Proficiency Tests

DLA

**food
cosmetics
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Evaluation Report
proficiency test

DLA 35/2017

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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Unteraufträge *Subcontractors*	Die Prüfung der Gehalte, Homogenität und Stabilität von EP-Parametern wird von DLA im Unterauftrag vergeben. The analysis of the content, homogeneity and stability of PT-parameters are 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 is a common in commerce rusk from a European supplier with added inulin. The basic matrix rusk was crushed (mesh < 1,5 mm) and an aliquot was mixed and homogenized with prior sieved inulin (mesh 400 µm). Afterwards additional basic matrix rusk was added in portions in three steps and each homogenized mechanically until the total amount was reached.

Afterwards the samples were packaged in portions to approximately 20 g into metallised PET film bags. The portions were numbered chronologic-ally.

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

Table 1: Composition of DLA-Samples

Ingredients	Content

Rusk
        Ingredients: Wheat flour, sugar, vegetable fats and
        oils, yeast, salt, wheat malt flour, wheat gluten
 	
        Nutrients per 100 g: fat 5,2 g, carbohydrates 72 g
        thereof sugar 14 g, protein 12 g
	96,9 g/100g *

- thereof dietary fiber (5,7 %) **	5,5 g/100g **
Inulin	3,08 g/100g *

*Ingredient contents according to gravimetric mixture
** Ingredient contents according to declaration (by AOAC-Method 2009.01)

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 samples showed a probability of 83%. Additionally particle number results were converted into concentrations, statistically evaluated according to normal distribution and compared to the standard deviation according to Horwitz. This gave a Hor-Rat value of 0,82 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 and inulin they are 8,5 % and 3,3 %. Thus they were similar to the repeatability standard deviations of the official methods (ASU L 00.00-18 / L 17.03-1 and L 00.00-94, see. 3.6.2) (see Tab. 3) [16-19]. The repeatability standard deviations of the participants' results are given in the documentation in the statistic data (see 4.1 and 4.7).

Furthermore, the homogeneity was characterized by the **trend line function of participants' results for chronological bottled single samples**. The maximum deviations from the mean value of the trend line was 40% of the target standard deviation for dietary fiber and 25% for inulin (s. 5.2 homogeneity) and can therefore be regarded as slightly increased for dietary fiber and low for 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

The experience with various DLA reference materials showed good storage stability with respect to the durability of the sample (spoilage) and the content of the PT parameters dietary fiber and inulin for comparable food matrices and water activity (a_w value <0,5). The stability of sample material is therefore given during the investigation period under consideration of given storage conditions.

2.2 Sample shipment and information to the test

Two portions of test material were sent to every participating laboratory in the 7th week of 2017. The testing method was optional. The tests should be finished at 31st March 2017 the latest.

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

The two portions are identical samples of a ground common in commerce rusk with added inulin.

Please note the attached information on the proficiency test.
(see documentation, section 5.4 Information on the PT)

2.3 Submission of results

The participants submitted their results in standard forms, which have been handed out with the samples (by email).

The finally calculated concentrations of the parameter as average of duplicate determinations of both numbered samples were used for the statistical evaluation. For the calculation of the repeatability- and reproducibility standard deviation the single values of the double determination were used.

Queried and documented were single results, recovery and the used testing methods. In case participants submitted several results for the same parameter obtained by different methods these results were evaluated with the same evaluation number with a letter as a suffix and indication of the related method.

12 out of 13 participants submitted the results in time. One participant obtained a sample by subsequent delivery and with submission extension in consultation with DLA.

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].

The condition is that the majority of the participants' results show a normal distribution or are distributed unimodal and symmetrically. To this end, an examination of the distribution is carried out, inter alia, using the kernel density estimate [3, 12].

In case there are indications for sources of higher variability such as a bimodal distribution of results, a cause analysis is performed. Frequently different analytical methods may cause an anomaly in results' distribution. If this is the case, separate evaluations with own assigned values (X_{pti}) are made whenever possible.

The statistical evaluation is carried out for all the parameters for a minimum of 7 values are present.

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 PT´s may differ from reproducibility standard deviations of ring trials, because the participating laboratories of a PT generally use different internal conditions and methods for determining the measured values.
In the present evaluation, the specification of the reproducibility standard deviation, therefore, does not refer to a specific method, but characterizes approximately the comparability of results between the laboratories, assumed the effect of homogeneity and stability of the sample are negligible.

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

3.5 Exclusion of results and outliers

Before statistical evaluation obvious blunders, such as those with incorrect units, decimal point errors, and results for a another proficiency test item can be removed from the data set [2]. Even if a result clearly deviates from the robust mean (e.g. factor >10) and has an influence on the robust statistics, a result can be excluded from statistical evaluation [3].

All results should be given at least with 2 significant digits. Specifying 3 significant digits is usually sufficient.

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

Results are identified as outliers by the use of robust statistics. If a value deviates from the robust mean by more than 3 times the robust standard deviation, it is classified as an outlier [3]. Detected outliers are stated for information only, when z-score are < -2 or > 2. Due to the use of robust statistics outliers are not excluded, provided that no other reasons are present [3].

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 dietary fiber and inulin the target standard deviation from section 3.6.2 (precision experiment) was applied. Additionally the standard uncertainty was considered by valuating with z´-scores (see 3.8).

Due to the number of <7 results for all other parameters, no statistical evaluation was performed.

3.6.1 General model (Horwitz)

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

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

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

3.6.2 Precision experiment

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

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

The relative repeatability standard deviations (RSD_r) and relative reproducibility standard deviation (RSD_R) given in Table 2 were determined in ring tests using the indicated methods.
The resulting target standard deviations σ_{pt}, which were identified there, were used to evaluate the results and to provide additional information for the statistical data.

Table 2: Relative repeatability standard deviations (RSD_r) and relative reproducibility standard deviation (RSD_R) according to selected evaluations of tests for precision and the resulting target standard deviation σ_{pt} [16, 17, 18, 19]

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 %[1]	ASU §64 L 17.03-1
Insoluble Dietary Fiber (enzymatic-gravimetric)	Mixed rye bread	5,45	5,14 %	8,44 %	7,6 %	ASU §64 L 17.03-1
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 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-gravimetric + liquid chromatography)	Whole grain bread Whole grain paste	11,6 12,7	12,3 % 4,47 %	18,0 % 11,3 %	15,8 % 10,8 %	AOAC 2009.01

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

For the determination of **total dietary fiber** the participants used 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 [20, 21].

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 [20, 21].

The method AOAC 2009.01 was only used by one participant in combination with the inulin determination for calculation of the total dietary fiber content after inulinase reaction.

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.

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].

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

<u>Table 3:</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
Soluble Diet-ary Fiber	Cereal product	2,57	0,694	27,0	–	DLA 26/2015
Soluble Diet-ary Fiber	Cereal product	2,17	0,380	17,5	–	DLA 36/2016
Soluble Diet-ary Fiber	Cereal product	1,17	0,896	76,6	–	DLA 35/2017
Insoluble Di-etary Fiber	Cereal product	3,31	1,06	32,0	–	DLA 26/2015
Insoluble Di-etary Fiber	Cereal product	6,06	0,460	7,59	–	DLA 36/2016
Insoluble Di-etary Fiber	Cereal product	2,99	0,965	32,3	–	DLA 35/2017
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

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

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

For information the z-scores below are calculated with a target standard deviation of 25%:

   i)   **z-Score - z_{ALL}**   (with respect to all methods)
   ii)   **z-Score - $z_{METHOD\ i}$** (with respect to single methods)

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. For example a fault isolation or a root cause analysis through the examination of transmission error or an error in the calculation, in the trueness and precision must be performed and if necessary appropriate corrective measures should be applied [3].

