Evaluation Report
proficiency test

DLA ptAU04 (2020)

Sugar Alcohols:

Sorbitol E420, Mannitol E421, Isomalt E953, Xylitol E967 and Erythritol E968

in Pudding Powder

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

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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.

Contents

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

The test material is a common in commerce pudding powder 'chocolate' from an European supplier.
The spiking materials sorbitol, mannitol, isomalt, xylitol and erythritol were sieved (mesh <600 µm), added to the basic mixture and the mixture was homogenized.

Afterwards the samples were portioned to approximately 10 g into metal-lised PET film bags and chronologically numbered.

The composition of the PT samples is given in table 1.

Table 1: Composition of DLA-Samples

Ingredients	**Content**
Chocolate Pudding Powder, Ingredients: Starch, light cocoa powder (18%), flavor Nutrients per 100 g: Protein 4,2 g, Carbohydrates 74 g, Fat 2,2 g	89,8 g/100 g
Sorbitol	1,80 g/100 g
Mannitol	2,40 g/100 g
Isomalt	1,98 g/100 g
Xylitol	2,16 g/100 g
Erythritol	1,86 g/100 g

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 sample showed a probability of 91%. 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) [16, 17]. This gave a HorRat value of 0,85 for the present PT sample. The results of microtracer analysis are given in the documentation.

The calculation of the **repeatability standard deviations S_r of the participants** was also used as an indicator of homogeneity. For all parameters the repeatability standard deviation was < 7,5% (see Table 2). Thus they were similar to corresponding repeatability standard deviations of precision data of the standardized methods (e.g. ASU- methods s. 3.6.2) [18-19]. The repeatability standard deviations of the participants' results are given in the documentation in the statistic data (see 4.1 to 4.5).

Table 2: Repeatability standard deviation S_r of double determinations of the participants (coefficient of variation CV_r in %)

Parameter	**CV_r**
E420 - Sorbit	2,29 %
E421 - Mannit	2,86 %
E953 – Isomalt	7,49 %
E967 – Xylit	2,32 %
E968 - Erythrit	4,88 %

Furthermore, the homogeneity was graphically characterized for information by the **trend line function of participants' results for chronological bottled single samples** (s. 5.2.2).

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. 0,48 (22,1°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

Two portions of test material were sent to every participating laboratory in the 26th week of 2020. The testing method was optional. The tests should be finished at 4th September 2020 the latest.

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

The two portions contain identical samples with the parameters Sugar Alcohols (Sorbitol E420, Mannitol E421, Isomalt E953, Xylitol E967, Erythritol E968) in the matrix of Pudding Powder. The analysis method is optional.

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).
The finally calculated concentrations of the parameter as average of duplicate determinations of both numbered samples were used for the statistical evaluation. For the calculation of the repeatability– and reproducibility standard deviation the single values of the double determination were used.
Queried and documented were single results, recovery and the used testing methods. In case participants submitted several results for the same parameter obtained by different methods these results were evaluated with the same evaluation number with a letter as a suffix and indication of the related method.

All 15 participants submitted their results in time.

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 statistical evaluation is carried out for all the parameters for a minimum of 7 values are present, in justified cases, an evaluation may also be carried out from 5 results onwards.
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*) 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 PT´s may differ from reproducibility standard deviations of ring trials, because the participating laboratories of a PT generally use different internal conditions and methods for determining the measured values.
In the present evaluation, the specification of the reproducibility standard deviation, therefore, does not refer to a specific method, but characterizes approximately the comparability of results between the laboratories, assumed the effect of homogeneity and stability of the sample are negligible.
In case single results from participants are available the calculation of the reproducibility standard deviation S_R is performed by: [3, 4].
The relative reproducibility standard deviation CV_R in percent of the mean is given as variation coefficient in the statistical data of participant for each parameter. The significance of CV_R is further explained in section 3.9.

3.5 Exclusion of results and outliers

Before statistical evaluation obvious blunders, such as those with incorrect units, decimal point errors, 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.

For valuation of sorbitol, mannitol, xylitol and erythritol in the present PT the target standard deviation according to the general model of Horwitz was applied (see 3.6.1).

The target standard deviation of the evaluation by a precision experiment (s. 3.6.2) was considered for isomalt (ASU §64 method L 00.00-59 / EN 15086) [18].

Additionally for isomalt and erythritol the standard uncertainty was considered by evaluation using z'-scores (see 3.6.8).

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 deviation (RSD_R) given in Table 3 were determined in ring tests using the indicated methods.
The resulting target standard deviations σ_{pt}, which were identified there, were used to evaluate the results and to provide additional information for the statistical data.

Table 3: Relative repeatability standard deviations (RSD_r) and relative reproducibility standard deviations (RSD_R) according to selected evaluations of tests for precision and the resulting target standard deviation σ_{pt} [18-19]

Parameter	Matrix	Mean	RSD_r	RSD_R	σpt	Method / Literature
E420 - Sorbitol	Cookies	3,73	1,52%	3,91%	3,76%[1]	HPLC-RI [18]
E420 - Sorbitol	Cookies	4,66	1,65%	2,66%	2,66%	Enzymatic [19]
E421 - Mannitol	Cookies	4,34	1,24%	3,55%	3,44%[1]	HPLC-RI [18]
E953 - GPS[2]	Cookies	13,5	0,52%	3,41%	3,41%	HPLC-RI [18]
E953 - GPM[2]	Cookies	12,6	0,66%	4,47%	4,45%[1]	HPLC-RI [18]
E967 - Xylitol	Cookies	3,03	1,62%	3,76%	3,58%[1]	HPLC-RI [18]

[1] *values used for evaluation (s. chapter 4*
[2] *Chemical isomalt (E953) is a mixture of 6-O-α-D-Gucopyranosyl-D-sorbitol (1,6-GPS) and 1-O-α-D-Glucopyranosyl-D-mannitol (1,1-GPM).*

3.6.3 Value by perception

The target standard deviation for proficiency assessment can be set at a value that corresponds to the level of performance that the coordinator would wish laboratories to be able to achieve [3].

For the present evaluation the target standard deviation according to 3.6.1 or 3.6.2 was regarded suitable.
Table 4 shows selected statistic data of participants results of present PT compared to PT results of previous years.

Table 4: Characteristics of the present PT (on dark gray) in comparison to a previous PT in 2018 (SD = standard deviation, CV = coefficient of variation, MV = MV)

Parameter	Matrix (Powder)	robust Mean	rob. SD (S*)	rel. SD (CV_{S*}) [%]	Quotient S*/σpt	DLA-report
E420 - Sorbitol	Pudding	2,93 g/100g	0,436 g/100g	14,9	2,3[1]	DLA 39/2018
E420 - Sorbitol	Pudding	1,82 g/100g	0,122 g/100g	6,74	1,8	ptAU04 (2020)
E421 - Mannitol	Pudding	1,93 g/100g	0,322 g/100g	16,7	2,3[1]	DLA 39/2018
E421 - Mannitol	Pudding	2,41 g/100g	0,126 g/100g	5,21	1,5	ptAU04 (2020)
E953 - Isomalt	Pudding	2,87 g/100g	0,765 g/100g	26,7	2,2[1]	DLA 39/2018
E953 – Isomalt	Pudding	1,96 g/100g	0,256 g/100g	13,1	2,0[1]	ptAU04 (2020)
E967 - Xylitol	Pudding	2,10 g/100g	0,304 g/100g	14,5	2,3[1]	DLA 39/2018
E967 - Xylitol	Pudding	2,17 g/100g	0,116 g/100g	5,33	1,5	ptAU04 (2020)
E968 - Erythri-tol	Pudding	3,18 g/100g	0,532 g/100g	16,7	2,3[1]	DLA 39/2018
E968 - Erythri-tol	Pudding	1,93 g/100g	0,185 g/100g	9,60	1,8[1]	ptAU04 (2020)

[1] *Calculated with z'-Score*

3.7 z-Score

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

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

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

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

The valid z-Score for each parameter is indicated as z-Score (σ_{pt}). The value indicated as z-Score (Info) only obtains a informative character. The both z-Scores were calculated with the different target standard deviations in accordance with 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 error or an error in the calculation, in the trueness and precision and use of reference material. If necessary, the problems must be addressed through appropriate corrective action [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 ($U_{(x_{pt})}$) [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 cofficient of variation (CV_R)

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*/σ_{pt}

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

3.11 Standard uncertainty of the assigned value

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

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

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

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

4. Results

All following tables are anonymized. With the delivering of the evaluation report the participants are informed about their individual evaluation number.

In the first table the characteristics are listed:

Statistic Data
Number of results
Number of outliers
Mean
Median
Robust mean(X_{pt})
Robust standard deviation (S^*)
Number with m replicate measurements
Repeatability standard deviation (S_r)
Coefficient of Variation (CV_r) in %
Reproducibility standard deviation (S_R)
Coefficient of Variation (CV_R) in %
Target range:
Target standard deviation σ_{pt} or σ_{pt}'
Target standard deviation for information
lower limit of target range ($X_{pt} - 2\sigma_{pt}$) or ($X_{pt} - 2\sigma_{pt}$') *
upper limit of target range ($X_{pt} + 2\sigma_{pt}$) or ($X_{pt} + 2\sigma_{pt}$´) *
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 σ_{pt}	z-Score (Info)	Hinweis Remark

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

4.1 E420 - Sorbitol in g/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results°	14
Number of outliers	1
Mean	1,82
Median	1,84
Robust Mean (X_{pt})	**1,82**
Robust standard deviation (S*)	**0,122**
Number with 2 replicates	14
Repeatability SD (S_r)	0,0414
Repeatability (CV_r)	2,29%
Reproducibility SD (S_R)	0,120
Reproducibility (CV_R)	6,64%
Target range:	
Target standard deviation σ_{pt}	**0,0664**
Target standard deviation (for Information)	0,0683
lower limit of target range	**1,68**
upper limit of target range	**1,95**
Quotient S/σ_{pt}*	*1,8*
Standard uncertainty $U_{(Xpt)}$	*0,0409*
Results in the target range	*11*
Percent in the target range	*79%*

° *without outlier (result no. 5)*

Comments to the statistic data:

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 00.00-59) is given for information.
The distribution of results showed a normal variability. The quotient S/σ_{pt} was below 2,0. The repeatability and reproducibility standard deviation were in the range of of established values for the used determination methods (s. 3.6.2). The comparability of results is given.*

79% of results were in the target range.

