Evaluation Report
proficiency test

DLA ptMYS1 (2020)

Mycotoxin-Screening:

Aflatoxins, Ochratoxin A, Deoxynivalenol, Zearalenone and Fumonisins

in Breakfast Cereals (Muesli)

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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 contain customary breakfast cereals "mueslie" from European suppliers. 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 1,5 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 to sample B 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 Ingredients: Oatmeal flakes, sugared cranberries, dried fruits (strawberries, raspberries, black currants, bananas, oranges), lemon juice concentrate, maltodextrin, whey powder, cereal flours (wheat, rice, oats, millet, barley, rye, corn), skimmed milk powder, vegetable fat, emulsifier: lecithins, cornflakes, vitamins, minerals Nutrients** per 100 g: Fat 7,5 g, carbohydrates 62 g therof sugar 7,6 g, fiber 8,5 g, protein 13 g, salt <0,1 g	80,0 g/100 g	88,2 g/100g
Maize, ground	20,0 g/100g	-
Almond flour, partially de-oiled	-	5,85 g/100g
Plant powder mixture	-	3,87 g/100g
Pistachio-almond mixture, ground	-	2,13 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 76% and 98%. Additionally particle number results were converted into concentrations, statistically evaluated according to normal distribution and compared to the standard deviation according to Horwitz. For the assessment HorRat values between 0,3 and 1,3 are to be accepted under repeat conditions (measurements within the laboratory) [17].
This gave a HorRat value of 1,0 and 0,6 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)	-	10,3 %
Aflatoxins Sum (AF Sum)	-	11,7 %
Ochratoxin A (OTA)	-	11,1 %
Deoxynivalenol (DON)	5,4 %	-
Fumonisins Sum (FUMO Sum)	16,3 %	-
Zearalenone (ZON)	17,6 %	-

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. 0,48 and 0,41 (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 2020. The testing method was optional. The tests should be finished at 3rd July 2020 the latest (extended).

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.*
Note: Please store samples at 2 - 10°C on arrival!

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

2.3 Submission of results

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

All 16 participants submitted their results in time.

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

3.2.5 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 the parameter below the target standard deviation from the general model of Horwitz, suitable for levels ≥ 120 µg/kg, was applied (s. 3.2.6.2):

- Fumonisins (ELISA results)

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

For information the target standard deviation from the general model of Horwitz, suitable for levels ≥ 120 µg/kg, was given additionally for the parameters Deoxynivalenol and Fumonisins (all results) (s. 3.2.6.2).

For the parameter Fumonisins (ELISA results) the standard uncertainty was considered evaluating the results by z'-scores (s. 3.2.6.8).

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] (AF = Aflatoxin, OTA = Ochratoxin, DON = Deoxynivalenol, FUMO = Fumonisins, ZON = Zearalenone)

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
ZON	Bread (including small bakery wares), pastries, biscuits, cereal snacks and breakfast cereals, excluding maize snacks and maize based breakfast cereals	100	25 [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^*)
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		
3	negative	<0,7	positive	3,65	2/2 (100%)	ELISA	
7		1,90		7,35		ELISA	not rated (sum of aflatoxins?)
8		1,40		6,05		ELISA	not rated (sum of aflatoxins?)
9		1,45		6,50		ELISA	not rated (sum of aflatoxins?)
10	negative	<1	positive	3,41	2/2 (100%)	ELISA	
14	negative	< 1	positive	4,70	2/2 (100%)	ELISA	
16	negative	<1	positive	5,03	2/2 (100%)	ELISA	
2	negative	<0,01	positive	3,41	2/2 (100%)	HPLC	
11	negative	<0,1	positive	3,52	2/2 (100%)	HPLC	
1	negative	<0,5	positive	5,57	2/2 (100%)	LC/MS	
5	negative	<0,2	positive	4,51	2/2 (100%)	LC/MS	
6	negative	<0,2	positive	5,10	2/2 (100%)	LC/MS	
15	negative	<LOQ	positive	7,47	1/2 (50%)	LC/MS	LOQ (2 µg/kg) is > acceptance level

	Sample A		Sample B	
Number positive	0		10	
Number negative	10		0	
Percent positive	0		100	
Percent negative	100		0	
Consensus value	negative		positive	

Methods:
w eitere Angaben s. Dokumentation
further details see documentation

positive: > 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	All Methods	LC-Methods
Number of results	10	6
Number of outliers	0	0
Mean	4,64	4,93
Median	4,61	4,81
Robust Mean (X_{pt})	**4,51**	**4,93**
Robust standard deviation (S*)	**1,12**	**1,71**
Number with 2 replicates	8	5
Repeatability SD (S_r)	0,496	0,606
Repeatability (CV_r)	10,3%	12,1%
Reproducibility SD (S_R)	1,39	1,72
Reproducibility (CV_R)	28,9%	34,3%
Target range:		
Target standard deviation σ_{pt}	**0,992**	**1,08**
Target standard deviation (for Information)	0,839	0,917
lower limit of target range	**2,52**	**2,76**
upper limit of target range	**6,49**	**7,10**
Quotient S/σ_{pt}*	*1,1*	*1,6*
Standard uncertainty $U_{(Xpt)}$	*0,441*	*0,873*
Results in the target range	*9*	*5*
Percent in the target range	*90%*	*83%*

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 and coefficients of variation *CV_r and CV_R* are in the range of established values of the applied methods (see 3.2.6.2).

90% of results of all methods and 83% of LC-methods were in the target range.

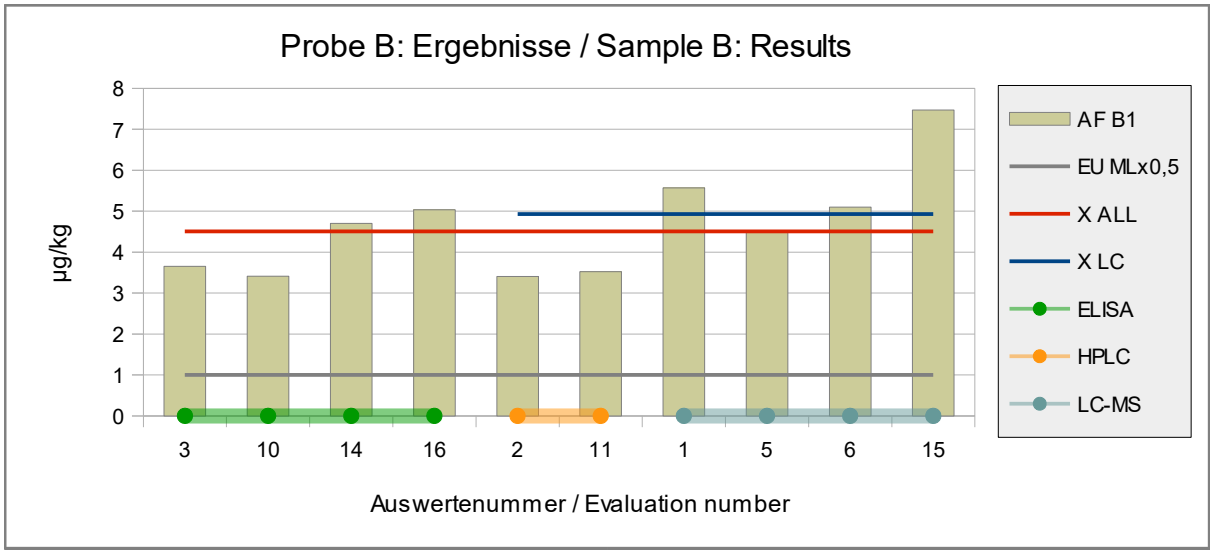

Abb./Fig. 1: Results Aflatoxin B1 (AF B1)
red line = Assigned value robust mean results all methods
blue line = Assigned value robust mean results LC methods
grey line = Qual. valuation as positive > 1,0 µg/kg
round symbols = Applied methods (see legend)

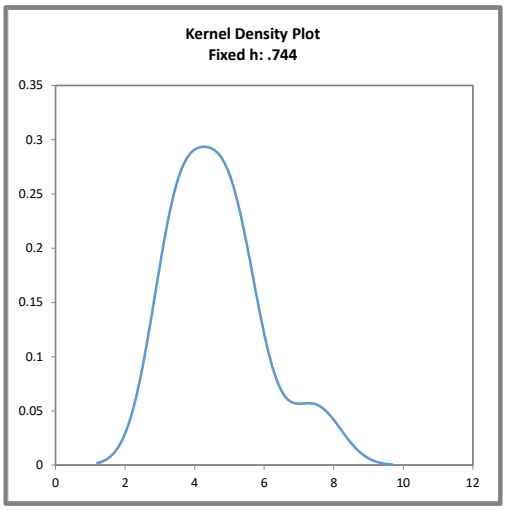

Abb. / Fig. 2:
Kerndichte-Schätzung aller ELISA-Ergebnisse (mit h = 0,75 x σpt von Xpt_{ALL})

Kernel density plot of all ELISA 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 side peak at 7,5 µg/kg.

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

Evaluation number	Sample B	Deviation	z-Score Xpt_{ALL}	Deviation	z-Score Xpt_{LC}	Method	Remarks
	[µg/kg]	X All		X LC			
3	3,65	-0,86	-0,87			ELISA	
10	3,41	-1,10	-1,1			ELISA	
14	4,70	0,19	0,19			ELISA	
16	5,03	0,52	0,53			ELISA	
2	3,41	-1,10	-1,1	-1,52	-1,4	HPLC	
11	3,52	-0,99	-1,0	-1,41	-1,3	HPLC	
1	5,57	1,06	1,1	0,64	0,59	LC/MS	
5	4,51	0,00	0,00	-0,42	-0,39	LC/MS	
6	5,10	0,59	0,60	0,17	0,16	LC/MS	
15	7,47	2,96	3,0	2,54	2,3	LC/MS	

Methods:
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further details see documentation

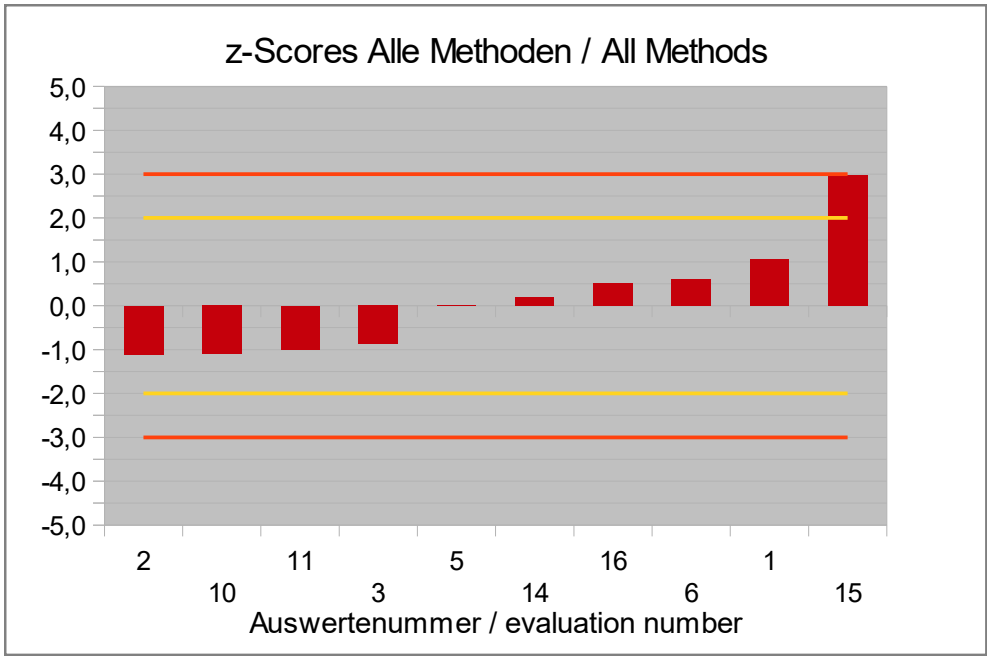

Abb./Fig. 3:
z-Scores Aflatoxin B1 (AF B1)
Assigned value robust mean results all methods

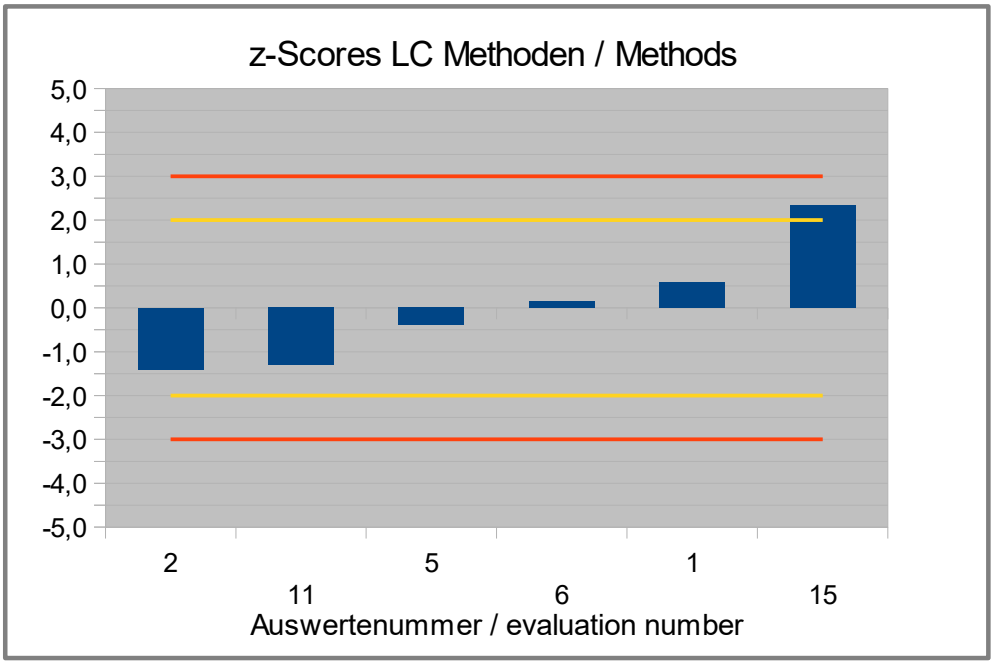

Abb./Fig. 4:
z-Scores Aflatoxin B1 (AF B1)
Assigned value robust mean results LC methods

