DLA
Proficiency Tests

Evaluation Report
proficiency test

DLA 35/2019

Dietary Fiber and Inulin:

in Cereal 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* Alexandra Scharf MSc. (QM-Beauftragte / Quality Manager) *- gezeichnet / signed A. Scharf* Datum / Date: 10 July 2019
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 of the food matrix samples are customary spelt waffles. The basic composition of samples A and B was the same (see table 1). Additionally inulin was added to sample B.
After crushing and sieving (mesh 2,0 mm) of the spelt waffles, the basic mixture was homogenized.
Afterwards the spiked sample B was produced as follows:
Previously sieved inulin (mesh 400 µm) was added to an aliquot of the matrix and the mixture was homogenized. Subsequently, the basic mixture was again added in steps and homogenized in each case until the total quantity had been reached.

The samples A and B were portioned to approximately 40 g in metallized PET film bags and numbered chronologically.

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

<u>Table 1:</u> Composition of DLA-Samples

Ingredients	Probe A	Probe B

Spelt Waffles, organic
    Ingredients: Spelt (99,5%), salt
 	
    Nutrients per 100 g:
    Fat 3,2 g Carbohydrates 65 g, thereof sugar
    2,5 g, Protein 14 g
 	
- thereof fiber (10 %) **
	100 g/100g *
	
	10,0 g/100g **
		95,7 g/100g *
		
		9,57 g/100g **

Inulin	–	4,34 g/100g *

* Contents according to gravimetric mixture
** Contents according to label

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

2.1.1 Homogeneity

The **mixture homogeneity before bottling** was examined 8-fold by **microtracer analysis**. It is a standardized method that is part of the international GMP certification system for feed [14].
Before mixing dye coated iron particles of µm size are added to the sample and the number of particles is determined after homogenization in taken aliquots. The evaluation of the mixture homogeneity is based on the Poisson distribution using the chi-square test. A probability of ≥ 5 % is equivalent to a good homogeneous mixture and of ≥ 25% to an excellent mixture [14, 15].
The microtracer analysis of the present PT samples A and the spiking level sample showed a probability of 96% and 85%. Additionally particle number results were converted into concentrations, statistically evaluated according to normal distribution and compared to the standard deviation according to Horwitz. For the assessment HorRat values between 0,3 and 1,3 are to be accepted under repeat conditions (measurements within the laboratory) [17].
This gave a HorRat value of 0,73 and 0,94 respectively. 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 total dietary fiber with and without inulin it is approx. 1,6% and 3,2%. For inulin it is 11,5% (sample A without inulin addition) and 2,5% (sample B with inulin addition). Thus they were similar to the repeatability standard deviations of the official methods ((ASU L 00.00-18 / L 17.03-1 and ASU L 00.00-94, see. 3.6.2) (see Tab. 3) [18-21]. The repeatability standard deviations of the participants' results are given in the documentation in the statistic data (see 4.1 to 4.3).

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

Parameter	CV_r Sample A	CV_r Sample B
Total dietary fiber without inulin	3,21 %	3,24 %
Total dietary fiber incl. inulin	1,56 %	1,77 %
Inulin	11,5 %*	2,49 %

* Sample A without addition of inulin

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. 0,16 / 0,17 (22,5°C) The stability of the sample material was thus ensured during the investigation period under the specified storage conditions.

2.2 Sample shipment and information to the test

The portions of test materials sample A, and B were sent to every participating laboratory in the 8th week of 2019. The testing method was optional. The tests should be finished at 5h April 2019 the latest.

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

There are two <u>different</u> samples of waffles from spelt. Inulin was added to one of the two samples A or B. The fat content is < 10%.

The parameters total dietary fiber and inulin should be analysed in duplicate for each sample.

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

2.3 Submission of results

The participants submitted their results in standard forms, which have been handed out with the samples (by email).
For statistical evaluation, the final contents of the analytes were indicated as the mean of the duplicate determinations. The individual values of the double determinations were also used to calculate the repeatability and comparison standard deviation.
Queried and documented were the indicated results and details of the test methods like specificity, test kit manufacturer and hints about the procedure.
In case participants submitted several results for the same parameter obtained by different methods these results were evaluated with the same evaluation number with a letter as a suffix and indication of the related method.

All 10 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 evaluation is usually carried out starting from 7 results, in justified cases a valuation is also allowed from 5 results.
In the present evaluation statistic data for the parameters total dietary fibers with and without inulin was done with a minimum of 5 values, because the statistical characteristics were sufficient. It should be considered that the significance can be limited due to the low number of results.
Single results giving values outside the measuring range of the participating laboratory or given as „0" are not considered for statistical evaluation (e.g. results given as > 25 mg/kg and < 2,5 mg/kg, respectively) [3].

3.2 Robust standard deviation

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

3.3 Repeatability standard deviation

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

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

The relative repeatability standard deviation as a percentage of the mean

value is indicated as coefficient of variation CV_r in the table of statistical characteristics in the results section in case single results from participants are available.

3.4 Reproducibility standard deviation

The reproducibility standard deviation S_R represents a inter-laboratory estimate of the standard deviation for the determination of each parameter on the bases of (outlier free) individual participant results. It takes into account both the repeatability standard deviation S_r and the within-laboratory standard deviation S_s. Reproducibility standard deviations of PTs may differ from reproducibility standard deviations of ring trials, because the participating laboratories of a PT generally use different internal conditions and methods for determining the measured values.
In the present evaluation, the specification of the reproducibility standard deviation, therefore, does not refer to a specific method, but characterizes approximately the comparability of results between the laboratories, assumed the effect of homogeneity and stability of the sample are negligible.

In case single results from participants are available the calculation of the reproducibility standard deviation S_R is performed by: [3, 4].

The relative reproducibility standard deviation as a percentage of the mean value is given as the coefficient of variation CV_R in the statistical characteristics in the results section, provided that the individual results of the participants are available, and the meaning is explained in more detail under 3.9.

3.5 Exclusion of results and outliers

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

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

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

3.6 Target standard deviation (for proficiency assessment)

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

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

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

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

For the valuation of <u>all parameters</u> the target standard deviation from section 3.6.2 (precision experiment) was applied (German ASU §64 Methods: L 17.03-1, L 00.00-94; AOAC 2009.01).

<u>Additionally</u> the standard uncertainty for <u>inulin</u> was considered by valuating with 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 deviations (RSD_R) given in table 3 were obtained in precision experiments by the indicated methods.
The resulting target standard deviations σ_{pt}, which were identified there, were used to evaluate the results and to provide additional information for the statistical data.

<u>Table 3:</u> Relative repeatability standard deviations (RSD_r) and relative reproducibility standard deviation (RSD_R) according to selected evaluations of tests for precision and the resulting target standard deviation σ_{pt} [18-21]

Parameter	Matrix	Mean [g/100g]	RSD_r	RSD_R	σ_{pt}	Method / Literature
Total Dietary Fiber (enzymatic-gravi-metric)	Mixed rye bread	8,83	2,49 %	5,10 %	4,8 %[1]	ASU §64 L 17.03-1
Insoluble Dietary Fiber (enzymatic-gravi-metric)	Mixed rye bread	5,45	5,14 %	8,44 %	7,6 %	ASU §64 L 17.03-1
Soluble Dietary Fiber (enzymatic-gravi-metric)	Mixed rye bread	3,02	14,6 %	20,9 %	18,2 %	ASU §64 L 17.03-1
Inulin (enzymatic-photo-metric)	Finished flour baby food chocolate	16,7 0,61 5,95	1,86 % 4,92 % 2,35 %	3,60 % 8,20 % 4,03 %	3,4 %[1] 7,4 % 3,7 %	ASU §64 L 00.00-94
Total Dietary Fiber including Inulin (enzymatic-gravi-metric + liquid chromatography)	Whole grain bread Whole grain paste	11,6 12,7	12,3 % 4,47 %	18,0 % 11,3 %	15,8 %[1] 10,8 %	AOAC 2009.01

[1] *in the evaluation (s. section 4) used values*

For the determination of **total dietary fiber** the participants used the following methods:

The **enzymatic-gravimetric methods** ASU L 00.00-18 (ASU L 17.03-1) or AOAC 991.43 (Lee et al. 1992) and AOAC 985.29. In which lower results of inulin, oligosaccharides and resistant starch can appear [23, 24].

The **enzymatic-gravimetric method combined with liquid chromatography** AOAC 2009.01, which includes soluble and insoluble dietary fiber, resistant starch and low molecular weight dietary fibers like inulin and oligosaccharides [23, 24]. One participant used the method AOAC 2011.25.

For **inulin determination** the following methods were used in this PT:

The **enzymatic-photometric methods** ASU L 00.00-94 or AOAC 999.03 as well as the e**nzymatic method in combination with ion exchange chromatography (IEC)** AOAC 997.08. One participant reported the results of the liquid chromatography from method AOAC 2009.01.

3.6.3 Value by perception

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

In the present PT, the target standard deviation of 3.6.2 were considered suitable.

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

Legal requirements:
In the guideline to the Food Information Regulation, the permitted tolerances for the nutrition information is for fiber of ± 2 g for contents of <10 g / 100g and of ± 20% for contents of 10-40 g / 100g [22].

3.7 z-Score

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

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

The requirements for the analytical performance are generally considered

as fulfilled if

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

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

3.7.1 Warning and action signals

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

An error or cause analysis can be carried out by checking the analysis process including understanding and implementation of the measurement by the staff, details of the measurement procedure, calibration of equipment and composition of reagents, transmission or calculation errors, trueness and precision and use of reference material. If necessary appropriate corrective measures should be applied [3].