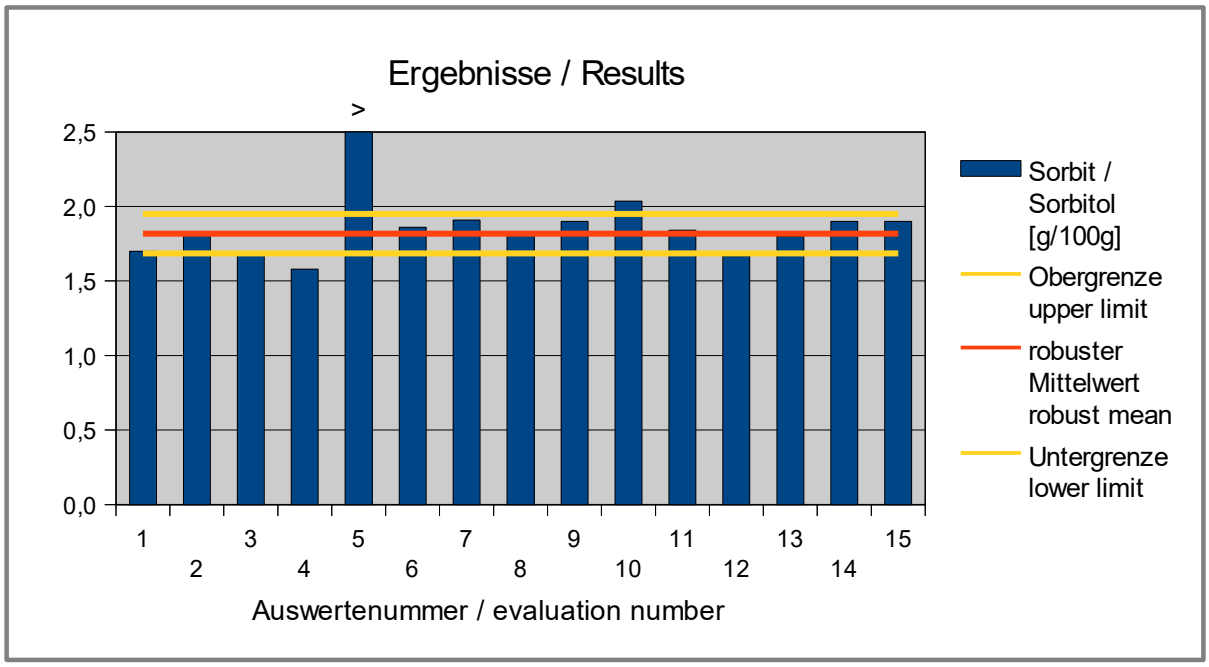


Abb. / Fig. 1: Ergebnisse Sorbit / Results sorbitol

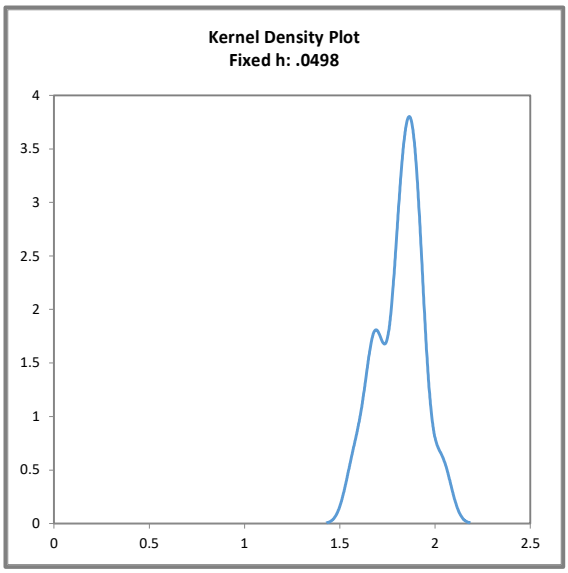


Abb. / Fig. 2:
Kerndichte-Schätzung der Ergebnisse
(mit h = 0,75 x σ_{pt} von X_{pt})
(ohne Ergebnis Nr. 5)

Kernel density plot of results
(with h = 0,75 x σ_{pt} of X_{pt})
(without result no. 5)

Comment:
The kernel density estimation shows nearly a symmetrical distribution of results with a side peak at approx. 1,7 g/100g and a shoulder at 2,0 g/100g, due to result outside or at the lower target range.

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Sorbit / Sorbitol [g/100g]	Abweichung [g/100g] Deviation [g/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	1,70	-0,117	-1,8	-1,7	
2	1,81	-0,007	-0,11	-0,11	
3	1,69	-0,129	-1,9	-1,9	
4	1,58	-0,237	-3,6	-3,5	
5	17,6				Ausreißer ausgeschlossen / Outlier excluded
6	1,86	0,043	0,64	0,62	
7	1,91	0,093	1,4	1,4	
8	1,83	0,013	0,19	0,19	
9	1,90	0,083	1,2	1,2	
10	2,04 *	0,218	3,3	3,2	
11	1,84	0,023	0,34	0,33	
12	1,67	-0,147	-2,2	-2,2	
13	1,80	-0,017	-0,26	-0,25	
14	1,90	0,083	1,2	1,2	
15	1,90	0,083	1,2	1,2	

* Mean calculated by DLA

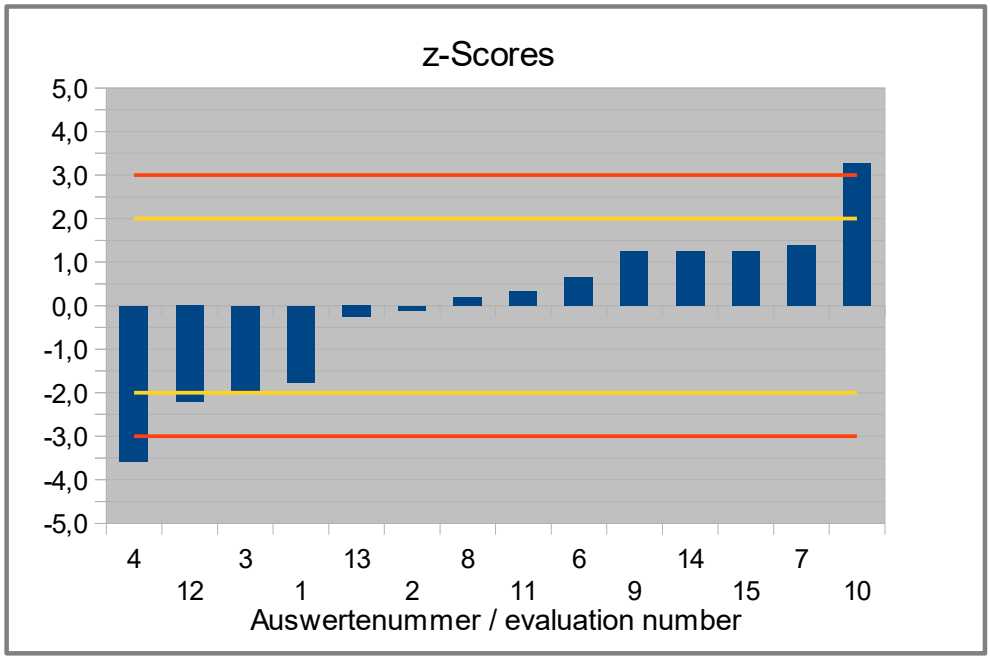


Abb. / Fig. 3: z-Scores Sorbit / sorbitol

4.2 E421 - Mannitol in g/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results°	13
Number of outliers	1
Mean	2,41
Median	2,40
Robust Mean (X_{pt})	**2,41**
Robust standard deviation (S*)	**0,126**
Number with 2 replicates	13
Repeatability SD (S_r)	0,0690
Repeatability (CV_r)	2,86%
Reproducibility SD (S_R)	0,122
Reproducibility (CV_R)	5,04%
Target range:	
Target standard deviation σ_{pt}	**0,0846**
Target standard deviation (for Information)	0,0831
lower limit of target range	**2,25**
upper limit of target range	**2,58**
Quotient S/σ_{pt}*	*1,5*
Standard uncertainty $U_{(Xpt)}$	*0,0436*
Results in the target range	*11*
Percent in the target range	*85%*

° *without outlier (result no. 5)*

Comments to the statistic data:

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 00.00-59) is given for information.
The distribution of results showed a normal variability. The quotient S/σ_{pt} was below 2,0. The repeatability and reproducibility standard deviation were in the range of of established values for the used determination methods (s. 3.6.2). The comparability of results is given.*

85% of results were in the target range.