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		
3	negative	<1	positive	4,55	2/2 (100%)	ELISA	
4	negative	<1	positive	3,00	2/2 (100%)	ELISA	
7	negative	1,90	positive	7,35	2/2 (100%)	ELISA	Mean of sample B calculated by DLA
8	negative	1,40	positive	6,05	2/2 (100%)	ELISA	
9	negative	1,45	positive	6,50	2/2 (100%)	ELISA	
10	negative	<1,75	positive	2,92	2/2 (100%)	ELISA	
13	negative	1,38	positive	4,31	2/2 (100%)	ELISA	
14	negative	< 1	positive	4,70	2/2 (100%)	ELISA	AF1 result taken as AF Sum by DLA
16	negative	<1	positive	5,03	2/2 (100%)	ELISA	AF1 result taken as AF Sum by DLA
2	negative	0	positive	3,86	2/2 (100%)	HPLC	Sum calculated by DLA
11	negative	<0,1	positive	3,78	2/2 (100%)	HPLC	
1	negative		positive	5,57	2/2 (100%)	LC/MS	
5	negative	<0,2	positive	5,42	2/2 (100%)	LC/MS	
6	negative	<0,2	positive	5,90	2/2 (100%)	LC/MS	
15		<LOQ	positive	8,27	2/2 (100%)	LC/MS	LOQ (11 µg/kg) is > acceptance level

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

Methods:
w eitere Angaben s. Dokumentation
further details see documentation

positive: > 2 µg/kg (EU maximum level x 0,5)
negative: < 2 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, with one exception 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	15	9	6
Number of outliers	0	0	0
Mean	5,15	4,93	5,47
Median	5,03	4,70	5,50
Robust Mean (x_{pt})	**5,10**	**4,93**	**5,46**
Robust standard deviation (S*)	**1,61**	**1,70**	**1,85**
Number with 2 replicates	11	8	4
Repeatability SD (S_r)	0,654	0,581	0,711
Repeatability (CV_r)	11,7%	11,2%	11,2%
Reproducibility SD (S_R)	1,56	1,44	1,41
Reproducibility (CV_R)	27,9%	27,8%	22,3%
Target range:			
Target standard deviation σ_{pt}	**1,12**	**1,09**	**1,20**
Target standard deviation (for Information)	0,968	0,937	1,04
lower limit of target range	**2,85**	**2,76**	**3,06**
upper limit of target range	**7,34**	**7,11**	**7,86**
Quotient S/σ_{pt}*	*1,4*	*1,6*	*1,5*
Standard uncertainty $U_{(Xpt)}$	*0,520*	*0,707*	*0,942*
Results in the target range	*14*	*8*	*5*
Percent in the target range	*93%*	*89%*	*83%*

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.

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

93% of results of all methods, 89% of ELISA-methods and 83% of LC-methods were in the target range.

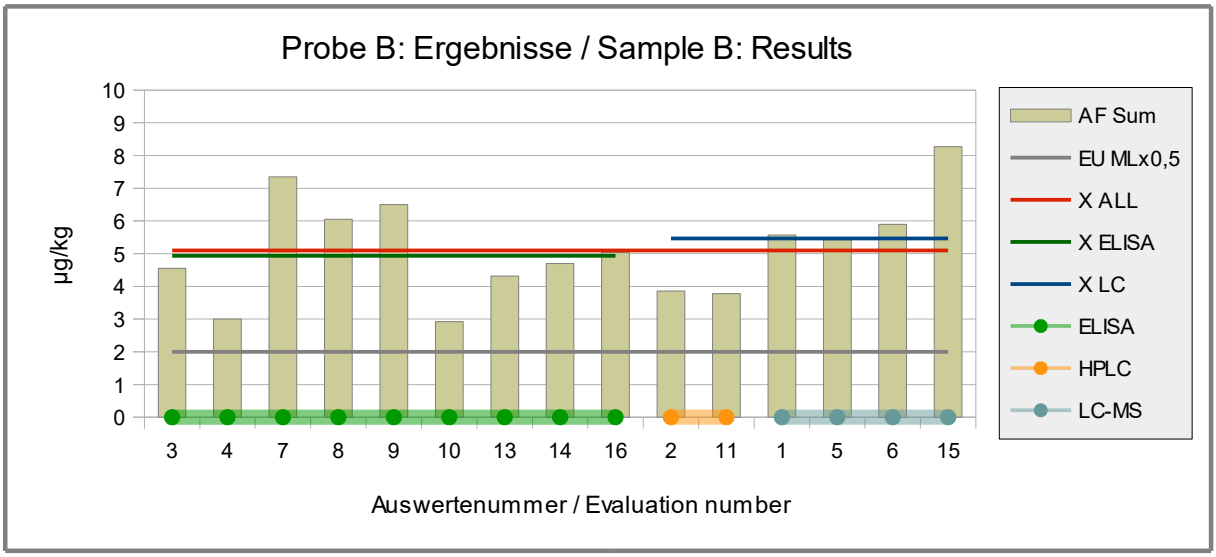

Abb./Fig. 5: Results Aflatoxins Sum (AF Sum)
red line = Assigned value robust mean results all methods
green line = Assigned value robust mean results ELSIA methods
blue line = Assigned value robust mean results LC methods
grey line = Qual. valuation as positive > 2,0 µg/kg
round symbols = Applied methods (see legend)

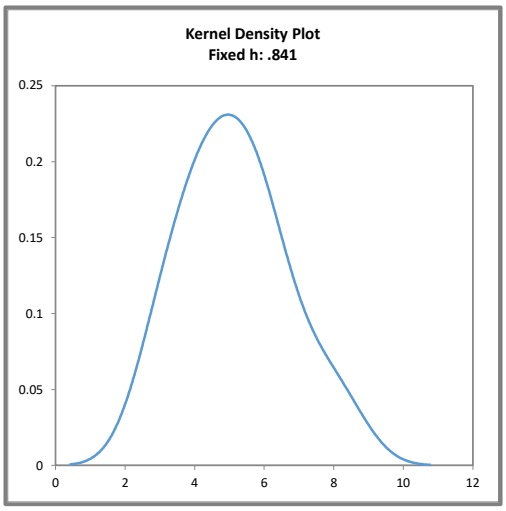

Abb. / Fig. 6:
Kerndichte-Schätzung aller Ergebnisse
(mit h = 0,75 x σ_{pt} von Xpt_{ALL})

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 slight shoulder at approx. 8 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			
3	4,55	-0,55	-0,49	-0,38	-0,35			ELISA	
4	3,00	-2,10	-1,9	-1,93	-1,8			ELISA	
7	7,35	2,25	2,0	2,42	2,2			ELISA	Mean of sample B calculated by DLA
8	6,05	0,95	0,85	1,12	1,0			ELISA	
9	6,50	1,40	1,3	1,57	1,4			ELISA	
10	2,92	-2,18	-1,9	-2,01	-1,9			ELISA	
13	4,31	-0,78	-0,70	-0,62	-0,57			ELISA	
14	4,70	-0,40	-0,35	-0,23	-0,22			ELISA	AF1 result taken as AF Sum by DLA
16	5,03	-0,07	-0,06	0,10	0,09			ELISA	AF1 result taken as AF Sum by DLA
2	3,86	-1,24	-1,1			-1,60	-1,3	HPLC	Sum calculated by DLA
11	3,78	-1,32	-1,2			-1,68	-1,4	HPLC	
1	5,57	0,47	0,42			0,11	0,09	LC/MS	
5	5,42	0,32	0,29			-0,04	-0,03	LC/MS	
6	5,90	0,80	0,72			0,44	0,37	LC/MS	
15	8,27	3,17	2,8			2,8	2,3	LC/MS	

Methoden:
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further details see documentation

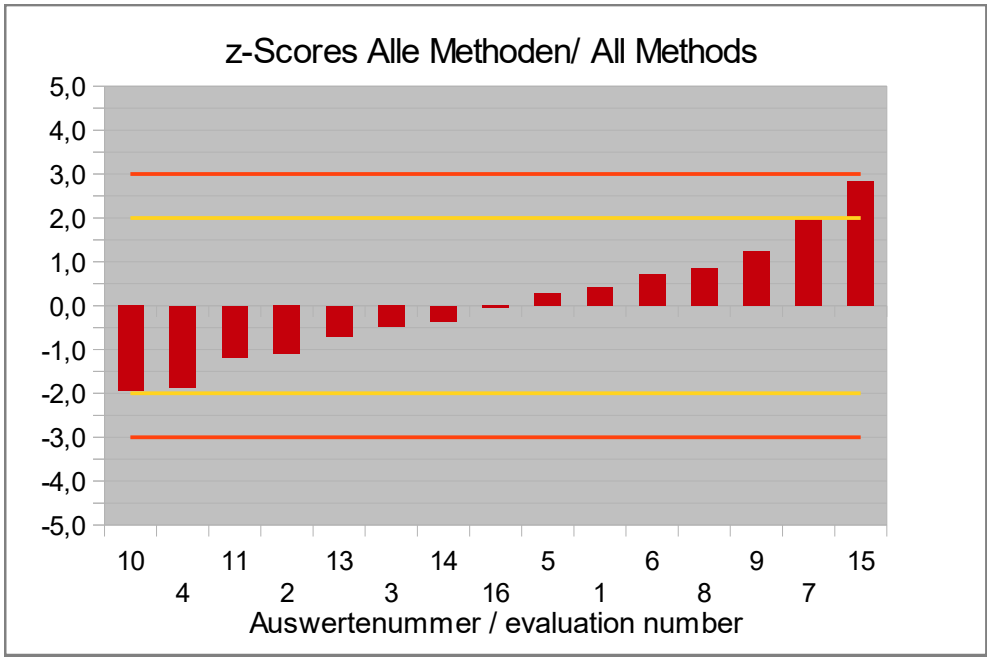

Abb./Fig. 7:
z-Scores Aflatoxins Sum (AF Sum)
Assigned value robust mean results all methods

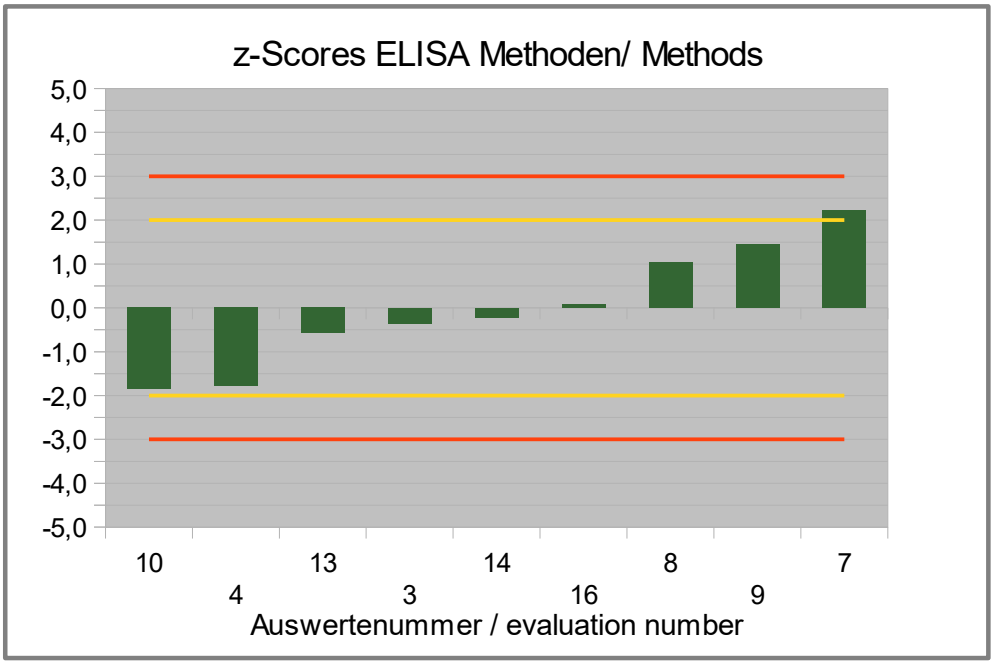

Abb./Fig. 8:
z-Scores Aflatoxins Sum (AF Sum)
Assigned value robust mean results ELISA methods

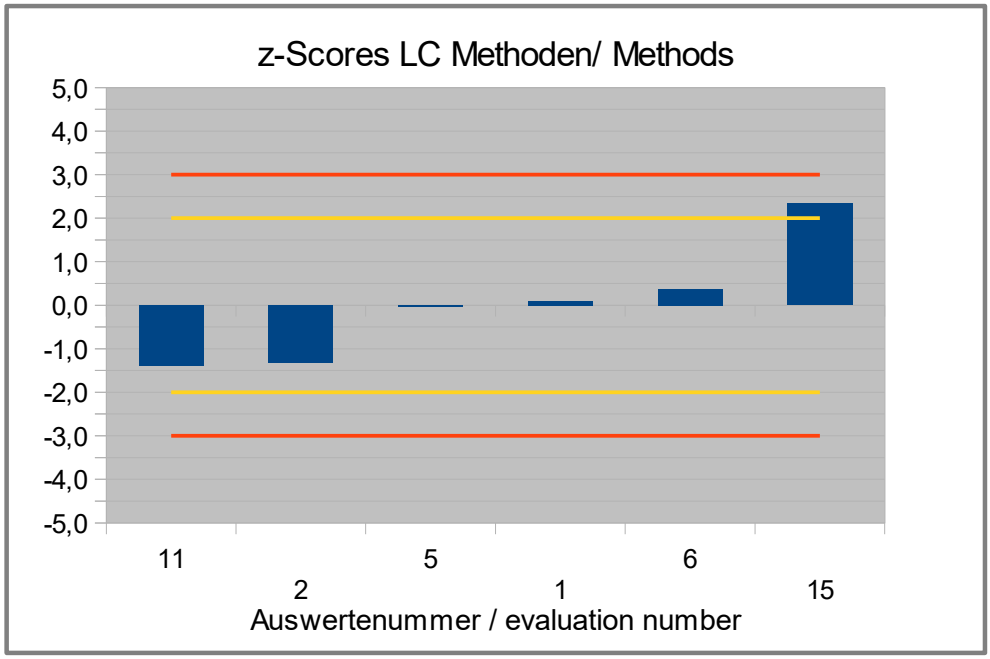

Abb./Fig. 9:
z-Scores Aflatoxins Sum (AF Sum)
Assigned value robust mean results LC methods

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		
3	negative	<1,5	positive	9,31	2/2 (100%)	ELISA	
4	-	<2	positive	5,60	1/2 (50%)	ELISA	LOQ (2 µg/kg) is > acceptance level
7	positive	2,40	positive	9,95	1/2 (50%)	ELISA	Mean of sample B calculated by DLA
8	positive	1,55	positive	6,30	1/2 (50%)	ELISA	
9	positive	2,90	positive	8,35	1/2 (50%)	ELISA	
10	positive	2,10	positive	14,4	1/2 (50%)	ELISA	
14	negative	< 1,25	positive	11,0	2/2 (100%)	ELISA	
2	negative	<0,03	positive	8,58	2/2 (100%)	HPLC	
11	negative	<0,1	positive	9,04	2/2 (100%)	HPLC	
1	negative	<0,5	positive	12,4	2/2 (100%)	LC/MS	
5	negative	<1	positive	10,0	2/2 (100%)	LC/MS	
6	negative	<0,5	positive	11,3	2/2 (100%)	LC/MS	
15	-	<BG	positive	12,3	1/2 (50%)	LC/MS	LOQ (10 µg/kg) is > acceptance level

	Sample A		Sample B	
Number positive	4		13	
Number negative	7		0	
Percent positive	36		100	
Percent negative	64		0	
Consensus value	none		positive	

Methods:
w eitere Angaben s. Dokumentation
further details see documentation

positive: > 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 with respect to all methods. The LC methods gave mostly negative and the ELISA methods mostly positive results.

