DLA
Dienstleistung
Lebensmittel
Analytik GbR

Evaluation Report
proficiency test

DLA 39/2018

Sugar Alcohols (E420, E421, E953, E 965, E 966, E967, E968) in vegetable product

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

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EP-Bericht Freigabe *PT-Report Authorization*	Dr. Matthias Besler-Scharf (Technischer Leiter / Technical Manager) *- gezeichnet / signed M. Besler-Scharf* Dr. Gerhard Wichmann (QM-Beauftragter / Quality Manager) *- gezeichnet / signed G. Wichmann* Datum / Date: 21 October 2018
Unteraufträge *Subcontractors*	Falls im Rahmen der Eignungsprüfung eine Prüfung der Gehalte, Homogenität und Stabilität von EP-Parametern durchgeführt wurde, hat DLA diese im Unterauftrag vergeben. 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 *Confidentiality*	Die Teilnehmerergebnisse sind im EP-Bericht in anonymisierter Form mit Auswertenummern benannt. Daten einzelner Teilnehmer werden ausschließlich nach vorheriger Zustimmung des Teilnehmers an Dritte weitergegeben. Participant result are named anonymously with evaluation numbers in the PT report. Data of individual participants will be passed on to third parties only with prior consent of the participant.

Inhalt / Content

1. Introduction

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

2. Realisation

2.1 Test material

The test material was a pudding powder "vanilla taste" (ingredients: corn starch, salt, flavour: natural vanilla flavour, colours: E 101 and E 160b), added were sorbitol (E 420/ 3,0%), mannitol (E 421/ 2,0%), isomalt (E 953/ 3,0%), lactitol (E 966/ 2,0%), xylitol (E 967/ 2,0%) and erythritol (E 968/ 3,0%). To the mixture were further added microtracer iron particles (FSS red lake) to homogeneity verification.

Approximately 1000 g of the material were mixed, sieved, homogenized and then packaged in portions to approximately 10 g. The portions were numbered chronologically.

2.1.1 Homogeneity

The **mixture homogeneity before bottling** was examined 10-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 showed a probability of 77%. 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 evaluation HorRat values between 0,3 and 1,3 are to be accepted under repeat conditions (measurements within a laboratory) [17]. This gave a HorRat value of 0,8. The results of microtracer analysis are given in the documentation.

The calculation of the **repeatability standard deviations S_r** of the participants were also used as an indicator of homogeneity. The result is similar to the repeatability standard deviation of the official method (e.g. ASU EN etc., see 3.6.2. The repeatability standard deviation of the participants is given in the documentation and in the statistic data (see 4.1 to 4.6).

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

2.1.2 Stability

A water activity (a_W) of < 0,6 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 <0,6).
The a_W value of the PT samples was approx. 0,53 (19,9°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 30th week of 2018. The testing method was optional. The tests should be finished at September 21th 2016 the latest.

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

The two portions contain identical samples of the same pudding powder with sugar alcohols (E420, E421, E953, E967, E968, other).

Please note the attached information on the proficiency test.
(see documentation, section 5.3 Information on the PT)

2.3 Results

The participants submitted their results in standard forms, which have been handed out with the samples (by email). The finally calculated concentrations as average of duplicate determinations of both numbered samples was 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 method.

From the 13 participants all participants submitted the result in time.

3. Evaluation

3.1 Consensus values 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. It should be noted that the significance may be limited due to the small number of results.

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

3.2 Robust standard deviation

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

3.3 Repeatability standard deviation

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

The calculation of the repeatability standard deviation S_r is performed by: [3, 4].

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

3.4 Reproducibility standard deviation

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

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

The calculation of the reproducibility standard deviation S_R is performed by: [3, 4].

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

3.5 Exclusion of results and outliers

Before statistical evaluation obvious blunders, such as those with incorrect units, decimal point errors, and results for a another proficiency test item can be removed from the data set [2]. Even if a result 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 identified as 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 is classified as an outlier [3]. Due to the using of robust statistics, outliers are generally excluded from the evaluation, unless there are other reasons (see above) [3]. Determined 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.

To evaluate the results, the target standard deviation was calculated for all sugar alcohols except erythritol according to the characteristics of a precision experiment (ASU §64 method, see 3.6.2). For the evaluation of erythritol the target standard deviation according to the general model of Horwitz was used (see 3.6.1). In addition, the standard uncertainty was taken into account for all PT-parameters and the results were evaluated by z´-score (see 3.8).

As additional information, the target standard deviation, calculated according to the Horwitz general model, was given for all sugar alcohols (except erythritol) (see 3.6.1).

3.6.1 General model (Horwitz)

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

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

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

3.6.2 Precision experiment

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

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

The values given in Table 1 relative repeatability standard deviation (RSD_r) and relative reproducibility standard deviation (RSD_R) were determined in collaborative trials using the specified methods.

The resulting target standard deviations σ_{pt} identified there (*) were used to evaluate the results or were additionally indicated in the statistical data for information purposes.

Table 1: Relative repeatability standard deviation (RSD_r) and relative reproducibility standard deviation (RSD_R) for the sugar alcohols according to evaluations of experiments for precision [16, 17]

Parameter	Matrix	Mean	RSD_r	RSD_R	σ_{pt}	Method/ Literature
Xylitol (E967)*	cookies	3,03%	1,62%	3,76%	3,58%	HPLC-RI/16
Sorbitol (E420)*	cookies	3,76%	1,52%	3,91%	3,76%	HPLC-RI/16
Sorbitol (E420)	cookies	4,66%	1,65%	2,66%	2,66%	Enzymatic/17
Mannitol (E421)*	cookies	4,34%	1,24%	3,55%	3,44%	HPLC-RI/16
GPS[1] (E953)	cookies	13,5%	0,52%	3,41%	3,41%	HPLC-RI/16
GPM[1] (E953)*	cookies	12,6%	0,66%	4,47%	4,45%	HPLC-RI/16
Lactitol (E966)*	cookies	6,11%	1,82%	7,83%	7,72%	HPLC-RI/16

[1] 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).

* The precision data are used to calculate the target standard deviation according ASU.

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].

3.7 z-Score

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

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

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

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

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

3.7.1 Warn- und Eingriffssignale

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

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

3.8 z'-Score

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

The calculation is performed by:

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

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

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

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

For warning and action signals see 3.7.1.

3.9 Reproducibility coefficient (CV)

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

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

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

3.10 Quotient S^*/σ_{pt}

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

3.11 Standard uncertainty

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

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

If $U(x_{pt}) \leq 0{,}3\ \sigma_{pt}$ the standard uncertainty of the consensus value needs not to be included in the interpretation of the results of the PT [3]. A clear exceeded the value of 0.3 is an indication that the target standard deviation was possibly set too low for the standard uncertainty of the assigned value.

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

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

4. Results

In the present PT the sugar alcohols E 420, E 421, E 953, E 966 E 967 and E 968 were added.
For comparison of the recoveries and precision of measurement, we have compiled the statistic data of the results in Table 2.

Table 2: Compilation of the characteristics of the sugar alcohols E 420, E 421, E 953, E 966 and E 967 (in g/100g)

Parameter	E 420	E 421	E 953	E 966	E 967	E 968
Spike (doping)	3,0	2,0	3,0	2,0	2,0	3,0
Rob. mean (X_{pt})	2,93	1,93	2,87	1,91	2,10	3,18
Recovery	97,7%	96,5%	95,7%	95,5%	105%	106%
Rob. Standard deviation (S*)	0,446	0,322	0,765	0,217	0,304	0,532
Repeatability standard deviation (S_r)	0,0904	0,0430	0,250	0,109	0,0715	0,0866
Reproducibility standard deviation (S_R)	0,413	0,355	0,693	0,227	0,297	0,643
Target standard deviation σ_{pt} or σ_{pt}'	0,192	0,138	0,343	0,185	0,130	0,236
Variation coefficient (CV_R)	14,1%	18,7%	24,3%	12,0%	14,2%	20,7%
Standard uncertainty $U_{(Xpt)}$	0,158	0,121	0,319	0,111	0,106	0,210
Quotient S*/σ_{pt} *or* S*/σ_{pt}'	2,3	2,3	2,2	1,2	2,3	2,3
Quotient $U_{(Xpt)}/\sigma_{pt}$'	0,82	0,88	0,93	0,60	0,81	0,89

The reproducibility coefficients are in the range of 12 – 24% and are therefore acceptable. The recovery is about 100% (96% – 106%).

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

In the first table the characteristics are listed:

Statistic Data
Number of results
Number of outliers
Mean
Median
Robust mean (X_{pt})
Robust standard deviation (S^x)
Number with 2 replicates
Repeatability standard deviation (S_r)
Repeatability (CV_r)
Reproducibility standard deviation (S_R)
Reproducibility (CV_R)
Target range:
Target standard deviation σ_{pt} or σ_{pt}'
Target standard deviation for information
lower limit of target range ($X_{pt} - 2\sigma_{pt}$) or ($X_{pt} - 2\sigma_{pt}$') *
upper limit of target range ($X_{pt} + 2\sigma_{pt}$) or ($X_{pt} + 2\sigma_{pt}$´) *
Quotient S/σ_{pt} or S*/σ_{pt}'*
Standard uncertainty $U(X_{pt})$
Quotient $U(X_{pt})/\sigma_{pt}$ or $U(X_{pt})/\sigma_{pt}$'
Number of results in the target range
Percent in the target range

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

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

Auswerte-nummer	**Parameter [Einheit/ Unit]**	**Abweichung**	**Z-Score** σ_{pt}	**z-Score** (Info)	**Hinweis**
Evaluation number		**Deviation**			**Remark**

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

4.1 Sorbitol (E420) in g/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	12
Number of outliers	
Mean	2,93
Median	2,97
Robust Mean (X)	**2,93**
Robust standard deviation (S*)	**0,436**
Number with 2 replicates	12
Repeatability SD (S_r)	0,0904
Repeatability (CV_r)	3,09%
Reproducibility SD (S_R)	0,413
Reproducibility (CV_R)	14,1%
Target range:	
Target standard deviation σ_{pt}'	**0,192**
Target standard deviation (for Information)	0,100
lower limit of target range	**2,54**
upper limit of target range	**3,31**
Quotient S/σ_{pt}'*	*2,3*
Standard uncertainty $U_{(Xpt)}$	*0,158*
Quotient $U_{(Xpt)}/\sigma_{pt}'$	*0,82*
Results in the target range	*8*
Percent in the target range	*67%*

Comments:

The target standard deviation was calculated using the target standard deviation according to ASU (precision experiment) and calculated as σ_{pt}'.

As additional information, the target standard deviation, calculated according to the Horwitz general model, was given.