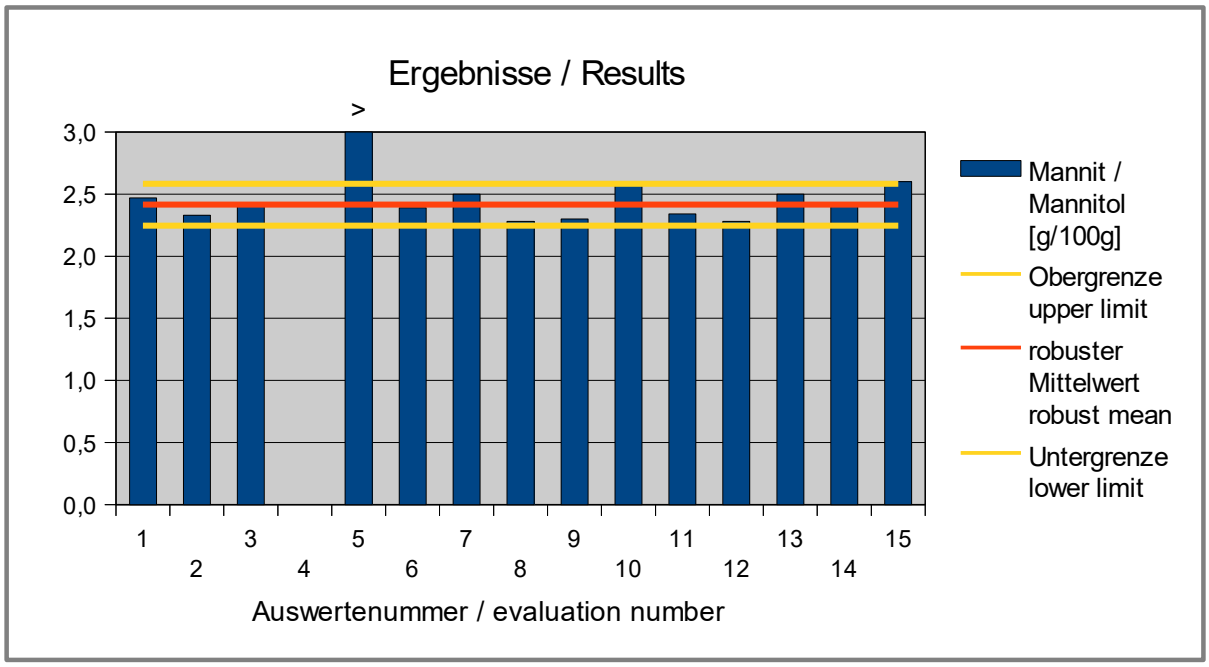


Abb. / Fig. 4: Ergebnisse Mannit / Results mannitol

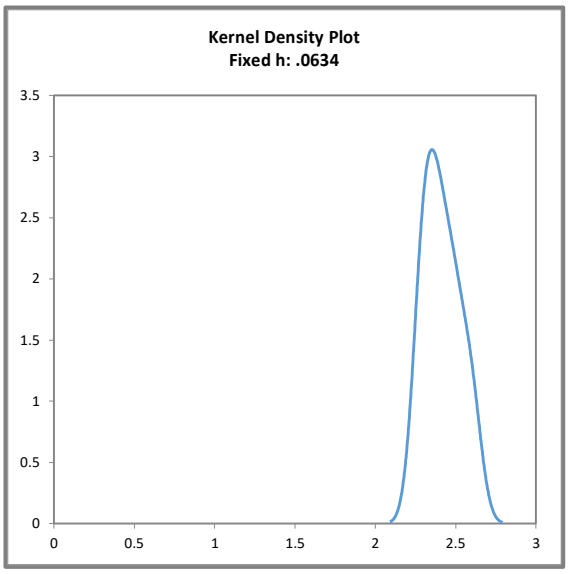


Abb. / Fig. 5:
Kerndichte-Schätzung der Ergebnisse
(mit h = 0,75 x σ_{pt} von Xpt)
(ohne Ergebnis Nr. 5)

Kernel density plot of results
(with h = 0,75 x σ_{pt} of Xpt)
(without result no. 5)

Comment:
The kernel density estimation shows nearly a symmetrical distribution of results.

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Mannit / Mannitol [g/100g]	Abweichung [g/100g] Deviation [g/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	2,47	0,056	0,66	0,67	
2	2,33	-0,084	-1,0	-1,0	
3	2,40	-0,012	-0,15	-0,15	
4					
5	25,3				Ausreißer ausgeschlossen / Outlier excluded
6	2,39	-0,024	-0,29	-0,29	
7	2,50	0,086	1,0	1,0	
8	2,28	-0,134	-1,6	-1,6	
9	2,30	-0,114	-1,4	-1,4	
10	2,60 *	0,181	2,1	2,2	
11	2,34	-0,074	-0,88	-0,90	
12	2,28	-0,134	-1,6	-1,6	
13	2,50	0,086	1,0	1,0	
14	2,40	-0,014	-0,17	-0,17	
15	2,60	0,186	2,2	2,2	

* Mean calculated by DLA

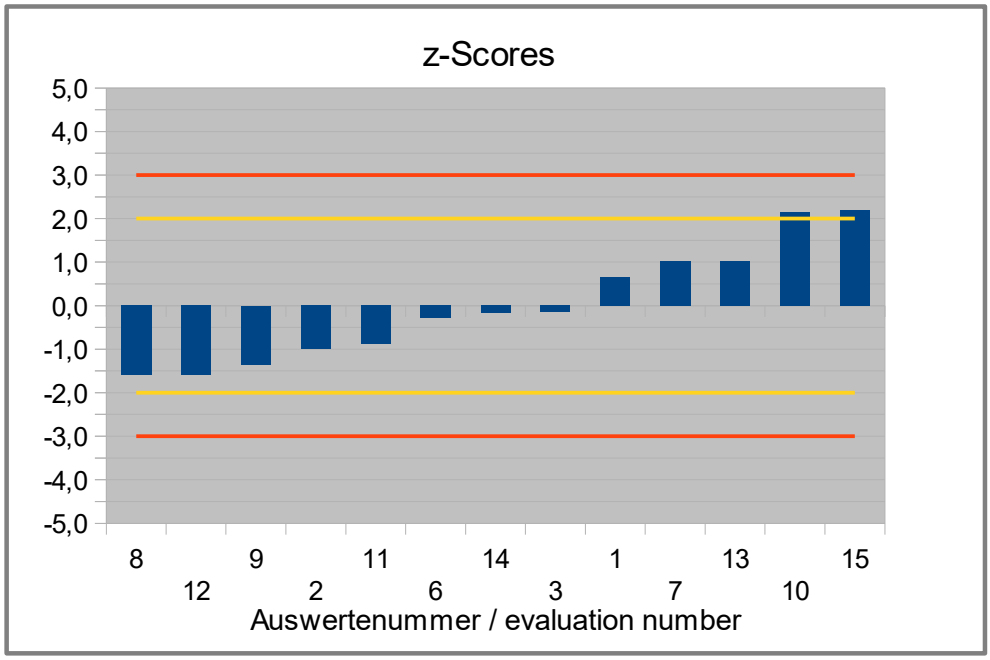


Abb. / Fig. 6: z-Scores Mannit / mannitol

4.3 E953 – Isomalt in g/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results°	11
Number of outliers	1
Mean	1,96
Median	1,90
Robust Mean (X_{pt})	**1,96**
Robust standard deviation (S*)	**0,256**
Number with 2 replicates	11
Repeatability SD (S_r)	0,147
Repeatability (CV_r)	7,49%
Reproducibility SD (S_R)	0,263
Reproducibility (CV_R)	13,4%
Target range:	
Target standard deviation σ_{pt}'	**0,130**
Target standard deviation (for Information)	0,0708
lower limit of target range	**1,70**
upper limit of target range	**2,22**
Quotient S/σ_{pt}'*	*2,0*
Standard uncertainty $U_{(Xpt)}$	*0,0963*
Results in the target range	*9*
Percent in the target range	*82%*

° *without outlier (result no. 4)*

Comments to the statistic data:

The target standard deviation was calculated using data from a precision experiment (ASU §64 L 00.00-59)(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 an increased variability. The quotient S/σpt was at 2,9. Therefore the valuation was done by z'-scores considering the standard uncertainty. The quotient S*/σpt' was then at 2,0. The repeatability and reproducibility standard deviation were above the range of established values for the used determination methods, while the relative standard deviation was lower than in previous proficiency tests (s. 3.6.2 and 3.6.3 Tab. 4). The comparability of results is given.*

82% of results were in the target range.

