



Final Report

evaluation of proficiency test

DLA ptAU01 (2022)

**Dietary Fiber, soluble / insoluble Fiber
and Inulin**

in Cereal Product (Bread Baking Mix)

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Allgemeine Informationen zur Eignungsprüfung (EP)
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<i>Vertraulichkeit</i> <i>Confidentiality</i>	<p>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.</p>

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The **List of Abbreviations** can be found in the
“DLA Evaluation Guide 02.01 (2022) General Proficiency Test Schemes”

1. Introduction

The participation in proficiency test (PT) 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].

The general procedure for evaluating the DLA proficiency tests can be found in the “**DLA Evaluation Guide 02.01 (2022) General Proficiency Test Schemes**”.

2. Realisation

2.1 Test material

The test material is a common in commerce bread baking mix from a European supplier. The basic composition of samples A and B was the same (see table 1).

After sieving and homogenisation of the raw material, the spiked sample B was produced as follows: Previously sieved inulin (mesh 400 µm) was added to an aliquot of the matrix and the mixture was homogenised. Subsequently, basic mixture was again added in further steps and homogenised in each case until the total amount was reached.

Afterwards, the samples were portioned to approximately 40 g into metallised PET film bags.

The composition (list of ingredients) of the samples is given in table 1.

Table 1: Composition of DLA-Samples

Ingredients	Sample A *	Sample B *
Bread Baking Mix, organic Ingredients: Wholemeal wheat flour 61%, wholemeal rye flour 33%, wholemeal rye sourdough powder 4%, sea salt Nutrients** per 100 g: Fat 1,7 g, carbohydrates 59 g thereof sugar 0,8 g, protein 10 g, salt 1,7 g	100 g/100 g	95,0 g/100 g
- thereof fiber (13 %) **	13,0 g/100 g **	12,4 g/100 g **
Inulin	-	4,98 g/100 g

* 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 testing results

A specific description of the procedures can be found in the “DLA Evaluation Guide 02.01 (2022) General Proficiency Test Schemes”.

Homogeneity testing by microtracer

The microtracer analysis showed an acceptable homogeneity of the present PT samples (see Table 2). The results of microtracer analysis are given in the documentation (see 5.2). The HorRat value for sample A with no addition of inulin was accepted, because of the good probability result.

Table 2: Results of microtracer analysis

Evaluation method	Criterion	Sample A	Sample B
Probability (poisson distribution)	≥ 5 % (good) ≥ 25% (excellent)	19 %	76 %
HorRat Value (normal distribution)	≤ 1,3	1,7	1,0

Homogeneity of the parameters

The calculation of the **repeatability standard deviations S_r of the participants** was also used as an indicator of homogeneity.

For total dietary fiber without inulin (method 1), it is approx. 1,3% and 1,1% and for inulin (methods 5, 6) it is 2,0% (sample A without inulin addition) and 4,0% (sample B with inulin addition) (see Table 3). Thus, they were similar to corresponding repeatability standard deviations of precision data of the standardized methods (e.g. ASU methods / EN ISO methods ASU L 00.00-18 / L 16.08-1 / L 17.03-1 and ASU L 00.00-94, s. 3.3) (see Table 6) [A-E].

The repeatability standard deviations of the participants' results are given in the documentation in the statistic data (see 4.1 to 4.6).

Table 3: 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)	1,34 %	1,10 %
Total dietary fiber incl. inulin (2+3)	11,9 %	15,7 %
Insoluble dietary fiber (1a, 2a, 3a)	1,97 %	3,01 %
Soluble dietary fiber (1b, 2b)	1,37 %	6,55 %
Inulin / Fructans (5+6)	2,01 %	4,04 %

* (1 - 6) Methods see page 10

** Sample B with addition of inulin

In case the criterion for sufficient homogeneity of the test items is not fulfilled, the impact on the target standard deviation will be verified. If necessary, the evaluation of results will be done considering the

standard uncertainty of the assigned value by z'-scores (s. DLA Evaluation Guide 02.01 (2022) 3.2.7.2 and 3.2.9) [3].

2.1.2 Stability

The a_w values of the PT **samples in form of powder** were below $< 0,5$ (see Table 4). Therefore, a good storage stability with respect to the durability of the sample (spoilage) and the content of the PT parameters as established for comparable food matrices can be expected. The stability of the sample material was thus ensured during the investigation period under the specified storage conditions.

Table 4: Results of water activity (a_w value).

Evaluation method	Criterion	Sample A	Sample B
a_w value	$\leq 0,5$	0,45 (RT)	0,45 (RT)

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 19th week of 2022. The testing method was optional. The tests should be finished at 08 July 2022 the latest. With the cover letter along with the sample shipment, the following information was given to the participants:

There are two different samples of bread baking mix. 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 (digital *result submission file*) which have been sent before by email in parallel to the sample shipment.

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

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

All 13 participants submitted at least one result.

3. Evaluation

A specific description of the concept and procedures can be found in the “DLA Evaluation Guide 02.01 (2022) General Proficiency Test Schemes”.

3.1 Quantitative Evaluation

From a total of 5 results on, a quantitative evaluation is carried out, provided that the conditions for a symmetrical distribution of the results and a joint evaluation are met. Frequently, different analytical methods may cause an anomaly in results' distribution. If this is the case, separate evaluations of the different methods with their own assigned values ($X_{pt\ METHOD\ i}$) are performed whenever possible.

Table 5 gives an overview of the evaluation characteristics, their calculation and related criteria. The procedure is described in detail in the accompanying document: DLA Evaluation Guide 02.01 (2022).

Table 5: Evaluation characteristics and criterions for consensus values from participants

Evaluation characteristics	Calculation or criterion
Assigned value ($X_{pt\ ALL}$ or $X_{pt\ METHOD\ i}$)	Robust mean (algorithm A) or median
Standard deviation (S^*_{ALL} or $S^*_{METHOD\ i}$)	Standard deviation (algorithm A)
Target standard deviation (σ_{pt})	From precision experiments according to: ASU L 00.00-18 / L 16.08-1 / L 17.03-1 and ASU L 00.00-94 (see 3.3) [A-E] for all parameters (see 3.2, Table 6)
Target standard deviation (for information)	General model according to: Horwitz ($X_{pt} \geq 120\ \mu\text{g}/\text{kg}$) for all parameters except: Horwitz ($X_{pt} \geq 13,8\ \text{g}/100\text{g}$) for total dietary fiber incl. inulin (methods 2 + 3)
Target range ($X_{pt} \pm 2\sigma_{pt}$ or $2\sigma_{pt}'$)	$X_{pt} \pm 2\sigma_{pt}$ or $2\sigma_{pt}'$
Quotient S^*/σ_{pt} or S^*/σ_{pt}'	$\leq 2,0$ (PT evaluation convincing)
z-Score or z'-Score	$-2 \leq z\text{-score} \leq 2$ (successful) $-2 > z\text{-score} > 2$ (warning signal) $-3 > z\text{-score} > 3$ (action signal)
Kernel density estimation Exclusion of outliers Repeatability standard deviation (S_r) Coefficient of Variation (CV_r) Reproducibility standard deviation (S_R) Coefficient of Variation (CV_R) Standard uncertainty of assigned value ($U_{(X_{pt})}$) Traceability	see DLA Evaluation Guide 02.01 (2022)

3.2 Additional information for the parameters

The following information is supplied in addition to the general information of the DLA Evaluation Guide 02.01 (2022).

Values by precision experiments

The relative repeatability standard deviations (RSD_r) and relative reproducibility standard deviations (RSD_R) given in **table 6** 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 6: Relative repeatability standard deviations (RSD_r) and relative reproducibility standard deviations (RSD_R) according to selected evaluations of tests for precision and the resulting target standard deviation σ_{pt} [A-F]

Parameter	Matrix	Mean [g/100g]	RSD_r	RSD_R	σ_{pt}	Method / Literature
Total Dietary Fiber (enzymatic-gravi- metric)	Mixed rye bread	8,83	2,49 %	5,10 %	4,8 %	ASU L 17.03-1 ASU L 16.08-1
	Grain bran	13,62	5,87 %	12,4 %	11,7 % ¹	
Insoluble Dietary Fiber (enzymatic-gravi- metric)	Mixed rye bread	5,45	5,14 %	8,44 %	7,6 %	ASU L 17.03-1 ASU L 16.08-1
	Grain bran	10,21	6,46 %	14,5 %	13,8 % ¹	
Soluble Dietary Fiber (enzymatic-gravi- metric)	Mixed rye bread	3,02	14,6 %	20,9 %	18,2 % ¹	ASU L 17.03-1
Inulin (enzymatic-photo- metric)	Finished flour baby food chocolate	16,7	1,86 %	3,60 %	3,4 %	ASU L 00.00-94
		0,61	4,92 %	8,20 %	7,4 % ¹	
		5,95	2,35 %	4,03 %	3,7 %	
Total Dietary Fiber including Inulin (enzymatic-gravi- metric + liquid chro- matography)	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 used for evaluation or information (cf. chapter 4)

Values by perception

Table 7 shows selected statistic data of participants' results of the present PT compared to PT results of previous years.

Table 7: Characteristics of the present PT (on grey) in comparison to the previous PT since 2019 (SD = standard deviation, CV = coefficient of variation)

Parameter	Matrix	rob. Mean [g/100g]	rob. SD (S*) [g/100g]	rel. SD (VK _S *) [%]	Quotient S*/opt	DLA Report
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 A+B***
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) A+B***
Total Dietary Fiber without Inulin (1)	Cereal product	10,9 10,9	0,714 0,935	6,55 8,61	0,56 0,74	DLA ptAU01 (2022) A+B****
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 A+B***
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) A+B***
Total Dietary Fiber with Inulin (2+3)	Cereal product	15,2 18,0	1,75 1,58	11,5 8,77	0,73 0,56	DLA ptAU01 (2022) A+B***
Insoluble dietary fiber (1a, 2a, 3a)	Cereal product	2,75 2,75	0,493 0,569	17,9 20,7	1,2* 1,3*	DLA ptAU01 (2021) A+B***
Insoluble dietary fiber (1a, 2a, 3a)	Cereal product	9,22 9,41	1,26 1,50	13,7 15,9	0,99 1,2	DLA ptAU01 (2022) A+B***
Inulin	Cereal product	0,730** 4,73	0,332 0,628	41,0 13,3	2,6* 2,2*	DLA 35/2019 A+B***
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) A+B***
Inulin/Fructans (5+6)	Cereal product	2,01 6,30	0,218 0,304	10,9 4,82	1,5 0,65	DLA ptAU01 (2022) A+B***

* with target standard deviation opt'

** Median

*** results: 1st line Sample A, 2nd line Sample B

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 result chapter, all quantitative results of the participants are displayed formatted to 3 valid digits. In the documentation, all results are given as they were transmitted by the participants. The result tables are structured as below:

Evaluation number	Parameter [Einheit / Unit]	Deviation	z-Score σ_{pt}	z-Score (Info)	Remark

4.1 Total Dietary Fiber without Inulin (in g/100g)

4.1.1 Sample A (Methods: 1)

Proficiency Test

Statistic Data	
Number of results	11
Number of outliers	-
Mean	10,5
Median	10,8
Robust Mean (X_{pt})	10,9
Robust standard deviation (S^*)	0,714
Number with 2 replicates	8
Repeatability SD (S_r)	0,146
Repeatability (CV_r)	1,34%
Reproducibility SD (S_R)	0,524
Reproducibility (CV_R)	4,82%
Target range:	
Target standard deviation σ_{pt}	1,27
Target standard deviation (for Information)	0,305
lower limit of target range	8,36
upper limit of target range	13,5
Quotient S^*/σ_{pt}	0,56
Standard uncertainty $U_{(X_{pt})}$	0,269
Results in the target range	10
Percent in the target range	91%

Comments:

The target standard deviation was calculated according to 3.3 evaluation of a precision experiment (ASU §64 L 16.08-1). In addition, the target standard deviation calculated according to the general model of Horwitz was given for information (s. DLA Evaluation Guide 02.01 (2022) 3.2.6.1).

The distribution of results showed a low variability. The quotient S^*/σ_{pt} was below 1,0. The robust standard deviation was in the range of previous PTs (see 3.3). The repeatability standard deviation was in the range of established values for the used determination methods (s. 3.3). The comparability of results is given.

91% of results were in the target range.

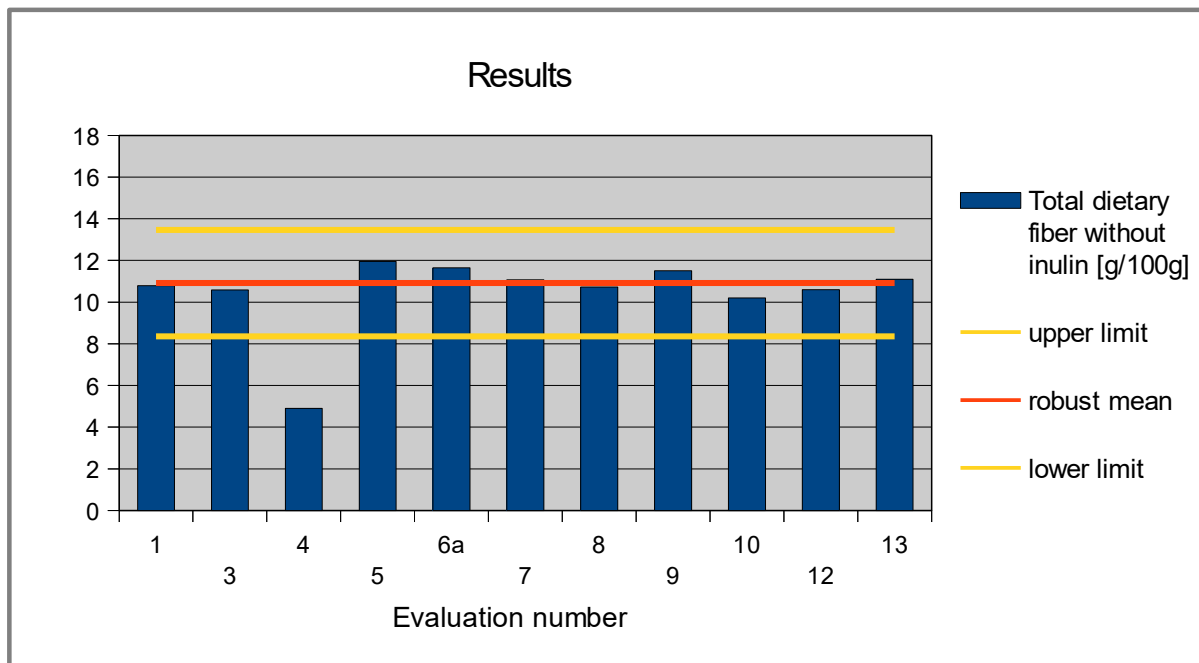


Fig. 1: Results total dietary fiber without inulin, sample A

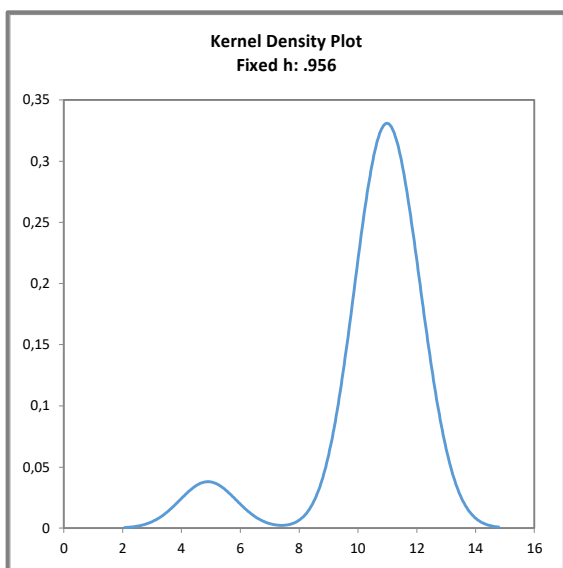


Fig. 2: Kernel density plot of all results (with $h = 0,75 \times \sigma_{pt}$ of $X_{pt,ALL}$)

Comments:

The kernel density estimation shows nearly a symmetrical distribution with a small side peak due to one result outside of the target range.