The evaluation of the results shows an acceptable variability of results, in particular because the tests using different methods (HPAEC-PAD,HPLC-RI, L-RID, GC-FID)). The quotient $S^x/\sigma_{pt}'$was slightly above 2,0. The quotient $U(X_{pt})/\sigma_p'$ is above 0,3 at 0,82, but to accept because of the different methods.

The reproducibility coefficient of variation is low (14%). The robust standard deviation is in the range of established values for the standard deviation of the methods used. The reproducibility of the results is given.

67 % of the results are within the target range.

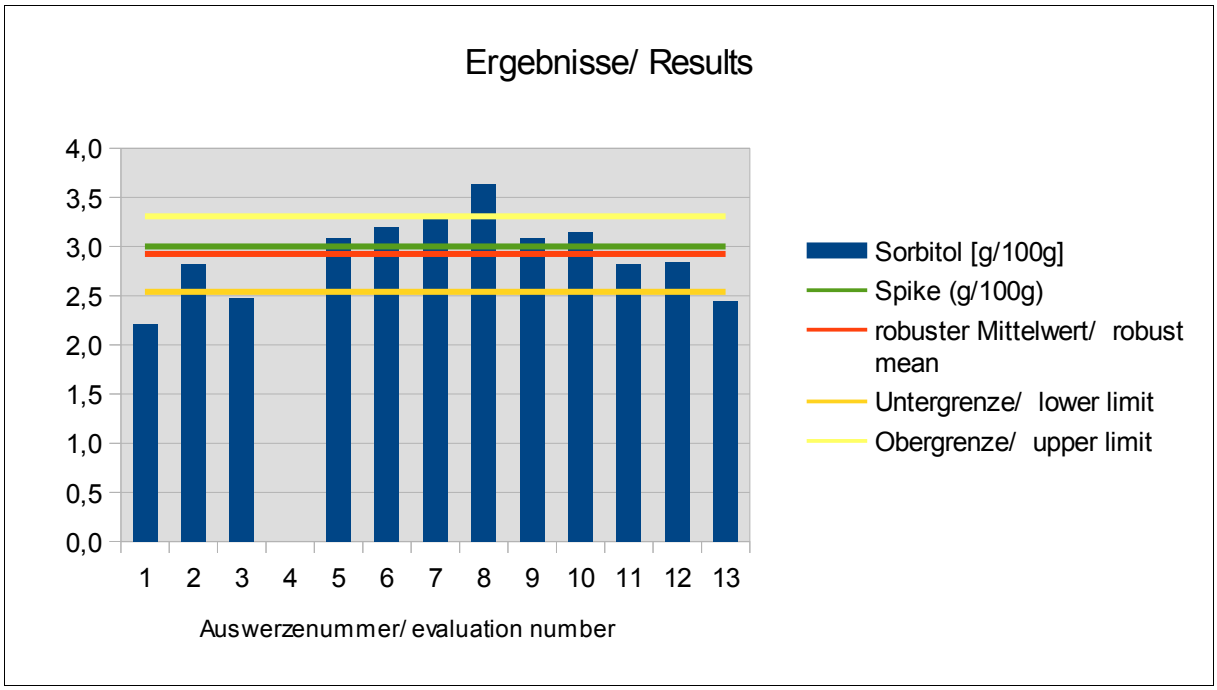

Abb. 1: Ergebnisse E 420
Fig. 1: Results E 420

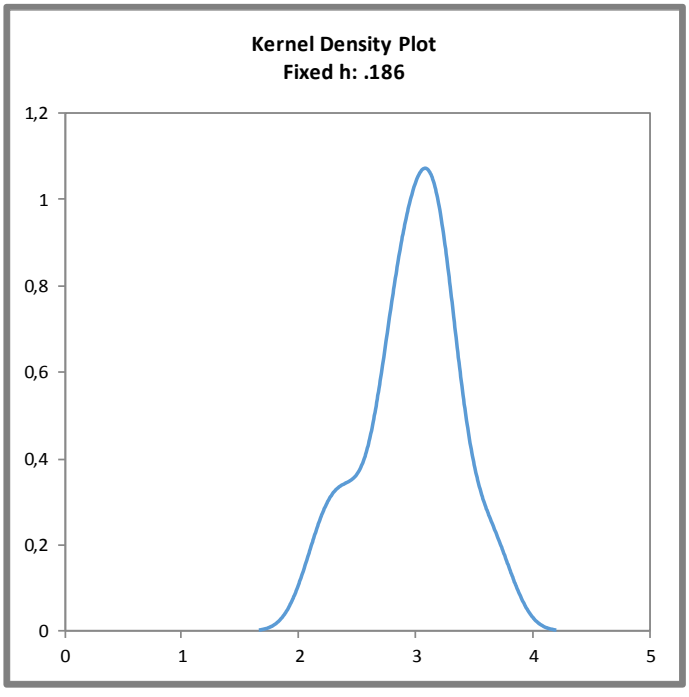

Abb. 2: Kerndichte Plot der Ergebnisse E 420 mit h = Ziel-standardabweichung
Fig. 2: Kernel density plot of the E 420 results with h = target standard deviation

Comments:
The kernel density plot shows a symmetrical distribution of results. The shoulder at 2,4 g/100g indicates results outside the target range.

Ergebnisse der teilnehmenden Institute:
Results of Participants:

Auswerte-nummer Evaluation number	Sorbitol [g/100g]	Abweichung [g/100g] Deviation [g/100g]	z´-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	2,21	-0,715	-3,7	-7,2	
2	2,83*	-0,100	-0,52	-1,0	
3	2,48	-0,445	-2,3	-4,5	
4					
5	3,09	0,165	0,86	1,7	
6	3,20	0,275	1,4	2,8	
7	3,30	0,375	2,0	3,8	
8	3,64	0,718	3,7	7,2	
9	3,09	0,165	0,86	1,7	
10	3,15	0,225	1,2	2,3	
11	2,82	-0,105	-0,5	-1,1	
12	2,85	-0,080	-0,4	-0,8	
13	2,45	-0,475	-2,5	-4,8	

* Mean calculaded by DLA

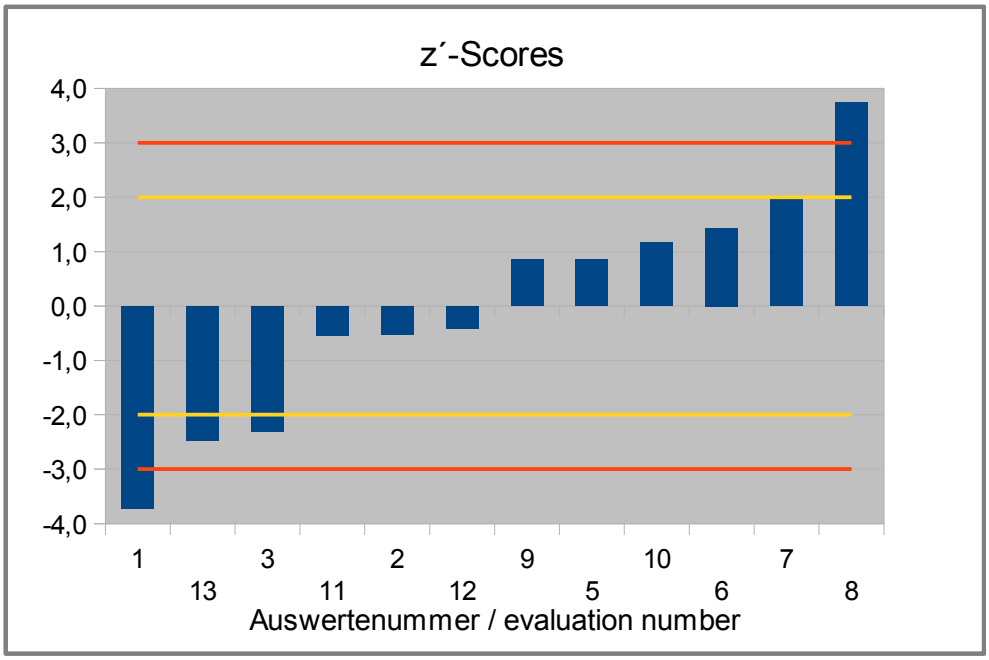

Abb. 3: Z-Scores E 420
Fig. 3: Z-Scores E 420

4.2 Mannitol (E421) in g/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	11
Number of outliers	
Mean	1,90
Median	1,90
Robust Mean (X)	**1,93**
Robust standard deviation (S*)	**0,322**
Number with 2 replicates	11
Repeatability SD (S_r)	0,0430
Repeatability (CV_r)	2,27%
Reproducibility SD (S_R)	0,355
Reproducibility (CV_R)	18,7%
Target range:	
Target standard deviation σ_{pt}´	**0,138**
Target standard deviation (for Information)	0,0699
lower limit of target range	**1,65**
upper limit of target range	**2,21**
Quotient S/σ_{pt}´*	*2,3*
Standard uncertainty $U_{(Xpt)}$	*0,121*
Quotient $U_{(Xpt)}/\sigma_{pt}$´	*0,88*
Results in the target range	*8*
Percent in the target range	*73%*

Comments:

The target standard deviation was calculated using the target standard deviation according to ASU (precision experiment) and calculated as σ_{pt}´.

As additional information, the target standard deviation, calculated according to the Horwitz general model, was giv**en.**

The evaluation of the results shows an acceptable variability of results, in particular because the tests using different methods (HPAEC-PAD,HPLC-RI, L-RID, GC-FID)). The quotient S^x/σ_{pt}´was slightly above 2,0. The quotient $U(X_{pt})/\sigma_p$´ is above 0,3 at 0,88, but to accept because of the different methods.

The reproducibility coefficient of variation is normal (19%). The robust standard deviation is in the range of established values for the standard deviation of the methods used. The reproducibility of the results is given.

73 % of the results are within the target range.