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

3.8 z'-Score

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

The calculation is performed by:

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

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

3.10 Quotient S*/σ_{pt}

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

3.11 Standard uncertainty of the assigned value

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

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

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

4. Results

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

In the first table the characteristics are listed:

Statistic Data
Number of results
Number of outliers
Mean
Median
Robust mean(X_{pt})
Robust standard deviation (S*)
Number with m replicate measurements
Repeatability standard deviation (S$_r$)
Coefficient of Variation (CV$_r$)in %
Reproducibility standard deviation (S$_R$)
Coefficient of Variation (CV$_R$)in %
Target range:
Target standard deviation σ_{pt} or σ_{pt}'
Target standard deviation for information
lower limit of target range $(X_{pt} - 2\sigma_{pt})$ or $(X_{pt} - 2\sigma_{pt}')$ *
upper limit of target range $(X_{pt} + 2\sigma_{pt})$ or $(X_{pt} + 2\sigma_{pt}')$ *
Variation coefficient V$_K$ in %
Quotient S/σ_{pt} or S*/σ_{pt}'*
Standard uncertainty U(X_{pt})
Quotient U(X_{pt})/σ_{pt} or U(X_{pt})/σ_{pt}'
Number of results in the target range
Percent in the target range

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

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

Auswerte-nummer Evaluation number	Parameter [Einheit / Unit]	Abweichung Deviation	z-Score σ$_{pt}$	z-Score (Info)	Hinweis Remark

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

4.1 Total Dietary Fiber in g/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	9
Number of outliers	0
Mean	4,36
Median	4,20
Robust Mean (X)	**4,32**
Robust standard deviation (S*)	**1,06**
Number with 2 replicates	9
Repeatability SD (S_r)	0,367
Repeatability (CV_r)	8,45%
Reproducibility SD (S_R)	1,05
Reproducibility (CV_R)	24,3%
Target range:	
Target standard deviation σ_{pt}'	**0,489**
Target standard deviation (for Information)	0,139
lower limit of target range	**3,34**
upper limit of target range	**5,30**
Quotient S*/σ_{pt}'	*2,2*
Standard uncertainty $U_{(Xpt)}$	*0,443*
Quotient $U_{(Xpt)}$/σ_{pt}'	*0,91*
Results in the target range	*6*
Percent in the target range	*67%*

Comments:

The target standard deviation was evaluated according to 3.6.2 by characteristics of a precision experiment (ASU §64 L 17.03-1). For information the target standard deviation using the general model of Horwitz is given (s. 3.6.1).

Exclusively enzymatic-gravimetric methods were used by the participants. The distribution of results showed an increased variability with a quotient S*/σ_{pt} of 5,2. Therefore valuation was done considering the standard uncertainty by z'-score. Then the quotient S*/σ_{pt}' of 2,2 was only slightly increased. The robust standard deviation and the repeatability and reproducibility standard deviation are comparable to those of prior PT´s (see 3.6.3). The comparability of results is given.

The quotient $U(X_{pt})$/σ_p of 0,9 is increased, but is acceptable on the basis of the other characteristics and the capability of applied methods.

67% of the results were in the target range.

Compared to the tolerances given in the guideline for the regulation (EU) No 1169/2011 for nutrition information all results, except the excluded result, were in the required range of ±2g for contents < 10 g/100g [22].

The robust mean of participant results is below the excepted value of approx. 5,5 g/100g for total dietary fiber without inulin (s. p. 4).
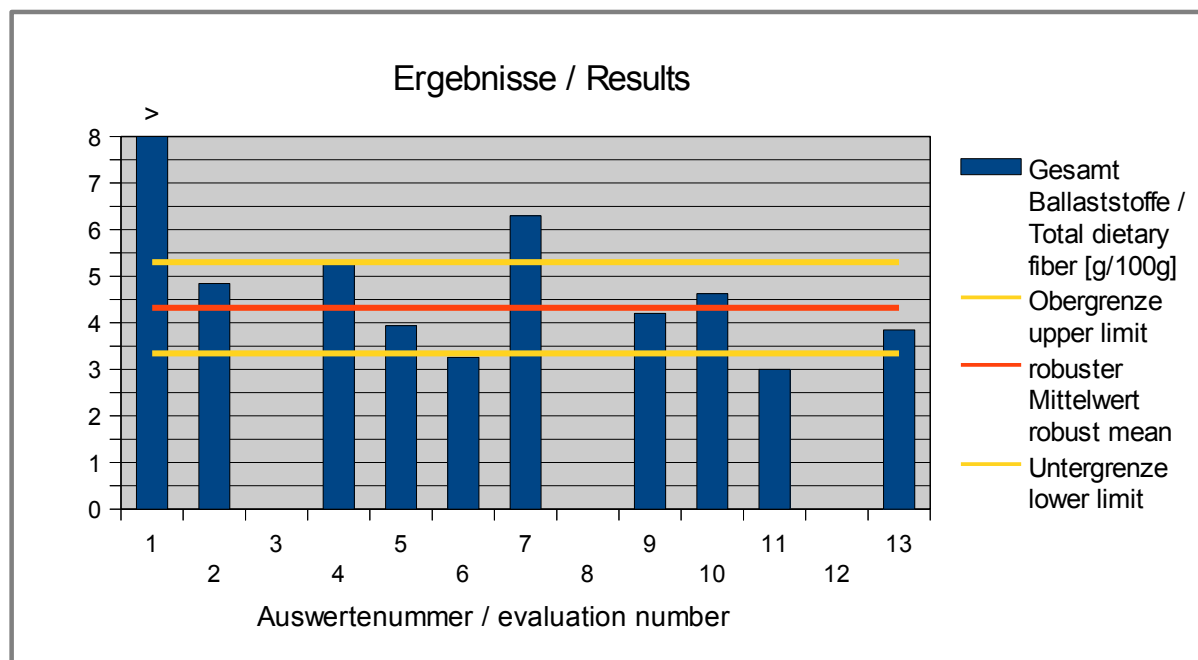
Abb. / Fig. 1: Ergebnisse Gesamtballaststoffe **/** Results Total dietary fiber
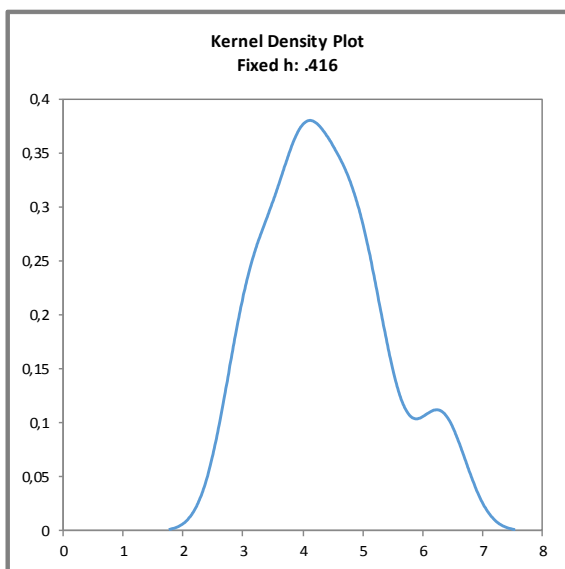
Abb. / Fig. 2:
Kerndichte-Schätzung der Ergebnisse (mit h = 0,75 x σ_{pt} von X_{pt})

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

Comment:
The kernel density shows almost a normal distribution of results with a shoulder at approx. 6,5 g/100g, due to a participant result above the target range. The result with the evaluation number 1 was excluded before.

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer / Evaluation number	Gesamt Ballaststoffe / Total dietary fiber [g/100g]		Abweichung [g/100g] Deviation [g/100g]	z'-Score (σ_{pt})	z-Score (Info)	Hinweis Remark
1	39,9	1)				Ergebnis ausgeschlossen / Result excluded (Faktor/factor 10?)
2	4,85	1)	0,525	1,1	3,8	
3						
4	5,25	2)	0,930	1,9	6,7	
5	3,94	1)	−0,380	−0,8	−2,7	
6	3,25	1)	−1,07	−2,2	−7,7	
7	6,30	3)	1,98	4,1	14,3	
8						
9	4,20	1)	−0,120	−0,2	−0,9	
10	4,63	1)	0,305	0,6	2,2	
11	3,00	1)	−1,32	−2,7	−9,5	
12						
13	3,85	1)	−0,470	−1,0	−3,4	

1) *ASU L 00.00-18 / AOAC 991.43 enzymatic-gravimetric*

2) *AOAC 985.29 enzymatic-gravimetric*

3) *AOAC 991.42 enzymatic-gravimetric*
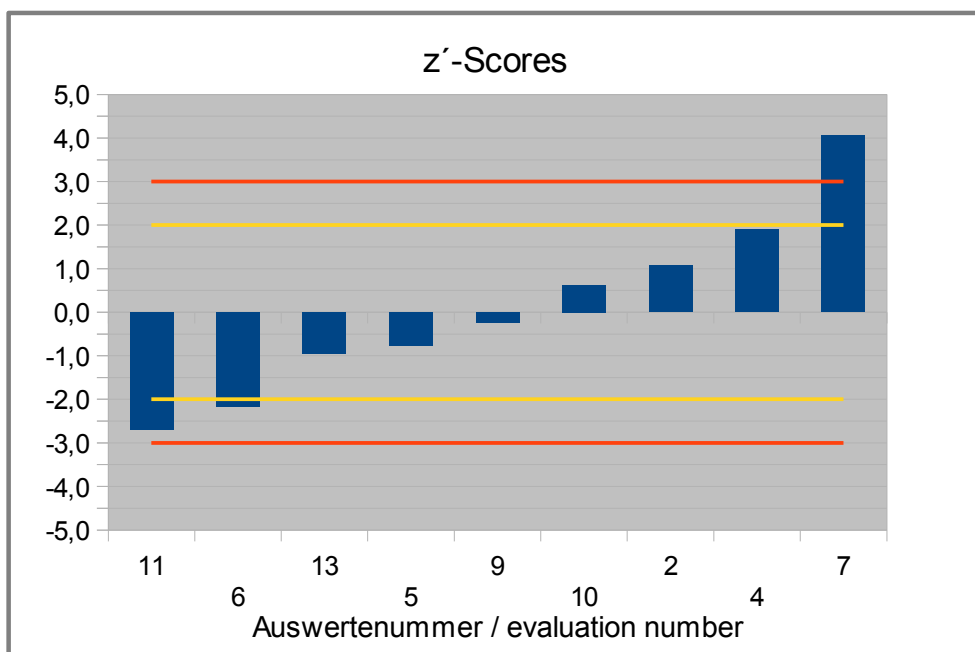
Abb. / Fig. 3: z´-Scores Gesamtballaststoffe / Total dietary fiber