Results of Participants:

Evaluation number	Total dietary fiber without inulin [g/100g]	Deviation [g/100g]	z-Score (opt)	z-Score (Info)	Method	Remark
1	10,8	-0,12	-0,09	-0,39	1	
3	10,6	-0,33	-0,26	-1,1	1	
4	4,90	-6,01	-4,7	-20	1	
5	12,0	1,05	0,82	3,4	1	
6a	11,7	0,74	0,58	2,4	1 / 2=1	Mean calculated by DLA °
7	11,1	0,16	0,13	0,53	1	
8	10,7	-0,19	-0,15	-0,62	1	
9	11,5	0,59	0,46	1,9	1	
10	10,2	-0,71	-0,56	-2,3	1	
12	10,6	-0,31	-0,24	-1,0	1	
13	11,1	0,19	0,15	0,62	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

° no obvious difference in methodology given (see documentation)

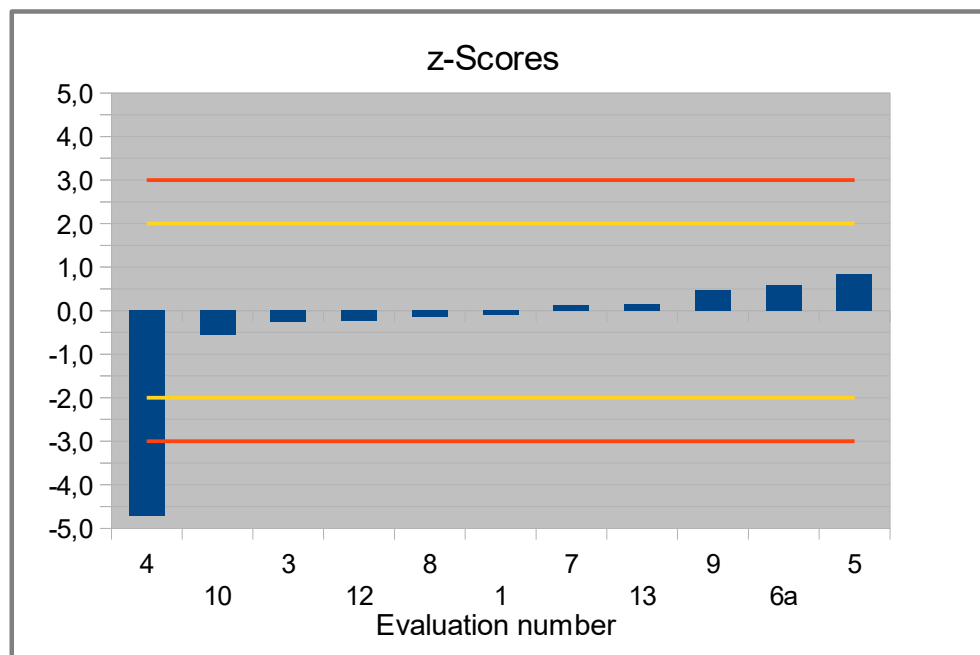


Fig. 3: z-Scores total dietary fiber without inulin, sample A

4.1.2 Sample B (Methods: 1)**Proficiency Test**

Statistic Data	
<i>Number of results</i>	10
<i>Number of outliers</i>	-
Mean	11,0
Median	10,7
Robust Mean (X_{pt})	10,9
Robust standard deviation (S^*)	0,935
<i>Number with 2 replicates</i>	7
Repeatability SD (S_r)	0,116
Repeatability (CV_r)	1,10%
Reproducibility SD (S_R)	0,694
Reproducibility (CV_R)	6,60%
<i>Target range:</i>	
Target standard deviation σ_{pt}	1,27
Target standard deviation (for Information)	0,303
lower limit of target range	8,32
upper limit of target range	13,4
<i>Quotient S^*/σ_{pt}</i>	<i>0,74</i>
<i>Standard uncertainty $U(X_{pt})$</i>	<i>0,370</i>
<i>Results in the target range</i>	9
<i>Percent in the target range</i>	90%

Comments:

The target standard deviation was calculated according to 3.3 evaluation of a precision experiment (ASU §64 L 16.08-1). In addition, the target standard deviation calculated according to the general model of Horwitz was given for information (s. DLA Evaluation Guide 02.01 (2022) 3.2.6.1).

The distribution of results showed a low variability. The quotient S^*/σ_{pt} was below 1,0. The robust standard deviation was in the range of previous PTs (see 3.3). The repeatability standard deviation was in the range of established values for the used determination methods (s. 3.3). The comparability of results is given.

90% of results were in the target range.

Note: For methods 1, the robust means for sample A (without addition of inulin) and sample B (with addition of 4,98g/100g inulin) are almost the same (each 10,9 g/100g).

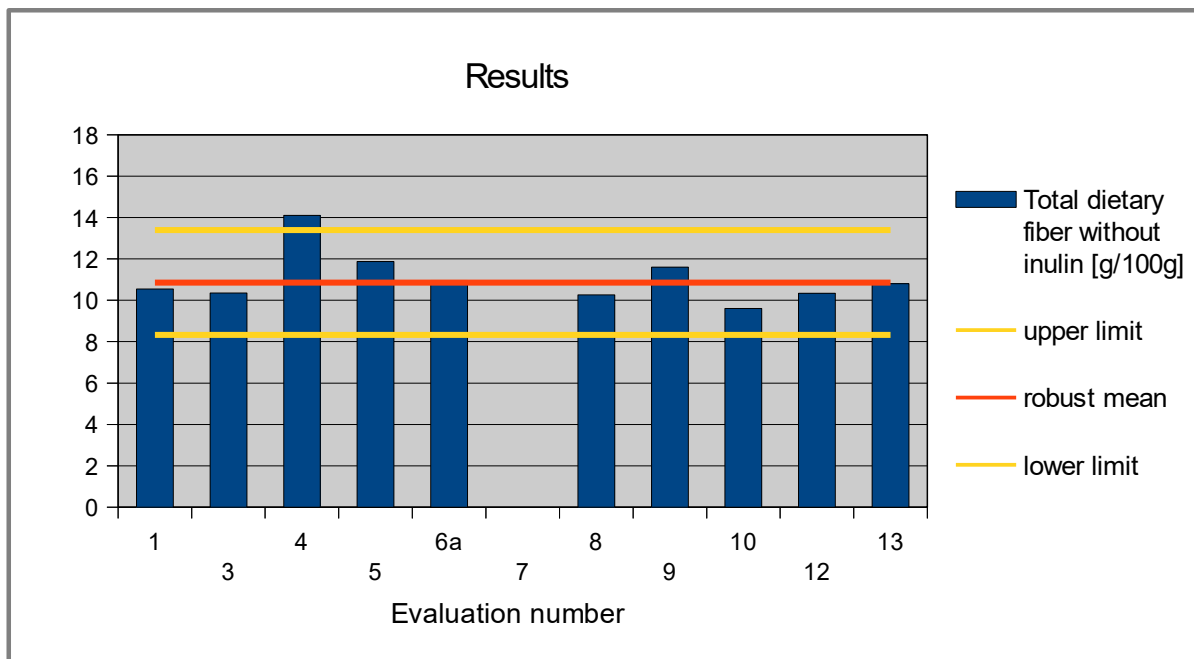


Fig. 4: Results total dietary fiber without inulin, sample B

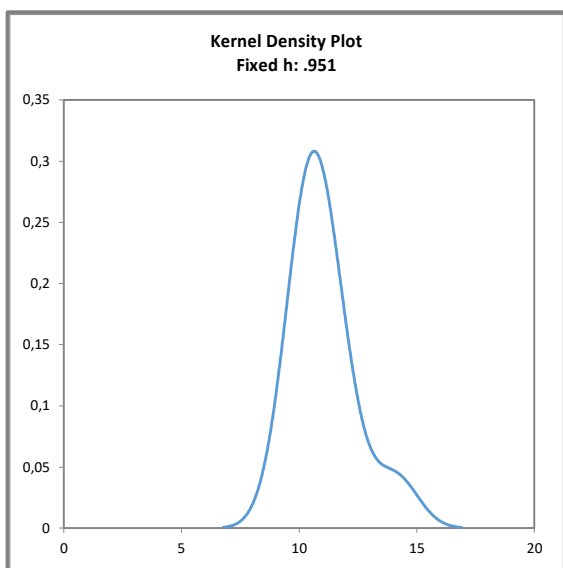


Fig. 5: Kernel density plot of all results (with $h = 0,75 \times \sigma_{pt}$ of $X_{pt,ALL}$)

Comments:

The kernel density estimation shows nearly a symmetrical distribution with a shoulder due to one result outside of the target range.

Results of Participants:

Evaluation number	Total dietary fiber without inulin [g/100g]	Deviation [g/100g]	z-Score (opt)	z-Score (Info)	Method	Remark
1	10,5	-0,32	-0,25	-1,0	1	
3	10,4	-0,51	-0,40	-1,7	1	
4	14,1	3,24	2,6	11	1	
5	11,9	1,01	0,80	3,3	1	
6a	11,0	0,09	0,07	0,31	1 / 2=1	Mean calculated by DLA °
7					1	
8	10,3	-0,60	-0,47	-2,0	1	
9	11,6	0,74	0,59	2,4	1	
10	9,60	-1,26	-0,99	-4,1	1	
12	10,3	-0,52	-0,41	-1,7	1	
13	10,8	-0,06	-0,04	-0,19	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

° no obvious difference in methodology given (see documentation)

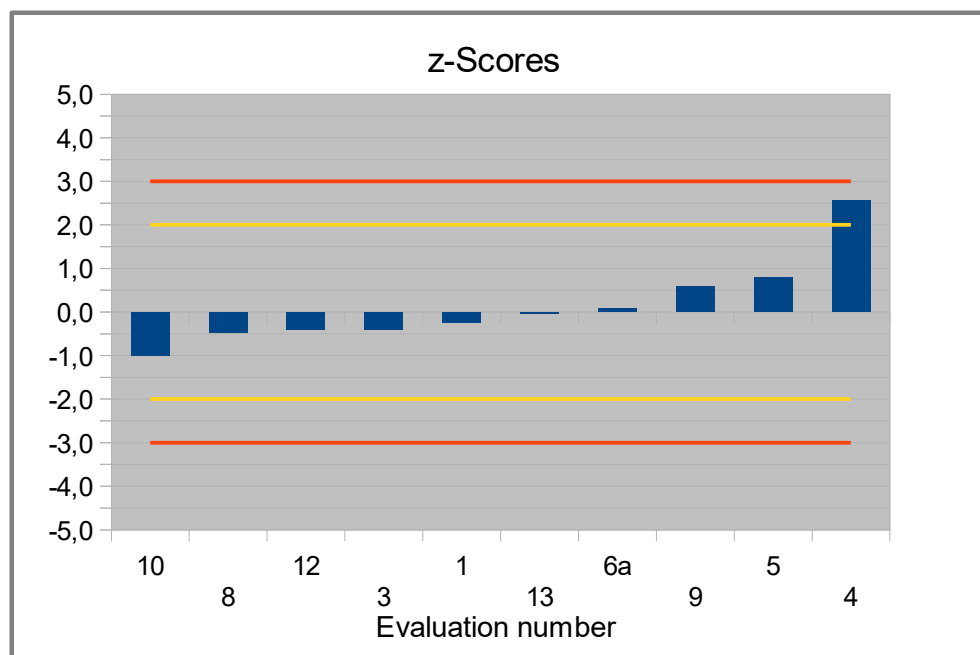


Fig. 6: z-Scores total dietary fiber without inulin, sample B

4.2 Total Dietary Fiber with Inulin (in g/100g)

4.2.1 Sample A (Methods: 2 + 3)

Proficiency Test

Statistic Data	
<i>Number of results</i>	9
<i>Number of outliers</i>	0
Mean	15,2
Median	14,7
Robust Mean (X_{pt})	15,2
Robust standard deviation (S^*)	1,75
<i>Number with 2 replicates</i>	4
Repeatability SD (S_r)	1,71
Repeatability (CV_r)	11,9%
Reproducibility SD (S_R)	-
Reproducibility (CV_R)	-
<i>Target range:</i>	
Target standard deviation σ_{pt}	2,39
Target standard deviation (for Information)	0,389
lower limit of target range	10,4
upper limit of target range	19,9
<i>Quotient S^*/σ_{pt}</i>	<i>0,73</i>
<i>Standard uncertainty $U_{(X_{pt})}$</i>	<i>0,728</i>
<i>Results in the target range</i>	9
<i>Percent in the target range</i>	100%

Comments:

The target standard deviation was calculated according to 3.3 evaluation of a precision experiment (AOAC 2009.01). In addition, the target standard deviation calculated according to the general model of Horwitz was given for information (s. DLA Evaluation Guide 02.01 (2022) 3.2.6.1).