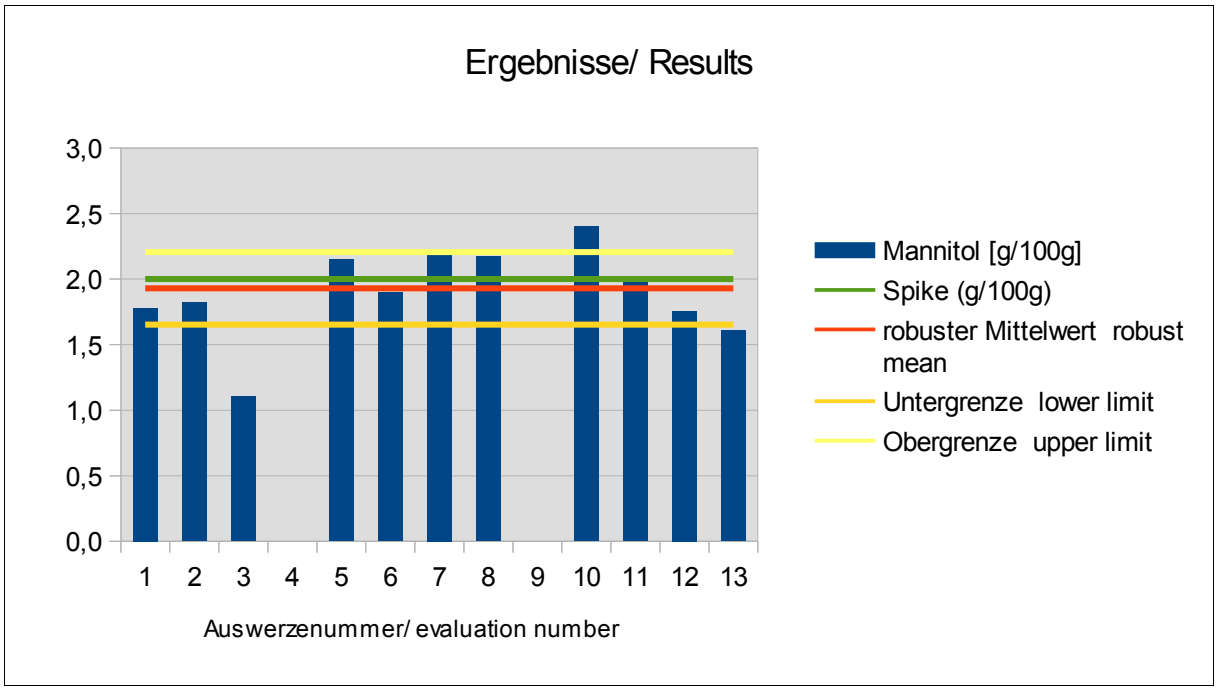

Abb. 4: Ergebnisse E 421
Fig. 4: Results E 421

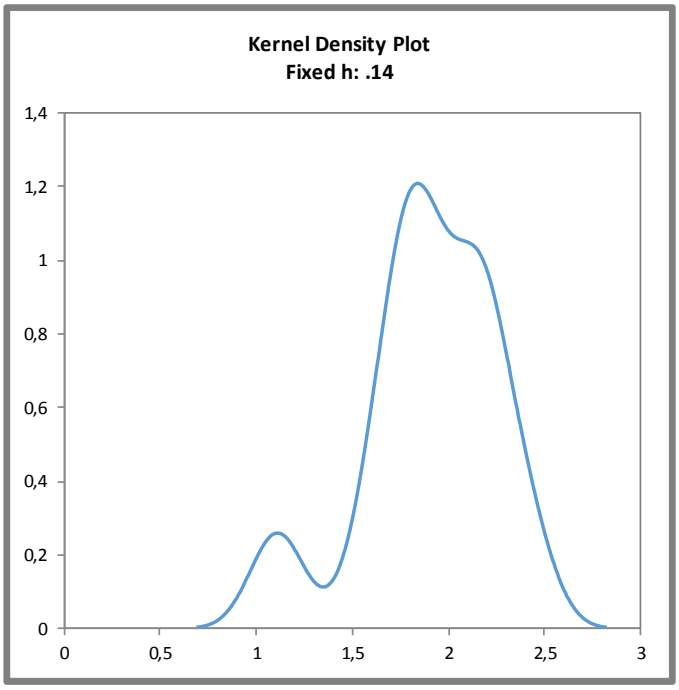

Abb. 5: Kerndichte Plot der Ergebnisse E 421 mit h = Zielstandardabweichung
Fig. 5: Kernel density plot of the E 421 results with h = target standard deviation

Comments:
The kernel density plot shows a symmetrical distribution of results. The shoulder at 2,4 g/100g and the second peak at 1,1 g/100g indicates results outside the target range.

Ergebnisse der teilnehmenden Institute:
Results of Participants:

Auswerte-nummer Evaluation number	Mannitol [g/100g]	Abweichung [g/100g] Deviation [g/100g]	z´-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	1,78	-0,150	-1,1	-2,1	
2	1,82*	-0,110	-0,79	-1,6	
3	1,11	-0,825	-6,0	-12	
4					
5	2,15	0,220	1,6	3,1	
6	1,90	-0,030	-0,22	-0,43	
7	2,20	0,270	2,0	3,9	
8	2,17	0,244	1,8	3,5	
9					
10	2,40	0,470	3,4	6,7	
11	1,99	0,0602	0,44	0,86	
12	1,76	-0,172	-1,2	-2,5	
13	1,61	-0,320	-2,3	-4,6	

* Mean calculated by DLA

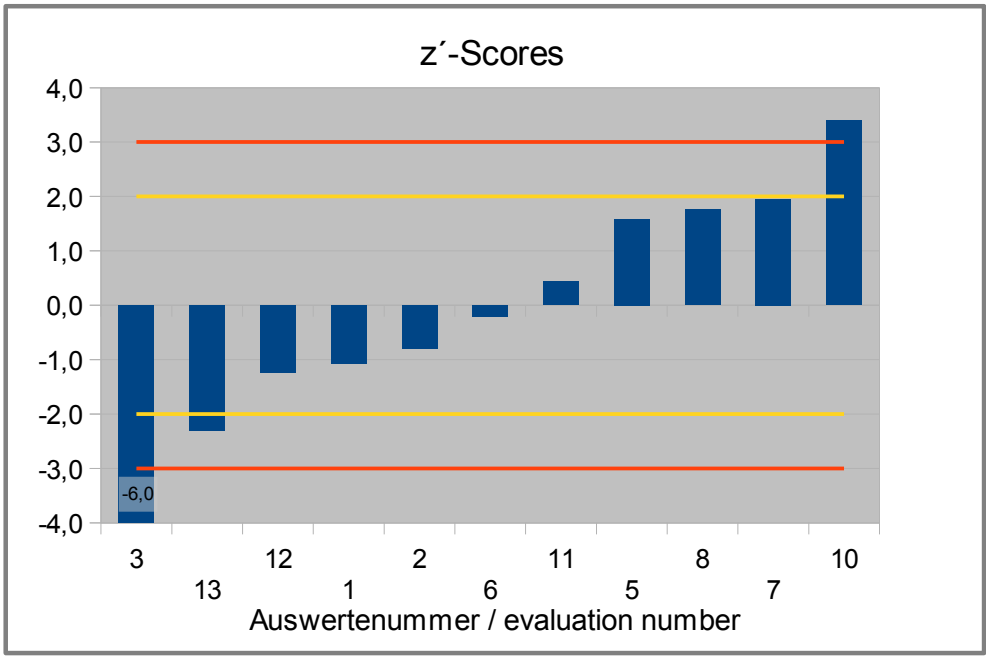

Abb. 6: Z-Scores E 421
Fig. 6: Z-Scores E 421

4.3 Isomalt (E953) in g/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	9
Number of outliers	
Mean	2,87
Median	3,03
Robust Mean (X)	**2,87**
Robust standard deviation (S*)	**0,765**
Number with 2 replicates	9
Repeatability SD (S_r)	0,250
Repeatability (CV_r)	8,74%
Reproducibility SD (S_R)	0,693
Reproducibility (CV_R)	24,3%
Target range:	
Target standard deviation σ_{pt}´	**0,343**
Target standard deviation (for Information)	0,0979
lower limit of target range	**2,18**
upper limit of target range	**3,56**
Quotient S/σ_{pt}´*	*2,2*
Standard uncertainty $U_{(Xpt)}$	*0,319*
Quotient $U_{(Xpt)}/\sigma_{pt}$´	*0,93*
Results in the target range	*5*
Percent in the target range	*56%*

Comments:

The target standard deviation was calculated using the target standard deviation according to ASU (precision experiment) and calculated as σ_{pt}´.

As additional information, the target standard deviation, calculated according to the Horwitz general model, was giv**en.**

The evaluation of the results shows an acceptable variability of results, in particular because the tests using different methods (HPAEC-PAD,HPLC-RI, L-RID, GC-FID)). The quotient S^x/σ_{pt}´was slightly above 2,0. The quotient $U(X_{pt})/\sigma_p$´ is above 0,3 at 0,93, but to accept because of the different methods.

The reproducibility coefficient of variation is acceptable (24%). The robust standard deviation is in the range of established values for the standard deviation of the methods used. The reproducibility of the results is given.

56 % of the results are within the target range.

