Evaluation Report
proficiency test

DLA ptAU01 (2021)

Dietary Fiber, soluble/insoluble Fiber and Inulin

in Cereal Product (Rusks)

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

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Unteraufträge *Subcontractors*	Im Rahmen dieser Eignungsprüfung wurden nachstehende Leistungen im Unterauftrag vergeben: Keine As part of the present proficency test the following services were subcontracted: none
Vertraulichkeit *Confidentiality*	Die Teilnehmerergebnisse sind im EP-Bericht in anonymisierter Form mit Auswertenummern benannt. Daten einzelner Teilnehmer werden ausschließlich nach vorheriger Zustimmung des Teilnehmers an Dritte weitergegeben. Participant result are named anonymously with evaluation numbers in the PT report. Data of individual participants will be passed on to third parties only with prior consent of the participant.

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1. Introduction

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

2. Realisation

2.1 Test material

The test material of the food matrix samples are customary spelt rusks. The basic composition of samples A and B was the same (see table 1). Additionally inulin was added to sample A.
After crushing and sieving (mesh 1,5 mm) of the rusks, the basic mixture was homogenized.
Afterwards the spiked sample A 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.

Table 1: Composition of DLA-Samples

Ingredients	**Probe A**	**Probe B**
Spelt Rusks, organic Ingredients: Spelled flour (83%), whole spelled meal (9%), sunflower oil, yeast, salt, coriander Nutrients per 100 g: Fat 6,8 g, carbohydrates 70 g, thereof sugar 2,2 g, protein 13 g, salt 0,99	96,7 g/100g *	100 g/100g *
- thereof fiber (10 %) **	4,2 g/100g **	4,3 g/100g **
Inulin	3,05 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 **micro-tracer analysis.** It is a standardized method that is part of the international GMP certification system for feed [14].
Before mixing dye coated iron particles of µm size are added to the sample and the number of particles is determined after homogenization in taken aliquots. The evaluation of the mixture homogeneity is based on the Poisson distribution using the chi-square test. A probability of ≥ 5 % is equivalent to a good homogeneous mixture and of ≥ 25% to an excellent mixture [14, 15].
The microtracer analysis of the present PT sample A showed a probability of 91%. Additionally particle number results were converted into concentrations, statistically evaluated according to normal distribution and compared to the standard deviation according to Horwitz. For the assessment HorRat values between 0,3 and 1,3 are to be accepted under repeat conditions (measurements within the laboratory) [17].
This gave a HorRat value of 1,1. 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 (method 1) it is approx. 5,2% and 5,4% and for inulin it is 5,3% (sample A with inulin addition) and 5,3% (sample B without inulin addition). Thus they were similar to the repeatability standard deviations of the official methods ((ASU L 00.00-18 / L 16.08-1 / 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 (1)	5,16 %	5,44 %
Total dietary fiber incl. inulin (2+3)	1,98 %	1,19 %
Insoluble dietary fiber (1a, 2a, 3a)	8,39 %	5,34 %
Inulin / Fructans (5+6)	2,00 %	5,31 %*

* Sample B without addition of inulin
(1 - 6) Methods see page 16

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. 0,17 and 0,19 (20-21°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 18th week of 2021. The testing method was optional. The tests should be finished at 2nd June 2021 the latest.

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

There are two different samples of rusk. Inulin was added to one of the two samples A or B. The fat content is < 10%.

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

Note: The analysis methods are optional. However, please carefully observe the given method lines in the result submission file. Directly below the table there are footnotes with information on the methods and standards.

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 17 participants submitted at least one result.

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 parameter total dietary fiber (methods 2+3) 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.

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

3.2 Robust standard deviation

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

3.3 Repeatability standard deviation

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

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

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

3.4 Reproducibility standard deviation

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

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

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

3.5 Exclusion of results and outliers

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

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

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

3.6 Target standard deviation (for proficiency assessment)

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

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

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

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

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 16.08-1, L 17.03-1, L 00.00-94; AOAC 2009.01).

<u>Additionally</u> the standard uncertainty was considered for methods 1 (total dietary fiber) and methods 1a+2a+3a (insoluble fiber) by valuating with z'-scores (see 3.6.8).

Note: Methods 1-6 see page 16

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.

Table 3: 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-gravimetric)	Mixed rye bread	8,83	2,49 %	5,10 %	4,8 %	ASU §64 L 17.03-1
	Grain bran	13,62	5,87 %	12,4 %	11,7 %[1]	L 16.08-1
Insoluble Dietary Fiber (enzymatic-gravimetric)	Mixed rye bread	5,45	5,14 %	8,44 %	7,6 %	ASU §64 L 17.03-1
	Grain bran	10,21	6,46 %	14,5 %	13,8 %[1]	L 16.08-1
Soluble Dietary Fiber (enzymatic-gravimetric)	Mixed rye bread	3,02	14,6 %	20,9 %	18,2 %	ASU §64 L 17.03-1
Inulin (enzymatic-photometric)	Finished flour	16,7	1,86 %	3,60 %	3,4 %	ASU §64 L 00.00-94
	baby food	0,61	4,92 %	8,20 %	7,4 %[1]	
	chocolate	5,95	2,35 %	4,03 %	3,7 %	
Total Dietary Fiber including Inulin (enzymatic-gravimetric + liquid chromatography)	Whole grain bread	11,6	12,3 %	18,0 %	15,8 %[1]	AOAC 2009.01
	Whole grain paste	12,7	4,47 %	11,3 %	10,8 %	

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

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

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].

Table 4: 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 ****
Total Dietary Fiber with Inulin (2+3)	Cereal product	7,51 5,41	0,765 0,480	10,2 8,88	0,65 0,56	DLA ptAU01 (2021) *Proben ****
Total Dietary Fiber without Inulin (1)	Cereal product	4,36 4,30	0,797 0,621	18,3 14,4	1,4* 1,2*	DLA ptAU01 (2021) *Proben ****
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 ****
Inulin/Fructans (5+6)	Cereal product	2,85 0,196	0,241 0,0925	8,46 47,3	1,1 2,1	DLA ptAU01 (2021) *Proben ****

** 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:

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

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

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

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

For warning and action signals see 3.7.1.

3.9 Reproducibility coefficient of variation (CV)

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

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

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

3.10 Quotient S*/σ_{pt}

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

3.11 Standard uncertainty and traceability

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

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

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

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

4. Results

For **submission** and **evaluation** of results, the following method classifications were specified / used by DLA in the present PT:

Parameter *(Methods)*
Total Dietary Fiber, without Inulin (1)
Insoluble Dietary Fiber, without Inulin (1a)
Soluble Dietary Fiber, without Inulin (1b)
Total Dietary Fiber, including Inulin (2)
Insoluble Dietary Fiber (2a)
Soluble Dietary Fiber, including Inulin (2b)
Total Dietary Fiber, Sum of IDF, HMW SDF, and LMW SDF (3)
Insoluble dietary fiber IDF (3a)
High molecular weight soluble dietary fiber HMW SDF (3b)
Low molecular weight soluble dietary fiber LMW SDF (3c)
Total Dietary Fiber, other (4)
Inulin (5)
Fructans (6)

(1) enzymatic-gravimetric methods like ASU L 00.00-18 (ASU L 17.03-1) or AOAC 991.43 and AOAC 985.29, in which lower results of inulin can occur

(1a + 1b) e.g. inulinase-reaction included

(2) enzymatic-gravimetric methods combined with liquid chromatography like AOAC 2009.01, which include inulin

(2a + 2b) e.g. without inulinase-reaction

(3) Total Dietary Fiber, as Sum of Insoluble DF, High Molecular Weight Soluble DF, and Low Molecular Weight Soluble DF (AOAC 2017.16)

(4) other methods

(5) enzymatic methods (e.g. ASU L 00.00-94)

(6) liquid-chromatography methods after enzymatic treatment (e.g. ISO 22579 (IDF 241):2020/AOAC 2016.14)

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

In the first table the characteristics are listed:

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

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

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

Auswerte-nummer	**Parameter [Einheit / Unit]**	**Abweichung**	**z-Score σpt**	**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 Total Dietary Fiber without Inulin in g/100g

4.1.1 Sample A (Methods: 1)

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	18
Number of outliers	0
Mean	4,32
Median	4,42
Robust Mean (X)	**4,36**
Robust standard deviation (S*)	**0,797**
Number with 2 replicates	17
Repeatability SD (S_r)	0,220
Repeatability (CV_r)	5,16%
Reproducibility SD (S_R)	0,802
Reproducibility (CV_R)	18,8%
Target range:	
Target standard deviation $\sigma_{pt'}$	**0,561**
Target standard deviation (for Information)	0,140
lower limit of target range	**3,24**
upper limit of target range	**5,49**
Quotient S/$\sigma_{pt'}$*	*1,4*
Standard uncertainty $U_{(Xpt)}$	*0,235*
Results in the target range	*16*
Percent in the target range	*89%*

Comments to the statistical characteristics:

The target standard deviation was calculated using data from a precision experiment (ASU §64 L 16.08-1) (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 slightly increased variability of results. Therefore the valuation was done by z'-scores considering the standard uncertainty. The quotient $S^*/\sigma_{pt'}$ was then < 2,0. The robust standard deviation was in the range of previous PTs (see 3.6.3). The robust standard deviation is comparable to those of prior PTs (see 3.6.3). The repeatability and reproducibility standard deviation are in the range of established values of the applied methods (see 3.6.2). The comparability of results is given.

89% of the results were in the target range.

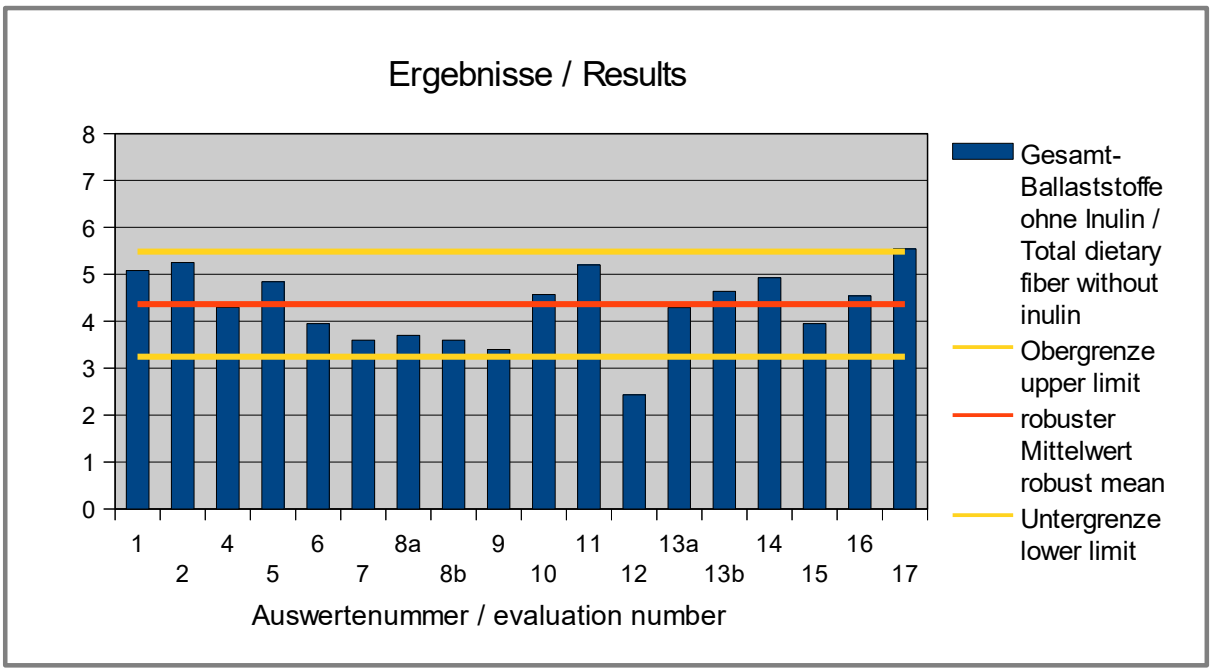

Abb. / Fig. 1: Ergebnisse Gesamt-Ballaststoffe ohne Inulin Probe A / Results total dietary fiber without inulin sample A

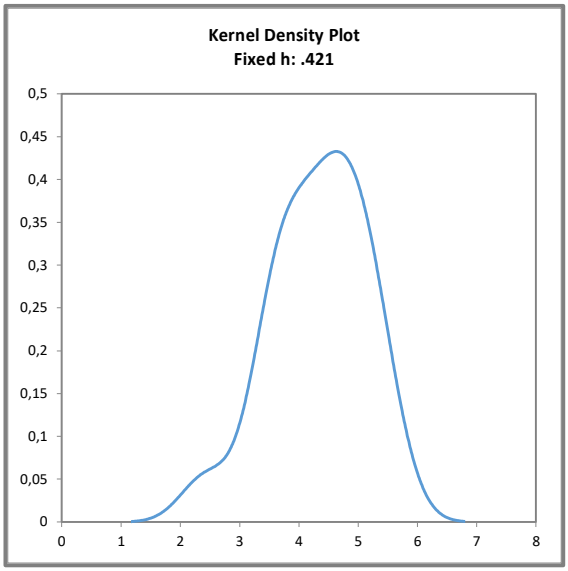

Abb. / Fig. 2:
Kerndichte-Schätzung der Ergebnisse (mit h = 0,75 x σ_{pt} von X_{pt})

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 small shoulders.

