Evaluation Report
proficiency test

DLA ptMYS1/2019

Mycotoxin-Screening:

Aflatoxins, Ochratoxin A, Deoxynivalenol, Zearalenone und Fumonisins

in Breakfast Cereals (Muesli)

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General Information on the proficiency test (PT)

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Vertraulichkeit *Confidentiality*	Die Teilnehmerergebnisse sind im EP-Bericht in anonymisierter Form mit Auswertenummern benannt. Daten einzelner Teilnehmer werden ausschließlich nach vorheriger Zustimmung des Teilnehmers an Dritte weitergegeben. Participant result are named anonymously with evaluation numbers in the PT report. Data of individual participants will be passed on to third parties only with prior consent of the participant.

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1. Introduction

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

2. Realisation

2.1 Test material

The test material is a customary breakfast cereal "mueslie" from a European supplier. The basic composition of samples A and B was the same. Additionally further ingredients with different natural levels of mycotoxins were added to sample A and B, respectively (see table 1).

After crushing and sieving (mesh 3,0 mm) of the muesli, the basic mixture was homogenized. Afterwards the samples A and B were produced as follows:

The further ingredients previously crushed and homogenized were added to an aliquot of the matrix for sample A or sample B and the mixture was homogenized. Subsequently, the basic mixture was again added in two steps and homogenized in each case until the total quantity had been reached.

The samples A and B were portioned to approximately 100 g in metallized PET film bags.

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

Table 1: Composition of DLA-Samples

Ingredients	**Sample A ***	**Sample B ***
Muesli with Fruits, organic Ingredients: Oat cereal flakes, raisins oiled, rice puffed, dried fruits (apricots, dates, plums, apples), rice flour, cinnamon Nutrients** per 100 g: Fat 5,0 g, carbohydrates 63 g therof sugar 17 g, fiber 8,8 g, protein 10 g, salt 0,03 g	84 g/100g	91 g/100g
Maize, ground	16 g/100g	-
Almond flour, partially de-oiled	-	5,0 g/100g
Plant powder mixture	-	2,5 g/100g
Pistachio-almond mixture, ground	-	2,0 g/100g

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

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

2.1.1 Homogeneity

The **mixture homogeneity before bottling** was examined 8-fold by **microtracer analysis.** It is a standardized method that is part of the international GMP certification system for feed [14].
Before mixing dye coated iron particles of µm size are added to the sample and the number of particles is determined after homogenization in taken aliquots. The evaluation of the mixture homogeneity is based on the Poisson distribution using the chi-square test. A probability of ≥ 5 % is equivalent to a good homogeneous mixture and of ≥ 25% to an excellent mixture [14, 15].
The microtracer analysis of the present PT samples showed a probability of 99% and 95%. Additionally particle number results were converted into concentrations, statistically evaluated according to normal distribution and compared to the standard deviation according to Horwitz. For the assessment HorRat values between 0,3 and 1,3 are to be accepted under repeat conditions (measurements within the laboratory) [17].
This gave a HorRat value of 0,63 and 0,80 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 all parameters it was in the range of 5% to 18% (see table 2). Thus they were similar to the repeatability standard deviations of the respective official methods (see. 3.6.2) (see Tab. 3) [20-27]. The repeatability standard deviations of the participants' results are given in the documentation in the statistic data (see 4.1 to 4.5).

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

Parameter	CV_r Sample A	CV_r Sample B
Aflatoxin B1 (AF B1)	-	10,6 %
Aflatoxins Sum (AF Sum)	-	11,5 %
Ochratoxin A (OTA)	-	18,4 %
Deoxynivalenol (DON)	5,2 %	-
Fumonisins Sum (FUMO Sum)	10,8 %	-
Zearalenone (ZON)	8,3 %	-

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. 3.2.8 and 3.2.11) [3].

2.1.2 Stability

A water activity (a_W) of < 0,5 is an important factor to ensure the stability of dry or dried products during storage. Optimum conditions for storage is the a_W value range of 0,15 - 0,3. In this range the lowest possible degradation rate is to be expected [16].

The experience with various DLA test materials showed good storage stability with respect to the durability of the sample (spoilage) and the content of the PT parameters for comparable food matrices and water activity (a_W value <0,5).
The a_W value of the EP samples was approx. 0,50 and 0,48 (20-21°C) The stability of the sample material was thus ensured during the investigation period under the specified storage conditions.

2.2 Sample shipment and information to the test

The portions of test materials sample A, and B were sent to every participating laboratory in the 17th week of 2019. The testing method was optional. The tests should be finished at 7h June 2019 the latest.

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

*There are **two different samples A and B** possibly containing the parameters Aflatoxins, Ochratoxin A, Deoxynivalenol, Zearalenon and Fumonisins in the range of µg/kg in the **matrix** of **cereal muesli with fruits**. The samples contain different ingredients with natural contents of the above mentioned mycotoxins.*

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

2.3 Submission of results

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

15 out of 16 participants submitted their results in time. One participant submitted the results with delay.

3. Evaluation

3.1 Qualitative consensus and valuation of results

The qualitative evaluation of the results of each participant was based on the agreement of the results classified as "negative" or "positive" with the **consensus values from participants.** A consensus value is determined unless ≥ 75% positive or negative results are present for a parameter.
The assessment will be in the form that the number of matching results followed by the number of samples for which a consensus value was obtained is indicated. Behind that the agreement is expressed as the percentage in parentheses.

For the **qualitative classification** of the participant results as "negative" or "positive" DLA derived acceptance levels in accordance with EU Regulation 401/2006 Annex II 4.4.1 (see this report 3.2.6.3 and Table 4). Under the EU Regulation, measurement results from mycotoxin screening methods that have levels less than 50% of the maximum permitted levels may be considered "compliant". Accordingly, "compliant" measurement results of <50% of the maximum level according to EU-VO 1881/2006 are classified as "negative" and measurement results >50% of the maximum level are classified as "positive" for the qualitative evaluation of the participant results in the present report.

3.2 Quantitative evaluation

3.2.1 Consensus value from participants (assigned value)

The **robust mean** of the submitted results was used as assigned value (X_{pt}) („consensus value from participants") providing a normal distribution. The calculation was done according to algorithm A as described in annex C of ISO 13528 [3]. If there are < 12 quantitative results and an increased difference between robust mean and median, the **median** may be used as the assigned value (criterion: Δ median - rob. mean > 0,3 σ_{pt}) [3].
The condition is that the majority of the participants' results show a normal distribution or are distributed unimodal and symmetrically. To this end, an examination of the distribution is carried out, inter alia, using the kernel density estimate [3, 12].
In case there are indications for sources of higher variability such as a bimodal distribution of results, a cause analysis is performed. Frequently different analytical methods may cause an anomaly in results' distribution. If this is the case, separate evaluations with own assigned values (X_{pti}) are made whenever possible.

In the present PT this was done, if possible, always for the results of all methods together (ELISA, HPLC, LC-MS) and separately for ELISA methods and LC methods (HPLC, LC-MS):

i) ***Assigned value of all methods*** - $X_{pt_{ALL}}$
ii) ***Assigned value of ELISA methods*** - $X_{pt_{ELISA}}$
iii) ***Assigned value of LC methods*** - $X_{pt_{LC}}$

Single results giving values outside the measuring range of the participating laboratory or given as „0" are not considered for statistical evaluation (e.g. results given as > 25 mg/kg and < 2,5 mg/kg,

respectively) [3].

3.2.2 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].

The following robust standard deviations were considered:

i) ***Robust standard deviation of all methods - S^*_{ALL}***
ii) ***Robust standard deviation of ELISA methods - S^*_{ELISA}***
iii) ***Robust standard deviation of LC methods - S^*_{LC}***

3.2.3 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.

3.2.4 Reproducibility standard deviation

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

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

The relative reproducibility standard deviation as a percentage of the mean value is given as the coefficient of variation CV_R in the statistic-

al characteristics in the results section, provided that the individual results of the participants are available, and the meaning is explained in more detail under 3.9.

3.2.5 Exclusion of results and outliers

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

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

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

3.2.6 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.

In the present PT the target standard deviation from the general model of* Horwitz / Thompson*, suitable for levels ≤ 120 µg/kg, was applied for the following parameters (s. 3.2.6.1):

- Aflatoxins, Ochratoxin A and Zearalenone.

For information the target standard deviation derived from a precision experiment was given additionally for the parameters Aflatoxins, Ochratoxin A and Zearalenone (s. 3.2.6.2).

In the present PT the target standard deviation derived from a precision experiment was applied for the following parameters (s. 3.2.6.2):

- Deoxynivalenol and Fumonisins.

For the parameter sum of fumonisins the standard uncertainty was considered by valuating with z'-scores (see 3.2.6.8).

For information the target standard deviation from the general model of* Horwitz*, suitable for levels ≥ 120 µg/kg, was given additionally for the parameters Aflatoxins, Ochratoxin A and Zearalenone (s. 3.2.6.2).

3.2.6.1 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)

3.2.6.2 Value by precision experiment

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

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

The relative repeatability standard deviations (RSD_r) and relative reproducibility standard deviations (RSD_R) given in table 3 were obtained in precision experiments by the indicated methods.
The resulting target standard deviations σ_{pt}, which were identified there, were used to evaluate the results and to provide additional information for the statistical data.

Table 3: Relative repeatability standard deviations (RSD_r) and relative reproducibility standard deviation (RSD_R) according to selected evaluations of tests for precision and the resulting target standard deviation σ_{pt} [20-27]

Parameter	**Matrix**	**Mean** [µg/kg]	**RSD_r**	**RSD_R**	**σ_{pt}**	**Method / Literature**
AF B1	Maize	14,9	5,8%	10%	9,12%[2]	ASU §64 L 15.00-2[20]
AF B1	Peanut paste	5,26	14,9%	30%	28,1%[2]	ASU §64 L 15.00-2[20]
AF B1	Peanut paste	0,80	6%	32%	31,7%	ASU §64 L 23.05-2[21]
AF Summe	Maize	24,5	7,3%	11,7%	10,5%[3]	ASU §64 L 15.00-2[20]
AF Summe	Peanut paste	8,42	17%	30%	27,5%[3]	ASU §64 L 15.00-2[20]
AF Summe	Peanut paste	1,3	6%	34%	33,7%	ASU §64 L 23.05-2[21]
OTA	Maize	16,3	20,1%	28,4%	24,6%[1]	ASU §64 L 15.00-1/2[22]
OTA	Barley	14,4	7,9%	26,5%	25,9%	ASU §64 L 15.00-1/2[22]
OTA	Sultanas	11,4	5,6%	14,3%	13,7%	ASU §64 L 30.00-5[23]
DON	Rice	458	6,5%	11,5%	11,5%	ASU §64 L 15.00-9[24]
DON	Wheat	678	6,0%	16,3%	15,7%	ASU §64 L 15.00-9[24]
DON	Wheat	165	21%	39%	36,1%	ASU §64 L 15.00-9[24]
DON	Maize	501	10%	23%	21,9%[1]	ASU §64 L 15.00-9[24]
FUMO Sum	Baby food	111,6	16,3%	26,6%	24,0%	ASU §64 L 48.02-5[25]
FUMO Sum	Baby food	293,4	6,9%	16,6%	15,9%	ASU §64 L 48.02-5[25]
FUMO Sum	Baby food	211,2	22,9%	26,6%	21,1%	ASU §64 L 48.02-5[25]
FUMO Sum	Baby food	322,5	14,0%	24,1%	22,0%[1]	ASU §64 L 48.02-5[25]
ZON	Maize	87,2	14,2%	20,6%	10,5%	ASU §64 L 48.02-3[26]
ZON	Maize	66,5	8,9%	16,4%	15,1%	ASU §64 L 48.02-3[26]
ZON	Wheat	26,3	8,9%	19,7%	18,7%	ASU §64 L 15.01/02-2 [27]
ZON	Wheat	58,3	3,8%	23,0%	22,8%[1]	ASU §64 L 15.01/02-2 [27]

[1] *in the evaluation (s. section 4) used values*
[2] *Mean applied = resulting target standard deviation σ_{pt} 18,6%*
[3] *Mean applied = resulting target standard deviation σ_{pt} 19,0%*

3.2.6.3 Value by perception

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

In the present PT, the target standard deviations according to 3.2.6.1 and 3.2.6.2 were considered suitable, respectively.

Legal requirements and acceptance levels for the qualitative assessment:

The maximum levels for mycotoxins in food stuffs are set out in EU Regulation 1881/2006 [19]. Table 4 shows the maximum levels for the parameters of the present screening PT in certain foods. The DLA-derived acceptance levels (50% of the target screening concentration according to EU Regulation 401/2006 Annex II 4.4.1) are also given in table 4 and were used for the qualitative assessment of the results (see 3.1 Qualitative consensus and valuation of results).

Note: The acceptance levels derived by DLA are not legally binding values. They were chosen for their suitability for the qualitative assessment of the PT samples. The actual food matrix of the PT samples may differ from the foodstuffs group specified in the EU Regulation.
For the qualitative assessment of fumonisins B1 and B2, 75% and 25% of the acceptance level for the sum of fumonisins were used, respectively.

Table 4: Maximum levels for mycotoxins in certain foods according to EU Regulation 1881/2006 and derived acceptance levels for the qualitative evaluation of the results in the present screening-PT based on EU Regulation 401/2006 [18, 19]

Mykotoxins	Foodstuffs	Maximum Levels	Acceptance Levels
		[µg/kg]	[µg/kg]
AF B1	All cereals and all products derived from cereals, including processed cereal products	2,0	1,0 [1]
AF B1	Almonds, pistachios and apricot kernels, intended for direct human consumption or use as an ingredient in foodstuffs	8,0	4,0
AF B1	Dried fruit, other than dried figs, and processed products thereof, intended for direct human consumption or use as an ingredient in foodstuffs	2,0	1,0
AF Sum	All cereals and all products derived from cereals, including processed cereal products	4,0	2,0 [1]
AF Sum	Almonds, pistachios and apricot kernels, intended for direct human consumption or use as an ingredient in foodstuffs	10,0	5,0
AF Sum	Dried fruit, other than dried figs, and processed products thereof, intended for direct human consumption or use as an ingredient in foodstuffs	4,0	2,0
OTA	All products derived from unprocessed cereals, including processed cereal products and cereals intended for direct human consumption	3,0	1,5 [1]
OTA	Dried vine fruit (currants, raisins and sultanas)	10,0	5,0
DON	Bread (including small bakery wares), pastries, biscuits, cereal snacks and breakfast cereals	500	250 [1]
FUMO Sum	Maize intended for direct human consumption, maize-based foods for direct human consumption	1000	500
FUMO Sum	Maize-based breakfast cereals and maize-based snacks	800	400
FUMO Sum	Processed maize-based foods and baby foods for infants and young children	200	100 [1]
ZON	Cereals intended for direct human consumption, cereal flour, bran and germ as end product marketed for direct human consumption	75	37,5
ZON	Maize intended for direct human consumption, maize-based snacks and maize-based breakfast cereals	100	50 [1]

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

(Maximum levels according to EU/1881/2006 (Annex) and acceptance levels based on EU/401/2006 (Annex II 4.4.1) for levels >50% below the maximum level)

3.2.7 z-Score

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

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

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

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

The z-score valid for the proficiency test is called z-score (σ_{pt}) in the evaluation, while the value called z-score (info) is purely informative. The two z scores are calculated with the different target standard deviations according to 3.2.6.

3.2.7.1 Warning and action signals

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

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

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

3.2.8 z'-Score

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

The calculation is performed by:

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

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

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

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

For warning and action signals see 3.2.7.1.

3.2.9 Reproducibility coefficient of variation (CV)

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

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

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

3.2.10 Quotient S*/σ_{pt}

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

3.2.11 Standard uncertainty and traceability

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

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

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

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

4. Results

All following tables are anonymized. With the delivering of the evaluation report the participants are informed about their individual evaluation number.
The results were grouped according to the applied methods (ELISA, HPLC, LC/MS) and sorted chronologically according to the evaluation number of the participants. First, the qualitative assessment of the results is shown followed by the quantitative evaluation. If at least 50% positive results and at least 5 quantitative results are available, the following statistical characteristics of the respective PT are listed:

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

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

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

Evaluation number	Result	Deviati-on	z-Score Xpt_{ALL}	Deviati-on	z-Score Xpt_{Mi}	Method	Remarks
	[µg/kg]	X All		X Mi			

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

4.1 Proficiency Test Aflatoxins

4.1.1 Results: Aflatoxin B1 (AF B1)

Qualitative valuation of results: Samples A and B

Evaluation number	Sample A	Sample A	Sample B	Sample B	Qualitative Valuation	Method	Remarks
	pos/neg	[µg/kg]	pos/neg	[µg/kg]	Agreement with consensus value		
5		0,80		4,08		ELISA	not rated (sum of aflatoxins?)
16		0,15		3,10		ELISA	not rated (sum of aflatoxins?)
2	negative	<0,1	positive	2,10	2/2 (100%)	HPLC	
4	negative	<0,12	positive	5,08	2/2 (100%)	HPLC	
6	negative	< 0,20	positive	6,60	2/2 (100%)	HPLC	
7	negative	<0,01	positive	3,38	2/2 (100%)	HPLC	
10	negative	<0,5	positive	5,10	2/2 (100%)	LC-MS	
13	negative	0 (<0,1)	positive	4,60	2/2 (100%)	LC-MS	
14	negative	<0.5	positive	5,30	2/2 (100%)	LC-MS	

	Sample A		Sample B	
Number positive	0		7	
Number negative	7		0	
Percent positive	0		100	
Percent negative	100		0	
Consensus value	negative		positive	

Methods:
further details see documentation

positve: > 1,0 µg/kg (EU maximum level x 0,5)
negative: < 1,0 µg/kg (EU maximum level x 0,5)

Comments:
The acceptance level for the classification of the results as positive or negative was set at 1.0 µg/kg (see 3.1 and Tab.4)
For sample A, all results were below and for sample B all results above the acceptance level.