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

<u>Table 4:</u> Characteristics of the present PT (on blue-grey) in comparison to previous PTs since 2015 (SD = standard deviation, CV = coefficient of variation)

Parameter	Matrix	robust Mean [g/100g]	rob. SD (S*) [g/100g]	rel. SD (VK$_{S*}$) [%]	Quotient S*/σ$_{pt}$	DLA-report
Total Dietary Fiber	Cereal product	6,06	0,840	13,9	2,0	DLA 26/2015
Total Dietary Fiber	Cereal product	8,15	0,579	7,10	1,5	DLA 36/2016
Total Dietary Fiber	Cereal product	4,32	1,06	24,5	2,2	DLA 35/2017
Total Dietary Fiber with Inulin	Cereal product	11,1 14,3	1,00 1,15	8,98 8,01	0,57 0,51	DLA 35/2019 *Samples ****
Total Dietary Fiber without Inulin	Cereal product	8,82 8,50	0,795 0,803	9,02 9,44	1,9 2,0	DLA 35/2019 *Samples ****
Inulin	Cereal product	3,68	0,690	18,8	1,9	DLA 26/2015
Inulin	Cereal product	3,14	0,524	16,7	1,8	DLA 36/2016
Inulin	Cereal product	3,35	0,468	14,0	2,2	DLA 35/2017
Inulin	Cereal product	0,730** 4,73	0,332 0,628	41,0 13,3	2,6* 2,2*	DLA 35/2019 *Samples ****

* with target standard deviation σ$_{pt}$'
** Median
*** results: 1st line Sample A, 2nd line Sample B

3.8 z'-Score

The z'-score can be used for the valuation of the results of the participants, in cases the standard uncertainty has to be considered (s. 3.11). The z'-score represents the relation of the deviation of the result (x_i) of the participant from the respective consensus value (X) to the square root of quadrat sum of the target standard deviation (σ_{pt}) and the standard uncertainty (Ux_{pt}) [3].

The calculation is performed by:

$$z'_i = \frac{x_i - x_{pt}}{\sqrt{\sigma^2_{pt} + u^2_{(x_{pt})}}}$$

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 ≤ z' ≤ 2 .

For warning and action signals see 3.7.1.

3.9 Reproducibility coefficient of variation (CV)

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

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

In contrast to the standard deviation as a measure of the absolute varia-bility 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 evalu-ation of the participating laboratories possibly can not be done [3].

3.10 Quotient S*/σpt

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

3.11 Standard uncertainty and traceability

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

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

If $U_{(Xpt)}$ ≤ 0,3 σpt 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} − 2σ_{pt}) or (X_{pt} − 2σ_{pt}') *
upper limit of target range   (X_{pt} + 2σ_{pt}) or (X_{pt} + 2σ_{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	Parameter	Abweichung	z-Score	z-Score	Hinweis
Evaluation number	[Einheit / Unit]	Deviation	σ_{pt}	(Info)	Remark

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

4.1 Total Dietary Fiber without Inulin in g/100g

4.1.1 Sample A

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	6
Number of outliers	–
Mean	8,49
Median	8,90
Robust Mean (X_{pt})	**8,82**
Robust standard deviation (S*)	**0,795**
Number with 2 replicates	4
Repeatability SD (S_r)	0,283
Repeatability (CV_r)	3,14%
Reproducibility SD (S_R)	0,545
Reproducibility (CV_R)	6,05%
Target range:	
Target standard deviation σ_{pt}	**0,422**
Target standard deviation (for Information)	0,254
lower limit of target range	**7,98**
upper limit of target range	**9,67**
Quotient S*/σ_{pt}	*1,9*
Standard uncertainty $U_{(Xpt)}$	*0,406*
Results in the target range	*5*
Percent in the target range	*83%*

Comments to the statistical characteristics:

The target standard deviation was calculated using data from a precision experiment (ASU §64 L 17.03-1) (3.6.2). For information the target stand-ard deviation using the general model of Horwitz is given (s. 3.6.1).

The evaluation showed a normal variability of results. The quotient S*/σ_{pt} was below 2,0.
The robust standard deviation is comparable to those of prior PTs (see 3.6.3). The comparability of results is given.
The repeatability and reproducibility standard deviation are in the range of established values of the applied methods (see 3.6.2).

83% of the results were in the target range.

Abb. / Fig. 1: Ergebnisse Gesamt-Ballaststoffe ohne Inulin Probe A **/** Results total dietary fiber without inulin sample A

Comment:
No kernel density was done due to the number of <8 results.

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer / Evaluation number	Gesamt-Ballaststoffe ohne Inulin / Total dietary fiber without inulin [g/100g]	Abweichung [g/100g] / Deviation [g/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis / Remark
1	9,40	0,579	1,4	2,3	
2	9,20	0,379	0,90	1,5	
3	5,62	−3,201	−7,6	−12,6	insoluble dietary fiber
4	8,60	−0,221	−0,52	−0,87	
5	9,50	0,679	1,6	2,7	
6					
7	8,60	−0,221	−0,52	−0,87	
8					
9					
10					

Methods: enzymatic-gravimetric methods (ASU L 00.00-18, ASU L 17.03-1, or AOAC 991.43, AOAC 985.29)

Abb. / Fig. 2: z-Scores Gesamt-Ballaststoffe ohne Inulin Probe A / Total dietary fiber without inulin sample A

4.1.2 Sample B

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	6
Number of outliers	–
Mean	8,24
Median	8,51
Robust Mean (X_{pt})	**8,50**
Robust standard deviation (S*)	**0,803**
Number with 2 replicates	4
Repeatability SD (S_r)	0,275
Repeatability (CV_r)	3,11%
Reproducibility SD (S_R)	0,461
Reproducibility (CV_R)	5,20%
Target range:	
Target standard deviation σ_{pt}	**0,407**
Target standard deviation (for Information)	0,246
lower limit of target range	**7,69**
upper limit of target range	**9,32**
Quotient S*/σ_{pt}	*2,0*
Standard uncertainty $U(X_{pt})$	*0,410*
Results in the target range	*5*
Percent in the target range	*83%*

Comments to the statistical characteristics:

The target standard deviation was calculated using data from a precision experiment (ASU §64 L 17.03-1) (3.6.2). For information the target standard deviation using the general model of Horwitz is given (s. 3.6.1).

The evaluation showed a normal variability of results. The quotient S*/σ_{pt} was below 2,0.
The robust standard deviation is comparable to those of prior PTs (see 3.6.3). The comparability of results is given.
The repeatability and reproducibility standard deviation are in the range of established values of the applied methods (see 3.6.2).

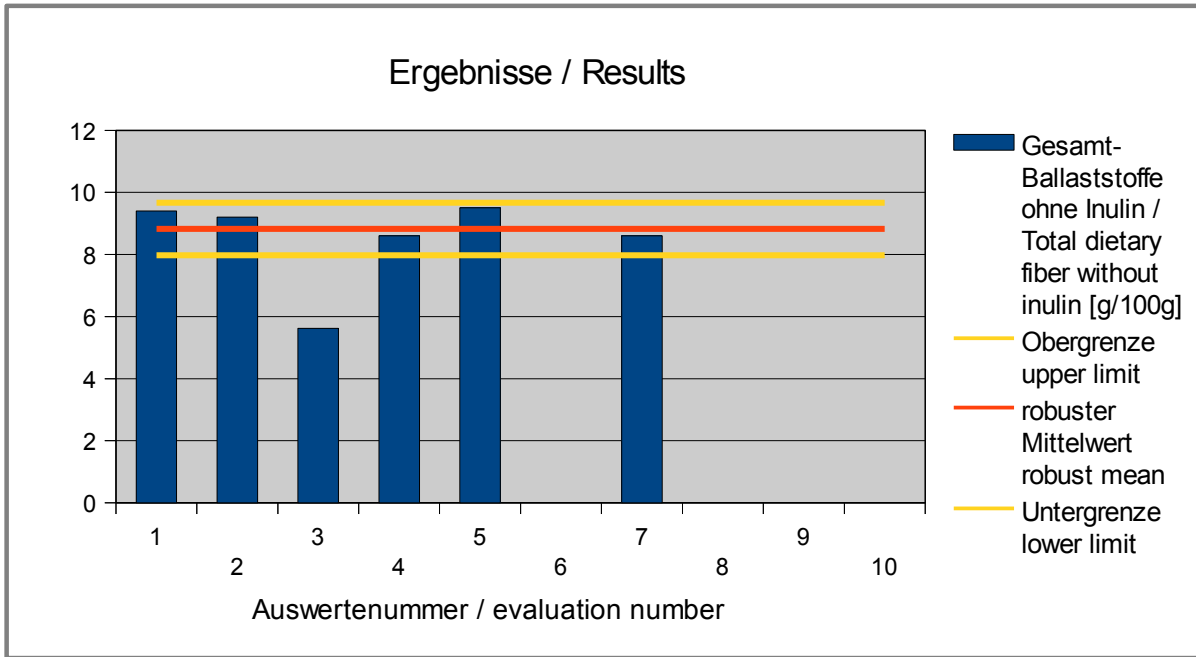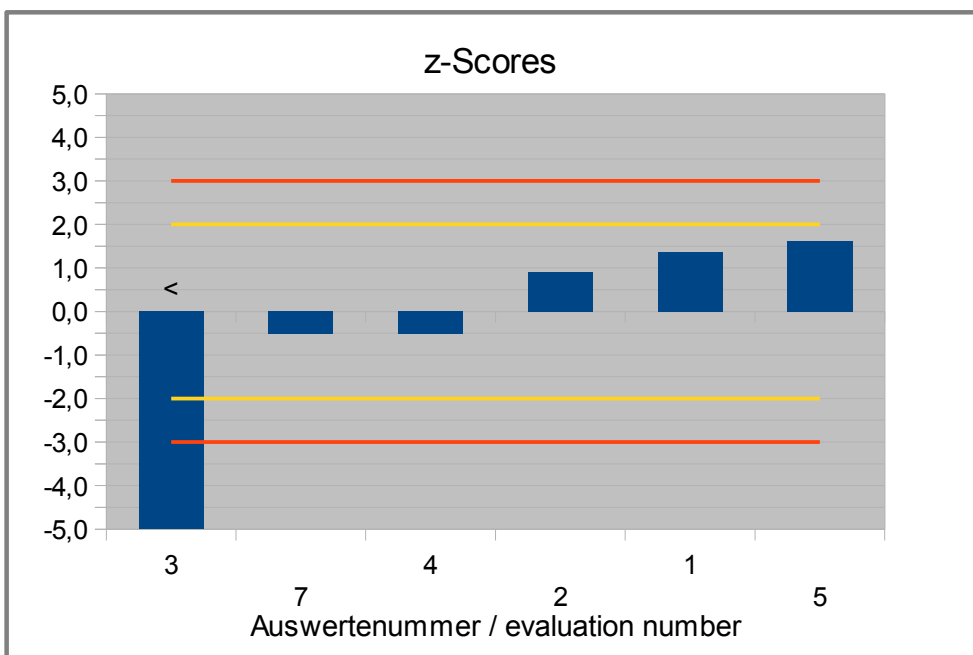83% of the results were in the target range.