Quantative valuation: Ochratoxin A in µg/kg

Sample B

Statistic Data	All Methods	ELISA-Methods	LC-Methods
Number of results	13	7	6
Number of outliers	0	0	0
Mean	9,88	9,27	10,6
Median	10,0	9,31	10,7
Robust Mean (x_{pt})	**9,86**	**9,24**	**10,6**
Robust standard deviation (S*)	**2,62**	**3,30**	**1,85**
Number with 2 replicates	12	7	5
Repeatability SD (S_r)	1,09	1,41	0,249
Repeatability (CV_r)	11,1%	15,4%	2,33%
Reproducibility SD (S_R)	2,65	3,09	1,80
Reproducibility (CV_R)	27,0%	33,6%	16,8%
Target range:			
Target standard deviation σ_{pt}	**2,43**	**2,03**	**2,33**
Target standard deviation (for Information)	2,17	2,27	2,61
lower limit of target range	**5,01**	**5,18**	**5,94**
upper limit of target range	**14,7**	**13,3**	**15,3**
Quotient S/σ_{pt}*	*1,1*	*1,6*	*0,80*
Standard uncertainty $U_{(Xpt)}$	*0,909*	*1,56*	*0,946*
Results in the target range	*13*	*6*	*6*
Percent in the target range	*100%*	*86%*	*100%*

Comments to the statistical characteristics:

For evaluation of the results of all methods the target standard deviation was calculated using data from a precision experiment (3.2.6.2). For evaluation of the results of the ELISA and LC methods the target standard deviation was calculated according to the general model of Horwitz/Thompson (3.2.6.1). In addition, the target standard deviation of the other model was given for information.

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

The repeatability and reproducibility standard deviation and coefficients of variation *CV_r and CV_R* are in the range of established values of the applied methods (see 3.2.6.2).
100% of results of all methods, 86% of ELISA-methods and 100% of LC-methods were in the target range.

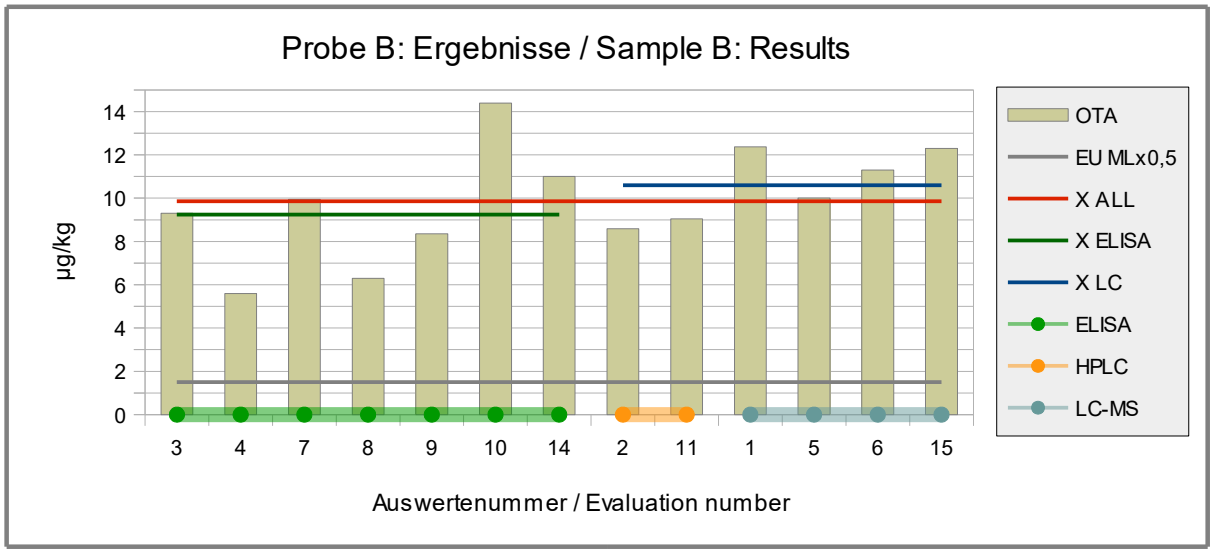

Abb./Fig. 10: Results Ochratoxin A (OTA)
red line = Assigned value robust mean results all methods
green line = Assigned value robust mean results ELSIA methods
blue line = Assigned value robust mean results LC methods
grey line = Qual. valuation as positive > 1,5 µg/kg
round symbols = Applied methods (see legend)

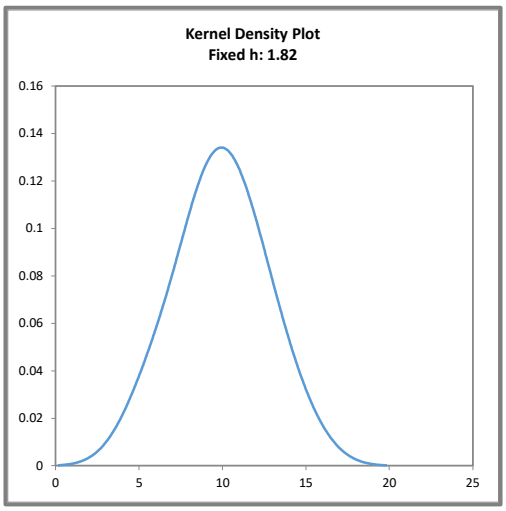

Abb. / Fig. 11:
Kerndichte-Schätzung aller Ergebnisse
(mit h = 0,75 x σpt von Xpt_{ALL})

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.

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 Alle		X ELISA		X LC			
3	9,31	-0,56	-0,23	0,06	0,03			ELISA	
4	5,60	-4,26	-1,8	-3,64	-1,8			ELISA	
7	9,95	0,09	0,04	0,71	0,35			ELISA	Mean of sample B calculated by DLA
8	6,30	-3,56	-1,5	-2,94	-1,4			ELISA	
9	8,35	-1,51	-0,62	-0,89	-0,44			ELISA	
10	14,4	4,54	1,9	5,16	2,5			ELISA	
14	11,0	1,14	0,47	1,76	0,86			ELISA	
2	8,58	-1,28	-0,53			-2,02	-0,86	HPLC	
11	9,04	-0,82	-0,34			-1,56	-0,67	HPLC	
1	12,4	2,51	1,0			1,77	0,76	LC/MS	
5	10,0	0,14	0,06			-0,60	-0,26	LC/MS	
6	11,3	1,44	0,59			0,70	0,30	LC/MS	
15	12,3	2,44	1,0			1,70	0,73	LC/MS	

Methods:
weitere Angaben s. Dokumentation
further details see documentation

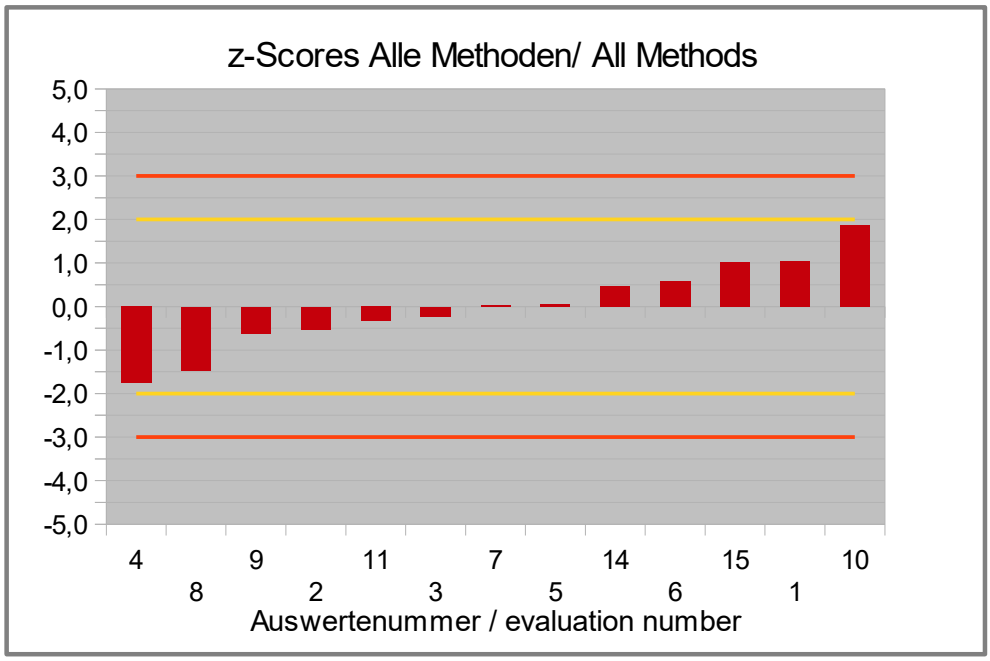

Abb./Fig. 12:
z-Scores Ochratoxin A (OTA)
Assigned value robust mean results all methods

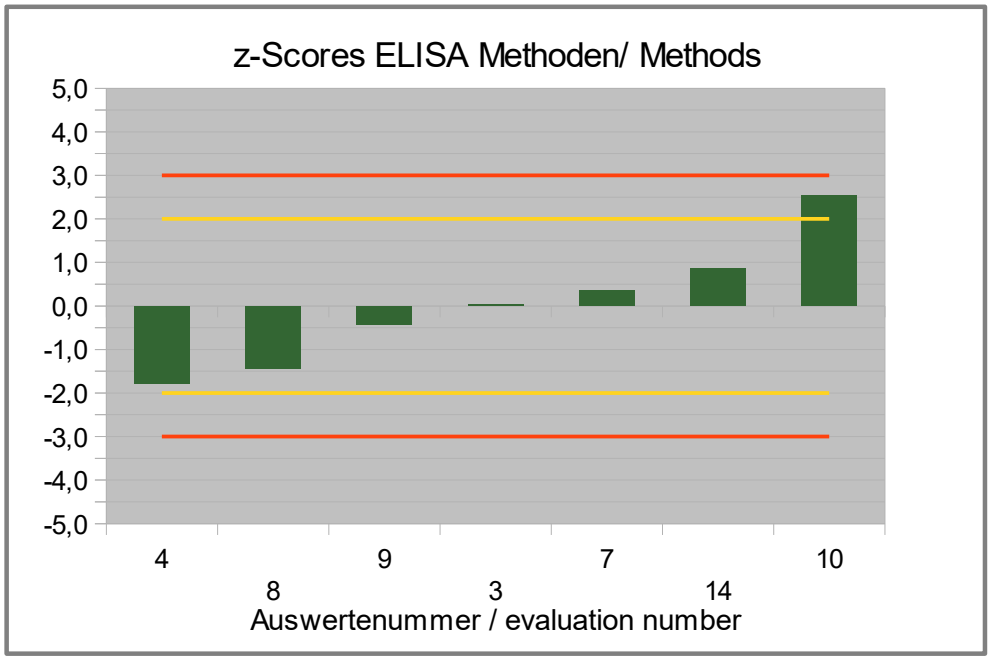

Abb./Fig. 13:
z-Scores Ochratoxin A (OTA)
Assigned value robust mean results ELISA methods

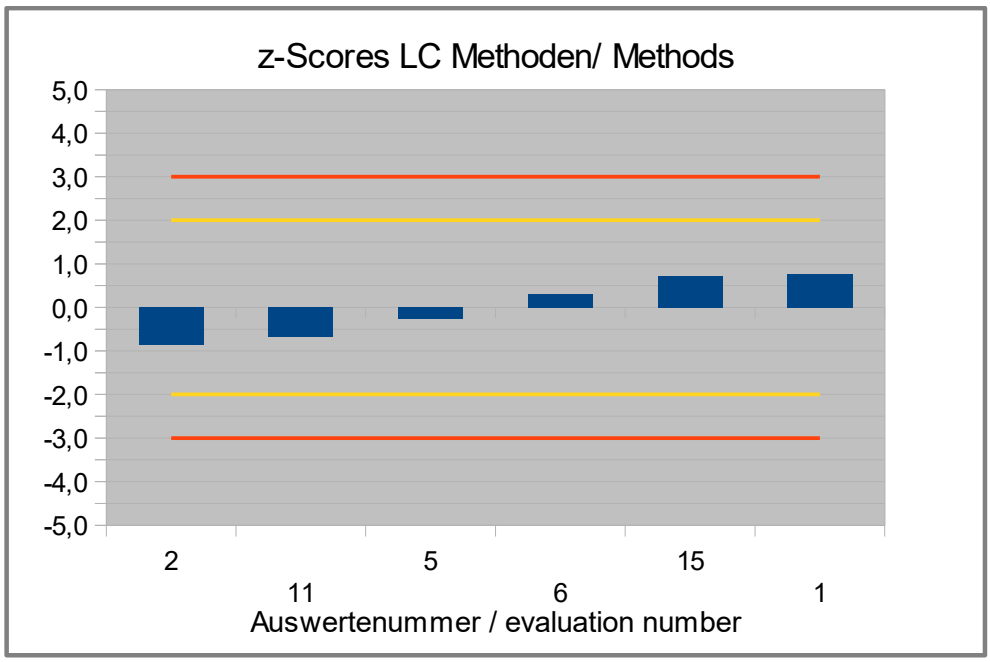

Abb./Fig. 14:
z-Scores Ochratoxin A (OTA)
Assigned value robust mean results LC methods

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		
3	positive	1194	negative	<100	2/2 (100%)	ELISA	
4	positive	780	negative	<250	2/2 (100%)	ELISA	
9	positive	926	negative	101	2/2 (100%)	ELISA	
10	positive	565	negative	<18,5	2/2 (100%)	ELISA	
12	positive	878	negative	<222	2/2 (100%)	ELISA	
13	positive	1294	negative	90,6	2/2 (100%)	ELISA	
14	positive	752	negative	< 30	2/2 (100%)	ELISA	
16	positive	643	negative	<40	2/2 (100%)	ELISA	
2	positive	758	negative	<40	2/2 (100%)	HPLC	
11	positive	613	negative	<50	2/2 (100%)	HPLC	
1	positive	1154	negative	187	2/2 (100%)	LC/MS	
6	positive	967	negative	22	2/2 (100%)	LC/MS	
15	positive	966	negative	<BG	2/2 (100%)	LC/MS	

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

Methods:
w eitere Angaben s. Dokumentation
further details see documentation

positive: > 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 B all results were below and for sample A all results above the acceptance level.