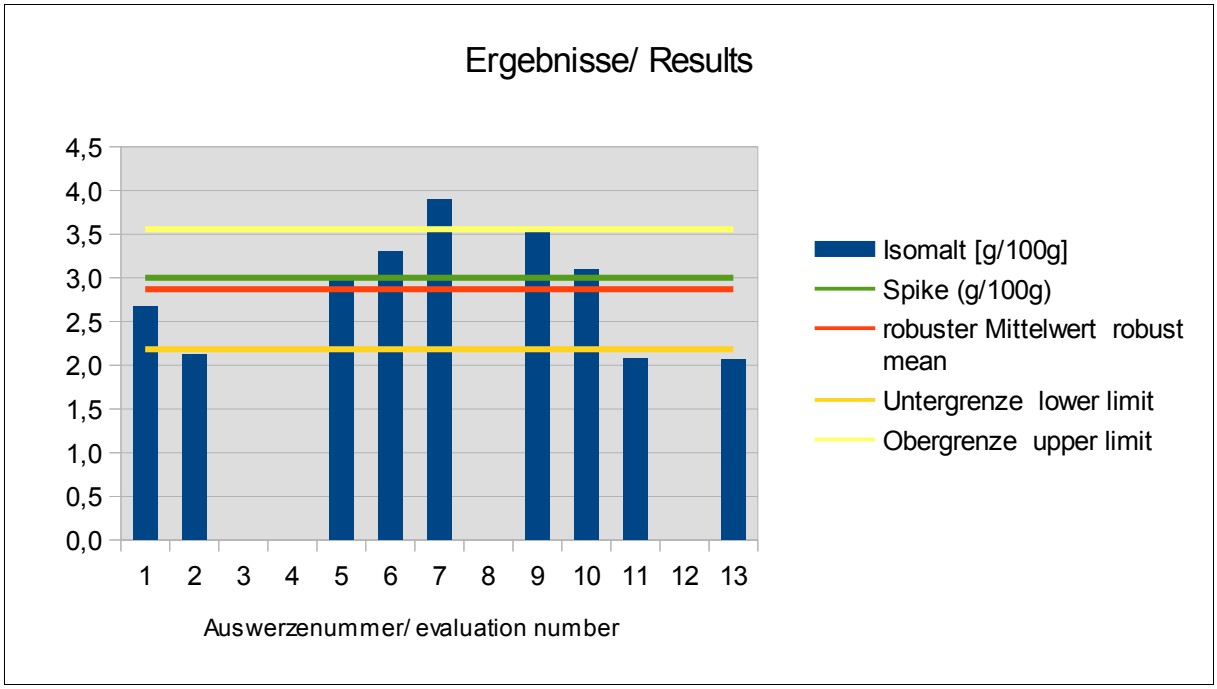

Abb. 7: Ergebnisse E 953
Fig. 7: Results E 953

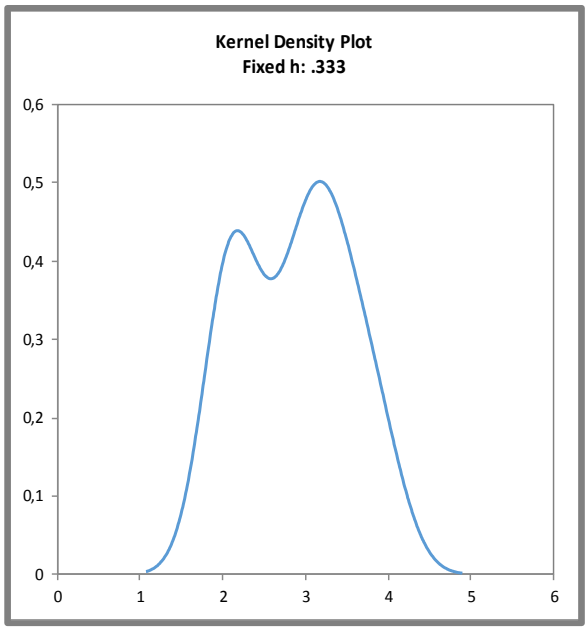

Abb. 8: Kerndichte Plot der Ergebnisse E 953 mit h = Zielstandardabweichung
Fig. 8: Kernel density plot of the E 953 results with h = target standard deviation

Comments:
The kernel density plot shows a symmetrical distribution of results. The second peak at 2,1 g/100g indicates results outside the target range. Systematic reasons for the relatively strong second peak are not clearly identifiable. Participant 2 used a HPLC method and participants 11 and 13 GC methods. The robust mean value (2,87 g/100g) corresponds quite well with the spiked level (3,0%) (-4.3%).

Ergebnisse der teilnehmenden Institute:
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	2,67	-0,199	-0,58	-2,0	
2	2,13*	-0,744	-2,2	-7,6	
3					
4					
5	3,03	0,161	0,47	1,6	
6	3,30	0,431	1,3	4,4	
7	3,90	1,03	3,0	10,5	
8					
9	3,55	0,676	2,0	6,9	
10	3,10	0,231	0,67	2,4	
11	2,08	-0,789	-2,3	-8,1	
12					
13	2,07	-0,799	-2,3	-8,2	

* Mean calculated by DLA

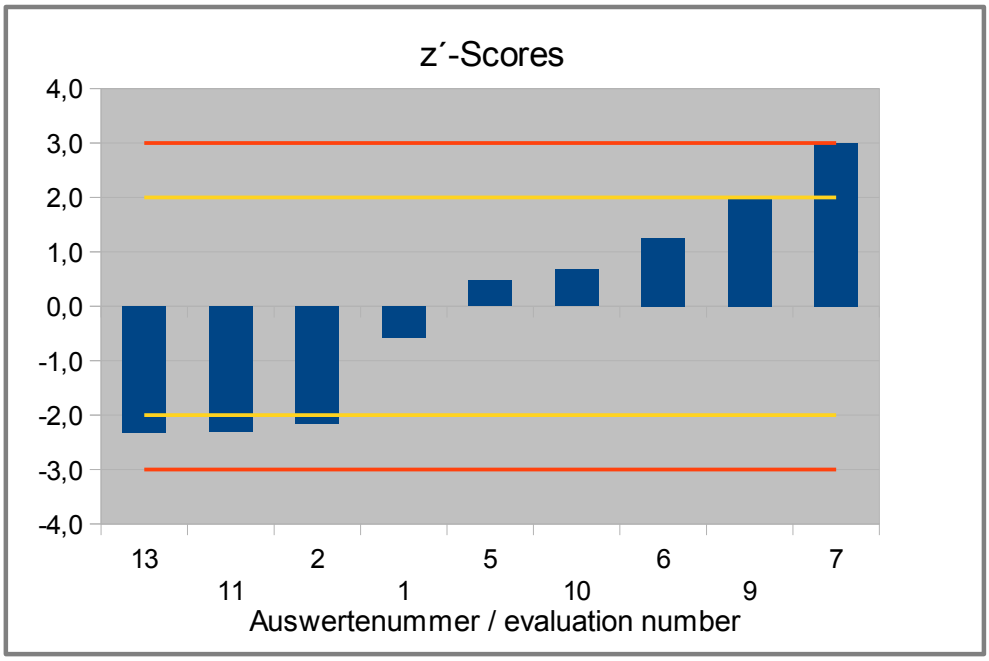

Abb. 9: Z-Scores E 953
Fig. 9: Z-Scores E 953

4.4 Lactitol (E966) in g/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	6
Number of outliers	
Mean	1,90
Median	1,98
Robust Mean (X)	**1,91**
Robust standard deviation (S*)	**0,217**
Number with 2 replicates	6
Repeatability SD (S_r)	0,109
Repeatability (CV_r)	5,78%
Reproducibility SD (S_R)	0,227
Reproducibility (CV_R)	12,0%
Target range:	
Target standard deviation σ_{pt}´	**0,185**
Target standard deviation (for Information)	0,0694
lower limit of target range	**1,54**
upper limit of target range	**2,28**
Quotient S/σ_{pt}´*	*1,2*
Standard uncertainty $U_{(Xpt)}$	*0,111*
Quotient $U_{(Xpt)}/\sigma_{pt}$´	*0,60*
Results in the target range	*5*
Percent in the target range	*83%*

Comments:

The target standard deviation was calculated using the target standard deviation according to ASU (precision experiment) and calculated as σ_{pt}´.

As additional information, the target standard deviation, calculated according to the Horwitz general model, was giv**en.**

The evaluation of the results shows an acceptable variability of results, in particular because the tests using different methods (HPAEC-PAD,HPLC-RI, L-RID, GC-FID)). The quotient S^*/σ_{pt}´ was below 2,0. The quotient $U(X_{pt})/\sigma_p$´ is above 0,3 at 0,60, but to accept because of the different methods.

The reproducibility coefficient of variation is low (12%). The robust standard deviation is in the range of established values for the standard deviation of the methods used. The reproducibility of the results is given.

83 % of the results are within the target range.

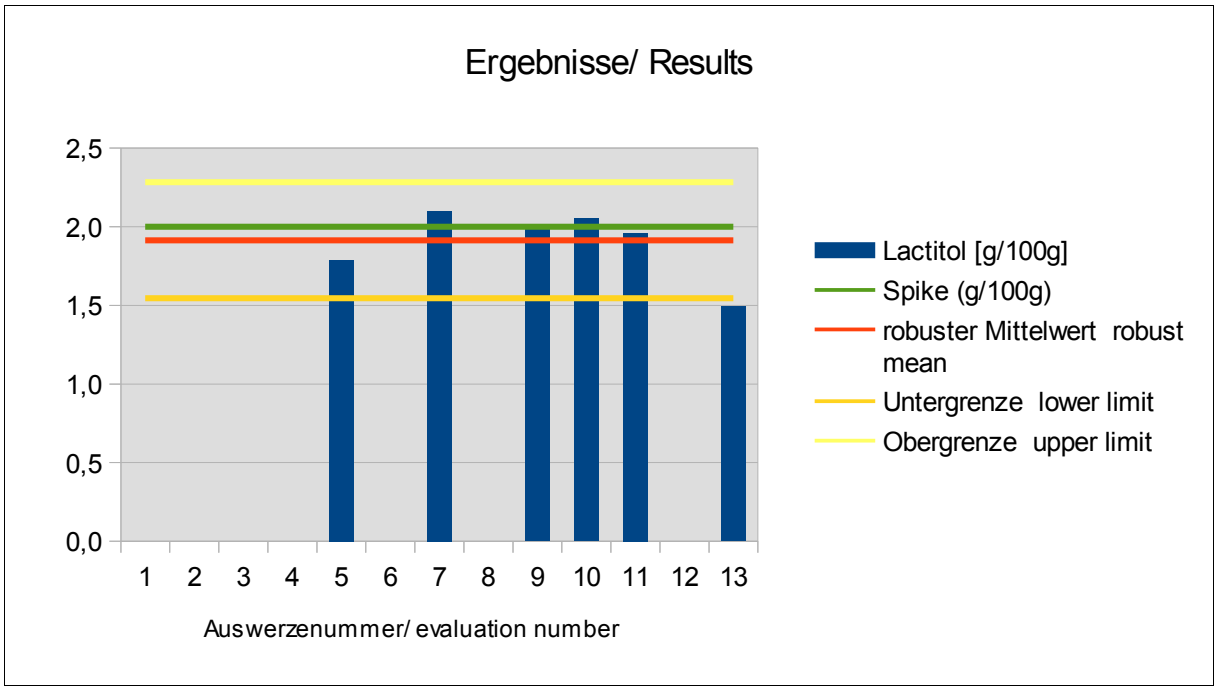

Abb. 10: Ergebnisse E 966
Fig. 10: Results E 966

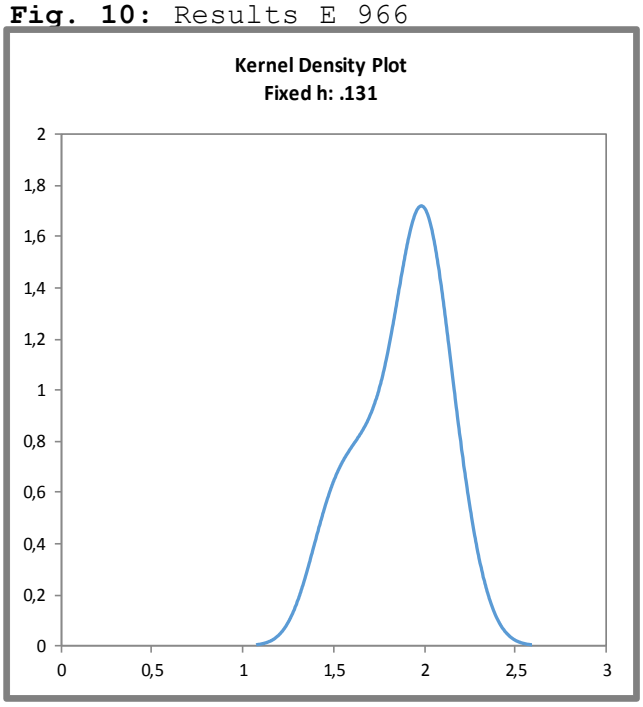

Abb. 11: Kerndichte Plot der Ergebnisse E 966 mit h = Zielstandardabweichung

Fig. 11: Kernel density plot of the E 966 results with h = target standard deviation

Comments:
The kernel density plot shows a symmetrical distribution of results. The
shoulder at 1,5 g/100g indicates a result outside the target range.