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Gesamt-Ballaststoffe ohne Inulin / Total dietary fiber without inulin	Abweichung [g/100g] Deviation [g/100g]	z'-Score (σ_{pt})	z-Score (Info)	Methode Method	Hinweis Remark
1	5,08	0,716	1,3	5,1	4 = 1	Method 1 assigned by DLA
2	5,25	0,886	1,6	6,3	1	
4	4,30	-0,069	-0,12	-0,49	2 = 1	Method 1 assigned by DLA
5	4,84	0,476	0,85	3,4	1	
6	3,95	-0,414	-0,74	-3,0	1	
7	3,60	-0,764	-1,4	-5,5	1	
8a	3,70	-0,664	-1,2	-4,7	1	
8b	3,60	-0,764	-1,4	-5,5	4 = 1	Method 1 assigned by DLA
9	3,40	-0,964	-1,7	-6,9	2 = 1	Method 1 assigned by DLA
10	4,57	0,206	0,37	1,5	1	
11	5,20	0,836	1,5	6,0	1	
12	2,43	-1,934	-3,4	-14	1	
13a	4,29	-0,074	-0,13	-0,53	1	
13b	4,64	0,276	0,49	2,0	4 = 1	Method 1 assigned by DLA
14	4,93	0,566	1,0	4,1	1	
15	3,95	-0,414	-0,74	-3,0	1	
16	4,54	0,176	0,31	1,3	1	
17	5,54	1,176	2,1	8,4	1	

(1) enzymatic-gravimetric methods like ASU L 00.00-18 (ASU L 17.03-1) or AOAC 991.43 and AOAC 985.29, in which lower results of inulin can occur

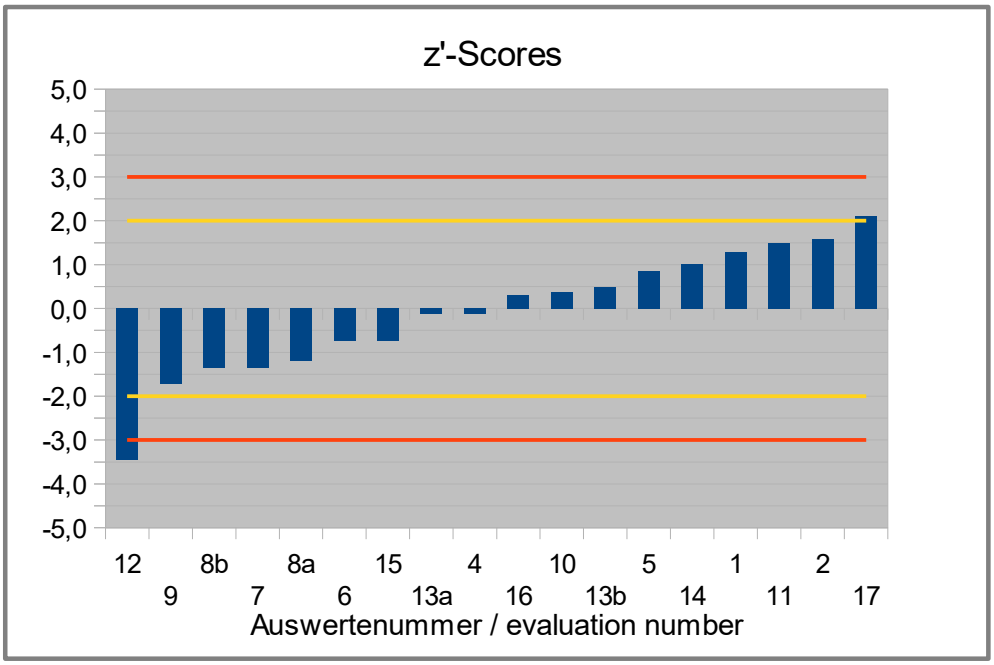

Abb. / Fig. 3: z'-Scores Gesamt-Ballaststoffe ohne Inulin Probe A / Total dietary fiber without inulin sample A

4.1.2 Sample B (Methods: 1)

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	18
Number of outliers	0
Mean	4,26
Median	4,19
Robust Mean (X)	**4,30**
Robust standard deviation (S*)	**0,621**
Number with 2 replicates	17
Repeatability SD (S_r)	0,230
Repeatability (CV_r)	5,44%
Reproducibility SD (S_R)	0,714
Reproducibility (CV_R)	16,9%
Target range:	
Target standard deviation $\sigma_{pt'}$	**0,535**
Target standard deviation (for Information)	0,138
lower limit of target range	**3,23**
upper limit of target range	**5,4**
Quotient S/$\sigma_{pt'}$*	*1,2*
Standard uncertainty $U_{(Xpt)}$	*0,183*
Results in the target range	*16*
Percent in the target range	*89%*

Comments to the statistical characteristics:

The target standard deviation was calculated using data from a precision experiment (ASU §64 L 16.08-1) (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 slightly increased variability of results. Therefore the valuation was done by z'-scores considering the standard uncertainty. The quotient S*/$\sigma_{pt'}$ was then < 2,0. The robust standard deviation was in the range of previous PTs (see 3.6.3). The robust standard deviation is comparable to those of prior PTs (see 3.6.3). The repeatability and reproducibility standard deviation are in the range of established values of the applied methods (see 3.6.2). The comparability of results is given.

89% of the results were in the target range.

Note: For methods 1 the difference of the robust means for sample A (with addition of 3,05 g/100g inulin) and sample B is 0,06 g/100g.

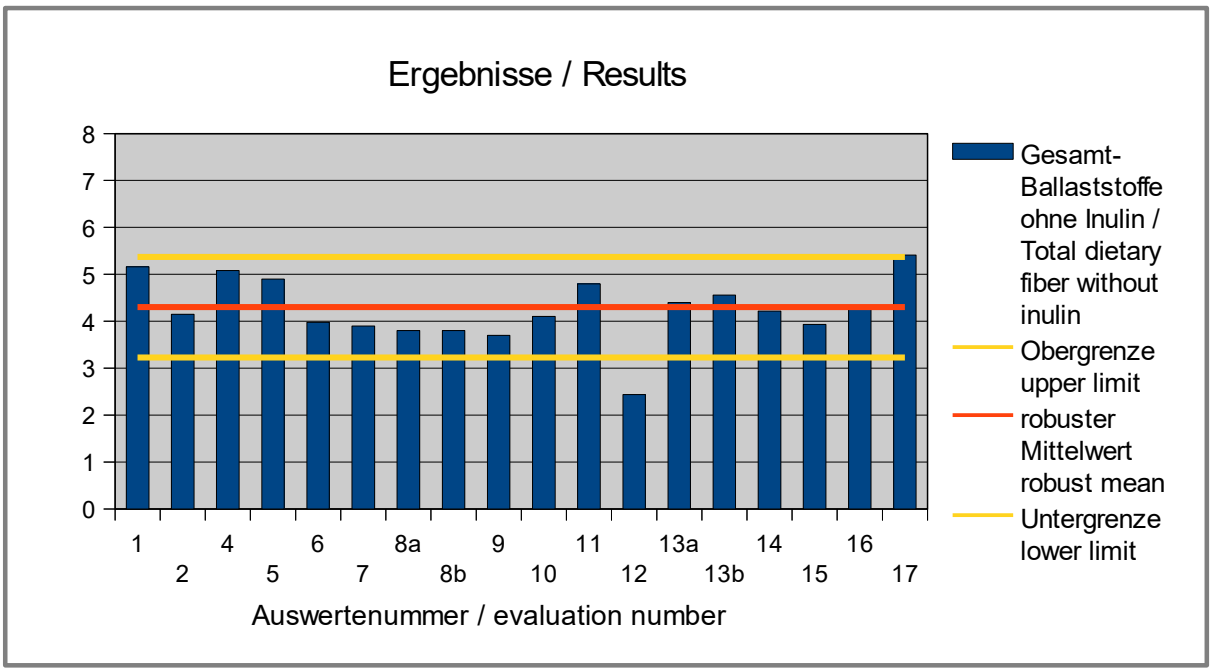

Abb. / Fig. 4: Ergebnisse Gesamt-Ballaststoffe ohne Inulin Probe B / Results total dietary fiber without inulin sample B

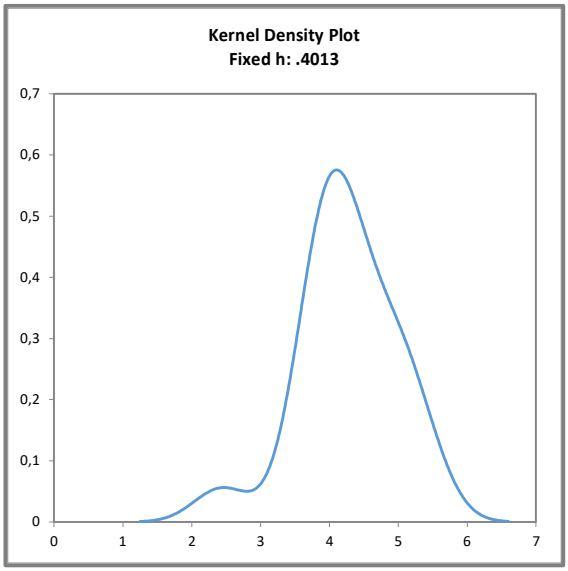

Abb. / Fig. 5:
Kerndichte-Schätzung der Ergebnisse (mit h = 0,75 x σ_{pt} von X_{pt})

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 slight shoulder and a small side peak.

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Gesamt-Ballaststoffe ohne Inulin / Total dietary fiber without inulin	Abweichung [g/100g] Deviation [g/100g]	z'-Score (σ_{pt})	z-Score (Info)	Methode Method	Hinweis Remark
1	5,16	0,861	1,6	6,2	4 = 1	Method 1 assigned by DLA
2	4,15	-0,149	-0,28	-1,1	1	
4	5,08	0,781	1,5	5,7	2 = 1	Method 1 assigned by DLA
5	4,90	0,601	1,1	4,4	1	
6	3,98	-0,319	-0,60	-2,3	1	
7	3,90	-0,399	-0,75	-2,9	1	
8a	3,80	-0,499	-0,93	-3,6	1	
8b	3,80	-0,499	-0,93	-3,6	4 = 1	Method 1 assigned by DLA
9	3,70	-0,599	-1,1	-4,3	2 = 1	Method 1 assigned by DLA
10	4,11	-0,194	-0,36	-1,4	1	
11	4,80	0,501	0,94	3,6	1	
12	2,44	-1,859	-3,5	-13	1	
13a	4,40	0,101	0,19	0,73	1	
13b	4,56	0,261	0,49	1,9	4 = 1	Method 1 assigned by DLA
14	4,22	-0,079	-0,15	-0,57	1	
15	3,93	-0,369	-0,69	-2,7	1	
16	4,30	0,001	0,00	0,01	1	
17	5,41	1,111	2,1	8,0	1	

(1) enzymatic-gravimetric methods like ASU L 00.00-18 (ASU L 17.03-1) or AOAC 991.43 and AOAC 985.29, in which lower results of inulin can occur