Quantative valuation: Deoxynivalenol 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	884	879	892
Median	878	829	966
Robust Mean (X_{pt})	**882**	**879**	**892**
Robust standard deviation (S*)	**255**	**289**	**238**
Number with 2 replicates	13	8	5
Repeatability SD (S_r)	47,6	58,5	20,6
Repeatability (CV_r)	5,39%	6,65%	2,31%
Reproducibility SD (S_R)	231	258	210
Reproducibility (CV_R)	26,2%	29,3%	23,6%
Target range:			
Target standard deviation σ_{pt}	**193**	**192**	**195**
Target standard deviation (for Information)	144	143	145
lower limit of target range	**496**	**494**	**501**
upper limit of target range	**1270**	**1260**	**1280**
Quotient S/σ_{pt}*	*1,3*	*1,5*	*1,2*
Standard uncertainty $U_{(Xpt)}$	*88,4*	*128*	*133*
Results in the target range	*12*	*7*	*5*
Percent in the target range	*92%*	*88%*	*100%*

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 and coefficients of variation *CV_r and CV_R* are in the range of established values of the applied methods (see 3.2.6.2).

92% of results of all methods, 88% of ELISA-methods and 100% of LC-methods were in the target range.

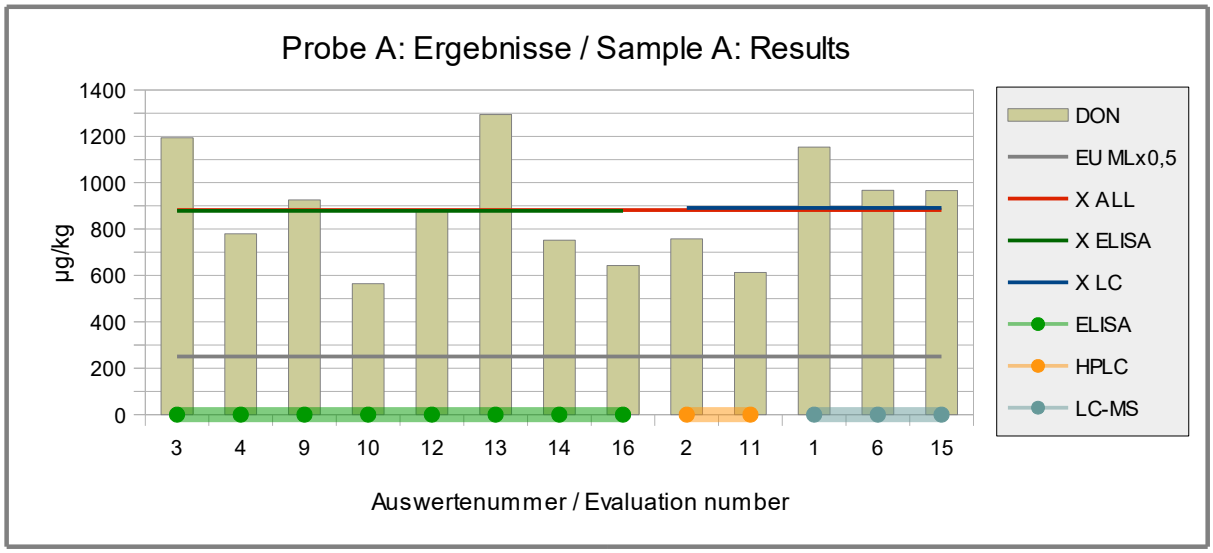

Abb./Fig. 15: Results Deoxynivalenol (DON)
red line = Assigned value robust mean results all methods
green line = Assigned value robust mean results ELSIA methods
blue line = Assigned value robust mean results LC methods
grey line = Qual. valuation as positive > 250 µg/kg
round symbols = Applied methods (see legend)

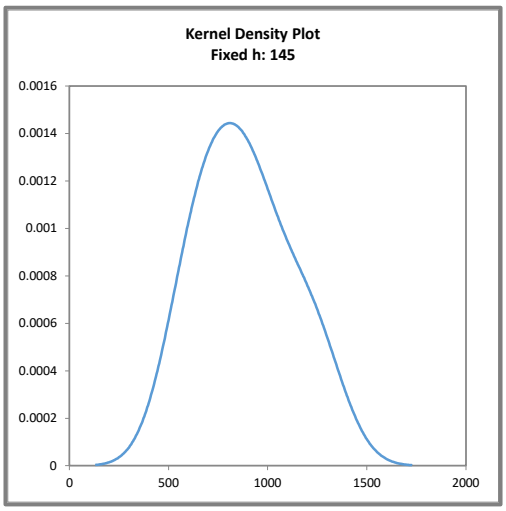

Abb. / Fig. 16:
Kerndichte-Schätzung aller Ergebnisse
(mit h = 0,75 x σ_{pt} von Xpt_{ALL})

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 slight at > 1100 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			
3	1194	312,8	1,6	315,4	1,6			ELISA	
4	780	-101,5	-0,53	-98,9	-0,51			ELISA	
9	926	44,6	0,23	47,1	0,25			ELISA	
10	565	-316,5	-1,6	-313,9	-1,6			ELISA	
12	878	-3,5	-0,02	-0,9	0,00			ELISA	
13	1294	412,0	2,1	414,6	2,2			ELISA	
14	752	-129,5	-0,67	-126,9	-0,66			ELISA	
16	643	-238,9	-1,2	-236,3	-1,2			ELISA	
2	758	-123,8	-0,64			-133,9	-0,69	HPLC	
11	613	-268,5	-1,4			-278,5	-1,4	HPLC	
1	1154	272,5	1,4			262,5	1,4	LC/MS	
6	967	85,5	0,44			75,5	0,39	LC/MS	
15	966	84,5	0,44			74,5	0,38	LC/MS	

Methods:
weitere Angaben s. Dokumentation
further details see documentation

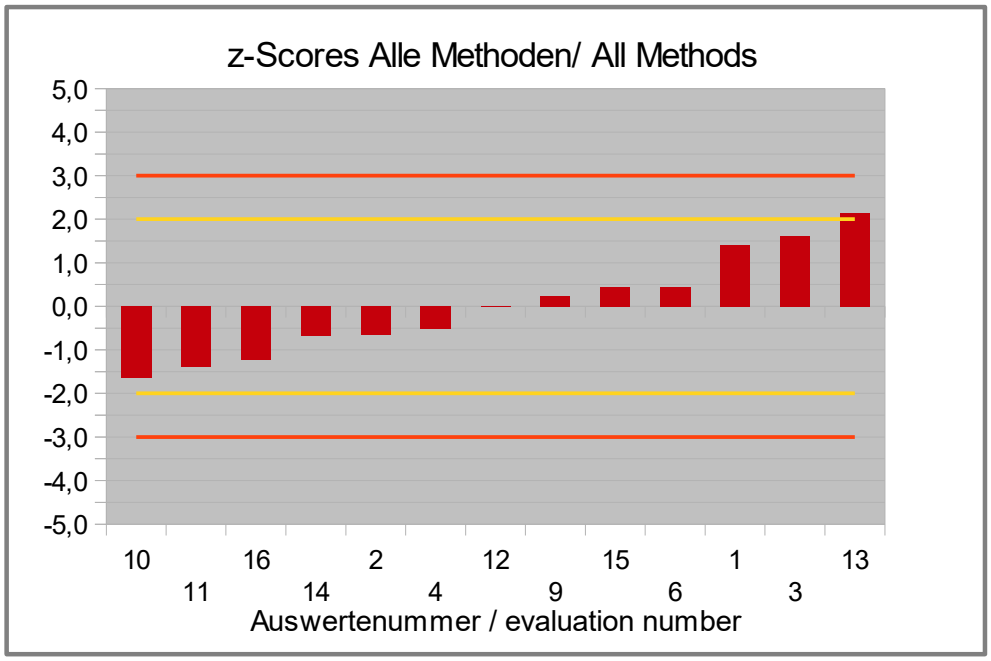

Abb./Fig. 17:
z-Scores Deoxynivalenol (DON)
Assigned value robust mean results all methods

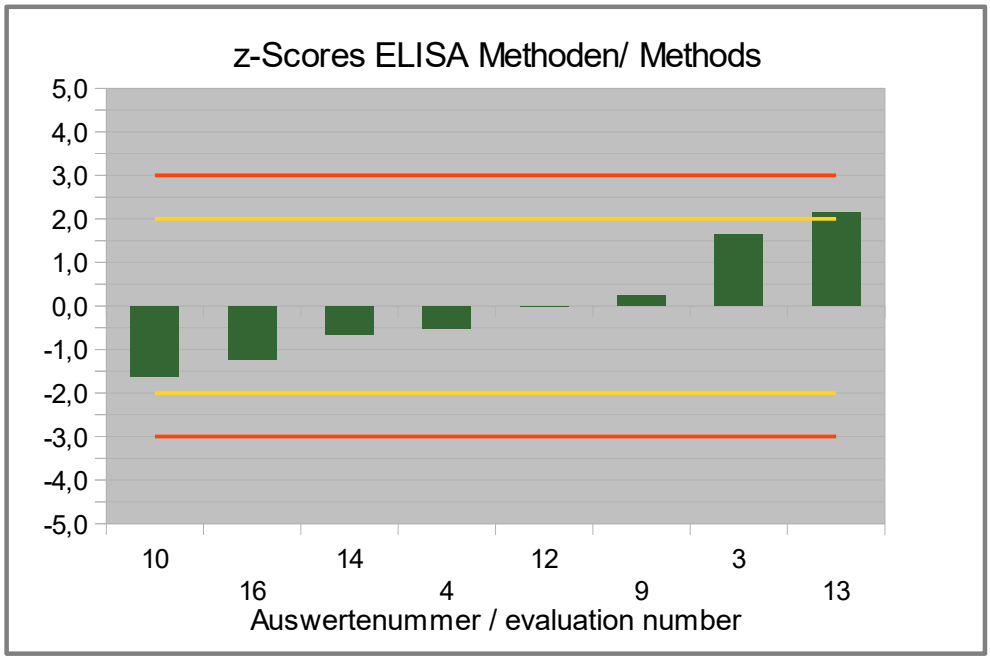

Abb./Fig. 18:
z-Scores Deoxynivalenol (DON)
Assigned value robust mean results ELISA methods

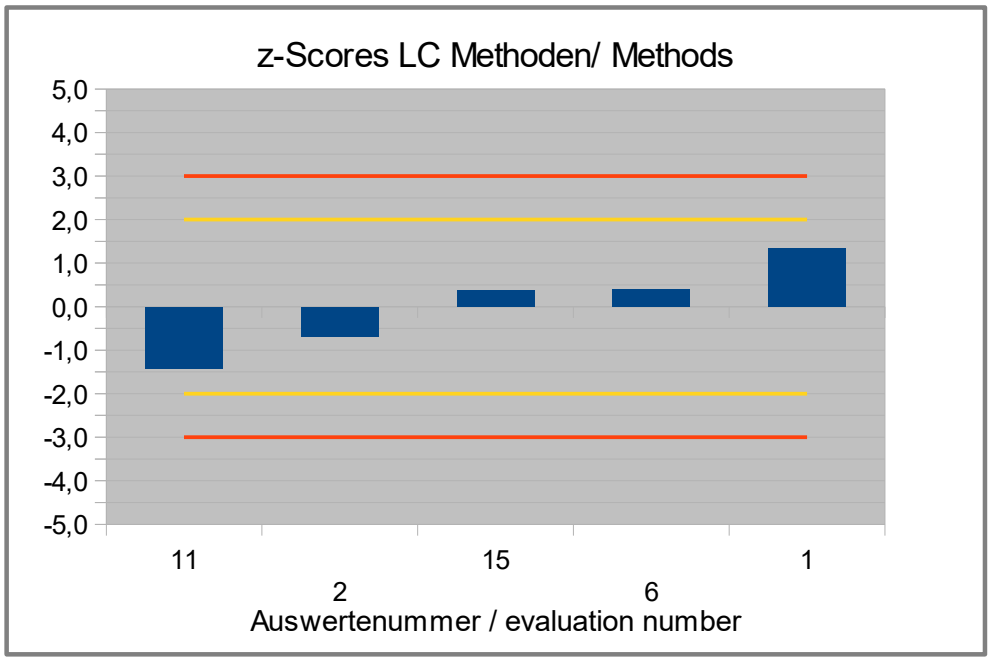

Abb./Fig. 19:
z-Scores Deoxynivalenol (DON)
Assigned value robust mean results LC methods

4.4 Proficiency Test Fumonisins

4.4.1 Results: Fumonisin B1 (FUMO B1)

Due to the small number of results no qualitative and quantitative evaluation was done (details see documentation).