Ergebnisse der teilnehmenden Institute:
Results of Participants:

Auswerte-nummer Evaluation number	Lactitol [g/100g]	Abweichung [g/100g] Deviation [g/100g]	z´-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1					
2					
3					
4					
5	1,79	-0,124	-0,671	-1,78	
6					
7	2,10	0,186	1,01	2,7	
8					
9	2,00	0,0811	0,439	1,17	
10	2,05	0,136	0,74	1,96	
11	1,96	0,0461	0,25	0,66	
12					
13	1,49	-0,424	-2,3	-6,1	

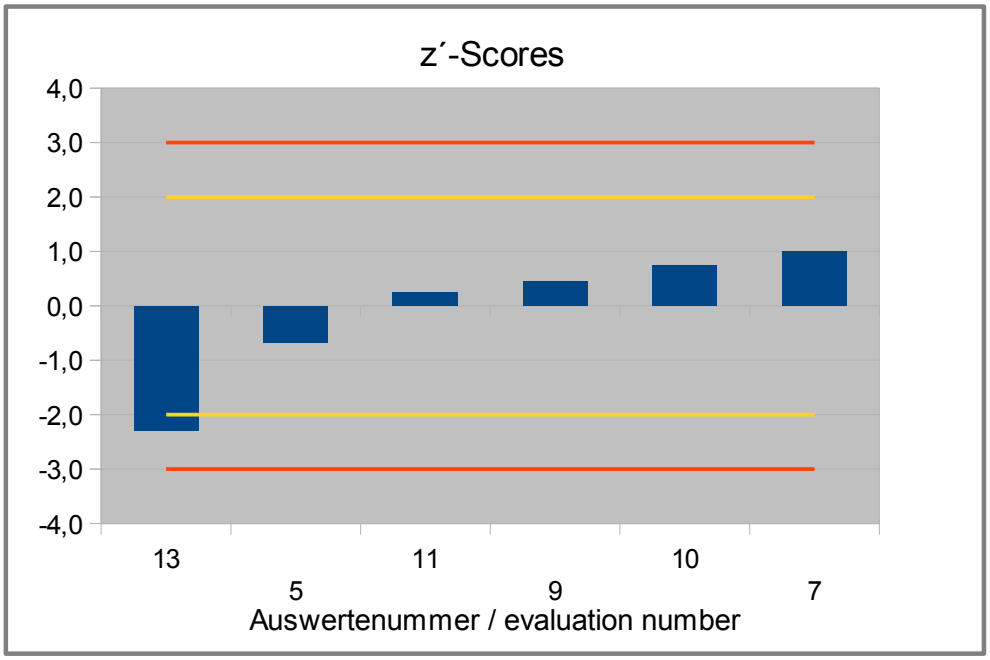

Abb. 12: Z-Scores E 966
Fig. 12: Z-Scores E 966

4.5 Xylitol (E 967) in g/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	13
Number of outliers	
Mean	2,10
Median	2,06
Robust Mean (X)	**2,10**
Robust standard deviation (S*)	**0,304**
Number with 2 replicates	13
Repeatability SD (S_r)	0,0715
Repeatability (CV_r)	3,42%
Reproducibility SD (S_R)	0,297
Reproducibility (CV_R)	14,2%
Target range:	
Target standard deviation σ_{pt}´	**0,130**
Target standard deviation (for Information)	0,0751
lower limit of target range	**1,84**
upper limit of target range	**2,36**
Quotient S/σ_{pt}´*	*2,3*
Standard uncertainty $U_{(Xpt)}$	*0,106*
Quotient $U_{(Xpt)}/\sigma_{pt}$´	*0,81*
Results in the target range	*9*
Percent in the target range	*69%*

Comments:

The target standard deviation was calculated using the target standard deviation according to ASU (precision experiment) and calculated as σ_{pt}´.

As additional information, the target standard deviation, calculated according to the Horwitz general model, was giv**en.**

The evaluation of the results shows an acceptable variability of results, in particular because the tests using different methods (HPAEC-PAD,HPLC-RI, L-RID, GC-FID)). The quotient S^x/σ_{pt}´was slightly above 2,0. The quotient $U(X_{pt})/\sigma_p$´ is above 0,3 at 0,81, but to accept because of the different methods.

The reproducibility coefficient of variation is low (14%). The robust standard deviation is in the range of established values for the standard deviation of the methods used. The reproducibility of the results is given.

69 % of the results are within the target range.

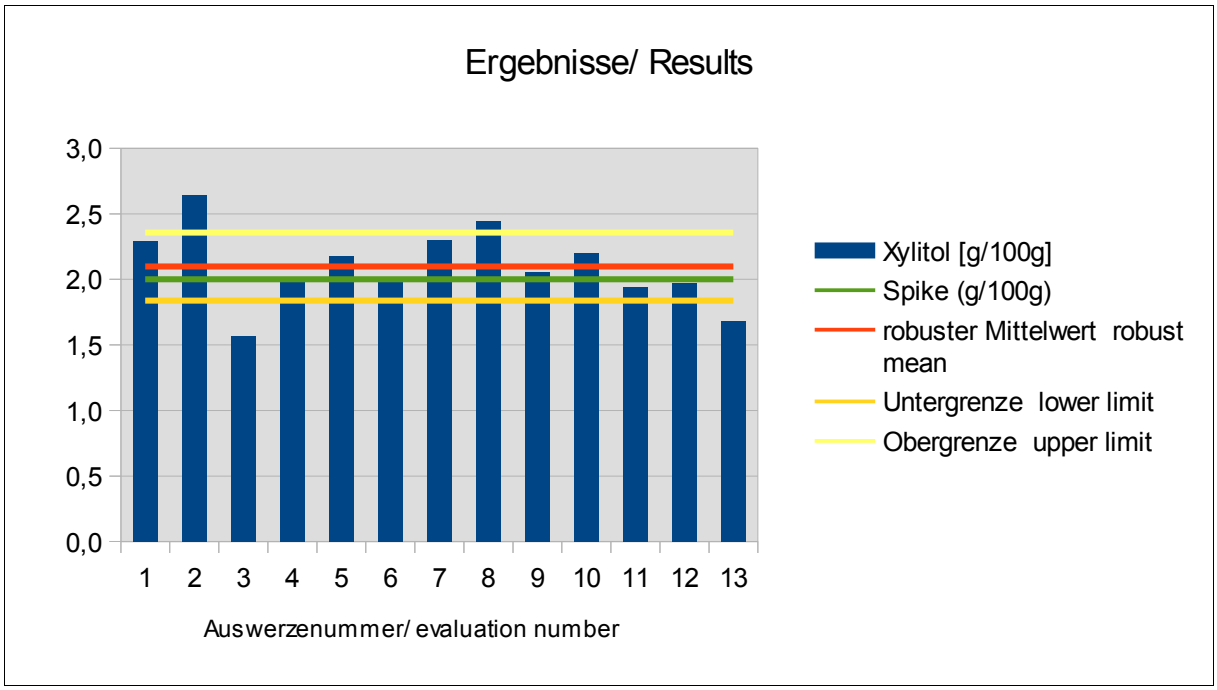

Abb. 13: Ergebnisse E 967
Fig. 13: Results E 967

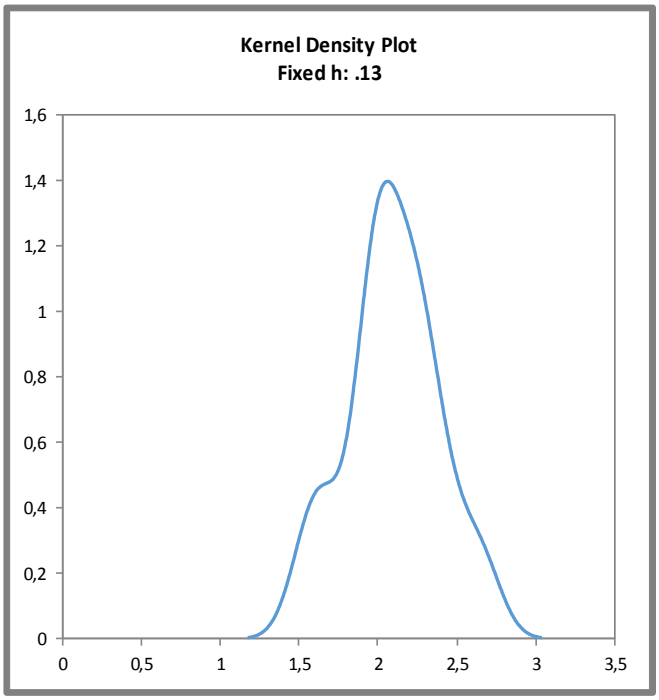

Abb. 14: Kerndichte Plot der Ergebnisse E 967 mit h = Zielstandardabweichung
Fig. 14: Kernel density plot of the E 967 results with h = target standard deviation

Comments:
The kernel density plot shows a symmetrical distribution of results. The
shoulder at 1,6 g/100g indicates results outside the target range.