4.2 Soluble Dietary Fiber in g/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	4
Number of outliers	0
Mean	1,17
Median	1,05
Robust Mean (X)	**1,17**
Robust standard deviation (S*)	**0,896**
Number with 2 replicates	3
Repeatability SD (S_r)	0,0952
Repeatability (CV_r)	7,20%
Reproducibility SD (S_R)	0,893
Reproducibility (CV_R)	67,5%

Due to the low number of results (<7) no statistical evaluation was done.
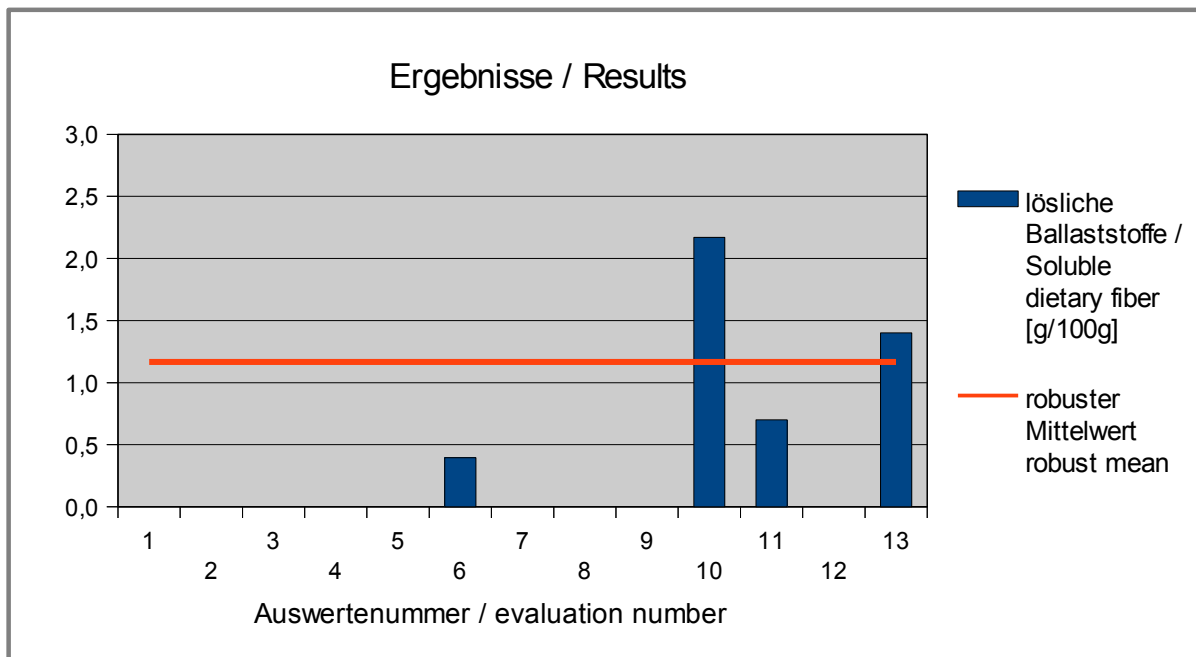
Abb. / Fig. 4: Ergebnisse lösliche Ballaststoffe **/** Results soluble dietary fiber

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	lösliche Ballaststoffe / Soluble dietary fiber [g/100g]	Abweichung [g/100g] Deviation [g/100g]	z-Score (σ_{pt})	Hinweis Remark
1				
2				
3				
4				
5				
6	0,40			
7				
8				
9				
10	2,17			
11	0,70			
12				
13	1,40			

4.3 Insoluble Dietary Fiber in g/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	5
Number of outliers	0
Mean	3,17
Median	2,86
Robust Mean (X)	**2,99**
Robust standard deviation (S*)	**0,965**
Number with 2 replicates	5
Repeatability SD (S_r)	0,234
Repeatability (CV_r)	7,35%
Reproducibility SD (S_R)	1,26
Reproducibility (CV_R)	39,5%

Due to the low number of results (<7) no statistical evaluation was done.
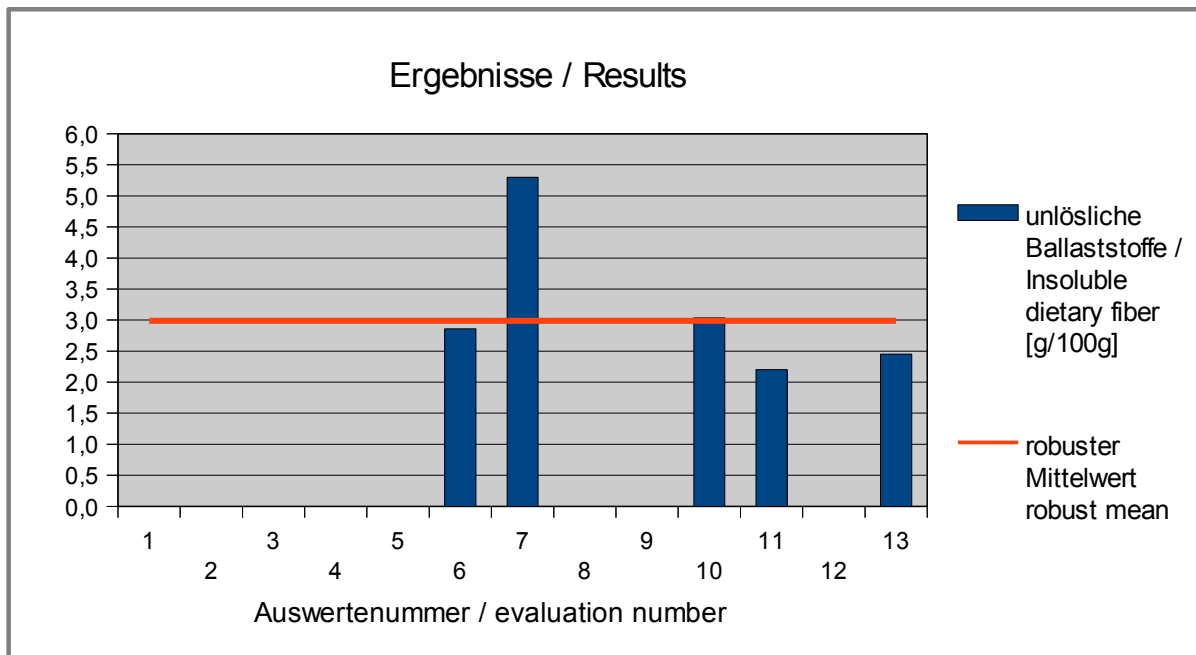
Abb. / Fig. 5: Ergebnisse unlösliche Ballaststoffe / Results insoluble dietary fiber

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer Evaluation number	unlösliche Ballaststoffe / Insoluble dietary fiber [g/100g]	Abweichung [g/100g] Deviation [g/100g]	z-Score (σ_{pt})	Hinweis Remark
1				
2				
3				
4				
5				
6	2,86			
7	5,30			
8				
9				
10	3,04			
11	2,20			
12				
13	2,45			

4.4 Total Dietary Fiber after Inulinase-Reac. in g/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	3
Number of outliers	0
Mean	4,89
Median	4,83
Robust Mean (X)	**4,89**
Robust standard deviation (S*)	**0,714**
Number with 2 replicates	3
Repeatability SD (S_r)	0,175
Repeatability (CV_r)	3,58%
Reproducibility SD (S_R)	0,642
Reproducibility (CV_R)	13,1%