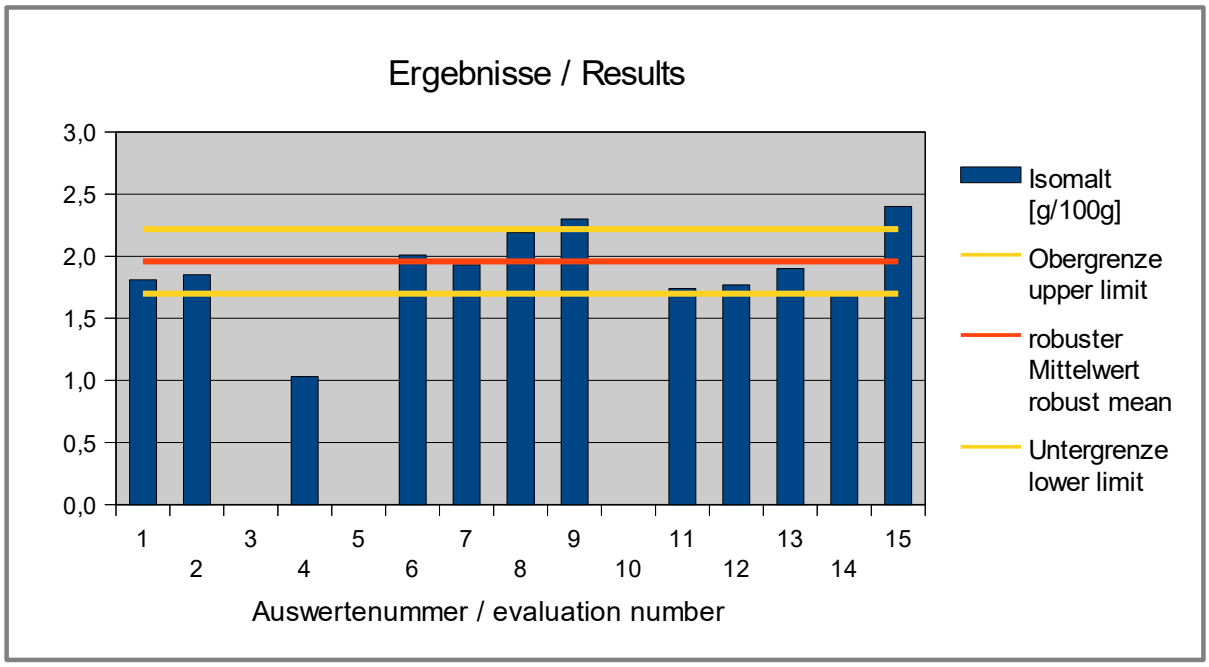


Abb. / Fig. 7: Ergebnisse Isomalt / Results isomalt

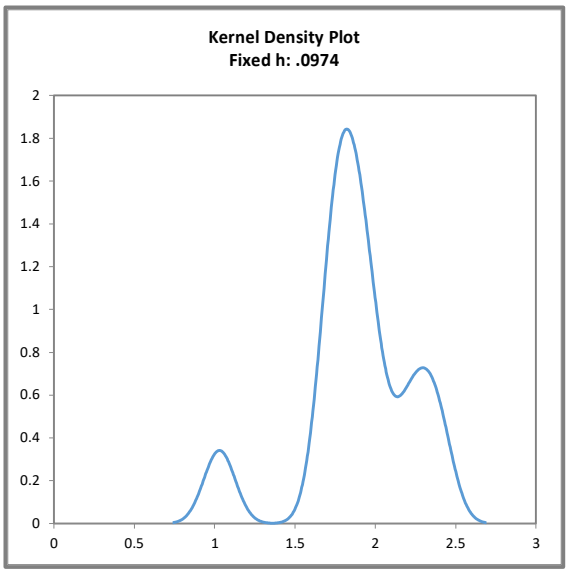


Abb. / Fig. 8:
Kerndichte-Schätzung der Ergebnisse (mit h = 0,75 x σ_{pt}' von Xpt)

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

Comment:
The kernel density estimation shows nearly a symmetrical distribution of results with two side peaks at approx. 1,0 g/100g and at approx. 2,3 g/100g, due to results outside the target range.

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Isomalt [g/100g]	Abweichung [g/100g] Deviation [g/100g]	z'-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	1,81	-0,148	-1,1	-2,1	
2	1,85	-0,108	-0,83	-1,5	
3					
4	1,03				Ausreißer ausgeschlossen / Outlier excluded
5					
6	2,01	0,052	0,40	0,73	
7	1,93	-0,028	-0,22	-0,40	
8	2,19	0,232	1,8	3,3	
9	2,30	0,342	2,6	4,8	
10					
11	1,74	-0,218	-1,7	-3,1	
12	1,77	-0,188	-1,5	-2,7	
13	1,90	-0,058	-0,45	-0,82	
14	1,70	-0,258	-2,0	-3,6	
15	2,40	0,442	3,4	6,2	

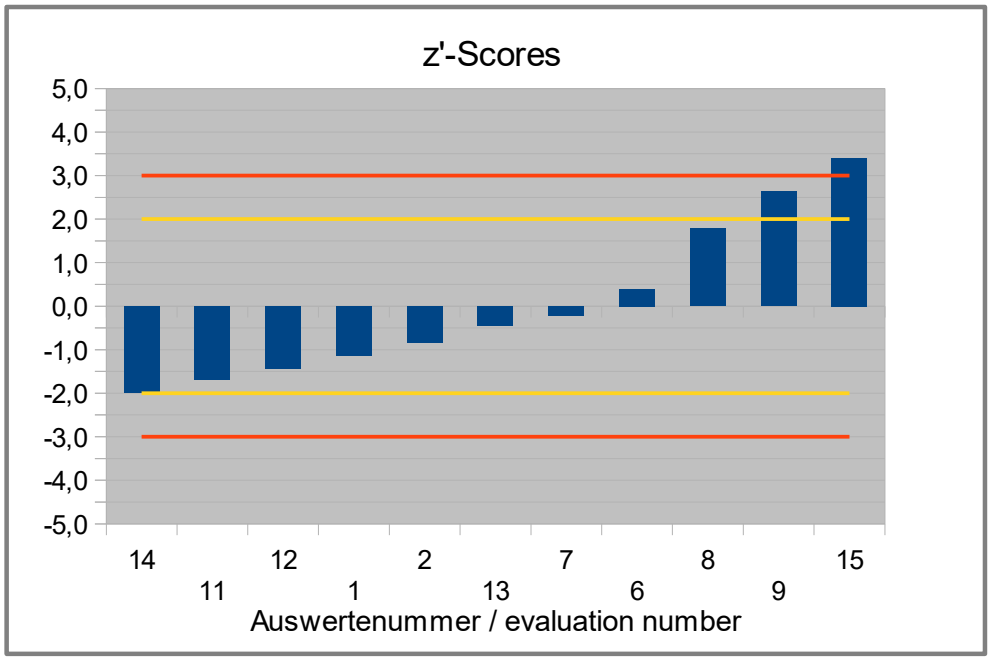


Abb. / Fig. 9: z'-Scores Isomalt

4.4 E967 - Xylitol in g/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results°	14
Number of outliers	1
Mean	2,18
Median	2,19
Robust Mean (X_{pt})	**2,17**
Robust standard deviation (S*)	**0,116**
Number with 2 replicates	14
Repeatability SD (S_r)	0,0503
Repeatability (CV_r)	2,32%
Reproducibility SD (S_R)	0,116
Reproducibility (CV_R)	5,35%
Target range:	
Target standard deviation σ_{pt}	**0,0774**
Target standard deviation (for Information)	0,0779
lower limit of target range	**2,02**
upper limit of target range	**2,33**
Quotient S/σ_{pt}*	*1,5*
Standard uncertainty $U_{(Xpt)}$	*0,0387*
Results in the target range	*12*
Percent in the target range	*86%*

° *without outlier (result no. 5)*

Comments to the statistic data:

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 00.00-59) is given for information.
The distribution of results showed a normal variability. The quotient S/σ_{pt} was below 2,0. The repeatability and reproducibility standard deviation were in the range of of established values for the used determination methods (s. 3.6.2). The comparability of results is given.*

86% of results were in the target range.

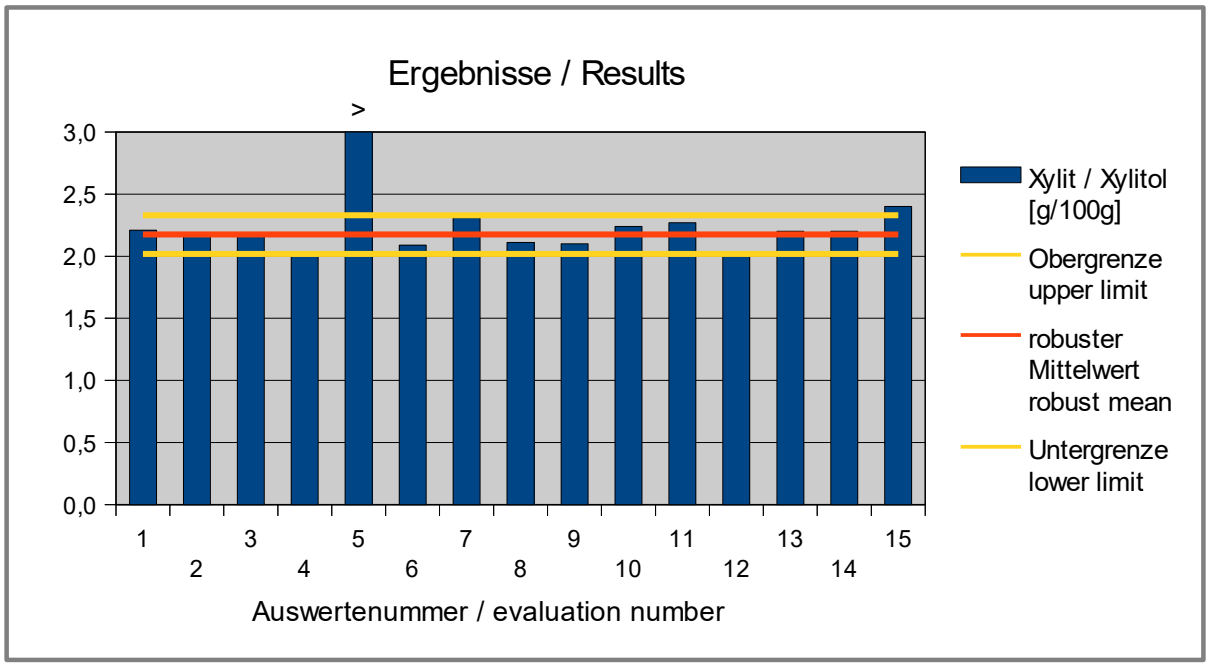


Abb. / Fig. 10: Ergebnisse Xylit / Results xylitol

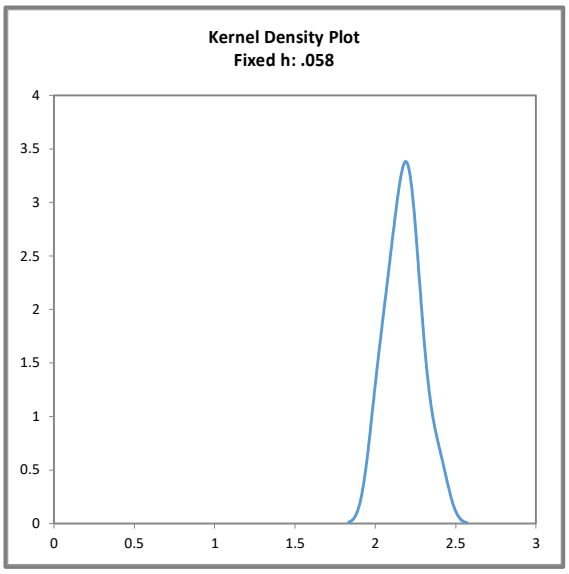


Abb. / Fig. 11:
Kerndichte-Schätzung der Ergebnisse
(mit h = 0,75 x σ_{pt} von X_{pt})
(ohne Ergebnis Nr. 5)

Kernel density plot of results
(with h = 0,75 x σ_{pt} of X_{pt})
(without result no. 5)

Comment:
The kernel density estimation shows nearly a symmetrical distribution of results.

