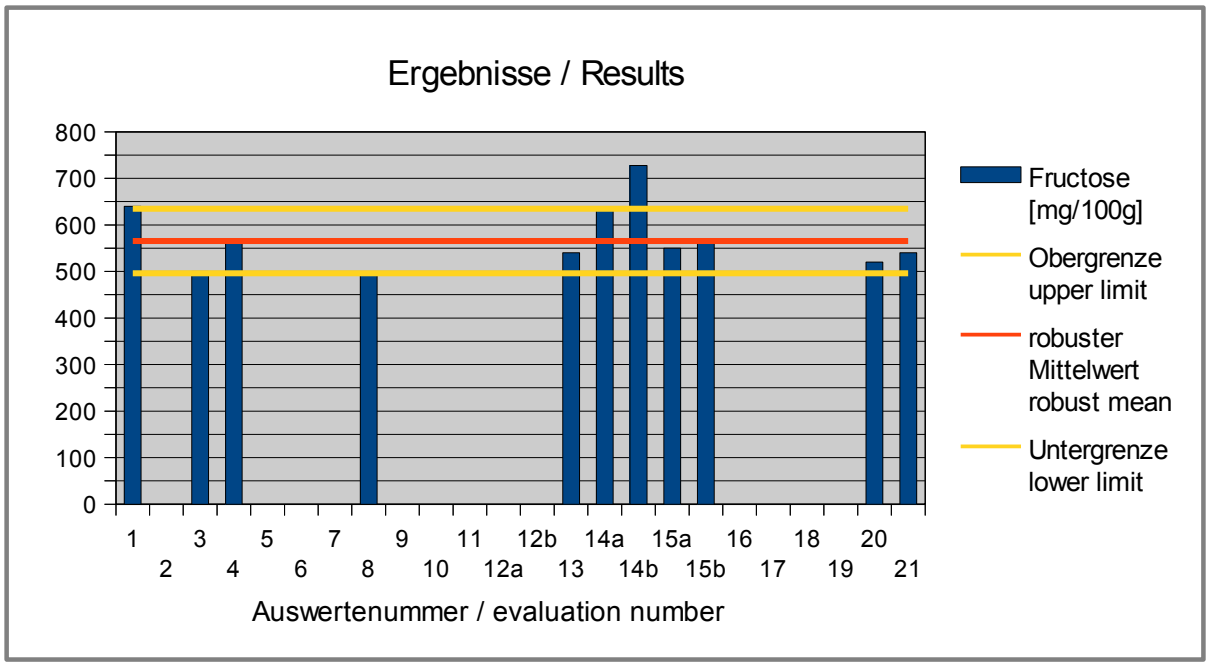


**Abb. / Fig. 4:** Ergebnisse Fructose Dotierungsniveauprobe / Results Fructose spiking level sample

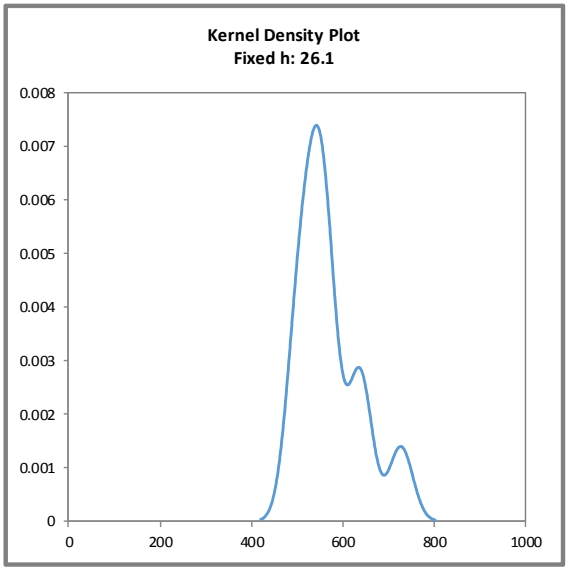


**Abb. / Fig. 5:**
Kerndichte-Schätzung der Ergebnisse (mit h = 0,75 x $\sigma_{pt}$ von $X_{pt}$)

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

*Comment:*
The kernel density shows almost a symmetrical distribution of results with two additional peaks at approx. 640 mg/100g and 730 mg/100g above the target range.

**Ergebnisse der Teilnehmer:**
**Results of Participants:**

| Auswerte-nummer<br>Evaluation number | Fructose [mg/100g] | Abweichung [mg/100g]<br>Deviation [mg/100g] | z'-Score ($\sigma_{pt}$) | z-Score (Info) | Hinweis<br>Remark |
|---|---|---|---|---|---|
| 1 | 640 | 74,5 | 2,1 | 5,6 | |
| 2 | | | | | |
| 3 | 500 | -65,5 | -1,9 | -5,0 | |
| 4 | 567 | 1,5 | 0,04 | 0,11 | |
| 5 | | | | | |
| 6 | | | | | |
| 7 | | | | | |
| 8 | 495 | -70,2 | -2,0 | -5,3 | |
| 9 | | | | | |
| 10 | | | | | |
| 11 | | | | | |
| 12a | | | | | |
| 12b | | | | | |
| 13 | 540 | -25,5 | -0,73 | -1,9 | |
| 14a | 636 | 70,0 | 2,0 | 5,3 | |
| 14b | 728 | 162,5 | 4,7 | 12 | |
| 15a | 550 | -15,5 | -0,45 | -1,2 | |
| 15b | 570 | 4,5 | 0,13 | 0,34 | |
| 16 | | | | | |
| 17 | | | | | |
| 18 | | | | | |
| 19 | | | | | |
| 20 | 520 | -45,5 | -1,3 | -3,4 | |
| 21 | 540 | -25,5 | -0,73 | -1,9 | |

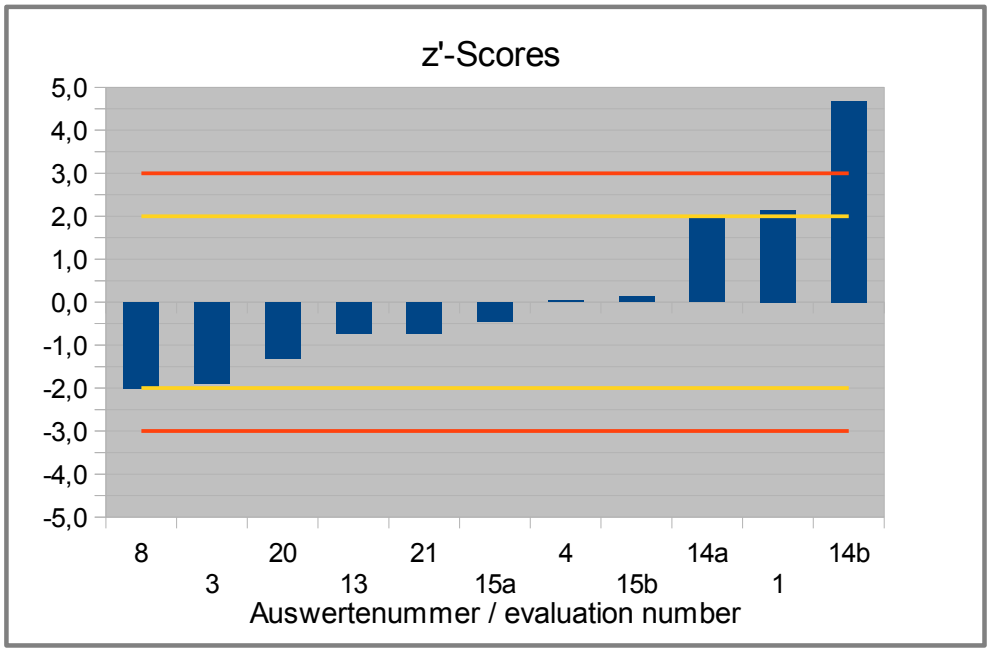


Abb. / Fig. 6: z'-Scores Fructose Dotierungsniveauprobe / fructose spiking level sample

## 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/100g] | pos/neg | [mg/100g] | Agreement with consensus value | |
| 1 | negative | <10 | positive | 100 | 2/2 (100%) | |
| 2 | negative | <LOQ | positive | 103 | 2/2 (100%) | |
| 3 | negative | <2 | positive | 94,9 | 2/2 (100%) | |
| 4 | negative | <6 | positive | 106 | 2/2 (100%) | |
| 5 | negative | <10 | positive | 438000 | 2/2 (100%) | |
| 6 | negative | <LOD | positive | 87,0 | 2/2 (100%) | |
| 7 | negative | | positive | 104 | 2/2 (100%) | |
| 8 | negative | | positive | 50,7 | 2/2 (100%) | |
| 9 | negative | <100 | positive | <100 | 2/2 (100%) | |
| 10 | negative | <3,4 | positive | 108 | 2/2 (100%) | |
| 11 | negative | < LOQ | positive | 112 | 2/2 (100%) | |
| 12a | negative | <20 | positive | 100 | 2/2 (100%) | for LOQ s. documentation |
| 12b | negative | <5 | positive | 107 | 2/2 (100%) | |
| 13 | negative | < 10 | positive | 100 | 2/2 (100%) | |
| 14a | negative | | positive | 99,0 | 2/2 (100%) | |
| 14b | | | positive | 115 | 1/1 (100%) | |
| 15a | negative | <50 | positive | 70,0 | 2/2 (100%) | |
| 15b | negative | <50 | positive | 110 | 2/2 (100%) | |
| 16 | positive | 158 | positive | 265 | 1/2 (50%) | |
| 17 | positive | <7,286 | positive | 118 | 1/2 (50%) | |
| 18 | negative | 0 | positive | 132 | 2/2 (100%) | |
| 19 | positive | 0,535 | positive | 0,660 | 1/2 (50%) | |
| 20 | negative | n.d. | positive | 90,0 | 2/2 (100%) | |
| 21 | negative | <5 | positive | 110 | 2/2 (100%) | |

| | Sample A | | Sample B | |
|---|---|---|---|---|
| Number positive | 3 | | 24 | |
| Number negative | 20 | | 0 | |
| Percent positive | 13 | | 100 | |
| Percent negative | 87 | | 0 | |
| Consensus value | negative | | positive | |