Due to the low number of results (<7) no statistical evaluation was done.
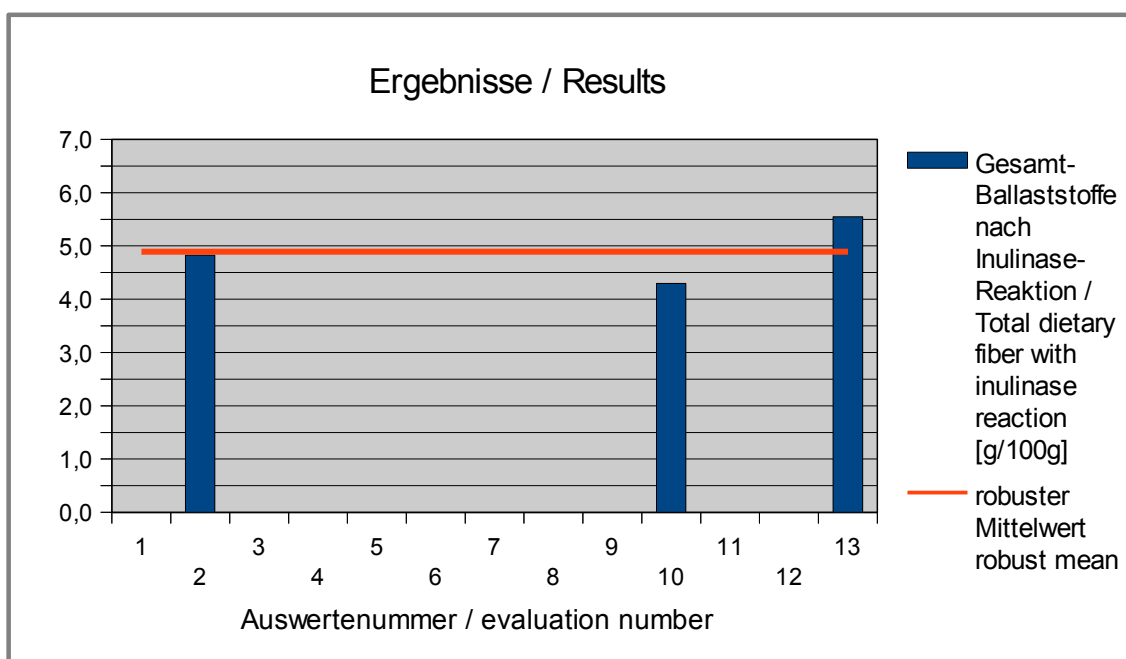
Abb. / Fig. 6: Ergebnisse Gesamtballaststoffe nach Inulinase-Behandlung / Results total dietary fiber after inulinase-reaction

Ergebnisse der Teilnehmer:
Results of Participants:

Auswerte-nummer / Evaluation number	Gesamt-Ballaststoffe nach Inulinase-Reaktion / Total dietary fiber with inulinase reaction[g/100g]	Abweichung [g/100g] Deviation [g/100g]	z-Score (σ_{pt})	Hinweis Remark
1				
2	4,83			
3				
4				
5				
6				
7				
8				
9				
10	4,30			
11				
12				
13	5,55			

4.5 Soluble Dietary Fiber after Inulinase-Reac. in g/100g

Vergleichsuntersuchung / Proficiency Test

Only one result was submitted (participant 10: 1,91 g/100g).
Further details are given in the documentation.

4.6 Insoluble Dietary Fiber after Inulinase-Reac. in g/100g

Vergleichsuntersuchung / Proficiency Test

Only one result was submitted (participant 10: 2,91 g/100g).
Further details are given in the documentation.

4.7 Inulin in g/100g

Vergleichsuntersuchung / Proficiency Test

Statistic Data	
Number of results	11
Number of outliers	0
Mean	3,57
Median	3,41
Robust Mean (X)	**3,53**
Robust standard deviation (S*)	**0,468**
Number with 2 replicates	10
Repeatability SD (S_r)	0,115
Repeatability (CV_r)	3,18%
Reproducibility SD (S_R)	0,494
Reproducibility (CV_R)	13,7%
Target range:	
Target standard deviation $\sigma_{pt'}$	**0,212**
Target standard deviation (for Information)	0,117
lower limit of target range	**3,10**
upper limit of target range	**3,95**
Quotient S*/$\sigma_{pt'}$	*2,2*
Standard uncertainty U(X_{pt})	*0,176*
Quotient U(X_{pt})/$\sigma_{pt'}$	*0,83*
Results in the target range	*8*
Percent in the target range	*73%*

Comment:

The target standard deviation was evaluated according to 3.6.2 by characteristics of a precision experiment (ASU §64 L 00.00-94). For information the target standard deviation using the general model of Horwitz is given (s. 3.6.1).
The distribution of results showed an increased variability with a quotient S*/σ_{pt} of 4,0. Therefore valuation was done considering the standard uncertainty by z'-score. Then the quotient S*/$\sigma_{pt}´$ of 2,2 was only slightly increased. The robust standard deviation and the repeatability and reproducibility standard deviation are comparable to those of prior PT´s (see 3.6.3). The comparability of results is given.

The quotient U(X_{pt})/σ_p of 0,8 is increased, but is acceptable on the basis of the other characteristics and the capability of applied methods.

73% of the results were in the target range.

Compared to the tolerances given in the guideline for the regulation (EU) No 1169/2011 for nutrition information all results, except the excluded result, were in the required range of ±2g for contents < 10 g/100g [22].

The robust mean of participant results is slightly above the excepted value from approx. 3,1 g/100g for added inulin (s. p. 4).
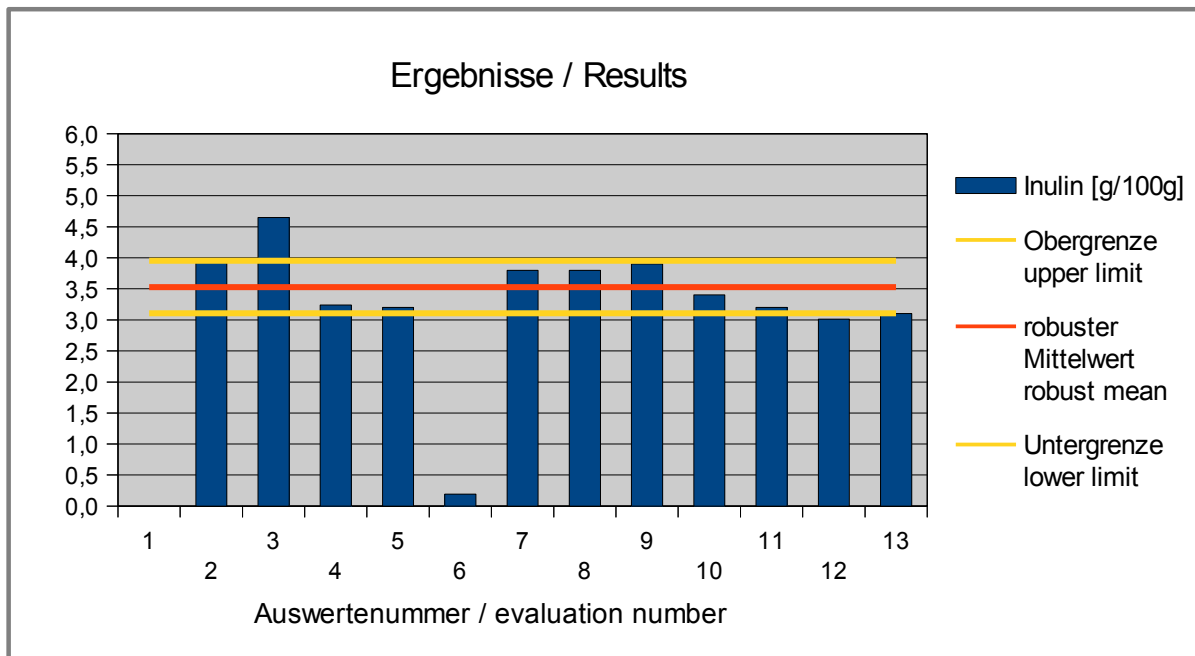
Abb. / Fig. 7: Ergebnisse Inulin **/** Results Inulin
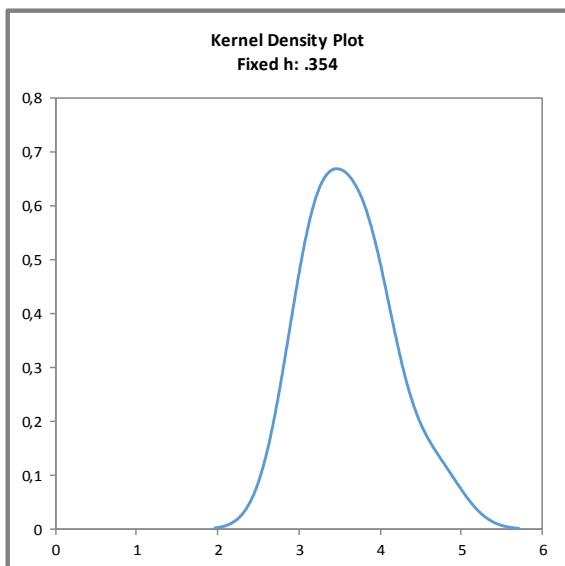
Abb. / Fig. 8:
Kerndichte-Schätzung der Ergebnisse (mit h = 0,75 x σ_{pt} von X_{pt})

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

Comment:
The kernel density shows almost a normal distribution of results. The result of participant number 6 was excluded before.