Quantative valuation: Aflatoxin B1 in µg/kg

Sample B

Statistic Data	**LC-Methods**
Number of results	7
Number of outliers	0
Mean	4,59
Median	5,08
Robust Mean (X)	**4,60**
Robust standard deviation (S*)	**1,63**
Number with 2 replicates	4
Repeatability SD (S_r)	0,420
Repeatability (CV_r)	10,6%
Reproducibility SD (S_R)	1,52
Reproducibility (CV_R)	38,5%
Target range:	
Target standard deviation σ_{pt}	**1,01**
Target standard deviation (for Information)	0,856
lower limit of target range	**2,58**
upper limit of target range	**6,63**
Quotient S/σ_{pt}*	*1,61*
Standard uncertainty $U_{(Xpt)}$	*0,772*
Results in the target range	*6*
Percent in the target range	*86%*

Comments to the statistical characteristics:

The target standard deviation was calculated according to the general model of Horwitz/Thompson (3.2.6.1). For information the target standard deviation using data from a precision experiment was given (s. 3.2.6.2).

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

The repeatability and reproducibility standard deviation are in the range of established values of the applied methods (see 3.2.6.2).

86% of the results were in the target range.

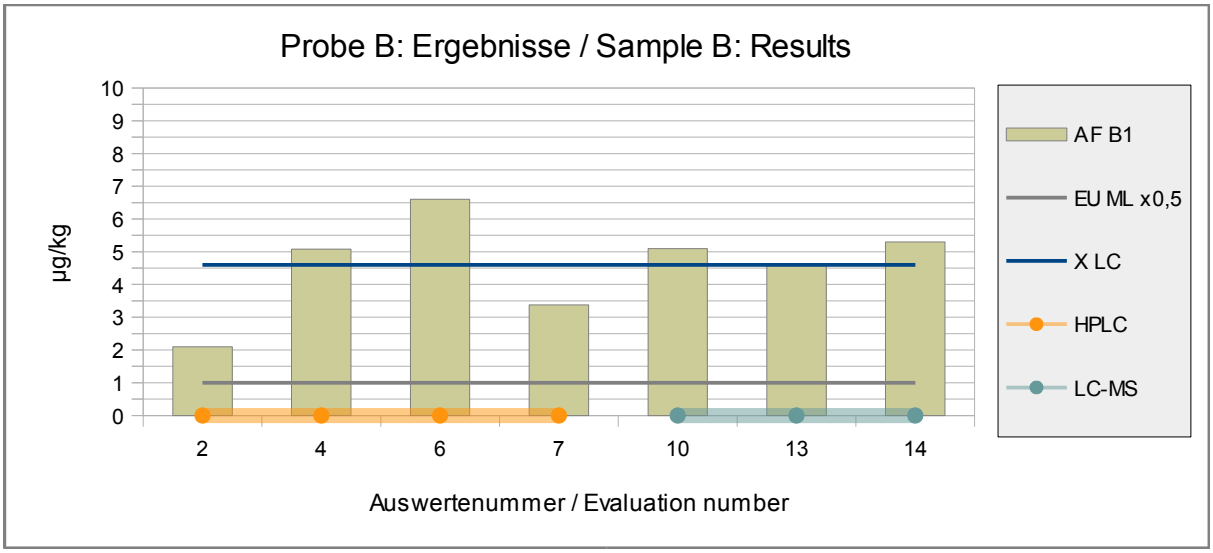

Abb./Fig. 1: Results Aflatoxin B1 (AF B1)
blue line = Assigned value robust mean results LC methods
grey line = Qual. valuation as positive > 1,0 µg/kg
round symbols = Applied methods (see legend)

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

z-Scores der Ergebnisse: Aflatoxin B1
z-Scores of Results: Aflatoxin B1

Evaluation number	Sample B	Devia-tion	z-Score Xpt_{LC}	Method	Remarks
	[µg/kg]	X LC			
2	2,10	-2,50	-2,5	HPLC	
4	5,08	0,48	0,47	HPLC	
6	6,60	2,00	2,0	HPLC	
7	3,38	-1,22	-1,2	HPLC	
10	5,10	0,50	0,49	LC-MS	
13	4,60	0,00	0,00	LC-MS	
14	5,30	0,70	0,69	LC-MS	

Methods:
further details see documentation

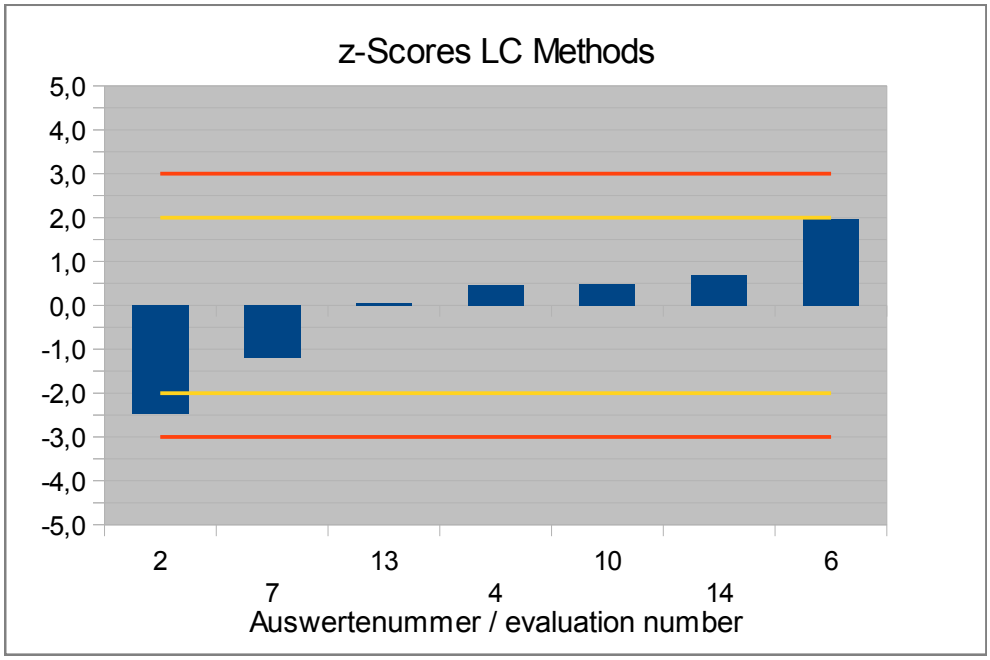

Abb./Fig. 2:
z-Scores Aflatoxin B1 (AF B1)
Assigned value robust mean results LC methods

4.1.2 Results: Aflatoxins Sum (AF Sum)

Qualitative valuation of results: Samples A and B

Evaluation number	Sample A	Sample A	Sample B	Sample B	Qualitative Valuation	Method	Remarks
	pos/neg	[µg/kg]	pos/neg	[µg/kg]	Agreement with consensus value		
1	negative	<BG (1,75)	positive	4,50	2/2 (100%)	ELISA	
3	negative	0 (<1)	positive	2,63	2/2 (100%)	ELISA	
5	negative	0,80	positive	4,08	2/2 (100%)	ELISA	
12	negative	< 1	positive	2,30	2/2 (100%)	ELISA	
15	negative	0,25	positive	3,80	2/2 (100%)	ELISA	Mean calculated by DLA
16	negative	0,15	positive	3,10	2/2 (100%)	ELISA	Mean calculated by DLA
2	negative	<0,3	positive	2,21	2/2 (100%)	HPLC	
4	negative	0 (<0,48)	positive	5,76	2/2 (100%)	HPLC	
6	negative	< 0,80	positive	7,20	2/2 (100%)	HPLC	
7	negative	<0,04	positive	4,02	2/2 (100%)	HPLC	Sum calculated by DLA
10	negative	<2	positive	5,10	2/2 (100%)	LC-MS	
13	negative	0 (<0,4)	positive	5,00	2/2 (100%)	LC-MS	
14	negative	<0,5	positive	5,30	2/2 (100%)	LC-MS	

	Sample A		Sample B	
Number positive	0		13	
Number negative	13		0	
Percent positive	0		100	
Percent negative	100		0	
Consensus value	negative		positive	

Methods:
further details see documentation

positive: > 2,0 µg/kg (EU maximum level x 0,5)
negative: < 2,0 µg/kg (EU maximum level x 0,5)

Comments:
The acceptance level for the classification of the results as positive or negative was set at 2.0 µg/kg (see 3.1 and Tab.4)
For sample A, all results were below and for sample B all results above the acceptance level.

Quantative valuation: Aflatoxins Sum in µg/kg

Sample B

Statistic Data	All Methods	ELISA Methods	LC Methods
Number of results	13	6	7
Number of outliers	0	0	0
Mean	4,23	3,40	4,94
Median	4,08	3,45	5,10
Robust Mean (X_{pt})	**4,17**	**3,40**	**4,99**
Robust standard deviation (S*)	**1,53**	**0,980**	**1,65**
Number with 2 replicates	10	6	4
Repeatability SD (S_r)	0,425	0,427	0,423
Repeatability (CV_r)	11,5%	12,6%	10,2%
Reproducibility SD (S_R)	1,14	0,921	1,42
Reproducibility (CV_R)	30,9%	27,1%	34,4%
Target range:			
Target standard deviation σ_{pt}	**0,918**	**0,748**	**1,10**
Target standard deviation (for Information)	0,793	0,646	0,947
lower limit of target range	**2,34**	**1,90**	**2,79**
upper limit of target range	**6,01**	**4,90**	**7,18**
Quotient S/σ_{pt}*	*1,7*	*1,3*	*1,5*
Standard uncertainty $U_{(Xpt)}$	*0,530*	*0,500*	*0,778*
Results in the target range	*11*	*6*	*6*
Percent in the target range	*85%*	*100%*	*86%*

Comments to the statistical characteristics:

The target standard deviation was calculated according to the general model of Horwitz/Thompson (3.2.6.1). For information the target standard deviation using data from a precision experiment was given (s. 3.2.6.2).

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

The repeatability and reproducibility standard deviation are in the range of established values of the applied methods (see 3.2.6.2).

85% of the results of all methods were in the target range.

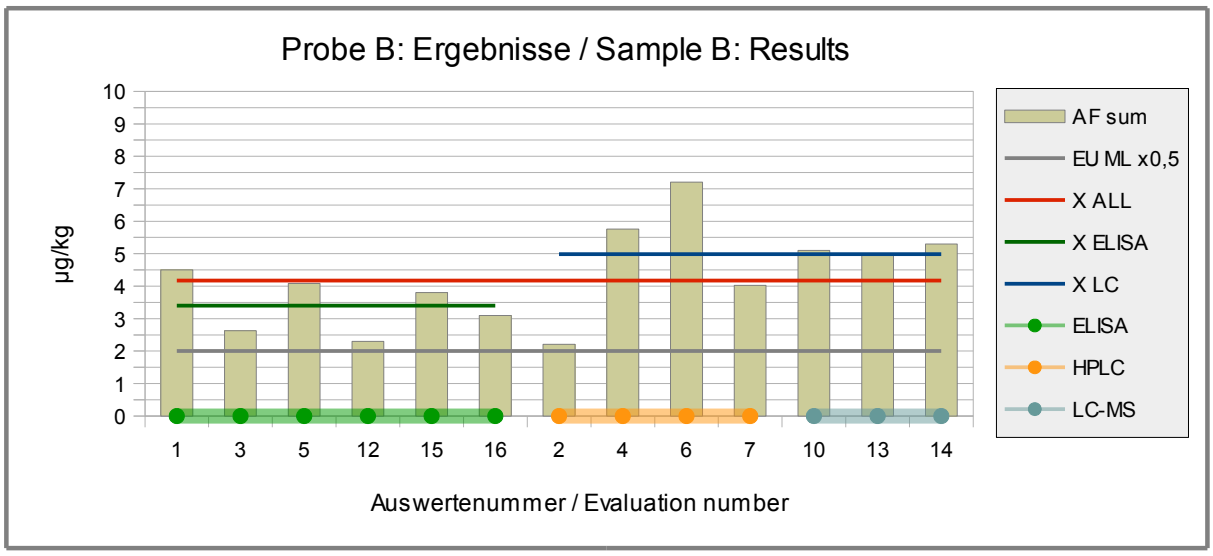

Abb./Fig. 3: Results Aflatoxins Sum (AF Sum)
red line = Assigned value robust mean results all methods
green line = Assigned value robust mean results ELSIA methods
blue line = Assigned value robust mean results LC methods
grey line = Qual. valuation as positive > 2,0 µg/kg
round symbols = Applied methods (see legend)

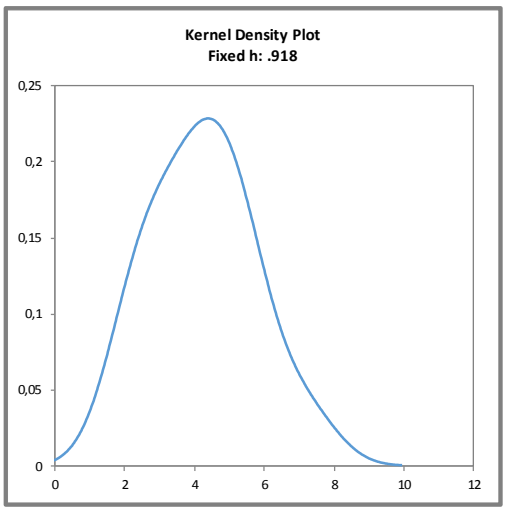

Abb. / Fig. 4:
Kerndichte-Schätzung aller Ergebnisse
(mit h = 0,75 x σpt von Xpt_{ALL})

Kernel density plot of all results
(with h = 0,75 x σpt of Xpt_{ALL})

Comments:
The kernel density estimation shows nearly a symmetrical distribution of results with a small shoulder at approx. 2 - 3,5 µg/kg.

z-Scores der Ergebnisse: Aflatoxine Summe
z-Scores of Results: Aflatoxins Sum

Evaluation number	Sample B	Deviation	z-Score Xpt_{ALL}	Deviation	z-Score Xpt_{ELISA}	Deviation	z-Score Xpt_{LC}	Method	Remarks
	[µg/kg]	X All		X ELISA		X LC			
1	4,50	0,33	0,36	1,10	1,5			ELISA	
3	2,63	-1,55	-1,7	-0,78	-1,0			ELISA	
5	4,08	-0,09	-0,10	0,68	0,91			ELISA	
12	2,30	-1,87	-2,0	-1,10	-1,5			ELISA	
15	3,80	-0,37	-0,41	0,40	0,53			ELISA	Mean calculated by DLA
16	3,10	-1,07	-1,2	-0,30	-0,40			ELISA	Mean calculated by DLA
2	2,21	-1,96	-2,1			-2,78	-2,5	HPLC	
4	5,76	1,59	1,7			0,78	0,71	HPLC	
6	7,20	3,03	3,3			2,22	2,0	HPLC	
7	4,02	-0,15	-0,17			-0,97	-0,88	HPLC	Sum calculated by DLA
10	5,10	0,93	1,0			0,12	0,10	LC-MS	
13	5,00	0,83	0,90			0,01	0,01	LC-MS	
14	5,30	1,13	1,2			0,32	0,29	LC-MS	

Methods:
further details see documentation

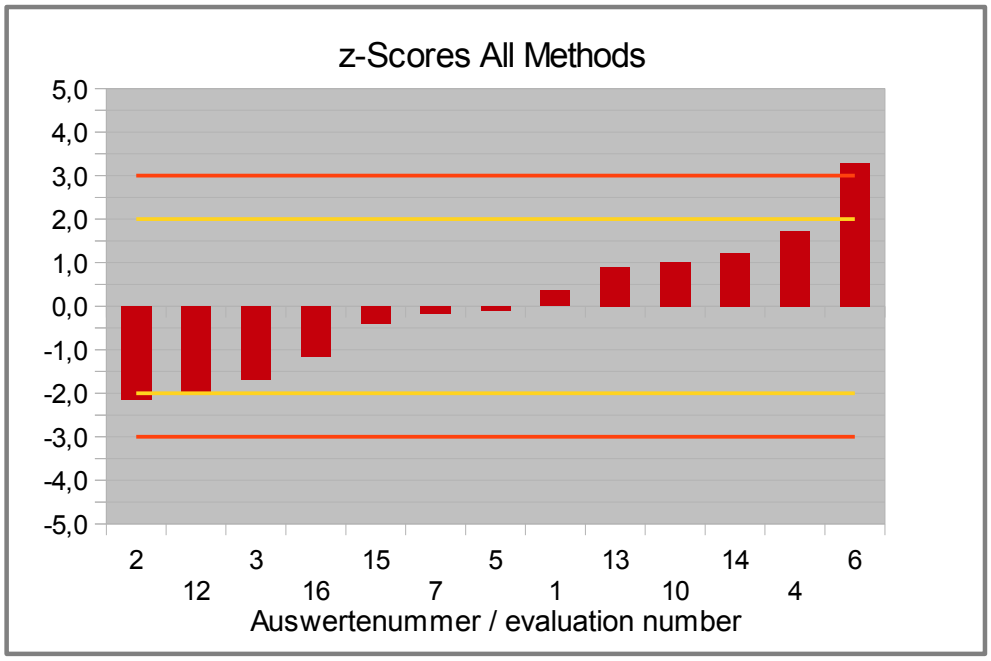

Abb./Fig. 5:
z-Scores Aflatoxins Sum (AF Sum)
Assigned value robust mean results all methods