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Xylit / Xylitol [g/100g]	Abweichung [g/100g] Deviation [g/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	2,21	0,036	0,47	0,46	
2	2,18	0,006	0,08	0,08	
3	2,16	-0,016	-0,21	-0,21	
4	2,02	-0,154	-2,0	-2,0	
5	24,6				Ausreißer ausgeschlossen / Outlier excluded
6	2,09	-0,084	-1,1	-1,1	
7	2,31	0,136	1,8	1,7	
8	2,11	-0,064	-0,83	-0,82	
9	2,10	-0,074	-0,96	-0,95	
10	2,24 *	0,066	0,85	0,85	
11	2,27	0,096	1,2	1,2	
12	2,00	-0,174	-2,2	-2,2	
13	2,20	0,026	0,34	0,33	
14	2,20	0,026	0,34	0,33	
15	2,40	0,226	2,9	2,9	

* Mean calculated by DLA

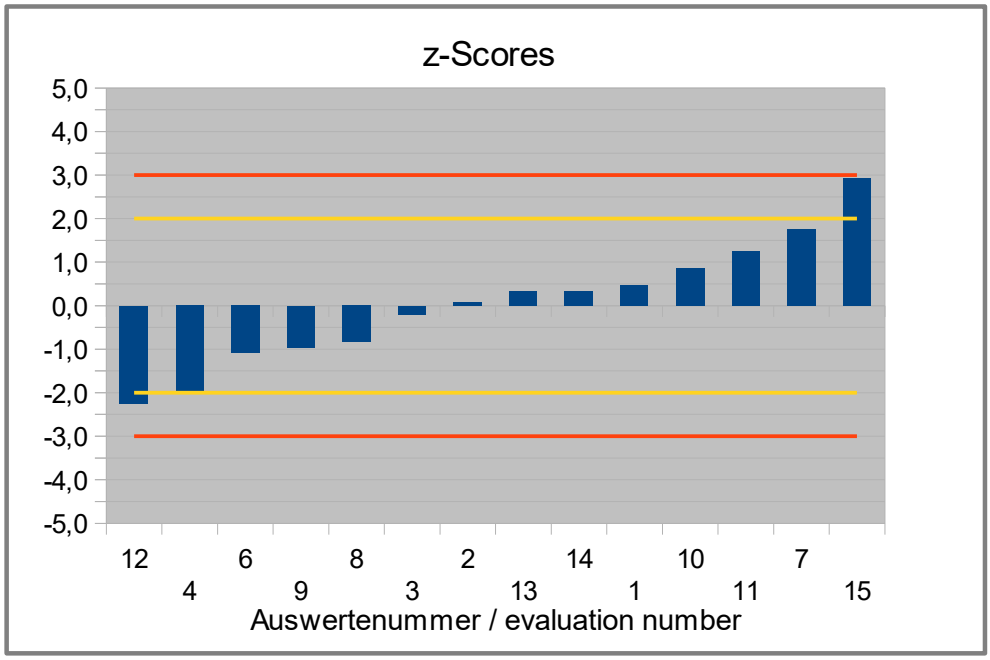


Abb. / Fig. 12: z-Scores Xylit / xylitol

4.5 E968 - Erythritol in g/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results°	10
Number of outliers	1
Mean	1,91
Median	1,92
Robust Mean (X_{pt})	**1,93**
Robust standard deviation (S*)	**0,185**
Number with 2 replicates	10
Repeatability SD (S_r)	0,0928
Repeatability (CV_r)	4,88%
Reproducibility SD (S_R)	0,214
Reproducibility (CV_R)	11,2%
Target range:	
Target standard deviation σ_{pt}'	**0,101**
lower limit of target range	**1,73**
upper limit of target range	**2,13**
Quotient S/σ_{pt}'*	*1,8*
Standard uncertainty $U_{(Xpt)}$	*0,0733*
Results in the target range	*8*
Percent in the target range	*80%*

° *without outlier (result no. 5)*

Comments to the statistic data:

The target standard deviation was calculated according to the model of Horwitz (s. 3.6.1).
The distribution of results showed an increased variability. The quotient S/σpt was at 2,7. Therefore the valuation was done by z'-scores considering the standard uncertainty. The quotient S*/σpt' was then below 2,0. The robust standard deviation is lower than in the previous PT (see 3.6.3). The comparability of results is given.*

80% of results were in the target range.

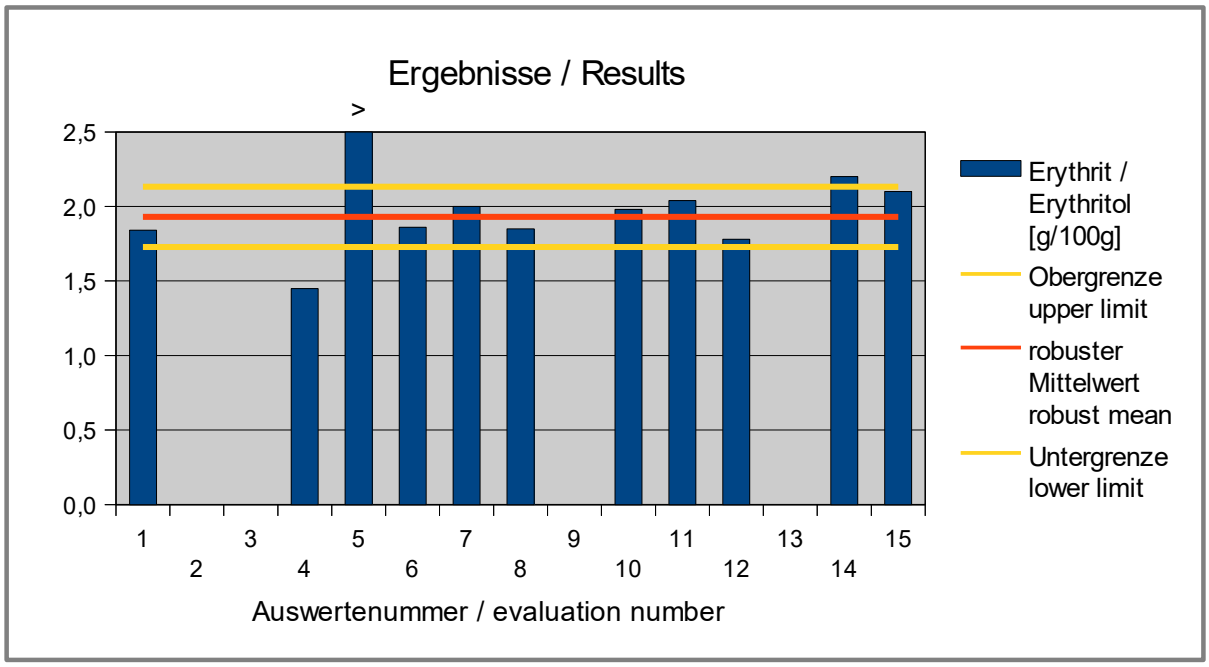


Abb. / Fig. 13: Ergebnisse Erythrit / Results erythritol

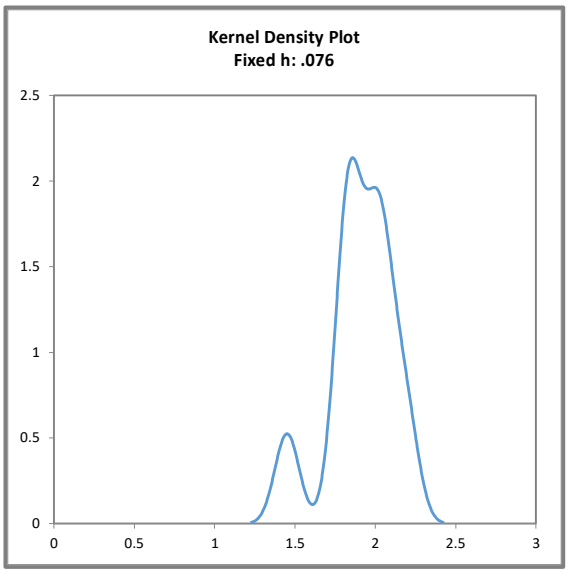


Abb. / Fig. 14:
Kerndichte-Schätzung der Ergebnisse
(mit h = 0,75 x σ_{pt}' von X_{pt})
(ohne Ergebnis Nr. 5)

Kernel density plot of results
(with h = 0,75 x σ_{pt}' of X_{pt})
(without result no. 5)

Comment:
The kernel density estimation shows nearly a symmetrical distribution of results with a slightly two-peaked maximum and a side peak at approx. 1,5 g/100g, due to a result outside the target range.