Abb. / Fig. 3: Ergebnisse Gesamt-Ballaststoffe ohne Inulin Probe B **/** Results total dietary fiber without inulin sample B

Comment:
No kernel density was done due to the number of <8 results..

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer / Evaluation number	Gesamt-Ballaststoffe ohne Inulin / Total dietary fiber without inulin [g/100g]	Abweichung [g/100g] / Deviation [g/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis / Remark
1	9,10	0,597	1,5	2,4	
2	8,30	−0,203	−0,50	−0,82	
3	5,74	−2,763	−6,8	−11	insoluble dietary fiber
4	8,40	−0,103	−0,25	−0,42	
5	8,62	0,117	0,29	0,47	
6					
7	9,30	0,797	2,0	3,2	
8					
9					
10					

Methods: enzymatic-gravimetric methods (ASU L 00.00-18, ASU L 17.03-1, or AOAC 991.43, AOAC 985.29)

Abb. / Fig. 4: z-Scores Gesamt-Ballaststoffe ohne Inulin Probe B / Total dietary fiber without inulin sample B

4.2 Total Dietary Fiber with Inulin in g/100g

4.2.1 Sample A

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	5
Number of outliers	–
Mean	10,7
Median	11,2
Robust Mean (X_{pt})	**11,1**
Robust standard deviation (S*)	**1,00**
Number with 2 replicates	4
Repeatability SD (S_r)	0,173
Repeatability (CV_r)	1,65%
Reproducibility SD (S_R)	1,84
Reproducibility (CV_R)	17,6%
Target range:	
Target standard deviation σ_{pt}	**1,75**
Target standard deviation (for Information)	0,309
lower limit of target range	**7,60**
upper limit of target range	**14,6**
Quotient S*/σ_{pt}	*0,57*
Standard uncertainty U$_{(Xpt)}$	*0,557*
Results in the target range	*5*
Percent in the target range	*100%*

Comments to the statistical characteristics:

The target standard deviation was calculated using data from a precision experiment (AOAC 2009.01) (3.6.2). For information the target standard deviation using the general model of Horwitz is given (s. 3.6.1).

The evaluation showed a low variability of results. The quotient S*/σ_{pt} was below 1,0.
The robust standard deviation is comparable to those of prior PTs (see 3.6.3). The comparability of results is given.
The repeatability and reproducibility standard deviation are in the range of established values of the applied methods (see 3.6.2).
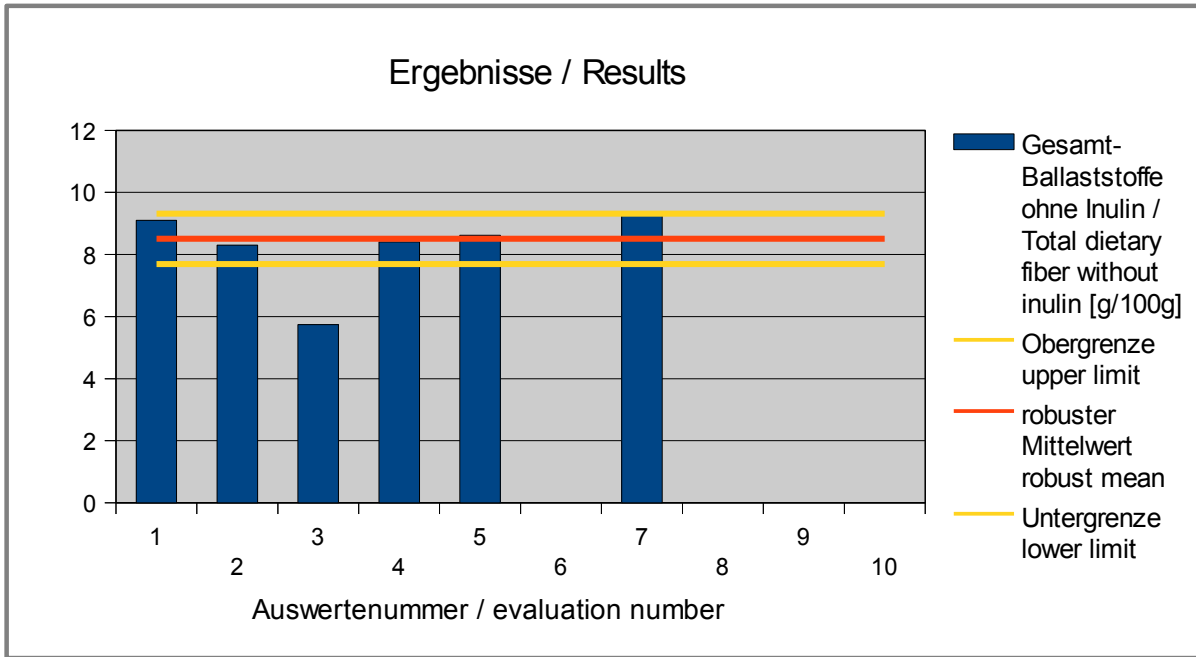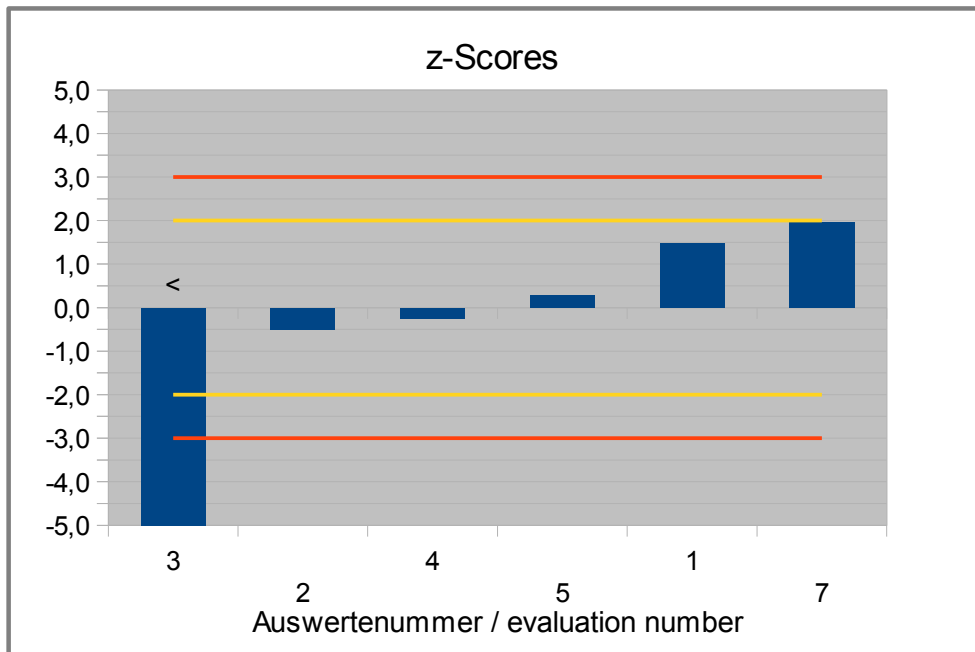
All results were in the target range.

Abb. / Fig. 5: Ergebnisse Gesamt-Ballaststoffe inkl. Inulin Probe A **/** Results total dietary fiber incl. inulin sample A

Comment:
No kernel density was done due to the number of <8 results.