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						
2	3,93		0,397	1,9	3,4	
3	4,65	2)	1,12	5,3	9,6	
4	3,24		−0,288	−1,4	−2,5	
5	3,20	1)	−0,328	−1,5	−2,8	
6	0,19	1)				Ergebnis ausgeschlossen / Result excluded
7	3,80	2)	0,272	1,3	2,3	
8	3,80		0,272	1,3	2,3	
9	3,90	2)	0,372	1,8	3,2	
10	3,41	1)	−0,123	−0,6	−1,1	
11	3,20	1)	−0,328	−1,5	−2,8	
12	3,01		−0,518	−2,4	−4,4	
13	3,10	2)	−0,428	−2,0	−3,7	single result

1) *ASU L 00.00-94 / AOAC 999.03 enzymatic-photometric*

2) *AOAC 997.08 enzymatic-IEC or mod.*
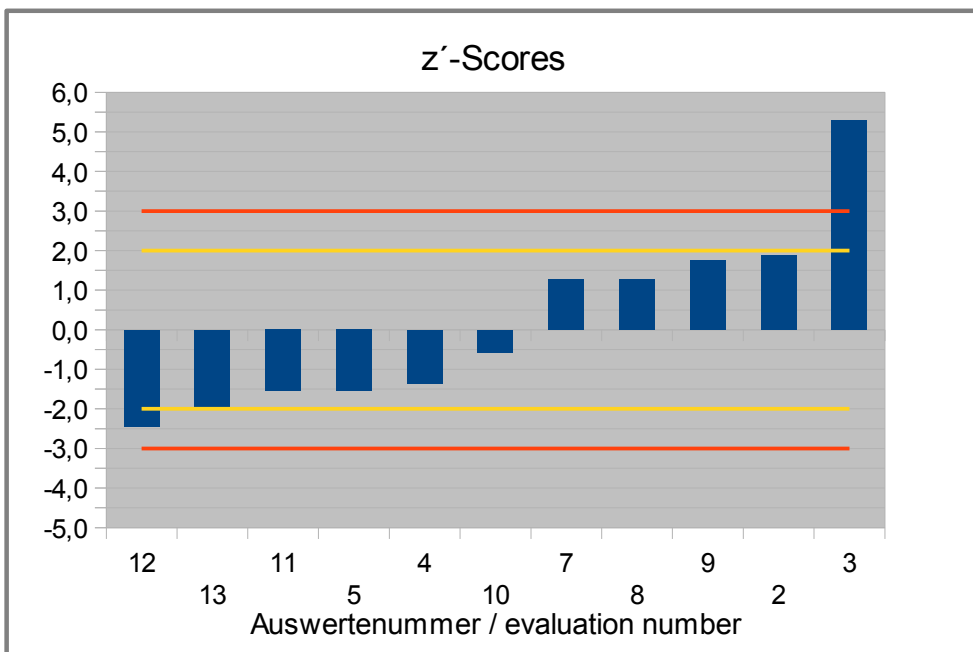
Abb. / Fig. 9: z´-Scores Inulin

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

Parameter	Teilnehmer	Einheit	Probe A DLA Nr.	Probe B DLA Nr.	Datum der Analyse	Abschließendes Ergebnis	Ergebnis Probe A	Ergebnis Probe B	Bestimmungs-grenze	Angabe inkl. Wiederfindung	Wiederfin-dungsrate	Methodenbeschreibung
Analyte	Participant	Unit	Sample A DLA No.	Sample B DLA No.	Date of analysis	Result (Mean)	Result A	Result B	Limit of quantifi-cation	Incl. Recovery rate	Recovery rate [%]	Method description
Gesamt Ballaststoffe / Total dietary fiber	1	g/100g	38	95		39,85						§64 LFGB L00.00-18
	2	g/100g	21	112	02/03 // 17/03	4,845	4,91	4,78		no		PV 2202-04
	3	g/100g	46	78								
	4	g/100g	41	92	14.03.17	5,25	5,6	4,9	0,5	N/A	N/A	AOAC 985.29
	5	g/100g	37	96	15.03.17	3,94	3,91	3,97		no		§64 LFGB L.00.00-18
	6	g/100g	53	80	08.03.17	3,254	3,349	3,159	0,05	no	99,6	ASU L00.00-18
	7	g/100g	66	68	16.02.17	6,3	6,4	6,1	0,5	no		PNTA0066/AOAC 991,42
	8	g/100g	57	76								
	9	g/100g	31	102	23.03.17	4,2	4,1	4,2	0,1	no		AOAC 991.43
	10	g/100g	35/2017 (103)	35/2017 (30)	27.02.17	4,625	4,63	4,62	0,02	no		SOP M 1010 nach ASU L 00.00-18, mod.
	11	g/100g	11	122	24.02.17	3	2,3	3,6	2	no	-	AOAC 991.43
	12	g/100g	54	79								
	13	g/100g	106	28	06.04.17	3,85	4	3,7		no		AOAC 991.43

Parameter / Analyte	Teilnehmer / Participant	Frittendurch-messer / Frit diameter	Porendurch-messer / Pore diameter	Angaben zu Amylase / Notes to Amylase	Angaben zu Protease / Notes to Protease	Angaben zu Amyloglucosidase / Notes to Amyloglucosidase	Angaben zur Inulinase / Notes to Inulinase	Kalibrierung und Referenzmaterial / Calibration and reference material	Methode akkreditiert nach ISO/IEC 17025 / Method accred. accord. ISO/IEC 17025	Sonstige Hinweise / Further remarks
Ballaststoffe / dietary fibre	1	4 cm	16-40 µm						yes	
	2	30 mm	40 - 100 µm	Bioquant 112979 LOT: HC 563819	Bioquant 112979 LOT: HC 563819	Bioquant 112979 LOT: HC 563819			yes	
	3									
	4	N/A	P.2	Megazyme K-TDFR	Megazyme K-TDFR	Megazyme K-TDFR	N/A	Internal Reference Material	yes	N/A
	5	Filterpapier MN 640W	Filterpapier MN 640W	Sigma A 3306; Lot SLBJ0135V	Sigma P 3910; Lot SLBQ9441V	Sigma A 9913; Lot SLBP1905V	Fluka 57620; Lot BCBP7930V	Bran Breakfast CerialNo. 0558	yes	
	6							yes	yes	keine
	7	4,5cm	40-60 µm	Megazyme E-BLAAM	Megazyme E-BSPRT	Megazyme E-AMGDF		Internally	yes	
	8									
	9		2	Megazyme Kit AOAC 991.43	Megazyme Kit AOAC 991.43	Megazyme Kit AOAC 991.43			no	
	10	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-Maltoside)	Fructanase Maxture Exo-Inulinase 2000 U/mL + Endo-Inulinase 80 U/mL ; In 50% Glycerol	control chart Weetabix	yes	Fiber (claculated): 5,17 % &
	11	3.3 cm	40-100 µm	α-amylase, 3000 units/ml (Megazyme)	protease, 350 tyrosine units/ml (Megazyme)	amyloglucosidase, 3300 units/ml (Megazyme)	not applicable	bd518 Dried Bran Breakfast Cereal	no	-
	12									
	13	30 mm	40-90um	Megazyme E-Blaam	Megazyme E-BSPRT	Megazyme E-AMGDF		no	_	

Parameter: Soluble Dietary Fiber

Parameter	Teilnehmer	Einheit	Probe A DLA Nr.	Probe B DLA Nr.	Datum der Analyse	Abschließendes Ergebnis	Ergebnis Probe A	Ergebnis Probe B	Bestimmungs-grenze	Angabe inkl. Wiederfindung	Wiederfin-dungsrate	Methodenbeschreibung
Analyte	Participant	Unit	Sample A DLA No.	Sample B DLA No.	Date of analysis	Result (Mean)	Result A	Result B	Limit of quantifi-cation	Incl. Recovery rate	Recovery rate [%]	Method description
Lösliche Ballaststoffe / Soluble dietary fiber	1	g/100g	38	95								
	2	g/100g	21	112								
	3	g/100g	46	78								
	4	g/100g	41	92	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
	5	g/100g	37	96								
	6	g/100g	53	80	08.03.17	0,395	0,394	0,396	0,05	no	99,6	ASU L00.00-18
	7	g/100g	66	68								
	8	g/100g	57	76								
	9	g/100g	31	102								
	10	g/100g	35/2017 (103)	35/2017 (30)	13.03.17	2,17	2,11	2,23	0,04	no		SOP M 2477 according to ASU L 00.00-18, mod.
	11	g/100g	11	122	24.02.17	0,7	0	1,4	2	no	-	
	12	g/100g	54	79								
	13	g/100g	106	28	06.04.17	1,4	1,5	1,3		no		AOAC 991.43