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Erythrit / Erythritol [g/100g]	Abweichung [g/100g] Deviation [g/100g]	z'-Score (σ_{pt})	Hinweis Remark
1	1,84	-0,090	-0,89	
2				
3				
4	1,45	-0,480	-4,7	
5	21,4			Ausreißer ausgeschlossen / Outlier excluded
6	1,86	-0,070	-0,69	
7	2,00	0,070	0,69	
8	1,85	-0,080	-0,79	
9				
10	1,98 *	0,050	0,49	
11	2,04	0,110	1,1	
12	1,78	-0,150	-1,5	
13				
14	2,20	0,270	2,7	
15	2,10	0,170	1,7	

* Mean calculated by DLA

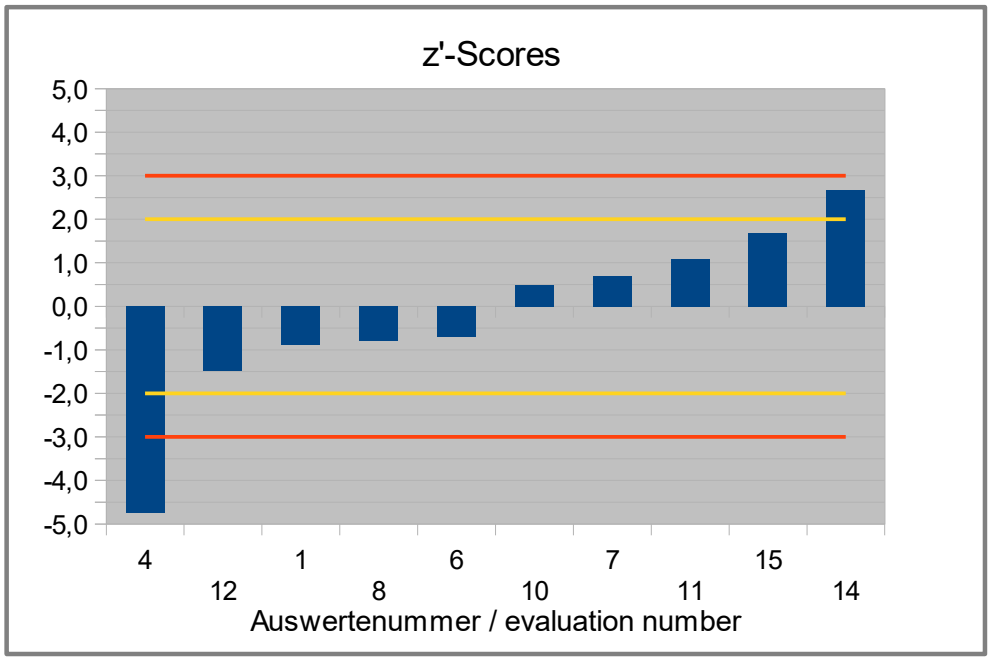


Abb. / Fig. 15: z'-Scores Erythrit / erythritol

4.6 Participant z-Scores: overview table

Evaluation number	E420 Sorbitol	E421 Mannitol	E953 Isomalt	E967 Xylitol	E968 Erythritol
	z-Score	z-Score	z'-Score	z-Score	z'-Score
1	-1,8	0,66	-1,1	0,47	-0,89
2	-0,11	-1,0	-0,83	0,08	-
3	-1,9	-0,15	-	-0,21	-
4	-3,6	-	-	-2,0	-4,7
5	-	-	-	-	-
6	0,64	-0,29	0,40	-1,1	-0,69
7	1,4	1,0	-0,22	1,8	0,69
8	0,19	-1,6	1,8	-0,83	-0,79
9	1,2	-1,4	2,6	-0,96	-
10	3,3	2,1	-	0,85	0,49
11	0,34	-0,88	-1,7	1,2	1,1
12	-2,2	-1,6	-1,5	-2,2	-1,5
13	-0,26	1,0	-0,45	0,34	-
14	1,2	-0,17	-2,0	0,34	2,7
15	1,2	2,2	3,4	2,9	1,7

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

5.1 Details by the participants

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

5.1.1 Primary Data

Parameter	Parti-cipant	Unit	Sample I DLA No.	Sample II DLA No.	Date of analysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifica-tion	Incl. Re-covery Rate	Recovery rate
					Day/Month					yes / no	in %
E420 - Sorbit / Sorbitol	1	g/100g	19	53	14.07.20	1,7	1,71	1,68	0,03	no	
	2	g/100g	24	48	17.07.20	1,81	1,84	1,79	0,1	no	
	3	g/100g	07	65	06.07.20	1,688	1,705	1,67	0,04	no	
	4	g/100g	3	69	31.08.20	1,58	1,56	1,6	0,0003	no	95
	5	g/100g	34	38	28.08.20	17,61	16,99	18,22	0,09	no	
	6	g/100g	8	22	09.07.20	1,86	1,88	1,84	0,1 g/100g	no	-
	7	g/100g	13	59	18.08.20	1,91	1,9	1,92	0,2	no	
	8	g/100g	6	66	16.07.20	1,83	1,83	1,83	<0.50	no	
	9	g/100g	28	44	13.07.20	1,9	1,9	1,9	0,5	no	
	10	g/100g	No. 33	No. 39	27.08. - 01.09.	yes	1,97	2,1	0,005	no	
	11	g/100g	No.68	No.04	08.08.20	1,84	1,81	1,86	0,1	no	100
	12	g/100g	17	55	03.08.20	1,67	1,66	1,68	0,1		
	13	g/100g	27	45	03.09.20	1,8	1,8	1,8	0,01	no	
	14	g/100g	29	43	11.07.20	1,9	1,9	1,8	0,5	no	-
	15	g/100g	42	30	24.07.20	1,9	1,9	1,8	0,5	no	-

Parameter	Parti-cipant	Unit	Sample I DLA No.	Sample II DLA No.	Date of analysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifica-tion	Incl. Re-covery Rate	Recovery rate
					Day/Month					yes / no	in %
E421 - Mannit / Mannitol	1	g/100g			14.07.20	2,47	2,47	2,47	0,03	no	
	2	g/100g	24	48	17.07.20	2,33	2,37	2,29	0,1	no	
	3	g/100g	07	65	06.07.20	2,402	2,337	2,466	0,04	no	
	4	g/100g									
	5	g/100g	34	38	28.08.20	25,33	25,18	25,47	0,09	no	
	6	g/100g	8	22	09.07.20	2,39	2,25	2,52	0,1 g/100g	no	-
	7	g/100g	13	59	18.08.20	2,5	2,48	2,51	0,2	no	
	8	g/100g	6	66	16.07.20	2,28	2,33	2,23	<0.50	no	
	9	g/100g	28	44	13.07.20	2,3	2,3	2,3	0,5	no	
	10	g/100g	No. 33	No. 39	27.08. - 01.09.	yes	2,66	2,53	0,005	no	
	11	g/100g	No.68	No.04	08.08.20	2,34	2,34	2,33	0,1	no	100
	12	g/100g	17	55	03.08.20	2,28	2,28	2,27	0,1		
	13	g/100g	27	45	03.09.20	2,5	2,5	2,5	0,01	no	
	14	g/100g	29	43	11.07.20	2,4	2,4	2,4	0,5	no	-
	15	g/100g	42	30	24.07.20	2,6	2,6	2,6	0,5	no	-

Parameter	Parti-cipant	Unit	Sample I DLA No.	Sample II DLA No.	Date of analysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifica-tion	Incl. Re-covery Rate	Recovery rate
					Day/Month					yes / no	in %
E953 - Isomalt	1	g/100g			14.07.20	1,81	1,88	1,75	0,03	no	
	2	g/100g	24	48	17.07.20	1,85	1,82	1,88	0,1	no	
	3	g/100g									
	4	g/100g	3	69	31.08.20	1,03	0,998	1,06	0,0003	no	90
	5	g/100g									
	6	g/100g	8	22	09.07.20	2,01	2,1	1,92	0,1 g/100g	no	-
	7	g/100g	13	59	18.08.20	1,93	1,89	1,96	0,2	no	
	8	g/100g	6	66	16.07.20	2,19	2,18	2,2	<0.50	no	
	9	g/100g	28	44	13.07.20	2,3	2,6	2	0,5	no	
	10	g/100g	No. 33	No. 39	27.08. - 01.09.	no					
	11	g/100g	No.68	No.04	08.08.20	1,74	1,78	1,71	0,1	no	100
	12	g/100g	17	55	03.08.20	1,77	1,75	1,78	0,1		
	13	g/100g	27	45	03.09.20	1,9	1,8	2,0	0,01	no	
	14	g/100g	29	43	11.07.20	1,7	1,7	1,6	0,5	no	-
	15	g/100g	42	30	24.07.20	2,4	2,4	2,4	0,5	no	-

Parameter	Parti-cipant	Unit	Sample I DLA No.	Sample II DLA No.	Date of analysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifica-tion	Incl. Re-covery Rate	Recovery rate
					Day/Month					yes / no	in %
E967 - Xylit / Xylitol	1	g/100g			14.07.20	2,21	2,23	2,19	0,03	no	
	2	g/100g	24	48	17.07.20	2,18	2,2	2,16	0,1	no	
	3	g/100g	07	65	06.07.20	2,158	2,152	2,164	0,04	no	
	4	g/100g	3	69	31.08.20	2,02	2,08	1,96	0,0003	no	96
	5	g/100g	34	38	28.08.20	24,61	24,68	24,54	0,04	no	
	6	g/100g	8	22	09.07.20	2,09	2,01	2,16	0,1 g/100g	no	-
	7	g/100g	13	59	18.08.20	2,31	2,29	2,33	0,2	no	
	8	g/100g	6	66	16.07.20	2,11	2,09	2,13	<0.50	no	
	9	g/100g	28	44	13.07.20	2,1	2,1	2,1	0,5	no	
	10	g/100g	No. 33	No. 39	27.08. - 01.09.	yes	2,21	2,27	0,005	no	
	11	g/100g	No.68	No.04	08.08.20	2,27	2,24	2,3	0,1	no	100
	12	g/100g	17	55	03.08.20	2	1,99	2	0,1		
	13	g/100g	27	45	03.09.20	2,2	2,2	2,1	0,01	no	
	14	g/100g	29	43	11.07.20	2,2	2,1	2,2	0,5	no	-
	15	g/100g	42	30	24.07.20	2,4	2,4	2,4	0,5	no	-

Parameter	Parti-cipant	Unit	Sample I DLA No.	Sample II DLA No.	Date of analysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifica-tion	Incl. Re-covery Rate	Recovery rate
					Day/Month					yes / no	in %
E968 - Erythrit / Erythritol	1	g/100g			14.07.20	1,84	1,84	1,84	0,03	no	
	2	g/100g									
	3	g/100g									
	4	g/100g	3	69	31.08.20	1,45	1,34	1,56	0,0003	no	99
	5	g/100g	34	38	28.08.20	21,39	21,87	20,9	0,08	no	
	6	g/100g	8	22	09.07.20	1,86	1,72	1,99	0,1 g/100g	no	-
	7	g/100g	13	59	18.08.20	2	1,99	2	0,2	no	
	8	g/100g	6	66	16.07.20	1,85	1,84	1,86	<0.50	no	
	9	g/100g									
	10	g/100g	No. 33	No. 39	27.08. - 01.09.	yes	1,97	1,99	0,005	no	
	11	g/100g	No.68	No.04	08.08.20	2,04	2,04	2,04	0,1	no	100
	12	g/100g	17	55	03.08.20	1,78	1,77	1,78	0,1		
	13	g/100g									
	14	g/100g	29	43	11.07.20	2,2	2,3	2,1	0,5	no	-
	15	g/100g	42	30	24.07.20	2,1	2	2,1	0,5	no	-