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer / Evaluation number	Gesamt-Ballaststoffe inkl. Inulin / Total dietary fiber incl. inulin [g/100g]	Abweichung [g/100g] / Deviation [g/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis / Remark
1					
2	11,7	0,60	0,34	1,9	
3					
4					
5					
6					
7a	11,8	0,70	0,4	2,3	
7b	11,2	0,10	0,06	0,32	
8	7,77	−3,33	−1,9	−10,8	
9	11,2	0,09	0,05	0,29	
10					

Methods: enzymatic-gravimetric methods combined with Liquidchromatography (AOAC 2009.01, AOAC 2011.25)

Abb. / Fig. 6: z-Scores Gesamt-Ballaststoffe inkl. Inulin Probe A / Total dietary fiber incl. inulin sample A

4.2.2 Sample B

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	5
Number of outliers	0
Mean	14,3
Median	14,4
Robust Mean (X_{pt})	**14,3**
Robust standard deviation (S*)	**1,15**
Number with 2 replicates	4
Repeatability SD (S_r)	0,254
Repeatability (CV_r)	1,76%
Reproducibility SD (S_R)	1,16
Reproducibility (CV_R)	8,07%
Target range:	
Target standard deviation σ_{pt}	**2,26**
Target standard deviation (for Information)	0,384
lower limit of target range	**9,82**
upper limit of target range	**18,9**
Quotient S*/σ_{pt}	*0,51*
Standard uncertainty $U_{(Xpt)}$	*0,642*
Results in the target range	*5*
Percent in the target range	*100%*

Comments to the statistical characteristics:

The target standard deviation was calculated using data from a precision experiment (AOAC 2009.01) (3.6.2). For information the target standard deviation using the general model of Horwitz is given (s. 3.6.1).

The evaluation showed a low variability of results. The quotient S*/σ_{pt} was below 1,0.
The robust standard deviation is comparable to those of prior PTs (see 3.6.3). The comparability of results is given.
The repeatability and reproducibility standard deviation are in the range of established values of the applied methods (see 3.6.2).
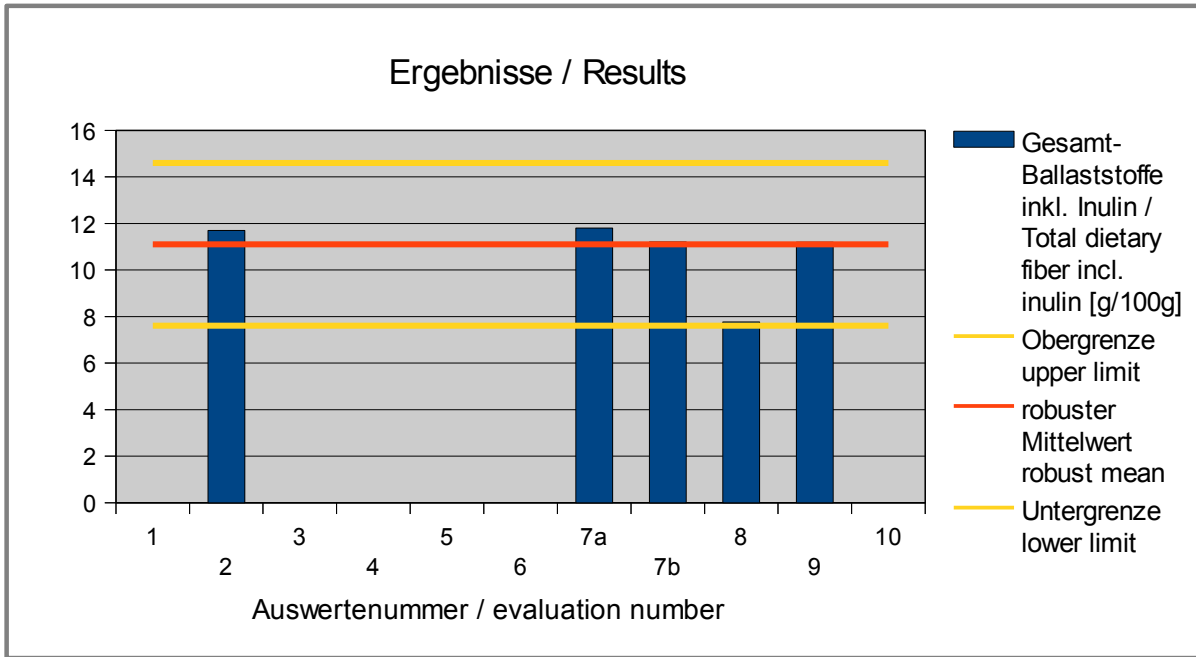
All results were in the target range.

Abb. / Fig. 7: Ergebnisse Gesamt-Ballaststoffe inkl. Inulin Probe B **/** Results total diatary fiber incl. inulin sample B

Comment:
No kernel density was done due to the number of <8 results.

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Gesamt-Ballaststoffe inkl. Inulin / Total dietary fiber incl. inulin [g/100g]	Abweichung [g/100g] Deviation [g/100g]	z-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1					
2	14,0	−0,35	−0,15	−0,90	
3					
4					
5					
6					
7a	15,3	0,95	0,42	2,5	
7b	14,4	0,05	0,02	0,14	
8	12,8	−1,53	−0,67	−4,0	
9	15,2	0,86	0,38	2,2	
10					

Methods: enzymatic-gravimetric methods combined with Liquidchromatography (AOAC 2009.01, AOAC 2011.25)
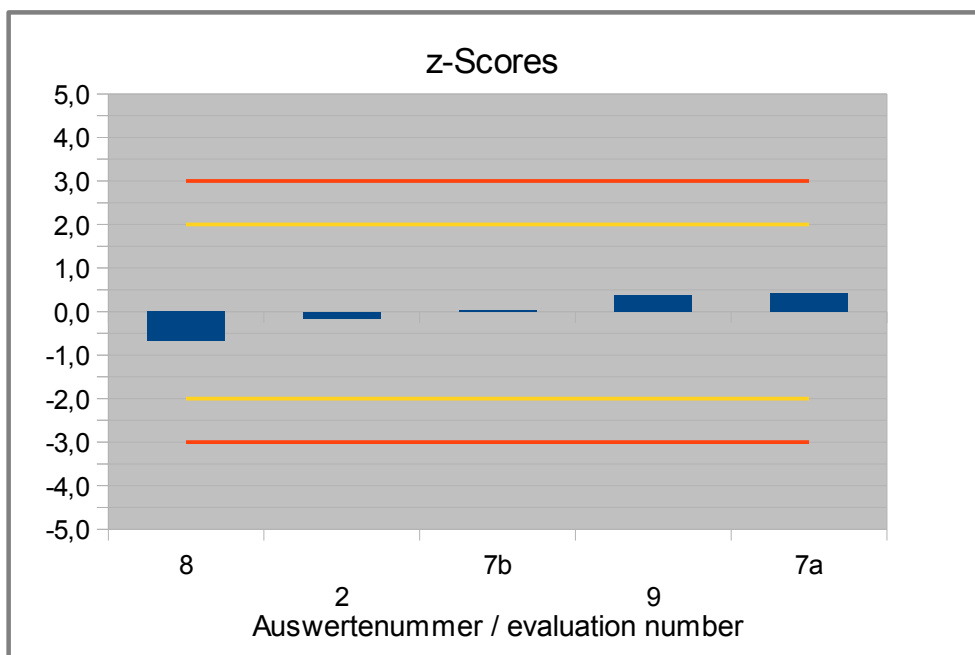
Abb. / Fig. 8:   z-Scores Gesamt-Ballaststoffe inkl. Inulin Probe B / Total dietary fiber incl. inulin sample B

4.3 Inulin in g/100g

4.3.1 Sample A

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	11
Number of outliers	–
Mean	0,860
Robust Mean	0,810
Median (X_{pt})	**0,730**
Robust standard deviation (S*)	**0,332**
Number with 2 replicates	10
Repeatability SD (S_r)	0,0840
Repeatability (CV_r)	9,84%
Reproducibility SD (S_R)	0,433
Reproducibility (CV_R)	50,8%
Target range:	
Target standard deviation σ_{pt}'	**0,127**
Target standard deviation (for Information)	0,0306
lower limit of target range	**0,475**
upper limit of target range	**0,985**
Quotient S*/σ_{pt}'	2,6
Standard uncertainty $U_{(Xpt)}$	0,125
Results in the target range	7
Percent in the target range	64%

Comments to the statistical characteristics:

The assigned value was the median (see 3.1).

The target standard deviation was calculated using data from a precision experiment (ASU §64 L 00.00-94)(3.6.2). For information the target standard deviation using the general model of Horwitz is given (s. 3.6.1).

The evaluation showed an increased variability of results, with a quotient S*/σ_{pt} of > 10,0. Therefore the evaluation of all methods was done by z'-score considering the standard uncertainty. The quotient S*/σ_{pt}' was then at 2,6.