Parameter Analyte	Teilnehmer Participant	Frittendurch-messer Frit diameter	Porendurch-messer Pore diameter	Angaben zu Amylase Notes to Amylase	Angaben zu Protease Notes to Protease	Angaben zu Amyloglucosidase Notes to Amyloglucosidase	Angaben zur Inulinase Notes to Inulinase	Kalibrierung und Referenzmaterial Calibration and reference material	Methode akkreditiert nach ISO/IEC 17025 Method accred. accord. ISO/IEC 17025	Sonstige Hinweise Further remarks
Lösliche Ballaststoffe / Soluble dietary fibre	1									
	2									
	3									
	4	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
	5									
	6							yes	yes	none
	7									
	8									
	9									
	10	40mm	Por. 3 16-40 µm / 25-50 µm	3000 U/mL; 54 U/mg	50 mg/mL	3,26 U/mL (souble starch); 200 U/mL 8p-NP Bets-Maltoside)	Fructanase Maxture Exo-Inulinase 2000 U/mL + Endo-Inulinase 80 U/mL ; In 50% Glycerol	control chart Weetabix	yes	
	11									
	12									
	13	30 mm	40-90um	Megazyme E-Blaam	Megazyme E-BSPRT	Megazyme E-AMGDF		no	_	

Parameter: Insoluble Dietary Fiber

Parameter	Teilnehmer	Einheit	Probe A DLA Nr.	Probe B DLA Nr.	Datum der Analyse	Abschließendes Ergebnis	Ergebnis Probe A	Ergebnis Probe B	Bestimmungs-grenze	Angabe inkl. Wiederfindung	Wiederfin-dungsrate	Methodenbeschreibung
Analyte	Participant	Unit	Sample A DLA No.	Sample B DLA No.	Date of analysis	Result (Mean)	Result A	Result B	Limit of quantifi-cation	Incl. Recovery rate	Recovery rate [%]	Method description
Unlösliche Ballaststoffe / Insoluble dietary fiber	1	g/100g	38	95								
	2	g/100g	21	112								
	3	g/100g	46	78								
	4	g/100g	41	92	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
	5	g/100g	37	96								
	6	g/100g	53	80	08.03.17	2,859	2,955	2,763	0,05	no	99,6	ASU L00.00-18
	7	g/100g	66	68	28.03.17	5,3	5,7	5	0,5	no		PNTA0066/AOAC 991,42
	8	g/100g	57	76								
	9	g/100g	31	102								
	10	g/100g	35/2017 (103)	35/2017 (30)	13.03.17	3,035	3,06	3,01	0,04	no		SOP M 2477 according to ASU L 00.00-18, mod.
	11	g/100g	11	122	24.02.17	2,2	2,3	2,2	2	no	-	
	12	g/100g	54	79								
	13	g/100g	106	28	06.04.17	2,45	2,5	2,4		no		AOAC 991.43

Parameter	Teilnehmer	Frittendurch-messer	Porendurch-messer	Angaben zu Amylase	Angaben zu Protease	Angaben zu Amyloglucosidase	Angaben zur Inulinase	Kalibrierung und Referenzmaterial	Methode akkreditiert nach ISO/IEC 17025	Sonstige Hinweise
Analyte	Participant	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
Unlösliche Ballaststoffe / Insoluble dietary fiber	1									
	2									
	3									
	4	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
	5									
	6							yes	yes	none
	7	4,5cm	40-60 µm	Megazyme E-BLAAM	Megazyme E-BSPRT	Megazyme E-AMGDF		internal	yes	
	8									
	9									
	10	40mm	Por. 3 16-40 µm / 25-50 µm	3000 U/mL; 54 U/mg	50 mg/mL	3,26 U/mL (souble starch); 200 U/mL 8p-NP Bets-Maltoside)	Fructanase Maxture Exo-Inulinase 2000 U/mL + Endo-Inulinase 80 U/mL ; In 50% Glycerol	control chart Weetabix	yes	
	11									
	12									
	13	30 mm	40-90um	Megazyme E-Blaam	Megazyme E-BSPRT	Megazyme E-AMGDF		no	_	

Parameter: Total Dietary Fiber after Inulinase-Reaction

Parameter	Teilnehmer	Einheit	Probe A DLA Nr.	Probe B DLA Nr.	Datum der Analyse	Abschließendes Ergebnis	Ergebnis Probe A	Ergebnis Probe B	Bestimmungs-grenze	Angabe inkl. Wiederfindung	Wiederfin-dungsrate	Methodenbeschreibung
Analyte	Participant	Unit	Sample A DLA No.	Sample B DLA No.	Date of analysis	Result (Mean)	Result A	Result B	Limit of quantifi-cation	Incl. Recovery rate	Recovery rate [%]	Method description
Gesamtballaststoffe nach Inulinase-Reaktion/ Total dietary fiber with inulinase reaction	1	g/100g	38	95								
	2	g/100g	21	112	02/03 // 08/03	4,83	4,71	4,95		no		PV 2202-04
	3	g/100g	46	78								
	4	g/100g	41	92	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
	5	g/100g	37	96								
	6	g/100g	53	80								
	7	g/100g	66	68								
	8	g/100g	57	76								
	9	g/100g	31	102								
	10	g/100g	35/2017 (103)	35/2017 (30)	06.03.17	4,295	4,2	4,39	0,02	no		ASU L 00.00-18 mod., enzymatic-gravimetric
	11	g/100g	11	122								
	12	g/100g	54	79								
	13	g/100g	106	28	06.04.17	5,55	5,4	5,7		no		AOAC2009.01 corrected with Inulin AOAC 997.08 modified value as reported below (see comment in cel M33 etc

Parameter Analyte	Teilnehmer Participant	Frittendurch-messer Frit diameter	Porendurch-messer Pore diameter	Angaben zu Amylase Notes to Amylase	Angaben zu Protease Notes to Protease	Angaben zu Amyloglucosidase Notes to Amyloglucosidase	Angaben zur Inulinase Notes to Inulinase	Kalibrierung und Referenzmaterial Calibration and reference material	Methode akkreditiert nach ISO/IEC 17025 Method accred. accord. ISO/IEC 17025	Sonstige Hinweise Further remarks
Gesamtballasts toffe nach Inulinase-Reaktion/ Total dietary fiber with inulinase reaction	1									
	2	30 mm	40 - 100 µm	Bioquant 112979 LOT: HC 563819	Bioquant 112979 LOT: HC 563819	Bioquant 112979 LOT: HC 563819	Megazyme Fructanase Mixture LOT: 121001		yes	
	3									
	4	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
	5									
	6									
	7									
	8									
	9									
	10	40mm	Por. 3 16-40 µm / 25-50 µm	3000 U/mL; 54 U/mg	50 mg/mL	3,26 U/mL (souble starch); 200 U/mL 8p-NP Bets-Maltoside)	Fructanase Maxture Exo-Inulinase 2000 U/mL + Endo-Inulinase 80 U/mL ; In 50% Glycerol	control chart Weetabix	yes	
	11									
	12									
	13	30 mm	40-90um	Megazyme E-PANAA	Megazyme E-BSPRT	Megazyme E-AMGDF	Megazyme FRMXLQ	no	_	

Parameter: Soluble Dietary Fiber after Inulinase-Reaction

Parameter	Teilnehmer	Einheit	Probe A DLA Nr.	Probe B DLA Nr.	Datum der Analyse	Abschließendes Ergebnis	Ergebnis Probe A	Ergebnis Probe B	Bestimmungs-grenze	Angabe inkl. Wiederfindung	Wiederfin-dungsrate	Methodenbeschreibung
Analyte	Participant	Unit	Sample A DLA No.	Sample B DLA No.	Date of analysis	Result (Mean)	Result A	Result B	Limit of quantifi-cation	Incl. Recovery rate	Recovery rate [%]	Method description
Lösliche Ballaststoffe nach Inulinase-Reaktion/ Soluble dietary fiber with inulinase reaction	1	g/100g	38	95								
	2	g/100g	21	112								
	3	g/100g	46	78								
	4	g/100g	41	92	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
	5	g/100g	37	96								
	6	g/100g	53	80								
	7	g/100g	66	68								
	8	g/100g	57	76								
	9	g/100g	31	102								
	10	g/100g	35/2017 (103)	35/2017 (30)	13.03.17	1,905	1,91	1,9	0,04	no		SOP M 2477 according to ASU L 00.00-18, mod.
	11	g/100g	11	122								
	12	g/100g	54	79								
	13	g/100g	106	28		not measured	not measured	not measured	not measured	-		