Parameter	Parti-cipant	Unit	Sample I DLA No.	Sample II DLA No.	Date of analysis	Result (Mean)	Result Sample I	Result Sample II	Limit of quantifica-tion	Incl. Re-covery Rate	Recovery rate
					Day/Month					yes / no	in %
E 965 – Maltit / Malitiol	2	g/100g	24	48	17.07.20	<0,1			0,1	no	
	6	g/100g	8	22	09.07.20	<0,1	<0,1	<0,1	0,1 g/100g	no	-
E 966 – Lactit / Lactitol	2	g/100g	24	48	17.07.20	<0,1			0,1	no	

5.1.2 Analytical Methods

Parameter	Partici-pant	Method description as in test report / norm / literature	Sample preparation and processing	Measuring method	Calibration / Reference material	Recovery rate with same matrix	Method accredited ISO/IEC 17025	Further Remarks
						yes / no	yes / no	
E420 - Sorbit / Sorbitol	1	HPAEC-PAD	aqueous extraction, dilution, membrane filtration		external standard		yes	
	2	SLMB Nr. 501.2:1976-01, Akt. 2008 mod.,	standard; no Carrez clarification; Ext. 60 ° C; Silylation using BSTFA; GC condition	GC-FID			yes	
	3	Determination of Sugar Alcohols in Confections and Fruit Juices by High-Performance Anion-Exchange Chromatography with Pulsed Amperometric Detection	Ultrasonic in Milli-Q	HPLC-IC	yes	no	no	
	4	Determination of sugar alcohols in food using GC-FID, internal method	none	none	IS: Xylose	yes	yes	
	5	HPLC-RID				NO	NO	
	6	house method SOP M 3701	Dissolve in ultrapure water	IC with amperometric detection	Sugar Alcohol Kit 47266, Sigma Aldrich	no	yes	
	7	ASU § 64 LFGB L 00.00-59, 2008-12, HPLC-RI	Sample weight: approx. 1 g/20 ml dist. Water, shake for 30 minutes at 40°C, clarify with Carrez	HPLC-RI	D-Sorbitol, Sigma Aldrich		yes	
	8	LC/PAD				NO	YES	
	9	§ 64 LFGB L 00.00-59: 2008-12[a]				yes	yes	
	10			HPIC-PAD			yes	
	11	AOAC 2018.16/ ISO 22184:2019	Extracting with mixture of water and ethanol	HPAEC-PAD		No	No	
	12						Yes	
	13	PV-AC-187		HPAEC-PAD	Solvent calibration	no	yes	
	14	Extraction with a basic mixture of CH3CN-H2O	-	-	-	no	yes	-
	15	HPLCLC/RI - internal method PNTA0177			external calib. curve and internal RM	no	yes	

Parameter	Partici-pant	Method description as in test report / norm / literature	Sample preparation and processing	Measuring method	Calibration / Reference material	Recovery rate with same matrix	Method accredited ISO/IEC 17025	Further Remarks
						yes / no	yes / no	
E421 - Mannit / Mannitol	1	HPAEC-PAD	aqueous extraction, dilution, membrane filtration		external Standard		yes	
	2	SLMB Nr. 501.2:1976-01, Akt. 2008 mod.,	Standard; no Carrez clarification; Ext. 60°C; Silylation using BSTFA; GC condition	GC-FID			yes	
	3	Determination of Sugar Alcohols in Confections and Fruit Juices by High-Performance Anion-Exchange Chromatography with Pulsed Amperometric Detection	Ultrasonic in Milli-Q	HPLC-IC	yes	no	no	
	4							
	5	HPLC-RID				NO	YES	
	6	house method SOP M 3701	Dissolve in ultrapure water	IC with amperometric detection	Sugar Alcohol Kit 47266, Sigma Aldrich	no	yes	
	7	ASU § 64 LFGB L 00.00-59, 2008-12, HPLC-RI	Sample weight: approx. 1 g/20 ml dist. Water, shake for 30 minutes at 40°C, clarify with Carrez	HPLC-RI	D-Mannitol, Sigma Aldrich		yes	
	8	LC/PAD				NO	YES	
	9	§ 64 LFGB L 00.00-59: 2008-12[a]				yes	yes	
	10			HPIC-PAD			yes	
	11	AOAC 2018.16/ ISO 22184:2019	Extracting with mixture of water and ethanol	HPAEC-PAD		No	No	
	12						Yes	
	13	PV-AC-187		HPAEC-PAD	Solvent calibration	no	yes	
	14	Extraction with a basic mixture of CH3CN-H2O	-	-	-	no	yes	-
	15	HPLCLC/RI - internal method PNTA0177			external calib. curve and internal RM	no	yes	

Parameter	Partici-pant	Method description as in test report / norm / literature	Sample preparation and processing	Measuring method	Calibration / Reference material	Recovery rate with same matrix	Method accredited ISO/IEC 17025	Further Remarks
						yes / no	yes / no	
E953 - Isomalt	1	HPAEC-PAD	aqueous extraction, dilution, membrane filtration		external Standard		yes	
	2	SLMB Nr. 501.2:1976-01, Akt. 2008 mod.,	Standard; no Carrez clarification; Ext. 60°C; Silylation using BSTFA; GC condition	GC-FID			yes	
	3							
	4	Determination of sugar alcohols in food using GC-FID, internal method	none	none	IS: Cellobiose	yes	yes	
	5							
	6	house method SOP M 3701	Dissolve in ultrapure water	IC with amperometric detection	Isomalt Pharmaceutical Secondary Standard; Certified Reference Material PHR1769-1G, Sigma-Aldrich	no	yes	
	7	ASU § 64 LFGB L 00.00-59, 2008-12, HPLC-RI	Sample weight: approx. 1 g/20 ml dist. Water, shake for 30 minutes at 40°C, clarify with Carrez	HPLC-RI	Isomalt CRS, Sigma Aldrich		yes	
	8	LC/PAD				NO	YES	
	9	§ 64 LFGB L 00.00-59: 2008-12[a]				yes	yes	
	10							
	11	AOAC 2018.16/ ISO 22184:2019	Extracting with mixture of water and ethanol	HPAEC-PAD		No	No	
	12						Yes	
	13	PV-AC-187		HPAEC-PAD	Solvent calibration	no	yes	
	14	Extraction with a basic mixture of CH3CN-H2O	-	-	-	no	yes	-
	15	HPLCLC/RI - internal method PNTA0177			external calib. curve and internal RM	no	yes	

Parameter	Partici-pant	Method description as in test report / norm / literature	Sample preparation and processing	Measuring method	Calibration / Reference material	Recovery rate with same matrix	Method accredited ISO/IEC 17025	Further Remarks
						yes / no	yes / no	
E967 - Xylit / Xylitol	1	HPAEC-PAD	aqueous extraction, dilution, membrane filtration		external Standard		yes	
	2	SLMB Nr. 501.2:1976-01, Akt. 2008 mod.,	Standard; no Carrez clarification; Ext. 60°C; Silylation using BSTFA; GC condition	GC-FID			yes	
	3	Determination of Sugar Alcohols in Confections and Fruit Juices by High-Performance Anion-Exchange Chromatography with Pulsed Amperometric Detection	Ultrasonic in Milli-Q	HPLC-IC	yes	no	no	
	4	Determination of sugar alcohols in food using GC-FID, internal method	none	none	IS: Xylose	yes	yes	
	5	HPLC-RID				NO	YES	
	6	house method SOP M 3701	Dissolve in ultrapure water	IC with amperometric detection	Sugar Alcohol Kit 47266, Sigma Aldrich	no	yes	
	7	ASU § 64 LFGB L 00.00-59, 2008-12, HPLC-RI	Sample weight: approx. 1 g/20 ml dist. Water, shake for 30 minutes at 40°C, clarify with Carrez	HPLC-RI	Xylitol, Alfa Aesar, Sigma-Aldrich		yes	
	8	LC/PAD				NO	YES	
	9	§ 64 LFGB L 00.00-59: 2008-12[a]				yes	yes	
	10			HPIC-PAD			yes	
	11	AOAC 2018.16/ ISO 22184:2019	Extracting with mixture of water and ethanol	HPAEC-PAD		No	No	
	12						Yes	
	13	PV-AC-187		HPAEC-PAD	Solvent calibration	no	yes	
	14	Extraction with a basic mixture of CH3CN-H2O	-	-	-	no	yes	-
	15	HPLCLC/RI - internal method PNTA0177			external calib. curve and internal RM	no	yes	

Parameter	Partici-pant	Method description as in test report / norm / literature	Sample preparation and processing	Measuring method	Calibration / Reference material	Recovery rate with same matrix	Method accredited ISO/IEC 17025	Further Remarks
						yes / no	yes / no	
E968 - Erythrit / Erythritol	1	HPAEC-PAD	aqueous extraction, dilution, membrane filtration		external Standard		yes	
	2							
	3							
	4	Determination of sugar alcohols in food using GC-FID, internal method	none	none	IS: Xylose	yes	yes	
	5	HPLC-RID				NO	YES	
	6	house method SOP M 3701	Dissolve in ultrapure water	IC with amperometric detection	Sugar Alcohol Kit 47266, Sigma Aldrich	no	yes	
	7	ASU § 64 LFGB L 00.00-59, 2008-12, HPLC-RI , modified	Sample weight: approx. 1 g/20 ml dist. Water, shake for 30 minutes at 40°C, clarify with Carrez	HPLC-RI	Erythritol, Alfa Aesar, Sigma-Aldrich		yes	
	8	LC/PAD				NO	YES	
	9							
	10			HPIC-PAD			yes	
	11	AOAC 2018.16/ ISO 22184:2019	Extracting with mixture of water and ethanol	HPAEC-PAD		No	No	
	12						Yes	
	13							not included in the bilacon spectrum
	14	Extraction with a basic mixture of CH3CN-H2O	-	-	-	no	yes	-
	15	HPLCLC/RI - internal method PNTA0177			external calib. curve and internal RM	no	yes	