*Comments:*
*The consensus values are in qualitative agreement with the spiking of sample B. 3 positive results for sample A were obained, partly below the limit of quantification.*

### 4.2.2 Lactose Sample B (in mg/100g)

**Vergleichsuntersuchung / Proficiency Test**

| **Statistic Data** | |
|---|---|
| *Number of results* | 21 |
| *Number of outliers* | 2 |
| Mean | 109 |
| Median | 104 |
| **Robust Mean (X)** | **104** |
| **Robust standard deviation (S*)** | **13,1** |
| *Target range:* | |
| **Target standard deviation $\sigma_{pt}$** | **8,15** |
| Target standard deviation (for Information) | 5,84 |
| **lower limit of target range** | **87,5** |
| **upper limit of target range** | **120** |
| *Quotient S*/$\sigma_{pt}$* | *1,6* |
| *Standard uncertainty $U_{(Xpt)}$* | *3,57* |
| *Results in the target range* | *16* |
| *Percent in the target range* | *76%* |

** without result No. 5 and 19 (excluded in advance)*

*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 $S^*/\sigma_{pt}$ 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.*

*76% of results were in the target range.*

*The robust mean of participant results was 91 % of the spiking level of lactose to sample B (s. p. 5).*

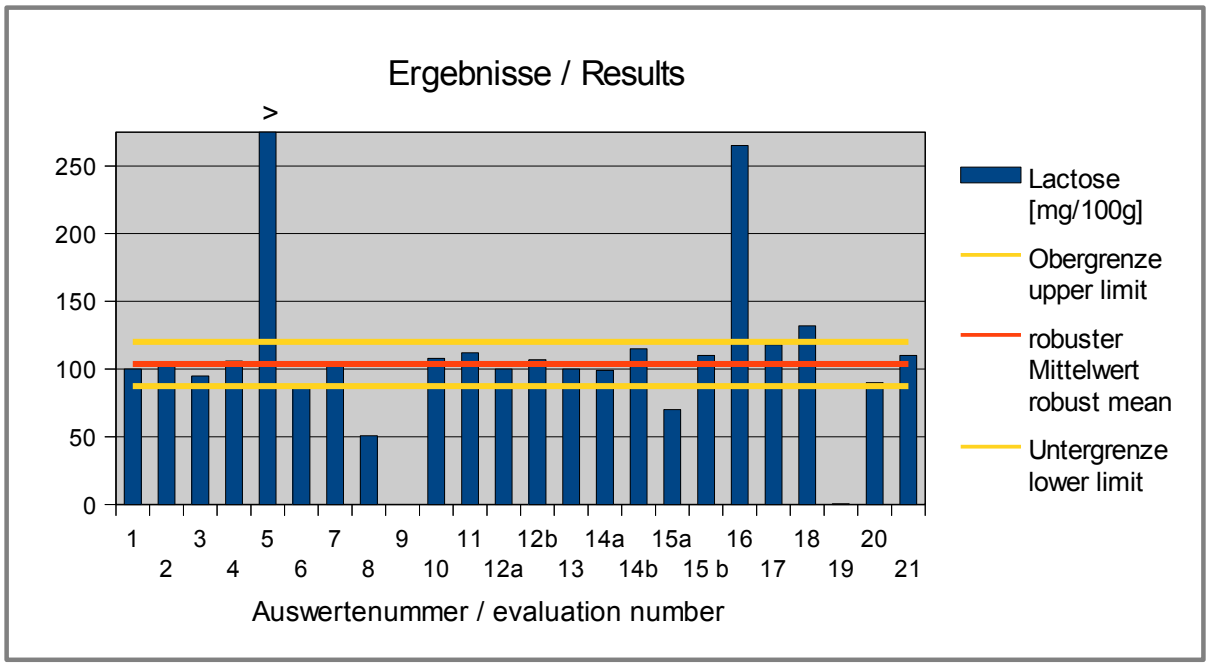


**Abb. / Fig. 7:** Ergebnisse Lactose Probe B / Results lactose sample B

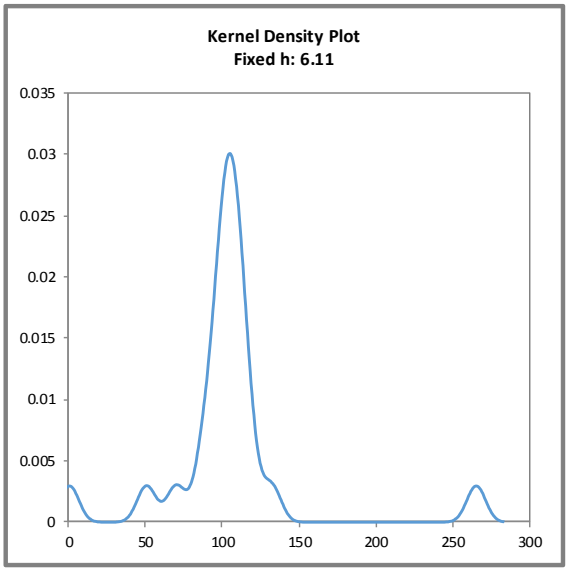


**Abb. / Fig. 8:**
Kerndichte-Schätzung der Ergebnisse
(mit h = 0,75 x $\sigma_{pt}$ von $X_{pt}$)

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

*Comment:*
The kernel density shows nearly a symmetrical distribution of results with three small side peaks < 80 mg/100g and one side peak at approx. 260 mg/100g, due to single results below and above the target range. Result no. 5 is not shown.

**Ergebnisse der Teilnehmer:**
**Results of Participants:**

| Auswerte-nummer<br>Evaluation number | Lactose [mg/100g] | Abweichung [mg/100g]<br>Deviation [mg/100g] | z-Score ($\sigma_{pt}$) | z-Score (Info) | Hinweis<br>Remark |
|---|---|---|---|---|---|
| 1 | 100 | -3,8 | -0,46 | -0,64 | |
| 2 | 103 | -0,8 | -0,09 | -0,13 | |
| 3 | 94,9 | -8,9 | -1,1 | -1,5 | |
| 4 | 106 | 2,2 | 0,27 | 0,38 | |
| 5 | 438000 | | | | Ergebnis ausgeschlossen / Result excluded |
| 6 | 87,0 | -16,8 | -2,1 | -2,9 | |
| 7 | 104 | 0,2 | 0,03 | 0,04 | |
| 8 | 50,7 | -53,1 | -6,5 | -9,1 | |
| 9 | <100 | | | | |
| 10 | 108 | 4,2 | 0,52 | 0,73 | |
| 11 | 112 | 8,2 | 1,0 | 1,4 | |
| 12a | 100 | -3,8 | -0,46 | -0,64 | LOQ s. documentation |
| 12b | 107 | 3,2 | 0,40 | 0,55 | |
| 13 | 100 | -3,8 | -0,46 | -0,64 | |
| 14a | 99,0 | -4,8 | -0,58 | -0,82 | |
| 14b | 115 | 11,2 | 1,4 | 1,9 | |
| 15a | 70,0 | -33,8 | -4,1 | -5,8 | |
| 15b | 110 | 6,2 | 0,77 | 1,1 | |
| 16 | 265 | 161,2 | 20 | 28 | |
| 17 | 118 | 14,3 | 1,8 | 2,4 | |
| 18 | 132 | 28,2 | 3,5 | 4,8 | |
| 19 | 0,660 | | | | Ergebnis ausgeschlossen / Result excluded |
| 20 | 90 | -13,8 | -1,7 | -2,4 | |
| 21 | 110 | 6,24 | 0,77 | 1,1 | |

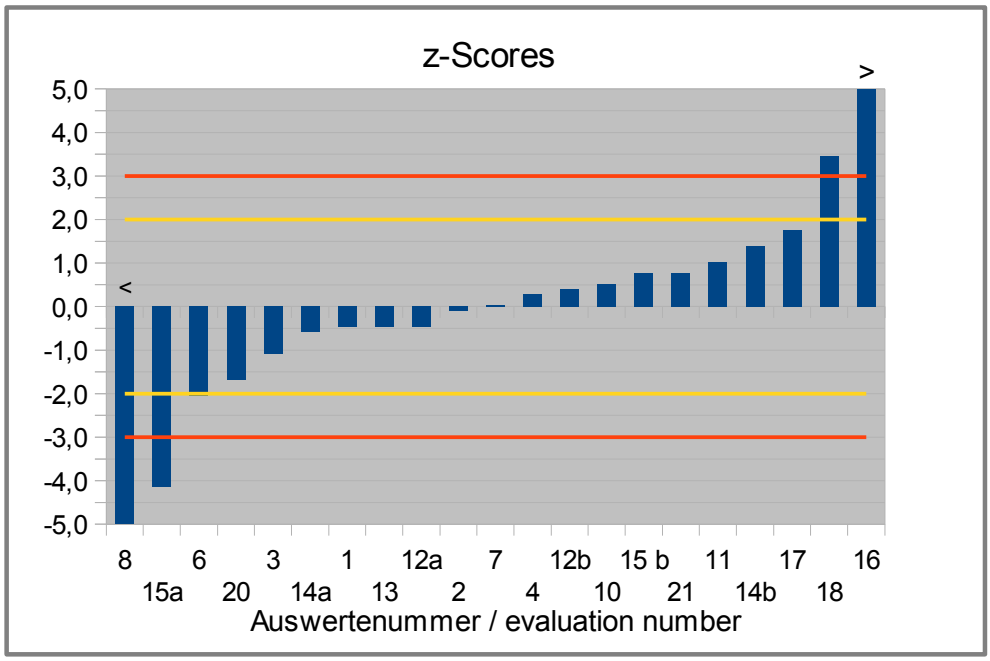


Abb. / Fig. 9: z-Scores Lactose Probe B / lactose sample B

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

**Vergleichsuntersuchung / Proficiency Test**

| **Statistic Data** | |
|---|---|
| *Number of results** | 20 |
| *Number of outliers* | 1 |
| Mean | 104 |
| Median | 98,3 |
| **Robust Mean (X)** | **96,7** |
| **Robust standard deviation (S*)** | **10,5** |
| *Target range:* | |
| **Target standard deviation $\sigma_{pt}$** | **7,60** |
| Target standard deviation (for Information) | 5,50 |
| **lower limit of target range** | **81,5** |
| **upper limit of target range** | **112** |
| *Quotient S*/$\sigma_{pt}$* | *1,4* |
| *Standard uncertainty $U_{(Xpt)}$* | *2,93* |
| *Results in the target range* | *17* |
| *Percent in the target range* | *85%* |