Parameter / Analyte	Teilnehmer / Participant	Frittendurch-messer / Frit diameter	Porendurch-messer / Pore diameter	Angaben zu Amylase / Notes to Amylase	Angaben zu Protease / Notes to Protease	Angaben zu Amyloglucosidase / Notes to Amyloglucosidase	Angaben zur Inulinase / Notes to Inulinase	Kalibrierung und Referenzmaterial / Calibration and reference material	Methode akkreditiert nach ISO/IEC 17025 / Method accred. accord. ISO/IEC 17025	Sonstige Hinweise / Further remarks
Lösliche Ballaststoffe nach Inulinase-Reaktion/ Soluble dietary fiber with inulinase reaction	1									
	2									
	3									
	4	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
	5									
	6									
	7									
	8									
	9									
	10	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-Maltoside)	Fructanase Maxture Exo-Inulinase 2000 U/mL + Endo-Inulinase 80 U/mL ; In 50% Glycerol	control chart Weetabix	yes	
	11									
	12									
	13							_	_	_	

Parameter: Insoluble Dietary Fiber after Inulinase-Reaction

Parameter	Teilnehmer	Einheit	Probe A DLA Nr.	Probe B DLA Nr.	Datum der Analyse	Abschließendes Ergebnis	Ergebnis Probe A	Ergebnis Probe B	Bestimmungs-grenze	Angabe inkl. Wiederfindung	Wiederfin-dungsrate	Methodenbeschreibung
Analyte	Participant	Unit	Sample A DLA No.	Sample B DLA No.	Date of analysis	Result (Mean)	Result A	Result B	Limit of quantifi-cation	Incl. Recovery rate	Recovery rate [%]	Method description
Unlösliche Ballaststoffe nach Inulinase-Reaktion/ Insoluble dietary fiber with inulinase reaction	1	g/100g	38	95								
	2	g/100g	21	112								
	3	g/100g	46	78								
	4	g/100g	41	92	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
	5	g/100g	37	96								
	6	g/100g	53	80								
	7	g/100g	66	68								
	8	g/100g	57	76								
	9	g/100g	31	102								
	10	g/100g	35/2017 (103)	35/2017 (30)	13.03.17	2,905	2,75	3,06	0,04	no		SOP M 2477 according to ASU L 00.00-18, mod.
	11	g/100g	11	122								
	12	g/100g	54	79								
	13	g/100g	106	28		not measured	not measured	not measured	not measured	-		

Parameter / Analyte	Teilnehmer / Participant	Frittendurch-messer / Frit diameter	Porendurch-messer / Pore diameter	Angaben zu Amylase / Notes to Amylase	Angaben zu Protease / Notes to Protease	Angaben zu Amyloglucosidase / Notes to Amyloglucosidase	Angaben zur Inulinase / Notes to Inulinase	Kalibrierung und Referenzmaterial / Calibration and reference material	Methode akkreditiert nach ISO/IEC 17025 / Method accred. accord. ISO/IEC 17025	Sonstige Hinweise / Further remarks
Unlösliche Ballaststoffe nach Inulinase-Reaktion/ Insoluble dietary fiber with inulinase reaction	1									
	2									
	3									
	4	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
	5									
	6									
	7									
	8									
	9									
	10	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-Maltoside)	Fructanase Maxture Exo-Inulinase 2000 U/mL + Endo-Inulinase 80 U/mL ; In 50% Glycerol	control chart Weetabix	yes	
	11									
	12									
	13									

Parameter: Inulin

Parameter	Teilnehmer	Einheit	Probe A DLA Nr.	Probe B DLA Nr.	Datum der Analyse	Abschließendes Ergebnis	Ergebnis Probe A	Ergebnis Probe B	Bestimmungs-grenze	Angabe inkl. Wiederfindung	Wiederfin-dungsrate	Methodenbeschreibung
Analyte	Participant	Unit	Sample A DLA No.	Sample B DLA No.	Date of analysis	Result (Mean)	Result A	Result B	Limit of quantifi-cation	Incl. Recovery rate	Recovery rate [%]	Method description
Inulin	1	g/100g	38	95								
	2	g/100g	21	112	24.02.17	3,925	3,82	4,03		no		PV 2255
	3	g/100g	46	78	31.03.17	4,65	4,6	4,7	0,05	no	-	AOAC 997.08: „Fructans in Food Products (Ion Exchange Chromatographic Method)", modified
	4	g/100g	41	92	14.03.17	3,24	3,28	3,2	<0.1	no	n/a	Fruktane total (inclusive Inulin)
	5	g/100g	37	96	28.03.17	3,2	3,15	3,24		no		§64 LFGB L.00.00-94
	6	g/100g	53	80	08.03.17	0,194	0,193	0,194	0,1	no	95	ASU L00.00-94
	7	g/100g	66	68	23.02.17	3,8	3,7	3,8	0,5	no		PNTQ1038/AOAC 997,08
	8	g/100g	57	76	22.03.17	3,8	3,8	3,8	0,3	no		LAV 21.0051.01 enzymatic
	9	g/100g	31	102	24.02.17	3,9	3,9	3,8	0,1			AOAC 997.08
	10	g/100g	35/2017 (103)	35/2017 (30)	17.03.17	3,405	3,6	3,21	0,2	no		ASU L 00.00-94 mod.
	11	g/100g	11	122	03.03.17	3,2	3,2	3,3	< 1	no	-	AOAC 999.03
	12	g/100g	54	79	27.02.	3,01	3,06	2,95				internal Method, HPLC-RID
	13	g/100g	106	28	17.03.17		not measured	3,1		no		AOAC 997.08 modified

Parameter	Teilnehmer	UV-Test Kit Hersteller	HPLC Säule	Detektor	Probenvorbereitung und -aufarbeitung	Kalibrierung und Referenzmaterial	Methode akkreditiert nach ISO/IEC 17025	Sonstige Hinweise
Analyte	Participant	UV-test kit manufacturer	HPLC column	Detector	Notes to Protease	Calibration and reference material	Method accred. accord. ISO/IEC 17025	Further remarks
Inulin	1							
	2	r-biopharm Art. Nr. 10139106035 LOT: 15244720	Eurospher 100-5 NH2	RI			yes	
	3	-	Carbopac™ PA20; 3 x 150 mm	PAD	-	Reference material: DLA 36/2016	yes	Following modifications were performed: Glucose-content was considered by calculation of the degree of polymerisation. Other composition of eluents, Gradientenprogram shortend from 83 to 47 min
	4	Megazyme	N/A	UV/VIS	no prep	Fructose Standard	No	This analysis does not include FOS
	5	r-biopharm 10716260; Lot 14111700 JUL17					yes	
	6	r-biopharm				yes	yes	none
	7		Zorbax Carbohydrate 5µ 4.6x150mm. Agilent 843300-908	RI		Fructose/glucose/sucrose from Sigma	no	
	8	r-Biopharm					yes	
	9	/		FID			no	GC
	10	Boehringer Mannheim von R-Biopharm	not used	not used	according to §64 LFGB, L 00.00-94	control chart Weetabix	yes	As it is a photometric method accoridng to § 64 LFGB, L 00.00-94 no HPLC column/detector was used
	11	not applicable	Dionex CarboPac PA1	PAD	-	Control Flour (Megazyme)	no	-
	12						yes	
	13	_	Dionex PA1	PAD	no		-	Eurofins Heerenveen routinely uses AOAC 2009.01 for low molecular weight dietary fiber containing materials (eg FOS, inulin GOS , polydextrose

5.2 Homogeneity

5.2.1 Mixture homogeneity before bottling

Microtracer Homogeneity Test
DLA 35-2017

Weight whole sample	2,80	kg
Microtracer	FSS-rot lake	
Particle size	75 – 300	µm
Weight per particle	2,0	µg
Addition of tracer	32,7	mg/kg

Result of analysis

Sample	Weight [g]	Particle number	Particles [mg/kg]
1	5,07	78	30,8
2	5,11	89	34,8
3	5,02	75	29,9
4	5,16	87	33,7
5	5,08	83	32,7
6	5,06	80	31,6
7	5,07	95	37,5
8	5,06	90	35,6

Poisson distribution		
Number of samples	8	
Degree of freedom	7	
Mean	84,6	Partikel
Standard deviation	6,54	Partikel
χ^2 (CHI-Quadrat)	3,54	
Probability	**83**	%
Recovery rate	102	%

Normal distribution		
Number of samples	8	
Mean	33,3	mg/kg
Standard deviation	2,58	mg/kg
rel. Standard deviaton	7,73	%
Horwitz standard deviation	9,44	%
HorRat-value	**0,82**	
Recovery rate	102	%

### 5.2.2 Comparison of sample numbers / test results and trend line

By comparison of the increasing sample numbers and the measurement results of participants, the homogeneity of the chronological bottled PT item can be characterized with the help of the trend line function:

Total Dietary Fiber				
Target standard deviation σ_{pt}	0,490			g/100g
Sample numbers	11 – 122			
Total numbers of samples	18			
Slope	0,0218			
Trend line range	4,14	–	4,53	g/100g
Deviation trend line	4,34	±	0,196	g/100g
Percent of σpt	40,0	%		

Homgenität / homogeneity

Gesamtballaststoffe / Total Dietary Fiber

DLA-Nr. / No.