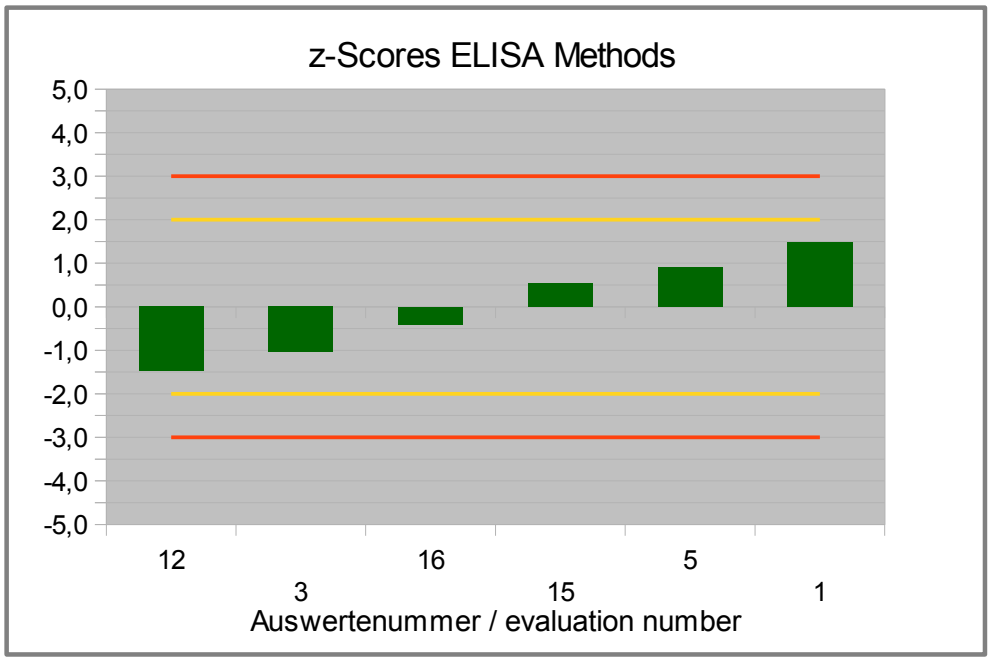

Abb./Fig. 6:
z-Scores Aflatoxins Sum (AF Sum)
Assigned value robust mean results ELISA methods

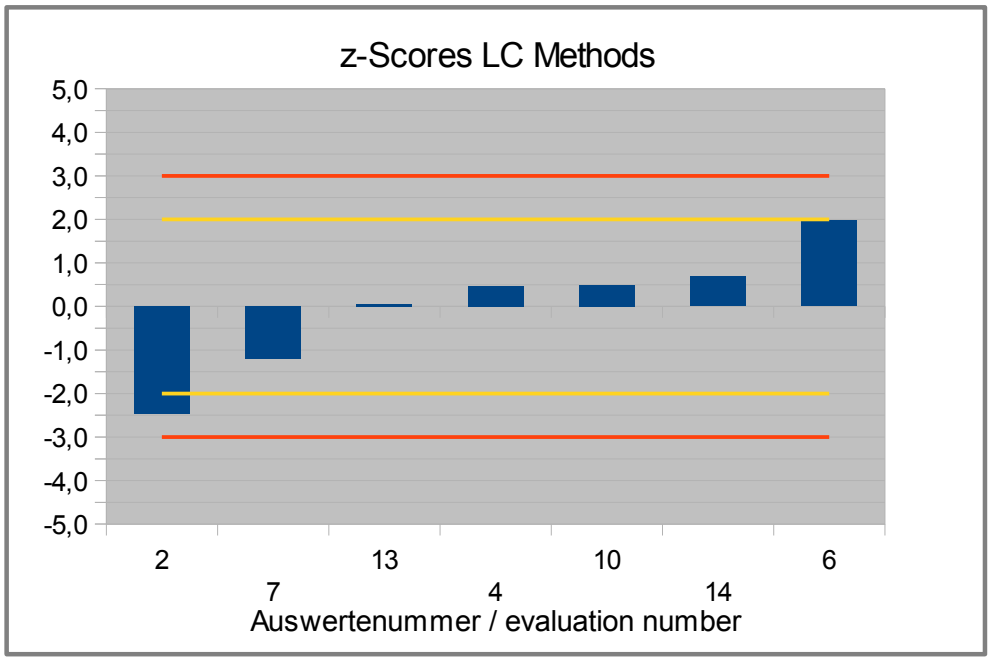

Abb./Fig. 7:
z-Scores Aflatoxins Sum (AF Sum)
Assigned value robust mean results LC methods

4.2 Proficiency Test Ochratoxin A

4.2.1 Results: Ochratoxin A (OTA)

Qualitative valuation of results: Samples A and B

Evaluation number	Sample A	Sample A	Sample B	Sample B	Qualitative Valuation	Method	Remarks
	pos/neg	[µg/kg]	pos/neg	[µg/kg]	Agreement with consensus value		
1	negative	1,1	positive	8,80	1/1 (100%)	ELISA	
3	positive	7,98	positive	8,80	1/1 (100%)	ELISA	
5	negative	<1,5	positive	7,11	1/1 (100%)	ELISA	
7	negative	<1,25	positive	4,05	1/1 (100%)	ELISA	
12	positive	4,8	positive	18,8	1/1 (100%)	ELISA	
15	positive	1,7	positive	7,95	1/1 (100%)	ELISA	Mean calculated by DLA
16	positive	3,15	positive	6,25	1/1 (100%)	ELISA	Mean calculated by DLA
2	negative	<0,3	positive	4,99	1/1 (100%)	HPLC	
4	negative	0,89	positive	7,06	1/1 (100%)	HPLC	
6	negative	< 0,50	positive	5,70	1/1 (100%)	HPLC	
10	negative	<1	positive	9,10	1/1 (100%)	LC-MS	
13	negative	0 (<0,4)	positive	8,10	1/1 (100%)	LC-MS	
14	negative	<0.5	positive	5,50	1/1 (100%)	LC-MS	

	Sample A		Sample B	
Number positive	4		13	
Number negative	9		0	
Percent positive	31		100	
Percent negative	69		0	
Consensus value	none		positive	

Methods:
further details see documentation

positve: > 1,5 µg/kg (EU maximum level x 0,5)
negative: < 1,5 µg/kg (EU maximum level x 0,5)

Comments:
The acceptance level for the classification of the results as positive or negative was set at 1.5 µg/kg (see 3.1 and Table 4).
For sample B, all results were above the acceptance level, while for sample A, no consensus value of ≥ 75% negative or positive results was obtained.

Quantative valuation: Ochratoxin A in µg/kg

Sample B

Statistic Data	All Methods	ELISA Methods	LC Methods
Number of results	12	6°	6
Number of outliers	0	1	0
Mean	6,95	7,16	6,74
Median	7,09	7,53	6,38
Robust Mean (X_{pt})	**6,96**	**7,17**	**6,74**
Robust standard deviation (S*)	**1,86**	**2,03**	**1,84**
Number with 2 replicates	9	6	3
Repeatability SD (S_r)	1,27	1,19	1,43
Repeatability (CV_r)	18,4%	16,6%	22,0%
Reproducibility SD (S_R)	2,05	1,99	2,44
Reproducibility (CV_R)	29,6%	27,9%	37,6%
Target range:			
Target standard deviation σ_{pt}	**1,53**	**1,58**	**1,48**
Target standard deviation (for Information)	1,71	1,76	1,66
lower limit of target range	**3,90**	**4,02**	**3,77**
upper limit of target range	**10,0**	**10,3**	**9,71**
Quotient S/σ_{pt}*	*1,2*	*1,3*	*1,2*
Standard uncertainty $U_{(Xpt)}$	*0,671*	*1,04*	*0,941*
Results in the target range	*12*	*6*	*6*
Percent in the target range	*100%*	*100%*	*100%*

° *without outliers (result no. 12)*

Comments to the statistical characteristics:

The target standard deviation was calculated according to the general model of Horwitz/Thompson (3.2.6.1). For information the target standard deviation using data from a precision experiment was given (s. 3.2.6.2).

The distribution of results showed a normal variability. The quotients S*/σ_{pt} were below 2,0 each.

The repeatability and reproducibility standard deviation are in the range of established values of the applied methods (see 3.2.6.2).

100% of the results of all methods were in the target range.

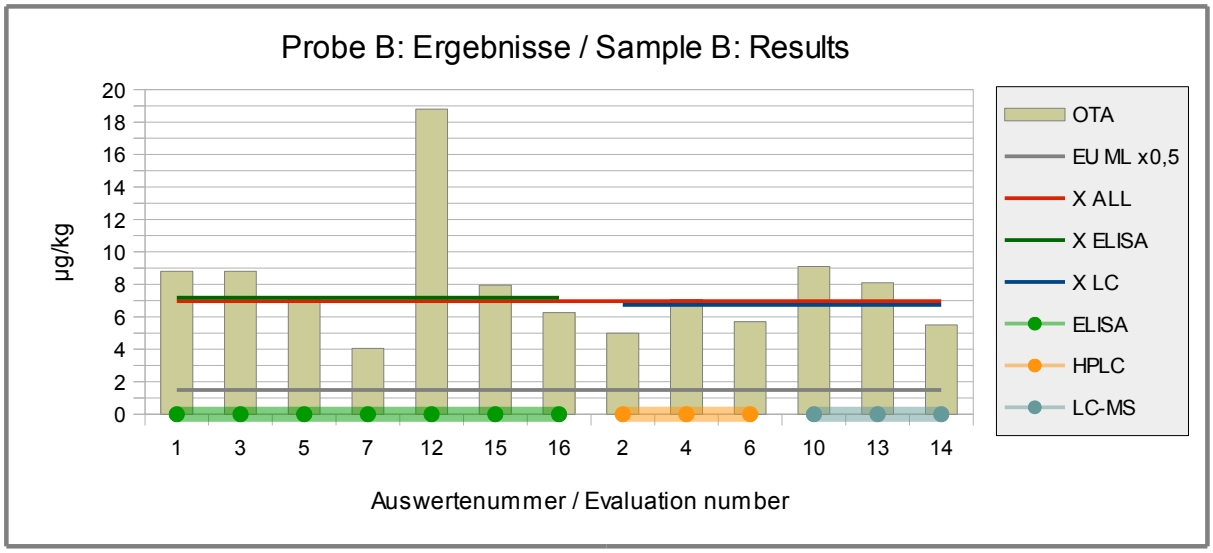

Abb./Fig. 8: Results Ochratoxin A (OTA)
red line = Assigned value robust mean results all methods
green line = Assigned value robust mean results ELSIA methods
blue line = Assigned value robust mean results LC methods
grey line = Qual. valuation as positive > 2,0 µg/kg
round symbols = Applied methods (see legend)

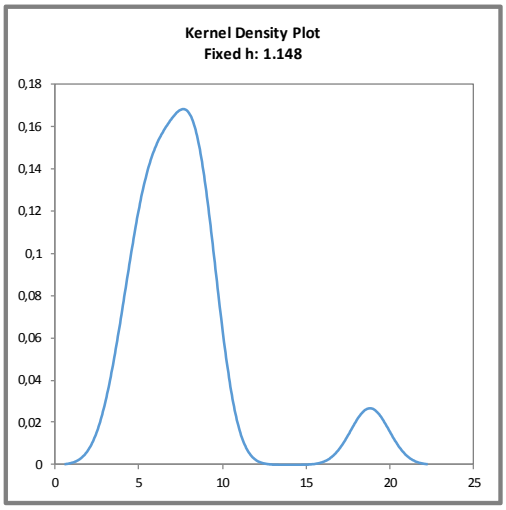

Abb. / Fig. 9:
Kerndichte-Schätzung aller Ergebnisse
(mit h = 0,75 x σ_{pt} von Xpt_{ALL})

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

Comments:
The kernel density estimation shows nearly a symmetrical distribution of results with a side peak >15 µg/kg due to an outlier.

z-Scores der Ergebnisse: Ochratoxin A
z-Scores of Results: Ochratoxin A

Evaluation number	Sample B	Deviation	z-Score Xpt_{ALL}	Deviation	z-Score Xpt_{ELISA}	Deviation	z-Score Xpt_{LC}	Method	Remarks
	[µg/kg]	X All		X ELISA		X LC			
1	8,80	1,84	1,2	1,63	1,0			ELISA	
3	8,80	1,84	1,2	1,63	1,0			ELISA	
5	7,11	0,15	0,1	-0,06	-0,04			ELISA	
7	4,05	-2,91	-1,9	-3,12	-2,0			ELISA	
12	18,8	11,84	7,7	11,63	7,37			ELISA	Outlier X_{all} a. X_{ELISA}
15	7,95	0,99	0,6	0,78	0,49			ELISA	Mean calculated by DLA
16	6,25	-0,71	-0,5	-0,92	-0,58			ELISA	Mean calculated by DLA
2	4,99	-1,98	-1,3			-1,76	-1,18	HPLC	
4	7,06	0,10	0,1			0,32	0,2	HPLC	
6	5,70	-1,26	-0,8			-1,04	-0,7	HPLC	
10	9,10	2,14	1,4			2,36	1,59	LC-MS	
13	8,10	1,14	0,7			1,36	0,92	LC-MS	
14	5,50	-1,46	-1,0			-1,24	-0,84	LC-MS	

Methods:
further details see documentation

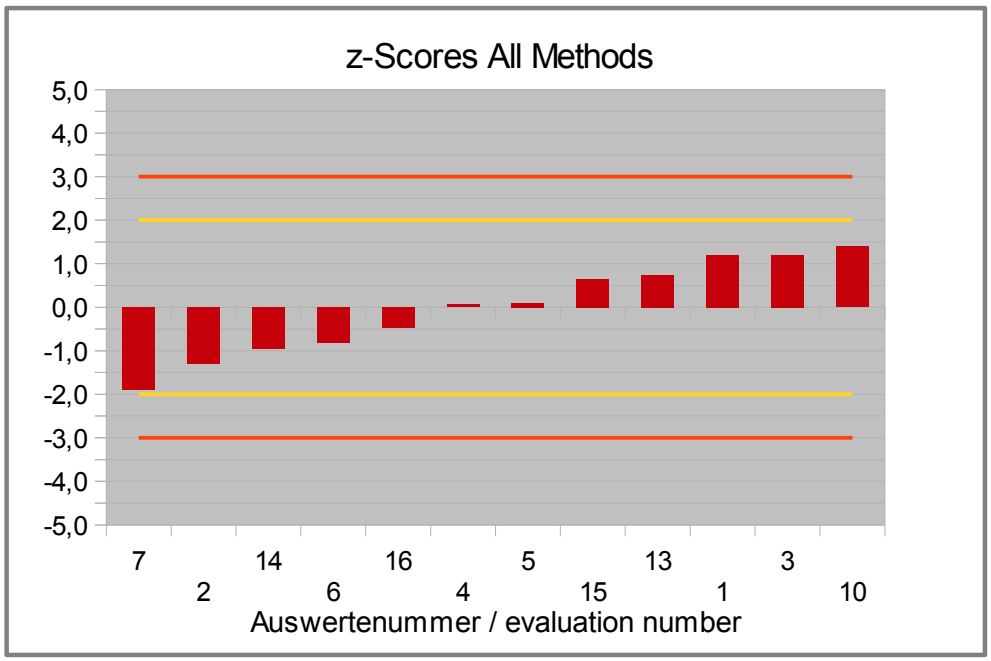

Abb./Fig. 10:
z-Scores Ochratoxin A (OTA)
Assigned value robust mean results all methods

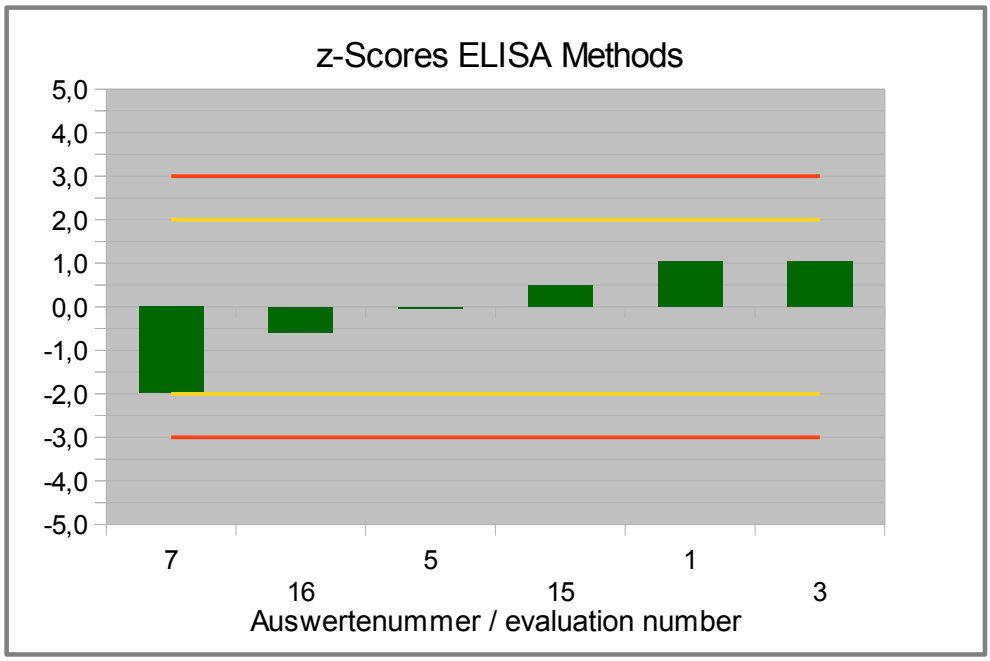

Abb./Fig. 11:
z-Scores Ochratoxin A (OTA)
Assigned value robust mean results ELISA methods