** without result No. 19 (excluded in advance)*

*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 $S^*/\sigma_{pt}$ 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.*

*85% of results were in the target range.*

*The robust mean of participant results was 87 % of the spiking level of lactose to the spiking level sample (s. p. 5).*

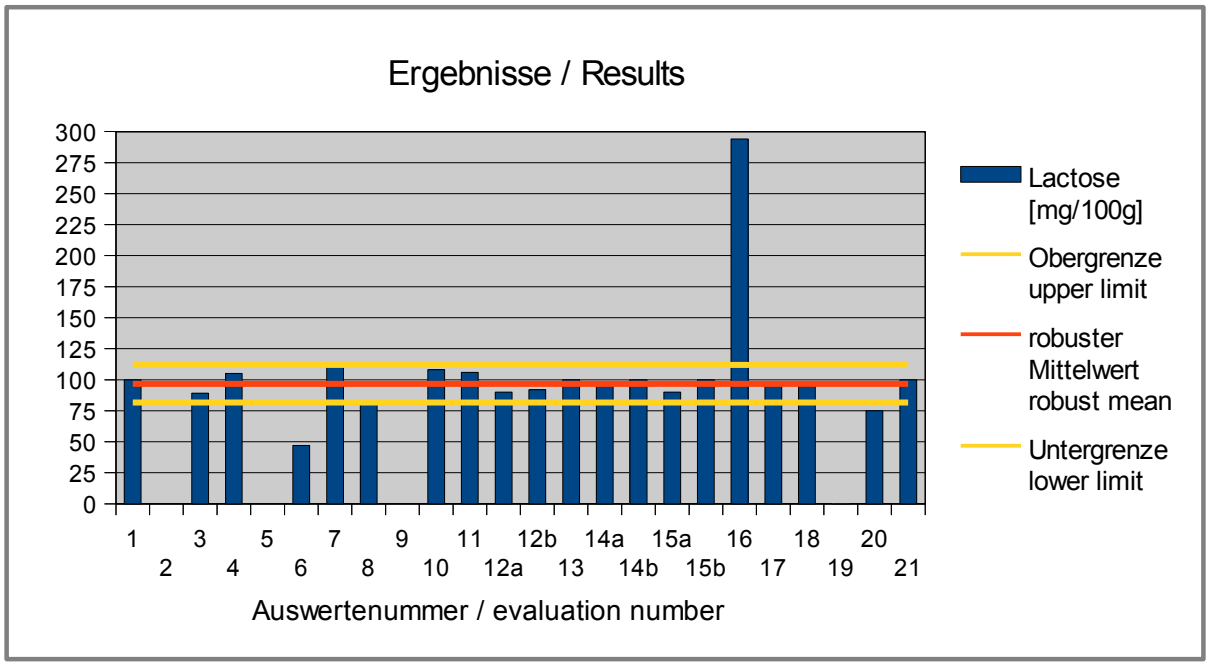


**Abb. / Fig. 10:** Ergebnisse Lactose Dotierungsniveauprobe / Results lactose spiking level sample

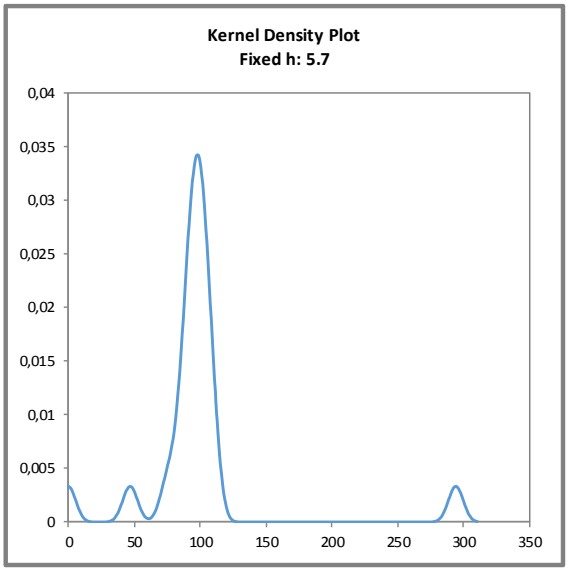


**Abb. / Fig. 11:**
Kerndichte-Schätzung der Ergebnisse (mit h = 0,75 x $\sigma_{pt}$ von $X_{pt}$)

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

*Comment:*
The kernel density shows nearly a symmetrical distribution of results with two side peaks < 60 mg/100g and a side peakt at approx. 290 mg/100g, due to three results above the target range and a shoulder, due to single results below and above the target range.

**Ergebnisse der Teilnehmer:**
**Results of Participants:**

| Auswerte-nummer<br>Evaluation number | Lactose [mg/100g] | Abweichung [mg/100g]<br>Deviation [mg/100g] | z-Score ($\sigma_{pt}$) | z-Score (Info) | Hinweis<br>Remark |
|---|---|---|---|---|---|
| 1 | 100 | 3,3 | 0,43 | 0,60 | |
| 2 | <LOQ | | | | |
| 3 | 89,2 | -7,5 | -1,0 | -1,4 | |
| 4 | 105 | 8,3 | 1,1 | 1,5 | |
| 5 | | | | | |
| 6 | 47,0 | -49,7 | -6,5 | -9,0 | |
| 7 | 110 | 13,3 | 1,7 | 2,4 | |
| 8 | 82,8 | -14,0 | -1,8 | -2,5 | |
| 9 | <100 | | | | |
| 10 | 108 | 11,3 | 1,5 | 2,1 | |
| 11 | 106 | 9,3 | 1,2 | 1,7 | |
| 12a | 90,0 | -6,7 | -0,88 | -1,2 | <LOQ |
| 12b | 92,0 | -4,7 | -0,62 | -0,86 | |
| 13 | 100 | 3,3 | 0,43 | 0,60 | |
| 14a | 96,5 | -0,2 | -0,03 | -0,04 | |
| 14b | 100 | 3,3 | 0,43 | 0,60 | |
| 15a | 90,0 | -6,7 | -0,88 | -1,2 | |
| 15b | 100 | 3,3 | 0,43 | 0,60 | |
| 16 | 294 | 197,3 | 26 | 36 | |
| 17 | 95,6 | -1,1 | -0,14 | -0,20 | |
| 18 | 94,9 | -1,8 | -0,24 | -0,33 | |
| 19 | 0,0100 | | | | Ergebnis ausgeschlossen / Result excluded |
| 20 | 75,0 | -21,7 | -2,9 | -4,0 | |
| 21 | 100 | 3,3 | 0,43 | 0,60 | |

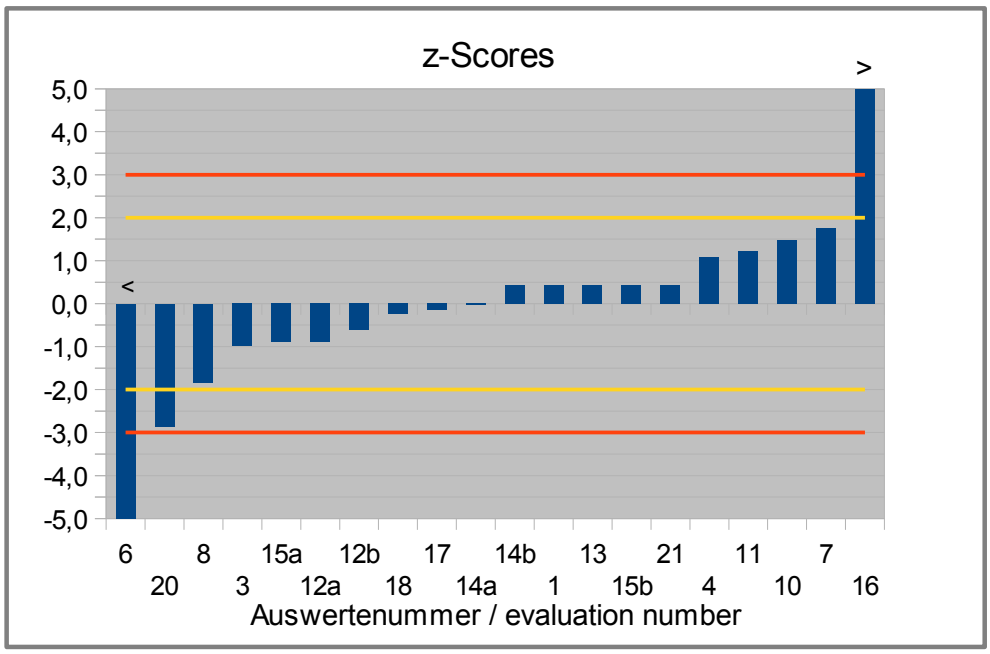


Abb. / Fig. 12: z-Scores Lactose Dotierungsniveauprobe / lactose spiking level sample