Ergebnis / result *10

Linear (Ergebnis / result *10)

f(x) = 0,2179x + 41,3904
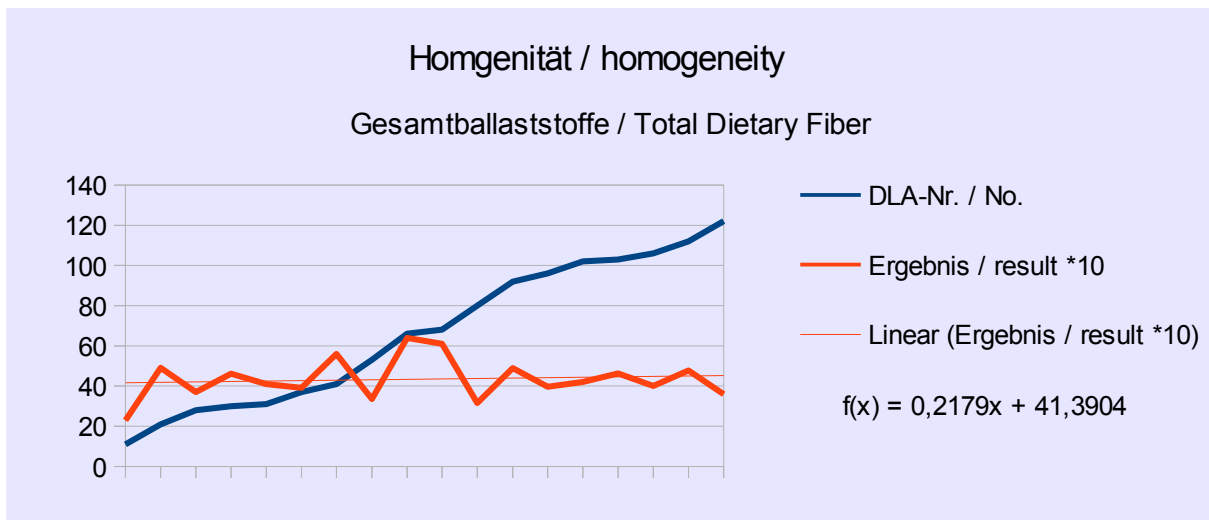
Abb./Fig. 10:
Trendfunktion Probennummern vs. Ergebnisse
trend line function sample number vs. results

Inulin				
Target standard deviation σ_{pt}	0,211			g/100g
Sample numbers	11 – 122			
Total numbers of samples	20			
Slope	0,00529			
Trend line range	3,55	–	3,66	g/100g
Deviation trend line	3,60	±	0,053	g/100g
Percent of σpt	25,1	%		
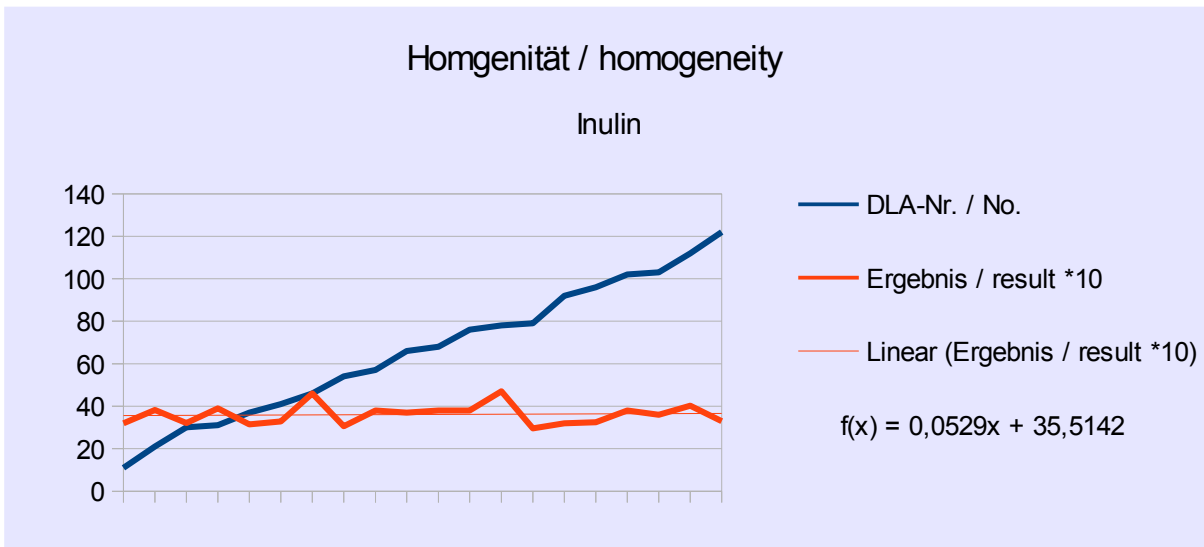
Abb./Fig. 11:
Trendfunktion Probennummern vs. Ergebnisse
trend line function sample number vs. results

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 35-2017**
PT name	**Dietary Fiber and Inulin in Cereal Product**
Sample matrix*	*Rusk, ground / Ingredients: wheat flour, sugar, vegetable fats and oils, yeast, salt, wheat malt flour, wheat gluten and further addition of inulin; fat content <10%*
Number of samples and sample amount	*2 identical samples: 20 g each.*
Storage	*room temperature*
Intentional use	*Laboratory use only (quality control samples)*
Parameter	*quantitative:* *total dietary fiber, soluble and insoluble dietary fiber (each without and with inulinase treatment) 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	*The results for sample A and B as well as the final results calculated as mean of the double determination (samples A and B) should be filled in the result submission file. The recovery rates, if carried out, has to be included in the calculation.*
Units	*g/100g*
Number of significant digits	*at least 2*
Further information	*For information please specify:* – *Date of analysis* – *DLA-sample-numbers (for sample A and B)* – *Limit of detection* – *Method description* – *Method is accredited* – *Further remarks*
Result submission	*The result submission file should be sent by e-mail to:* *pt@dla-lvu.de*
Deadline	*the latest <u>March 31st 2017</u>*
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. Testing of the content, homogeneity and stability of PT parameters is subcontracted by DLA.

6. Index of participant laboratories in alphabetical order

Teilnehmer / Participant	Ort / Town	Land / Country
		GREAT BRITAIN
		Germany
		NETHERLANDS
		Germany
		Germany
		NETHERLANDS
		Germany
		Germany
		Germany
		Germany
		Germany
		SPAIN
		BELGIUM

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

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

7. Index of references

1. DIN EN ISO/IEC 17025:2005; Allgemeine Anforderungen an die Kompetenz von Prüf- und Kalibrierlaboratorien / General requirements for the competence of testing and calibration laboratories
2. DIN EN ISO/IEC 17043:2010; Konformitätsbewertung – Allgemeine Anforderungen an Eignungsprüfungen / Conformity assessment – General requirements for proficiency testing
3. ISO 13528:2015 & DIN ISO 13528:2009; Statistische Verfahren für Eignungsprüfungen durch Ringversuche / Statistical methods for use in proficiency testing by interlaboratory comparisons
4. ASU §64 LFGB: Planung und statistische Auswertung von Ringversuchen zur Methodenvalidierung / DIN ISO 5725 series part 1, 2 and 6 Accuracy (trueness and precision) of measurement methods and results
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15. MTSE SOP No. 010.01 (2014): Quantitative measurement of mixing uniformity and carry-over in powder mixtures with the rotary detector technique, MTSE Micro Tracers Services Europe GmbH
16. ASU §64 LFGB: L 00.00-18: Bestimmung der Ballaststoffe in Lebensmitteln [Determination of dietary fieber in food]
17. ASU § 64 LFGB L 17.03-1 Bestimmung der Ballaststoffe in Mischbrot [Determination of dietary fieber in mixed-type bread].
18. ASU § 64 LFGB L 00.00-94 Bestimmung von Inulin in Lebensmitteln – Enzymatisches Verfahren [Determination of inulin in food – Enzymatic method]
19. 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
20. Positionspapier der Lebensmittelchemischen Gesellschaft [der GDCh] „Ballaststoffe: aktuelle Betrachtung aus rechtlicher und analytischer Sicht" erarbeitet durch die Arbeitsgruppe "Fragen der Ernährung", (2012) [Position paper on dietary fiber legal and analytical issues, Food Chemistry Society of the GDCh]
21. Dietary fibre fractions in cereal foods measured by a new integrated AOAC

method, Hollmann et al. (2013), Food Chem 140(3):586-9
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)