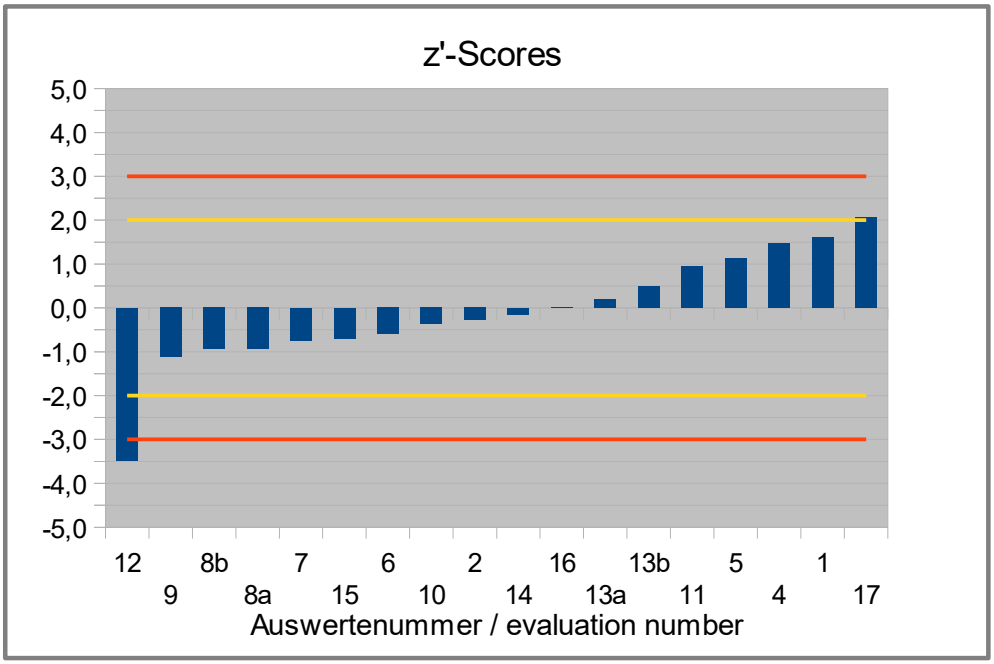

Abb. / Fig. 6: z'-Scores Gesamt-Ballaststoffe ohne Inulin Probe B / Total dietary fiber without inulin sample B

4.2 Total Dietary Fiber with Inulin in g/100g

4.2.1 Sample A (Methods: 2+3)

Vergleichsuntersuchung / Proficiency Test

Kenndaten	
Anzahl der Messergebnisse	5
Anzahl der Ausreißer	0
Mittelwert	7,51
Median	7,50
Robuster Mittelwert (X_{pt})	**7,51**
Robuste Standardabweichung (S*)	**0,765**
Anzahl mit 2 Wiederholmessungen	3
Wiederholstandardabweichung (S_r)	0,143
Variationskoeffizient (VK_r)	1,98%
Vergleichsstandardabweichung (S_R)	0,727
Variationskoeffizient (VK_R)	10,1%
Zielkenndaten:	
Zielstandardabweichung σ_{pt}	**1,18**
Zielstandardabweichung (zur Information)	0,222
Untere Grenze des Zielbereichs	**5,14**
Obere Grenze des Zielbereichs	**9,87**
Quotient S/σ_{pt}*	*0,65*
Standardunsicherheit $U_{(Xpt)}$	*0,428*
Ergebnisse im Zielbereich	*5*
Prozent im Zielbereich	*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 repeatability and reproducibility standard deviation are in the range of established values of the applied methods (see 3.6.2). The comparability of results is given.

All results were in the target range.

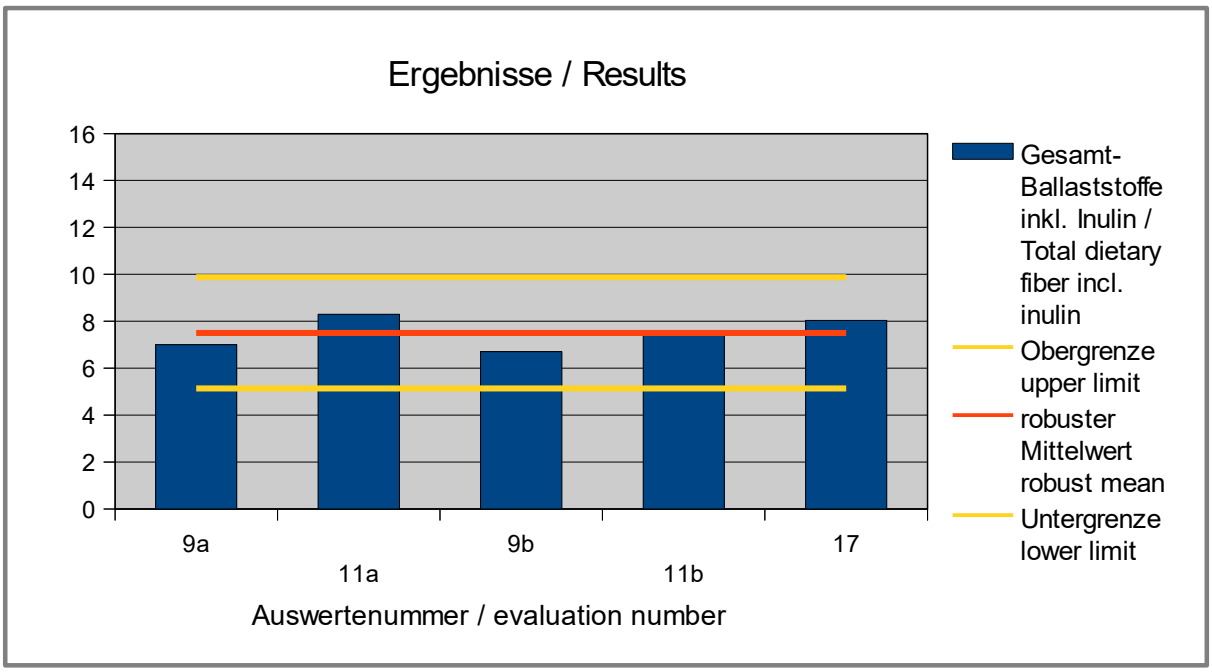

Abb. / Fig. 7: Ergebnisse Gesamt-Ballaststoffe inkl. Inulin Probe A / Results total dietary fiber with inulin sample A

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	Abweichung [g/100g] Deviation [g/100g]	z-Score (σ_{pt})	z-Score (Info)	Methode Method	Hinweis Remark
9a	7,00	-0,508	-0,43	-2,3	4 = 2	Method 2 assigned by DLA
11a	8,30	0,792	0,67	3,6	2	
9b	6,70	-0,808	-0,68	-3,6	3	
11b	7,50	-0,008	-0,01	-0,04	3	
17	8,04	0,532	0,45	2,4	3	

(2) enzymatic-gravimetric methods combined with liquid chromatography like AOAC 2009.01, which include inulin
(3) Total Dietary Fiber, as Sum of Insoluble DF, High Molecular Weight Soluble DF, and Low Molecular Weight Soluble DF (AOAC 2017.16)

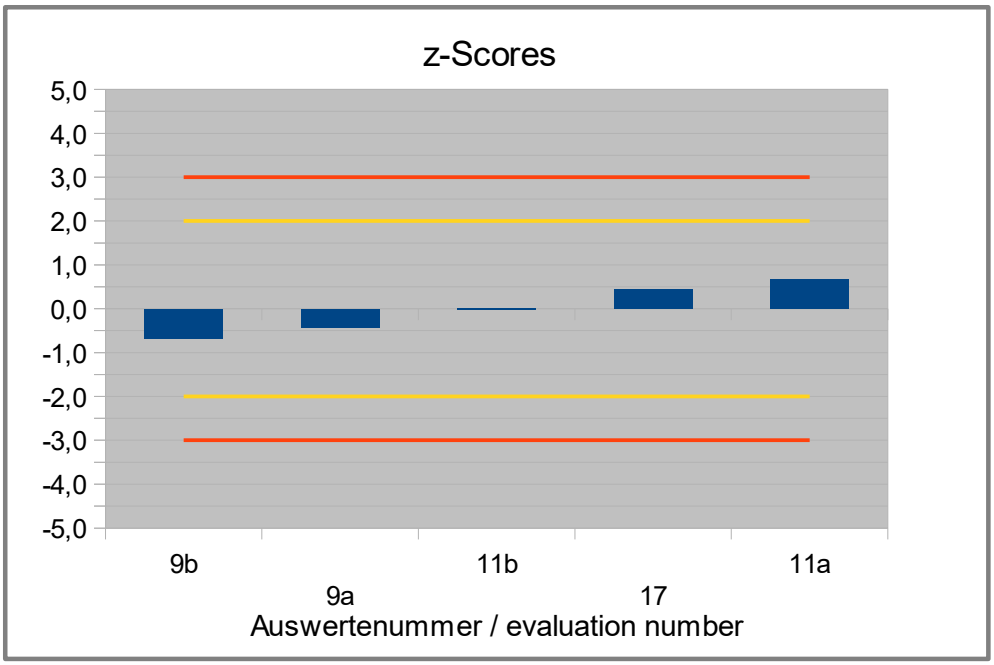

Abb. / Fig. 8: z-Scores Gesamt-Ballaststoffe inkl. Inulin Probe A / Total dietary fiber with inulin sample A

4.2.1 Sample B (Methodes: 2+3)

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	5
Number of outliers	0
Mean	5,41
Median	5,40
Robust Mean (X)	**5,41**
Robust standard deviation (S*)	**0,480**
Number with 2 replicates	3
Repeatability SD (S_r)	0,063
Repeatability (CV_r)	1,19%
Reproducibility SD (S_R)	0,129
Reproducibility (CV_R)	2,45%
Target range:	
Target standard deviation σ_{pt}	**0,852**
Target standard deviation (for Information)	0,168
lower limit of target range	**3,70**
upper limit of target range	**7,11**
Quotient S/σ_{pt}*	*0,56*
Standard uncertainty $U_{(Xpt)}$	*0,268*
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 repeatability and reproducibility standard deviation are in the range of established values of the applied methods (see 3.6.2). The comparability of results is given.

All results were in the target range.

Note: For methods 2+3 the difference of the robust means for sample A (with addition of 3,05 g/100g inulin) and sample B is 2,10 g/100g.

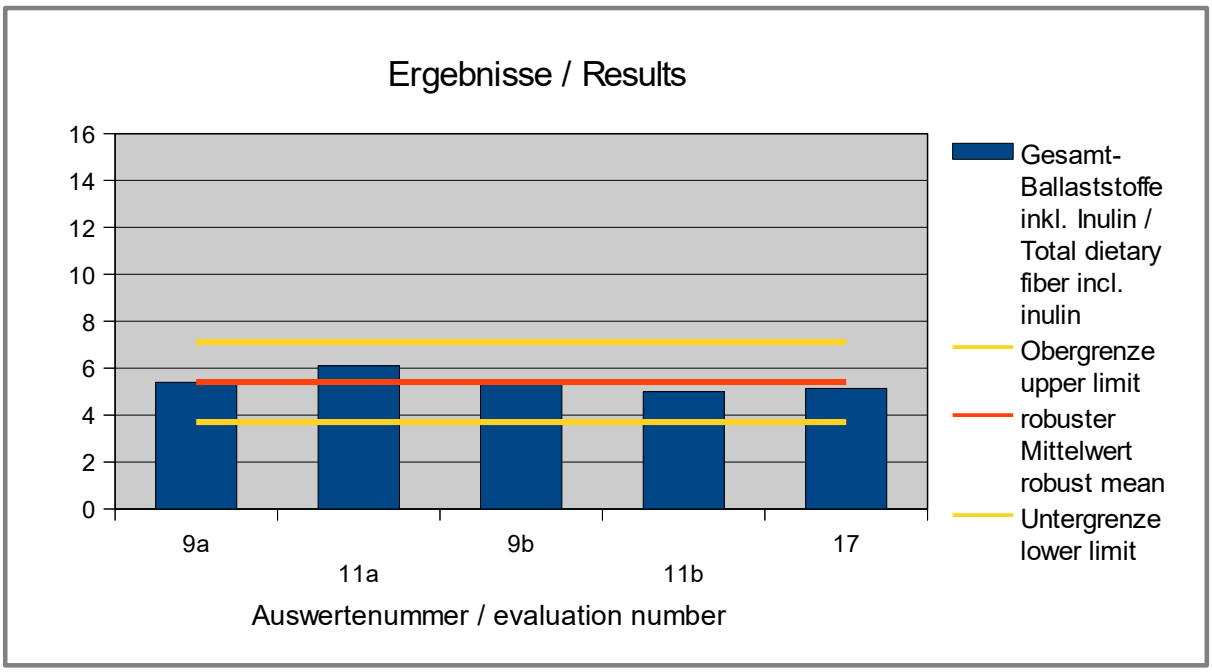

Abb. / Fig. 9: Ergebnisse Gesamt-Ballaststoffe inkl. Inulin Probe B / Results total dietary fiber with inulin sample B