### 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.

***Spiking Level Sample and Sample B***

| Evaluation number | Spiking Level Sample | Recovery rate* | Sample B | Recovery rate* | Remarks |
|---|---|---|---|---|---|
| | [mg/100g] | [%] | [mg/100g] | [%] | |
| 1 | 100 | **90** | 100 | **87** | |
| 2 | <LOQ | | 103 | **90** | |
| 3 | 89,2 | 80 | 94,9 | 83 | |
| 4 | 105 | **95** | 106 | **93** | |
| 5 | | | 438000 | 382533 | |
| 6 | 47,0 | 42 | 87,0 | 76 | |
| 7 | 110 | **99** | 104 | **91** | |
| 8 | 82,8 | 75 | 50,7 | 44 | |
| 9 | <100 | | <100 | | |
| 10 | 108 | **97** | 108 | **94** | |
| 11 | 106 | **95** | 112 | **98** | |
| 12a | 90,0 | 81 | 100 | **87** | LOQ s. documentation |
| 12b | 92,0 | 83 | 107 | **93** | |
| 13 | 100 | **90** | 100 | **87** | |
| 14a | 96,5 | **87** | 99,0 | **86** | |
| 14b | 100 | **90** | 115 | **100** | |
| 15a | 90,0 | 81 | 70,0 | 61 | |
| 15b | 100 | **90** | 110 | **96** | |
| 16 | 294 | 265 | 265 | 231 | |
| 17 | 95,6 | **86** | 118 | **103** | |
| 18 | 94,9 | **85** | 132 | **115** | |
| 19 | 0,0100 | 0,0090 | 0,660 | 0,58 | |
| 20 | 75,0 | 68 | 90,0 | 79 | |
| 21 | 100 | **90** | 110 | **96** | |

| RA** | 85-115 % | RA** | 85-115 % |
|---|---|---|---|
| Numbers in RA | **12** | Numbers in RA | **15** |
| | | | |
| Percent in RA | **57** | Percent in RA | **65** |
| | | | |

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

** Range of acceptance 3.12 (s. page 14)

***Comments:***

*For the spiking level sample 57% (12) of the participants obtained a recovery rate within the range of 85-115%. For the spiked food matrix sample A 65% (15) of the recovery rates were in this range.*

## 4.3 Galactose

*For galactose no results above the detection or quantification limits were reported for samples A and B (see documentation).*

### *4.3.1 Galactose Spiking Level Sample (in mg/100g)*

**Vergleichsuntersuchung / Proficiency Test**

Due to the small number of results (<7) no statistical evaluation was done.

**Ergebnisse der Teilnehmer:**
**Results of Participants:**

| Auswerte-nummer<br>Evaluation number | Galactose [mg/100g] | Abweichung [mg/100g]<br>Deviation [mg/100g] | z-Score ($\sigma_{pt}$) | z-Score (Info) | Hinweis<br>Remark |
|---|---|---|---|---|---|
| 1 | | | | | |
| 2 | | | | | |
| 3 | <50 | | | | |
| 4 | 5 | | | | |
| 5 | | | | | |
| 6 | | | | | |
| 7 | | | | | |
| 8 | | | | | |
| 9 | <100 | | | | |
| 10 | | | | | |
| 11 | | | | | |
| 12a | | | | | |
| 12b | | | | | |
| 13 | | | | | |
| 14a | | | | | |
| 14b | | | | | |
| 15a | not applicable | | | | |
| 15b | <20 | | | | |
| 16 | | | | | |
| 17 | | | | | |
| 18 | | | | | |
| 19 | | | | | |
| 20 | 5 | | | | |
| 21 | <100 | | | | |

*Comments:*
Two participants obtained amounts of 5 mg/100g in the spiking level sample.

# 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**

| Analyte | Partici-pant | Unit | Date of analysis | Final result | Detectable | LOD | LOQ | Incl. RR | Recovery rate [%] |
|---|---|---|---|---|---|---|---|---|---|
| Sample A | | | Day /Month | mg/100g | yes / no | | | | |
| Fructose | 1 | mg/100g | 10.05.19 | <0,01 g/100g | no | 0,003 | 0,01 | | |
| | 2 | mg/100g | | | | | | | |
| | 3 | mg/100g | 15.05.19 | <500 | no | - | 500 | no | - |
| | 4 | mg/100g | | <100 | | | <100 | no | |
| | 5 | mg/100g | | | | | | | |
| | 6 | mg/100g | 13.05.19 | <Limit of detection | no | 100 | 300 | no | |
| | 7 | mg/100g | | | | | | | |
| | 8 | mg/100g | 16.05.19 | 15,47 | | 25 | 25 | no | |
| | 9 | mg/100g | | | | | | | |
| | 10 | mg/100g | | | | | | | |
| | 11 | mg/100g | | | | | | | |
| | 12a | mg/100g | | | | | | | |
| | 12b | mg/100g | | | | | | | |
| | 13 | mg/100g | 24.04.19 | < 25 | no | 10 | 25 | no | |
| | 14 | mg/100g | 08.05.19 | | no | 10 | 30 | no | |
| | 15a | mg/100g | 09.05.19 | <50 | no | | 50 | | |
| | 15 b | mg/100g | 11.4. | <100 | no | | 100 | | |
| | 16 | mg/100g | | | | | | | |
| | 17 | mg/100g | | | | | | | |
| | 18 | mg/100g | | | | | | | |
| | 19 | mg/100g | | | | | | | |
| | 20 | mg/100g | 11.04.19 | 30 | yes | | | no | |
| | 21 | mg/100g | 10.04.19 | <100 | no | 100 | | | |

**Fructose Sample B**

| Analyte | Partici-pant | Unit | Date of analysis | Final result | Detectable | LOD | LOQ | Incl. RR | Recovery rate [%] |
|---|---|---|---|---|---|---|---|---|---|
| Sample B | | | Day /Month | mg/100g | yes / no | | | | |
| Fructose | 1 | mg/100g | 10.05.19 | 0,69 g/100g | yes | 0,003 | 0,01 | | |
| | 2 | mg/100g | | | | | | | |
| | 3 | mg/100g | 15.05.19 | 500 | yes | | 500 | no | |
| | 4 | mg/100g | | 558 | yes | | <100 | no | |
| | 5 | mg/100g | | | | | | | |
| | 6 | mg/100g | 13.05.19 | 487 | yes | 100 | 300 | no | |
| | 7 | mg/100g | | | | | | | |
| | 8 | mg/100g | 16.05.19 | 494,46 | | 25 | 25 | no | |
| | 9 | mg/100g | | | | | | | |
| | 10 | mg/100g | | | | | | | |
| | 11 | mg/100g | | | | | | | |
| | 12a | mg/100g | | | | | | | |
| | 12b | mg/100g | | | | | | | |
| | 13 | mg/100g | 24.04.19 | 520 | Yes | 10 | 25 | no | |
| | 14 | mg/100g | 08.05.19 | 537 | yes | 10 | 30 | no | |
| | 15a | mg/100g | 09.05.19 | 570 | yes | | 50 | | |
| | 15 b | mg/100g | 11.4. | 530 | yes | | 100 | | |
| | 16 | mg/100g | | | | | | | |
| | 17 | mg/100g | | | | | | | |
| | 18 | mg/100g | | | | | | | |
| | 19 | mg/100g | | | | | | | |
| | 20 | mg/100g | 11.04.19 | 490 | yes | | | no | |
| | 21 | mg/100g | 10.04.19 | 490 | yes | 100 | | | |

**Fructose Spiking Level Sample**

| Analyte | Partici-pant | Unit | Date of analysis | Final result | Detectable | LOD | LOQ | Incl. RR | Recovery rate [%] |
|---|---|---|---|---|---|---|---|---|---|
| Spiking Level Sample | | | Day /Month | mg/100g | yes / no | | | | |
| Fructose | 1 | mg/100g | 10.05.19 | 0,64 g/100g | yes | 0,003 | 0,01 | | |
| | 2 | mg/100g | | | | | | | |
| | 3 | mg/100g | 15.05.19 | 500 | yes | | 500 | no | - |
| | 4 | mg/100g | | 567 | yes | | <100 | no | |
| | 5 | mg/100g | | | | | | | |
| | 6 | mg/100g | 13.05.19 | <Limit of quantification | yes | 100 | 300 | no | |
| | 7 | mg/100g | | | | | | | |
| | 8 | mg/100g | 16.05.19 | 495,3 | | 25 | 25 | no | |
| | 9 | mg/100g | | | | | | | |
| | 10 | mg/100g | | | | | | | |
| | 11 | mg/100g | | | | | | | |
| | 12a | mg/100g | | | | | | | |
| | 12b | mg/100g | | | | | | | |
| | 13 | mg/100g | 24.04.19 | 540 | Yes | 10 | 25 | no | 101 |
| | 14 | mg/100g | 08.05.19 | 635,5 | yes | 10 | 30 | no | |
| | 15a | mg/100g | 09.05.19 | 550 | yes | | 50 | | |
| | 15 b | mg/100g | 11.4. | 570 | yes | | 100 | | |
| | 16 | mg/100g | | | | | | | |
| | 17 | mg/100g | | | | | | | |
| | 18 | mg/100g | | | | | | | |
| | 19 | mg/100g | | | | | | | |
| | 20 | mg/100g | 11.04.19 | 520 | yes | | | no | |
| | 21 | mg/100g | 10. Apr | 540 | yes | 100 | | | |