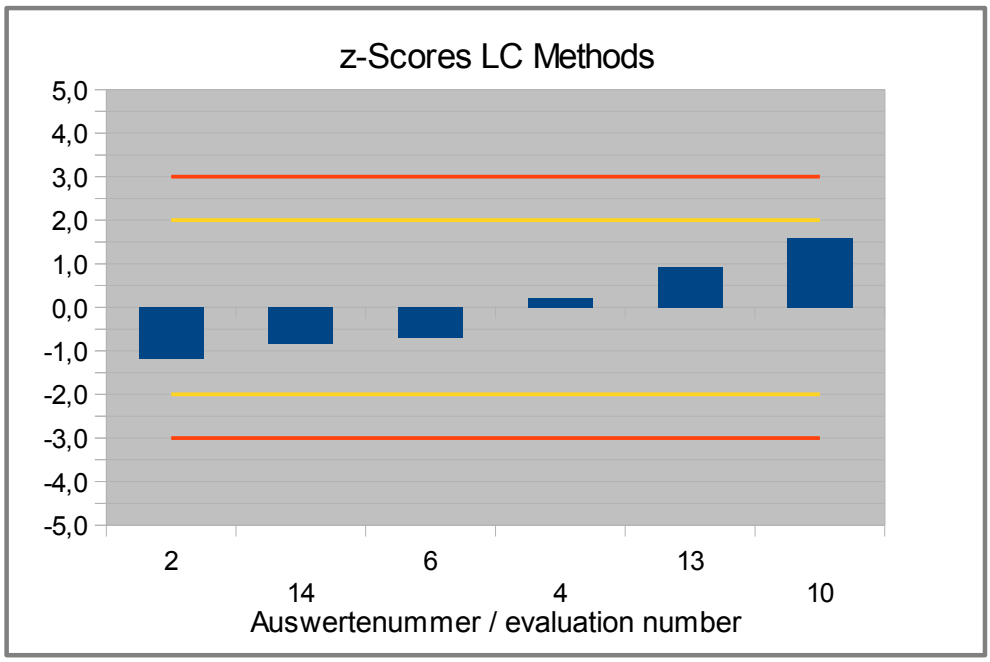

Abb./Fig. 12:
z-Scores Ochratoxin A (OTA)
Assigned value robust mean results LC methods

4.3 Proficiency Test Deoxynivalenol

4.3.1 Results: Deoxynivalenol (DON)

Qualitative valuation of results: Samples A and B

Evaluation number	Sample A	Sample A	Sample B	Sample B	Qualitative Valuation	Method	Remarks
	pos/neg	[µg/kg]	pos/neg	[µg/kg]	Agreement with consensus value		
1	positive	838	negative	29,2	2/2 (100%)	ELISA	
2	negative	1,42	negative	0,24	1/2 (50%)	ELISA	
3	negative	77,2	negative	5,57	1/2 (50%)	ELISA	
5	positive	1191	negative	<100	2/2 (100%)	ELISA	
7	positive	353	negative	<18,5	2/2 (100%)	ELISA	
8	positive	670	negative	222	2/2 (100%)	ELISA	
9	positive	827		<600	1/1 (100%)	ELISA	
12	positive	1765	negative	75,3	2/2 (100%)	ELISA	
15	positive	825	negative	120	2/2 (100%)	ELISA	Mean calculated by DLA
16	positive	834	negative	189	2/2 (100%)	ELISA	Mean calculated by DLA
4	positive	718	negative	<25	2/2 (100%)	LC-MS	
6	positive	721	negative	< 20	2/2 (100%)	LC-MS	
10	positive	430	negative	<100	2/2 (100%)	LC-MS	
13	positive	848	negative	0 (<10)	2/2 (100%)	LC-MS	
14	positive	716	negative	(<20)	2/2 (100%)	LC-MS	
11	positive	507	negative	12	2/2 (100%)	div	

	Sample A		Sample B	
Number positive	14		0	
Number negative	2		15	
Percent positive	88		0	
Percent negative	13		100	
Consensus value	positive		negative	

Methods:
further details see documentation

positve: > 250 µg/kg (EU maximum level x 0,5)
negative: < 250 µg/kg (EU maximum level x 0,5)

Comments:
The acceptance level for the classification of the results as positive or negative was set at 250 µg/kg (see 3.1 and Table 4).
For sample A, 88% of the results were above and for sample B all results were below the acceptance level (note: the indication <600 µg/kg was not considered).

Quantative valuation: Deoxynivalenol in µg/kg

Sample A

Statistic Data	**All Methods**	**ELISA Methods**	**LC Methods**
Number of results	14°	8°	5
Number of outliers	2	2	0
Mean	803	913	687
Median	773	831	718
Robust Mean (X_{pt})	**755**	**868**	**702**
Robust standard deviation (S*)	**250**	**360**	**139**
Number with 2 replicates	10	7	2
Repeatability SD (S_r)	37,2	40,1	35,3
Repeatability (CV_r)	5,18%	5,07%	6,19%
Reproducibility SD (S_R)	247	251	207
Reproducibility (CV_R)	34,3%	31,7%	36,3%
Target range:			
Target standard deviation σ_{pt}	**165**	**190**	**154**
Target standard deviation (for Information)	126	142	118
lower limit of target range	**425**	**488**	**395**
upper limit of target range	**1090**	**1250**	**1010**
Quotient S/σ_{pt}*	*1,5*	*1,9*	*0,90*
Standard uncertainty $U_{(Xpt)}$	*83,6*	*159*	*77,5*
Results in the target range	*11*	*6*	*5*
Percent in the target range	*79%*	*75%*	*100%*

° *without outliers (results no. 2 and 3)*

Comments to the statistical characteristics:

The target standard deviations were calculated using data from a precision experiment (3.2.6.2). For information the target standard deviations according to the general model of Horwitz were given (s. 3.2.6.1).

The distributions of results showed a normal to low variability. The quotients S*/σ_{pt} were below 2,0 each.

The repeatability and reproducibility standard deviation are in the range of established values of the applied methods (see 3.2.6.2).

79% of the results of all methods were in the target range.

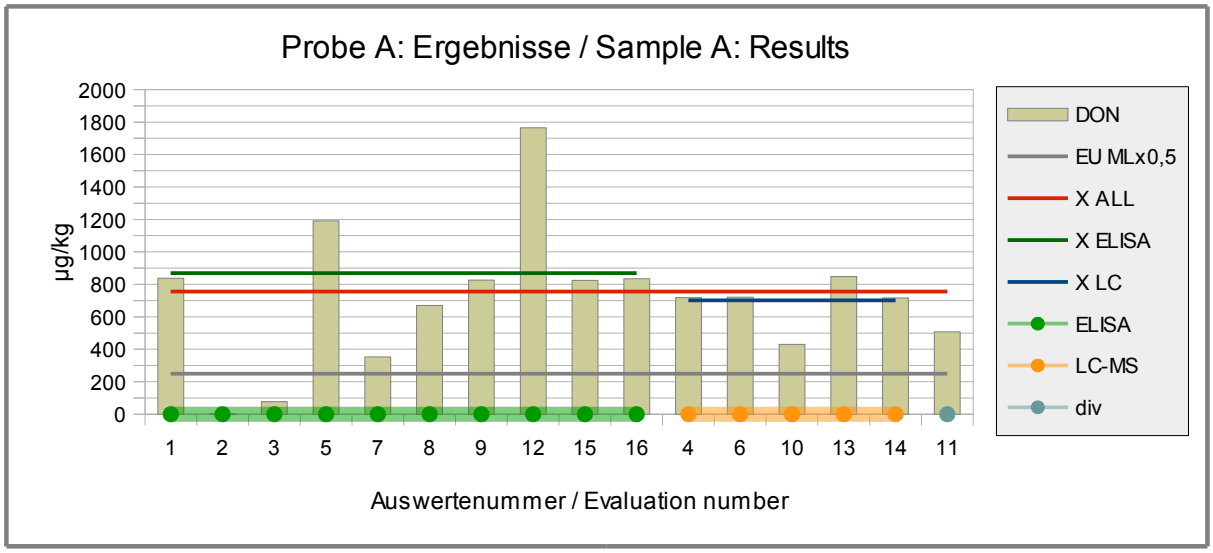

Abb./Fig. 13: Results Deoxynivalenol (DON)
red line = Assigned value robust mean results all methods
green line = Assigned value robust mean results ELSIA methods
blue line = Assigned value robust mean results LC methods
grey line = Qual. valuation as positive > 250 µg/kg
round symbols = Applied methods (see legend)

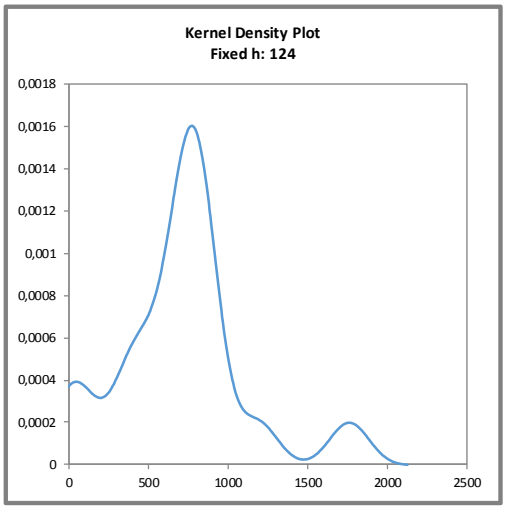

Abb. / Fig. 14:
Kerndichte-Schätzung aller Ergebnisse
(mit h = 0,75 x σpt von Xpt_{ALL})

Kernel density plot of all results
(with h = 0,75 x σpt of Xpt_{ALL})

Comments:
The kernel density estimation shows nearly a symmetrical distribution of results with two shoulders (at <500 and >1000 µg/kg) as well as two side peaks at <100 and approx. 1700 µg/kg.

z-Scores der Ergebnisse: Deoxynivalenol
z-Scores of Results: Deoxynivalenol

Evaluation number	Sample A	Deviation	z-Score Xpt_{ALL}	Deviation	z-Score Xpt_{ELISA}	Deviation	z-Score Xpt_{LC}	Method	Remarks
	[µg/kg]	X All		X ELISA		X LC			
1	838	82,6	0,5	-30,2	-0,16			ELISA	
2	1,42	-754,0	-4,6	-866,8	-4,6			ELISA	Outlier X_{all} a. X_{ELISA}
3	77,2	-678,2	-4,1	-791,0	-4,2			ELISA	Outlier X_{all} a. X_{ELISA}
5	1191	435,1	2,6	322,3	1,7			ELISA	
7	353	-402,7	-2,4	-515,5	-2,7			ELISA	
8	670	-85,4	-0,5	-198,2	-1,0			ELISA	
9	827	71,6	0,4	-41,2	-0,22			ELISA	
12	1765	1009,6	6,1	896,8	4,7			ELISA	
15	825	69,6	0,4	-43,2	-0,23			ELISA	Mean calculated by DLA
16	834	78,6	0,5	-34,2	-0,18			ELISA	Mean calculated by DLA
4	718	-37,4	-0,2			15,8	0,10	LC-MS	
6	721	-34,4	-0,2			18,8	0,12	LC-MS	
10	430	-325,4	-2,0			-272,2	-1,8	LC-MS	
13	848	92,6	0,6			145,8	0,95	LC-MS	
14	716	-39,4	-0,2			13,8	0,09	LC-MS	
11	507	-248,4	-1,5					div	

Methods:
further details see documentation

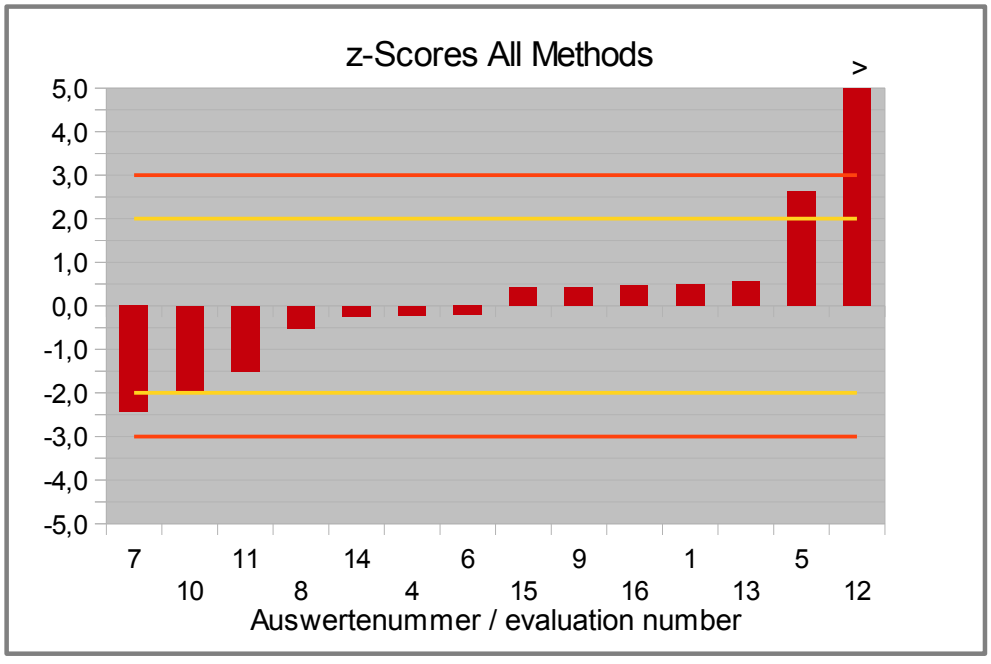

Abb./Fig. 15:
z-Scores Deoxynivalenol (DON)
Assigned value robust mean results all methods

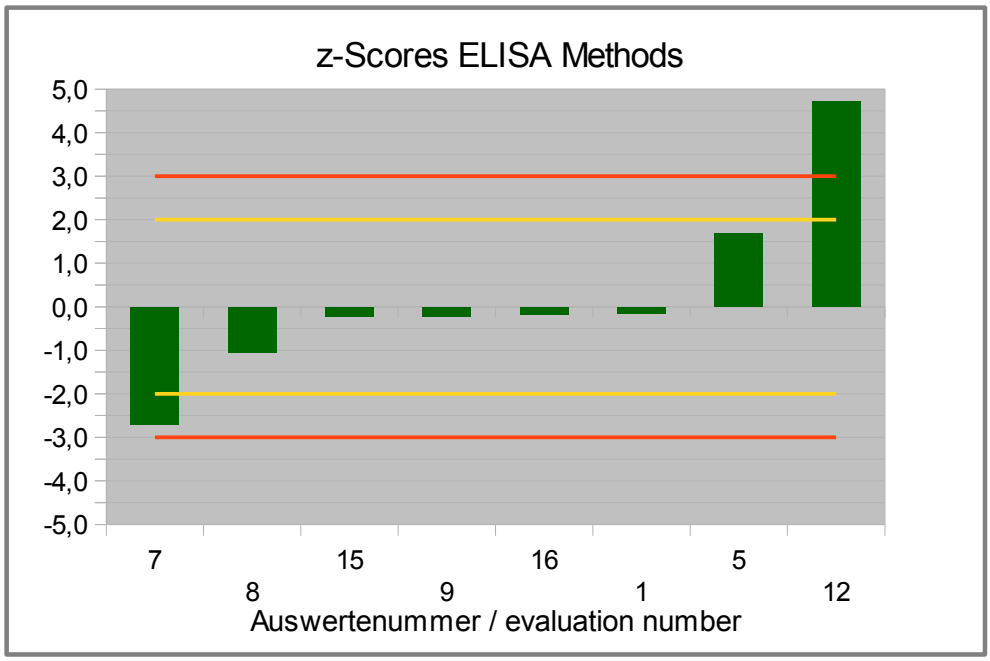

Abb./Fig. 16:
z-Scores Deoxynivalenol (DON)
Assigned value robust mean results ELISA methods