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	Abweichung [g/100g] Deviation [g/100g]	z-Score (σ_{pt})	z-Score (Info)	Methode Method	Hinweis Remark
9a	5,40	-0,008	-0,01	-0,05	4 = 2	Method 2 assigned by DLA
11a	6,10	0,692	0,81	4,1	2	
9b	5,40	-0,008	-0,01	-0,05	3	
11b	5,00	-0,408	-0,48	-2,4	3	
17	5,14	-0,268	-0,31	-1,6	3	

(2) enzymatic-gravimetric methods combined with liquid chromatography like AOAC 2009.01, which include inulin
(3) Total Dietary Fiber, as Sum of Insoluble DF, High Molecular Weight Soluble DF, and Low Molecular Weight Soluble DF (AOAC 2017.16)

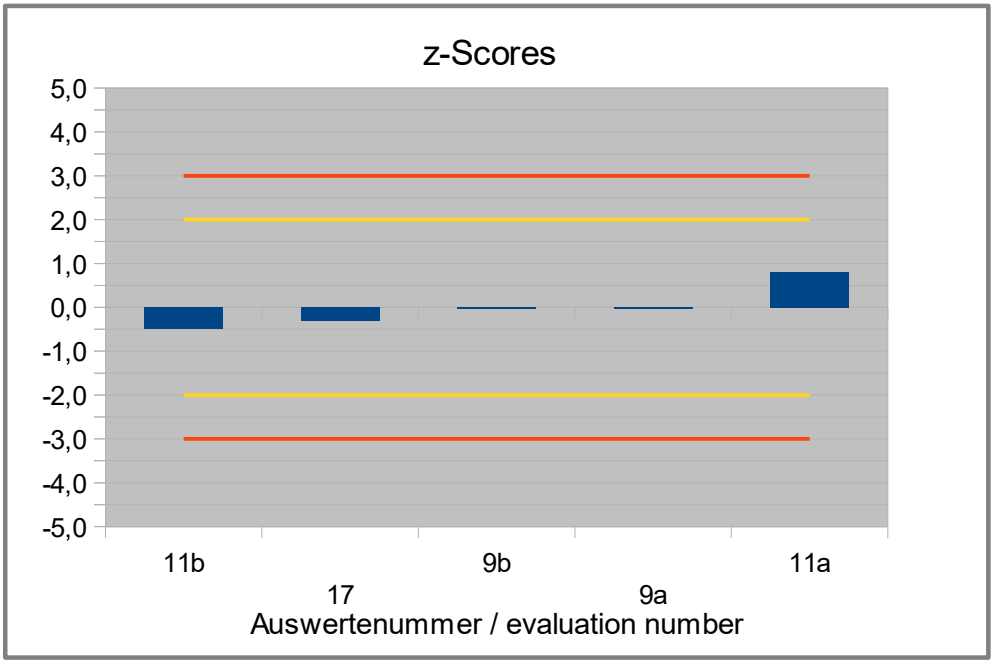

Abb. / Fig. 10: z-Scores Gesamt-Ballaststoffe inkl. Inulin Probe B / Total dietary fiber with inulin sample B

4.3 Insoluble Dietary Fiber in g/100g

4.3.1 Sample A (Methods: 1a, 2a, 3a)

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	11
Number of outliers	0
Mean	2,72
Median	2,90
Robust Mean (X)	**2,75**
Robust standard deviation (S*)	**0,493**
Number with 2 replicates	11
Repeatability SD (S_r)	0,227
Repeatability (CV_r)	8,39%
Reproducibility SD (S_R)	0,523
Reproducibility (CV_R)	19,3%
Target range:	
Target standard deviation $\sigma_{pt'}$	**0,421**
Target standard deviation (for Information)	0,094
lower limit of target range	**1,91**
upper limit of target range	**3,59**
Quotient $S^*/\sigma_{pt'}$	*1,17*
Standard uncertainty $U_{(Xpt)}$	*0,186*
Results in the target range	*10*
Percent in the target range	*91%*

Comments to the statistical characteristics:

The target standard deviation was calculated using data from a precision experiment (ASU §64 L 16.08-1) (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 slightly increased variability of results. Therefore the valuation was done by z'-scores considering the standard uncertainty. The quotient $S^*/\sigma_{pt'}$ was then < 2,0.
The robust standard deviation is comparable to those of prior PTs (see 3.6.3). The repeatability and reproducibility standard deviation are in the range of established values of the applied methods (see 3.6.2). The comparability of results is given.

91% of the results were in the target range.

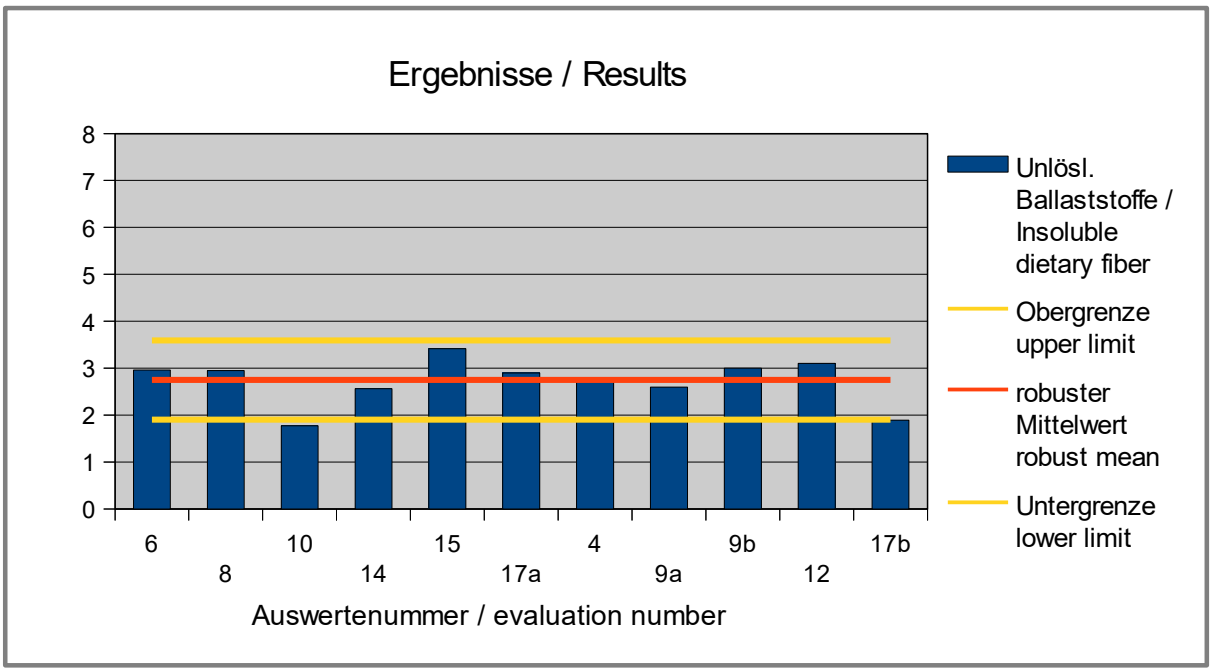

Abb. / Fig. 11: Ergebnisse unlösliche Ballaststoffe Probe A / Results insoluble dietary fiber sample A

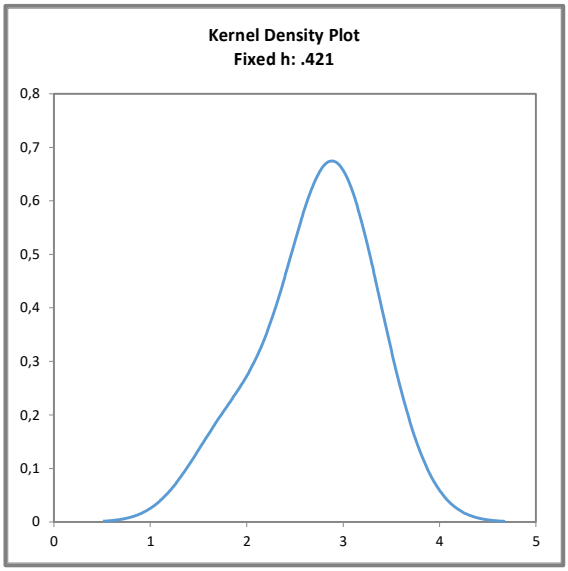

Abb. / Fig. 12:
Kerndichte-Schätzung der Ergebnisse (mit h = 0,75 x σ_{pt} von X_{pt})

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

Comment:
The kernel density shows almost a symmetrical distribution of results with a shoulder.

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Unlösl. Ballaststoffe / Insoluble dietary fiber	Abweichung [g/100g] Deviation [g/100g]	z'-Score (σ_{pt})	z-Score (Info)	Methode Method	Hinweis Remark
6	2,96	0,211	0,50	2,2	1a	
8	2,95	0,201	0,48	2,1	1a	
10	1,77	-0,979	-2,3	-10	1a	
14	2,56	-0,189	-0,45	-2,0	1a	
15	3,42	0,666	1,6	7,1	1a	
17a	2,90	0,151	0,36	1,6	1a	
4	2,74	-0,014	-0,03	-0,15	2a = 1a	Method 1a assigned by DLA
9a	2,60	-0,149	-0,35	-1,6	2a = 1a	Method 1a assigned by DLA
9b	3,00	0,251	0,60	2,7	3a	
12	3,10	0,351	0,83	3,7	3a	
17b	1,89	-0,859	-2,0	-9,1	3a	

(1) enzymatic-gravimetric methods like ASU L 00.00-18 (ASU L 17.03-1) or AOAC 991.43 and AOAC 985.29, in w hich low er results of inulin can occur
(1a + 1b) e.g. inulinase-reaction included
(2) enzymatic-gravimetric methods combined w ith liquid chromatography like AOAC 2009.01, w hich include inulin
(2a + 2b) e.g. w ithout inulinase-reaction
(3) Total Dietary Fiber, as Sum of Insoluble DF, High Molecular Weight Soluble DF, and Low Molecular Weight Soluble DF (AOAC 2017.16)
(3a) Insoluble Dietary Fiber (Insoluble DF)

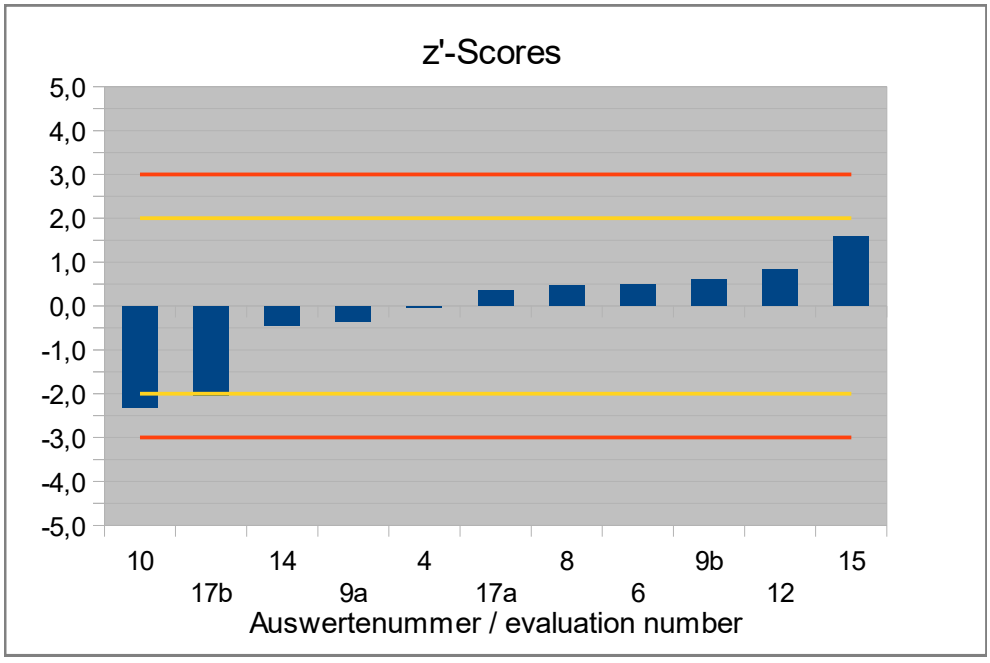

Abb. / Fig. 13: z'-Scores unlösliche Ballaststoffe Probe A / insoluble dietary fiber sample A

4.3.2 Sample B (Methods: 1a, 2a, 3a)

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	11
Number of outliers	-
Mean	2,64
Median	2,90
Robust Mean (X)	**2,75**
Robust standard deviation (S*)	**0,569**
Number with 2 replicates	10
Repeatability SD (S_r)	0,150
Repeatability (CV_r)	5,34%
Reproducibility SD (S_R)	0,445
Reproducibility (CV_R)	15,8%
Target range:	
Target standard deviation $\sigma_{pt'}$	**0,434**
Target standard deviation (for Information)	0,094
lower limit of target range	**1,88**
upper limit of target range	**3,61**
Quotient $S^*/\sigma_{pt'}$	*1,3*
Standard uncertainty $U_{(Xpt)}$	*0,214*
Results in the target range	*10*
Percent in the target range	*91%*

Comments to the statistical characteristics:

The target standard deviation was calculated using data from a precision experiment (ASU §64 L 16.08-1) (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 slightly increased variability of results. Therefore the valuation was done by z'-scores considering the standard uncertainty. The quotient $S^*/\sigma_{pt'}$ was then < 2,0.
The robust standard deviation is comparable to those of prior PTs (see 3.6.3). The repeatability and reproducibility standard deviation are in the range of established values of the applied methods (see 3.6.2). The comparability of results is given.