Ergebnisse der teilnehmenden Institute:
Results of Participants:

Auswerte-nummer Evaluation number	Xylitol [g/100g]	Abweichung [g/100g] Deviation [g/100g]	z´-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	2,29	0,192	1,5	2,6	
2	2,65*	0,547	4,2	7,3	
3	1,57	-0,533	-4,1	-7,1	
4	2,02	-0,0780	-0,60	-1,0	
5	2,18	0,0820	0,63	1,1	
6	2,00	-0,098	-0,76	-1,3	
7	2,30	0,202	1,6	2,7	
8	2,44	0,345	2,7	4,6	
9	2,06	-0,0430	-0,33	-0,57	
10	2,20	0,102	0,79	1,4	
11	1,94	-0,158	-1,2	-2,1	
12	1,97	-0,128	-0,99	-1,7	
13	1,68	-0,418	-3,2	-5,6	

* Mean calculated by DLA

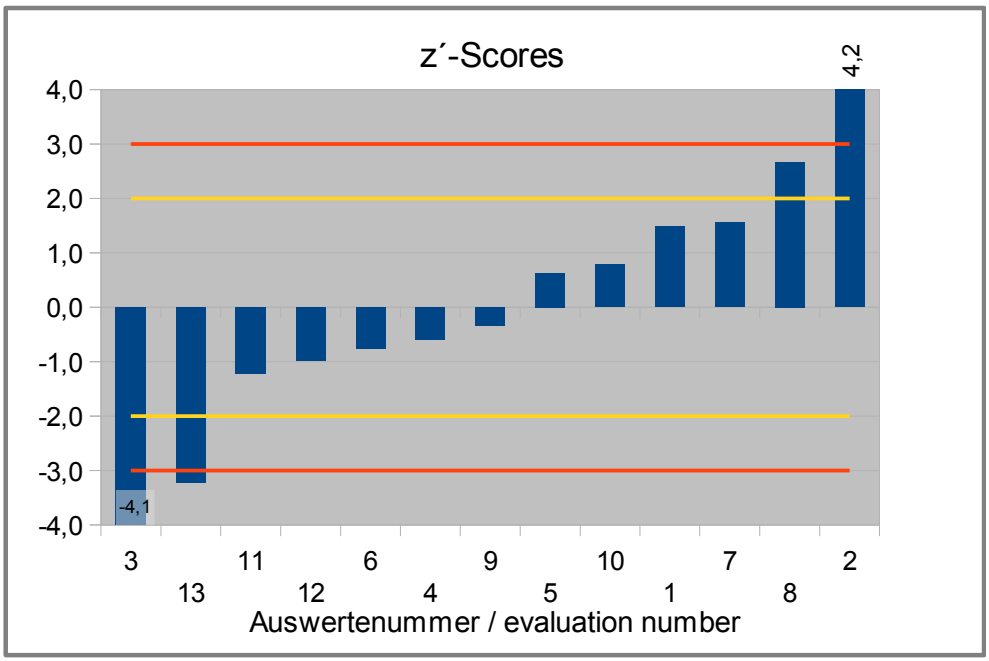

Abb. 15: Z-Scores E 967
Fig. 15: Z-Scores E 967

4.6 Erythritol (E 968) in g/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	10
Number of outliers	
Mean	3,11
Median	3,22
Robust Mean (X)	**3,18**
Robust standard deviation (S*)	**0,532**
Number with 2 replicates	10
Repeatability SD (S_r)	0,0866
Repeatability (CV_r)	2,79%
Reproducibility SD (S_R)	0,643
Reproducibility (CV_R)	20,7%
Target range:	
Target standard deviation σ_{pt}´	**0,236**
lower limit of target range	**2,71**
upper limit of target range	**3,66**
Quotient S/σ_{pt}´*	*2,3*
Standard uncertainty $U_{(Xpt)}$	*0,210*
Quotient $U_{(Xpt)}/\sigma_{pt}$´	*0,89*
Results in the target range	*6*
Percent in the target range	*60%*

Comments:

The target standard deviation was calculated using the Horwitz general model and calculated as σ_{pt}´.

The evaluation of the results shows an acceptable variability of results, in particular because the tests using different methods (HPAEC-PAD,HPLC-RI, L-RID, GC-FID)). The quotient S^x/σ_{pt}´was slightly above 2,0. The quotient $U(X_{pt})/\sigma_p$´ is above 0,3 at 0,89, but to accept because of the different methods.

The reproducibility coefficient of variation is acceptable (21%). The robust standard deviation is in the range of established values for the standard deviation of the methods used. The reproducibility of the results is given.

60 % of the results are within the target range.

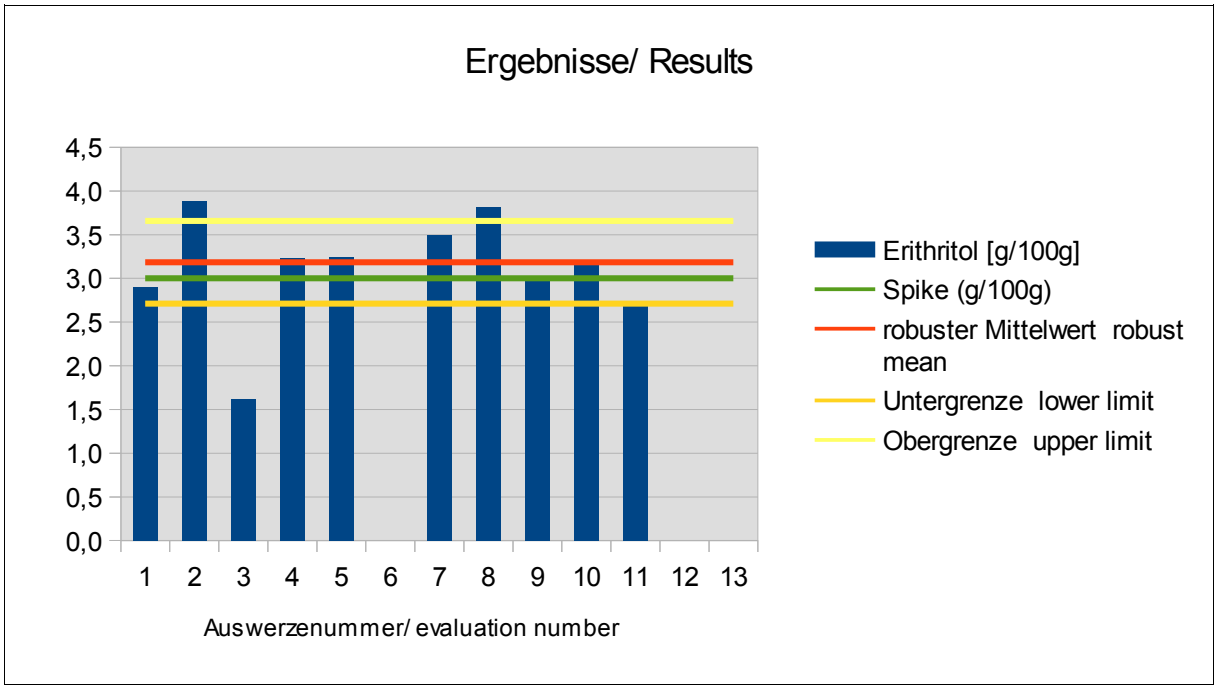

Abb. 16: Ergebnisse E 968
Fig. 16: Results E 968

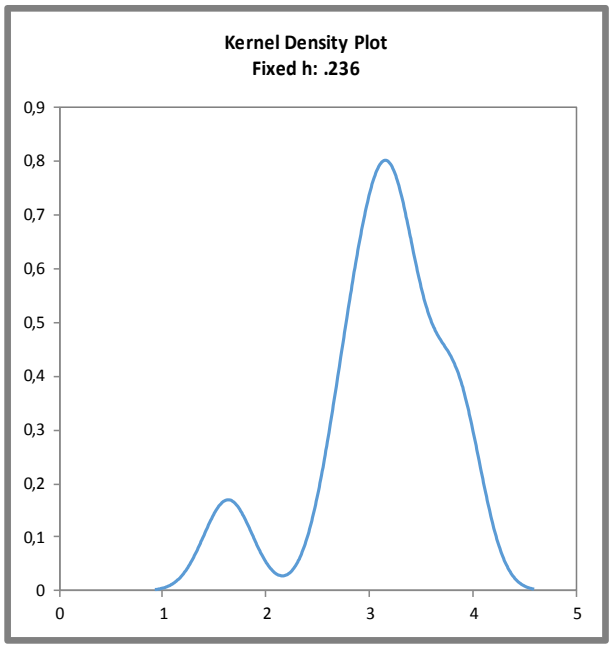

Abb. 14: Kerndichte Plot der Ergebnisse E 968 mit h = Zielstandardabweichung
Fig. 14: Kernel density plot of the E 968 results with h = target standard deviation

Comments:
The kernel density plot shows a symmetrical distribution of results. The
shoulder at 3,8 g/100g and the second peak at 1,6 g/100g indicates results outside the target range.

Ergebnisse der teilnehmenden Institute:
Results of Participants:

Auswerte-nummer Evaluation number	Erithritol [g/100g]	Abweichung [g/100g] Deviation [g/100g]	z´-Score (σ_{pt})	Hinweis Remark
1	2,90	-0,285	-1,2	
2	3,88*	0,695	2,9	
3	1,63	-1,56	-6,6	
4	3,23	0,0455	0,19	
5	3,24	0,0555	0,24	
6				
7	3,50	0,315	1,3	
8	3,82	0,634	2,7	
9	2,99	-0,195	-0,82	
10	3,20	0,0155	0,066	
11	2,70	-0,485	-2,1	
12				
13				

* Mean calculated by DLA

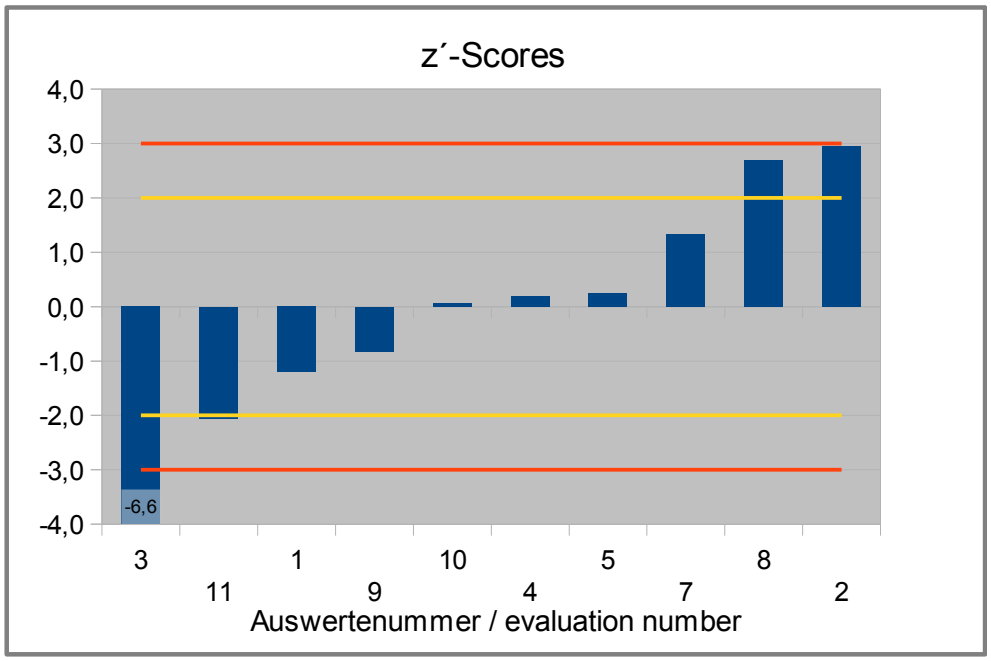

Abb. 18: Z-Scores E 968
Fig. 18: Z-Scores E 968

4.7 Maltitol (E 965) in g/100g

Under "Further sugar alcohols" the PT samples for maltitol (E 965) were examined by 3 participants.