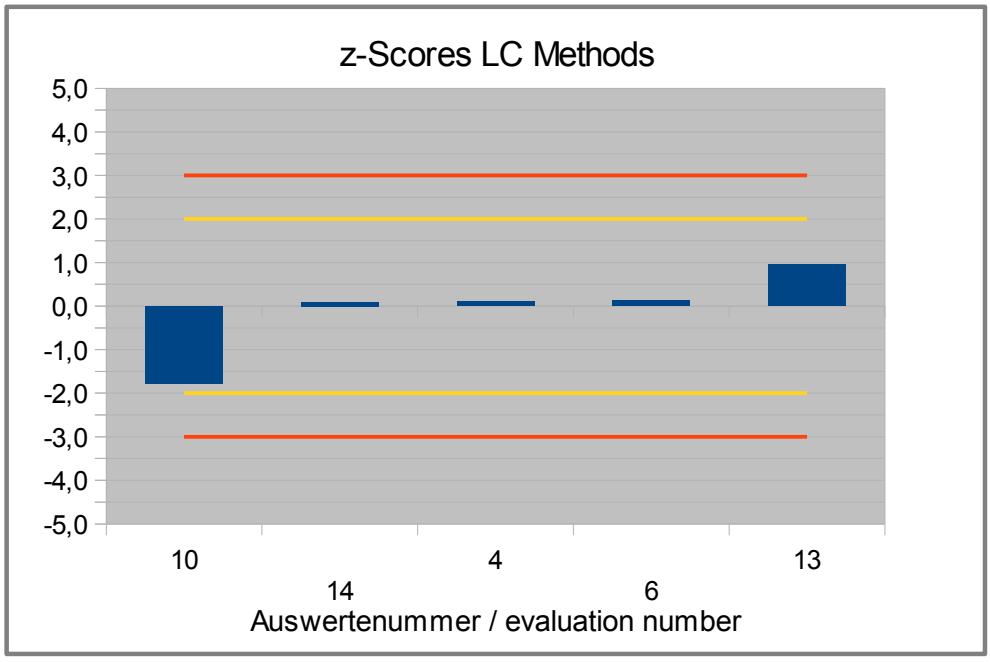

Abb./Fig. 17:
z-Scores Deoxynivalenol (DON)
Assigned value robust mean results LC methods

4.4 Proficiency Test Fumonisins

4.4.1 Results: Fumonisin B1 (FUMO B1)

Qualitative valuation of results: Samples A and B

Evaluation number	Sample A	Sample A	Sample B	Sample B	Qualitative Valuation	Method	Remarks
	pos/neg	[µg/kg]	pos/neg	[µg/kg]	Übereinstimmungen mit Konsenswerten		
4	positive	223	negative	23,3	2/2 (100%)	HPLC	
6	positive	149	negative	<20	2/2 (100%)	LC-MS	
13	positive	293	negative	0 (<20)	2/2 (100%)	LC-MS	
14	positive	80	negative	<10	2/2 (100%)	LC-MS	

	Sample A		Sample B	
Number positive	4		0	
Number negative	0		4	
Percent positive	100		0	
Percent negative	0		100	
Consensus value	positive		negative	

Methods:
further details see documentation

positve: > 75 µg/kg (0,75 x EU maximum level x 0,5)
negative: < 75 µg/kg (0,75 x EU maximum level x 0,5)

Comments:
The acceptance level for the classification of the results as positive or negative was set at 75 µg/kg (see 3.1 and Table 4).
For sample A all the results were above and for sample B all results were below the acceptance level.

Quantative evaluation: Fumonisin B1 in µg/kg

Due to the small number of results no quantitative evaluation was done.

4.4.2 Results: Fumonisin B2 (FUMO B2)

Qualitative valuation of results: Samples A and B

Evaluation number	Sample A	Sample A	Sample B	Sample B	Qualitative Valuation	Method	Remarks
	pos/neg	[µg/kg]	pos/neg	[µg/kg]	Übereinstimmungen mit Konsenswerten		
4	positive	35,0	negative	< 10	2/2 (100%)	HPLC	
6	positive	34,0	negative	<20	2/2 (100%)	LC-MS	
13	positive	47,0	negative	0 (<12)	2/2 (100%)	LC-MS	
14	positive	36,0	negative	16,0	2/2 (100%)	LC-MS	

	Sample A		Sample B	
Number positive	4		0	
Number negative	0		4	
Percent positive	100		0	
Percent negative	0		100	
Consensus value	positive		negative	

Methods:
further details see documentation

positve: > 25 µg/kg (0,25 x EU maximum level x 0,5)
negative: < 25 µg/kg (0,25 x EU maximum level x 0,5)

Comments:
The acceptance level for the classification of the results as positive or negative was set at 25 µg/kg (see 3.1 and Table 4).
For sample A all the results were above and for sample B all results were below the acceptance level.

Quantative evaluation: Fumonisin B2 in µg/kg

Due to the small number of results no quantitative evaluation was done.

4.4.3 Results: Fumonisins Sum (FUMO Sum)

Qualitative valuation of results: Samples A and B

Evaluation number	Sample A	Sample A	Sample B	Sample B	Qualitative Valuation	Method	Remarks
	pos/neg	[µg/kg]	pos/neg	[µg/kg]	Agreement with consensus value		
1	negative	0,21	negative	<BG (<25)	1/2 (50%)	ELISA	
3	positive	189	negative	1,78	2/2 (100%)	ELISA	
5	positive	527	positive	123	1/2 (50%)	ELISA	
7	positive	230	negative	<25	2/2 (100%)	ELISA	
8	positive	442	positive	251	1/2 (50%)	ELISA	
12	positive	200	negative	< 50	2/2 (100%)	ELISA	
15	positive	214	negative	12,8	2/2 (100%)	ELISA	Mean calculated by DLA
16	positive	271	negative	3,2	2/2 (100%)	ELISA	Mean calculated by DLA
4	positive	258	negative	23,3	2/2 (100%)	HPLC	
6	positive	183	negative	< 40	2/2 (100%)	LC-MS	
13	positive	340	negative	0 (<32)	2/2 (100%)	LC-MS	
14	positive	116	negative	16	2/2 (100%)	LC-MS	

	Sample A		Sample B	
Number positive	11		2	
Number negative	1		10	
Percent positive	92		17	
Percent negative	8		83	
Consensus value	positive		negative	

Methods:
further details see documentation

positve: > 100 µg/kg (EU maximum level x 0,5)
negative: < 100 µg/kg (EU maximum level x 0,5)

Comments:
The acceptance level for the classification of the results as positive or negative was set at 100 µg/kg (see 3.1 and Table 4).
For sample A, 92% of the results were above and for sample B 83% of the results were below the acceptance level.

Quantative valuation: Fumonisins Sum in µg/kg

Sample A

Statistic Data	All Methods	ELISA Methods
Number of results	11°	7°
Number of outliers	1	1
Mean	270	296
Robust Mean	262	295
Median (X_{pt})	**230**	**230**
Robust standard deviation (S*)	**119**	**148**
Number with 2 replicates	8	7
Repeatability SD (S_r)	29,5	31,3
Repeatability (CV_r)	10,8%	10,6%
Reproducibility SD (S_R)	141	135
Reproducibility (CV_R)	51,5%	45,7%
Target range:		
Target standard deviation σ_{pt}'	**67,5**	**86,4**
Target standard deviation (for Information)	45,9	45,9
lower limit of target range	**95,1**	**57,2**
upper limit of target range	**365**	**403**
Quotient S/σ_{pt}'*	*1,8*	*1,7*
Standard uncertainty $U_{(Xpt)}$	*44,7*	*70,1*
Results in the target range	*9*	*5*
Percent in the target range	*82%*	*71%*

° *without outliers (result no. 1)*

Comments to the statistical characteristics:

The median was used as the assigned value each (s. 3.2.1).

The target standard deviations were calculated using data from a precision experiment (3.2.6.2). For information the target standard deviations according to the general model of Horwitz were given (s. 3.2.6.1).

The distributions of results showed a slightly increased variability. The quotients S*/σ_{pt} were >2,0 each. Therefore both evaluations were done by z'-scores considering the standard uncertainty (s. 3.2.8). The quotients S*/σ_{pt}' were each below 2,0 then.

The repeatability standard deviations are in the range of established values of the applied methods (see 3.2.6.2).

82% of the results of all methods were in the target range.

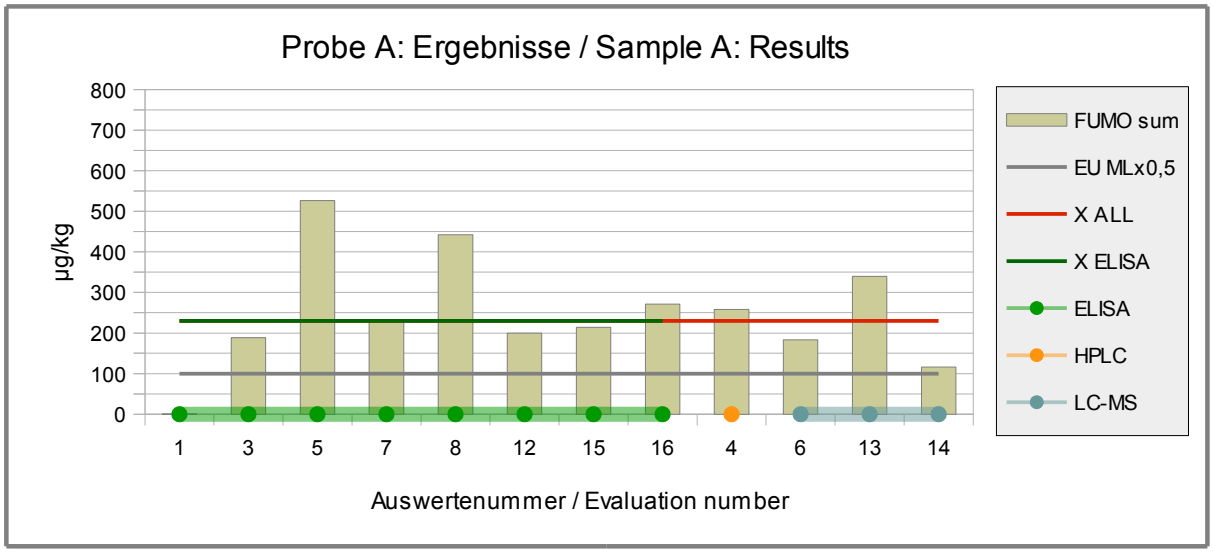

Abb./Fig. 18: Results Fumonisins Sum (FUMO Sum)
red line = Assigned value robust mean results all methods
green line = Assigned value robust mean results ELSIA methods
grey line = Qual. valuation as positive > 100 µg/kg
round symbols = Applied methods (see legend)

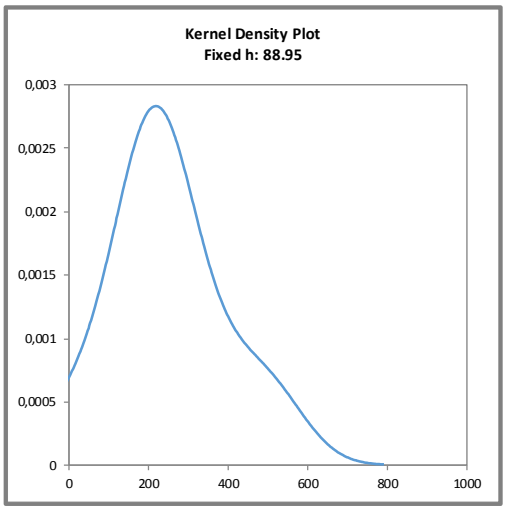

Abb. / Fig. 19:
Kerndichte-Schätzung aller Ergebnisse
(mit h = 0,75 x σpt von Xpt_{ALL})

Kernel density plot of all results
(with h = 0,75 x σpt of Xpt_{ALL})

Comments:
The kernel density estimation shows nearly a symmetrical distribution of results with a shoulder at 400-600 µg/kg.

z-Scores der Ergebnisse: Fumonisine Summe
z-Scores of Results: Fumonisins Sum

Evaluation number	Sample A	Deviation	z-Score Xpt_{ALL}	Deviation	z-Score Xpt_{ELISA}	Method	Remarks
	[µg/kg]	X All		X ELISA			
1	0,21	-229,8	-3,4	-229,8	-2,7	ELISA	Outlier X_{all} a. X_{ELISA}
3	189	-41,5	-0,62	-41,5	-0,48	ELISA	
5	527	296,8	4,4	296,8	3,4	ELISA	
7	230	0,0	0,00	0,0	0,00	ELISA	
8	442	212,0	3,1	212,0	2,5	ELISA	
12	200	-30,0	-0,44	-30,0	-0,35	ELISA	
15	214	-16,0	-0,24	-16,0	-0,19	ELISA	Mean calculated by DLA
16	271	41,0	0,61	41,0	0,47	ELISA	Mean calculated by DLA
4	258	28,0	0,42			HPLC	
6	183	-47,0	-0,70			LC-MS	
13	340	110,0	1,6			LC-MS	
14	116	-114,0	-1,7			LC-MS	

Methods:
further details see documentation

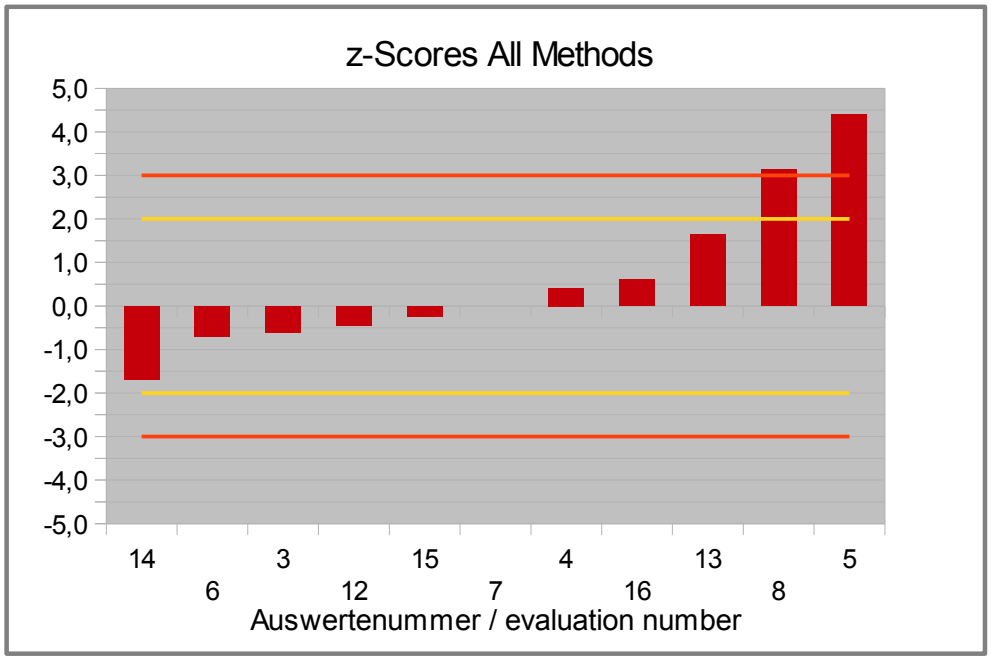

Abb./Fig. 20:
z-Scores Fumonisins Sum (FUMO Sum)
Assigned value robust mean results all methods

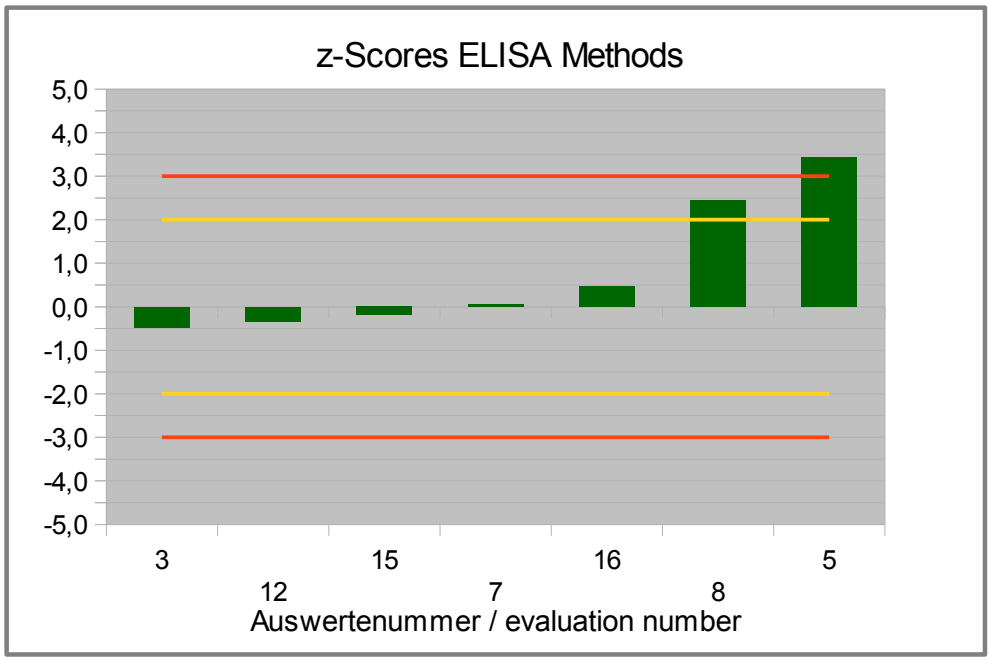

Abb./Fig. 21:
z-Scores Fumonisins Sum (FUMO Sum)
Assigned value robust mean results ELISA methods

4.5 Proficiency Test Zearalenone

4.5.1 Results: Zearalenone (ZON)

Qualitative valuation of results: Samples A and B

Evaluation number	Sample A	Sample A	Sample B	Sample B	Qualitative Valuation	Method	Remarks
	pos/neg	[µg/kg]	pos/neg	[µg/kg]	Agreement with consensus value		
1	positive	73,0	negative	<BG (<1,75)	2/2 (100%)	ELISA	
3	positive	36,4	negative	13,4	2/2 (100%)	ELISA	
5	positive	47,0	negative	14,5	2/2 (100%)	ELISA	
7	positive	49,2	negative	<1,75	2/2 (100%)	ELISA	
8	positive	62,0	positive	50	1/2 (50%)	ELISA	
12	positive	44,9	negative	< 25	2/2 (100%)	ELISA	
15	positive	72,2	negative	14,1	2/2 (100%)	ELISA	Mean calculated by DLA
16	positive	51,9	negative	10,2	2/2 (100%)	ELISA	Mean calculated by DLA
4	positive	62,3	negative	<5	2/2 (100%)	LC-MS	
6	positive	60,0	negative	< 10	2/2 (100%)	LC-MS	
10	negative	23,0	negative	<10	1/2 (50%)	LC-MS	
13	positive	64,0	negative	0 (<4)	2/2 (100%)	LC-MS	
14	positive	61,0	negative	5,50	2/2 (100%)	LC-MS	

	Sample A		Sample B	
Number positive	12		1	
Number negative	1		12	
Percent positive	92		8	
Percent negative	8		92	
Consensus value	positive		negative	

Methods:
further details see documentation

positve: > 25 µg/kg (EU maximum level x 0,5)
negative: < 25 µg/kg (EU maximum level x 0,5)

Comments:
The acceptance level for the classification of the results as positive or negative was set at 25 µg/kg (see 3.1 and Table 4).
For sample A, 92% of the results were above and for sample B below the acceptance level.