The distribution of results showed a low variability. The quotient S^*/σ_{pt} was below 1,0. The robust standard deviation was in the range of previous PTs (see 3.3). The repeatability standard deviation was in the range of established values for the used determination methods (s. 3.3). The comparability of results is given. This conclusion can be limited for the evaluation across different methods.

100% of results were in the target range.

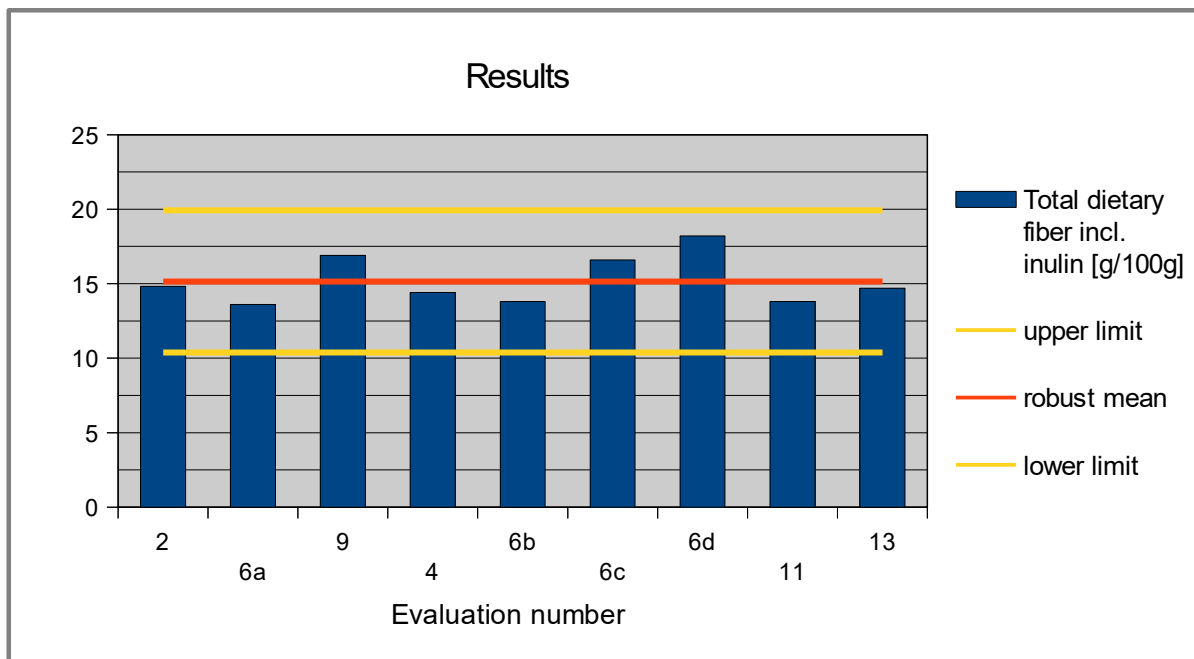


Fig. 7: Results total dietary fiber with inulin, sample A

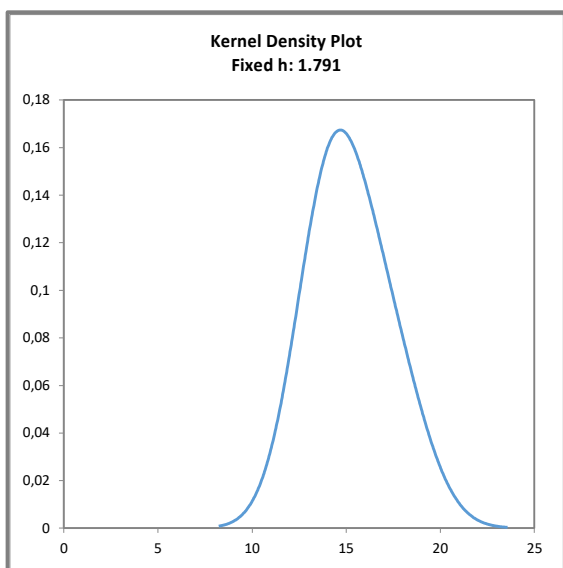


Fig. 8: Kernel density plot of all results (with $h = 0,75 \times \sigma_{pt}$ of $X_{pt,ALL}$)

Comments:

The kernel density estimation shows nearly a symmetrical distribution.

Results of Participants:

Evaluation number	Total dietary fiber incl. inulin [g/100g]	Deviation [g/100g]	z-Score (opt)	z-Score (Info)	Method	Remark
2	14,8	-0,33	-0,14	-0,86	4=2	Method 2 assigned by DLA
6a	13,6	-1,55	-0,65	-4,0	2	
9	16,9	1,75	0,73	4,5	2	
4	14,4	-0,75	-0,32	-1,9	3	
6b	13,8	-1,35	-0,57	-3,5	3	
6c	16,6	1,45	0,61	3,7	3	
6d	18,2	3,05	1,3	7,8	1=3	Method 3 assigned by DLA
11	13,8	-1,35	-0,57	-3,5	4=3	Method 3 assigned by DLA
13	14,7	-0,45	-0,19	-1,2	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)

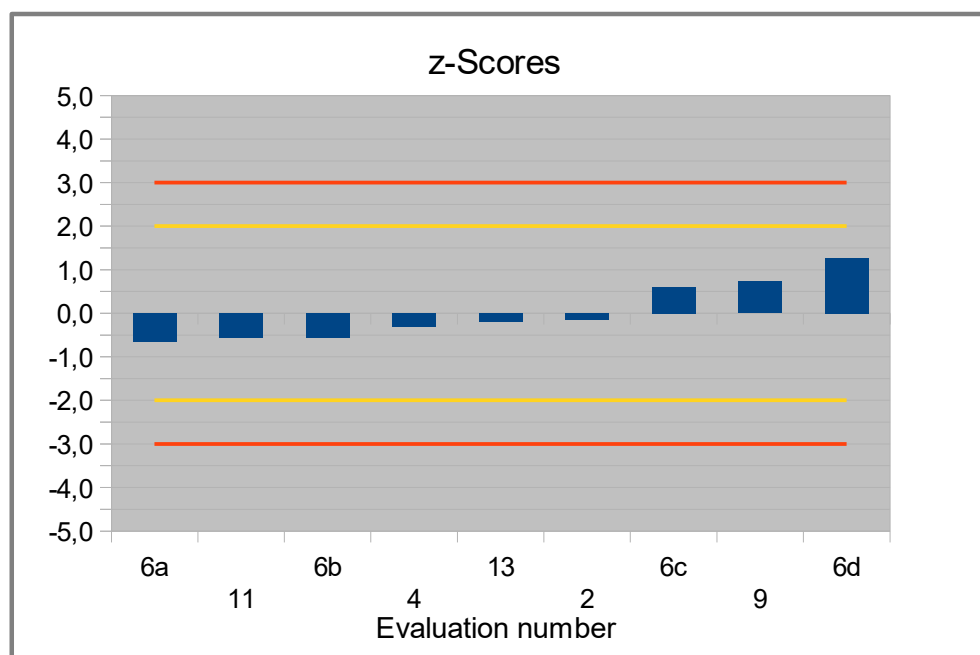


Fig. 9: z-Scores total dietary fiber with inulin, sample A

4.2.2 Sample B (Methods: 2 + 3)**Proficiency Test**

Statistic Data	
<i>Number of results</i>	9
<i>Number of outliers</i>	0
Mean	18,1
Median	17,7
Robust Mean (X_{pt})	18,0
Robust standard deviation (S^*)	1,58
<i>Number with 2 replicates</i>	4
Repeatability SD (S_r)	2,73
Repeatability (CV_r)	15,7%
Reproducibility SD (S_R)	-
Reproducibility (CV_R)	-
<i>Target range:</i>	
Target standard deviation σ_{pt}	2,84
Target standard deviation (for Information)	0,424
lower limit of target range	12,3
upper limit of target range	23,7
<i>Quotient S^*/σ_{pt}</i>	<i>0,56</i>
<i>Standard uncertainty $U_{(X_{pt})}$</i>	<i>0,658</i>
<i>Results in the target range</i>	9
<i>Percent in the target range</i>	100%

Comments:

The target standard deviation was calculated according to 3.3 evaluation of a precision experiment (AOAC 2009.01). In addition, the target standard deviation calculated according to the general model of Horwitz was given for information (s. DLA Evaluation Guide 02.01 (2022) 3.2.6.1).

The distribution of results showed a low variability. The quotient S^*/σ_{pt} was below 1,0. The robust standard deviation was in the range of previous PTs (see 3.3). The repeatability standard deviation was in the upper range of established values for the used determination methods (s. 3.3). The comparability of results is given. This conclusion can be limited for the evaluation across different methods.

100% of results were in the target range.

Note: For methods 2 and 3, the difference of the robust means for sample A (without addition of inulin) and sample B (with addition of 4,98g/100g inulin) is 2,80g/100g.

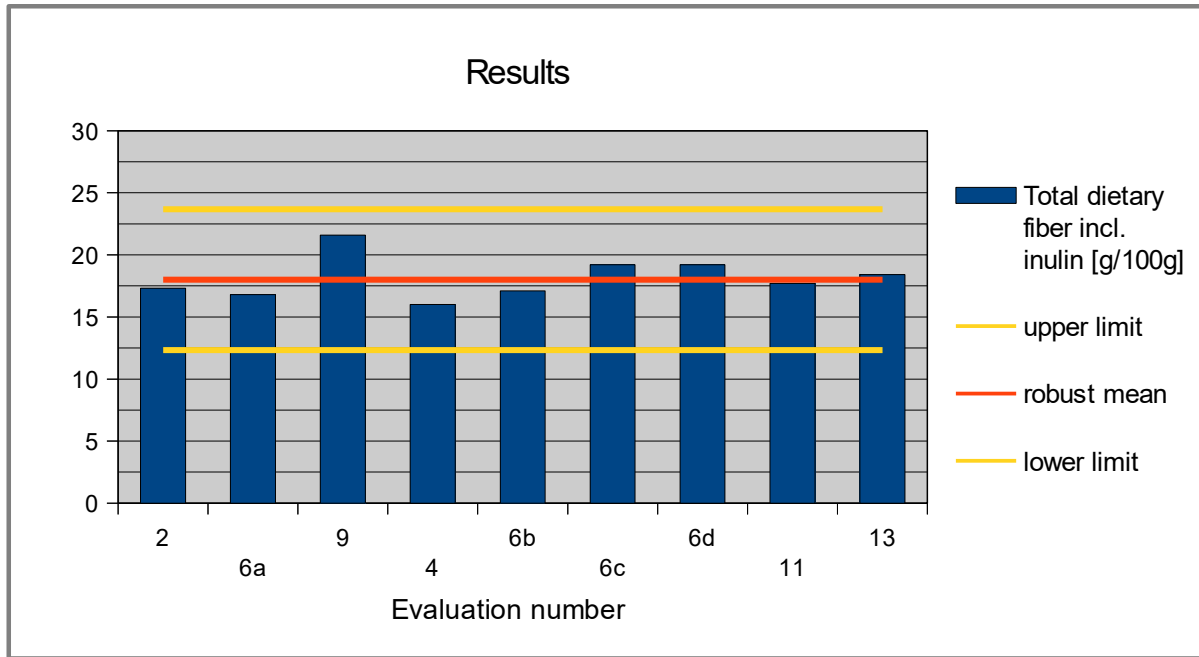


Fig. 10: Results total dietary fiber with inulin, sample B

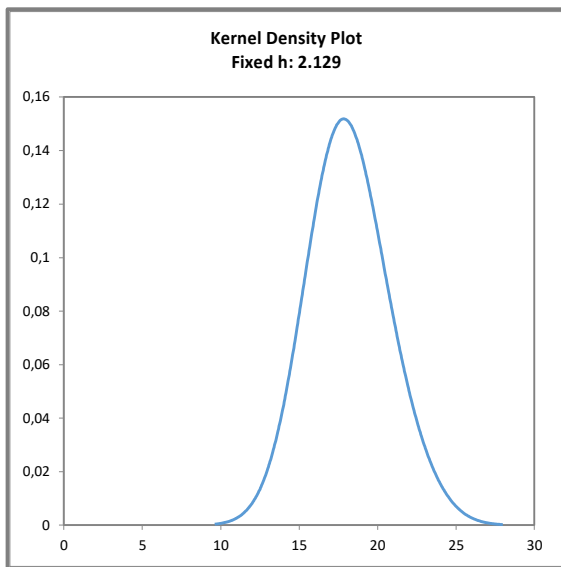


Fig. 11: Kernel density plot of all results (with $h = 0,75 \times \sigma_{pt}$ of $X_{pt,ALL}$)

Comments:

The kernel density estimation shows nearly a symmetrical distribution.