No inulin was added to sample A, the measured levels are thus due to the natural contents of the matrix spelt. The repeatability and reproducibility standard deviation are above the range of established values of the applied methods (see 3.6.2).
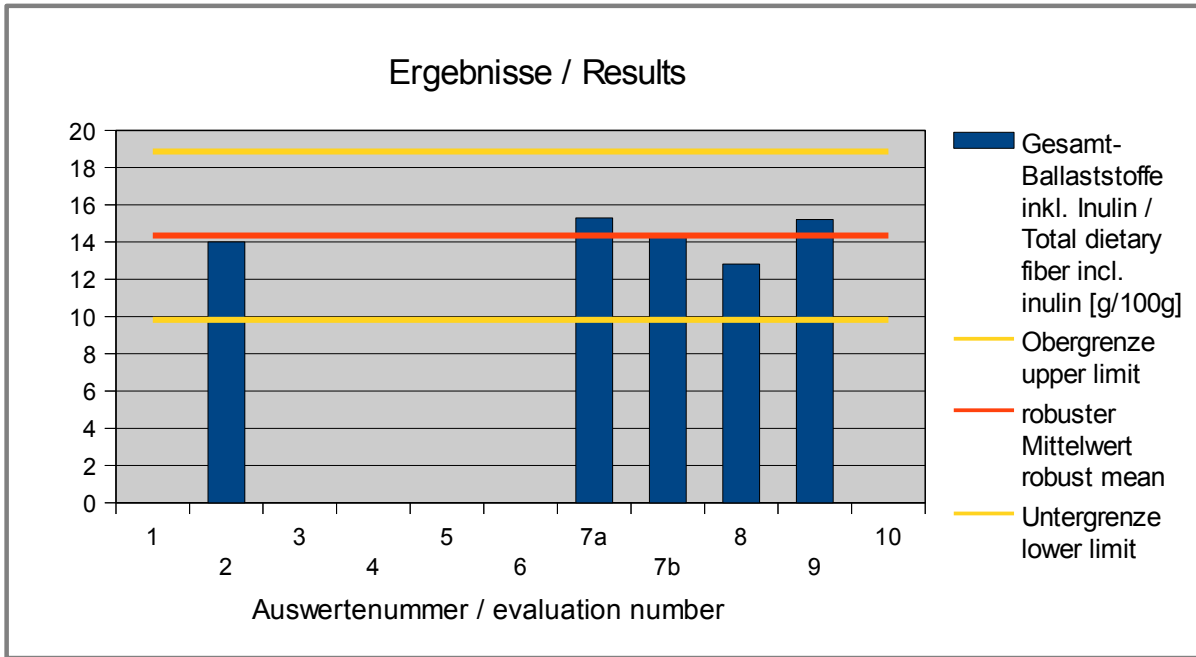
64% of the results were in the target range.

Abb. / Fig. 9: Ergebnisse Inulin Probe A **/** Results inulin sample A

Abb. / Fig. 10:
Kerndichte-Schätzung der Ergebnisse
(mit h = 0,75 x σ_{pt} von X_{pt})

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

Comment:
The kernel density shows almost a symmetrical distribution of results with a shoulder at approx. 1,0 g/100g and two smaller peaks at approx. 1,3 g/100g and 1,8 g/100g, due to results out of the target range.

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Inulin [g/100g]	Abweichung [g/100g] Deviation [g/100g]	z'-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	1,28	0,550	4,3	18	
2	0,900	0,170	1,3	5,6	
3	0,565	−0,165	−1,3	−5,4	
4	1,04	0,310	2,4	10	
5	0,390	−0,340	−2,7	−11	
6	0,620	−0,110	−0,86	−3,6	
7a	0,730	0,000	0,00	0,00	
7b	0,730	0,000	0,00	0,00	
8	1,85	1,120	8,8	37	
9	0,720	−0,010	−0,08	−0,33	
10	0,630	−0,100	−0,79	−3,3	

.
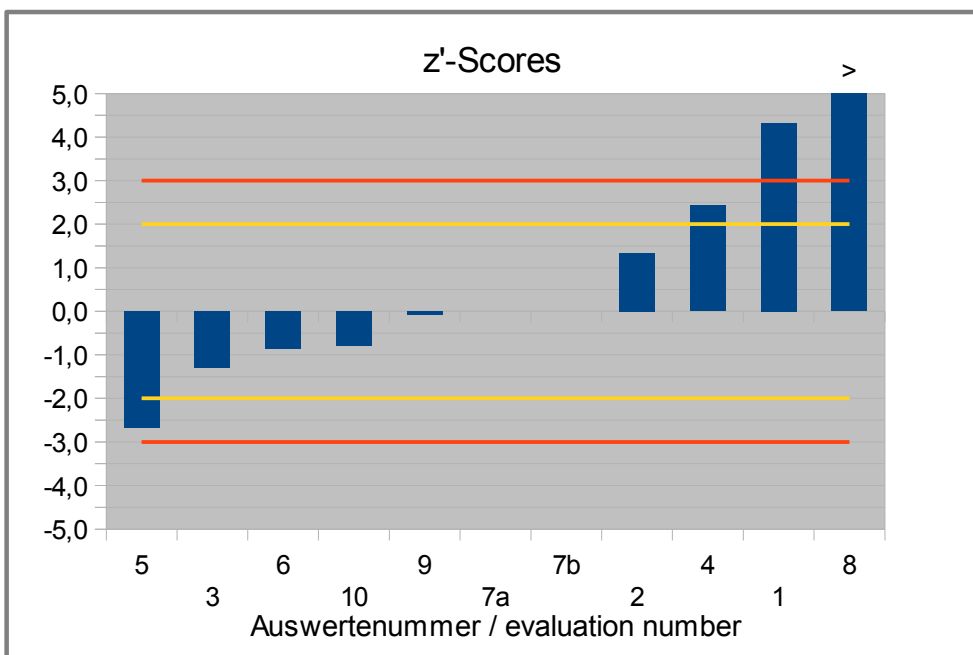
Abb. / Fig. 11: z'-Scores Inulin Probe A / inulin sample A

4.3.2 Sample B

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	11
Number of outliers	–
Mean	5,03
Robust Mean	4,65
Median (X_{pt})	**4,73**
Robust standard deviation (S*)	**0,628**
Number with 2 replicates	10
Repeatability SD (S_r)	0,118
Repeatability (CV_r)	2,34%
Reproducibility SD (S_R)	1,28
Reproducibility (CV_R)	25,4%
Target range:	
Target standard deviation $\sigma_{pt'}$	**0,285**
Target standard deviation (for Information)	0,150
lower limit of target range	**4,16**
upper limit of target range	**5,30**
Quotient S*/$\sigma_{pt'}$	*2,2*
Standard uncertainty $U_{(X_{pt})}$	*0,237*
Results in the target range	*8*
Percent in the target range	*73%*

Comments to the statistical characteristics:

The assigned value was the median (see 3.1).

The target standard deviation was calculated using data from a precision experiment (ASU §64 L 00.00-94)(3.6.2). For information the target standard deviation using the general model of Horwitz is given (s. 3.6.1).

The evaluation showed an increased variability of results, with a quotient S*/σ_{pt} of 4,0. Therefore the evaluation was done by z'-score considering the standard uncertainty. The quotient S*/$\sigma_{pt'}$ was then at 2,2. The robust standard deviation is comparable to those of prior PTs (see 3.6.3). The comparability of results is given.

The repeatability standard deviation is in the range of established values of the applied methods while the reproducibility standard deviation is increased (see 3.6.2).