## Lactose Sample A

| Analyte | Partici-pant | Unit | Date of analysis | Final result | Detectable | LOD | LOQ | Incl. RR | Recovery rate [%] |
|---|---|---|---|---|---|---|---|---|---|
| Sample A | | | Day /Month | mg/100g | yes / no | | | | |
| Lactose | 1 | mg/100g | 10.05.19 | <0,01 g/100g | no | 0,003 | 0,01 | | |
| | 2 | mg/100g | 14.05.19 | <LOQ | NO | 40 | 75 | NO | |
| | 3 | mg/100g | 13.05.19 | <2 | no | - | 2 | no | - |
| | 4 | mg/100g | 13.05.19 | <6 | | | <6 | no | |
| | 5 | mg/100g | 08.05.19 | <10 | no | 10 | 10 | no\ | |
| | 6 | mg/100g | 13.05.19 | <Limit of detection | no | 1,4 | 5 | no | |
| | 7 | mg/100g | 09.05.19 | | no | 14 | 70 | no | 103 |
| | 8 | mg/100g | 16.05.19 | | no | 25 | 25 | no | |
| | 9 | mg/100g | 16.05. | <100 | no | 10 | 100 | no | 99 |
| | 10 | mg/100g | 15.05.19 | <3.4 | no | 1,1 | 3,4 | yes | 98,56 |
| | 11 | mg/100g | 11.04.19 | < LOQ | no | 2 | 5 | no | 0,98 |
| | 12a | mg/100g | 08.05.19 | <20 | no | 20 | 50 | no | |
| | 12b | mg/100g | 10.05.19 | <5 | no | 5 | 15 | no | |
| | 13 | mg/100g | 24.04.19 | < 10 | no | 3 | 10 | no | |
| | 14 | mg/100g | 08.05.19 | | no | 10 | 30 | no | |
| | 15a | mg/100g | 09.05.19 | <50 | no | | 50 | | |
| | 15 b | mg/100g | 12.4. | <50 | no | | 50 | | |
| | 16 | mg/100g | 13.05.19 | 158 | yes | | 10 | no | |
| | 17 | mg/100g | 24.05.19 | <7.286 | Yes | 7,286 | 7,286 | No | |
| | 18 | mg/100g | 23.04.19 | 0 | no | | 2 | yes | 99 |
| | 19 | mg/100g | 19.04.19 | 0,535 | yes | | | | |
| | 20 | mg/100g | 08.04.19 | n.n. | no | 4 | 14 | no | |
| | 21 | mg/100g | 10.04.19 | <5 | no | 5 | | | |

**Lactose Sample B**

| Analyte | Partici-pant | Unit | Date of analysis | Final result | Detectable | LOD | LOQ | Incl. RR | Recovery rate [%] |
|---|---|---|---|---|---|---|---|---|---|
| Sample B | | | Day /Month | mg/100g | yes / no | | | | |
| Lactose | 1 | mg/100g | 10.05.19 | 0,1 g/100g | yes | 0,003 | 0,01 | | |
| | 2 | mg/100g | 14.05.19 | 103 | YES | 40 | 75 | NO | |
| | 3 | mg/100g | 13.05.19 | 94,9 | yes | - | 2 | no | - |
| | 4 | mg/100g | 13.05.19 | 106 | yes | | <6 | no | |
| | 5 | mg/100g | 08.05.19 | 438000 | yes | 10 | 10 | no\ | |
| | 6 | mg/100g | 13.05.19 | 87 | yes | 1,4 | 5 | no | |
| | 7 | mg/100g | 09.05.19 | 104 | yes | 14 | 70 | no | 103 |
| | 8 | mg/100g | 16.05.19 | 50,71 | | 25 | 25 | no | |
| | 9 | mg/100g | 16.05. | <100 | yes | 10 | 100 | no | 99,4 |
| | 10 | mg/100g | 15.05.19 | 108 | yes | 1,1 | 3,4 | yes | 98,56 |
| | 11 | mg/100g | 11.04.19 | 112 | yes | 2 | 5 | no | 0,98 |
| | 12a | mg/100g | 08.05.19 | <200 | yes | 50 | 200 | no | |
| | 12b | mg/100g | 10.05.19 | 107 | yes | 3 | 10 | no | |
| | 13 | mg/100g | 24.04.19 | 100 | Yes | 3 | 10 | no | 103 |
| | 14 | mg/100g | 08.05.19 | 99 | yes | 10 | 30 | no | |
| | 15a | mg/100g | 09.05.19 | 70 | yes | | 50 | | |
| | 15 b | mg/100g | 12.4. | 110 | yes | | 20 | | |
| | 16 | mg/100g | 13.05.19 | 265 | yes | | 10 | no | |
| | 17 | mg/100g | 24.05.19 | 118,035 | Yes | 7,286 | 7,286 | No | |
| | 18 | mg/100g | 07.05.19 | 132 | yes | | 2 | yes | 99 |
| | 19 | mg/100g | 19.04.19 | 0,66 | yes | | | | |
| | 20 | mg/100g | 08.04.19 | 90 | yes | 4 | 14 | no | |
| | 21 | mg/100g | 10.04.19 | 110 | yes | 5 | | | |

**Lactose Spiking Level Sample**

| Analyte | Partici-pant | Unit | Date of analysis | Final result | Detectable | LOD | LOQ | Incl. RR | Recovery rate [%] |
|---|---|---|---|---|---|---|---|---|---|
| Spiking Level Sample | | | Day /Month | mg/100g | yes / no | | | | |
| Lactose | 1 | mg/100g | 10.05.19 | 0,1 g/100g | yes | 0,003 | 0,01 | | |
| | 2 | mg/100g | 14.05.19 | <LOQ | YES | 40 | 75 | NO | |
| | 3 | mg/100g | 13.05.19 | 89,2 | yes | - | 2 | no | - |
| | 4 | mg/100g | 13.05.19 | 105 | yes | | <6 | no | |
| | 5 | mg/100g | | | | | | | |
| | 6 | mg/100g | 13.05.19 | 47 | yes | 1,4 | 5 | no | |
| | 7 | mg/100g | 09.05.19 | 110 | yes | 29 | 144 | no | 103 |
| | 8 | mg/100g | 16.05.19 | 82,75 | | 25 | 25 | no | |
| | 9 | mg/100g | 16.05. | <100 | yes | 10 | 100 | no | 99,2 |
| | 10 | mg/100g | 15.05.19 | 108 | yes | 1,1 | 3,4 | yes | 98,56 |
| | 11 | mg/100g | 11.04.19 | 106 | yes | 2 | 5 | no | 0,98 |
| | 12a | mg/100g | 08.05.19 | <200 | yes | 50 | 200 | no | |
| | 12b | mg/100g | 10.05.19 | 92 | yes | 3 | 10 | no | |
| | 13 | mg/100g | 24.04.19 | 100 | Yes | 3 | 10 | no | 100 |
| | 14 | mg/100g | 08.05.19 | 96,5 | yes | 10 | 30 | no | |
| | 15a | mg/100g | 09.05.19 | 90 | yes | | 50 | | |
| | 15 b | mg/100g | 12.4. | 100 | yes | | 20 | | |
| | 16 | mg/100g | 13.05.19 | 294 | yes | | 10 | no | |
| | 17 | mg/100g | 24.05.19 | 95,63 | Yes | 7,286 | 7,286 | No | |
| | 18 | mg/100g | 30.04.19 | 94,9 | yes | | 2 | yes | 99 |
| | 19 | mg/100g | 19.04.19 | 0,01 | yes | | | | |
| | 20 | mg/100g | 08.04.19 | 75 | yes | 4 | 14 | no | |
| | 21 | mg/100g | 10. Apr | 100 | yes | 5 | | | |

## Galactose Sample A

| Analyte | Partici-pant | Unit | Date of analysis | Final result | Detectable | LOD | LOQ | Incl. RR | Recovery rate [%] |
|---|---|---|---|---|---|---|---|---|---|
| Sample A | | | Day /Month | mg/100g | yes / no | | | | |
| Galactose | 1 | mg/100g | | | | | | | |
| | 2 | mg/100g | | | | | | | |
| | 3 | mg/100g | 15.05.19 | <50 | no | - | 50 | no | - |
| | 4 | mg/100g | | <3 | | | <3 | no | |
| | 5 | mg/100g | | | | | | | |
| | 6 | mg/100g | | | | | | | |
| | 7 | mg/100g | 09.05.19 | | no | 7,5 | 38 | no | 103 |
| | 8 | mg/100g | 16.05.19 | | no | 25 | 25 | no | |
| | 9 | mg/100g | 16.05. | <100 | no | 10 | 100 | no | |
| | 10 | mg/100g | | | | | | | |
| | 11 | mg/100g | | | | | | | |
| | 12a | mg/100g | | | | | | | |
| | 12b | mg/100g | | | | | | | |
| | 13 | mg/100g | | | | | | | |
| | 14 | mg/100g | | | | | | | |
| | 15a | mg/100g | 09.05.19 | not applicable | | | 50 | | |
| | 15 b | mg/100g | 12.4. | <50 | no | | 50 | | |
| | 16 | mg/100g | | | | | | | |
| | 17 | mg/100g | | | | | | | |
| | 18 | mg/100g | | | | | | | |
| | 19 | mg/100g | | | | | | | |
| | 20 | mg/100g | 08.04.19 | n.n. | no | | | no | |
| | 21 | mg/100g | 10.04.19 | <100 | no | 100 | | | |