Results:

Auswertenummer/ Evaluation number	Maltitol (g/100g)	Ergebnis Probe A/ Result sample A	Ergebnis Probe B/ Result sample B	Bestimmungs-grenze/ limit of determination
7	not detectable	<0,5	<0,5	0,5
9	not detectable	< 0,2	< 0,2	0,2
13	not detectable	0	0	0,1

Comment:

No maltitol was added to the samples. All three participants confirmed that no maltitol was detectable above the respective limit of determination.

The determination limits were in the range of 0,1 – 0,5 g/100g.

5. Documentation

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

5.1 Primary data

5.1.1.1 Sorbitol ((E 420)

Teil-nehmer	Proben Nr. A	Proben Nr.B	Datum der Analyse	Ergebnis (Mittel)	Ergebnis A	Ergebnis B	Bestim-mungs-grenze	Inkl. WF	Wiederfin-dungsrate [%]
Partici-pant	Sample No.A	Sample No. B	Date of analysis	Result (Mean)	Result A	Result B	Limit of determina-tion	Incl. RR	Recovery rate [%]
				g/100g	g/100g	g/100g	g/100g		in %
1	20	50	18.09.18	2,21	2,12	2,28		no	
2	8	62	02.08.-13.08.	yes*	2,98	2,67	0,005	no	
3	23	47	09.18.	2,48	2,45	2,51		yes	-
4	24	46							
5	14	56	11.9.	3,09	3,07	3,11	0,05	no	
6	19	51	12.09.18	3,2	3,2	3,2	0,4	no	104
7	26	44	01.08.18	3,3	3,3	3,3	0,5	no	-
8	12	58	23.08.18	3,6425	3,5465	3,7368	1	no	
9	34	36	20.01.00	3,09	3,08	3,1	0,2	no	99
10	5	65	23.08.18	3,15	3,2	3,1	0,5	no	
11	32	38	08.08.18	2,82	2,87	2,76		no	
12	10	60	27.07.18	2,845	2,835	2,855	0,04	no	
13	6	64	01.08.	2,45	2,38	2,48	0,1	no	

** Mean calculated by DLA*

5.1.1.2 Mannitol (E 421)

Teil-nehmer	Proben Nr. A	Proben Nr.B	Datum der Analyse	Ergebnis (Mittel)	Ergebnis A	Ergebnis B	Bestim-mungs-grenze	Inkl. WF	Wiederfin-dungsrate [%]
Partici-pant	Sample No.A	Sample No. B	Date of analysis	Result (Mean)	Result A	Result B	Limit of determina-tion	Incl. RR	Recovery rate [%]
				g/100g	g/100g	g/100g	g/100g		in %
1	20	50	18.09.18	1,78	1,78	1,77		no	
2	8	62	02.08.-13.08.	yes*	1,86	1,78	0,005	no	
3	23	47	09.18.	1,105	1,05	1,16		yes	-
4	24	46							
5	14	56	11.9.	2,15	2,14	2,16	0,05	no	
6	19	51	12.09.18	1,9	1,9	1,8	0,4	no	97
7	26	44	01.08.18	2,2	2,2	2,2	0,5	no	-
8	12	58	23.08.18	2,1736	2,2155	2,1316	1	no	
9	34	36	20.01.00	n.a.	n.a.	n.a.			
10	5	65	23.08.18	2,4	2,4	2,4	0,5	no	
11	32	38	08.08.18	1,99	1,98	1,99		no	
12	10	60	27.07.18	1,7575	1,735	1,78	0,04	no	
13	6	64	01.08.	1,61	1,58	1,63	0,1	no	

** Mean calculated by DLA*

5.1.1.3 Isomalt (E 953)

Teil-nehmer	Proben Nr. A	Proben Nr.B	Datum der Analyse	Ergebnis (Mittel)	Ergebnis A	Ergebnis B	Bestim-mungsgrenze	Inkl. WF	Wiederfin-dungsrate [%]
Partici-pant	Sample No.A	Sample No. B	Date of analysis	Result (Mean)	Result A	Result B	Limit of determina-tion	Incl. RR	Recovery rate [%]
				g/100g	g/100g	g/100g	g/100g		in %
1	20	50	18.09.18	2,67	2,77	2,56		no	
2	8	62	02.08.-13.08.	yes*	2,29	1,96	0,005	no	
3	23	47	-	n.a.	n.a.	n.a.	-	-	-
4	24	46							
5	14	56	11.9.	3,03	2,91	3,15	0,05	no	
6	19	51	12.09.18	3,3	3,6	3	0,4	no	89
7	26	44	01.08.18	3,9	3,7	4	0,5	no	-
8	12	58							
9	34	36	20.01.00	3,545	3,34	3,75	0,2	no	95
10	5	65	23.08.18	3,1	3,2	3	0,5	no	
11	32	38	08.08.18	2,08	2,3	1,86		no	
12	10	60							
13	6	64	01.08.	2,07	1,91	2,16	0,1	no	

** Mean calculated by DLA*

5.1.1.4 Xylitol (E 967)

Teil-nehmer	Proben Nr. A	Proben Nr.B	Datum der Analyse	Ergebnis (Mittel)	Ergebnis A	Ergebnis B	Bestim-mungsgren-ze	Inkl. WF	Wiederfin-dungsrate [%]
Parti-ci-pant	Sample No.A	Sample No. B	Date of analysis	Result (Mean)	Result A	Result B	Limit of determina-tion	Incl. RR	Recovery rate [%]
				g/100g	g/100g	g/100g	g/100g		in %
1	20	50	18.09.18	2,29	2,33	2,1		no	
2	8	62	02.08.-13.08.	yes*	2,68	2,61	0,005	no	
3	23	47	09.18.	1,565	1,56	1,57		yes	-
4	24	46	22.08.18	2,02	1,99	2,05		no	
5	14	56	11.9.	2,18	2,17	2,2	0,05	no	
6	19	51	12.09.18	2	2	2	0,4	no	99
7	26	44	01.08.18	2,3	2,3	2,3	0,5	no	-
8	12	58	23.08.18	2,4435	2,4073	2,4797	1	no	
9	34	36	20.01.00	2,055	2,02	2,09	0,2	no	98
10	5	65	23.08.18	2,2	2,2	2,2	0,5	no	
11	32	38	08.08.18	1,94	1,82	2,05		no	
12	10	60	27.07.18	1,97	1,985	1,955	0,04	no	
13	6	64	01.08.	1,68	1,62	1,7	0,1	no	

** Mean calculated by DLA*

5.1.1.5 Erythritol (E 968)

Teil-nehmer	Proben Nr. A	Proben Nr.B	Datum der Analyse	Ergebnis (Mittel)	Ergebnis A	Ergebnis B	Bestim-mungsgrenze	Inkl. WF	Wiederfin-dungsrate [%]
Partici-pant	Sample No.A	Sample No. B	Date of analysis	Result (Mean)	Result A	Result B	Limit of determina-tion	Incl. RR	Recovery rate [%]
				g/100g	g/100g	g/100g	g/100g		in %
1	20	50	18.09.18	2,9	2,83	2,97		no	
2	8	62	02.08.-13.08.	yes*	3,9	3,86	0,005	no	
3	23	47	09.18.	1,625	1,6	1,65		yes	-
4	24	46	22.08.18	3,23	3,16	3,3		no	
5	14	56	11.9.	3,24	3,26	3,23	0,05	no	
6	19	51							
7	26	44	01.08.18	3,5	3,4	3,5	0,5	no	-
8	12	58	23.08.18	3,8188	3,9008	3,7368	1	no	
9	34	36	20.01.00	2,99	3,03	2,95	0,1	no	xx
10	5	65	23.08.18	3,2	3,1	3,3	0,5	no	
11	32	38	08.08.18	2,7	2,62	2,77		no	
12	10	60							
13	6	64							

** Mean calculated by DLA*

5.1.1.6 Further

Paramter	Teil-nehmer	Proben Nr. A	Proben Nr.B	Datum der Analyse	Ergebnis (Mittel)	Ergebnis A	Ergebnis B	Bestim-mungsgrenze	Inkl. WF	Wiederfin-dungsrate [%]
Paramter	Partici-pant	Sample No.A	Sample No. B	Date of analysis	Result (Mean)	Result A	Result B	Limit of determina-tion	Incl. RR	Recovery rate [%]
					g/100g	g/100g	g/100g	g/100g		in %
Sorbitol + Xylitol combined	1	20	50	18.09.18	4,49	4,47	4,52		no	
E 966 Lactitol	4	24	46							
E 966 Lactitol	5	14	56	11.9.	1,79	1,71	1,88	0,05	no	
E 966 Lactitol	7	26	44	01.08.18	2,1	2,1	2	0,5	no	-
E 965 Maltitol	7	26	44	01.08.18	<0,5	<0,5	<0,5	0,5	no	-
E 996 Lactitol	9	34	36	20.01.00	1,995	1,98	2,01	0,2	no	96
E 965 Maltitol	9	34	36	20.01.00	< 0,2	< 0,2	< 0,2	0,2	no	90
E 966 Lactitol	10	5	65	23.08.18	2,05	2,2	1,9	0,5	no	
E 966 Lactitol	11	32	38	08.08.18	1,96	2,01	1,91		no	
E 966 Lactitol	13	6	64	01.08.	1,49	1,53	1,47	0,1	no	
E 965 Maltitol	13	6	64	01.08.	0	0	0	0,1	no	

5.1.2 Analytical methods

5.1.2.1 Sorbitol (E 420)

Teilnehmer	Methodenbeschreibung	Probenvorbereitung	Messmethode	Kalibrierung und Referenzmaterial	Wiederfindung wurde mit gleicher Matrix bestimmt	Methode akkreditiert	Sonstige Hinweise
Participant	**Method description**	**Sample preparation**	**Measuring method**	**Calibration and reference material**	**Recovery** with same matrix	**Method** accredited	**Further remarks**
1	ASU L 00.00-59		HPLC-RI			yes	
2			HPIC-PAD			yes	
3	MSZ EN 15086:2006	-	-	standard addition	no	yes	-
4							
5	HPAEC-PAD	aqueous extraction, membrane filtration		external standard		yes	
6	ASU L 00.00-59 (2008-12)	no carrez preciptation			yes	yes	
7	HPLC/RI - internal method PNTA0177			external calib. curve and intern RM	no	yes	
8	LC-RID	Homogenization with water		0,05-1 g/100ml	no	yes	
9	L00.00-59	Extractionnwith warm water, filtration, Carrez clarification	HPLC-RI, Cation exchanger column, 70°C	external calibration, VWR	no	yes	
10	in house method			Sigma	no	no	
11	GC-FID	TMS-derivatisation				no	
12		Extraction with water	HPLC		no	no	
13	SLMB Nr. 501.2:1976-01, upd. 2008 mod.,	Standard; no Carrez clarification; Ext. 60 °C; Silylation using BSTFA; GC				yes	