Results of Participants:

Evaluation number	Total dietary fiber incl. inulin [g/100g]	Deviation [g/100g]	z-Score (opt)	z-Score (Info)	Method	Remark
2	17,3	-0,70	-0,25	-1,6	4=2	Method 2 assigned by DLA
6a	16,8	-1,21	-0,43	-2,9	2	
9	21,6	3,59	1,3	8,5	2	
4	16,0	-2,01	-0,71	-4,7	3	
6b	17,1	-0,91	-0,32	-2,1	3	
6c	19,2	1,19	0,42	2,8	3	
6d	19,2	1,19	0,42	2,8	1=3	Method 3 assigned by DLA
11	17,7	-0,31	-0,11	-0,73	4=3	Method 3 assigned by DLA
13	18,4	0,39	0,14	0,92	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)

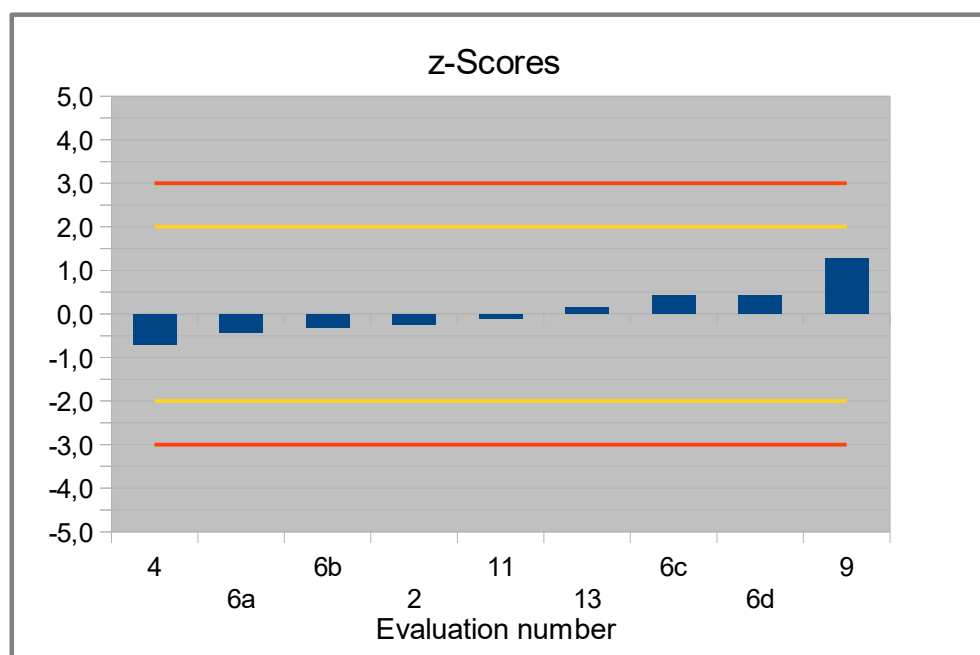


Fig. 12: z-Scores total dietary fiber with inulin, sample B

4.3 Insoluble Dietary Fiber (in g/100g)

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

Proficiency Test

Statistic Data	
Number of results	11
Number of outliers	-
Mean	9,05
Median	9,27
Robust Mean (X_{pt})	9,22
Robust standard deviation (S^*)	1,26
Number with 2 replicates	5
Repeatability SD (S_r)	0,183
Repeatability (CV_r)	1,97%
Reproducibility SD (S_R)	0,616
Reproducibility (CV_R)	6,63%
Target range:	
Target standard deviation σ_{pt}	1,27
Target standard deviation (for Information)	0,264
lower limit of target range	6,68
upper limit of target range	11,8
Quotient S^*/σ_{pt}	0,99
Standard uncertainty $U_{(X_{pt})}$	0,475
Results in the target range	9
Percent in the target range	82%

Comments:

The target standard deviation was calculated according to 3.3 evaluation of a precision experiment (ASU §64 L 16.08-1). In addition, the target standard deviation calculated according to the general model of Horwitz was given for information (s. DLA Evaluation Guide 02.01 (2022) 3.2.6.1).

The distribution of results showed a low variability. The quotient S^*/σ_{pt} was below 1,0. The robust standard deviation was in the range of previous PTs (see 3.3). The repeatability standard deviation was in the range of established values for the used determination methods (s. 3.3). The comparability of results is given. This conclusion can be limited for the evaluation across different methods.

82% of results were in the target range.

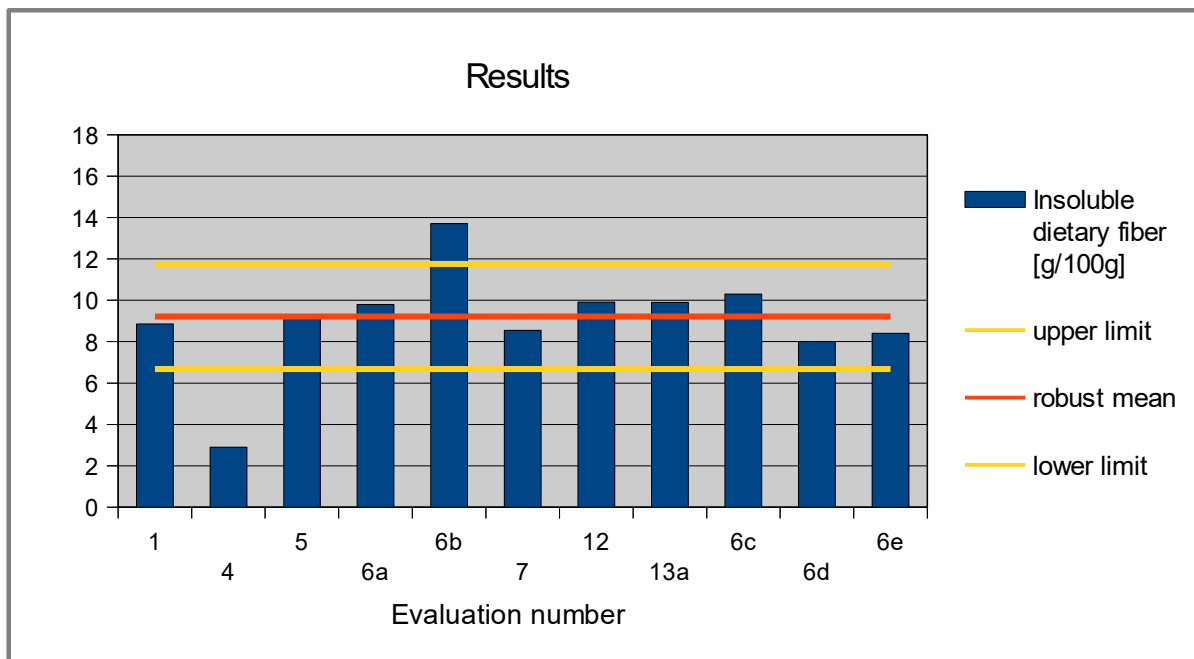


Fig. 13: Results insoluble dietary fiber, sample A

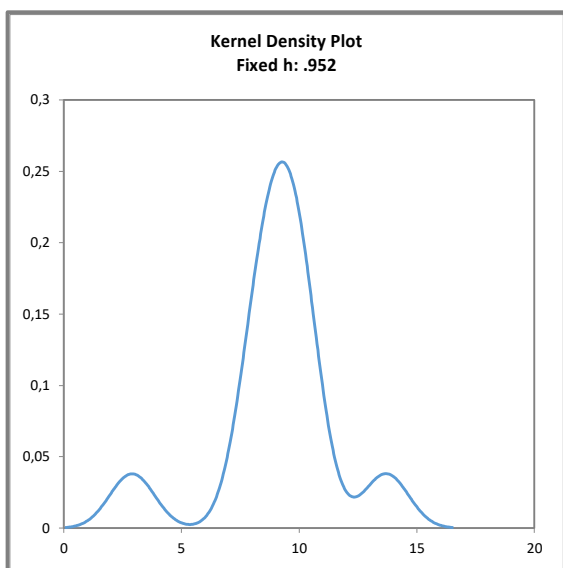


Fig. 14: Kernel density plot of all results (with $h = 0,75 \times \sigma_{pt,ALL}$)

Comments:

The kernel density estimation shows nearly a symmetrical distribution with two small side peaks due to two results outside of the target range.

Results of Participants:

Evaluation number	Insoluble dietary fiber [g/100g]	Deviation [g/100g]	z-Score (opt)	z-Score (Info)	Method	Remark
1	8,85	-0,369	-0,29	-1,4	1a	
4	2,90	-6,32	-5,0	-24	1a	
5	9,27	0,051	0,04	0,19	1a	
6a	9,80	0,581	0,46	2,2	2a=1a	Method 1a assigned by DLA
6b	13,7	4,48	3,5	17	1a	
7	8,54	-0,679	-0,54	-2,6	1a	
12	9,91	0,691	0,54	2,6	1a	
13a	9,90	0,681	0,54	2,6	1a	
6c	10,3	1,08	0,85	4,1	2a	
6d	8,00	-1,22	-0,96	-4,6	3a	
6e	8,40	-0,819	-0,65	-3,1	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 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)



Fig. 15: z-Scores insoluble dietary fiber, sample A

4.3.2 Sample B (Methods: 1a + 2a + 3a)**Proficiency Test**

Statistic Data	
Number of results	10
Number of outliers	0
Mean	9,45
Median	9,38
Robust Mean (X_{pt})	9,41
Robust standard deviation (S^*)	1,50
Number with 2 replicates	5
Repeatability SD (S_r)	0,289
Repeatability (CV_r)	3,01%
Reproducibility SD (S_R)	1,40
Reproducibility (CV_R)	14,6%
Target range:	
Target standard deviation σ_{pt}	1,30
Target standard deviation (for Information)	0,269
lower limit of target range	6,82
upper limit of target range	12,0
Quotient S^*/σ_{pt}	1,2
Standard uncertainty $U_{(X_{pt})}$	0,592
Results in the target range	10
Percent in the target range	100%

Comments:

The target standard deviation was calculated according to 3.3 evaluation of a precision experiment (ASU §64 L 16.08-1). In addition, the target standard deviation calculated according to the general model of Horwitz was given for information (s. DLA Evaluation Guide 02.01 (2022) 3.2.6.1).

The distribution of results showed a normal variability. The quotient S^*/σ_{pt} was below 2,0. The robust standard deviation was in the range of previous PTs (see 3.3). The repeatability standard deviation was in the range of established values for the used determination methods (s. 3.3). The comparability of results is given. This conclusion can be limited for the evaluation across different methods.

100% of results were in the target range.

Note: For methods 1a, 2a and 3a, the difference of the robust means for sample A (without addition of inulin) and sample B (with addition of 4,98g/100g inulin) is 0,19g/100g.

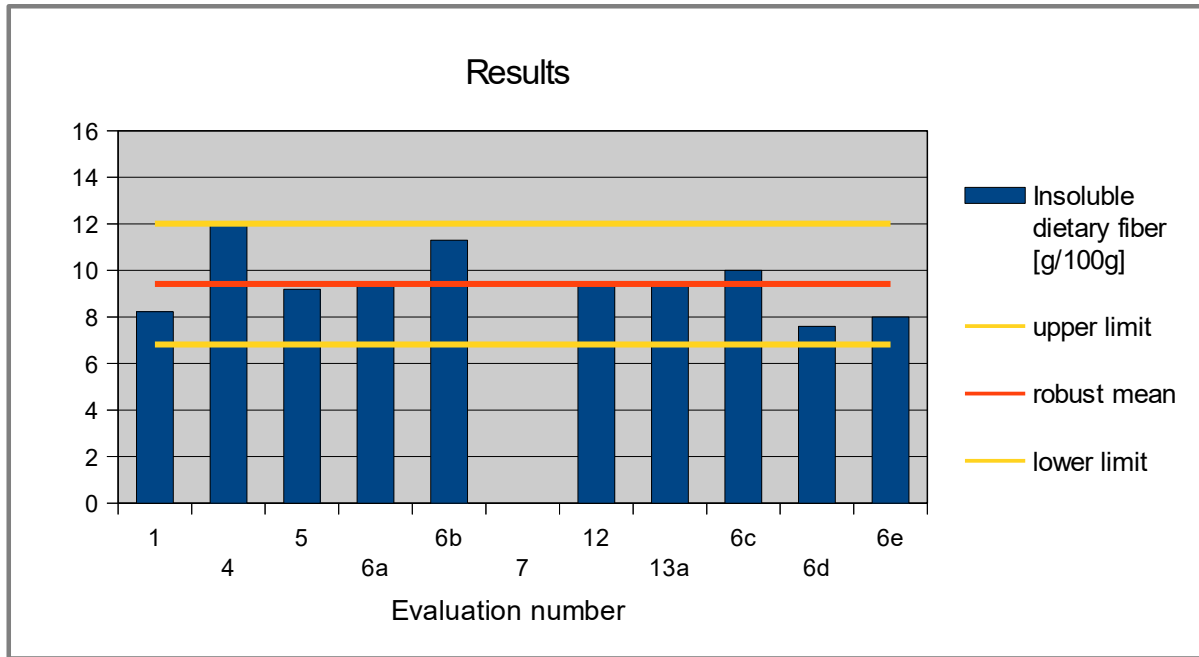


Fig. 16: Results insoluble dietary fiber, sample B

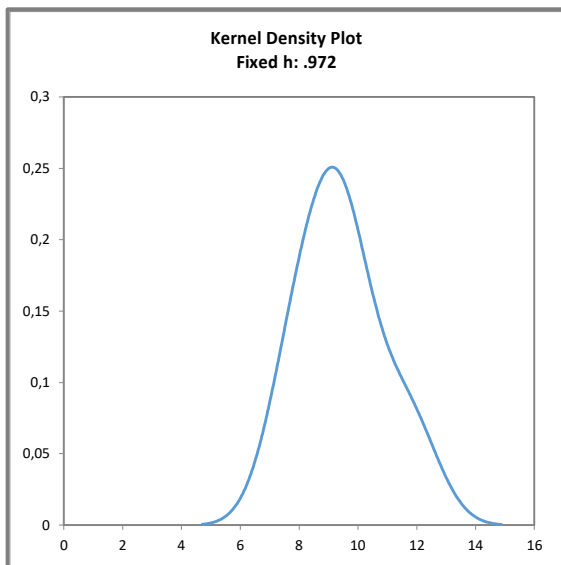


Fig. 17: Kernel density plot of all results (with $h = 0,75 \times \sigma_{pt,ALL}$)

Comments:

The kernel density estimation shows nearly a symmetrical distribution with a slight shoulder.