4.4.2 Results: Fumonisin B2 (FUMO B2)

Due to the small number of results no qualitative and quantitative evaluation was done (details see documentation).

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		
3	positive	279		<200	1/2 (50%)	ELISA	LOQ (200 µg/kg) is > acceptance level
4	positive	140	negative	<50	2/2 (100%)	ELISA	
9	positive	270	negative	8,80	2/2 (100%)	ELISA	
12	positive	278		<222	1/2 (50%)	ELISA	LOQ (222 µg/kg) is > acceptance level
14	positive	534	negative	< 25	2/2 (100%)	ELISA	
15	positive	392	negative	<BG	2/2 (100%)	HPLC	
5	positive	279	negative	<80	2/2 (100%)	LC/MS	Sum calculated by DLA
6	positive	189	negative	<50	2/2 (100%)	LC/MS	

	Sample A		Sample B	
Number positive	8		0	
Number negative	0		6	
Percent positive	100		0	
Percent negative	0		100	
Consensus value	positive		negative	

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

Methods:
w eitere Angaben s. Dokumentation
further details see documentation

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 all results were above and for sample B, as far as evaluable, below the acceptance level.

Quantative valuation: Fumonisins Sum in µg/kg

Sample A

Statistic Data	All Methods	ELISA-Methods
Number of results	8	5
Number of outliers	0	0
Mean	295	300
Median	279	278
Robust Mean (X_{pt})	**286**	**300**
Robust standard deviation (S*)	**115**	**163**
Number with 2 replicates	7	5
Repeatability SD (S_r)	48,5	53,7
Repeatability (CV_r)	16,3%	17,9%
Reproducibility SD (S_R)	135	148
Reproducibility (CV_R)	45,5%	49,3%
Target range:		
Target standard deviation σ_{pt}	**62,8**	**108**
Target standard deviation (for Information)	55,2	66,0
lower limit of target range	**160**	**85**
upper limit of target range	**411**	**515**
Quotient S/σ_{pt}*	*1,8*	*1,5*
Standard uncertainty $U_{(Xpt)}$	*50,8*	*90,9*
Results in the target range	*6*	*4*
Percent in the target range	*75%*	*80%*

Comments to the statistical characteristics:

For evaluation of the results of all methods the target standard deviation was calculated using data from a precision experiment (3.2.6.2). For evaluation of the results of the ELISA methods the target standard deviation was calculated according to the general model of Horwitz/Thompson (3.2.6.1). In addition, the target standard deviation of the other model was given for information.

The distributions of all results showed a normal variability. The distribution of the ELISA results showed a slightly increased variability. The quotient S*/σ_{pt} was >2,0. Therefore the ELISA methods evaluation was done by z'-scores considering the standard uncertainty (s. 3.2.8). The quotient S*/σ_{pt}' was below 2,0 then.

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

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

75% of results of all methods and 80% of ELISA-methods were in the target range.

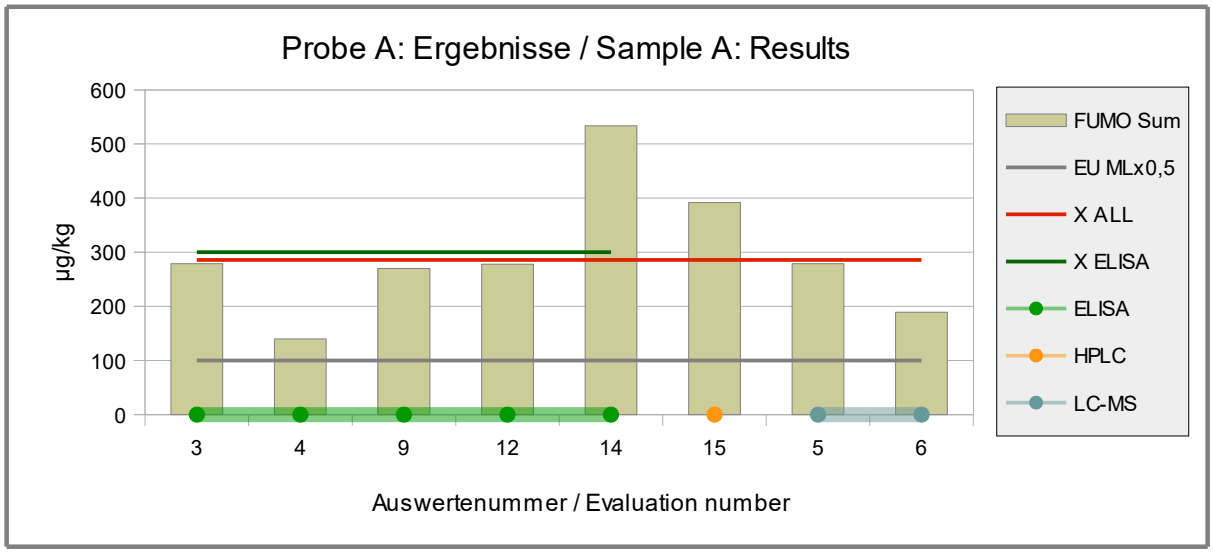

Abb./Fig. 20: Results Fumonisins Sum (FUMO Sum)
red line = Assigned value robust mean results all methods
green line = Assigned value robust mean results ELSIA methods
grey line = Qual. valuation as positive > 100 µg/kg
round symbols = Applied methods (see legend)

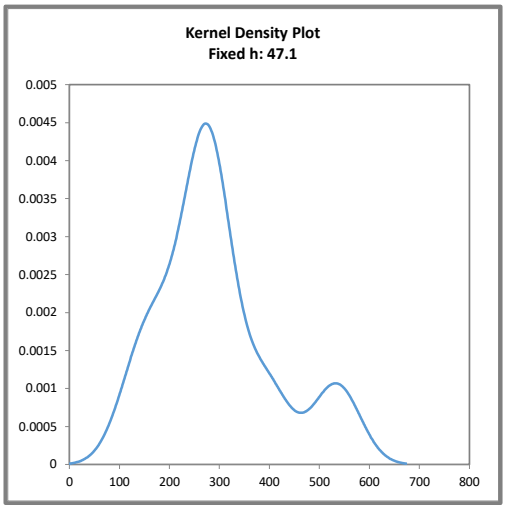

Abb. / Fig. 21:
Kerndichte-Schätzung aller Ergebnisse
(mit h = 0,75 x σpt von Xpt_{ALL})

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 < 200 µg/kg and a small peak at approx. 500 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 Alle		X ELISA			
3	279	-6,6	-0,10	-20,9	-0,19	ELISA	
4	140	-145,7	-2,3	-160,0	-1,5	ELISA	
9	270	-15,7	-0,25	-30,0	-0,28	ELISA	
12	278	-7,7	-0,12	-22,0	-0,20	ELISA	
14	534	248,3	4,0	234,0	2,2	ELISA	
15	392	106,3	1,7			HPLC	
5	279	-6,7	-0,11			LC/MS	Sum calculated by DLA
6	189	-96,7	-1,5			LC/MS	

Methoden:
weitere Angaben s. Dokumentation
further details see documentation

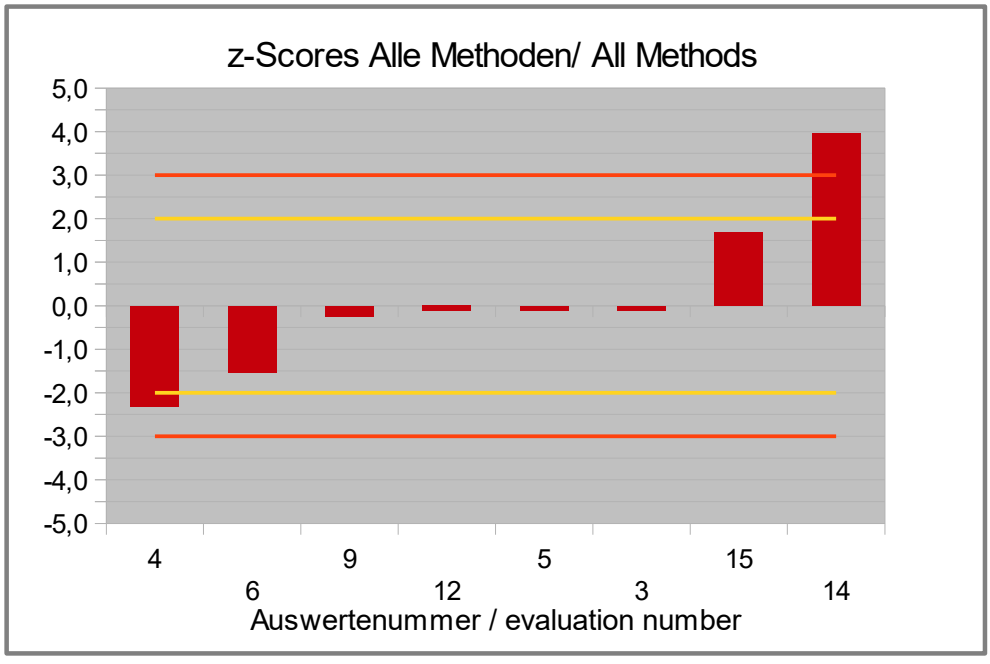

Abb./Fig. 22:
z-Scores Fumonisins Sum (FUMO Sum)
Assigned value robust mean results all methods

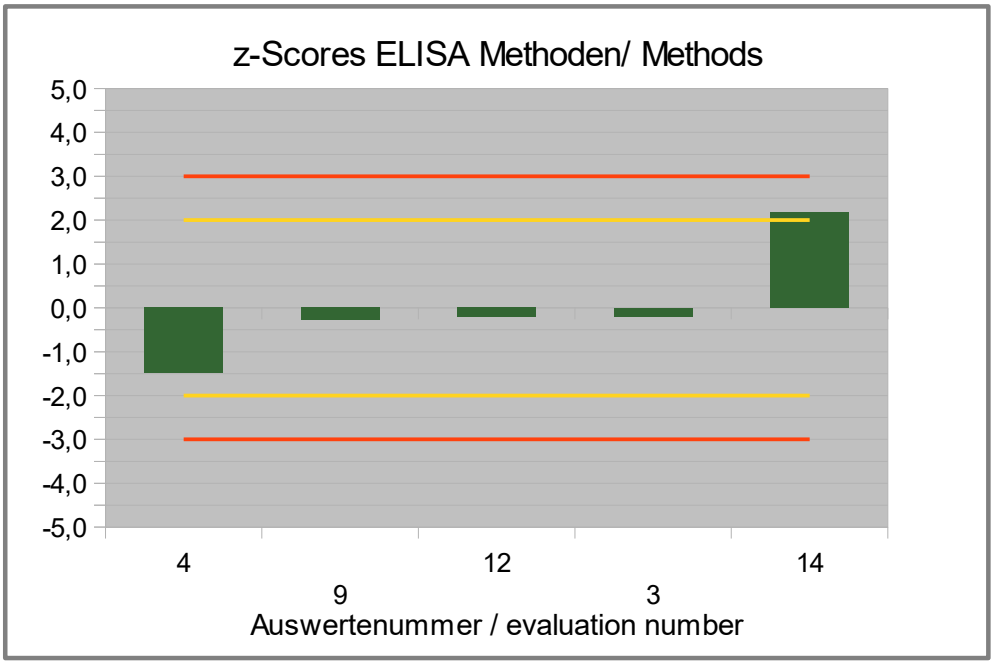

Abb./Fig. 23:
z-Scores Fumonisins Sum (FUMO Sum)
Assigned value robust mean results ELISA methods

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		
3	positive	73,1	negative	<15	2/2 (100%)	ELISA	
4	positive	51,0	positive	42,0	2/2 (100%)	ELISA	
7	negative	0	positive	31,1	0/2 (0%)	ELISA	Mean of sample B calculated by DLA
8	negative	22,0	negative	9,70	1/2 (50%)	ELISA	
9	positive	65,2	positive	27,5	1/2 (50%)	ELISA	
10	positive	47,1	negative	<1,75	2/2 (100%)	ELISA	
12	positive	54,0		<50	2/2 (100%)	ELISA	
13	positive	42,6	negative	14,7	2/2 (100%)	ELISA	
14	positive	68,0	negative	< 1,75	2/2 (100%)	ELISA	
2	positive	35,2	negative	<10	2/2 (100%)	HPLC	
11	positive	42,2	negative	<1	2/2 (100%)	HPLC	
1	positive	57,9	negative	<10	2/2 (100%)	LC/MS	
5	positive	84,8		<50	2/2 (100%)	LC/MS	
6	positive	66,2	negative	<5,0	2/2 (100%)	LC/MS	
15	positive	45,0	negative	<LOQ	2/2 (100%)	LC/MS	

	Sample A		Sample B	
Number positive	13		3	
Number negative	2		10	
Percent positive	87		23	
Percent negative	13		77	
Consensus value	positive		negative	

Methods:
w eitere Angaben s. Dokumentation
further details see documentation

positive: > 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, 87% of the results were above and for sample B 77% below the acceptance level.