5.1.2.2 Mannitol (E 421)

Teilnehmer	**Methodenbeschrei-bung**	**Probenvorbereitung**	**Messmethode**	**Kalibrierung und Referenzmaterial**	**Wiederfindung** w urde mit gleicher Matrix bestimmt	**Methode** akkreditiert	**Sonstige Hinweise**
Participant	**Method description**	**Sample preparation**	**Measuring method**	**Calibration and reference mate-rial**	**Recovery** w ith same matrix	**Method** accredited	**Further remarks**
1	2008-12		HPLC-RI			yes	
2			HPIC-PAD			yes	
3	MSZ EN 15086:2006	-	-	external stan-dard	no	yes	-
4							
5	HPAEC-PAD	aqueous extraction, membrane filtration		external stan-dard		yes	
6	ASU L 00.00-59 (2008-12)	no carrez precipta-tion			yes	yes	
7	HPLC/RI - internal method PNTA0177			external calib. curve and intern RM	no	yes	
8	LC-RID	Homogenization with water		0,05-1 g/100ml	no	yes	
9							
10	in house method			Sigma	no	no	
11	GC-FID	TMS-derivatisation				no	
12		Extraction with wa-ter	HPLC		no	no	
13	SLMB Nr. 501.2:1976-01, upd. 2008 mod.,	Standard; no Carrez clarification; Ext. 60 °C; Silylation using BSTFA; GC				yes	

5.1.2.3 Isomalt (E 953)

Teilnehmer	**Methodenbeschrei-bung**	**Probenvorbereitung**	**Messmethode**	**Kalibrie-rung und Referenzma-terial**	**Wiederfindung** w urde mit gleicher Matrix bestimmt	**Methode** ak-kreditiert	**Sonstige Hinweise**
Participant	**Method description**	**Sample preparation**	**Measuring method**	**Calibration and refe-rence mate-rial**	**Recovery** w ith same matrix	**Method** ac-credited	**Further remarks**
1	Column: MN Ca2+		HPLC-RI			yes	
2			HPIC-PAD			yes	
3	-	-	-	-	-	-	-
4							
5	HPAEC-PAD	aqueous extraction, membrane filtration		external standard		yes	
6	ASU L 00.00-59 (2008-12)	no carrez precipta-tion			yes	yes	
7	HPLC/RI - internal method PNTA0177			external calib. cur-ve and in-tern RM	no	yes	
8							
9	L00.00-59			Alcum	no	yes	
10	in house method			Sigma	no	no	
11	GC-FID	TMS-derivatisation				no	
12							
13	SLMB Nr. 501.2:1976-01, upd. 2008 mod.,	Standard; no Carrez clarification; Ext. 60 °C; Silylation using BSTFA; GC				yes	

5.1.2.4 Xylitol (E 967)

Teilnehmer	**Methodenbeschreibung**	**Probenvorbereitung**	**Messmethode**	**Kalibrierung und Referenz-material**	**Wiederfindung** w urde mit gleicher Ma-trix bestimmt	**Methode** akkreditiert	**Sonstige Hinweise**
Participant	**Method description**	**Sample preparation**	**Measuring method**	**Calibration and reference material**	**Recovery** w ith same matrix	**Method** accredited	**Further remarks**
1	s.o.		HPLC-RI			yes	
2			HPIC-PAD			yes	
3	MSZ EN 15086:2006	-	-	standard addi-tion	no	yes	-
4	ASU L 00.00-59 mod.	Preparation with MeOH	HPLC/RI	extern, yes	no	yes	
5	HPAEC-PAD	aqueous extraction, membrane filtration		external stan-dard		yes	
6	ASU L 00.00-59 (2008-12)	no carrez precipta-tion			yes	yes	
7	HPLC/RI - internal method PNTA0177			external ca-lib. curve and intern RM	no	yes	
8	LC-RID	Homogenization with water		0,05-1 g/100ml	no	yes	
9	L00.00-59			Sigma Aldrich	no	yes	
10	in house method			Sigma	no	no	
11	GC-FID	TMS-derivatisation				no	
12		Extraction with wa-ter	HPLC		no	no	
13	SLMB Nr. 501.2:1976-01, upd. 2008 mod.,	Standard; no Carrez clarification; Ext. 60 °C; Silylation using BSTFA; GC				yes	

5.1.2.5 Erythritol (E 968)

Teilnehmer	**Methodenbeschreibung**	**Probenvorbereitung**	**Messmethode**	**Kalibrierung und Referenzmaterial**	**Wiederfindung** wurde mit gleicher Matrix bestimmt	**Methode** akkreditiert	**Sonstige Hinweise**
Participant	**Method description**	**Sample preparation**	**Measuring method**	**Calibration and reference material**	**Recovery** with same matrix	**Method** accredited	**Further remarks**
1	see above		HPLC-RI			yes	
2			HPIC-PAD			yes	
3	MSZ EN 15086:2006	-	-	standard addition	no	yes	-
4	ASU L 00.00-59 mod.	Preparation with MeOH	HPLC/RI	extern, yes	no	yes	
5	HPAEC-PAD	aqueous extraction, membrane filtration		external standard		no	
6							
7	HPLC/RI - internal method PNTA0177			external calib. curve and intern RM	no	yes	
8	LC-RID	Homogenization with water		0,05-1 g/100ml	no	yes	
9	L00.00-59 (in addition)			Serva	no	yes	
10	in house method			Sigma	no	no	
11	GC-FID	TMS-derivatisation				no	
12							
13							

5.1.2.6 Further

Teilnehmer	Parameter	Methodenbeschreibung	Probenvorbereitung	Messmethode	Kalibrierung und Referenzmaterial	Wiederfindung wurde mit gleicher Matrix bestimmt	Methode akkreditiert	Sonstige Hinweise
Participant	**Parameter**	**Method description**	**Sample preparation**	**Measuring method**	**Calibration and reference material**	**Recovery** with same matrix	**Method** accredited	**Further remarks**
1	Sorbitol + Xylitol combined	see above		HPLC-RI			yes	Xylitol and sorbitol overlap. The individual results are given according to the content according to the standard, divided by 2
4	E 966 Lactitol							
5	E 966 Lactitol	HPAEC-PAD	aqueous extraction, membrane filtration		external standard		yes	
7	E 966 Lactitol	HPLC/RI - internal method PNTA0177			external calib. curve and intern RM	no	yes	
7	E965 Maltitol	HPLC/RI - internal method PNTA0177			external calib. curve and intern RM	no	yes	
9	E 966 Lactitol	L00.00-59			Alfa Aesar	no	yes	
9	E965 Maltitol	L00.00-59			Sigma Aldrich	no	yes	
10	E 966 Lactitol	in house method			Sigma	no	no	
11	E 966 Lactitol	GC-FID	TMS-derivatisation				no	
13	E 966 Lactitol	SLMB Nr. 501.2:1976-01, Akt. 2008 mod.,	Standard; no Carrez clarification; Ext. 60 °C; Silylation using BSTFA; GC				yes	
13	E965 Maltitol	SLMB Nr. 501.2:1976-01, Akt. 2008 mod.,	Standard; no Carrez clarification; Ext. 60 °C; Silylation using BSTFA; GC				yes	

5.2 Homogeneity

5.2.1 Mixture homogeneity before bottling

Microtracer Homogeneity test
DLA 39-2018

Weight whole sample	1,000	kg
Microtracer	FSS-rot lake	
Particle size	75 – 300	µm
Weight per particle	2,0	µg
Addition of tracer	32,1	mg/kg

Results of analysis

Sample	Weight [g]	Particle number	Particles [mg/kg]
1	6,24	102	32,7
2	5,70	99	34,7
3	8,73	142	32,5
4	8,71	134	30,8
5	8,40	122	29,0
6	7,94	149	37,5
7	5,41	82	30,3
8	5,65	88	31,2
9	6,04	94	31,1
10	5,77	96	33,3

Poisson destribution		
Number of samples	10	
Degree of freedom	9	
mean	110,8	Particle
Standard deviation	8,40	Particle
χ^2 (CHI-Quadrat)	5,73	
Probability	**77**	%
Recovery rate	101	%

Normal distribution		
Number of samples	10	
Mean	32,3	mg/kg
Standard deviation	2,45	mg/kg
rel. Standard deviation	7,6	%
Horwitz Standard deviation	9,5	%
HorRat value	**0,8**	
Revocery rate	101	%

5.3 Information on the Proficiency Test (PT)

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

Information on the Proficiency Test (PT)

PT number	***DLA 39-2018***
PT name	***Sugar Alcohols (E420, E421, E953, E967, E968) in Plant Product***
*Sample matrix**	*Samples I + II:* ***Pudding powder***
Number of samples and sample amount	*2 identical samples I+ II, 10 g each.*
Storage	*Samples I + II cooled 2 - 10°C*
Intentional use	*Laboratory use only (quality control samples)*
Parameter	*quantitative:* ***Sugar Alcohols (E420, E421, E953, E967, E968, other)***
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 IIas 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***
Deadline	***the latest 21st September 2018***
Evaluation report	*The evaluation report is expected to be completed 6 weeks after deadline of result submission and sent as PDF file by e-mail.*
Coordinator and contact person of PT	*Dr. Gerhard Wichmann*

* 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

Teilnehmer/ Participant	Ort/ Town	Land/ Country
		Hungary
		Deutschland
		Italy
		Sweden
		Deutschland
		Deutschland
		Spain
		Deutschland
		Deutschland
		Deutschland
		Deutschland
		Spain
		Deutschland

[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 literature

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
6. Evaluation of analytical methods used for regulation of food and drugs; W. Horwitz; 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; Analyst, 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 estimates, 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 Messungen (2003); Quantifying Uncertainty in Analytical Measurement (1999)
14. GMP+ Feed Certification scheme, Module: Feed Safety Assurance,

chapter 5.7 Checking 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. ASU §64 LFGB: L 00.00-59; Bestimmung von Isomalt, Lactit, Maltit, Mannit, Sorbit und Xylit in Lebensmitteln; HPLC-Verfahren (2008)

17. ASU §64 LFGB: L 18.00-14; Bestimmung von D-Sorbit in Feinen Backwaren; Enzymatische Verfahren (1994)