91% of the results were in the target range.

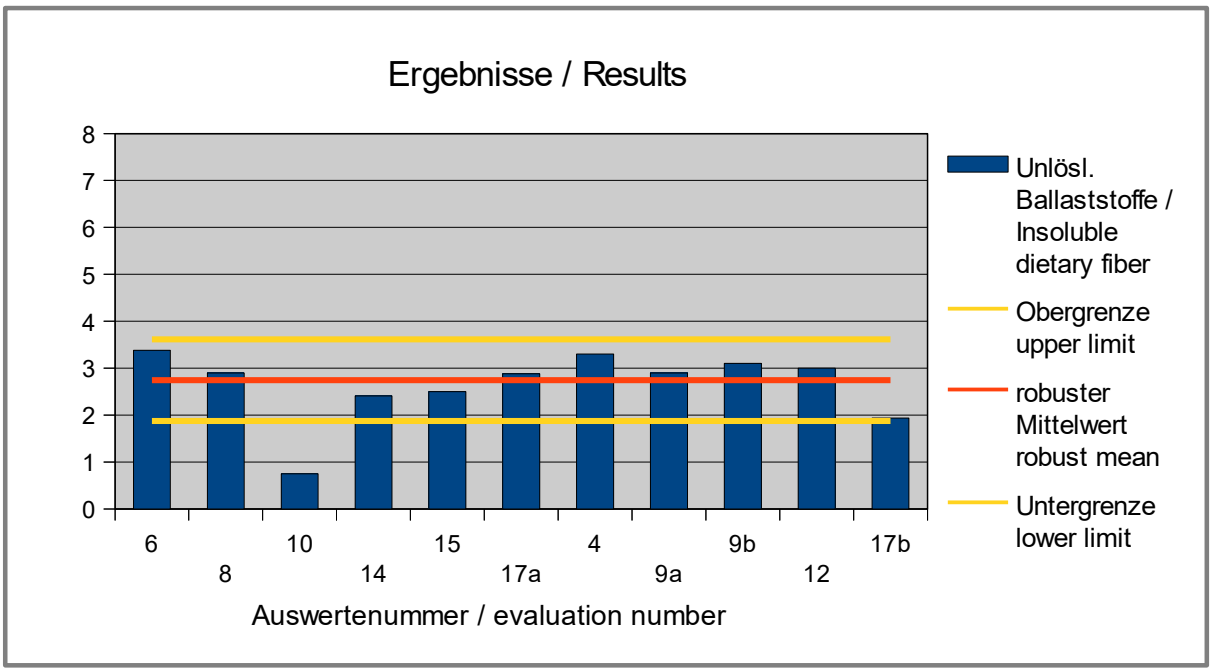

Abb. / Fig. 14: Ergebnisse unlösliche Ballaststoffe Probe B / Results insoluble dietary fiber sample B

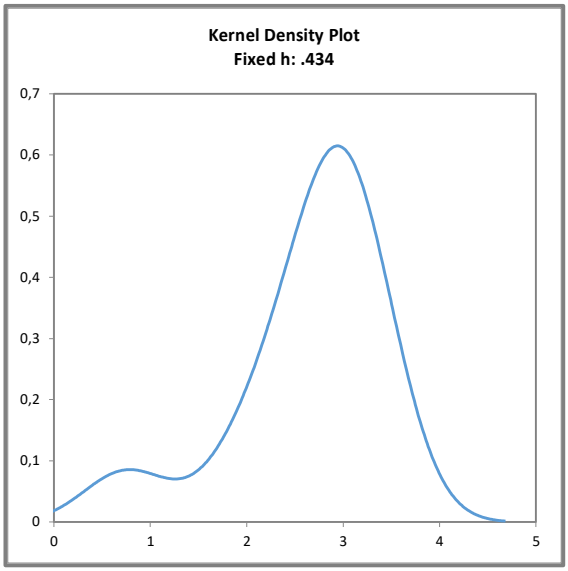

Abb. / Fig. 15:
Kerndichte-Schätzung der Ergebnisse (mit h = 0,75 x σ_{pt} von X_{pt})

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 small side peak.

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Unlösl. Ballaststoffe / Insoluble dietary fiber	Abweichung [g/100g] Deviation [g/100g]	z'-Score (σ_{pt})	z-Score (Info)	Methode Method	Hinweis Remark
6	3,38	0,634	1,5	6,7	1a	
8	2,90	0,154	0,36	1,6	1a	
10	0,75	-1,996	-4,6	-21	1a	
14	2,41	-0,336	-0,77	-3,6	1a	
15	2,50	-0,246	-0,57	-2,6	1a	
17a	2,88	0,134	0,31	1,4	1a	
4	3,30	0,554	1,3	5,9	2a = 1a	Method 1a assigned by DLA
9a	2,90	0,154	0,36	1,6	2a = 1a	Method 1a assigned by DLA
9b	3,10	0,354	0,82	3,8	3a	
12	3,00	0,254	0,59	2,7	3a	
17b	1,94	-0,806	-1,9	-8,5	3a	

(1) enzymatic-gravimetric methods like ASU L 00.00-18 (ASU L 17.03-1) or AOAC 991.43 and AOAC 985.29, in which lower results of inulin can occur
(1a + 1b) e.g. inulinase-reaction included
(2) enzymatic-gravimetric methods combined with liquid chromatography like AOAC 2009.01, which include inulin
(2a + 2b) e.g. without inulinase-reaction
(3) Total Dietary Fiber, as Sum of Insoluble DF, High Molecular Weight Soluble DF, and Low Molecular Weight Soluble DF (AOAC 2017.16)
(3a) Insoluble Dietary Fiber (Insoluble DF)

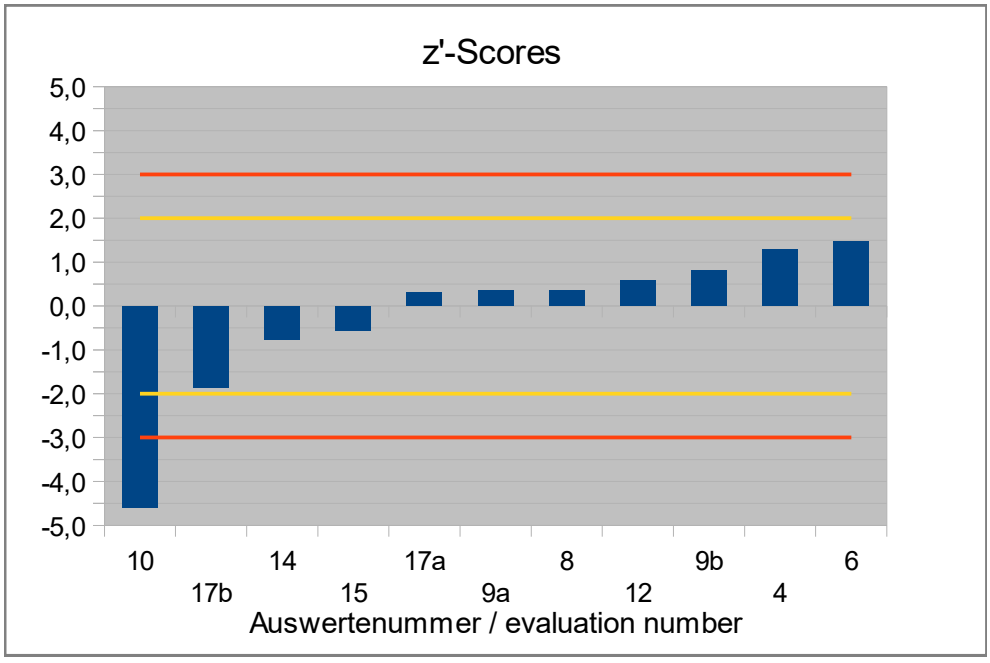

Abb. / Fig. 16: z'-Scores unlösliche Ballaststoffe Probe B / insoluble dietary fiber sample B

4.4 Insoluble Dietary Fiber in g/100g

4.4.1 Sample A (Methods: 1b, 2b, 3b+c)

Vergleichsuntersuchung / Proficiency Test

Comments:
The distribution of the results showed a highly increased variability within the methods (1b, 3b). And in some cases fewer than 5 results were available for the methods (3b, 3c). Therefore a statistical evaluation of the results was not carried out.

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer / Evaluation number	Lösliche Ballaststoffe / Soluble dietary fiber	Abweichung [g/100g] / Deviation [g/100g]	z'-Score (σ_{pt})	z-Score (Info)	Methode / Method	Hinweis / Remark
6	< 1,0				1b	
8	0,75				1b	
10	2,80				1b	
14	1,82				1b	
15	0,92				1b	
17a	2,64				1b	
4	1,56				2b = 1b	Method 1b assigned by DLA
9a	0,80				2b = 1b	Method 1b assigned by DLA
9b	1,20				3b	
12a	< 0,50				3b	
17b	2,44				3b	
9c	2,60				3c	
12b	3,12				3c	
17c	3,71				3c	

(1) enzymatic-gravimetric methods like ASU L 00.00-18 (ASU L 17.03-1) or AOAC 991.43 and AOAC 985.29, in w hich low er results of inulin can occur
(1a + 1b) e.g. inulinase-reaction included
(2) enzymatic-gravimetric methods combined w ith liquid chromatography like AOAC 2009.01, w hich include inulin
(2a + 2b) e.g. w ithout inulinase-reaction
(3) Total Dietary Fiber, as Sum of Insoluble DF, High Molecular Weight Soluble DF, and Low Molecular Weight Soluble DF (AOAC 2017.16)
(3b + 3c) Soluble Dietary Fiber: b) High Molecular Weight Soluble DF and c) Low Molecular Weight Soluble DF) (AOAC 2017.16)

4.4.2 Sample B (Methods: 1b, 2b, 3b+c)

Vergleichsuntersuchung / Proficiency Test

Comments:
The distribution of the results showed a highly increased variability within the methods (1b, 3b). And in some cases fewer than 5 results were available for the methods (3b, 3c). Therefore a statistical evaluation of the results was not carried out.

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer / Evaluation number	Lösliche Ballaststoffe / Soluble dietary fiber	Abweichung [g/100g] / Deviation [g/100g]	z'-Score (σ_{pt})	z-Score (Info)	Methode / Method	Hinweis / Remark
6	< 1,0				1b	
8	0,90				1b	
10	3,36				1b	
14	2,09				1b	
15	1,47				1b	
17a	2,53				1b	
4	1,78				2b = 1b	Method 1b assigned by DLA
9a	0,90				2b = 1b	Method 1b assigned by DLA
9b	1,40				3b	
12a	< 0,50				3b	
17b	2,35				3b	
9c	0,90				3c	
12b	1,70				3c	
17c	0,85				3c	

(1) enzymatic-gravimetric methods like ASU L 00.00-18 (ASU L 17.03-1) or AOAC 991.43 and AOAC 985.29, in which lower results of inulin can occur
(1a + 1b) e.g. inulinase-reaction included
(2) enzymatic-gravimetric methods combined with liquid chromatography like AOAC 2009.01, which include inulin
(2a + 2b) e.g. without inulinase-reaction
(3) Total Dietary Fiber, as Sum of Insoluble DF, High Molecular Weight Soluble DF, and Low Molecular Weight Soluble DF (AOAC 2017.16)
(3b + 3c) Soluble Dietary Fiber: b) High Molecular Weight Soluble DF and c) Low Molecular Weight Soluble DF) (AOAC 2017.16)

4.5 Inulin / Fructans in g/100g

4.5.1 Sample A (Methods: 5 + 6)

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	16
Number of outliers	0
Mean	2,86
Median	2,82
Robust Mean (X)	**2,85**
Robust standard deviation (S*)	**0,241**
Number with 2 replicates	15
Repeatability SD (S_r)	0,0569
Repeatability (CV_r)	2,00%
Reproducibility SD (S_R)	0,245
Reproducibility (CV_R)	8,63%
Target range:	
Target standard deviation σ_{pt}	**0,212**
Target standard deviation (for Information)	0,097
lower limit of target range	**2,43**
upper limit of target range	**3,28**
Quotient S/σ_{pt}*	*1,1*
Standard uncertainty $U_{(Xpt)}$	*0,075*
Results in the target range	*15*
Percent in the target range	*94%*

Comments to the statistical characteristics:

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 a low 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 repeatability and reproducibility standard deviation are in the range of established values of the applied methods (see 3.6.2). The comparability of results is given.