Quantative valuation: Zearalenone in µg/kg

Sample A

Statistic Data	All Methods	ELISA-Methods	LC-Methods
Number of results	14	8	6
Number of outliers	0	0	0
Mean	53,9	52,9	55,2
Median	52,5	52,5	51,5
Robust Mean (X_{pt})	**54,0**	**53,7**	**55,2**
Robust standard deviation (S*)	**16,9**	**16,7**	**20,8**
Number with 2 replicates	12	7	5
Repeatability SD (S_r)	9,00	11,0	4,94
Repeatability (CV_r)	17,6%	20,9%	10,0%
Reproducibility SD (S_R)	16,5	19,4	13,0
Reproducibility (CV_R)	32,2%	36,8%	26,3%
Target range:			
Target standard deviation σ_{pt}	**11,9**	**11,8**	**12,1**
Target standard deviation (for Information)	12,3	12,3	12,6
lower limit of target range	**30,2**	**30,1**	**30,9**
upper limit of target range	**77,7**	**77,3**	**79,5**
Quotient S/σ_{pt}*	*1,4*	*1,4*	*1,7*
Standard uncertainty $U_{(Xpt)}$	*5,66*	*7,36*	*10,6*
Results in the target range	*12*	*7*	*5*
Percent in the target range	*86%*	*88%*	*83%*

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 and coefficients of variation *CV_r and CV_R* are in the range of established values of the applied methods (see 3.2.6.2).

86% of results of all methods, 88% of ELISA-methods and 83% of LC-methods were in the target range.

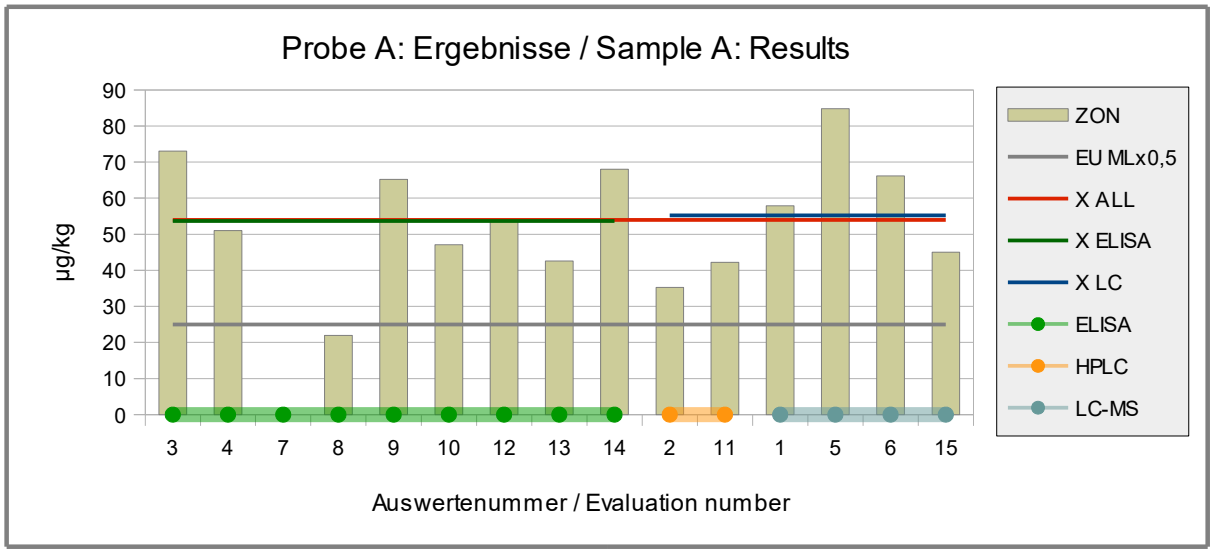

Abb./Fig. 24: Results Zearalenone (ZON)
red line = Assigned value robust mean results all methods
green line = Assigned value robust mean results ELSIA methods
blue line = Assigned value robust mean results LC methods
grey line = Qual. valuation as positive > 25 µg/kg
round symbols = Applied methods (see legend)

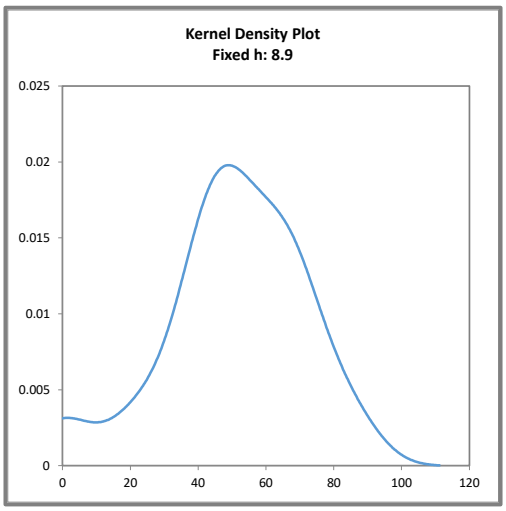

Abb. / Fig. 25:
Kerndichte-Schätzung aller Ergebnisse
(mit h = 0,75 x σ_{pt} von Xpt_{ALL})

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 slight shoulder at > 60 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 Alle		X ELISA		X LC			
3	73,1	19,11	1,6	19,36	1,6			ELISA	
4	51,0	-2,95	-0,25	-2,70	-0,23			ELISA	
7	0							ELISA	Mean of sample B calculated by DLA
8	22,0	-32,00	-2,7	-31,75	-2,7			ELISA	
9	65,2	11,25	0,95	11,50	0,97			ELISA	
10	47,1	-6,85	-0,58	-6,60	-0,56			ELISA	
12	54,0	0,01	0,00	0,26	0,02			ELISA	
13	42,6	-11,39	-0,96	-11,13	-0,94			ELISA	
14	68,0	14,05	1,2	14,30	1,2			ELISA	
2	35,2	-18,71	-1,6			-19,98	-1,6	HPLC	
11	42,2	-11,75	-0,99			-13,02	-1,1	HPLC	
1	57,9	3,95	0,33			2,68	0,22	LC/MS	
5	84,8	30,85	2,6			29,58	2,4	LC/MS	
6	66,2	12,25	1,0			10,98	0,90	LC/MS	
15	45,0	-8,95	-0,75			-10,22	-0,84	LC/MS	

Methoden:
weitere Angaben s. Dokumentation
further details see documentation

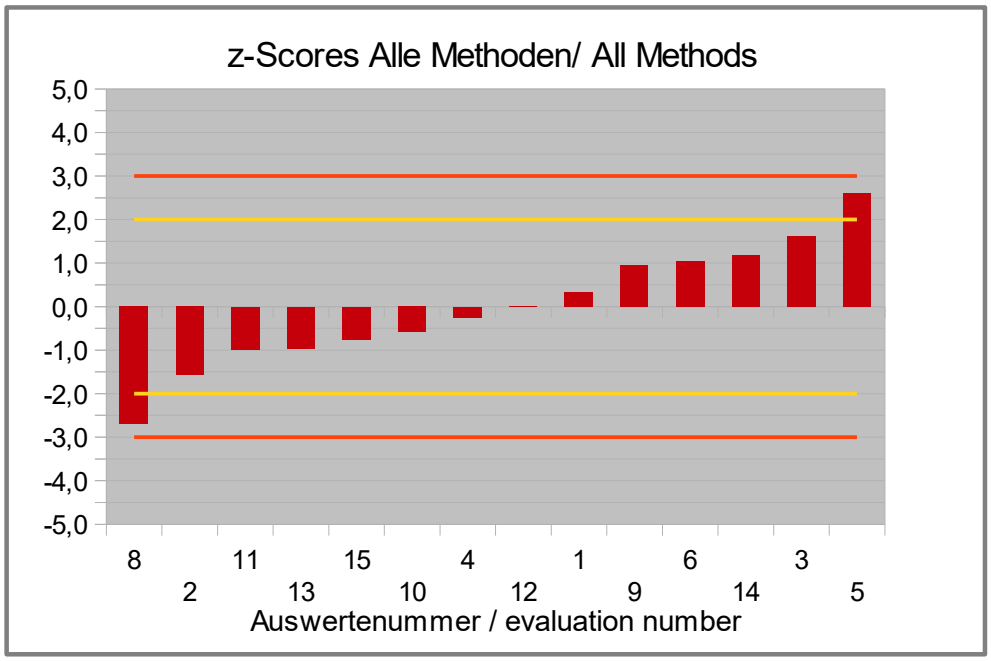

Abb./Fig. 26:
z-Scores Zearalenone (ZON)
Assigned value robust mean results all methods

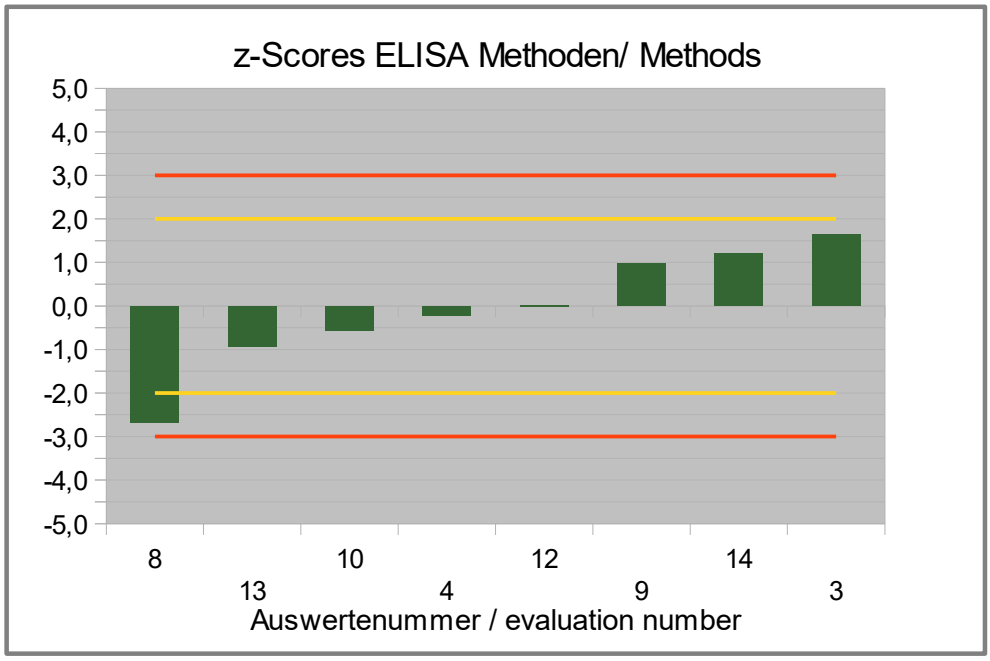

Abb./Fig. 27:
z-Scores Zearalenone (ZON)
Assigned value robust mean results ELISA methods

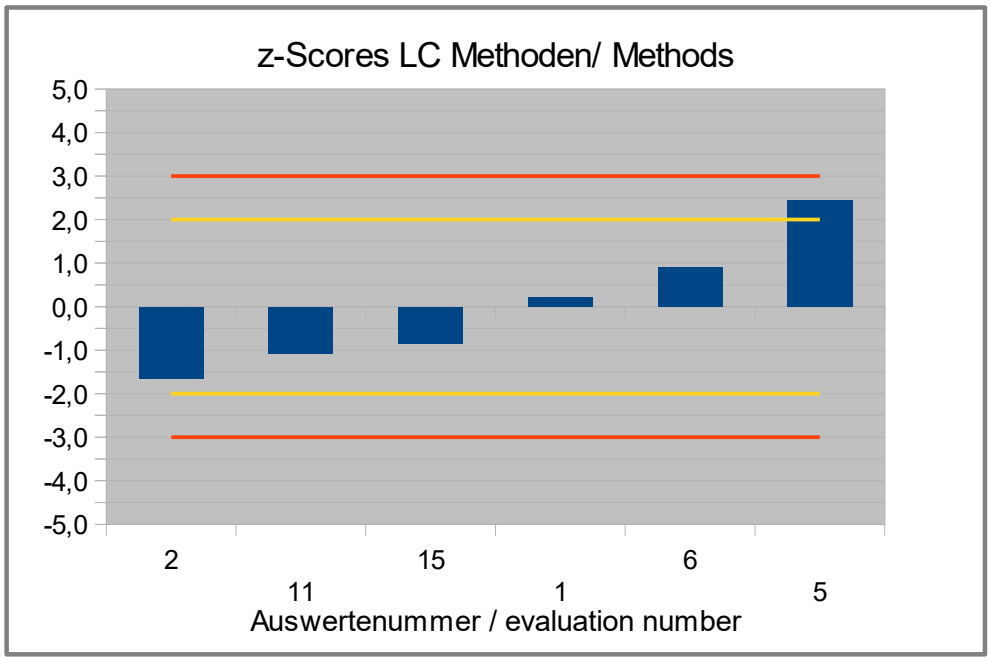

Abb./Fig. 28:
z-Scores Zearalenone (ZON)
Assigned value robust mean results LC methods

4.6 z-Scores of participants: Summary table

Auswerte-nummer	AF B1	AF B1	AF Sum	AF Sum	AF Sum	OTA	OTA	OTA	DON	DON	DON	FUMO Sum	FUMO Sum	ZON	ZON	ZON
Methoden	ALL	LC	All	ELISA	LC	All	ELISA	LC	All	ELISA	LC	All	ELISA	All	ELISA	LC
1	1,1	0,59	0,42	-	0,09	1,0	-	0,76	1,4	-	1,4	-	-	0,33	-	0,22
2	-1,1	-1,4	-1,1	-	-1,3	-0,53	-	-0,86	-0,64	-	-0,69	-	-	-1,6	-	-1,6
3	-0,87	-	-0,49	-0,35	-	-0,23	0,03	-	1,6	1,6	-	-0,10	-0,19	1,6	1,6	-
4	-	-	-1,9	-1,8	-	-1,8	-1,8	-	-0,53	-0,51	-	-2,3	-1,5	-0,25	-0,23	-
5	0,00	-0,39	0,29	-	-0,03	0,06	-	-0,26	-	-	-	-0,11	-	2,6	-	2,4
6	0,60	0,16	0,72	-	0,37	0,59	-	0,30	0,44	-	0,39	-1,5	-	1,0	-	0,90
7	-	-	2,0	2,2	-	0,04	0,35	-	-	-	-	-	-	-	-	-
8	-	-	0,85	1,0	-	-1,5	-1,4	-	-	-	-	-	-	-2,7	-2,7	-
9	-	-	1,3	1,4	-	-0,62	-0,44	-	0,23	0,25	-	-0,25	-0,28	0,95	0,97	-
10	-1,1	-	-1,9	-1,9	-	1,9	2,5	-	-1,6	-1,6	-	-	-	-0,58	-0,56	-
11	-1,0	-1,3	-1,2	-	-1,4	-0,34	-	-0,67	-1,4	-	-1,4	-	-	-0,99	-	-1,1
12	-	-	-	-	-	-	-	-	-0,02	0,00	-	-0,12	-0,20	0,00	0,02	-
13	-	-	-0,70	-0,57	-	-	-	-	2,1	2,2	-	-	-	-0,96	-0,94	-
14	0,19	-	-0,35	-0,22	-	0,47	0,86	-	-0,67	-0,66	-	4,0	2,2	1,2	1,2	-
15	3,0	2,3	2,8	-	2,3	1,0	-	0,73	0,44	-	0,38	1,7	-	-0,75	-	-0,84
16	0,53	-	-0,06	0,09	-	-	-	-	-1,2	-1,2	-	-	-	-	-	-