Parameter	Partici-pant	Method description as in test report / norm / literature	Sample preparation and processing	Measuring method	Calibration / Reference material	Recovery rate with same matrix	Method accredited ISO/IEC 17025	Further Remarks
						yes / no	yes / no	
E 965 – Maltit / Malitiol	2	SLMB Nr. 501.2:1976-01, Akt. 2008 mod.,	Standard; no Carrez clarification; Ext. 60°C; Silylation using BSTFA; GC condition	GC-FID			yes	
	6	house method SOP M 3701	Dissolve in ultrapure water	IC with amperometric detection	Sugar Alcohol Kit 47266, Sigma Aldrich	no	yes	
E 966 – Lactit / Lactitol	2	SLMB Nr. 501.2:1976-01, Akt. 2008 mod.,	Standard; no Carrez clarification; Ext. 60°C; Silylation using BSTFA; GC condition	GC-FID			yes	

5.2 Homogeneity

5.2.1 Mixture homogeneity before bottling

Microtracer Homogeneity Test
DLA ptAU4

Weight whole sample	1,00	kg
Microtracer	FSS-rot lake	
Particle size	75 – 300	µm
Weight per particle	2,0	µg
Addition of tracer	24,2	mg/kg

Result of analysis

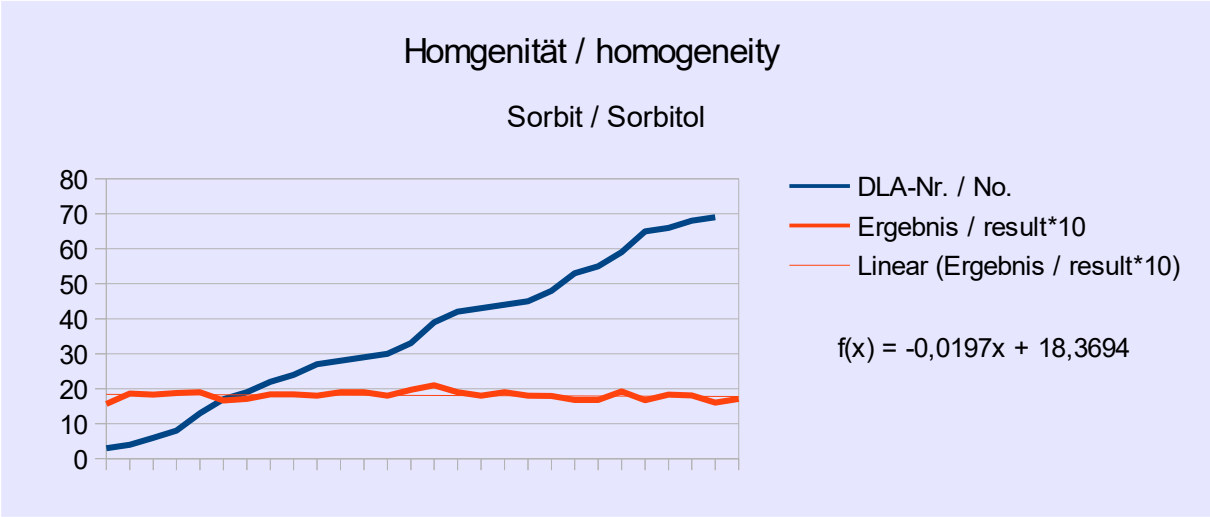
Sample	Weight [g]	Particle number	Particles [mg/kg]
1	4,95	52	21,0
2	5,01	52	20,8
3	5,03	56	22,3
4	5,04	59	23,4
5	4,99	50	20,0
6	4,98	52	20,9
7	5,05	46	18,2
8	5,01	46	18,4

Poisson distribution		
Number of samples	8	
Degree of freedom	7	
Mean	51,6	Particles
Standard deviation	4,43	Particles
χ^2 (CHI-Quadrat)	2,67	
Probability	**91**	%
Recovery rate	85	%

Normal distribution		
Number of samples	8	
Mean	20,6	mg/kg
Standard deviation	1,77	mg/kg
rel. Standard deviaton	8,59	%
Horwitz standard deviation	10,1	%
HorRat-value	**0,85**	
Recovery rate	85	%

5.2.2 Trend line function of the participants results

By comparison of the increasing sample numbers and the measurement results of participants, the homogeneity of the chronological bottled PT items can be shown by the trend line for information:



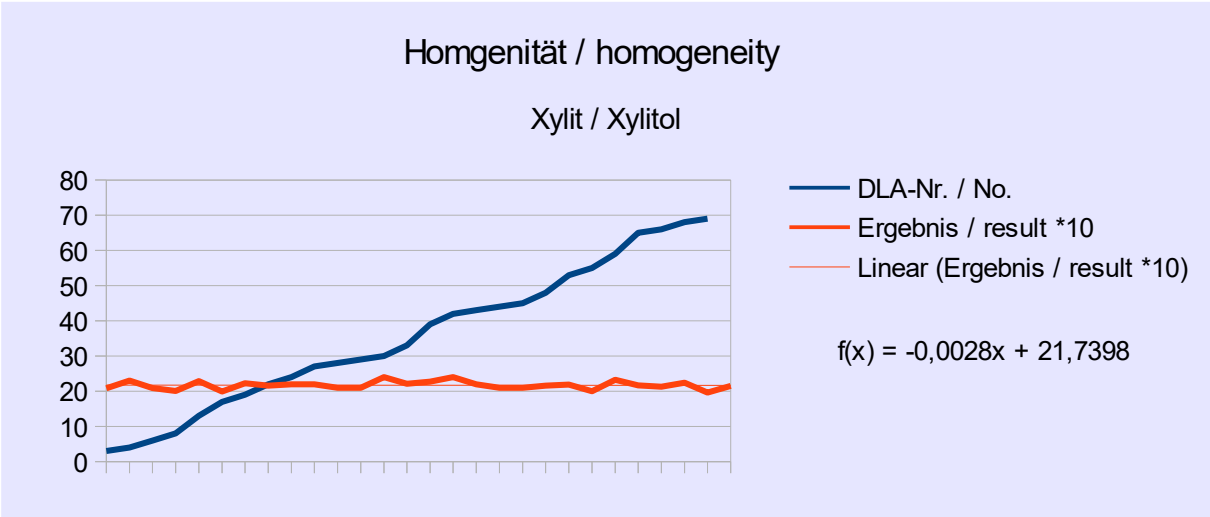


Abb./Fig. 16: Trendfunktion Probennummern vs. Ergebnisse von Sorbit und Xylit (1*10 dargestellt)
trend line function sample number vs. results of sorbitol and xylitol (1*10 shown)

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 ptAU04 - 2020***
PT name	***Sugar Alcohols (E420, E421, E953, E967, E968) in Pudding Powder***
*Sample matrix**	***Samples I + II:*** *Pudding Powder Chocolate / Ingredients: starch, low-fat cocoa powder, flavor and sugar alcohols*
Number of samples and sample amount	*2 identical samples I + II, 10 g each.*
Storage	*Samples I + II:* *room temperature*
Intentional use	*Laboratory use only (quality control samples)*
Parameter	*quantitative:* ***Sugar Alcohols (E420, E421, E953, E967, E968)***
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.*
Result sheet	*The results for sample I and II as well as the final results calculated as mean of the double determination (samples I and II) should be filled in the result submission file. The recovery rates, if carried out, has to be included in the calculation.*
Units	*g/100g*
Number of significant digits	*at least 2*
Further information	*For information please specify:* – *Date of analysis* – *DLA-sample-numbers (for sample I and II)* – *Limit of detection* – *Assignment incl. Recovery* – *Recovery with the same matrix* – *Method is accredited*
Result submission	*The result submission file should be sent by e-mail to:* ***pt@dla-lvu.de***
Last Deadline	***the latest September 04th 2020***
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, Ph.D.*

** 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
		GREAT BRITAIN
		Germany
		Germany
		GREECE
		SPAIN
		ITALY
		SWEDEN
		Germany
		Germany
		Germany
		Germany
		Germany
		Germany
		VIETNAM
		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 interlaboratory comparisons
4. ASU §64 LFGB: Planung und statistische Auswertung von Ringversuchen zur Methodenvalidierung / DIN ISO 5725 series part 1, 2 and 6 Accuracy (trueness and precision) of measurement methods and results
5. Verordnung / Regulation 882/2004/EU; Verordnung über über amtliche Kontrollen zur Überprüfung der Einhaltung des Lebensmittel- und Futtermittelrechts sowie der Bestimmungen über Tiergesundheit und Tierschutz / Regulation on official controls performed to ensure the verification of compliance with feed and food law, animal health and animal welfare rules
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16. 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 Requirements, Appendix F, p. 2, AOAC Int (2016)
18. ASU §64 LFGB: L 00.00-59 (2008): Bestimmung von Isomalt, Lactit, Maltit, Mannit, Sorbit und Xylit in Lebensmitteln, HPLC-Verfahren / EN 15086 (2006): Foodstuffs - Determination of isomalt, lactitol, maltitol, mannitol, sorbitol and xylitol in foodstuffs
19. ASU §64 LFGB: L 18.00-14 (1994): Bestimmung von D-Sorbit in Feinen Backwaren, Enzymatische Verfahren (1994) [Determination of D-sorbitol in fine baked goods, Enzymatic method]