Results of Participants:

Evaluation number	Insoluble dietary fiber [g/100g]	Deviation [g/100g]	z-Score (opt)	z-Score (Info)	Method	Remark
1	8,23	-1,18	-0,91	-4,4	1a	
4	12,0	2,59	2,0	9,6	1a	
5	9,19	-0,223	-0,17	-0,83	1a	
6a	9,40	-0,013	-0,01	-0,05	2a=1a	Method 1a assigned by DLA
6b	11,3	1,89	1,5	7,0	1a	
7					1a	
12	9,35	-0,063	-0,05	-0,23	1a	
13a	9,40	-0,013	-0,01	-0,05	1a	
6c	10,0	0,587	0,45	2,2	2a	
6d	7,60	-1,81	-1,4	-6,7	3a	
6e	8,00	-1,41	-1,1	-5,3	3a	

- (1) enzymatic-gravimetric methods like ASUL 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 with 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)

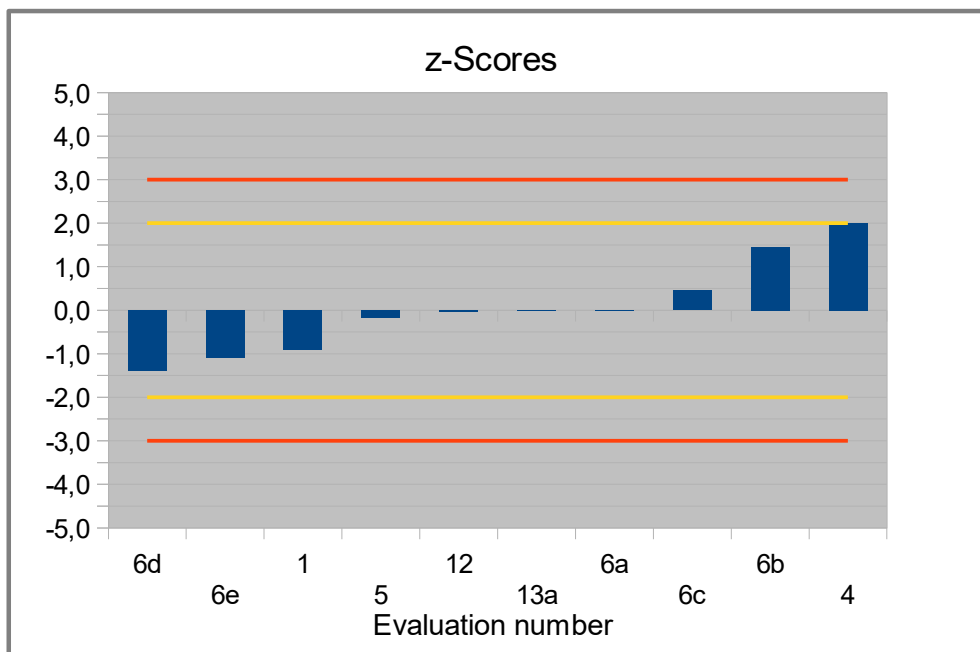


Fig. 18: z-Scores insoluble dietary fiber, sample B

4.4 Soluble Dietary Fiber (in g/100g)

4.4.1 Sample A (Methods: 1b + 2b)

Proficiency Test

Statistic Data	
<i>Number of results</i>	8
<i>Number of outliers</i>	0
Mean	2,68
Median	2,60
Robust Mean (X_{pt})	2,59
Robust standard deviation (S^*)	0,834
<i>Number with 2 replicates</i>	5
Repeatability SD (S_r)	0,0315
Repeatability (CV_r)	1,37%
Reproducibility SD (S_R)	0,513
Reproducibility (CV_R)	22,3%
<i>Target range:</i>	
Target standard deviation σ_{pt}	0,471
Target standard deviation (for Information)	0,0899
lower limit of target range	1,65
upper limit of target range	3,54
<i>Quotient S^*/σ_{pt}</i>	1,8
<i>Standard uncertainty $U_{(X_{pt})}$</i>	0,369
<i>Results in the target range</i>	6
<i>Percent in the target range</i>	75%

Comments:

The target standard deviation was calculated according to 3.3 evaluation of a precision experiment (ASU §64 L 17.03-1). In addition, the target standard deviation calculated according to the general model of Horwitz was given for information (s. DLA Evaluation Guide 02.01 (2022) 3.2.6.1).

The distribution of results showed a normal variability. The quotient S^*/σ_{pt} was below 2,0.

The robust standard deviation was in the range of previous PTs (see 3.3).

The comparability of results can be limited due to the low number of results and evaluation across different methods.

75% of results were in the target range.

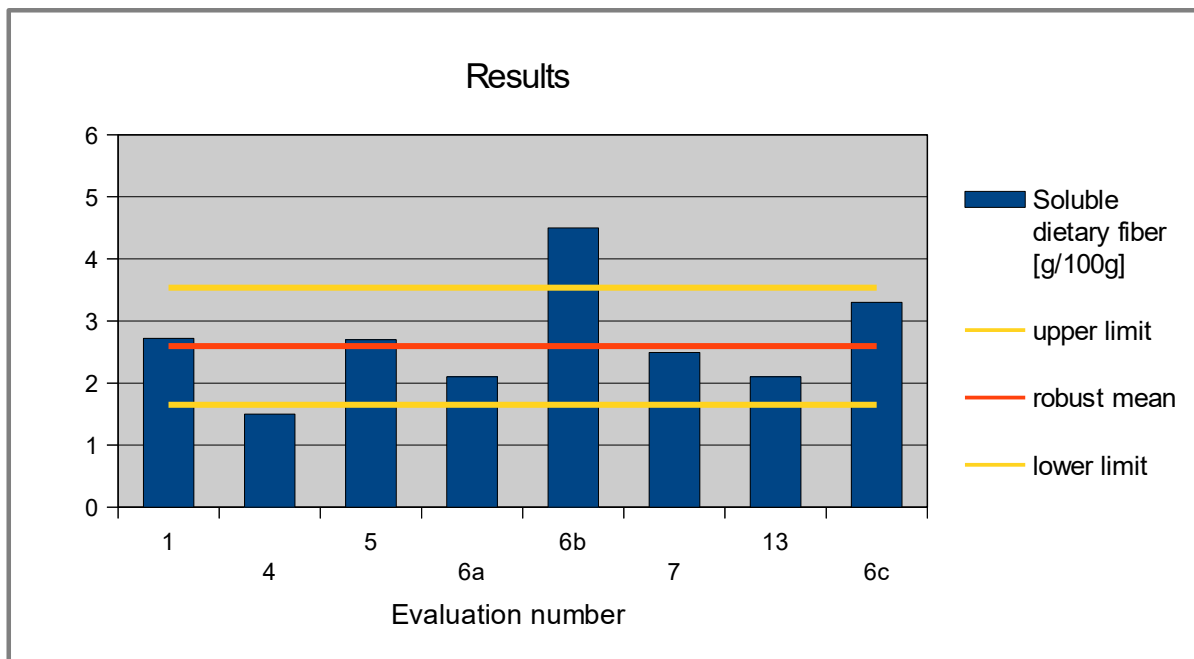


Fig. 19: Results soluble dietary fiber, sample A

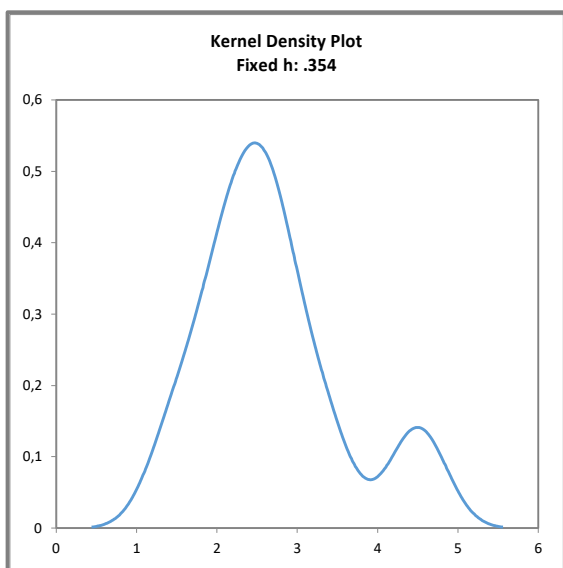


Fig. 20: Kernel density plot of all results (with $h = 0,75 \times \sigma_{pt}$ of $X_{pt,ALL}$)

Comments:

The kernel density estimation shows nearly a symmetrical distribution with a smaller side peak due to one higher single value.

Results of Participants:

Evaluation number	Soluble dietary fiber [g/100g]	Deviation [g/100g]	z-Score (opt)	z-Score (Info)	Method	Remark
1	2,72	0,126	0,27	1,4	1b	
4	1,50	-1,09	-2,3	-12	1b	
5	2,70	0,106	0,22	1,2	1b	
6a	2,10	-0,494	-1,0	-5,5	2b=1b	Method 1b assigned by DLA
6b	4,50	1,91	4,0	21	1b	
7	2,49	-0,104	-0,22	-1,2	1b	
13	2,10	-0,494	-1,0	-5,5	1b	
6c	3,30	0,706	1,5	7,8	2b	

(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

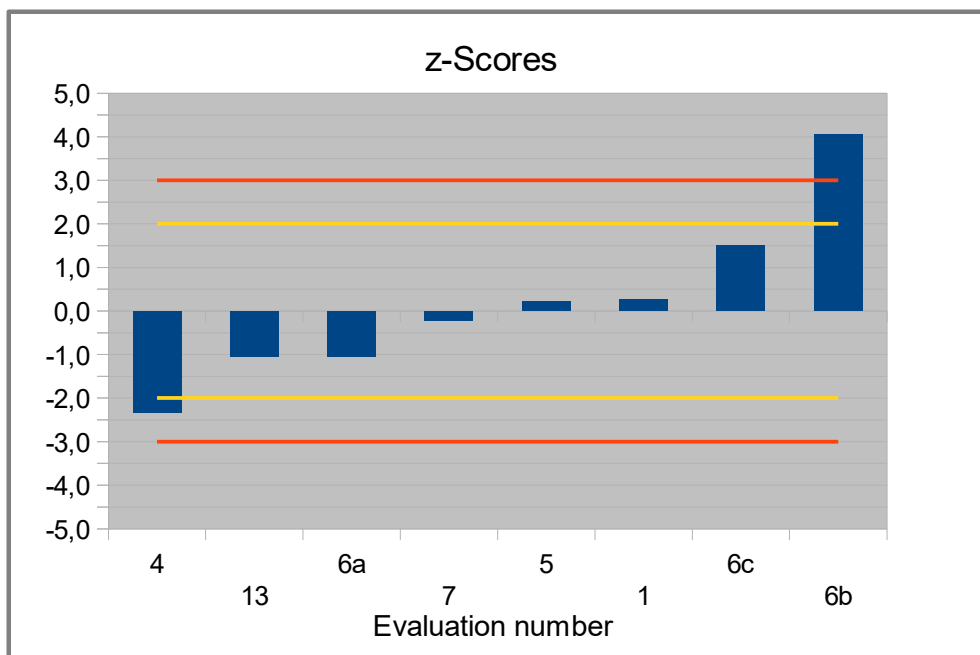


Fig. 21: z-Scores soluble dietary fiber, sample A

4.4.2 Sample B (Methods: 1b + 2b)**Proficiency Test**

Statistic Data	
Number of results	5 °
Number of outliers	2
Mean	2,24
Median	2,10
Robust Mean (X_{pt})	2,24
Robust standard deviation (S^*)	0,557
Number with 2 replicates	4
Repeatability SD (S_r)	0,153
Repeatability (CV_r)	6,55%
Reproducibility SD (S_R)	0,514
Reproducibility (CV_R)	22,0%
Target range:	
Target standard deviation σ_{pt}	0,407
Target standard deviation (for Information)	0,0794
lower limit of target range	1,43
upper limit of target range	3,06
Quotient S^*/σ_{pt}	1,4
Standard uncertainty $U(X_{pt})$	0,311
Results in the target range	5
Percent in the target range	100%

° number without outliers

Comments:

The target standard deviation was calculated according to 3.3 evaluation of a precision experiment (ASU §64 L 17.03-1). In addition, the target standard deviation calculated according to the general model of Horwitz was given for information (s. DLA Evaluation Guide 02.01 (2022) 3.2.6.1).

The distribution of results showed a normal variability. The quotient S^*/σ_{pt} was below 2,0.

The robust standard deviation was in the range of previous PTs (see 3.3).

The comparability of results can be limited due to the low number of results and evaluation across different methods.

100% of results were in the target range.

Note: For methods 1b and 2b, the difference of the robust means for sample A (without addition of inulin) and sample B (with addition of 4,98g/100g inulin) is 0,35g/100g.

The difference of the results for sample A and B of evaluation no. 6b and 6c was 3,4 g/100g and 3,5 g/100g, respectively.

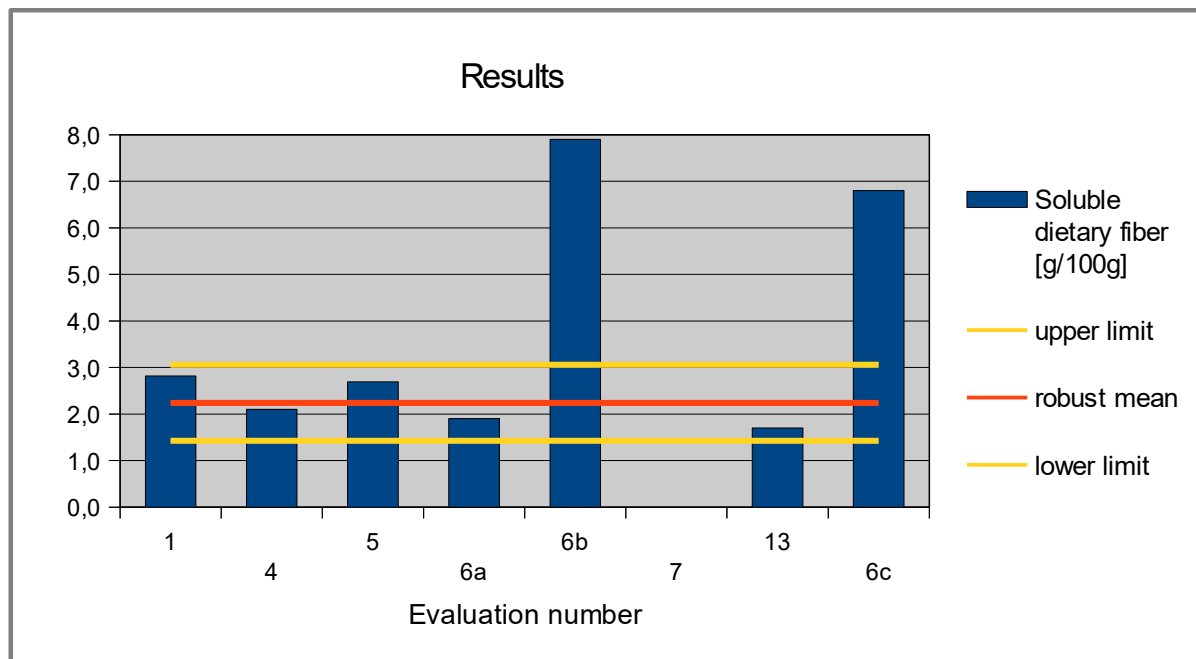


Fig. 22: Results soluble dietary fiber, sample B

Note:

Due to the low number of < 8 results a kernel density estimation could not be carried out.