Quantative valuation: Zearalenone in µg/kg

Sample A

Statistic Data	All Methods	ELISA Methods	LC Methods
Number of results	13	8	5
Number of outliers	0	0	0
Mean	54,4	54,6	54,1
Median	60,0	50,6	61,0
Robust Mean (X_{pt})	**55,2**	**54,6**	**60,0**
Robust standard deviation (S*)	**14,1**	**15,0**	**4,98**
Number with 2 replicates	10	8	2
Repeatability SD (S_r)	4,30	4,03	5,22
Repeatability (CV_r)	8,26%	7,39%	12,5%
Reproducibility SD (S_R)	15,8	13,5	26,8
Reproducibility (CV_R)	30,5%	24,7%	64,1%
Target range:			
Target standard deviation σ_{pt}	**12,1**	**12,0**	**13,2**
Target standard deviation (for Information)	12,6	12,5	13,7
lower limit of target range	**30,9**	**30,6**	**33,6**
upper limit of target range	**79,5**	**78,6**	**86,4**
Quotient S/σ_{pt}*	*1,2*	*1,2*	*0,38*
Standard uncertainty $U_{(Xpt)}$	*4,89*	*6,63*	*2,79*
Results in the target range	*12*	*8*	*4*
Percent in the target range	*92%*	*100%*	*80%*

Comments to the statistical characteristics:

The target standard deviation was calculated according to the general model of Horwitz/Thompson (3.2.6.1). For information the target standard deviation using data from a precision experiment was given (s. 3.2.6.2).

The distributions of results showed a normal to low variability. The quotients S*/σ_{pt} were below 2,0 each.

The repeatability and reproducibility standard deviation are in the range of established values of the applied methods (see 3.2.6.2).

92% of the results of all methods were in the target range.

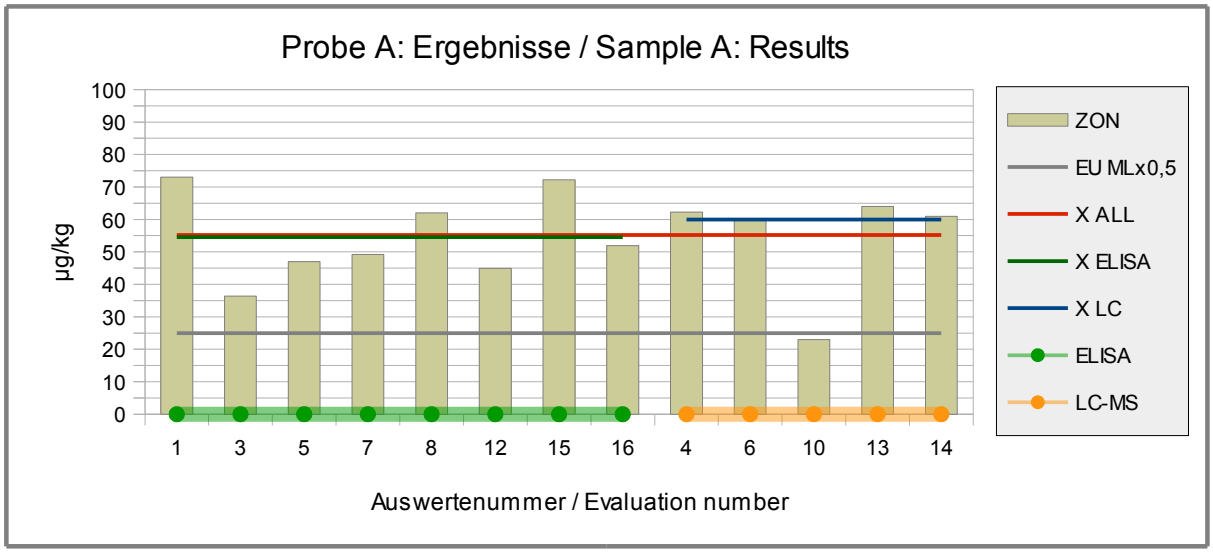

Abb./Fig. 22: Results Zearalenone (ZON)
red line = Assigned value robust mean results all methods
green line = Assigned value robust mean results ELSIA methods
blue line = Assigned value robust mean results LC methods
grey line = Qual. valuation as positive > 25 µg/kg
round symbols = Applied methods (see legend)

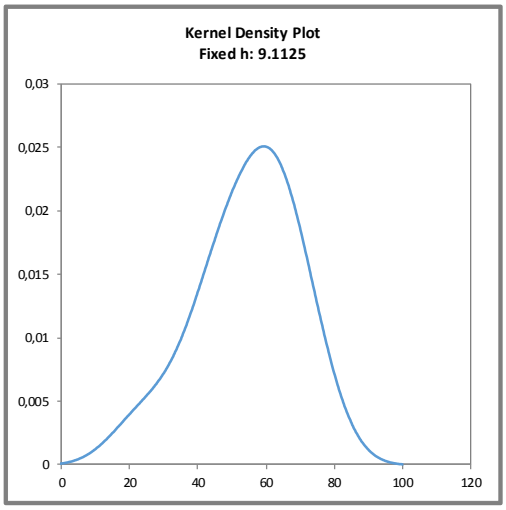

Abb. / Fig. 23:
Kerndichte-Schätzung aller Ergebnisse
(mit h = 0,75 x σpt von Xpt_{ALL})

Kernel density plot of all results
(with h = 0,75 x σpt of Xpt_{ALL})

Comments:
The kernel density estimation shows nearly a symmetrical distribution of results with a slight shoulder at 20-30 µg/kg.

z-Scores der Ergebnisse: Zearalenon
z-Scores of Results: Zearalenone

Evaluation number	Sample A	Deviation	z-Score Xpt_{ALL}	Deviation	z-Score Xpt_{ELISA}	Deviation	z-Score Xpt_{LC}	Method	Remarks
	[µg/kg]	X All		X ELISA		X LC			
1	73,0	17,78	1,5	18,43	1,5			ELISA	
3	36,4	-18,87	-1,6	-18,22	-1,5			ELISA	
5	47,0	-8,22	-0,68	-7,57	-0,63			ELISA	
7	49,2	-6,01	-0,49	-5,36	-0,45			ELISA	
8	62,0	6,78	0,56	7,43	0,62			ELISA	
12	44,9	-10,32	-0,85	-9,67	-0,81			ELISA	
15	72,2	16,98	1,4	17,63	1,5			ELISA	Mean calculated by DLA
16	51,9	-3,32	-0,27	-2,67	-0,22			ELISA	Mean calculated by DLA
4	62,3	7,08	0,58			2,32	0,18	LC-MS	
6	60,0	4,78	0,39			0,02	0,00	LC-MS	
10	23,0	-32,22	-2,7			-36,98	-2,8	LC-MS	
13	64,0	8,78	0,72			4,02	0,30	LC-MS	
14	61,0	5,78	0,48			1,02	0,08	LC-MS	

Methods:
further details see documentation

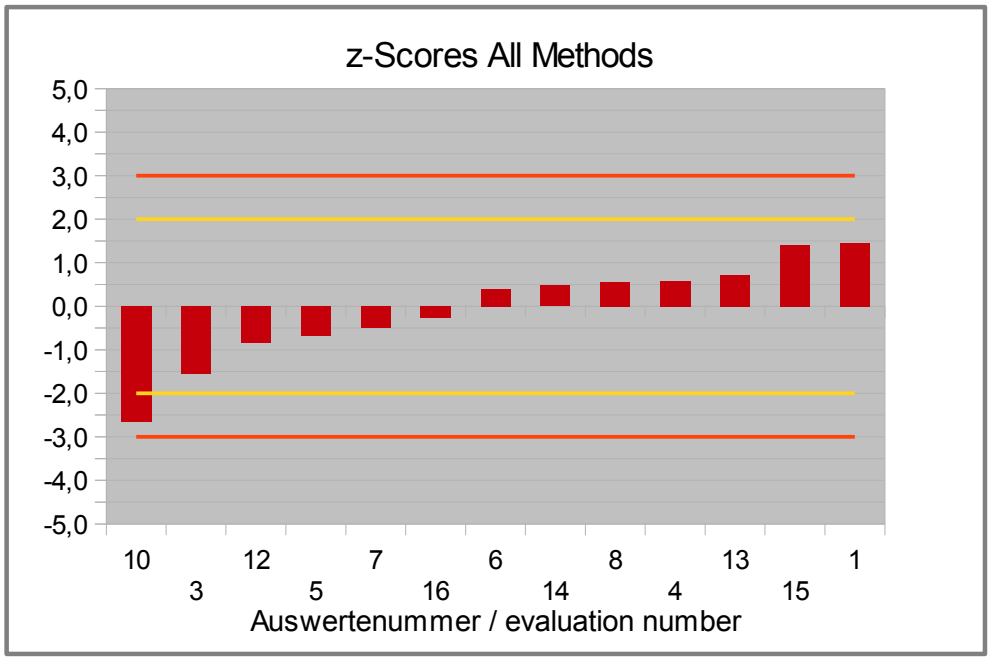

Abb./Fig. 24:
z-Scores Zearalenone (ZON)
Assigned value robust mean results all methods

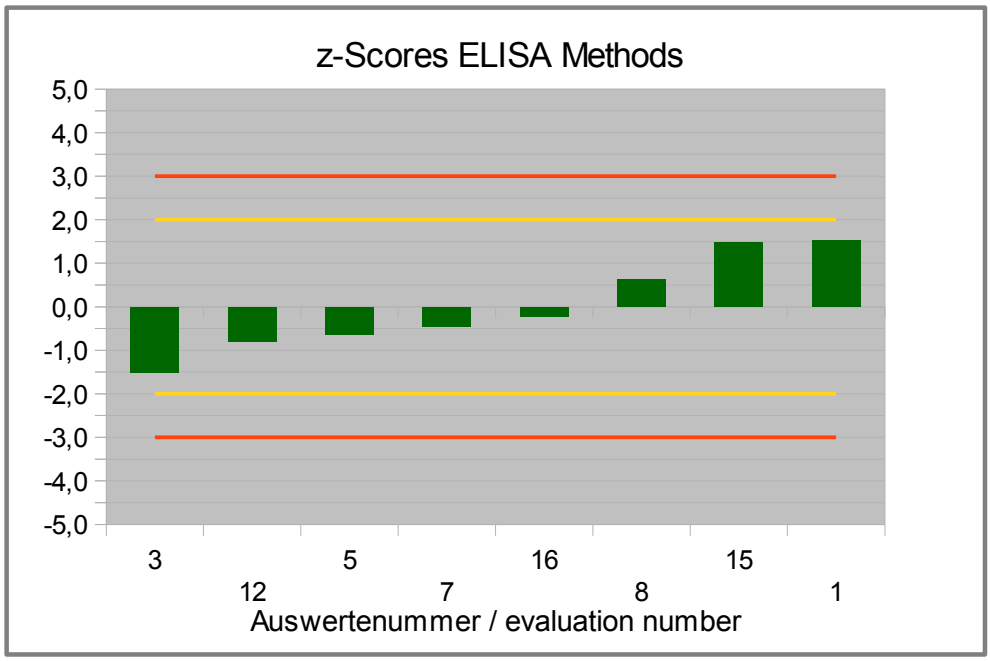

Abb./Fig. 25:
z-Scores Zearalenone (ZON)
Assigned value robust mean results ELISA methods

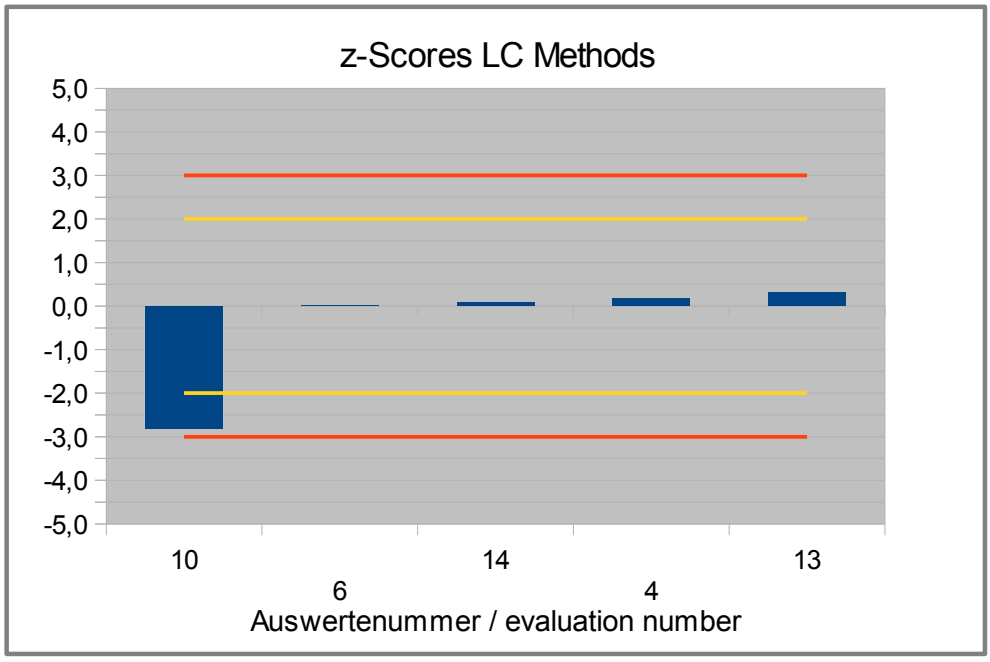

Abb./Fig. 26:
z-Scores Zearalenone (ZON)
Assigned value robust mean results LC methods

4.6 z-Scores of participants: Summary table

Evaluation number	AF B1	AF Sum	AF Sum	AF Sum	OTA	OTA	OTA	DON	DON	DON	FUMO Sum	FUMO Sum	ZON	ZON	ZON
Methods	LC	All	ELISA	LC	All	ELISA	LC	All	ELISA	LC	All	ELISA	All	ELISA	LC
1	-	0,36	1,47	-	1,20	1,03	-	0,50	-0,16	-	-3,41	-2,66	1,46	1,54	-
2	-2,47	-2,14	-	-2,53	-1,29	-	-1,18	-4,56	-4,56	-	-	-	-	-	-
3	-	-1,69	-1,04	-	1,20	1,03	-	-4,10	-4,16	-	-0,62	-0,48	-1,55	-1,52	-
4	0,47	1,73	-	0,71	0,06	-	0,22	-0,23	-	0,10	0,42	-	0,58	-	0,18
5	-	-0,10	0,91	-	0,10	-0,04	-	2,63	1,70	-	4,40	3,43	-0,68	-0,63	-
6	1,97	3,30	-	2,02	-0,82	-	-0,70	-0,21	-	0,12	-0,70	-	0,39	-	0,00
7	-1,21	-0,17	-	-0,88	-1,90	-1,98	-	-2,44	-2,71	-	0,00	0,00	-0,49	-0,45	-
8	-	-	-	-	-	-	-	-0,52	-1,04	-	3,14	2,45	0,56	0,62	-
9	-	-	-	-	-	-	-	0,43	-0,22	-	-	-	-	-	-
10	0,49	1,01	-	0,10	1,40	-	1,59	-1,97	-	-1,77	-	-	-2,65	-	-2,80
11	-	-	-	-	-	-	-	-1,50	-	-	-	-	-	-	-
12	-	-2,04	-1,47	-	7,73	7,37	-	6,11	4,72	-	-0,44	-0,35	-0,85	-0,81	-
13	0,00	0,90	-	0,01	0,74	-	0,92	0,56	-	0,95	1,63	-	0,72	-	0,30
14	0,69	1,23	-	0,29	-0,95	-	-0,84	-0,24	-	0,09	-1,69	-	0,48	-	0,08
15	-	-0,41	0,53	-	0,65	0,49	-	0,42	-0,23	-	-0,24	-0,19	1,40	1,47	-
16	-	-1,17	-0,40	-	-0,46	-0,58	-	0,48	-0,18	-	0,61	0,47	-0,27	-0,22	-