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

5. Documentation

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 Quantitation	Incl. Recovery	Recovery Rate
				Day/Month	Sample A	Sample A	Sample A	Sample B	Sample B	Sample B		yes/no	in %
Aflatoxin B1	ELISA	3	µg/kg	02/07	<0,7	<0,7	<0,7	3,65	3,8	3,5	0,7	no	
	ELISA	7	µg/kg	19.05.20	1,9	1,7	2,1		7,1	7,6			
	ELISA	8	µg/kg	25.04.20	1,4	1,6	1,2	6,05	6,5	5,6	10	no	
	ELISA	9	µg/kg	01.05.20	1,45	1,2	1,7	6,5	6,1	6,9			
	ELISA	10	µg/kg	14.05.20	<1			3,41			1	no	
	ELISA	14	µg/kg	28.05.20	< 1	<1	<1	4,7	4,5	4,9	1	no	A: 108, B: 88
	ELISA	16	µg/kg	20.05.20	<1	<1	<1	5,03	5,1	5	1		
	HPLC	2	µg/kg	13.05.	<0,01	<0,01	<0,01	3,406	3,484	3,327	0,01	no	
	HPLC	11	µg/kg	07.05.20	<0,1			3,52	3,58	3,46	0,1	no	116
	LC/MS	1	µg/kg		<0,5	<0,5	<0,5	5,57	5,5	5,64	0,5	yes	
	LC/MS	5	µg/kg	12.05.20	<0,2			4,51			<0.5	no	
	LC/MS	6	µg/kg	12.05.20	<0,2	<0,2	<0,2	5,1	5,1	5,1	0,2	yes	100
	LC/MS	15	µg/kg	13.05.20	<LOQ	<LOQ	<LOQ	7,47	6,5	8,4	2	no	

Parameter	Meth. Abr.	Partici-pant	Unit	Date of Analysis	Result (Mean)	Result I	Result II	Result (Mean)	Result I	Result II	Limit of Quantitation	Incl. Recovery	Recovery Rate
				Day/Month	Sample A	Sample A	Sample A	Sample B	Sample B	Sample B		yes/no	in %
Aflatoxin B2	HPLC	2	µg/kg	13.05.	<0,01	<0,01	<0,01	0,273	0,282	0,264	0,01	no	
	HPLC	11	µg/kg	07.05.20	<0,1			0,15	0,16	0,15	0,1	no	75
	LC/MS	1	µg/kg		<0,5	<0,5	<0,5	<0,5	<0,5	<0,5	0,5	yes	
	LC/MS	5	µg/kg	12.05.20	<0,2			0,55			<0.5	no	
	LC/MS	6	µg/kg	12.05.20	<0,2	<0,2	<0,2	0,49	0,47	0,51	0,2	yes	100
	LC/MS	15	µg/kg	13.05.20	<LOQ	<LOQ	<LOQ	0,8	0,75	0,85	3	no	

Parameter	Meth. Abr.	Partici-pant	Unit	Date of Analysis	Result (Mean)	Result I	Result II	Result (Mean)	Result I	Result II	Limit of Quantitation	Incl. Recovery	Recovery Rate
				Day/Month	Sample A	Sample A	Sample A	Sample B	Sample B	Sample B		yes/no	in %
Aflatoxin G1	HPLC	2	µg/kg	13.05.	<0,01	<0,01	<0,01	0,155	0,151	0,158	0,01	no	
	HPLC	11	µg/kg	07.05.20	<0,1			0,11	0,1	0,11	0,1	no	76
	LC/MS	1	µg/kg		<0,5	<0,5	<0,5	<0,5	<0,5	<0,5	0,5	yes	
	LC/MS	5	µg/kg	12.05.20	<0,2			0,36			<0.5	no	
	LC/MS	6	µg/kg	12.05.20	<0,2	<0,2	<0,2	<0,2	<0,2	<0,2	0,2	yes	100
	LC/MS	15	µg/kg	13.05.20	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	3	no	

Parameter	Meth. Abr.	Partici-pant	Unit	Date of Analysis	Result (Mean)	Result I	Result II	Result (Mean)	Result I	Result II	Limit of Quantitation	Incl. Recovery	Recovery Rate
				Day/Month	Sample A	Sample A	Sample A	Sample B	Sample B	Sample B		yes/no	in %
Aflatoxin G2	HPLC	2	µg/kg	13.05.	<0,01	<0,01	<0,01	0,026	0,025	0,026	0,01	no	
	HPLC	11	µg/kg	07.05.20	<0,1			<0,1			0,1	no	12
	LC/MS	1	µg/kg		<0,5	<0,5	<0,5	<0,5	<0,5	<0,5	0,5	yes	
	LC/MS	5	µg/kg	12.05.20	<0,2			<0,2			<0.5		
	LC/MS	6	µg/kg	12.05.20	<0,2	<0,2	<0,2	0,32	0,32	0,32	0,2	yes	100
	LC/MS	15	µg/kg	13.05.20	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	3	no	

Parameter	Meth. Abr.	Partici-pant	Unit	Date of Analysis	Result (Mean)	Result I	Result II	Result (Mean)	Result I	Result II	Limit of Quantitation	Incl. Recovery	Recovery Rate
				Day/Month	Sample A	Sample A	Sample A	Sample B	Sample B	Sample B		yes/no	in %
Summe Aflatoxine/ Sum of Aflatoxins	ELISA	3	µg/kg	02/07	<1	<1	<1	4,55	4,6	4,5	1	no	
	ELISA	4	µg/kg	10.6.20, 18.6.20	<1	<1	<1	3	3,93	2,07	1	no	
	ELISA	7	µg/kg	19.05.20	1,9	1,7	2,1		7,1	7,6			
	ELISA	8	µg/kg	25.04.20	1,4	1,6	1,2	6,05	6,5	5,6	10	no	
	ELISA	9	µg/kg	01.05.20	1,45	1,2	1,7	6,5	6,1	6,9			
	ELISA	10	µg/kg	02.06.20	<1,75			2,92			1,75	no	
	ELISA	13	µg/kg	28.05.20	1,378	1,335	1,421	4,313	4,437	4,189	1 ppb	yes	94,7
	ELISA	14	µg/kg	28.05.20	< 1	<1	<1	4,7	4,5	4,9	1	no	
	ELISA	16	µg/kg	20.05.20	<1	<1	<1	5,03	5,1	5	1		
	HPLC	2	µg/kg		0			3,86					
	HPLC	11	µg/kg	07.05.20	<0,1			3,78			0,1	no	
	LC/MS	1	µg/kg					5,57	5,5	5,64		yes	
	LC/MS	5	µg/kg	12.05.20	<0,2			5,42					
	LC/MS	6	µg/kg	12.05.20	<0,2	<0,2	<0,2	5,9	5,89	5,93	0,2	yes	100
	LC/MS	15	µg/kg	13.05.20	<LOQ	<LOQ	<LOQ	8,27	7,3	9,3	11	no	

Parameter	Meth. Abr.	Partici-pant	Unit	Date of Analysis	Result (Mean)	Result I	Result II	Result (Mean)	Result I	Result II	Limit of Quantitation	Incl. Recovery	Recovery Rate
				Day/Month	Sample A	Sample A	Sample A	Sample B	Sample B	Sample B		yes/no	in %
Ochratoxin A	ELISA	3	µg/kg	02/07	<1,5	<1,5	<1,5	9,305	9,54	9,07	1,5	no	
	ELISA	4	µg/kg	10.6.20, 19.6.20	<2	<2	<2	5,6	6,58	4,61	2	no	
	ELISA	7	µg/kg	19.05.20	2,4	2,3	2,5		9,6	10,3			
	ELISA	8	µg/kg	25.04.20	1,55	0,7	2,4	6,3	6,8	5,8	10	no	
	ELISA	9	µg/kg	01.05.20	2,9	2,8	3	8,35	8,1	8,6			
	ELISA	10	µg/kg	02.06.20	2,1	1,5	2,5	14,4	16,7	12,1	1	no	200
	ELISA	14	µg/kg	27.05.	< 1,25	< 1,25	< 1,25	11	11	10	1,25	no	A: 82, B: 104
	HPLC	2	µg/kg	12.06.	<0,03	<0,03	<0,03	8,583	8,666	8,499	0,03	no	
	HPLC	11	µg/kg	19.05.20	<0,1			9,04	8,91	9,16	0,1	no	
	LC/MS	1	µg/kg		<0,5	<0,5	<0,5	12,37	12,35	12,39	0,5	yes	
	LC/MS	5	µg/kg	12.05.20	<1			10			<1	no	
	LC/MS	6	µg/kg	12.05.20	<0,5	<0,5	<0,5	11,3	11,2	11,4	0,5	yes	100
	LC/MS	15	µg/kg	13.05.20	<LOQ	<LOQ	<LOQ	12,3	11,9	12,6	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 Quantitation	Incl. Recovery	Recovery Rate
				Day/Month	Sample A	Sample A	Sample A	Sample B	Sample B	Sample B		yes/no	in %
Deoxynivalenol	ELISA	3	µg/kg	02/07	1194,35	1164,1	1224,6	<100	<100	<100	100	no	
	ELISA	4	µg/kg	28.5.20, 16.6.20	780	825	740	<250	<250	<250	250	no	
	ELISA	9	µg/kg	01.05.20	926,08	954,28	897,87	101,27	117,24	85,3			
	ELISA	10	µg/kg	26.05.20	565	590	540	<18,5			18,5	no	81
	ELISA	12	µg/kg	26.05.20	878	849	907	<222	<222	<222	222	---	---
	ELISA	13	µg/kg	09.06.2020.	1293,5	1280,6	1305,5	90,636	99,169	82,103	0,1 ppm	yes	106,1
	ELISA	14	µg/kg	12.05.	752	844	660	< 30	< 30	< 30	30	no	A: 116, B: 144
	ELISA	16	µg/kg	20.05.20	642,6	634,1	651,1	<40	<40	<40	40		
	HPLC	2	µg/kg	08.06.	757,68	762,97	752,39	<40	<40	<40	40	no	
	HPLC	11	µg/kg	19.05.20	613	615	611	<50			50	no	92
	LC/MS	1	µg/kg		1154	1179	1129	187	187	-	100	yes	
	LC/MS	6	µg/kg	12.05.20	967	965	969	22	22,4	21,8	10	yes	100
	LC/MS	15	µg/kg	13.05.20	966	946	986	<LOQ	<BG	<LOQ	80	no	

Parameter	Meth. Abr.	Partici-pant	Unit	Date of Analysis	Result (Mean)	Result I	Result II	Result (Mean)	Result I	Result II	Limit of Quantitation	Incl. Recovery	Recovery Rate
				Day/Month	Sample A	Sample A	Sample A	Sample B	Sample B	Sample B		yes/no	in %
Fumonisin B1	HPLC	15	µg/kg	10.06.20	352	321	383	<LOQ	<LOQ		100	no	
	LC/MS	5	µg/kg	19.06.20	279			<80			<80	no	
	LC/MS	6	µg/kg	15.05.20	189	186	192	<50	<50	<50	50	yes	100

Parameter	Meth. Abr.	Partici-pant	Unit	Date of Analysis	Result (Mean)	Result I	Result II	Result (Mean)	Result I	Result II	Limit of Quantitation	Incl. Recovery	Recovery Rate
				Day/Month	Sample A	Sample A	Sample A	Sample B	Sample B	Sample B		yes/no	in %
Fumonisin B2	HPLC	15	µg/kg	10.06.20	40	39	41	<LOQ	<LOQ		100	no	
	LC/MS	5	µg/kg	19.06.20	<80			<80			<80		
	LC/MS	6	µg/kg	15.05.20	<50	<50	<50	<50	<50	<50	50	yes	100

Parameter	Meth. Abr.	Partici-pant	Unit	Date of Analysis	Result (Mean)	Result I	Result II	Result (Mean)	Result I	Result II	Limit of Quantitation	Incl. Recovery	Recovery Rate
				Day/Month	Sample A	Sample A	Sample A	Sample B	Sample B	Sample B		yes/no	in %
Gesamt Fumonisine/ Total Fumonisins	ELISA	3	µg/kg	02/07	279,1	276,5	281,7	<200	<200	<200	200	no	
	ELISA	4	µg/kg	16.6.20, 11.6.20	140	129,5	151,2	<50	<50	<50	50	no	
	ELISA	9	µg/kg	01.05.20	270	327	213	8,8	11,9	5,7			
	ELISA	12	µg/kg	26.05.20	278	246	310	<222	<222	<222	222	---	---
	ELISA	14	µg/kg	29.05.20	534	481	587	< 25	< 25	< 25	25	no	A: 138, B: 129
	HPLC	15	µg/kg	10.06.20	392	360	424	<LOQ	<LOQ				
	LC/MS	5	µg/kg	19.06.20	279			<80			<80		
	LC/MS	6	µg/kg	15.05.20	189	186	192	<50	<50	<50	50	yes	100