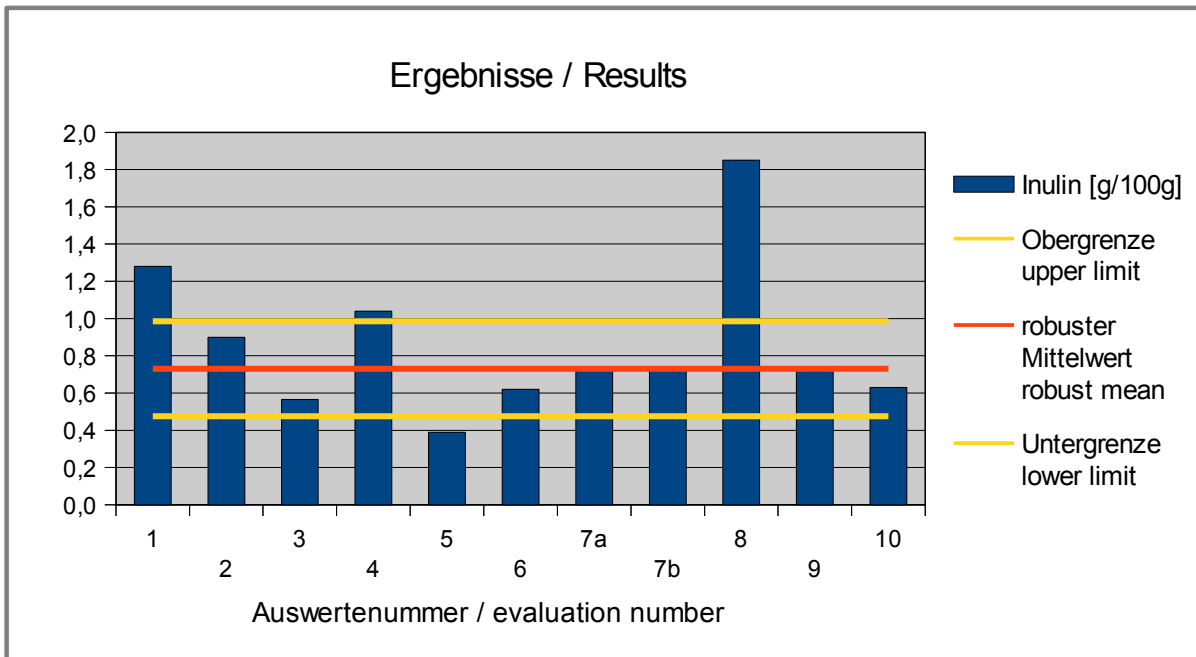73% of the results were in the target range.
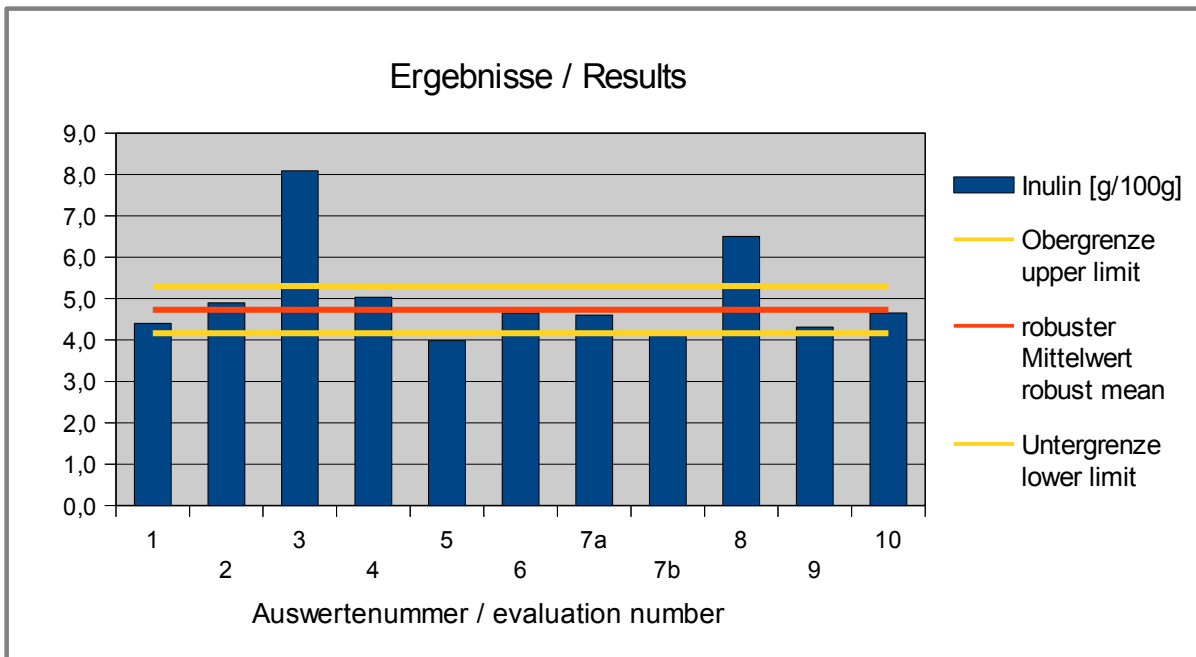
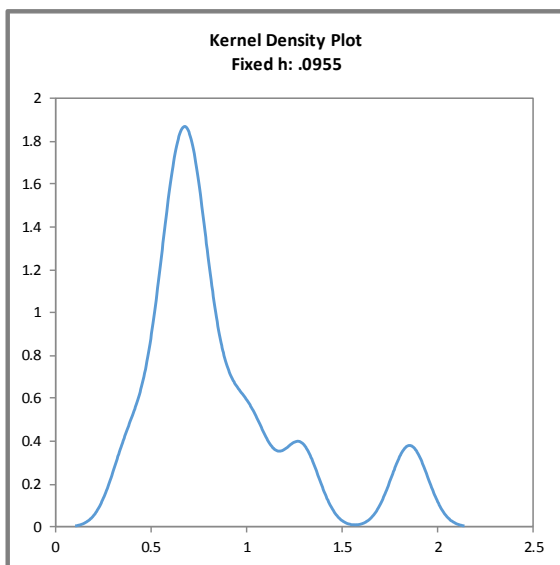**Abb. / Fig. 12:** Ergebnisse Inulin Probe B **/** Results inulin sample B
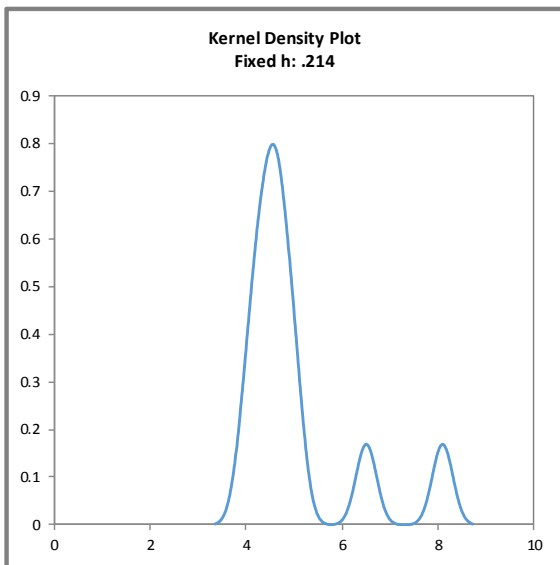
Abb. / Fig. 13:
Kerndichte-Schätzung der Ergebnisse
(mit h = 0,75 x σ_{pt} von X_{pt})

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

Comment:
The kernel density shows almost a symmetrical distribution of results
with two smaller peaks at approx. 7 g/100g and 8 g/100g, due to results
out of the target range.

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer / Evaluation number	Inulin [g/100g]	Abweichung [g/100g] / Deviation [g/100g]	z'-Score (σ_{pt})	z-Score (Info)	Hinweis / Remark
1	4,40	−0,334	−1,2	−2,2	
2	4,90	0,166	0,58	1,1	
3	8,09	3,356	12	22	
4	5,03	0,296	1,0	2,0	
5	3,98	−0,754	−2,6	−5,0	
6	4,65	−0,084	−0,29	−0,56	
7a	4,60	−0,134	−0,47	−0,89	
7b	4,20	−0,534	−1,9	−3,6	
8	6,50	1,766	6,2	12	
9	4,31	−0,424	−1,5	−2,8	
10	4,65	−0,084	−0,29	−0,56	
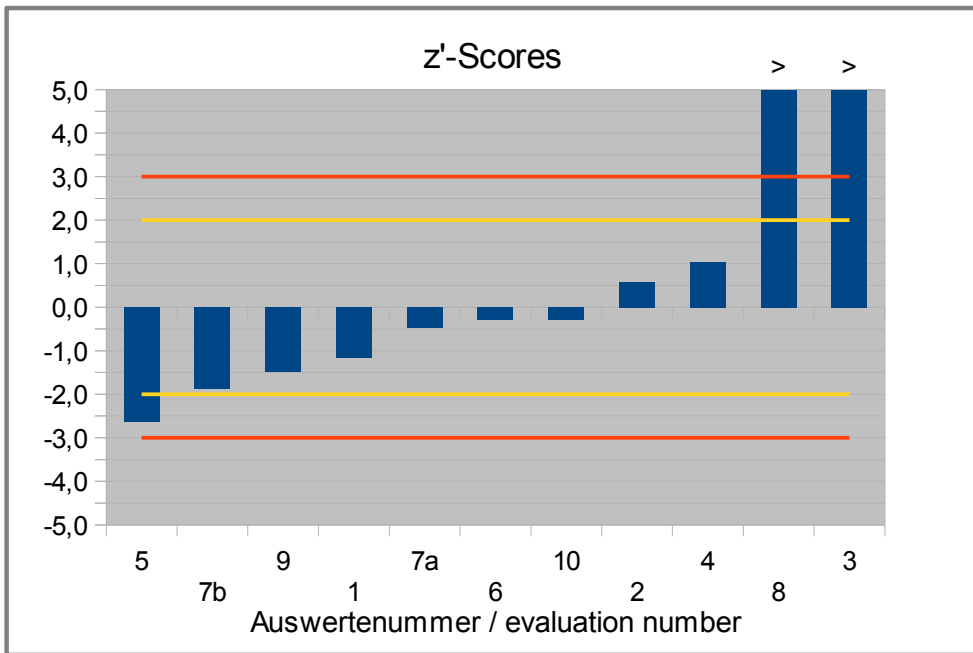
Abb. / Fig. 14: z'-Scores Inulin Probe B / inulin sample B

4.3.3 Difference Sample B – Sample A (Delta Inulin)

The basic matrix spelt waffles was the same for sample A and sample B. Inulin was added to sample B only. Therefore, the difference of the inulin results of sample B and sample A was calculated by DLA and statistically evaluated for information.

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	11
Number of outliers	1
Mean	4,17
Robust Mean	3,99
Median (X_{pt})	**3,91**
Robust standard deviation (S*)	**0,541**
Target range:	
Target standard deviation $\sigma_{pt'}$	**0,243**
Target standard deviation (for Information)	0,127
lower limit of target range	**3,43**
upper limit of target range	**4,40**
Quotient S*/$\sigma_{pt'}$	*2,2*
Standard uncertainty $U_{(Xpt)}$	*0,204*
Results in the target range	*8*
Percent in the target range	*73%*

Comments to the statistical characteristics:

The assigned value was the median (see 3.1).

The target standard deviation was calculated using data from a precision experiment (ASU §64 L 00.00-94)(3.6.2). For information the target standard deviation using the general model of Horwitz is given (s. 3.6.1).

The evaluation of all methods showed an increased variability of results with a quotient S*/σ_{pt} of 4,1. Therefore the evaluation was done by z'-score considering the standard uncertainty. The quotient S*/$\sigma_{pt'}$ was then at 2,2. The robust standard deviation is comparable to those of prior PTs (see 3.6.3). The comparability of results is given.