94% of the results were in the target range.

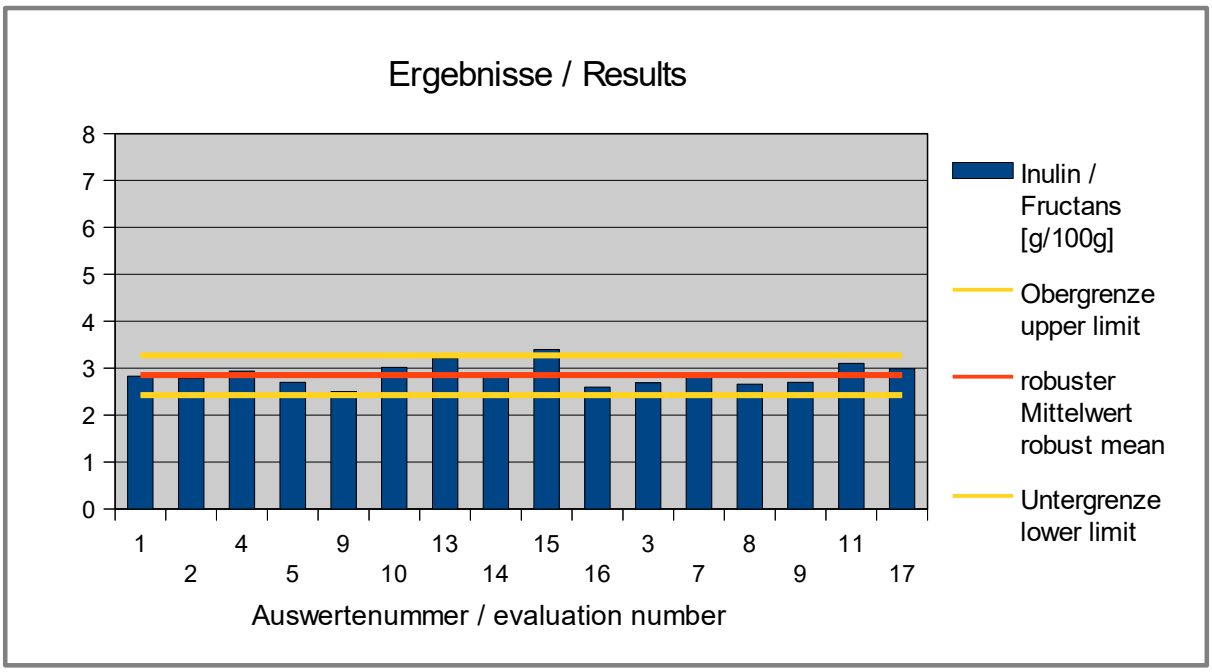

Abb. / Fig. 17: Ergebnisse Inulin/Fructane Probe A / Results inulin/fructans sample A

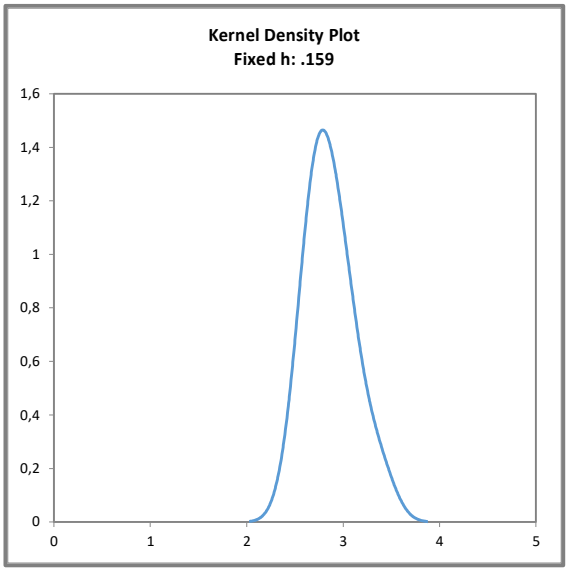

Abb. / Fig. 18:
Kerndichte-Schätzung der Ergebnisse (mit h = 0,75 x σ_{pt} von Xpt)

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

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

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Inulin / Fructans [g/100g]	Abweichung [g/100g] Deviation [g/100g]	z-Score (σ_{pt})	z-Score (Info)	Methode Method	Hinweis Remark
1	2,83	-0,022	-0,11	-0,23	5	
2	2,79	-0,062	-0,29	-0,64	5	
4	2,94	0,088	0,41	0,90	5	
5	2,70	-0,152	-0,72	-1,6	5	
9a	2,50	-0,352	-1,7	-3,6	5	
10	3,02	0,163	0,77	1,7	5	
13	3,21	0,358	1,7	3,7	5	
14	2,81	-0,042	-0,20	-0,44	5	
15	3,40	0,548	2,6	5,6	5	
16	2,60	-0,252	-1,2	-2,6	5	
3	2,69	-0,162	-0,77	-1,7	6	
7	2,89	0,038	0,18	0,39	6	
8	2,66	-0,192	-0,91	-2,0	6	
9b	2,70	-0,152	-0,72	-1,6	6	
11	3,10	0,248	1,2	2,5	6	
17	2,99	0,138	0,65	1,4	6	

(5) enzymatic methods (e.g. ASU L 00.00-94)

(6) liquid-chromatography methods after enzymatic treatment (e.g. ISO 22579 (IDF 241):2020/AOAC 2016.14)

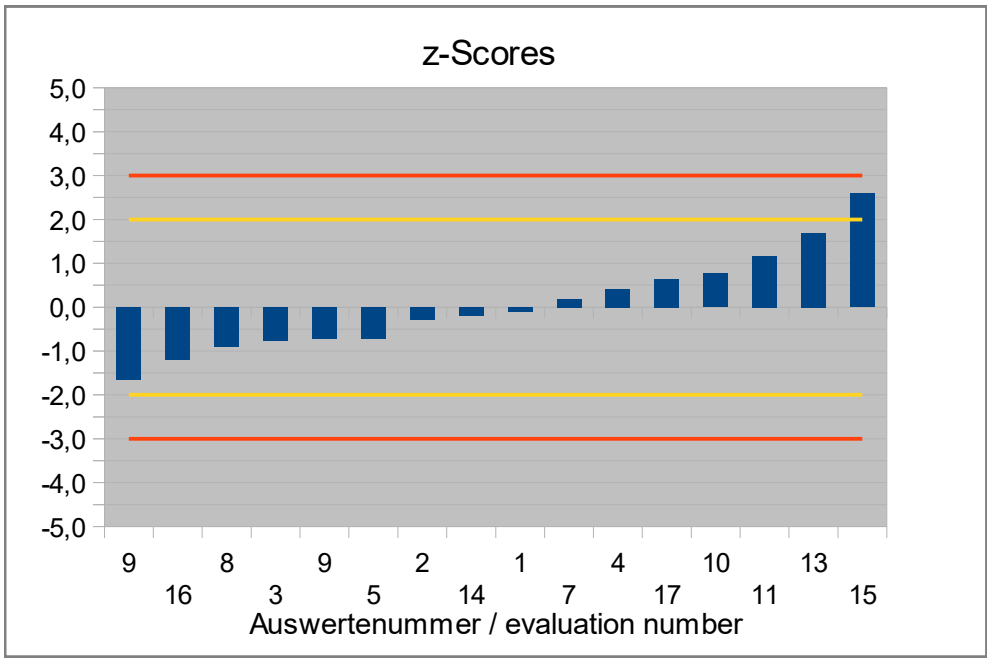

Abb. / Fig. 19: z-Scores Inulin/Fructane Probe A / inulin/fructans sample A

4.5.2 Sample B (Methods: 5 + 6)

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	8
Number of outliers	–
Mean	0,228
Median	0,175
Robust Mean (X)	**0,196**
Robust standard deviation (S*)	**0,0925**
Number with 2 replicates	8
Repeatability SD (S_r)	0,0120
Repeatability (CV_r)	5,31%
Reproducibility SD (S_R)	0,157
Reproducibility (CV_R)	69,8%
Target range:	
Target standard deviation $\sigma_{pt'}$	**0,0434**
Target standard deviation (for Information)	0,010
lower limit of target range	**0,109**
upper limit of target range	**0,282**
Quotient $S^*/\sigma_{pt'}$	*2,1*
Standard uncertainty $U_{(Xpt)}$	*0,041*
Results in the target range	*5*
Percent in the target range	*63%*

Comments to the statistical characteristics:

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 > 6,0. Therefore the evaluation of all methods was done by z'-score considering the standard uncertainty. The quotient $S^*/\sigma_{pt'}$ was then at 2,1.

No inulin was added to sample B, 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).

63% of the results were in the target range.

Note: For methods 5+6 the difference of the robust means for sample A (with addition of 3,05 g/100g inulin) and sample B is 2,65 g/100g.

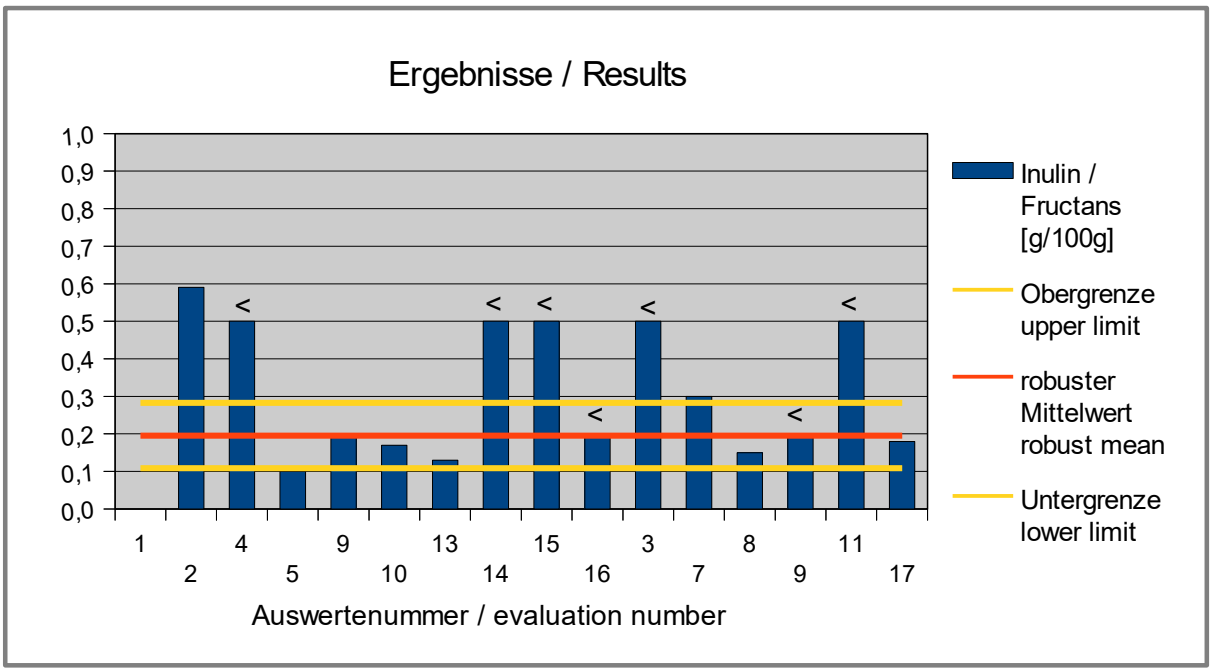

Abb. / Fig. 20: Ergebnisse Inulin/Fructane Probe B / Results inulin/fructans sample B

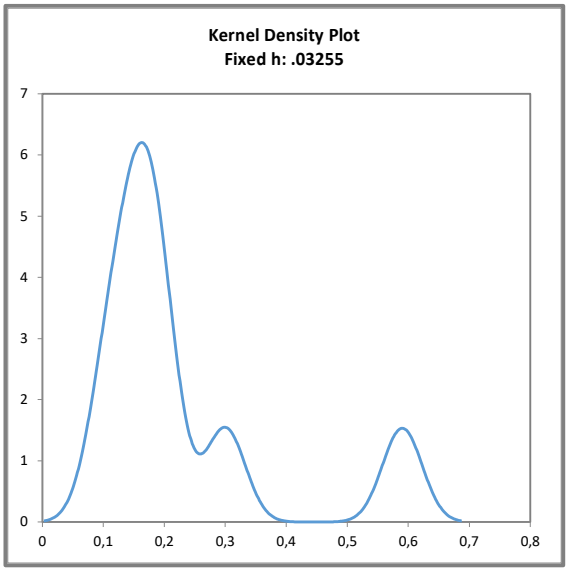

Abb. / Fig. 21:
Kerndichte-Schätzung der Ergebnisse (mit h = 0,75 x σ_{pt} von X_{pt})

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 side peaks.