Results of Participants:

Evaluation number	Soluble dietary fiber [g/100g]	Deviation [g/100g]	z-Score (opt)	z-Score (Info)	Method	Remark
1	2,82	0,578	1,4	7,3	1b	
4	2,10	-0,142	-0,35	-1,8	1b	
5	2,69	0,448	1,1	5,6	1b	
6a	1,90	-0,342	-0,84	-4,3	2b=1b	Method 1b assigned by DLA
6b	7,90				1b	outlier, result excluded
7					1b	
13	1,70	-0,542	-1,3	-6,8	1b	
6c	6,80				2b	outlier, result excluded

(1) enzymatic-gravimetric methods like ASUL 00.00-18 (ASUL 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

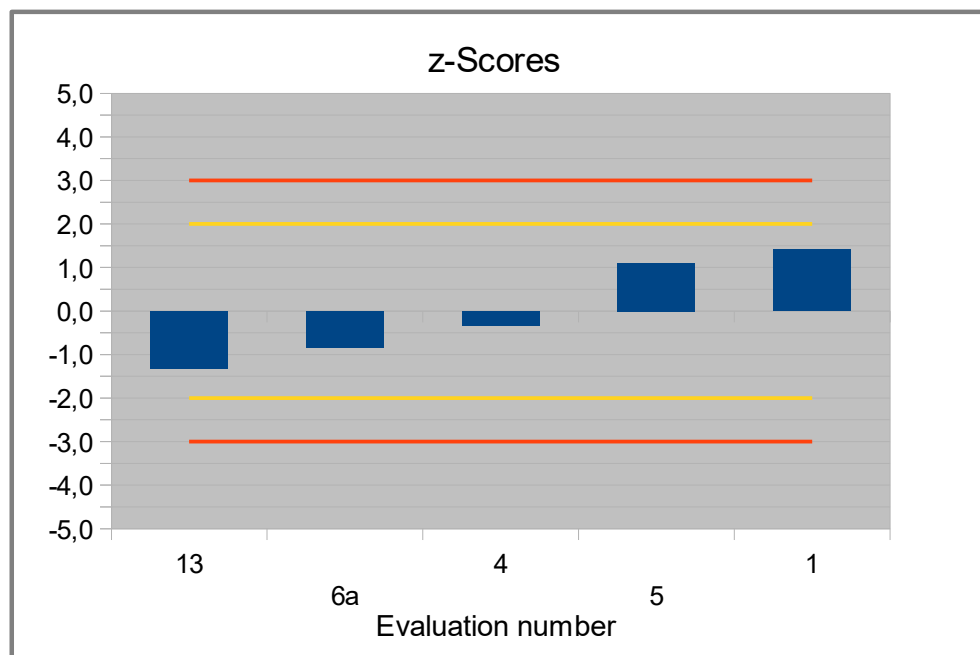


Fig. 23: z-Scores soluble dietary fiber, sample B

4.5 Soluble HMW and LMW Dietary Fiber (in g/100g)

In the cases of methods 3b and 3c for soluble high molecular weight (HMW) and low molecular weight (LMW) dietary fiber fewer than 5 results were available. Therefore a statistical evaluation of the results was not carried out. The results are given in the documentation.

4.6 Inulin / Fructans (in g/100g)

4.6.1 Sample A (Methods: 5 + 6)

Proficiency Test

Statistic Data	
<i>Number of results</i>	8
<i>Number of outliers</i>	0
Mean	2,01
Median	2,05
Robust Mean (X_{pt})	2,01
Robust standard deviation (S^*)	0,218
<i>Number with 2 replicates</i>	5
Repeatability SD (S_r)	0,0535
Repeatability (CV_r)	2,59%
Reproducibility SD (S_R)	0,218
Reproducibility (CV_R)	10,6%
<i>Target range:</i>	
Target standard deviation σ_{pt}	0,149
Target standard deviation (for Information)	0,0722
lower limit of target range	1,71
upper limit of target range	2,30
<i>Quotient S^*/σ_{pt}</i>	1,5
<i>Standard uncertainty $U_{(X_{pt})}$</i>	0,0963
<i>Results in the target range</i>	8
<i>Percent in the target range</i>	100%

Comments:

The target standard deviation was calculated according to 3.3 evaluation of a precision experiment (ASU §64 L 00.00-94). In addition, the target standard deviation calculated according to the general model of Horwitz was given for information (s. DLA Evaluation Guide 02.01 (2022) 3.2.6.1).

The distribution of results showed a normal variability. The quotient S^*/σ_{pt} was below 2,0. The robust standard deviation was in the range of previous PTs (see 3.3). The repeatability standard deviation was in the range of established values for the used determination methods (s. 3.3). The comparability of results can be limited due to the low number of results and evaluation across different methods.

100% of results were in the target range.

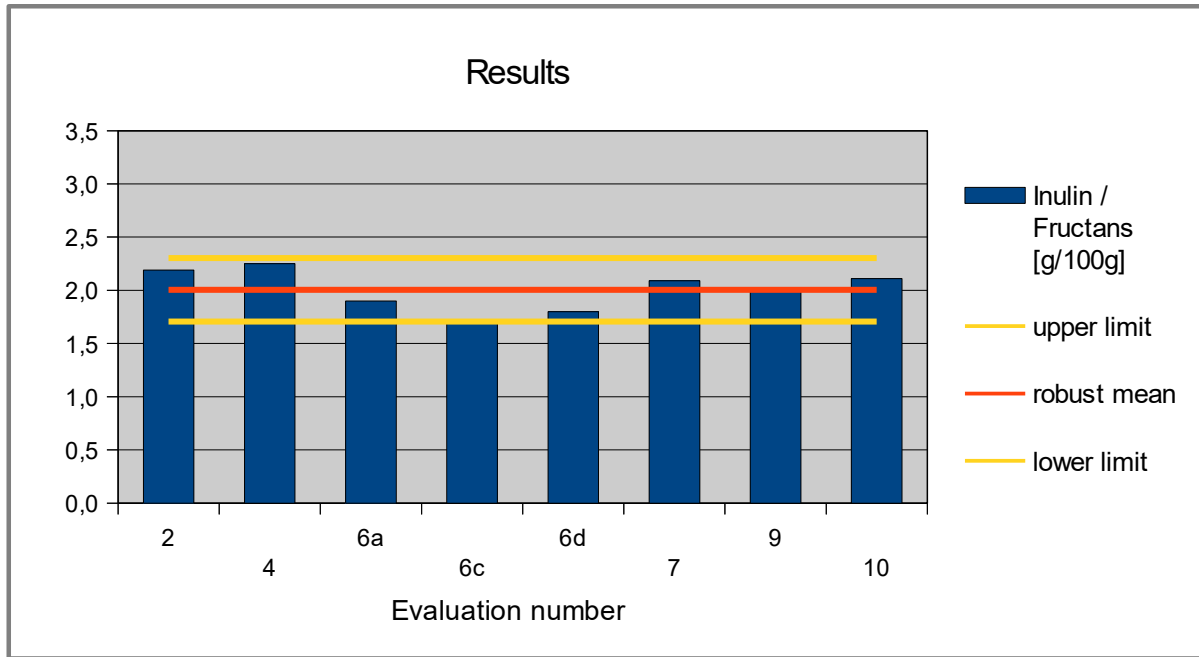


Fig. 24: Results inulin / fructans, sample A

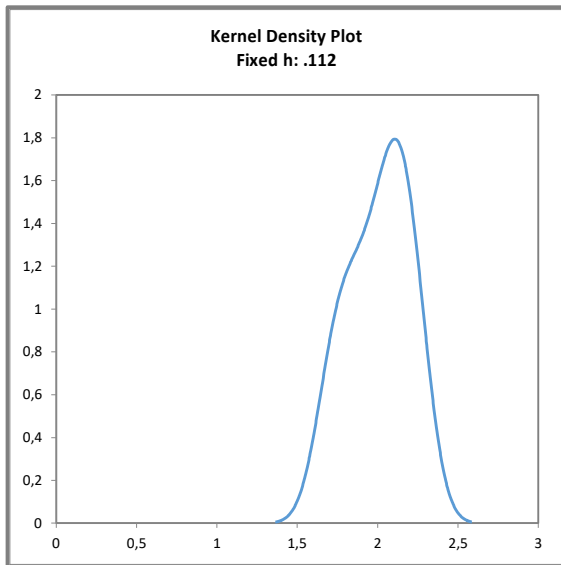


Fig. 25: Kernel density plot of all results (with $h = 0,75 \times \sigma_{pt\text{ALL}}$)

Comments:

The kernel density estimation shows nearly a symmetrical distribution with a slight shoulder.

Results of Participants:

Evaluation number	Inulin / Fructans [g/100g]	Deviation [g/100g]	z-Score (opt)	z-Score (Info)	Method	Remark
2	2,19	0,185	1,2	2,6	5	
4	2,25	0,245	1,6	3,4	5	
6a	1,90	-0,105	-0,71	-1,5	5	
6c	1,70	-0,305	-2,0	-4,2	6	
6d	1,80	-0,205	-1,4	-2,8	6	
7	2,09	0,085	0,57	1,2	6	
9	2,00	-0,005	-0,03	-0,07	6	
10	2,11	0,105	0,71	1,5	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)

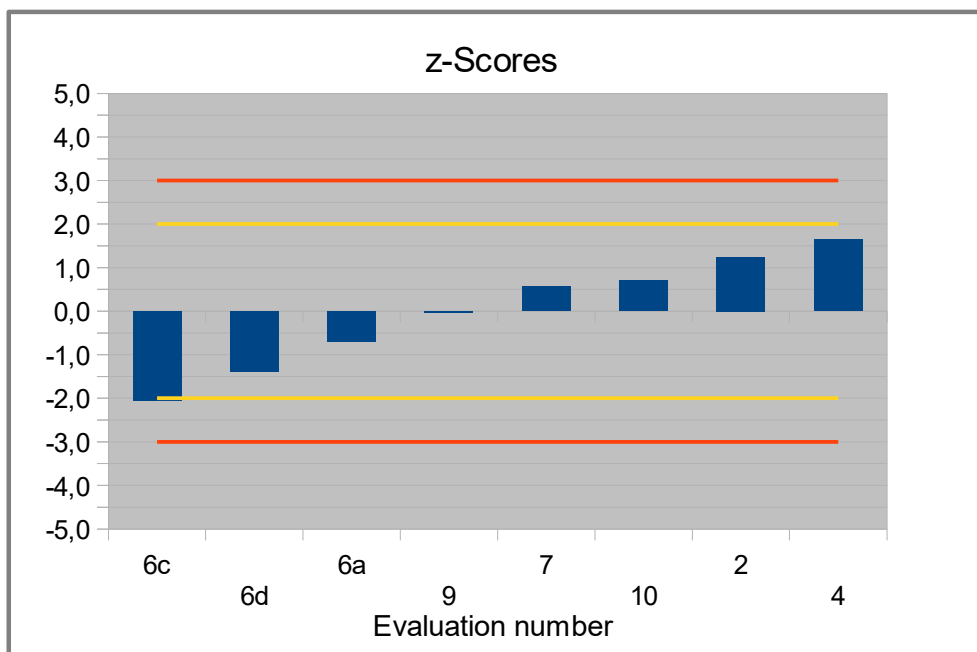


Fig. 26: z-Scores inulin / fructans, sample A

4.6.2 Sample B (Methods: 5 + 6)**Proficiency Test**

Statistic Data	
Number of results	7
Number of outliers	0
Mean	6,30
Median	6,22
Robust Mean (X_{pt})	6,30
Robust standard deviation (S^*)	0,304
Number with 2 replicates	4
Repeatability SD (S_r)	0,261
Repeatability (CV_r)	4,04%
Reproducibility SD (S_R)	0,263
Reproducibility (CV_R)	4,07%
Target range:	
Target standard deviation σ_{pt}	0,467
Target standard deviation (for Information)	0,191
lower limit of target range	5,36
upper limit of target range	7,23
Quotient S^*/σ_{pt}	0,65
Standard uncertainty $U(X_{pt})$	0,143
Results in the target range	7
Percent in the target range	100%

Comments:

The target standard deviation was calculated according to 3.3 evaluation of a precision experiment (ASU §64 L 00.00-94). In addition, the target standard deviation calculated according to the general model of Horwitz was given for information (s. DLA Evaluation Guide 02.01 (2022) 3.2.6.1).

The distribution of results showed a low variability. The quotient S^*/σ_{pt} was below 1,0. The robust standard deviation was in the range of previous PTs (see 3.3). The repeatability standard deviation was in the range of established values for the used determination methods (s. 3.3).

100% of results were in the target range.

Note: For methods 5 and 6, the difference of the robust means for sample A (without addition of inulin) and sample B (with addition of 4,98g/100g inulin) is 4,29g/100g.

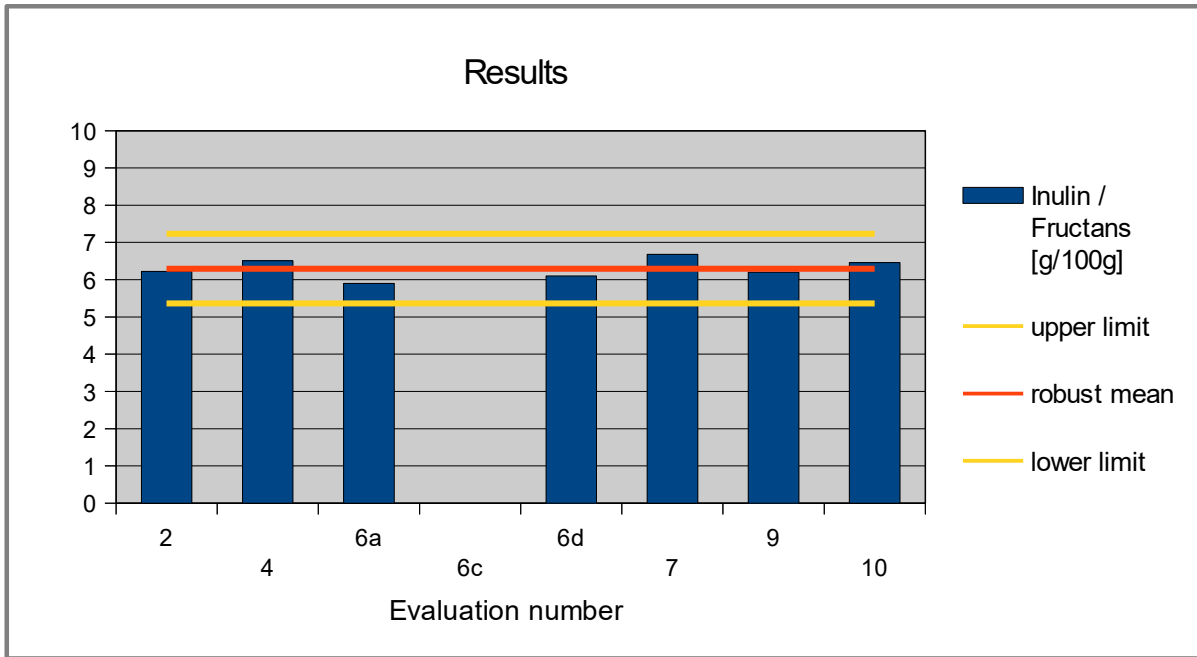


Fig. 27: Results inulin / fructans, sample B

Note:

Due to the low number of < 8 results, a kernel density estimation could not be carried out.