73% of the results were in the target range.
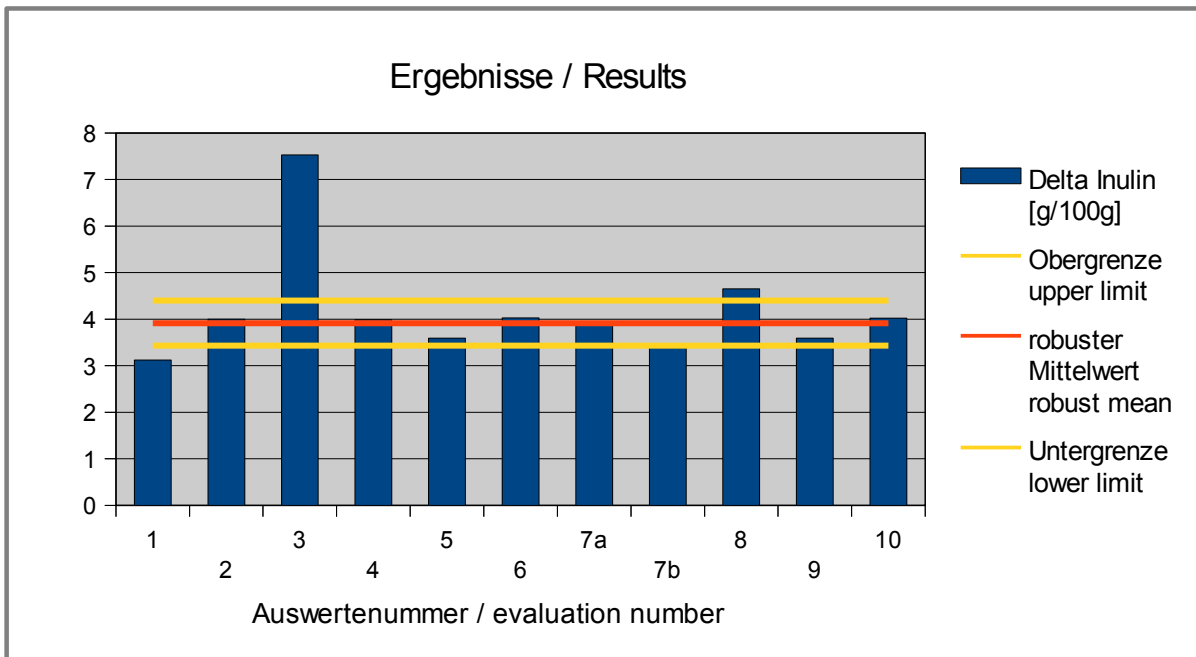
**Abb. / Fig. 15:** Ergebnisse Delta Inulin (Probe B - Probe A) **/** Results delta inulin (sample B - sample A)
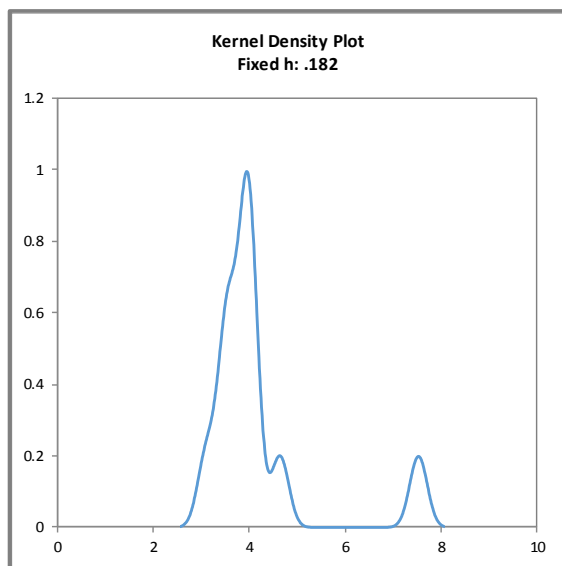
Abb. / Fig. 16:
Kerndichte-Schätzung der Ergebnisse (mit h = 0,75 x σ_{pt} von X$_{pt}$)

Kernel density plot of results (with h = 0,75 x σ_{pt} von X$_{pt}$)

Comment:
The kernel density shows almost a symmetrical distribution of results with a shoulder at approx. 3,0 g/100g and two smaller peaks at approx. 4,6 g/100g and 7,5 g/100g, due to results out of the target range.

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Delta Inulin [g/100g]	Abweichung g/100g] Deviation [g/100g]	z'-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	3,12	−0,794	−3,3	−6,2	
2	4,00	0,086	0,35	0,67	
3	7,53	3,611	15	28	
4	3,99	0,076	0,31	0,59	
5	3,59	−0,324	−1,3	−2,5	
6	4,03	0,116	0,48	0,91	
7a	3,87	−0,044	−0,18	−0,35	
7b	3,47	−0,444	−1,8	−3,5	
8	4,65	0,736	3,0	5,8	
9	3,59	−0,324	−1,3	−2,5	
10	4,02	0,106	0,44	0,83	
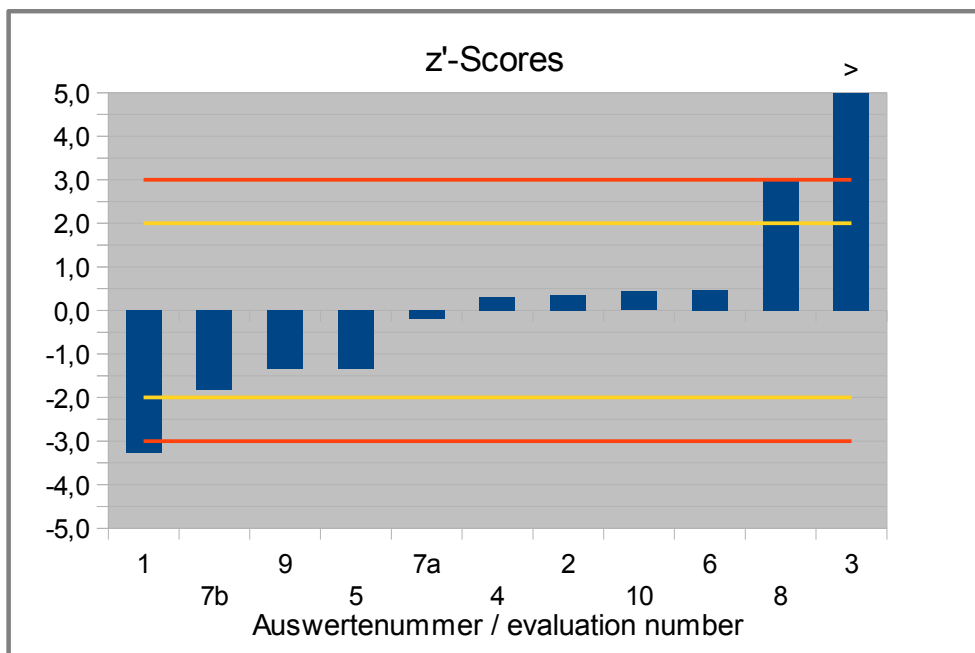
Abb. / Fig. 17:   z'-Scores Delta Inulin (Sample B - Sample A)

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 and analytical Methods

Parameter: Total Dietary Fiber, without inulin

Analyte	Parti-cipant	Unit	Date of analysis	Result (Mean)	Result I	Result II	Result (Mean)	Result I	Result II	Limit of quantification	Incl. Recovery rate	Recovery rate [%]	Method specification, as in test report / standard / literature
			Day / Month	**Sample A**	**Sample A**	**Sample A**	**Sample B**	**Sample B**	**Sample B**		yes / no	in %	
Gesamt-Ballaststoffe, ohne Inulin/ Total Dietary Fiber, without Inulin	1	g/100g	26.02.19	9,4	9,3	9,5	9,1	9,05	9,15	0,5	no		AOAC 991.43
	2	g/100g	21.03.19	9,2			8,3			0,5	no		
	3	g/100g	19.03.19	5,62			5,74				no		L00.00-18:1997-01
	4	g/100g	21.03.19	8,6	8,9	8,3	8,4	8,5	8,3	0,5	no		AOAC991.43
	5	g/100g	12.03.19	9,5	9,26	9,74	8,62	8,3	8,93		no	93,5	ASU L 00.00-18 1997-01 Correction 2002-12; Analysis of food - Determination of fiber in food
	6	g/100g											
	7	g/100g	14.03.19	8,6	8,5	8,6	9,3	9,1	9,5	0,4	no		AOAC 991.43
	8	g/100g											
	9	g/100g											
	10	g/100g											

Continuation:

Analyte	Parti-cipant	Frit diameter	Pore diameter	Notes to Amylase	Notes to Protease	Notes to Amyloglucosidase	Notes to Inulinase	Calibration and reference material	Method accred. accord. ISO/IEC 17025	Further remarks
									yes / no	
Gesamt-Ballaststoffe, ohne Inulin/ Total Dietary Fiber, without Inulin	1							Reference material DLA 2018	yes	
	2								yes	
	3		2						yes	The result for insoluble fiber is given. Normally, soluble & insoluble are not determined separately by us.
	4	/	2	Megazyme kit	Megazyme kit	Megazyme kit	not used		no	sample not dried and defatted (<10%) before determination
	5	41 mm	2	a-Amylase by Merck	Testkit by Merck	Testkit by Merck	Fructanase mixture by Megazyme		yes	Sample preaparation: was degreased
	6									
	7	30 mm	40-90um	Megazyme E-Blaam	Megazyme E-BSPRT	Megazyme E-AMGDF	no inulinase	no	–	
	8									
	9									
	10									