**Galactose Sample B**

| Analyte | Partici-pant | Unit | Date of analysis | Final result | Detectable | LOD | LOQ | Incl. RR | Recovery rate [%] |
|---|---|---|---|---|---|---|---|---|---|
| Sample B | | | Day /Month | mg/100g | yes / no | | | | |
| Galactose | 1 | mg/100g | | | | | | | |
| | 2 | mg/100g | | | | | | | |
| | 3 | mg/100g | 15.05.19 | <50 | no | - | 50 | no | - |
| | 4 | mg/100g | | <3 | no | | <3 | no | |
| | 5 | mg/100g | | | | | | | |
| | 6 | mg/100g | | | | | | | |
| | 7 | mg/100g | 09.05.19 | | no | 7,5 | 38 | no | 103 |
| | 8 | mg/100g | 16.05.19 | | no | 25 | 25 | no | |
| | 9 | mg/100g | 16.05. | <100 | no | 10 | 100 | no | |
| | 10 | mg/100g | | | | | | | |
| | 11 | mg/100g | | | | | | | |
| | 12a | mg/100g | | | | | | | |
| | 12b | mg/100g | | | | | | | |
| | 13 | mg/100g | | | | | | | |
| | 14 | mg/100g | | | | | | | |
| | 15a | mg/100g | 09.05.19 | not applicable | | | 50 | | |
| | 15 b | mg/100g | 12.4. | <20 | no | | 20 | | |
| | 16 | mg/100g | | | | | | | |
| | 17 | mg/100g | | | | | | | |
| | 18 | mg/100g | | | | | | | |
| | 19 | mg/100g | | | | | | | |
| | 20 | mg/100g | 08.04.19 | n.d. | no | | | no | |
| | 21 | mg/100g | 10.04.19 | <100 | no | 100 | | | |

**Galactose Spiking Level Sample**

| Analyte | Partici-pant | Unit | Date of analysis | Final result | Detectable | LOD | LOQ | Incl. RR | Recovery rate [%] |
|---|---|---|---|---|---|---|---|---|---|
| Spiking Level Sample | | | Day /Month | mg/100g | yes / no | | | | |
| Galactose | 1 | mg/100g | | | | | | | |
| | 2 | mg/100g | | | | | | | |
| | 3 | mg/100g | 15.05.19 | <50 | no | - | 50 | no | - |
| | 4 | mg/100g | | 5 | yes | | <3 | no | |
| | 5 | mg/100g | | | | | | | |
| | 6 | mg/100g | | | | | | | |
| | 7 | mg/100g | 08.05.19 | | no | 38 | 189 | no | 103 |
| | 8 | mg/100g | 16.05.19 | | no | 25 | 25 | no | |
| | 9 | mg/100g | 16.05. | <100 | no | 10 | 100 | no | |
| | 10 | mg/100g | | | | | | | |
| | 11 | mg/100g | | | | | | | |
| | 12a | mg/100g | | | | | | | |
| | 12b | mg/100g | | | | | | | |
| | 13 | mg/100g | | | | | | | |
| | 14 | mg/100g | | | | | | | |
| | 15a | mg/100g | 09.05.19 | not applicable | | | 50 | | |
| | 15 b | mg/100g | 12.4. | <20 | no | | 20 | | |
| | 16 | mg/100g | | | | | | | |
| | 17 | mg/100g | | | | | | | |
| | 18 | mg/100g | | | | | | | |
| | 19 | mg/100g | | | | | | | |
| | 20 | mg/100g | 08.04.19 | 5 | no | | | no | |
| | 21 | mg/100g | 10. Apr | <100 | no | 100 | | | |

## 5.1.2 Analytical Methods

### Fructose Sample A

| Analyte | Partici-pant | Method description as in test report / norm / litera-ture | Sample preparati-on | Measuring me-thod | Calibration / Reference ma-terial | Recovery rate with same matrix | Method accredited ISO/IEC 17025 | Further Remarks |
|---|---|---|---|---|---|---|---|---|
| | | | | | | | | |
| Fructose | 1 | Fructose, HPLC SOP Code: 084L02xx (inhouse) | extraction aqueous, Carrez Klärung | HPAEC-PAD | yes | | yes | given as g/100g |
| | 2 | | | | | | | |
| | 3 | HPLC-IR - internal method PNTA0101 | | | external calib. curve and internal RM | no | yes | |
| | 4 | enzymatic | | | | | yes | |
| | 5 | | | | | | | |
| | 6 | MP.0002.R1.2018 | Extraction with water | IC quantification of sample extract | External std Fructosa Sigma | no | yes | |
| | 7 | | | | | | | |
| | 8 | Carbohydrates by ion chromatography | | | | | | |
| | 9 | | | | | | | |
| | 10 | | | | | | | |
| | 11 | | | | | | | |
| | 12a | | | | | | | |
| | 12b | | | | | | | |
| | 13 | internal method A/75 | | HPLC/PAD | Sigma Aldrich | | yes | |
| | 14 | Enzymatic | homogenisation, aqueouse extraction, Carrez-precipitation, filtration | | Standards from Enzyme-Kit r-biopharm | no | yes | |
| | 15a | Internal method HPAEC-PAD | extraction with water and precipitation by Carrez-solution | HPAEC-PAD | | | yes | |
| | 15 b | r-biopharm Test-Combination 10 139 106 035:2011-05 | | | | | yes | |
| | 16 | | | | | | | |
| | 17 | | | | | | | |
| | 18 | | | | | | | |
| | 19 | | | | | | | |
| | 20 | ASU L 48.01-3, modified | | | | no | yes | |
| | 21 | HPAEC-PAD | | | | | no | |

## Fructose Sample B

| Analyte | Partici-pant | Method description as in test report / norm / litera-ture | Sample preparati-on | Measuring me-thod | Calibration / Reference ma-terial | Recovery rate with same matrix | Method accredited ISO/IEC 17025 | Further Remarks |
|---|---|---|---|---|---|---|---|---|
| | | | | | | | | |
| Fructose | 1 | Fructose, HPLC SOP Code: 084L02xx (inhouse) | extraction aqueous, Carrez Klärung | HPAEC-PAD | yes | | yes | given as g/100g |
| | 2 | | | | | | | |
| | 3 | HPLC-IR - internal method PNTA0101 | | | external calib. curve and internal RM | no | yes | |
| | 4 | enzymatic | | | | | yes | |
| | 5 | | | | | | | |
| | 6 | MP.0002.R1.2018 | Extraction with water | IC quantification of sample extract | External std Fructosa Sigma | no | yes | |
| | 7 | | | | | | | |
| | 8 | Carbohydrates by ion chromatography | | | | | | |
| | 9 | | | | | | | |
| | 10 | | | | | | | |
| | 11 | | | | | | | |
| | 12a | | | | | | | |
| | 12b | | | | | | | |
| | 13 | internal method A/75 | | HPLC/PAD | Sigma Aldrich | | yes | |
| | 14a | Enzymatic | homogenisation, aqueouse extraction, Carrez-precipitation, filtration | | Standards from Enzyme-Kit r-biopharm | no | yes | |
| | 14b | | | | | | | HPAEC-PAD: 544 mg/100g |
| | 15a | Internal method HPAEC-PAD | extraction with water and precipitation by Carrez-solution | HPAEC-PAD | | | yes | |
| | 15 b | r-biopharm Test-Combination 10 139 106 035:2011-05 | | | | | yes | |
| | 16 | | | | | | | |
| | 17 | | | | | | | |
| | 18 | | | | | | | |
| | 19 | | | | | | | |
| | 20 | ASU L 48.01-3, modified | | | | no | yes | |
| | 21 | HPAEC-PAD | | | | | no | |

## Fructose Spiking Level Sample

| Analyte | Partici-pant | Method description as in test report / norm / litera-ture | Sample preparati-on | Measuring me-thod | Calibration / Reference ma-terial | Recovery rate with same matrix | Method accredited ISO/IEC 17025 | Further Remarks |
|---|---|---|---|---|---|---|---|---|
| | | | | | | | | |
| Fructose | 1 | Fructose, HPLC SOP Code: 084L02xx (inhouse) | extraction aqueous, Carrez precipitation | HPAEC-PAD | yes | | yes | given as g/100g |
| | 2 | | | | | | | |
| | 3 | HPLC-IR - internal method PNTA0101 | | | external calib. curve and internal RM | no | yes | |
| | 4 | enzymatic | | | | | yes | |
| | 5 | | | | | | | |
| | 6 | MP.0002.R1.2018 | Extraction with water | IC quantification of sample extract | External std Fructosa Sigma | no | yes | |
| | 7 | | | | | | | |
| | 8 | Carbohydrates by ion chromatography | | | | | | |
| | 9 | | | | | | | |
| | 10 | | | | | | | |
| | 11 | | | | | | | |
| | 12a | | | | | | | |
| | 12b | | | | | | | |
| | 13 | internal method A/75 | | HPLC/PAD | Sigma Aldrich | | yes | |
| | 14a | Enzymatic | homogenisation, aqueouse extraction, Carrez-precipitation, filtration | | Standards from Enzyme-Kit r-biopharm | no | yes | |
| | 14b | | | | | | | HPAEC-PAD: 728 mg/100g |
| | 15a | Internal method HPAEC-PAD | extraction with water and precipitation by Carrez-solution | HPAEC-PAD | | | yes | |
| | 15 b | r-biopharm Test-Combination 10 139 106 035:2011-05 | | | | | yes | |
| | 16 | | | | | | | |
| | 17 | | | | | | | |
| | 18 | | | | | | | |
| | 19 | | | | | | | |
| | 20 | ASU L 48.01-3, modified | | | | no | yes | |
| | 21 | HPAEC-PAD | | | | | no | |