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	Inulin / Fructans [g/100g]	Abweichung [g/100g] Deviation [g/100g]	z'-Score (σ_{pt})	z-Score (Info)	Methode Method	Hinweis Remark
1					5	
2	0,59	0,394	9,1	39	5	
4	< 0,50				5	
5	0,10	-0,096	-2,2	-9,6	5	
9a	0,20	0,004	0,10	0,4	5	
10	0,17	-0,026	-0,59	-2,6	5	
13	0,13	-0,066	-1,5	-6,6	5	
14	< 0,50				5	
15	< 0,50				5	
16	< 0,20				5	
3	< 0,50				6	
7	0,30	0,104	2,4	10,4	6	
8	0,15	-0,046	-1,0	-4,6	6	
9b	< 0,20				6	
11	< 0,50				6	
17	0,18	-0,016	-0,36	-1,6	6	

(5) enzymatic methods (e.g. ASU L 00.00-94)

(6) liquid-chromatography methods after enzymatic treatment (e.g. ISO 22579 (IDF 241):2020/AOAC 2016.14)

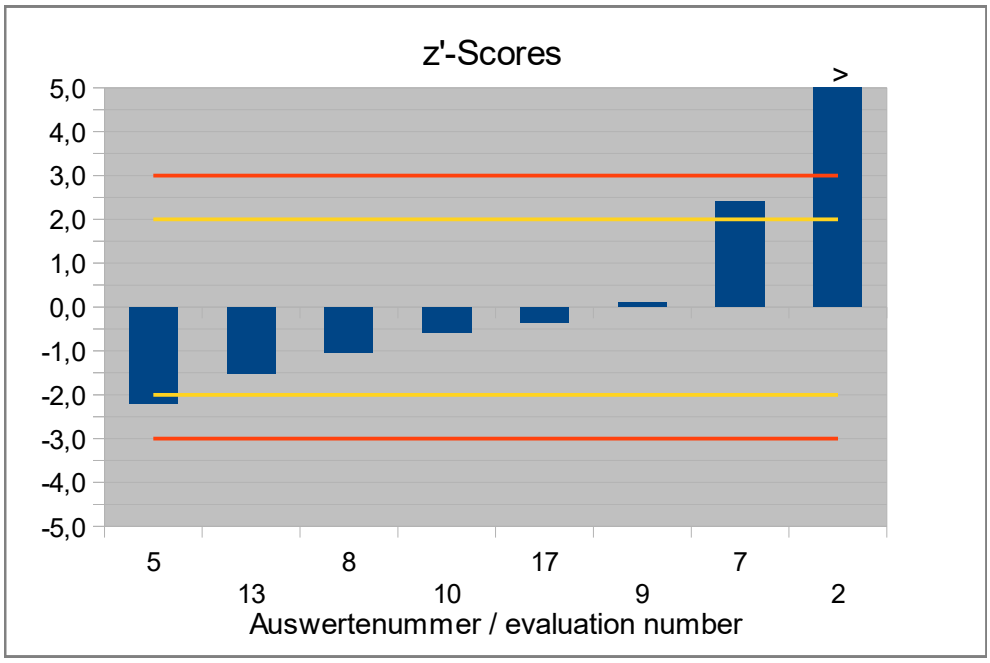

Abb. / Fig. 22: z'-Scores Inulin/Fructane Probe B / inulin/fructans sample B

4.4 Participant z-Scores: overview table

Evaluation number	TDF (1)°	TDF (1)°	TDF (2+3)	TDF (2+3)	IDF (1a,2a,3a)°	IDF (1a,2a,3a)°	Inulin / Fructans (5+6)	Inulin / Fructans (5+6)°
	Sample A	Sample B	Sample A	Sample B	Sample A	Sample B	Sample A	Sample B
1	1,3	1,6					-0,11	
2	1,6	-0,28					-0,29	9,1
3							-0,77	
4	-0,12	1,5			-0,03	1,30	0,41	
5	0,85	1,1					-0,72	-2,2
6	-0,74	-0,60			0,50	1,5		
7	-1,4	-0,75					0,18	2,4
8/8a	-1,2	-0,93			0,48	0,36	-0,91	-1,0
8b	-1,4	-0,93						
9/9a	-1,7	-1,1	-0,43	-0,01	-0,35	0,36	-1,7	0,10
9b			-0,68	-0,01	0,60	0,82	-0,72	
10	0,37	-0,36			-2,3	-4,6	0,77	-0,59
11/11a	1,5	0,94	0,67	0,81			1,2	
11b			-0,01	-0,48				
12	-3,4	-3,5			0,83	0,59		
13/13a	-0,13	0,19					1,7	-1,5
13b	0,49	0,49						
14	1,0	-0,15			-0,45	-0,77	-0,20	
15	-0,74	-0,69			1,6	-0,57	2,6	
16	0,31	0,00					-1,2	
17/17a	2,1	2,1	0,45	-0,31	0,36	0,31	0,65	-0,36
17b					-2,0	-1,9		

° z'-Score

(1 - 6) Methods see page 17

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

5. Documentation

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

5.1 Details by the participants

5.1.1 Primary Data

Parameter: Total Dietary Fiber without Inulin (Methods: 1)

Meth.	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 %	
4 = 1	1	g/100g	31.05.21	5,08	5	5,15	5,16	5,02	5,29		no		Testkit Total Dietary Fiber, Merck (1.12979.0001)
1	2	g/100g	07.06.21	5,25	5	5,5	4,15	4	4,3	0,5	no		AOAC 991.43
2 = 1	4	g/100g	10.05.21	4,295	4,48	4,11	5,08	5,03	5,13	2			calculation
1	5	g/100g	16.06.21	4,84	5,12	4,57	4,9	5,22	4,58		no		ASU § 64 LFGB, L 00.00-18, 2017-10
1	6	g/100g	20.05.21	3,95	3,88	4,01	3,98	4,04	3,93	1	no		AOAC 991.43 mod
1	7	g/100g	09.06.21	3,6	3,6	3,7	3,9	3,9	3,9		no		AOAC991.43
1	8a	g/100g	18.06.21	3,7	3,8	3,6	3,8	3,8	3,8	0,5	no		ASU L 00.00-18: 1997-01
4 = 1	8b	g/100g	18.06.21	3,6	3,6	3,6	3,8	3,8	3,8	0,5	no		ASU L 00.00-18: 1997-01
2 = 1	9	g/100g	04.06.21	3,4	3,7	3	3,7	3,9	3,5	0,5	no		AOAC 991.43
1	10	g/100g	09.06.21	4,57	4,81	4,33	4,105	4,21	4		no		ASU L 00.00-18 modified
1	11	g/100g	21.05.21	5,20			4,80						
1	12	g/100g	25.06.21	2,43	2,53	2,32	2,44	2,47	2,4	0,5	no		AOAC 991.43
1	13a	g/100g	18.05.//20.05.	4,29	4,31	4,26	4,4	4,32	4,48		no		PV 2202; anaolog ASU L 00.00-18, with addition of inulinase
4 = 1	13b	g/100g	18.05.//20.05.	4,64	4,65	4,62	4,56	4,7	4,42		no		PV 2202; anaolog ASU L 00.00-18, without addition of inulinase
1	14	g/100g	22.06.21	4,93	4,8	5,05	4,22	4,56	3,87	< 0,5	no		ASU L 00.00-18 : 1997-01 corr. 2002-12
1	15	g/100g	30.06.21	3,95	3,9	4, 00	3,93	3,84	4,02	0,1	no		
1	16	g/100g	17.06.21	4,54	4,49	4,58	4,3	4,26	4,36	0,50 g/100g	no		SOP M 1010 according to ASU L 00.00-18, mod.
1	17	g/100g	21/06	5,54	5,46	5,62	5,41	5,72	5,11		No		

Continuation:

Meth.	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	
4 = 1	1	40mm	G2	as per kit instructions					yes	Measurement by Ankom Fibreanalyzer
1	2							Referenzmaterial DLA 2020	yes	
2 = 1	4								yes	
1	5	30 mm	2	Bioquant Total Dietary Fiber Merck 1.12979.001					yes	
1	6								yes	
1	7	/	2	Megazyme kit	Megazyme kit	Megazyme kit	Novozyme SP230		no	results expressed on sample as such in g/100g and not defatted (<10%) before determination
1	8a	30 mm	40 - 90 µm	Merck Kit	Merck Kit	Merck Kit	Mix of exo- and en-do-Inulinase		yes	
4 = 1	8b	30 mm	40 - 90 µm	Merck Kit	Merck Kit	Merck Kit	Mix of exo- and en-do-Inulinase		yes	without Inulin, GS-Method
2 = 1	9	30 mm	40-90um	Megazyme E-Blaam	Megazyme E-BSPRT	Megazyme E-AMGDF	no inulinase	no	–	
1	10								yes	
1	11								yes	
1	12								yes	
1	13a	30 mm	40 - 100 µm	Merck 1.12979.001; LOT: HC041950			Megazyme Fructa-nase Mixture; LOT: 170301a		yes	
4 = 1	13b	30 mm	40 - 100 µm	Merck 1.12979.001; LOT: HC041950					yes	
1	14	40 mm	40-100 µm	Dietary Fiber kit from Merck					yes	sample defatted with PE
1	15	Behrotest CFT 30 3 cm	Porosity 2	Testkit from Megazyme					yes	behrotest filter crucible 30 ml
1	16	40mm	Por. 3 16-40 µm / 25-50 µm	3000 U/mL; 54 U/mg	50 mg/mL	3,26 U/mL (soluble starch); 200 U/mL 8p-NP Bets-Mal-toside)	Fructanase Maxture Exo-Inulinase 2000 U/mL + Endo-Inuli-nase 80 U/mL ; In 50% Glycerol	control chart Weetabix	yes	
1	17								Yes	

Parameter: Insoluble and soluble Dietary Fiber (Methods: 1a + 1b)

Meth.	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 %	
1a	6	g/100g	25.05.21	2,96	2,99	2,92	3,38	3,35	3,42	1	no		AOAC 991.43 mod
1a	8	g/100g	18.06.21	2,95	3	2,9	2,9	2,7	3	0,5	no		ASU L 00.00-18: 1997-01
1a	10	g/100g	23.06.21	1,77	1,73	1,81	0,75	0,68	0,82		no		ASU L 00.00-18 modified
1a	14	g/100g	22.06.21	2,56	2,44	2,67	2,41	2,56	2,25	< 0,5	no		ASU L 00.00-18 : 1997-01 corr. 2002-12
1a	15	g/100g	18.06.21	3,415	3,25	3,58	2,5	2,47	2,53	0,1	no		
1a	17	g/100g	21/06	2,9	3,02	2,79	2,88	3,05	2,71		No		
2a = 1a	4	g/100g	10.05.21	2,735	2,46	3,01	3,3	3,22	3,38	1			ASU L 00.00-18, 2017-10
2a = 1a	9	g/100g	04.06.21	2,6	2,9	2,2	2,9	3	2,7	0,3	no		AOAC 991.43
1b	6	g/100g	-	<1			<1			1	no		
1b	8	g/100g	18.06.21	0,75	0,8	0,7	0,9	1,1	0,8	0,5	no		ASU L 00.00-18: 1997-01
1b	10	g/100g	23.06.21	2,8	3	2,6	3,355	3,39	3,32		no		ASU L 00.00-18 modified
1b	14	g/100g	22.06.21	1,82	1,61	2,03	2,09	2,15	2,03	< 0,5	no		ASU L 00.00-18 : 1997-01 corr. 2002-12
1b	15	g/100g	18.06.21	0,92	0,91	0,92	1,47	1,45	1,48	0,1	no		
1b	17	g/100g	21/06	2,64	2,44	2,83	2,53	2,67	2,4		No		
2b = 1b	4	g/100g	10.05.21	1,56	2,02	1,1	1,78	1,81	1,75	1			ASU L 00.00-18, 2017-10
2b = 1b	9	g/100g	04.06.21	0,8	0,8	0,8	0,9	0,9	0,8	0,2	no		AOAC 991.43