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

Parameter	Meth. Abr.	Partici-pant	Unit	Date of Analysis	Result (Mean)	Result I	Result II	Result (Mean)	Result I	Result II	Limit of Quanti-tation	Incl. Re-covery	Recovery Rate
				Day/Month	Sample A	Sample A	Sample A	Sample B	Sample B	Sample B		yes/no	in %
Aflatoxin B1	ELISA	1	µg/kg										
Aflatoxin B1	ELISA	3	µg/kg										
Aflatoxin B1	ELISA	5	µg/kg	05/06	0,797	0,803	0,79	4,08	3,78	4,38	0,7	no	
Aflatoxin B1	ELISA	12	µg/kg										
Aflatoxin B1	ELISA	15	µg/kg										
Aflatoxin B1	ELISA	16	µg/kg	05.06./ 07.06.		0,1	0,2		2,7	3,5	0-8 ppb		
Aflatoxin B1	HPLC	2	µg/kg	29.04. - 31.05.	<0,1	<0,1	<0,1	2,1	2,45	1,74	0,1	no	
Aflatoxin B1	HPLC	4	µg/kg	04.06.19	<0,12			5,08			0,12	yes	91,8
Aflatoxin B1	HPLC	6	µg/kg	May/2019	< 0,20 µg/kg			6,6 µg/kg			< 0,20 µg/kg		
Aflatoxin B1	HPLC	7	µg/kg	07.05.19	<0,01	<0,01	<0,01	3,38	3,34	3,42	0,02	no	-
Aflatoxin B1	LC-MS	10	µg/kg	14.05.19	<0,5	<0,5	<0,5	5,1	5,5	4,6	<0,5	yes	100
Aflatoxin B1	LC-MS	13	µg/kg	03.05.19	0			4,6			0,1	yes	ISTD 13C
Aflatoxin B1	LC-MS	14	µg/kg		<0.5			5,3	5,1	5,4	0,5	no	88

Parameter	Meth. Abr.	Partici-pant	Unit	Date of Analysis	Result (Mean)	Result I	Result II	Result (Mean)	Result I	Result II	Limit of Quanti-tation	Incl. Re-covery	Recovery Rate
				Day/Month	Sample A	Sample A	Sample A	Sample B	Sample B	Sample B		yes/no	in %
Aflatoxin B2	ELISA	1	µg/kg										
Aflatoxin B2	ELISA	3	µg/kg										
Aflatoxin B2	ELISA	5	µg/kg										
Aflatoxin B2	ELISA	12	µg/kg										
Aflatoxin B2	ELISA	15	µg/kg										
Aflatoxin B2	ELISA	16	µg/kg										
Aflatoxin B2	HPLC	2	µg/kg	29.04. - 31.05.	<0,1	<0,1	<0,1	0,116	0,124	0,107	0,1	no	
Aflatoxin B2	HPLC	4	µg/kg	04.06.19	<0,12			0,4			0,12	yes	101,3
Aflatoxin B2	HPLC	6	µg/kg	May/2019	< 0,20 µg/kg			0,41 µg/kg			< 0,20 µg/kg		
Aflatoxin B2	HPLC	7	µg/kg	07.05.19	<0,01	<0,01	<0,01	0,34	0,35	0,33	0,01	no	
Aflatoxin B2	LC-MS	10	µg/kg	14.05.19	<0,5	<0,5	<0,5	<0,5	<0,5	<0,5	<0,5	yes	100
Aflatoxin B2	LC-MS	13	µg/kg	03.05.19	0			0,21			0,1	yes	ISTD 13C
Aflatoxin B2	LC-MS	14	µg/kg										

Parameter	Meth. Abr.	Partici-pant	Unit	Date of Analysis	Result (Mean)	Result I	Result II	Result (Mean)	Result I	Result II	Limit of Quanti-tation	Incl. Re-covery	Recovery Rate
				Day/Month	Sample A	Sample A	Sample A	Sample B	Sample B	Sample B		yes/no	in %
Aflatoxin G1	ELISA	1	µg/kg										
Aflatoxin G1	ELISA	3	µg/kg										
Aflatoxin G1	ELISA	5	µg/kg										
Aflatoxin G1	ELISA	12	µg/kg										
Aflatoxin G1	ELISA	15	µg/kg										
Aflatoxin G1	ELISA	16	µg/kg										
Aflatoxin G1	HPLC	2	µg/kg	29.04. - 31.05.	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1	0,1	no	
Aflatoxin G1	HPLC	4	µg/kg	04.06.19	<0,12			0,28			0,12	yes	90,3
Aflatoxin G1	HPLC	6	µg/kg	May/2019	< 0,20 µg/kg			0,25 µg/kg			< 0,20 µg/kg		
Aflatoxin G1	HPLC	7	µg/kg	07.05.19	<0,01	<0,01	<0,01	0,24	0,22	0,26	0,05	no	
Aflatoxin G1	LC-MS	10	µg/kg	14.05.19	<0,5	<0,5	<0,5	<0,5	<0,5	<0,5	<0,5	yes	100
Aflatoxin G1	LC-MS	13	µg/kg	03.05.19	0			0,16			0,1	yes	ISTD 13C
Aflatoxin G1	LC-MS	14	µg/kg										

Parameter	Meth. Abr.	Partici-pant	Unit	Date of Analysis	Result (Mean)	Result I	Result II	Result (Mean)	Result I	Result II	Limit of Quanti-tation	Incl. Re-covery	Recovery Rate
				Day/Month	Sample A	Sample A	Sample A	Sample B	Sample B	Sample B		yes/no	in %
Aflatoxin G2	ELISA	1	µg/kg										
Aflatoxin G2	ELISA	3	µg/kg										
Aflatoxin G2	ELISA	5	µg/kg										
Aflatoxin G2	ELISA	12	µg/kg										
Aflatoxin G2	ELISA	15	µg/kg										
Aflatoxin G2	ELISA	16	µg/kg										
Aflatoxin G2	HPLC	2	µg/kg	29.04. - 31.05.	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1	0,1	no	
Aflatoxin G2	HPLC	4	µg/kg	04.06.19	< 0,03			< 0,03			0,12	yes	100
Aflatoxin G2	HPLC	6	µg/kg	May/2019	< 0,20 µg/kg			< 0,20 µg/kg			< 0,20 µg/kg		
Aflatoxin G2	HPLC	7	µg/kg	07.05.19	<0,01	<0,01	<0,01	0,058	0,056	0,06	0,01	no	
Aflatoxin G2	LC-MS	10	µg/kg	14.05.19	<0,5	<0,5	<0,5	<0,5	<0,5	<0,5	<0,5	yes	100
Aflatoxin G2	LC-MS	13	µg/kg	03.05.19	0			0			0,1	yes	ISTD 13C
Aflatoxin G2	LC-MS	14	µg/kg										

Parameter	Meth. Abr.	Partici-pant	Unit	Date of Analysis	Result (Mean)	Result I	Result II	Result (Mean)	Result I	Result II	Limit of Quanti-tation	Incl. Re-covery	Recovery Rate
				Day/Month	Sample A	Sample A	Sample A	Sample B	Sample B	Sample B		yes/no	in %
Sum Aflatoxins	ELISA	1	µg/kg	06.06.19	< BG	< BG	< BG	4,5	4,7	4,2	1,75		
Sum Aflatoxins	ELISA	3	µg/kg	06.06.19	0	0	0	2,625	2,45	2,8			
Sum Aflatoxins	ELISA	5	µg/kg	05/06	0,797	0,803	0,79	4,08	3,78	4,38	0,7	no	
Sum Aflatoxins	ELISA	12	µg/kg	16.05.19	< 1	< 1	< 1	2,3	2,2	2,3	1	no	
Sum Aflatoxins	ELISA	15	µg/kg	6.6./7.6.		0,2	0,3		4,3	3,4			
Sum Aflatoxins	ELISA	16	µg/kg	05.06./ 07.06.		0,1	0,2		2,7	3,5	0-8 ppb		
Sum Aflatoxins	HPLC	2	µg/kg	29.04. - 31.05.	<0,3	<0,3	<0,3	2,21	2,57	1,85	0,3	no	
Sum Aflatoxins	HPLC	4	µg/kg	04.06.19	0			5,76					
Sum Aflatoxins	HPLC	6	µg/kg	May/2019	< 0,80 µg/kg			7,2 µg/kg			< 0,80 µg/kg		
Sum Aflatoxins	HPLC	7	µg/kg	07.05.19	<0,04	<0,04	<0,04	4,02°	3,97	4,07	0,02	no	-
Sum Aflatoxins	LC-MS	10	µg/kg	14.05.19	<2	<2	<2	5,1	5,5	4,6	<2	yes	100
Sum Aflatoxins	LC-MS	13	µg/kg	03.05.19	0			5			0,4	yes	ISTD 13C
Sum Aflatoxins	LC-MS	14	µg/kg		<0.5			5,3	5,1	5,4	0,5	no	88

Parameter	Meth. Abr.	Partici-pant	Unit	Date of Analysis	Result (Mean)	Result I	Result II	Result (Mean)	Result I	Result II	Limit of Quanti-tation	Incl. Re-covery	Recovery Rate
				Day/Month	Sample A	Sample A	Sample A	Sample B	Sample B	Sample B		yes/no	in %
Ochratoxin A	ELISA	1	µg/kg	03.06.19	1,1	1,1	1,1	8,8	8,4	9,1	1		
Ochratoxin A	ELISA	3	µg/kg	06.06.19	7,975	6,4	9,55	8,8	9,05	8,55			
Ochratoxin A	ELISA	5	µg/kg	05/06	<1,5	<1,5	<1,5	7,11	7,17	7,06	1,5	no	
Ochratoxin A	ELISA	7	µg/kg	07.05.19	<1,25	<1,25	<1,25	4,05	4,05	4,05	1,25	no	
Ochratoxin A	ELISA	12	µg/kg	16.05.19	4,8	5,5	4,1	18,8	19,6	18	2	no	
Ochratoxin A	ELISA	15	µg/kg	6.6./7.6.		1,4	2		6,3	9,6			
Ochratoxin A	ELISA	16	µg/kg	05.06./ 07.06		3,7	2,6		7,4	5,1	0-25 ppb		
Ochratoxin A	HPLC	2	µg/kg	29.04. - 31.05.	<0,3	<0,3	<0,3	4,985	5,075	4,895	0,3	no	
Ochratoxin A	HPLC	4	µg/kg	05.06.19	0,89			7,06			0,1	yes	80
Ochratoxin A	HPLC	6	µg/kg	May/2019	< 0,50 µg/kg			5,7 µg/kg			< 0,50 µg/kg		
Ochratoxin A	LC-MS	10	µg/kg	14.05.19	<1	<1	<1	9,1	7,3	10,8	<1	yes	100
Ochratoxin A	LC-MS	13	µg/kg	07.05.19	0			8,1			0,4	yes	ISTD 13C
Ochratoxin A	LC-MS	14	µg/kg		<0.5			5,5	5,5	5,4	0,5	no	90

Parameter	Meth. Abr.	Partici-pant	Unit	Date of Analysis	Result (Mean)	Result I	Result II	Result (Mean)	Result I	Result II	Limit of Quanti-tation	Incl. Re-covery	Recovery Rate
				Day/Month	Sample A	Sample A	Sample A	Sample B	Sample B	Sample B		yes/no	in %
Deoxynivalenol	ELISA	1	µg/kg	04.06.19	838	838	838	29,2	26,7	31,6	18,5		
Deoxynivalenol	ELISA	2	µg/kg	29.04. - 31.05.	1,42			0,244				no	
Deoxynivalenol	ELISA	3	µg/kg	06.06.19	77,225	72,3	82,15	5,57	4,3	6,85			
Deoxynivalenol	ELISA	5	µg/kg	05/06	1190,5	1227	1154	<100	<100	<100	100	no	
Deoxynivalenol	ELISA	7	µg/kg	07.05.19	352,69	352,69	352,69	<18,50	<18,50	<18,50	15,5	no	
Deoxynivalenol	ELISA	8	µg/kg	29.05.19	670	663	676	222	222	222	222	—	—
Deoxynivalenol	ELISA	9	µg/kg	14.05.19	827	783	871	<600	<600	<600	600	yes	93 and 107
Deoxynivalenol	ELISA	12	µg/kg	16.05.19	1765	1610	1920	75,3	80,6	70	25 / 250	no	
Deoxynivalenol	ELISA	15	µg/kg	6.6./7.6.		798,21	852,54		100,43	139,7			
Deoxynivalenol	ELISA	16	µg/kg	05.06./ 07.06.		793,77	873,46		204,58	172,84	0-2 ppm		
Deoxynivalenol	LC-MS	4	µg/kg	05.06.19	718			<25			50	no	
Deoxynivalenol	LC-MS	6	µg/kg	May/2019	721 µg/kg			< 20 µg/kg			< 20 µg/kg		
Deoxynivalenol	LC-MS	10	µg/kg	14.05.19	430	410	440	<100	<100	<100	<100	yes	100
Deoxynivalenol	LC-MS	13	µg/kg	10.05.19	848			0			10	yes	ISTD 13C
Deoxynivalenol	LC-MS	14	µg/kg		716	684	748				20	no	113
Deoxynivalenol	div	11	µg/kg	14.05.19	507	501	512	12	19	5	102	yes	A=125; B=89

Parameter	Meth. Abr.	Partici-pant	Unit	Date of Analysis	Result (Mean)	Result I	Result II	Result (Mean)	Result I	Result II	Limit of Quanti-tation	Incl. Re-covery	Recovery Rate
				Day/Month	Sample A	Sample A	Sample A	Sample B	Sample B	Sample B		yes/no	in %
Fumonisin B1	HPLC	4	µg/kg	10.08.00	223			23,3			20	yes	99,9
Fumonisin B1	LC-MS	6	µg/kg	May/2019	149 µg/kg			< 20 µg/kg			< 20 µg/kg		
Fumonisin B1	LC-MS	13	µg/kg	07.05.19	293			0			20	yes	ISTD 13C
Fumonisin B1	LC-MS	14	µg/kg		80	84	75	<10			10	no	102
Fumonisin B2	HPLC	4	µg/kg	03.02.00	35			< 10			20	yes	94,1
Fumonisin B2	LC-MS	6	µg/kg	May/2019	34 µg/kg			< 20 µg/kg			< 20 µg/kg		
Fumonisin B2	LC-MS	13	µg/kg	07.05.19	47			0			12	yes	ISTD 13C
Fumonisin B2	LC-MS	14	µg/kg		36	40	32	16	16	15	10	no	92

Parameter	Meth. Abr.	Partici-pant	Unit	Date of Analysis	Result (Mean)	Result I	Result II	Result (Mean)	Result I	Result II	Limit of Quanti-tation	Incl. Re-covery	Recovery Rate
				Day/Month	Sample A	Sample A	Sample A	Sample B	Sample B	Sample B		yes/no	in %
Fumonisine Sum	ELISA	1	µg/kg	06.06.19	0,21	0,22	0,2	< BG	< BG	< BG	25		
Fumonisine Sum	ELISA	3	µg/kg	06.06.19	188,5	186,55	190,45	1,775	1,35	2,2			
Fumonisine Sum	ELISA	5	µg/kg	05/06	526,75	536	517,5	123,3	133,3	113,3	120	no	
Fumonisine Sum	ELISA	7	µg/kg	07.05.19	230	230	230	<25	<25	<25	<25	no	
Fumonisine Sum	ELISA	8	µg/kg	29.05.19	442	495	388	251	222	280	222	__	__
Fumonisine Sum	ELISA	12	µg/kg	16.05.19	200	202,7	197,3	< 50	< 50	< 50	50	no	
Fumonisine Sum	ELISA	15	µg/kg	6.6./7.6.		206,8	220,9		11,1	14,4			
Fumonisine Sum	ELISA	16	µg/kg	05.06./ 07.06		291	250,5		2,8	3,6	0-600 ppb		
Fumonisine Sum	HPLC	4	µg/kg	14.09.00	258			23,3					
Fumonisine Sum	LC-MS	6	µg/kg	May/2019	183 µg/kg			< 40 µg/kg			< 40 µg/kg		
Fumonisine Sum	LC-MS	13	µg/kg	07.05.19	340			0			32	yes	ISTD 13C
Fumonisine Sum	LC-MS	14	µg/kg		116	124	107	16	16	15	10	no	