## Lactose Sample A

| Analyte | 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 |
|---|---|---|---|---|---|---|---|---|
| | | | | | | | | |
| Lactose | 1 | Fructose, HPLC SOP Code: 084L02xx (inhouse) | extraction aqueous, Carrez precipitation | HPAEC-PAD | yes | | yes | given as g/100g |
| | 2 | ENZYMATIC | | | R-BIOPHARM | | NO | |
| | 3 | LC/MS/MS - internal method PNTA0189 | | | external calib. curve and internal RM | no | yes | |
| | 4 | FOOD-PA 591, according to ASU L07.00-23, enzymatic | | | | | yes | |
| | 5 | Roche Lactose/Galactose Kit (Id N° 10 176 303 035) | | IC quantification of sample extract | | | yes | |
| | 6 | MP.0002.R1.2018 | Extraction with water | | External std Lactose Sigma | no | yes | |
| | 7 | r-biopharm 10 176 303 035 | | | Lactose-Test control solution | no | yes | |
| | 8 | Carbohydrates by ion chromatography | | | | | | |
| | 9 | enzymatic | according to manual | | | yes | yes | |
| | 10 | 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. | | Internal reference sample, spiked | no | no | no |
| | 11 | in house method | | | | yes | yes | |
| | 12a | ISO 22662 | | HPLC-ELSD | DLA 18/2018 Sample A | no | yes | |
| | 12b | Internal Method (Determination of Lactose by LC-MS) | | LC-MS | DLA 18/2018 Sample A | no | no | |
| | 13 | internal method A/75 | | HPLC/PAD | Sigma Aldrich | | yes | |
| | 14 | Enzymatic | homogenisation, aqueouse extraction, Carrez-precipitation, filtration | | Standards from Enzyme-Kit r-biopharm | no | yes | |
| | 15a | Internal method HPAEC-PAD | extraction with water and precipitation by Carrez-solution | HPAEC-PAD | | | yes | |
| | 15 b | r-biopharm Test-Combination 10 176 303 035:2011-06 | | | | | yes | |
| | 16 | Megazyme K-LACGAR | | | | | yes | |
| | 17 | Enzymatic Test Megazyme LOLAC | Water Extraction Carrez Clean up | Readings taken at 340nm | In House Verified Material | | No | |
| | 18 | HPLC-MS | | recovery calculated by C13-Lactose internal standard | Anhydrous lactose (Sigma) | yes | yes | |
| | 19 | Foodlab Jr. | | Photometer | ext. Standards | | | |
| | 20 | ASU L 01.00-17, modified | | | | no | yes | |
| | 21 | HPAEC-PAD | | | | | no | |

## Lactose Sample B

| Analyte | 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 |
|---|---|---|---|---|---|---|---|---|
| | | | | | | | | |
| Lactose | 1 | Fructose, HPLC SOP Code: 084L02xx (inhouse) | extraction aqueous, Carrez precipitation | HPAEC-PAD | yes | | yes | given as g/100g |
| | 2 | ENZYMATIC | | | R-BIOPHARM | | NO | |
| | 3 | LC/MS/MS - internal method PNTA0189 | | | external calib. curve and internal RM | no | yes | |
| | 4 | FOOD-PA 591, in Anl. an ASU L07.00-23, enzymatisch | | | | | yes | |
| | 5 | Roche Lactose/Galactose Kit (Id N° 10 176 303 035) | | | | | yes | |
| | 6 | MP.0002.R1.2018 | Extraction with water | IC quantification of sample extract | External std Lactose Sigma | no | yes | |
| | 7 | r-biopharm 10 176 303 035 | | | Lactose-Test control solution | no | yes | |
| | 8 | Carbohydrates by ion chromatography | | | | | | |
| | 9 | enzymatic | according to manual | | | yes | yes | |
| | 10 | 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. | | Internal reference sample, spiked | no | no | no |
| | 11 | in house method | | | | yes | yes | |
| | 12a | ISO 22662 | | HPLC-ELSD | DLA 18/2018 Sample A | no | yes | despite below LOQ quantified: 100mg/100g |
| | 12b | Internal Method (Determination of Lactose by LC-MS) | | LC-MS | DLA 18/2018 Sample A | no | no | |
| | 13 | internal method A/75 | | HPLC/PAD | Sigma Aldrich | Yes | yes | |
| | 14a | Enzymatic | homogenisation, aqueouse extraction, Carrez-precipitation, filtration | | Standards from Enzyme-Kit r-biopharm | no | yes | |
| | 14b | | | | | | | HPAEC-PAD: 115 mg/100g |
| | 15a | Internal method HPAEC-PAD | extraction with water and precipitation by Carrez-solution | HPAEC-PAD | | | yes | |
| | 15 b | r-biopharm Test-Combination 10 176 303 035:2011-06 | | | | | yes | |
| | 16 | Megazyme K-LACGAR | | | | | yes | |
| | 17 | Enzymatic Test Megazyme LOLAC | Water Extraction Carrez Clean up | Readings taken at 340nm | In House Verified Material | | No | |
| | 18 | HPLC-MS | | recovery calculated by C13-Lactose internal standard | Anhydrous lactose (Sigma) | yes | yes | |
| | 19 | Foodlab Jr. | | Photometer | ext. Standards | | | |
| | 20 | ASU L 01.00-17, modified | | | | no | yes | |
| | 21 | HPAEC-PAD | | | | | no | |

## Lactose Spiking Level Sample

| Analyte | 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 |
|---|---|---|---|---|---|---|---|---|
| | | | | | | | | |
| Lactose | 1 | Fructose, HPLC SOP Code: 084L02xx (inhouse) | extraction aqueous, Carrez precipitation | HPAEC-PAD | yes | | yes | given as g/100g |
| | 2 | ENZYMATIC | | | R-BIOPHARM | | NO | |
| | 3 | LC/MS/MS - internal method PNTA0189 | | | external calib. curve and internal RM | no | yes | |
| | 4 | FOOD-PA 591, in Anl. an ASU L07.00-23, enzymatisch | | | | | yes | |
| | 5 | | | | | | | |
| | 6 | MP.0002.R1.2018 | Extraction with water | IC quantification of sample extract | External std Lactose Sigma | no | yes | |
| | 7 | r-biopharm 10 176 303 035 | | | Lactose-Test control solution | no | yes | |
| | 8 | Carbohydrates by ion chromatography | | | | | | 43601 |
| | 9 | enzymatic | according to manual | | | yes | yes | |
| | 10 | 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. | | Internal reference sample, spiked | no | no | no |
| | 11 | in house method | | | | yes | yes | |
| | 12a | ISO 22662 | | HPLC-ELSD | DLA 18/2018 Sample A | no | yes | despite below LOQ quantified: 90mg/100g |
| | 12b | Internal Method (Determination of Lactose by LC-MS) | | LC-MS | DLA 18/2018 Sample A | no | no | |
| | 13 | internal method A/75 | | HPLC/PAD | Sigma Aldrich | Yes | yes | |
| | 14a | Enzymatic | homogenisation, aqueouse extraction, Carrez-precipitation, filtration | | Standards from Enzyme-Kit r-biopharm | no | yes | |
| | 14b | | | | | | | HPAEC-PAD: 100 mg/100g |
| | 15a | Internal method HPAEC-PAD | extraction with water and precipitation by Carrez-solution | HPAEC-PAD | | | yes | |
| | 15 b | r-biopharm Test-Combination 10 176 303 035:2011-06 | | | | | yes | |
| | 16 | Megazyme K-LACGAR | | | | | yes | |
| | 17 | Enzymatic Test Megazyme LOLAC | Water Extraction Carrez Clean up | Readings taken at 340nm | In House Verified Material | | No | |
| | 18 | HPLC-MS | | recovery calculated by C13-Lactose internal standard | Anhydrous lactose (Sigma) | yes | yes | |
| | 19 | Foodlab Jr. | | Photometer | ext. Standards | | | |
| | 20 | ASU L 01.00-17, modified | | | | no | yes | |
| | 21 | HPAEC-PAD | | | | | no | |

## Galactose Sample A

| Analyte | Partici-pant | Method description as in test report / norm / litera-ture | Sample preparati-on | Measuring me-thod | Calibration / Reference ma-terial | Recovery rate with same matrix | Method accredited ISO/IEC 17025 | Further Remarks |
|---|---|---|---|---|---|---|---|---|
| | | | | | | | | |
| Galactose | 1 | | | | | | | |
| | 2 | | | | | | | |
| | 3 | HPAE-PAD - internal method PNTA0179 | | | external calib. curve and internal RM | no | no | |
| | 4 | FOOD-PA 591, in Anl. an ASU L07.00-23, enzymatisch | | | | | yes | |
| | 5 | | | | | | | |
| | 6 | | | | | | | |
| | 7 | r-biopharm 10 176 303 035 | | | Lactose-Test control solution | no | yes | |
| | 8 | Carbohydrates by ion chro-matography | | | | | | |
| | 9 | enzymatic | according to manual | | | | | |
| | 10 | | | | | | | |
| | 11 | | | | | | | |
| | 12a | | | | | | | |
| | 12b | | | | | | | |
| | 13 | | | | | | | |
| | 14 | | | | | | | |
| | 15a | Internal method HPAEC-PAD | extraction with water and precipitation by Carrez-solution | HPAEC-PAD | | | yes | |
| | 15 b | r-biopharm Test-Combination 10 176 303 035:2011-06 | | | | | yes | |
| | 16 | | | | | | | |
| | 17 | | | | | | | |
| | 18 | | | | | | | |
| | 19 | | | | | | | |
| | 20 | Enzymatic by testkit | | | | no | no | Galactose not validated |
| | 21 | HPAEC-PAD | | | | | no | |