Parameter	Meth. Abr.	Partici-pant	Unit	Date of Analysis	Result (Mean)	Result I	Result II	Result (Mean)	Result I	Result II	Limit of Quantitation	Incl. Recovery	Recovery Rate
				Day/Month	Sample A	Sample A	Sample A	Sample B	Sample B	Sample B		yes/no	in %
Zearalenone	ELISA	3	µg/kg	02/07	73,06	72,33	73,79	<15	<15	<15	7,5	no	
	ELISA	4	µg/kg	11.6.20, 16.6.20	51	60,6	41,4	42	44,6	39,1	25	no	
	ELISA	7	µg/kg	19.05.20	0	0	0		29,5	32,7			
	ELISA	8	µg/kg	25.04.20	21,95	13,9	30	9,7	8,8	10,6	100	no	
	ELISA	9	µg/kg	01.05.20	65,2	78,2	52,2	27,5	31,9	23,1			
	ELISA	10	µg/kg	28.05.20	47,1	50,7	43,5	<1,75			1,75	no	131
	ELISA	12	µg/kg	26.05.20	53,96	<50	57,92	<50	<50	<50	50	---	---
	ELISA	13	µg/kg	15.05.20	42,567	40,347	44,788	14,705	14,504	14,906	15 ppb	ja	99,9
	ELISA	14	µg/kg	28.05.	68	77	59	< 1,75	< 1,75	< 1,75	1,75	no	A: 108, B: 130
	HPLC	2	µg/kg	19.05.	35,24	34,44	36,03	<10	<10	<10	10	no	
	HPLC	11	µg/kg	19.05.20	42,2	49,2	35,3	<1			1	no	102
	LC/MS	1	µg/kg		57,9	60,9	54,9	<10	<10	<10	10	ja	
	LC/MS	5	µg/kg	12.05.20	84,8			<50			<50	no	95
	LC/MS	6	µg/kg	12.05.20	66,2	67,9	64,5	<5,0	<5,0	<5,0	5	ja	100
	LC/MS	15	µg/kg	13.05.20	45	44,7	45,3	<BG	<LOQ	<LOQ	4	no	

5.1.2 Analytical Methods

Parameter	Meth. Abr.	Partici-pant	Method description as in test report / 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	
Aflatoxin B1	ELISA	3	quantitative ELISA	extraction with methanol 70%		standard solutions by the manufacturer	no	yes	
	ELISA	7			Elisa (Neogen)				
	ELISA	8			ELISA				
	ELISA	9			Elisa Screening by Neogen				
	ELISA	10	in-house method SOP Q 37-02		• r-biopharm Testkit RIDASCREEN Aflatoxin B1 30/15			yes	
	ELISA	14	ELISA, RIDASCREEN® Aflatoxin B1 30/15, Art. No. R1211			DLA_2019_ptMYS1	yes	yes	
	ELISA	16	Aflatoxin B1 ELISA Tecna Celer Afla B1 HU0040004, Lot H07119					no	
	HPLC	2	ASU L 15.00-2 (2014-02)					yes	
	HPLC	11	DIN EN ISO 16050				yes	yes	
	LC/MS	1			LC-MS				
	LC/MS	5	HPLC-MS/MS					yes	recovery correction by int. standard
	LC/MS	6	SOP M 3650	Extraction with Acetonitril and Water	LC-MS/MS	6 Point Calibration	no	yes	
	LC/MS	15	LC-MS / MS (ESI). Stable isotope dilution analysis with single-stage, wet chemical extraction and Dilute & Shoot.					yes	

Parameter	Meth. Abr.	Partici-pant	Method description as in test report / 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	
Aflatoxin B2, G1, G2	HPLC	2	ASU L 15.00-2 (2014-02)					yes	
	HPLC	11	DIN EN ISO 16050				yes	yes	
	LC/MS	1			LC-MS				
	LC/MS	5	HPLC-MS/MS					yes	recovery correction by int. standard
	LC/MS	6	SOP M 3650	Extraction with Acetonitril and Water	LC-MS/MS	6 Point Calibration	no	yes	
	LC/MS	15	LC-MS / MS (ESI). Stable isotope dilution analysis with single-stage, wet chemical extraction and Dilute & Shoot.					yes	

Parameter	Meth. Abr.	Partici-pant	Method description as in test report / 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	
Summe Aflatoxine/ Sum of Aflatoxins	ELISA	3	quantitative ELISA	extraction with methanol 70%		standard solutions by the manufacturer	no	yes	
	ELISA	4	ELISA	according to protocol for dry fruits					
	ELISA	7			Elisa (Neogen)				
	ELISA	8							
	ELISA	9							
	ELISA	10	in-house method SOP Q 217-04		• r-biopharm Testkit RIDASCREEN Aflatoxin total			yes	
	ELISA	13	Like in a analysis report		ELISA			no	
	ELISA	14							
	ELISA	16							
	HPLC	2							
	HPLC	11							
	LC/MS	1			LC-MS				
	LC/MS	5							
	LC/MS	6	SOP M 3650	Extraction with Acetonitril and Water	LC-MS/MS	6 Point Calibration	no	yes	
	LC/MS	15	LC-MS / MS (ESI). Stable isotope dilution analysis with single-stage, wet chemical extraction and Dilute & Shoot.					yes	

Parameter	Meth. Abr.	Partici-pant	Method description as in test report / 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	3	quantitative ELISA	extraction with methanol 70%		standard solutions by the manufacturer	no	yes	
	ELISA	4	ELISA	according to protocol for dry fruits					
	ELISA	7			Elisa (Neogen)				
	ELISA	8			ELISA				
	ELISA	9							
	ELISA	10	in-house method SOP Q 47-04	Sample preparation for fast and easy screening	r-biopharm Testkit RIDASCREEN Ochratoxin A 30/15		no	yes	
	ELISA	14	ELISA, RIDASCREEN® Ochratoxin A 30/15, Art. No. R1311	according to 9.2 Testkit manual		DLA_2019_ptMYS1	yes	yes	
	HPLC	2	A. Thellmann, W. Weber: DLR 93 (1), 1997, S. 1-3 (1997)					yes	
	HPLC	11	L15.03-1					yes	
	LC/MS	1			LC-MS				
	LC/MS	5	HPLC-MS/MS				no	yes	recovery correction by int. standard
	LC/MS	6	SOP M 3650	Extraction with Acetonitril and Water	LC-MS/MS	6 Point Calibration	no	yes	
	LC/MS	15	LC-MS / MS (ESI). Stable isotope dilution analysis with single-stage, wet chemical extraction and Dilute & Shoot.					yes	

Parameter	Meth. Abr.	Partici-pant	Method description as in test report / 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	3	quantitative ELISA	extraction with deionized water		standard solutions by the manufacturer	no	yes	
	ELISA	4	ELISA						
	ELISA	9							
	ELISA	10	in-house method SOP Q 42-04		• r-biopharm Testkit RIDASCREEN DON		no	yes	
	ELISA	12	r-biopharm Fast-DON R5901	as per kit insturctions	as per kit insturctions	---	---	see test kit	
	ELISA	13	Like in a analysis report		ELISA			no	
	ELISA	14	ELISA, RIDASCREEN® DON, Art. No. R5906			DLA_2019_ptMYS1	yes	yes	Amounts > LOQ detected in sample B by ELISA. They are due to cross-reactivities. Confirmation by HPLC resulted in levels of <30 µg/kg. Therefore the LOQ of the HPLC method was given.
	ELISA	16	DON ELISA Tecna Celer DON HU0040009, Lot 01030					no	
	HPLC	2	WES 072 (in-house method)					yes	
	HPLC	11	L 15.00-9				no	yes	
	LC/MS	1			LC-MS				
	LC/MS	6	SOP M 3650	Extraction with Acetonitril and Water	LC-MS/MS	6 Point Calibration	no	yes	
	LC/MS	15	LC-MS / MS (ESI). Stable isotope dilution analysis with single-stage, wet chemical extraction and Dilute & Shoot.					yes	

Parameter	Meth. Abr.	Partici-pant	Method description as in test report / 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 and B2	HPLC	15	HPLC-FLD according to EN 14352					yes	
	LC/MS	5	HPLC-MS/MS					no	
	LC/MS	6	SOP M 2919	Extraction with Ammonium acetate solution and Methanol	LC-MS/MS	5 Point Calibration	no	yes	

Parameter	Meth. Abr.	Partici-pant	Method description as in test report / 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	
Gesamt Fumonisine/ Total Fumonisins	ELISA	3	quantitative ELISA	extraction with methanol 70%		standard solutions by the manufacturer	no	yes	
	ELISA	4	ELISA						
	ELISA	9							
	ELISA	12	r-biopharm Fast-FUM R5602	as per kit instutructions	as per kit instutructions	---	---	see test kit	
	ELISA	14	ELISA, RIDASCREEN® Fumonisin, Art. No. R3401		Specifity to Fumonisin B2 approx. 40%	DLA_2019_ptMYS1	yes	yes	
	HPLC	15							
	LC/MS	5							
	LC/MS	6	SOP M 2919	Extraction with Ammonium acetate solution and Methanol	LC-MS/MS	5 Point Calibration	no	yes	

Parameter	Meth. Abr.	Partici-pant	Method description as in test report / 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	3	quantitative ELISA	extraction with methanol 70%		standard solutions by the manufacturer	no	yes	
	ELISA	4	ELISA						
	ELISA	7			Elisa (Neogen)				
	ELISA	8			ELISA				
	ELISA	9							
	ELISA	10	in-house method SOP Q 49-03		• r-biopharm Testkit RIDASCREEN Zearalenone		no		
	ELISA	12	r-biopharm Fast-ZEA R5502	as per kit instructions	as per kit instructions	---	---	see test kit	
	ELISA	13	Like in a analysis report		ELISA			no	
	ELISA	14	ELISA, RIDASCREEN® Zearalenon, Art. No. R1401			DLA_2019_ptMYS1	yes	yes	
	HPLC	2	WES 128 (in-house method)					yes	
	HPLC	11	DIN EN 15792				no	yes	
	LC/MS	1			LC-MS				
	LC/MS	5	HPLC-MS/MS				yes	yes	
	LC/MS	6	SOP M 3650	Extraction with Acetonitril and Water	LC-MS/MS	6 Point Calibration	no	yes	
	LC/MS	15	LC-MS / MS (ESI). Stable isotope dilution analysis with single-stage, wet chemical extraction and Dilute & Shoot.					yes	

5.2 Homogeneity

5.2.1 Mixture homogeneity before bottling

Microtracer Homogeneity Test
DLA - ptMYS1 Sample A

Weight whole sample	6,25	kg
Microtracer	FSS-rot lake	
Particle size	75 – 300	µm
Weight per particle	2,0	µg
Addition of tracer	24,0	mg/kg

Result of analysis

Sample	Weight [g]	Particle number	Particles [mg/kg]
1	5,03	59	23,5
2	5,01	54	21,6
3	5,04	50	19,8
4	5,04	56	22,2
5	4,98	69	27,7
6	4,98	59	23,7
7	4,97	62	24,9
8	5,00	60	24,0

Poisson distribution		
Number of samples	8	
Degree of freedom	7	
Mean	58,6	Particles
Standard deviation	5,90	Particles
χ^2 (CHI-Quadrat)	4,15	
Probability	**76**	%
Recovery rate	98	%

Normal distribution		
Number of samples	8	
Mean	23,4	mg/kg
Standard deviation	2,36	mg/kg
rel. Standard deviaton	10,1	%
Horwitz standard deviation	10,0	%
HorRat-value	**1,0**	
Recovery rate	98	%

Microtracer Homogeneity Test
DLA - ptMYS1 Sample B

Weight whole sample	6,81	kg
Microtracer	FSS-rot lake	
Particle size	75 – 300	µm
Weight per particle	2,0	µg
Addition of tracer	22,0	mg/kg

Result of analysis

Sample	Weight [g]	Particle number	Particles [mg/kg]
1	4,99	71	28,5
2	5,03	68	27,0
3	5,02	65	25,9
4	5,02	74	29,5
5	5,03	66	26,2
6	5,04	76	30,2
7	4,98	72	28,9
8	5,02	72	28,7

Poisson distribution		
Number of samples	8	
Degree of freedom	7	
Mean	70,5	Particles
Standard deviation	3,88	Particles
χ^2 (CHI-Quadrat)	1,49	
Probability	**98**	%
Recovery rate	128	%

Normal distribution		
Number of samples	8	
Mean	28,1	mg/kg
Standard deviation	1,55	mg/kg
rel. Standard deviaton	5,5	%
Horwitz standard deviation	9,7	%
HorRat-value	**0,57**	
Recovery rate	128	%

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 (2020)***
PT name	***Mycotoxin-Screening: Aflatoxins, Ochratoxin A, Deoxynivalenol, Zearalenon and Fumonisins in Breakfast Cereals***
*Sample matrix**	***Samples A + B:*** *Cereal muesli with fruits / Ingredients: oatmeal flakes, sugared cranberries, dried fruits (strawberries, raspberries, black currants, bananas, oranges), lemon juice concentrate, maltodextrin, whey powder, cereal flours (wheat, rice, oats, millet, barley, rye, corn), skimmed milk powder, vegetable fat, emulsifier: lecithins, cornflakes, vitamins, minerals 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* – *DLA-sample-numbers (for sample I and II)* – *Limit of detection* – *Assignment incl. Recovery* – *Recovery with the same matrix* – *Method is accredited*
Result submission	*The result submission file should be sent by e-mail to:* ***pt@dla-lvu.de***
Last Deadline	***the latest 03rd July 2020***
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
		AUSTRIA
		AUSTRIA
		Germany
		Germany
		SWITZERLAND
		Germany
		Germany
		CROATIA
		Germany
		HUNGARY
		Germany
		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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19. Verordnung EG/1881/2006 zur Festsetzung der Höchstgehalte für bestimmte Kontamin-anten in Lebensmitteln / Regulation EC/1881/2006 setting maximum levels for cer-tain contaminants in foodstuffs (Version 19.03.2018)
20. ASU §64 LFGB 15.00-2 (Feb. 2014): Bestimmung von Aflatoxin B1 und der Summe von Aflatoxin B1, B2, G1 und G2 in Getreiden, Schalenfrüchten und verwandten Produkten / EN ISO 16050 (2011) Foodstuffs - Determination of aflatoxin B1, and the total content of aflatoxins B1, B2, G1 and G2 in cereals, nuts and derived products - High performance liquid chromatographic method
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in B_1, B_2, G_1 and G_2 in hazelnuts, peanuts, pistachios, figs and paprika powder - High performance liquid chromatographic method with post-column derivatisation and immunoaffinity column cleanup

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