Parameter	Meth. Abr.	Partici-pant	Unit	Date of Analysis	Result (Mean)	Result I	Result II	Result (Mean)	Result I	Result II	Limit of Quanti-tation	Incl. Re-covery	Recovery Rate
				Day/Month	Sample A	Sample A	Sample A	Sample B	Sample B	Sample B		yes/no	in %
Zearalenone	ELISA	1	µg/kg	05.06.19	73	70	75	< BG	< BG	< BG	1,75		
Zearalenone	ELISA	3	µg/kg	06.06.19	36,35	40,85	31,85	13,35	13,05	13,65			
Zearalenone	ELISA	5	µg/kg	05/06	47	49,9	44,05	14,5	14,1	15,03	7,5	no	
Zearalenone	ELISA	7	µg/kg	07.05.19	49,21	49,21	49,21	<1,75	<1,75	<1,75	<1,75	no	
Zearalenone	ELISA	8	µg/kg	29.05.19	62	65	59	50	50	50	50	__	__
Zearalenone	ELISA	12	µg/kg	16.05.19	44,9	41,5	48,4	< 25	< 25	< 25	25	no	
Zearalenone	ELISA	15	µg/kg	6.6./7.6.		71,6	73,2		13,3	14,8			
Zearalenone	ELISA	16	µg/kg	05.06./ 07.06		49	54,8		6	14,3	0-500 ppb		
Zearalenone	LC-MS	4	µg/kg	05.06.19	62,3			<5			10	no	
Zearalenone	LC-MS	6	µg/kg	May/2019	60 µg/kg			< 10 µg/kg			< 10 µg/kg		
Zearalenone	LC-MS	10	µg/kg	14.05.19	23	28	18	<10	<10	<10	<10	yes	100
Zearalenone	LC-MS	13	µg/kg	10.05.19	64			0			4	yes	ISTD 13C
Zearalenone	LC-MS	14	µg/kg		61	62	59				20	no	87

5.1.2 Analytical Methods

Parameter	Meth. Abr.	Partici-pant	Method description as in test report / norm / literature	Sample preparation	Measuring method	Calibration / Reference material	Recovery rate with same matrix	Method accredited ISO/IEC 17025	Further Remarks
							yes / no	yes / no	
Aflatoxin B1 - G2	ELISA	1							
Aflatoxin B1 - G2	ELISA	3	Elisa						
Aflatoxin B1 - G2	ELISA	5	quantitative ELISA	extraction with methanol 70%		standard solutions by the manufacturer	no	no	
Aflatoxin B1 - G2	ELISA	12							
Aflatoxin B1 - G2	ELISA	15	Elisa						
Aflatoxin B1 - G2	ELISA	16	Elisa		Testkit 8031 Veratox Aflatoxin HS from Neogen				
Aflatoxin B1 - G2	HPLC	2	In-house method, according to Aflaprep-Test method from R-Biopharm for detection of Aflatoxins by HPLC; IK0007	As per manufacturers instructions, Immunoaffinity columns Aflaprep, Art.-No.: RBRP07	HPLC	yes	no	yes	
Aflatoxin B1 - G2	HPLC	4	Aflatoxins in Cereals, Cereal products, selected Spices, Dried Fruits, Nuts, Oil Seeds, HPLC, 03-41-MAA-M-AFLA_HPLC, 2017-08	Extraction MeOH/H2O (80/20) + PBS-Buffer + IAC	HPLC-FLD, post column derivatisation	solvent calibration, no RM	yes	yes	Material had to be homogenized, because inhomogenous
Aflatoxin B1 - G2	HPLC	6	DIN EN ISO 16050 : 2011-09		HPLC-FLD			yes	
Aflatoxin B1 - G2	HPLC	7	ASU L 15.00-2:2014-02	-	-	-	-	yes	-
Aflatoxin B1 - G2	LC-MS	10	yes		LC-MS-MS	Standardaddition	yes	yes	
Aflatoxin B1 - G2	LC-MS	13	LC-MS/MS			Extern	ISTD 13C	yes	
Aflatoxin B1 - G2	LC-MS	14			LC-MS	Biopure	no	no	

Parameter	Meth. Abr.	Partici-pant	Method description as in test re-port / norm / literature	Sample preparation	Measuring method	Calibration / Refe-rence material	Recovery rate with same matrix	Method accredited ISO/IEC 17025	Further Remarks
							yes / no	yes / no	
Sum Aflatoxins	ELISA	1	§64 L 01.00-34					yes	
Sum Aflatoxins	ELISA	3							
Sum Aflatoxins	ELISA	5	quantitative ELISA	extraction with methanol 70%		standard solutions by the manufacturer	no	no	
Sum Aflatoxins	ELISA	12	Aflatoxin total HS ELISA #8031 (Neogen)	70% Methanol					
Sum Aflatoxins	ELISA	15							
Sum Aflatoxins	ELISA	16	Elisa		Testkit 8031 Veratox Aflatoxin HS from Neogen				
Sum Aflatoxins	HPLC	2	In-house method, according to Afla-prep-Test method from R-Biopharm for detection of Aflatoxins by HPLC; IK0007	As per manufacturers instruc-tions, Immunoaffinity columns Aflaprep, Art.-No.: RBRP07	HPLC	yes	no	yes	
Sum Aflatoxins	HPLC	4							
Sum Aflatoxins	HPLC	6	DIN EN ISO 16050 : 2011-09		HPLC-FLD			yes	
Sum Aflatoxins	HPLC	7	ASU L 15.00-2:2014-02	-	-	-	-	yes	-
Sum Aflatoxins	LC-MS	10	yes		LC-MS-MS	Standard addition	yes	yes	
Sum Aflatoxins	LC-MS	13	LC-MS/MS			Extern	ISTD 13C	yes	
Sum Aflatoxins	LC-MS	14			LC-MS	Biopure	no	no	

Parameter	Meth. Abr.	Partici-pant	Method description as in test re-port / norm / literature	Sample preparation	Measuring method	Calibration / Refe-rence material	Recovery rate with same matrix	Method accredited ISO/IEC 17025	Further Remarks
							yes / no	yes / no	
Ochratoxin A	ELISA	1	§64 L 01.00-34					yes	
Ochratoxin A	ELISA	3							
Ochratoxin A	ELISA	5	quantitative ELISA	extraction with methanol 70%		standard solutions by the manufacturer	no	no	
Ochratoxin A	ELISA	7	R-Biopharm, R1311:2009-10				nein	yes	
Ochratoxin A	ELISA	12	Veratox OTA ELISA #8610	70% Methanol					
Ochratoxin A	ELISA	15							
Ochratoxin A	ELISA	16	Elisa		Testkit 8610 Veratox Ochratoxin from N.				
Ochratoxin A	HPLC	2	modified according to §64 LFGB method L-46.02-5 (Januar 2010); IK0002	As per manufacturers instructions, Immunoaffinity columns Ochraprep, Art.-No.: RBRP14B	HPLC	yes	nein	yes	
Ochratoxin A	HPLC	4	Ochratoxin A in foodstuffs, HPLC, 03-41-MAA-M-OTA_CARB, 2017-08	Extraktion NaHCO3 + H2O + PBS-Buffer + IAC	HPLC-FLD, post column derivatisation	solvent calibration, no RM	yes	yes	
Ochratoxin A	HPLC	6	DIN EN ISO 16050 : 2011-09		HPLC-FLD			yes	
Ochratoxin A	LC-MS	10	yes		LC-MS-MS	Standard addition	yes	yes	
Ochratoxin A	LC-MS	13	LC-MS/MS			External	ISTD 13C	yes	
Ochratoxin A	LC-MS	14			LC-MS	Biopure	nein	nein	

Parameter	Meth. Abr.	Partici-pant	Method description as in test re-port / norm / literature	Sample preparation	Measuring method	Calibration / Refe-rence material	Recovery rate with same matrix	Method accredited ISO/IEC 17025	Further Remarks
							yes / no	yes / no	
Deoxynivalenol	ELISA	2	in-house method IK0124		ELISA	calibration yes, reference material no	no	no	
Deoxynivalenol	ELISA	3							
Deoxynivalenol	ELISA	5	quantitative ELISA	extraction with deionized water		standard solutions by the manufacturer	no	no	
Deoxynivalenol	ELISA	7	R-Biopharm, R5906:2009-06				no	yes	
Deoxynivalenol	ELISA	8	r-biopharm Fast-DON R5901	as per kit instructions	as per kit instructions	__	__	see Testkit	
Deoxynivalenol	ELISA	9	ELISA Method		fastDON r-biopharm	Bonner Enquete 2014	yes	yes	
Deoxynivalenol	ELISA	12	Veratox DON HS #8332 / Veratox DON 5/5 #8331NE	aqua dest.					
Deoxynivalenol	ELISA	15							
Deoxynivalenol	ELISA	16	Elisa		Testkit 8831 NE Veratox DON 5/5 from Neogen				
Deoxynivalenol	LC-MS	4	Fusarium toxins in cereals, cereal products and beer, LC-MS/MS, 03-41-MAA-M-DON_ZON, 2017-07	Extraction with ACN/H2O (84/16) + BondElut-SPE	LC-MS/MS	Matrix calibration (50-800)			
Deoxynivalenol	LC-MS	6	in-house method		HPLC-MS/MS			yes	
Deoxynivalenol	LC-MS	10	yes		LC-MS-MS	Standard addition	yes	yes	
Deoxynivalenol	LC-MS	13	LC-MS/MS			Extern	ISTD 13C	yes	
Deoxynivalenol	LC-MS	14			LC-MS	Biopure	no	no	
Deoxynivalenol	div	11	in-house method			Biopure	yes	yes	Sample B below our limit of detection

Parameter	Meth. Abr.	Partici-pant	Method description as in test re-port / norm / literature	Sample preparation	Measuring method	Calibration / Refe-rence material	Recovery rate with same matrix	Method accredited ISO/IEC 17025	Further Remarks
							yes / no	yes / no	
Fumonisin B1	HPLC	4	Fumonisins in cereals an cereal products, HPLC, 03-41-MAA-M-FUMO_HPLC, 2017-07	Extraction ACN/MeOH/H2O (25/25/50) + PBS-Buffer + IAC	HPLC-FLD, pre-column derivatisation	solvent calibration, no RM	yes	yes	
Fumonisin B1	LC-MS	6	in-house method		HPLC-MS/MS			yes	
Fumonisin B1	LC-MS	13	LC-MS/MS			Extern	ISTD 13C	yes	
Fumonisin B1	LC-MS	14			LC-MS	Biopure	no	no	
Fumonisin B2	HPLC	4							
Fumonisin B2	LC-MS	6	in-house method		HPLC-MS/MS			yes	
Fumonisin B2	LC-MS	13	LC-MS/MS			Extern	ISTD 13C	yes	
Fumonisin B2	LC-MS	14			LC-MS	Biopure	no	no	

Parameter	Meth. Abr.	Partici-pant	Method description as in test re-port / norm / literature	Sample preparation	Measuring method	Calibration / Refe-rence material	Recovery rate with same matrix	Method accredited ISO/IEC 17025	Further Remarks
							yes / no	yes / no	
Fumonisins Sum	ELISA	1	§64 L 01.00-34					yes	
Fumonisins Sum	ELISA	3							
Fumonisins Sum	ELISA	5	quantitative ELISA	extraction with methanol 70%		standard solutions by the manufacturer	no	no	
Fumonisins Sum	ELISA	7	R-Biopharm, R3401:2011-05				no	yes	
Fumonisins Sum	ELISA	8	r-biopharm Fast-FUM R5602	as per kit instructions	as per kit instructions	__	__	see Testkit	
Fumonisins Sum	ELISA	12	Veratox HS Fumonisin ELISA #8832	70% Methanol					
Fumonisins Sum	ELISA	15							
Fumonisins Sum	ELISA	16	Elisa		Testkit 8832 Veratox Fumonisin HS from Neogen				
Fumonisins Sum	HPLC	4							
Fumonisins Sum	LC-MS	6	in-house method		HPLC-MS/MS			yes	
Fumonisins Sum	LC-MS	13	LC-MS/MS			External	ISTD 13C	yes	
Fumonisins Sum	LC-MS	14							

Parameter	Meth. Abr.	Partici-pant	Method description as in test re-port / norm / literature	Sample preparation	Measuring method	Calibration / Refe-rence material	Recovery rate with same matrix	Method accredited ISO/IEC 17025	Further Remarks
							yes / no	yes / no	
Zearalenone	ELISA	1	§64 L 01.00-34					yes	
Zearalenone	ELISA	3							
Zearalenone	ELISA	5	quantitative ELISA	extraction with methanol 70%		standard solutions by the manufacturer	no	no	
Zearalenone	ELISA	7	R-Biopharm, R1401:2012-09				no	yes	
Zearalenone	ELISA	8	r-biopharm Fast-ZEA R5502	as per kit instructions	as per kit instructions	__	__	see Testkit	
Zearalenone	ELISA	12	Veratox Zearalenone ELISA #8110 (Neogen)	70% Methanol					
Zearalenone	ELISA	15							
Zearalenone	ELISA	16	Elisa		Testkit 8110 Veratox Zearalenone				
Zearalenone	LC-MS	4	Fusarium toxins in cereals, cereal products and beer, LC-MS/MS, 03-41-MAA-M-DON_ZON, 2017-07	Extraction with ACN/H2O (84/16) + BondElut-SPE	LC-MS/MS	Matrix calibration (5-80)			
Zearalenone	LC-MS	6	in-house method		HPLC-MS/MS			yes	
Zearalenone	LC-MS	10	yes		LC-MS-MS	Standarda ddition	yes	yes	
Zearalenone	LC-MS	13	LC-MS/MS			Extern	ISTD 13C	yes	
Zearalenone	LC-MS	14			LC-MS	Biopure	no	no	

5.2 Homogeneity

5.2.1 Mixture homogeneity before bottling

Microtracer Homogeneity Test
DLA ptMYS1-2019 Sample A

Weight whole sample	7,95	kg
Microtracer	FSS-rot lake	
Particle size	75 – 300	µm
Weight per particle	2,0	µg
Addition of tracer	15,6	mg/kg

Result of analysis

Sample	Weight [g]	Particle number	Particles [mg/kg]
1	5,02	42	16,7
2	5,00	44	17,6
3	5,09	41	16,1
4	5,00	38	15,2
5	5,05	40	15,8
6	5,06	39	15,4
7	5,05	38	15,0
8	5,06	36	14,2

Poisson distribution		
Number of samples	8	
Degree of freedom	7	
Mean	39,8	Particles
Standard deviation	2,65	Particles
χ^2 (CHI-Quadrat)	1,24	
Probability	**99**	%
Recovery rate	101	%

Normal distribution		
Number of samples	8	
Mean	15,8	mg/kg
Standard deviation	1,05	mg/kg
rel. Standard deviaton	6,67	%
Horwitz standard deviation	10,6	%
HorRat-value	**0,63**	
Recovery rate	101	%

Microtracer Homogeneity Test
DLA ptMYS1-2109 Sample B

Weight whole sample	8,01	kg
Microtracer	FSS-rot lake	
Particle size	75 – 300	µm
Weight per particle	2,0	µg
Addition of tracer	15,0	mg/kg

Result of analysis

Sample	Einwaage [g]	Partikel Anzahl	Partikel [mg/kg]
1	5,04	54	21,4
2	4,98	43	17,3
3	5,03	52	20,7
4	4,98	43	17,3
5	5,01	44	17,6
6	5,03	48	19,1
7	5,00	48	19,2
8	5,00	48	19,2

Poisson distribution		
Number of samples	8	
Degree of freedom	7	
Mean	47,5	Particles
Standard deviation	3,88	Particles
χ^2 (CHI-Quadrat)	2,22	
Probability	**95**	%
Recovery rate	126	%

Normal distribution		
Number of samples	8	
Mean	19,0	mg/kg
Standard deviation	1,55	mg/kg
rel. Standard deviaton	8,18	%
Horwitz standard deviation	10,3	%
HorRat-value	**0,80**	
Recovery rate	126	%

5.3 Information on the Proficiency Test (PT)

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

PT number	***ptMYS1***
PT name	***Mycotoxin-Screening: Aflatoxins, Ochratoxin A, Deoxynivalenol, Zearalenon and Fumonisins in Breakfast Cereals***
*Sample matrix**	***Samples A + B:*** *Cereal muesli with fruits / Ingredients: Oat wholemeal flakes, raisins oiled, rice puffed, dried fruits (apricots, dates, plums, apples), rice flour, cinnamon and other ingredients from corn, almonds, pistachios and plant powder*
Number of samples and sample amount	*2 different samples A + B: 200 g each (2x100g each).*
Storage	*Samples A + B: cooled 2 - 10°C*
Intentional use	*Laboratory use only (quality control samples)*
Parameter	*Quantitative+ qualitative: Aflatoxins (< 50 µg/kg), Ochratoxin A (< 100 µg/kg), Deoxynivalenol (< 1500 µg/kg), Zearalenon (< 500 µg/kg) and Fumonisins (< 1000 µg/kg)*
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 final results for sample A and B should be filled in the result submission file. The specification of individual results from a double determination can be made additionally. The recovery rates, if carried out, has to be included in the calculation.*
Units	*µg/kg*
Number of significant digits	*at least 2*
Further information	*For information please specify:* – *Date of analysis* – *Final results for sample A and B* – *Limit of detection* – *Assignment incl. Recovery* – *Recovery with the same matrix* – *Method is accredited*
Result submission	*The result submission file should be sent by e-mail to:* ***pt@dla-lvu.de***
Deadline	***the latest 07th June 2019***
Evaluation report	*The evaluation report is expected to be completed 6 weeks after deadline of result submission and sent as PDF file by e-mail.*
Coordinator and contact person of PT	*Matthias Besler-Scharf PhD*

* Control of mixture homogeneity and qualitative testings are carried out by DLA. Any testing of the content, homogeneity and stability of PT parameters is subcontracted by DLA.

6. Index of participant laboratories in alphabetical order

Teilnehmer / Participant	Ort / Town	Land / Country
		SWITZERLAND
		Germany
		Germany
		Germany
		Germany
		Germany
		SWITZERLAND
		Germany
		Germany
		Germany
		HUNGARY
		Germany
		Germany
		Germany

[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

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immunoaffinity column cleanup

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