Parameter: Total Dietary Fiber, including inulin

Analyte	Parti-cipant	Unit	Date of analysis	Result (Mean)	Result I	Result II	Result (Mean)	Result I	Result II	Limit of quantification	Incl. Recovery rate	Recovery rate [%]	Method specification, as in test report / standard / literature
			Day / Month	**Sample A**	**Sample A**	**Sample A**	**Sample B**	**Sample B**	**Sample B**		yes / no	in %	
Gesamt-Ballaststoffe, inklusive Inulin/ Total Dietary Fiber, including Inulin	1	g/100g	26.02.19										
	2	g/100g		11,7			14			0,1	no		
	3	g/100g											
	4	g/100g											
	5	g/100g											
	6	g/100g											
	7a	g/100g	13.03.19	11,8	11,8	11,8	15,3	15,1	15,5	0,3	no		AOAC 2009.01
	7b	g/100g	13.03.19	11,2	11,4	11	14,4	14,6	14,2	0,5	no		AOAC 2011.25
	8	g/100g	20.03.19	7,77	7,76	7,77	12,82	12,72	12,91	0.144 g/100 g	no	N/A	AOAC 2009.01
	9	g/100g	26.2-5.3.19	11,19	11,33	11,05	15,21	15,41	15,01	1,0 g/100g	no		AOAC 2001.03
	10	g/100g											

Continuation:

Analyte	Parti-cipant	Frit diameter	Pore diameter	Notes to Amylase	Notes to Protease	Notes to Amyloglucosidase	Notes to Inulinase	Calibration and reference material	Method accred. accord. ISO/IEC 17025	Further remarks
									yes / no	
	1									
	2								yes	
	3									
	4									
	5									
Gesamt-Ballaststoffe, inklusive Inulin/ Total Dietary Fiber, including Inulin	6									
	7a	30 mm	40-90um	Megazyme E-PANAA	Megazyme E-BSPRT	Megazyme E-AMGDF	no inulinase	no	–	
	7b	30 mm	40-90um	Megazyme E-PANAA	Megazyme E-BSPRT	Megazyme E-AMGDF	no inulinase	no	–	
	8	30 mm	40-60 µm	alpha- amylase 50 units/mL + 3.4 units/mL AMG (Megazyme)	350 tyrosine units/mL (Megazyme)	AMG 3300 units/mL (Megazyme)	N/A	Sigma-Aldrich	Yes	N/A
	9	3,0 cm	40-60 µm	A 3306 of TDF100A Sigma	P 3910 of TDF100A Sigma	A 9913 of TDF100A Sigma			no	
	10									

Parameter: Inulin

Analyte	Parti-cipant	Unit	Date of analysis	Result (Mean)	Result I	Result II	Result (Mean)	Result I	Result II	Limit of quantification	Incl. Recovery rate	Recovery rate [%]	Method specification, as in test report / standard / literature
			Day / Month	**Sample A**	**Sample A**	**Sample A**	**Sample B**	**Sample B**	**Sample B**		yes / no	in %	
Inulin	1	g/100g	27.02.19	1,28	1,15	1,4	4,4	4,3	4,5	0,05	no		AOAC 997.08: „Fructans in Food Products (Ion Exchange Chromatographic Method)", modifiziert
	2	g/100g	22.03.19	0,9			4,9			0,5	no		
	3	g/100g	31.03.+03.04.	0,565	0,68	0,45	8,09	8,23	7,95		no		L00.00-94:2008-12
	4	g/100g	26.03.19	1,04	1	1,1	5,03	5,1	4,9	0,1	no		AOAC997.08
	5	g/100g	28.03.19	0,39	0,42	0,36	3,98	4,03	3,92		no	109,98	ASU L 00.00-94 2006-09; Analysis of food - Determinatin of inulin in food - Enzymatic method Deviation: Use of the test kit R-Biopharm AG, sucrose, D-glucose and D-fructose, 10 716 260 035, 2014-01, For bakery products also extraction at RT
	6	g/100g	27.02.19	0,62	0,59	0,64	4,65	4,64	4,66	0,3	no		ASU § 64 LFGB 00.00-94
	7a	g/100g	19.03.19	0,73	0,77	0,68	4,6	4,6	4,6	0,3	no		AOAC 997.08 modified
	7b	g/100g	20.03.19	0,73	0,72	0,73	4,2	4,3	4,1	0,2	no		AOAC 999.03 modified
	8	g/100g	20.03.19	1,85	1,84	1,85	6,5	6,4	6,59	0.138 g/100 g	No	N/A	AOAC 2009.01
	9	g/100g	11.-18.3.19	0,72	0,71	0,73	4,31	4,35	4,27	0,1 g/100g	no		AOAC 997.08
	10	g/100g	13.03.19	0,63	0,61	0,64	4,65	4,57	4,73		no		ASU L 00.00-94 (modified)

Continuation:

Analyte	Parti-cipant	UV-test kit manufacturer	HPLC column	Detector	Notes on sample preparation and prozessing	Calibration and reference material	Method accred. accord. ISO/IEC 17025	Further remarks
							yes / no	
Inulin	1		Carbopac™ PA20; 3 x 150 mm	PAD	-	Reference material DLA 2018	yes	Following modifications were done: Glucose content is calculated on the basis of the degree of polymerisation, Other eluent composition, Gradient program shortened from 83 to 47 min
	2						no	
	3		NUCLEOGEL Sugar 810 Ca	RI	Purification with C18 SPE cartridges		no	
	4		GC	FID			no	
	5	R-Biopharm					yes / no	
	6	r-Biopharm					yes	
	7a	_	Dionex PA1	PAD	no		-	
	7b	_	Dionex PA1	PAD	no		-	
	8	N/A	TSK Gel (G2500PWXL)	RID	N/A	Sigma-Aldrich	No	LC results from AOAC 2009.01
	9	_	Carbopac PA20	PAD	aqueous extraction, hydrolysis with fructanase	DLA-material 36-2018	no	
	10	r-Biopharm		UV/Vis-Spectrometer	Inulinase by Sigma Aldrich		yes	

5.2 Homogeneity

5.2.1 Mixture homogeneity before bottling

Microtracer Homogeneity Test
DLA 35-2019 Sample A

Weight whole sample	1,71	kg
Microtracer	FSS-rot lake	
Particle size	75 – 300	µm
Weight per particle	2,0	µg
Addition of tracer	19,8	mg/kg

Result of analysis

Sample	Weight [g]	Particle number	Particles [mg/kg]
1	5,08	58	22,8
2	5,09	56	22,0
3	5,00	52	20,8
4	5,06	46	18,2
5	4,96	56	22,6
6	5,05	50	19,8
7	4,99	51	20,4
8	4,97	52	20,9

Poisson distribution		
Number of samples	8	
Degree of freedom	7	
Mean	52,6	Partikel
Standard deviation	3,87	Partikel
χ^2 (CHI-Quadrat)	1,99	
Probability	**96**	%
Recovery rate	106	%

Normal distribution		
Number of samples	8	
Mean	20,9	mg/kg
Standard deviation	1,54	mg/kg
rel. Standard deviaton	7,35	%
Horwitz standard deviation	10,1	%
HorRat-value	**0,73**	
Recovery rate	106	%

Microtracer Homogeneity Test
DLA 35-2019 Sample B

Weight whole sample	1,92	kg
Microtracer	FSS-rot lake	
Particle size	75 – 300	µm
Weight per particle	2,0	µg
Addition of tracer	19,4	mg/kg

Result of analysis

Sample	Weight [g]	Particle number	Particles [mg/kg]
1	5,06	54	21,3
2	4,97	49	19,7
3	4,99	50	20,0
4	5,00	62	24,8
5	5,00	53	21,2
6	5,01	47	18,8
7	4,97	54	21,7
8	5,05	60	23,8

Poisson distribution		
Number of samples	8	
Degree of freedom	7	
Mean	53,6	Partikel
Standard deviation	5,09	Partikel
χ^2 (CHI-Quadrat)	3,38	
Probability	**85**	%
Recovery rate	110	%

Normal distribution		
Number of samples	8	
Mean	21,4	mg/kg
Standard deviation	2,03	mg/kg
rel. Standard deviaton	9,49	%
Horwitz standard deviation	10,1	%
HorRat-value	**0,94**	
Recovery rate	110	%

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 35-2019**
PT name	**Dietary Fiber and Inulin in Cereal Product**
Sample matrix*	Samples A + B: Waffles / Ingredients: Spelt, salt and inulin (one of the two samples), fat content: < 10%
Number of samples and sample amount	2 different samples A + B, 40 g each.
Storage	Samples A + B: room temperature
Intentional use	Laboratory use only (quality control samples)
Parameter	quantitative: **total dietary fiber and inulin**
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	For each sample A and B, two results (double determination) per parameter are queried. The individual results as well as the mean value for sample A and for sample B should be entered in the result file.
Units	g/100g
Number of significant digits	at least 2
Further information	For information please specify: – Date of analysis – 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 05th April 2019**
Evaluation report	The evaluation report is expected to be completed 6 weeks after deadline of result submission and sent as PDF file by e-mail.
Coordinator and contact person of PT	Matthias Besler-Scharf PhD

* 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
		CANADA
		Germany
		Germany
		NETHERLANDS
		Germany
		Germany
		Germany
		Germany
		BELGIUM
		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.]

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20. ASU § 64 LFGB L 00.00-94 Bestimmung von Inulin in Lebensmitteln – Enzymatisches Verfahren [Determination of inulin in food – Enzymatic method]
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