Continuation:

Meth.	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	
1a	6								yes	
1a	8	30 mm	40 - 90 µm	Merck Kit	Merck Kit	Merck Kit	Mix of exo- and en-do-Inulinase		yes	
1a	10								yes	
1a	14	40 mm	40-100 µm	Dietary Fiber kit from Merck					yes	sample defatted with PE
1a	15	Behrotest CFT 30 3 cm	Porosity 2	Testkit from Megazyme					yes	behrotest filter crucible 30 ml
1a	17								Yes	
2a = 1a	4								yes	
2a = 1a	9	30 mm	40-90um	Megazyme E-Blaam	Megazyme E-BSPRT	Megazyme E-AMGDF	no inulinase	no	–	
1b	6								yes	Calculation from Total and insoluble
1b	8	30 mm	40 - 90 µm	Merck Kit	Merck Kit	Merck Kit	Mix of exo- and en-do-Inulinase		yes	
1b	10								yes	
1b	14	40 mm	40-100 µm	Dietary Fiber kit from Merck					yes	sample defatted with PE
1b	15	Behrotest CFT 30 3 cm	Porosity 2	Testkit from Megazyme					yes	behrotest filter crucible 30 ml
1b	17								Yes	
2b = 1b	4								yes	
2b = 1b	9	30 mm	40-90um	Megazyme E-Blaam	Megazyme E-BSPRT	Megazyme E-AMGDF	no inulinase	no	–	

Parameter: Total Dietary Fiber including Inulin (Method: 2)

Meth.	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 %	
4 = 2	9	g/100g	04.06.21	7	6,9	7,1	5,4	5,3	5,4	0,3	no		AOAC 2009.01
2	11	g/100g	12.05.21	8,30			6,10						

Meth.	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	
4 = 2	9	30 mm	40-90um	Megazyme E-PANAA	Megazyme E-BSPRT	Megazyme E-AMGDF	no inulinase	no		
2	11								yes	

For information from other participants, see Method 1.

Parameter: Insoluble and soluble Dietary Fiber (Methods: 2a + 2b)

For information from other participants, see Methods 1a and 1b.

Parameter: Total Dietary Fiber, IDF, HMW SDF and LMW SDF (Methods: 3, 3a, 3b + 3c)

Meth.	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 %	
3	9	g/100g	07.06.21	6,7	6,6	6,7	5,4	5,4	5,3	0,5	no		AOAC 2011.25
3	11	g/100g	21.06.21	7,50			5,00						
3	17	g/100g	22/06	8,04	8,17	7,9	5,14	5,11	5,17		No		
3a	9	g/100g	07.06.21	3	2,9	3	3,1	3	3,1	0,2	no		AOAC 2011.25
3a	12	g/100g	11.05.21	3,1	3,1	3,1	3	2,9	3	0,5	no		AOAC2009.01
3a	17	g/100g	22/06	1,89	1,73	2,04	1,94	1,9	1,97		No		
3b	9	g/100g	07.06.21	1,2	1,3	1	1,4	1,5	1,3	0,2	no		AOAC 2011.25
3b	12	g/100g	11.05.21	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	0,5	no		AOAC2009.01
3b	17	g/100g	22/06	2,44	2,63	2,24	2,35	2,37	2,33		No		
3c	9	g/100g	07.06.21	2,6	2,4	2,7	0,9	0,9	0,9	0,3	no		AOAC 2011.25
3c	12	g/100g	01.06.21	3,12	3,2	3,05	1,7	1,76	1,64	0,32	no		AOAC2009.01
3c	17	g/100g	22/06	3,71	3,81	3,62	0,85	0,84	0,87		No		

Continuation:

Meth.	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	
3	9	30 mm	40-90um	Megazyme E-PANAA	Megazyme E-BSPRT	Megazyme E-AMGDF	no inulinase	no	–	
3	11									
3	17								Yes	
3a	9	30 mm	40-90um	Megazyme E-PANAA	Megazyme E-BSPRT	Megazyme E-AMGDF	no inulinase	no	–	
3a	12								yes	
3a	17								Yes	
3b	9	30 mm	40-90um	Megazyme E-PANAA	Megazyme E-BSPRT	Megazyme E-AMGDF	no inulinase	no	–	
3b	12								yes	
3b	17								Yes	
3c	9	30 mm	40-90um	Megazyme E-PANAA	Megazyme E-BSPRT	Megazyme E-AMGDF	no inulinase	no	–	
3c	12							NIST 3233	yes	
3c	17								Yes	

Parameter: Total Dietary Fiber, other Methods (4)

For information from other participants, see Methods 1 and 2.

Parameter: Inulin / Fructans (Methods: 5 + 6)

Meth.	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 %	
5	1	g/100g	20.05.21	2,83	2,8	2,85	-	-	-		no	-	ASU L 00.00-94 mod.
5	2	g/100g	07.06.21	2,79	2,78	2,79	0,59	0,58	0,59	0,05	no		AOAC 997.08: „Fructans in Food Products (Ion Exchange Chromatographic Method)“, modified
5	4	g/100g	10.05.21	2,94	2,95	2,93	<0,50	<0,50	<0,50	0,5			ASU L 00.00--94, 2006-09
5	5	g/100g	16.06.21	2,7	2,686	2,71	0,1	0,11	0,09		no		ASU § 64 LFGB, L 00.00-94, 2006-09
5	9	g/100g	03.06.21	2,5	2,5	2,4	0,2	0,2	0,2	0,2	no		AOAC 999.03 modified
5	10	g/100g	01.06.21	3,015	3,02	3,01	0,17	0,17	0,17		no		ASU L 00.00-94
5	13	g/100g	02.06.	3,21	3,14	3,27	0,13	0,14	0,11		no		PV 2202; anaolog ASU L 00.00-94 modified
5	14	g/100g	23.06.21	2,81	2,87	2,74	< 0,5	< 0,5	< 0,5	< 0,5	no		ASU L 00.00-94 : 2006-09
5	15	g/100g	02.06.21	3,4	3,5	3,3	<0,5	<0,5	<0,5	0,5	no		HH-MA-M 02-051, HPLC: 2008-10ªÅ
5	16	g/100g	05.07.21	2,6	2,59	2,61	<0,20	<0,20	<0,20	0,20 g/100g	no		ASU L 00.00-94 mod.
6	3	g/100g	08.06.21	2,69	2,72	2,66	<0,5	<0,5	<0,5	0,5			internal Method LC-RI
6	7	g/100g	27 may -	2,89	2,88	2,9	0,3	0,31	0,29		no		AOAC997.08 with GC
6	8	g/100g	07.06.21	2,66	2,63	2,69	0,15	0,15	0,14	0,1	no		AOAC 997.08: 2013-08, mod.
6	9	g/100g	14.06.21	2,7	2,7	2,7	<0.2	<0.2	<0.2	0,2	no		AOAC2016.14
6	11	g/100g	01.06.21	3,10			<0,5%						
6	17	g/100g	13/06	2,99	2,97	3,01	0,18	0,19	0,17		No		

Continuation:

Meth.	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	
5	1	Test-Kit r-Biopharm No. 10 716 260 035	-	-	according to ASU L 00.00-94 mod. (particle size specification is neglected)		yes	Inulinase: Sigma Aldrich
5	2		Carbopac™ PA20; 3 x 150 mm	PAD	-	Reference material DLA 2020	yes	with modifications as follows: Glucose content calculated on basis of degree of polymerization. Other composition of eluent. Gradient program shortened from 83 to 47 min
5	4	r-biopharm					yes	
5	5	r-biopharm 10716260035					yes	
5	9	Megazyme	Thermo PA1	PAD			yes	
5	10	r-biopharm					no	
5	13	r-biopharm 10139106035; LOT: 42288200	Metrohm Metrosep Carb 2 with pre-column	PAD 35°C	determination of saccharose by IC-PAD		yes (IC-part applied for)	
5	14						yes	
5	15		Dr. Maisch Reprosil 100 NH2, 5um, 250*4mm	RID			yes	too less material
5	16	Boehringer Mannheim from R-Biopharm	-	-	according to §64 LFGB, L 00.00-94	control chart Weetabix	yes	Since it is a photometric method according to § 64 LFGB, L 00.00-94, there is no HPLC column and detector.
6	3				enzymatic treatment, determination of released sugars by HPLC		yes	as Inulin
6	7			FID	after silylation	standards+reference inulin	no	
6	8		Carbopac PA20	electrochemical (AEC)	determination of pre-exis. sugars	external calibration, cert. reference material	no	as Inulin
6	9	Megazyme	Thermo PA1	PAD			no	
6	11						no	Internal method - AOAC 997.08
6	17						Yes	

5.2 Homogeneity

5.2.1 Mixture homogeneity before bottling

Microtracer Homogeneity Test
DLA ptAU01 (2021) Sample A

Weight whole sample	2,79	kg
Microtracer	FSS-rot lake	
Particle size	75 – 300	µm
Weight per particle	2,0	µg
Addition of tracer	27,2	mg/kg

Result of analysis

Sample	Weight [g]	Particle number	Particles [mg/kg]
1	5,02	69	27,5
2	4,99	71	28,5
3	4,96	61	24,6
4	5,04	61	24,2
5	5,00	62	24,8
6	4,99	55	22,0
7	4,95	63	25,5
8	5,01	50	20,0

Poisson distribution		
Number of samples	8	
Degree of freedom	7	
Mean	61,5	Particle
Standard deviation	6,82	Particle
χ^2 (CHI-Quadrat)	5,29	
Probability	**62**	%
Recovery rate	91	%

Normal distribution		
Number of samples	8	
Mean	24,6	mg/kg
Standard deviation	2,73	mg/kg
rel. Standard deviaton	11,1	%
Horwitz standard deviation	9,88	%
HorRat-value	**1,1**	
Recovery rate	91	%

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 ptAU01 - 2021***
PT name	***Dietary Fiber, soluble / insoluble Fiber and Inulin in Cereal Product***
*Sample matrix**	*Samples A + B: Rusks / Ingredients: spelled flour, whole spelled meal, sunflower oil, yeast, salt, coriander and inulin (one of the two samples)*
Number of samples and sample amount	*2 different samples A + B, 40 g each.*
Storage	*Samples A + B:* *room temperature (PT period)*
Intentional use	*Laboratory use only (quality control samples)*
Parameter	*quantitative: total dietary fiber, soluble / insoluble 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* – *DLA-sample-numbers (for sample I and II)* – *Limit of detection* – *Assignment incl. Recovery* – *Recovery with the same matrix* – *Method is accredited*
Result submission	*The result submission file should be sent by e-mail to:* ***pt@dla-lvu.de***
Last Deadline	***the latest July 02nd 2021***
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.*

* 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
		Germany
		Germany
		Germany
		Germany
		SWEDEN
		NETHERLANDS
		Germany
		Germany
		Germany
		Germany
		BELGIUM
		Germany
		Germany
		BELGIUM
		Germany
		VIETNAM
		SPAIN

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

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

7. Index of references

1. DIN EN ISO/IEC 17025:2005; Allgemeine Anforderungen an die Kompetenz von Prüf- und Kalibrierlaboratorien / General requirements for the competence of testing and calibration laboratories
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18. ASU §64 LFGB: L 00.00-18: Bestimmung der Ballaststoffe in Lebensmitteln [Determination of dietary fieber in food]
19. ASU § 64 LFGB L 17.03-1 Bestimmung der Ballaststoffe in Mischbrot [Determination of dietary fieber in mixed-type bread].
20. ASU § 64 LFGB L 00.00-94 Bestimmung von Inulin in Lebensmitteln – Enzymatisches Verfahren [Determination of inulin in food – Enzymatic method]
21. Determination of total dietary fiber (CODEX definition) by Enzymatic-Gravimetric method and liquid chromatography: Collaborative Study, McCleary et al. (2010), J AOAC Int 93:221
22. LEITFADEN FÜR ZUSTÄNDIGE BEHÖRDEN – KONTROLLE DER EINHALTUNG DER EU-RECHTSVORSCHRIFTEN: Verordnung (EU) Nr. 1169/2011 über Information der Verbraucher über Lebensmittel (Dezember 2012) / GUIDANCE DOCUMENT FOR COMPETENT AUTHORITIES FOR THE CONTROL OF COMPLIANCE WITH EU LEGISLATION ON: Regulation (EU) No 1169/2011 on the provision of food information to consumers (December 2012)

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