## Galactose Sample B

| Analyte | 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 |
|---|---|---|---|---|---|---|---|---|
| | | | | | | | | |
| Galactose | 1 | | | | | | | |
| | 2 | | | | | | | |
| | 3 | HPAE-PAD - internal method PNTA0179 | | | external calib. curve and internal RM | no | no | |
| | 4 | FOOD-PA 591, in Anl. an ASU L07.00-23, enzymatisch | | | | | yes | |
| | 5 | | | | | | | |
| | 6 | | | | | | | |
| | 7 | r-biopharm 10 176 303 035 | | | Lactose-Test control solution | no | yes | |
| | 8 | Carbohydrates by ion chromatography | | | | | | |
| | 9 | enzymatic | according to manual | | | | | |
| | 10 | | | | | | | |
| | 11 | | | | | | | |
| | 12a | | | | | | | |
| | 12b | | | | | | | |
| | 13 | | | | | | | |
| | 14 | | | | | | | |
| | 15a | Internal method HPAEC-PAD | extraction with water and precipitation by Carrez-solution | HPAEC-PAD | | | yes | |
| | 15 b | r-biopharm Test-Combination 10 176 303 035:2011-06 | | | | | yes | |
| | 16 | | | | | | | |
| | 17 | | | | | | | |
| | 18 | | | | | | | |
| | 19 | | | | | | | |
| | 20 | Enzymatic by testkit | | | | no | no | Galactose not validated |
| | 21 | HPAEC-PAD | | | | | no | |

## Galactose Spiking Level Sample

| Analyte | Partici-pant | Method description as in test report / norm / litera-ture | Sample preparati-on | Measuring me-thod | Calibration / Reference ma-terial | Recovery rate with same matrix | Method accredited ISO/IEC 17025 | Further Remarks |
|---|---|---|---|---|---|---|---|---|
| | | | | | | | | |
| Galactose | 1 | | | | | | | |
| | 2 | | | | | | | |
| | 3 | HPAE-PAD - internal method PNTA0179 | | | external calib. curve and internal RM | no | no | |
| | 4 | FOOD-PA 591, in Anl. an ASU L07.00-23, enzymatisch | | | | | yes | |
| | 5 | | | | | | | |
| | 6 | | | | | | | |
| | 7 | r-biopharm 10 176 303 035 | | | Lactose-Test control solution | no | yes | |
| | 8 | Carbohydrates by ion chro-matography | | | | | | |
| | 9 | enzymatic | according to manual | | | | | |
| | 10 | | | | | | | |
| | 11 | | | | | | | |
| | 12a | | | | | | | |
| | 12b | | | | | | | |
| | 13 | | | | | | | |
| | 14 | | | | | | | |
| | 15a | Internal method HPAEC-PAD | extraction with water and precipitation by Carrez-solution | HPAEC-PAD | | | yes | |
| | 15 b | r-biopharm Test-Combination 10 176 303 035:2011-06 | | | | | yes | |
| | 16 | | | | | | | |
| | 17 | | | | | | | |
| | 18 | | | | | | | |
| | 19 | | | | | | | |
| | 20 | Enzymatic by testkit | | | | no | no | Galactose not validated |
| | 21 | HPAEC-PAD | | | | | no | |

## 5.2 Homogeneity

### 5.2.1 Mixture homogeneity before bottling

**Microtracer Homogeneity Test**
**DLA 18-2019 Sample B**

| | | |
|---|---|---|
| Weight whole sample | 2,63 | kg |
| Microtracer | FSS-rot lake | |
| Particle size | 75 – 300 | µm |
| Weight per particle | 2,0 | µg |
| Addition of tracer | 28,6 | mg/kg |

**Result of analysis**

| Sample | Weight [g] | Particle number | Particles [mg/kg] |
|---|---|---|---|
| 1 | 5,06 | 82 | 32,4 |
| 2 | 5,05 | 80 | 31,7 |
| 3 | 4,99 | 82 | 32,9 |
| 4 | 5,07 | 83 | 32,7 |
| 5 | 4,98 | 85 | 34,1 |
| 6 | 5,00 | 81 | 32,4 |
| 7 | 5,00 | 85 | 34,0 |
| 8 | 5,01 | 84 | 33,5 |

| **Poisson distribution** | | |
|---|---|---|
| Number of samples | 8 | |
| Degree of freedom | 7 | |
| Mean | 82,8 | Particle |
| Standard deviation | 2,14 | Particle |
| $\chi^2$ (CHI-Quadrat) | 0,39 | |
| **Probability** | **100** | % |
| Recovery rate | 115 | % |

| **Normal distribution** | | |
|---|---|---|
| Number of samples | 8 | |
| Mean | 33,0 | mg/kg |
| Standard deviation | 0,85 | mg/kg |
| rel. Standard deviaton | 2,59 | % |
| Horwitz standard deviation | 9,45 | % |
| **HorRat-value** | **0,27** | |
| Recovery rate | 115 | % |

**Microtracer Homogeneity Test**
**DLA 18-2019 Spiking Level Sample**

| | | |
|---|---|---|
| Weight whole sample | 1,82 | kg |
| Microtracer | FSS-rot lake | |
| Particle size | 75 – 300 | µm |
| Weight per particle | 2,0 | µg |
| Addition of tracer | 31,9 | mg/kg |

**Result of analysis**

| Sample | Weight [g] | Particle number | Particles [mg/kg] |
|---|---|---|---|
| 1 | 4,97 | 87 | 35,0 |
| 2 | 5,09 | 104 | 40,9 |
| 3 | 5,04 | 102 | 40,5 |
| 4 | 5,03 | 103 | 41,0 |
| 5 | 5,02 | 96 | 38,2 |
| 6 | 5,04 | 97 | 38,5 |
| 7 | 5,00 | 90 | 36,0 |
| 8 | 5,09 | 108 | 42,4 |

| **Poisson distribution** | | |
|---|---|---|
| Number of samples | 8 | |
| Degree of freedom | 7 | |
| Mean | 98,3 | Particle |
| Standard deviation | 6,52 | Particle |
| $\chi^2$ (CHI-Quadrat) | 3,03 | |
| **Probability** | **88** | % |
| Recovery rate | 122 | % |

| **Normal distribution** | | |
|---|---|---|
| Number of samples | 8 | |
| Mean | 39,1 | mg/kg |
| Standard deviation | 2,59 | mg/kg |
| rel. Standard deviaton | 6,63 | % |
| Horwitz standard deviation | 9,22 | % |
| **HorRat-value** | **0,72** | |
| Recovery rate | 122 | % |

## 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 - 2019*** |
| *PT name* | ***Lactose + Fructose in "lactose-free" food with "Spiking Level Sample"*** |
| *Sample matrix** | ***Samples A + B:*** *Cake baking mix / ingredients: wheat flour, sugar, starch, low-fat cocoa powder, acidifier: diphospates, raising agent: sodium carbonates, emulsifiers: E475 and E471, flavor, salt and lactose and fructose (one of both samples)*<br>***Spiking Level Sample:*** *potato powder, lactose and fructose* |
| *Number of samples and sample amount* | *2 different Samples A + B: 25 g each*<br>*+ 1 Spiking Level Sample: 25 g* |
| *Storage* | *Samples A + B: room temperature (long term 2 - 10°C)*<br>*Spiking Level Sample: room temperature* |
| *Intentional use* | *Laboratory use only (quality control samples)* |
| *Parameter* | *qualitative + quantitative: Lactose (optional: Galactose) + Fructose*<br>*Samples A + B: Lactose < 500 mg/100g*<br>*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.*<br>*In general we recommend to homogenize a representative sample amount before analysis according to good laboratory practice, especially in case of low sample weights.* |
| *Result sheet* | *One result each should be determined for Samples A and B and the Spiking Level Sample for submission.*<br>*The results should be filled in the result submission file. In case of several determinations the mean.* |
| *Units* | *mg/100g* |
| *Number of significant digits* | *at least 2* |
| *Further information* | *For information please specify:*<br>– *Date of analysis*<br>– *DLA-sample-numbers (for sample A and B)*<br>– *Limit of detection*<br>– *Assignment incl. Recovery*<br>– *Recovery with the same matrix*<br>– *Method is accredited* |
| *Result submission* | *The result submission file should be sent by e-mail to:*<br>***pt@dla-lvu.de*** |
| *Deadline* | ***the latest 17th May 2019*** |
| *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* | *Matthias Besler-Scharf PhD / 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 |
| | | CZECH REPUBLIC |
| | | Germany |
| | | ITALY |
| | | NETHERLANDS |
| | | GREAT BRITAIN |
| | | ITALY |
| | | ITALY |
| | | Germany |
| | | Germany |
| | | Germany |
| | | Germany |
| | | GREAT BRITAIN |
| | | NETHERLANDS |
| | | AUSTRIA |
| | | AUSTRIA |
| | | SPAIN |
| | | CZECH REPUBLIC |
| | | Germany |

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

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

# 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)
7. The International Harmonised Protocol for the Proficiency Testing of Ananlytical Laboratories ; J.AOAC Int., 76(4), 926 – 940 (1993)
8. A Horwitz-like funktion describes precision in proficiency test; M. Thompson, P.J. Lowthian; Analyst, 120, 271-272 (1995)
9. Protocol for the design, conduct and interpretation of method performance studies; W. Horwitz; Pure & Applied Chemistry, 67, 331-343 (1995)
10. Recent trends in inter-laboratory precision at ppb and sub-ppb concentrations in relation to fitness for purpose criteria in proficiency testing; M. Thompson; Ana-lyst, 125, 385-386 (2000)
11. The International Harmonised Protocol for the Proficiency Testing of Analytical Chemistry Laboratories; Pure Appl Chem, 78, 145 – 196 (2006)
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)