Results of Participants:

Evaluation number	Inulin / Fructans [g/100g]	Deviation [g/100g]	z-Score (opt)	z-Score (Info)	Method	Remark
2	6,22	-0,076	-0,16	-0,40	5	
4	6,51	0,214	0,46	1,1	5	
6a	5,90	-0,396	-0,85	-2,1	5	
6c					6	
6d	6,10	-0,196	-0,42	-1,0	6	
7	6,68	0,384	0,82	2,0	6	
9	6,20	-0,096	-0,20	-0,50	6	
10	6,46	0,164	0,35	0,86	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)

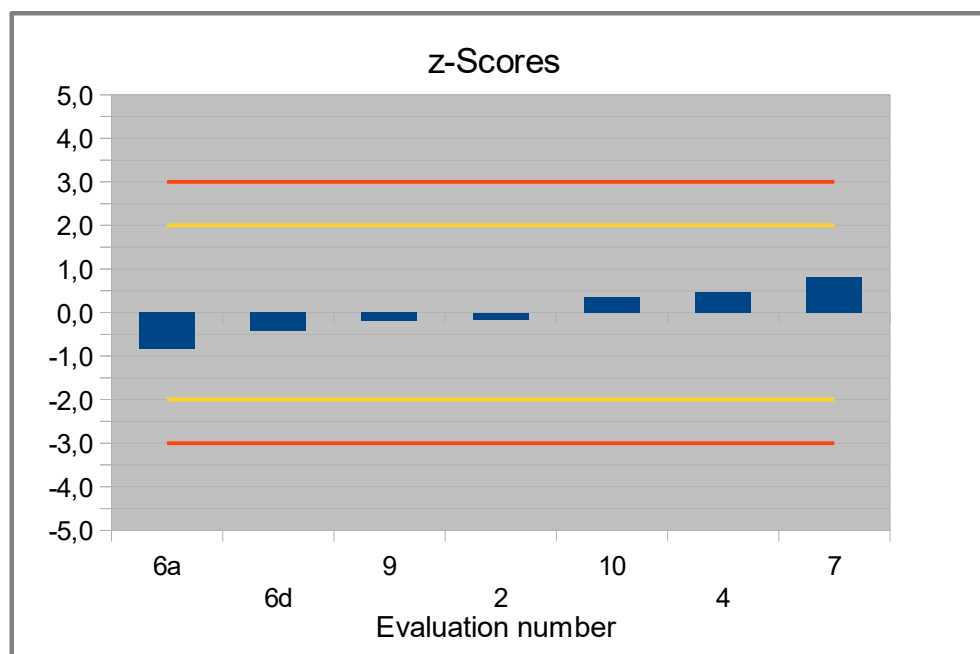


Fig. 28: z-Scores inulin / fructans, sample B

4.7 Participant z-Scores: overview table

Z-Scores for the assigned values from participants' results
(consensus values)

Evaluation number	Total dietary fiber without inulin (1)*		Total dietary fiber incl. Inulin (2+3)*		Insoluble dietary fiber (1a, 2a, 3a)*		Soluble dietary fiber (1b, 2b)*		Inulin / Fructans (5, 6)*	
	Sample A	Sample B	Sample A	Sample B	Sample A	Sample B	Sample A	Sample B	Sample A	Sample B
1	-0,09	-0,25			-0,29	-0,91	0,27	1,4		
2			-0,14	-0,25					1,2	-0,16
3	-0,26	-0,40								
4	-4,7	2,6	-0,32	-0,71	-5,0	2,0	-2,3	-0,35	1,6	0,46
5	0,82	0,80			0,04	-0,17	0,22	1,1		
6 / 6a	0,58	0,07	-0,65	-0,43	0,46	-0,01	-1,0	-0,84	-0,71	-0,85
6b			-0,57	-0,32	3,5	1,5	4,0			
6c			0,61	0,42	0,85	0,45	1,5		-2,0	
6d			1,3	0,42	-0,96	-1,4			-1,4	-0,42
6e					-0,65	-1,1				
7	0,13				-0,54		-0,22		0,57	0,82
8	-0,15	-0,47								
9	0,46	0,59	0,73	1,3					-0,03	-0,20
10	-0,56	-0,99							0,71	0,35
11			-0,57	-0,11						
12	-0,24	-0,41			0,54	-0,05				
13 / 13a	0,15	-0,04	-0,19	0,14	0,54	-0,01	-1,0	-1,3		
13b										

Valuation of z-score (DIN ISO 13528:2009-01):

-2 ≤ z-score ≤ 2 successful (in green)

-2 > z-score > 2 warning signal (in yellow)

-3 > z-score > 3 action signal (in red)

5. Documentation

5.1 Details by the participants

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

Primary data and Analytical Methods

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

Method	Participant	Unit	Date of analysis	Result (Mean)	Result Sample I	Result Sample II	Result (Mean)	Result Sample I	Result Sample II	Limit of determination	Incl. RR	Recovery rate
			Day/Month	Sample A	Sample A	Sample A	Sample B	Sample B	Sample B		yes / no	in %
1	1	g/100g	17.06.2022	10,79	10,65	10,93	10,54	10,46	10,62	0,1	no	
1	3	g/100g	14.06.2022	10,58	10,71	10,45	10,35	10,26	10,43	-	no	-
1	4	g/100g	17/Jun	4,9	5	4,8	14,1	14,3	13,8			
1	5	g/100g	25/May	11,96	11,97	11,95	11,87	11,95	11,79	1	no	-
1	6a	g/100g	01/Jul	11,4			10,6			0,2	no	-
2=1	6a	g/100g	01/Jul	11,9			11,3			0,5	no	-
1	7	g/100g	14.06.2022	11,07	11,01	11,13				0,1 g/100g	no	
1	8	g/100g	17/Jun	10,72	10,61	10,83	10,26	10,17	10,34	0,3	no	
1	9	g/100g	20.05.2022	11,50%			11,60%					
1	10	g/100g	09.06.2022	10,2	10,1	10,4	9,6	9,7	9,5		no	
1	12	g/100g	03/Jun	10,6	10,57	10,63	10,34	10,43	10,26	1	no	
1	13	g/100g	04.07.2022	11,1	11,2	11	10,8	10,8	10,7	0,1	no	

Continuation:

Method	Participant	Method description as in test report / norm / literature	Frit diameter	Pore	Details to Amylase	Details to Protease	Details to Amyloglucosidase	Details to Inulinase	Calibration and reference material	Method accredited ISO/IEC 17025	Further Remarks
									yes / no	yes / no	
1	1	§ 64 LFGB L 00.00-18: 1997-01	Behrotest CFT 30 3 cm	porosity 2	test kit from Megazyme	test kit from Megazyme	test kit from Megazyme			yes	behrotest filter crucible 30 ml
1	3	determination of dietary fiber in food; enzymatic digestion / gravimetry (deviation: filtration with Fibrebags); Total dietary fiber enzyme test kit Bioquant (1.12979.Bioquant	-	-	-	-	-	-	-	yes	Kit: Total dietary fiber enzyme test kit Bioquant (1.12979.Bioquant) ; modification: without frit, filtration with fibrebags
1	4									Yes	
1	5	ASU L 00.00-18	-	-	-	-	-	-	DLA 35-2017	no	
1	6a	AOAC 991.43	Ankom	Ankom	Megazyme E-Blaam	Megazyme E-BSPRT	Megazyme E-AMGDF	no inulinase	Control sample	yes	TDF part 991.43 LOQ is mentioned not LOD
2=1	6a	AOAC 991.43	Ankom	Ankom	Megazyme E-Blaam	Megazyme E-BSPRT	Megazyme E-AMGDF	no inulinase	Control sample	yes	LOQ is mentioned not LOD
1	7	The method allows for the determination of dietary fiber (high molecular weight fraction, TDF), soluble fiber (SDF) and insoluble fiber (IDF) in food for human use using a combination of enzymatic and gravimetric methods. the reference standard is AOAC 991.43 1994 "Determination of total, soluble and insoluble dietary fiber in foods by gravimetry"; which includes degreasing of samples, enzymatic digestion and filtration.			alfa-amylase Megazyme	protease Megazyme	Amyloglucosidase Solution / Megazyme	/		yes	
1	8	inhouse method	40 mm	40-90 µm	Sternzym BA	Sternzym BP	Sternzym GA	-		yes	
1	9										
1	10	AOAC991.43	/	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	12	AOAC 991.43	30mm	P2 (40-60µm)	Novo Nordisk	Sigma	Megazyme	-	-	yes	
1	13	ASU L 00.00-18			Megazyme E-BLAAM	Megazyme E-BSPRT	Megazyme E-AMGDF				

Parameter: Total Dietary Fiber incl. Inulin (Methods: 2, 3)

Method	Participant	Unit	Date of analysis	Result (Mean)	Result Sample I	Result Sample II	Result (Mean)	Result Sample I	Result Sample II	Limit of determination	Incl. RR	Recovery rate
			Day/Month	Sample A	Sample A	Sample A	Sample B	Sample B	Sample B		yes / no	in %
4=2	2	g/100g	01.07.2022	14,82	14,9	14,74	17,31	17,28	17,35	N/A	N/A	N/A
2	6a	g/100g	01/Jul	13.6 (13.4)			16.8 (16.7)			0,3	No	
2	9	g/100g	18.05.2022	16,90%			21,60%					
3	4	g/100g	17/Jun	14,4	12,1	16,7	16	12,2	19,8			
3	6b	g/100g	01/Jul	13.8 (13.5)			17.1 (16.9)			0,5	no	
3	6c	g/100g	01/Jul	16,6			19,2			0,5		
1=3	6d	g/100g	01/Jul	18,2			19,2					
4=3	11	g/100g	10/Jun	13,8	13,8	13,8	17,7	17,5	17,9	0,6	no	
3	13	g/100g	04.07.2022	14,7	15,4	13,9	18,4	17,7	19	0,1	no	

Method	Participant	Method description as in test report / norm / literature	Frit diameter	Pore	Details to Amylase	Details to Protease	Details to Amyloglucosidase	Details to Inulinase	Calibration and reference material	Method accredited ISO/IEC 17025	Further Remarks
									yes / no	yes / no	
4=2	2	Sum of Fibre = AOAC991.43modified+ Fructans	N/A	N/A	Megazyme	Megazyme	Megazyme	Megazyme	in house	Yes	Total amount of fibre is calculated
2	6a	AOAC 2009.01	30 mm	40-90um	Megazyme E-PANAA	Megazyme E-BSPRT	Megazyme E-AMGDF	no inulinase	Control sample	yes	LOQ is given not LOD
2	9										
3	4									No	
3	6b	AOAC 2011.25	30 mm	40-90um	Megazyme E-PANAA	Megazyme E-BSPRT	Megazyme E-AMGDF	no inulinase	Control sample	yes	LOQ is given not LOD
3	6c	AOAC 2022.xx (accepted to be published still)	30 mm	40-90um	Megazyme E-PANAA	Megazyme E-BSPRT	Megazyme E-AMGDF	no inulinase	Control sample	No	
1=3	6d	AOAC 2017.16	30 mm	40-90um	Megazyme E-PANAA	Megazyme E-BSPRT	Megazyme E-AMGDF	no inulinase	Control sample	yes	LOQ is given not LOD - NB METHOD Incl Inulin method 2017.16 !!
4=3	11	AOAC 2011.25								yes	
3	13	AOAC Official Method 2017.16, 2022-02			Megazyme E-PANAA	Romerlabs E-EBSPT-A	Romerlabs E-AMGDF-A	no Inulase			

Parameter: Insoluble Dietary Fiber (Methods: 1a, 2a, 3a)

Method	Participant	Unit	Date of analysis	Result (Mean)	Result Sample I	Result Sample II	Result (Mean)	Result Sample I	Result Sample II	Limit of determination	Incl. RR	Recovery rate
			Day/Month	Sample A	Sample A	Sample A	Sample B	Sample B	Sample B		yes / no	in %
1a	1	g/100g	15.06.2022	8,85	9,08	8,63	8,23	8,19	8,27	0,1	no	
1a	4	g/100g	17/Jun	2,9	3,3	2,5	12	12,4	11,5			
1a	5	g/100g	25/May	9,27	9,32	9,21	9,19	9,23	9,14	1	no	-
2a=1a	6a	g/100g		9,8			9,4			0,3		
1a	6b			13,7			11,3					
1a	7	g/100g	14.06.2022	8,54	8,39	8,69					no	
1a	12	g/100g	03/Jun	9,91	9,84	9,98	9,35	9,37	9,33	1	no	
1a	13a	g/100g	04.07.2022	9,9	9,9	9,8	9,4	9,4	9,3	0,1	no	
2a	6c	g/100g		10,3			10			0,2		
3a	6d	g/100g		8			7,6			0,2		
3a	6e	g/100g		8,4			8			0,2		
3a	13b	g/100g	04.07.2022								no	

Continuation:

Method	Participant	Method description as in test report / norm / literature	Frit diameter	Pore	Details to Amylase	Details to Protease	Details to Amyloglucosidase	Details to Inulinase	Calibration and reference material	Method accredited ISO/IEC 17025	Further Remarks
									yes / no	yes / no	
1a	1	§ 64 LFGB L 00.00-18: 1997-01	Behrotest CFT 30 3 cm	porosity 2	test kit from Megazyme	test kit from Megazyme	test kit from Megazyme			yes	behrotest filter crucible 30 ml
1a	4									Yes	
1a	5	ASU L 00.00-18	-	6 µm	-	-	-	-	DLA 35-2017	no	
2a=1a	6a										
1a	6b	For some unknown reason still we see that native wheat flour "uncooked" gives high values icw other methods.									IDF+SDFP = HMWDF
1a	7									yes	
1a	12	AOAC 991.43	30mm	P2 (40-60µm)	Novo Nordisk	Sigma	Megazyme	-	-	yes	
1a	13a	ASU L 00.00-18			Megazyme E-BLAAM	Megazyme E-BSPRT	Megazyme E-AMGDF				
2a	6c										IDF+SDFP = HMWDF
3a	6d										remark 3 refers to 2017.16 which does not report 4 parameters (TDF +,(HMWDF and LMWDF) but 3 should be 2011.25 or 2022.xx
3a	6e	see same remark as 2017.16 . Method uses same enzymic conditions. This might need further investigation by the experts in AOAC ERP.									remark 3 refers to 2017.16 which does not report 4 parameters (TDF +,(HMWDF and LMWDF) but 3 should be 2011.25 or 2022.xx
3a	13b	AOAC Official Method 2017.16, 2022-02			Megazyme E-PANAA	Romerlabs E-EBSPRT-A	Romerlabs E-AMGDF-A	no Inulase			IDF + HMW SDFS calculated as sum

Parameter: Soluble Dietary Fiber (Methods: 1b, 2b)

Method	Participant	Unit	Date of analysis	Result (Mean)	Result Sample I	Result Sample II	Result (Mean)	Result Sample I	Result Sample II	Limit of determination	Incl. RR	Recovery rate
			Day/Month	Sample A	Sample A	Sample A	Sample B	Sample B	Sample B		yes / no	in %
1b	1	g/100g	16.06.2022	2,72	2,71	2,74	2,82	2,76	2,88	0,1	no	
1b	4	g/100g	17/Jun	1,5	1,5	1,5	2,1	1,9	2,3			
1b	5	g/100g	25/May	2,7	2,65	2,74	2,69	2,72	2,66	1	no	-
2b=1b	6a	g/100g		2,1			1,9			0,2		
1b	6b			4,5			7,9					
1b	7	g/100g	14.06.2022	2,49	2,47	2,5					no	
1b	12	g/100g	03/Jun	<1	<1	<1	<1	1,06	<1	1	no	
1b	13	g/100g	04.07.2022	2,1	2,1	2,1	1,7	1,7	1,8	0,1	no	
2b	6c	g/100g		3.3 (3.1)			6.8* (6.7)			0,1		

Continuation:

Method	Participant	Method description as in test report / norm / literature	Frit diameter	Pore	Details to Amylase	Details to Protease	Details to Amyloglucosidase	Details to Inulinase	Calibration and reference material	Method accredited ISO/IEC 17025	Further Remarks
									yes / no	yes / no	
1b	1	§ 64 LFGB L 00.00-18: 1997-01	Behrotest CFT 30 3 cm	porosity 2	test kit from Megazyme	test kit from Megazyme	test kit from Megazyme			yes	behrotest filter crucible 30 ml
1b	4									Yes	
1b	5	ASU L 00.00-18	-	3 µm	-	-	-	-	DLA 35-2017	no	
2b=1b	6a										The reference below does not ft refers to 2009.01 which is not IDF and SDFS/SDF but determines ({IDF+SDFP} =HMWDF) and LMWDF=SDFS
1b	6b	We participated in MLT 2017.16-2022.xx study new methods where no uncooked wheat flour was included - native regular starch is same as in 2009.01/2011.25 methods									SDFS=LMWDF
1b	7									yes	
1b	12	AOAC 991.43	-	-	-	-	-	-	-	no	Calculated from Total and Insoluble.
1b	13	ASU L 00.00-18			Megazyme E-BLAAM	Megazyme E-BSPRT	Megazyme E-AMGDF				
2b	6c										SDFS=LMWDF - -- In brackets the modified 2011.25 with additional enzyme AMG step as acc. Optimized method acc. McCleary, J AOAC Intern 97 (2014) 896; Brunt & Sanders, Food Chem. 140 (2011) 574.

Parameter (not evaluated methods):

High molecular weight soluble dietary fiber HMW SDF (3b)

Low molecular weight soluble dietary fiber LMW SDF (3c)

Method	Participant	Unit	Date of analysis	Result (Mean)	Result Sample I	Result Sample II	Result (Mean)	Result Sample I	Result Sample II	Limit of determination	Incl. RR	Recovery rate
			Day/Month	Sample A	Sample A	Sample A	Sample B	Sample B	Sample B			
3b	6a	g/100g		2,4			2,3			0,2		
3b	6b	g/100g		4,3			3,6			0,2		
3b	13	g/100g	04.07.2022								no	
3c	6a	g/100g		3.4 (3.1)			7.2 (7.0)			0,3		
3c	6b	g/100g		3,9			7,6			0,3		
3c	13	g/100g	04.07.2022	3,8	4	3,6	8,1	7,7	8,5	0,1	no	

Method	Participant	Method description as in test report / norm / literature	Frit diameter	Pore	Details to Amylase	Details to Protease	Details to Amyloglucosidase	Details to Inulinase	Calibration and reference material	Method accredited ISO/IEC 17025	Further Remarks
									yes / no	yes / no	
3b	6a										
3b	6b										
3b	13	AOAC Official Method 2017.16, 2022-02			Megazyme E-PANAA	Romerlabs E-EBSPRT-A	Romerlabs E-AMGDF-A	no Inulase			IDF + HMW SDFS calculated as sum
3c	6a										In brackets the modified 2011.25 with additional enzyme AMG step as acc. Optimized method acc. McCleary, J AOAC Intern 97 (2014) 896; Brunt & Sanders, Food Chem. 140 (2011) 574.
3c	6b										
3c	13	AOAC Official Method 2017.16, 2022-02			Megazyme E-PANAA	Romerlabs E-EBSPRT-A	Romerlabs E-AMGDF-A	no Inulase			

Parameter: Total Dietary Fiber, other Methods (4)

For information of results originally assigned by the participants to other methods (4) see methods 1 and 2+3.

Parameter: Inulin / Fructans (5, 6)

Method	Participant	Unit	Date of analysis	Result (Mean)	Result Sample I	Result Sample II	Result (Mean)	Result Sample I	Result Sample II	Limit of determination	Incl. RR	Recovery rate
			Day/Month	Sample A	Sample A	Sample A	Sample B	Sample B	Sample B		yes / no	in %
5	2	g/100g	01.07.2022	2,19	2,11	2,27	6,22	6,21	6,23	N/A	No	N/A
5	4	g/100g	06/Jun	2,25	2,24	2,26	6,51	6,87	6,14			
5	6a	g/100g	01/Jul	1,9			5,9			0,2	no	
5	6b	g/100g	see part 1 reporting									
6	6c	g/100g	01/Jul	1,7	1,7	1,7	not done lab moving to new bldg			0,2	no	
6	6d		01/Jul	1,8			6,1			0,3	no	
6	7	g/100g	16.06.2022	2,09	2,11	2,06	6,68	6,62	6,73	0,1 g/100g	no	
6	9	g/100g	17.06.2022	2,00%			6,20%					
6	10	g/100g	09.06.2022	2,11	2,11	2,1	6,46	6,46	6,46	0,1		

Continuation:

Method	Participant	Method description as in test report / norm / literature	Frit diameter	Pore	Details to Amylase	Details to Protease	Details to Amyloglucosidase	Details to Inulinase	Calibration and reference material	Method accredited ISO/IEC 17025	Further Remarks
									yes / no	yes / no	
5	2	AOAC999.03modified	Megazyme	N/A	N/A			Yes		AOAC999.03modified	Megazyme
5	4							No			
5	6a	AOAC 999.03 modified	Megazyme	Thermo PA1	PAD		Control sample	Yes		AOAC 999.03 modified	Megazyme
5	6b										
6	6c	AOAC2016.14	Megazyme	Thermo PA1	PAD		Control sample	No		AOAC2016.14	Megazyme
6	6d	AOAC 997.08 modified	none	Thermo PA1	PAD		Control sample	No	Calculation fructan content based on using fructose content (Fi) only (%=Fi*factor k*g ~ 10/9*0.91=1.01). inulin with avg. DP=10 & ratio fructose/glucose (F/G) 9:1	AOAC 997.08 modified	none
6	7	The sample to be analysed is treated with water, and the extract is hydrolysed with suitable enzymes; the different units obtained are subjected to ion chromatography with pulsed amperometric detector (HPAED/PAD). (like method AOAC 997.08)	inulinase enzyme	CarbopacTM PA 100	Pulsent amperometric detector PAD	Treated with water, and the extract is hydrolysed with suitable enzymes	Calibration: Glucose, fructose, sucrose and arabinose (internal reference material)	yes		The sample to be analysed is treated with water, and the extract is hydrolysed with suitable enzymes; the different units obtained are subjected to ion chromatography with pulsed amperometric detector (HPAED/PAD). (like method AOAC 997.08)	inulinase enzyme
6	9										
6	10	AOAC997.08						no		AOAC997.08	

5.2 Homogeneity

5.2.1 Mixture homogeneity during bottling

Microtracer Homogeneity Test

DLA ptAU01 (2022) Sample A

Weight whole sample	3,10	kg
Microtracer	FSS-red lake	
Particle size	75 – 300	µm
Weight per particle	2,0	µg
Addition of tracer	23,8	mg/kg

Result of analysis

Sample	Weight [g]	Particle number	Particles [mg/kg]
1	5,00	63	25,2
2	5,00	44	17,6
3	5,00	51	20,4
4	5,01	40	16,0
5	5,04	50	19,8
6	4,95	44	17,8
7	4,98	56	22,5
8	5,02	39	15,5

Poisson distribution

Number of samples	8	
Degree of freedom	7	
Mean	48,4	Particles
Standard deviation	8,29	Particles
χ^2 (CHI-Square)	9,94	
Probability	19	%
Recovery rate	81	%

Normal distribution

Number of samples	8	
Mean	19,4	mg/kg
Standard deviation	3,32	mg/kg
rel. Standard deviation	17,1	%
Horwitz standard deviation	10,2	%
HorRat-value	1,7	
Recovery rate	81	%

Microtracer Homogeneity Test

DLA ptAU01 (2022) Sample B

Weight whole sample	3,10	kg
Microtracer	FSS-red lake	
Particle size	75 – 300	µm
Weight per particle	2,0	µg
Addition of tracer	24,5	mg/kg

Result of analysis

Sample	Weight [g]	Particle number	Particles [mg/kg]
1	5,03	71	28,2
2	4,97	58	23,3
3	5,00	52	20,8
4	5,04	66	26,2
5	4,96	55	22,2
6	4,98	63	25,3
7	5,00	65	26,0
8	4,99	59	23,6

Poisson distribution

Number of samples	8	
Degree of freedom	7	
Mean	61,1	Particles
Standard deviation	6,03	Particles
χ^2 (CHI-Square)	4,17	
Probability	76	%
Recovery rate	100	%

Normal distribution

Number of samples	8	
Mean	24,5	mg/kg
Standard deviation	2,41	mg/kg
rel. Standard deviation	9,9	%
Horwitz standard deviation	9,9	%
HorRat-value	1,0	
Recovery rate	100	%

5.3 Information on the Proficiency Test (PT)

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

<i>PT number</i>	DLA ptAU01 - 2022
<i>PT name</i>	Dietary Fiber, soluble / insoluble Fiber and Inulin in Cereal Product
<i>Sample matrix*</i>	<i>Samples A + B: Bread baking mix/ Ingredients: Wholegrain wheat flour 61%, wholegrain rye flour 33%, wholegrain rye sourdough powder 4%, sea salt</i>
<i>Number of samples and sample amount</i>	<i>2 different samples A + B, 40 g each.</i>
<i>Storage</i>	<i>Samples A + B: room temperature (PT period)</i>
<i>Intentional use</i>	<i>Laboratory use only (quality control samples)</i>
<i>Parameter</i>	<i>quantitative: total dietary fiber, soluble / insoluble Fiber and Inulin</i>
<i>Methods of analysis</i>	<i>Analytical methods are optional</i>
<i>Notes to analysis</i>	<i>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.</i>
<i>Result sheet</i>	<i>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.</i>
<i>Units</i>	<i>g/100g</i>
<i>Number of significant digits</i>	<i>at least 2</i>
<i>Further information</i>	<i>For information please specify:</i> <ul style="list-style-type: none"> - <i>Date of analysis</i> - <i>DLA-sample-numbers (for sample I and II)</i> - <i>Limit of detection</i> - <i>Assignment incl. Recovery</i> - <i>Recovery with the same matrix</i> - <i>Method is accredited</i>
<i>Result submission</i>	<i>The result submission file should be sent by e-mail to: pt@dla-lvu.de</i>
<i>Last Deadline</i>	the latest July 08th 2022.
<i>Evaluation report</i>	<i>The evaluation report is expected to be completed 6 weeks after deadline of result submission and sent as PDF file by e-mail.</i>
<i>Coordinator and contact person of PT</i>	<i>Matthias Besler-Scharf PhD</i>

* 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

Participant	Town	Country
		GREAT BRITAIN
		GERMANY
		GERMANY
		ITALY
		GERMANY
		SWEDEN
		NETHERLANDS
		GERMANY
		GERMANY
		CANADA
		GREAT BRITAIN
		BELGIUM
		SPAIN

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

7. Index of references

The list of **references no. 1-21** can be found in the
“**DLA Evaluation Guide 02.01 (2022) General Proficiency Test Schemes**”.

Additional specific references:

- A) ASU §64 LFGB: L 00.00-18: Bestimmung der Ballaststoffe in Lebensmitteln [Determination of dietary fiber in food]
- B) ASU §64 LFGB: L 16.08-1: Bestimmung der Ballaststoffe in Getreidekleie [Determination of dietary fiber in cereal bran]
- C) ASU § 64 LFGB L 17.03-1 Bestimmung der Ballaststoffe in Mischbrot [Determination of dietary fiber in mixed-type bread].
- D) ASU §64 LFGB: L 48.01-25: Bestimmung der Ballaststoffe in Säuglings- und Kindernahrung auf Milchbasis [Determination of dietary fiber in milk-based foods for infants and children]
- E) ASU § 64 LFGB L 00.00-94 Bestimmung von Inulin in Lebensmitteln – Enzymatisches Verfahren [Determination of inulin in food – Enzymatic method]
- F) 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
- G) 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)
- H) 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]
- I) Dietary fibre fractions in cereal foods measured by a new integrated AOAC method, Hollmann et al. (2013), Food Chem 140(3):586-9