Proficiency test for mycotoxins in the cereals oats and maize

EURLPT-MP06 (2021)

D.P.K.H. Pereboom, M. de Nijs, J.G.J. Mol

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Summary

A proficiency test (PT) for the quantitative determination of multiple mycotoxins in oats flour and maize flour was organised by the European Union Reference Laboratory for mycotoxins & plant toxins in food and feed (EURLMP) between May and September 2021. This PT was carried out by Wageningen Food Safety Research (WFSR) under accreditation (R013, Dutch Accreditation Council RvA, ISO/IEC 17043:2010).

Mycotoxins mandatory for quantification in this this PT were fumonisin B1 (FB1) and fumonisin B2 (FB2) based on the occurrence in the matrix maize, deoxynivalenol (DON) and zearalenone (ZEN) based on their ubiquitous occurrence in both matrices and T-2 toxin and HT-2 toxin based on upcoming regulation in the matrices.

In addition, the NRLs were encouraged, on a voluntary basis, to also analyse the samples for 10 additional mycotoxins: 3-acetyl-deoxynivalenol (3-Ac-DON), 15-acetyl-deoxinivalenol (15-Ac-DON), deoxynivalenol-3-glucoside (DON-3-G), nivalenol (NIV), the *Alternaria* toxins: alternariol (AOH) and alternariol monomethyl ether (AME), and the enniatins: enniatin A (Enn-A), enniatin A1 (Enn-A1), enniatin B (Enn-B) and enniatin B1 (Enn-B1). These mycotoxins were naturally present in the oats flour.

The participants were provided with one oats sample (material A) that was naturally contaminated with DON, T-2 toxin, HT-2 toxin and ZEN and most of the voluntary mycotoxins, but not with FB1 and FB2. The second sample provided was maize (material B) in which DON, FB1, FB2, HT-2 and ZEN were either natural present or spiked to the sample (T-2 toxin was absent).

The provided oats sample (sample A) was naturally contaminated with quantifiable levels of 3-Ac-DON, 15-Ac-DON, DON-3-G, NIV, Enn-A, Enn-A1, Enn-B, Enn-B1, AOH and AME. Material B, maize flour, was naturally contaminated with quantifiable levels of 15-Ac-DON, NIV, AOH, and AME.

The six mandatory mycotoxins and the 10 voluntary mycotoxins were sufficiently homogeneous and stable in both samples prepared during the PT. Each participant received one test sample of each material.

The primary goal was to assess the proficiency of the National Reference Laboratories (NRLs) and OLs that participated. The participants were asked to quantify the above-mentioned mycotoxins in both materials.

The participant's performance was assessed as z-score in both materials for the individual six mandatory mycotoxins. Maximum score was 4 out of 4 for material A and 5 out 5 for material B. False positives (FP) for FB1 and FB2 in material A and for T-2 toxin in material B were considered as unsatisfactory z-scores. False negatives (FN) were considered as unsatisfactory z-scores. Z-scores were calculated for each of the 10 voluntary mycotoxins when 7 or more participants submitted a result, and when uncertainty was below $0.7\sigma_p$. The results are for information only.

Forty-five participants, of which 38 NRLs for mycotoxins and/or plant toxins in food and feed (from 24 EU Member States, the EFTA MS Iceland, Norway and Switzerland and the candidate MS Serbia) and 7 Official Laboratories (all from 5 EU Member States) participated in the PT.

Almost two-third of the participants used one multi-method to cover the mandatory mycotoxins, in all cases using MS/MS. The other third of the participants used use two, three or even four different methods, often measuring the fumonisins separately, or using dedicated immuno-affinity clean-up columns (IAC) for individual or subgroups of mycotoxins, or using dedicated detection (fluorescence). Instrumental measurements were based on LC (only one exception: GC-MS, after derivatisation). Three participants used ELISA for analysis (ZEN, fumonisins).

In this PT the robust mean was used as consensus value. The consensus value based on the participants' results was used as the assigned value. The proficiency of the participants was assessed through z-scores, calculated using the assigned values and a relative target standard deviation of 25%. Characteristics of the PT materials and the outcome of this PT are summarised in Table 1.

A total of 44 participants analysed material A. Of those, 42 participants submitted a result for DON, 33 participants for T-2 toxin, 36 participants for HT-2 toxin and 43 participants for ZEN. 98% of the results for DON, 94% of the results for T-2 toxin, 84% of the results for HT-2 toxin and 88% of the results for ZEN were rated as satisfactory z-scores (|z|≤ 2). Respectively, 0%, 3%, 3% and 12% fell in into the questionable range with 2<|z|<3. Z-scores in the unsatisfactory range with |z|≥ 3 were reported for respectively 2%, 3%, 14% and 0%. Remarkably, 9 participants reported a quantitative result for FB1 and 5 participants also for FB2, which are considered as false positive results in this PT.

All 45 participants reported 2 or more results for material B. Of those, 42 participants submitted a result for DON, 41 participants for FB1, 39 participants for FB2, 38 participants for HT-2 toxin and 44 participants for ZEN. 90% of the results for DON, 90% of the results for FB1, 88% of the results for FB2, 85% of the results for HT-2 toxin and 86% of the results for ZEN were rated as satisfactory z-scores (|z|≤ 2). Respectively, 5%, 2%, 8%, 3% and 5% fell in into the questionable range with 2<|z|<3. Z-scores in the unsatisfactory range with |z|≥ 3 were reported for respectively 5%, 7%, 5%, 13% and 9%.

Of the 45 participant, 18 (40%) achieved optimal performance by detecting all mandatory mycotoxins with correct quantification and absence of false negative or false positive results in the two materials.

Table 1 *Summary of proficiency test materials parameters and participants' performance.*

Mycotoxins (mandatory)	Matrix	Assigned value (µg/kg)	Uncertainty (µg/kg)	Robust RSD_R 1) (%)	No of labs reporting			
					Quant. value	<LOQ	FN	FP
DON	A	3694	129	18.1	42			
	B	692	19.1	14.3	42			
FB1	A							9
	B	3863	217	28.8	41	1	1	
FB2	A							5
	B	222	12.8	28.8	39	3	1	
T-2 toxin	A	17.8	0.762	19.6	33	7		
HT-2 toxin	A	45.2	1.91	20.3	36	4	1	
	B	104	5.29	25.0	38	2	2	
ZEN	A	240	10.4	22.6	43			
	B	88.6	4.65	27.8	44			
Mycotoxins (voluntary)								
3-Ac-DON	A	484	26.6	19.6	20	1	1	
15-Ac-DON	B	66.0	4.15	18.1	13	6	1	
DON-3-G	A	853	78.0	30.2	17			
Enn-A1	A	16.7	1.76	26.6	10	1		
Enn-B	A	96.0	12.9	34.0	10	1	1	
Enn-B1	A	60.7	2.82	11.8	10	1	1	
NIV	A	60.5	9.43	39.5	10	5		
	B	121	8.04	20.0	14	1		

Mycotoxins	Matrix	Assigned value (µg/kg)	z-scores[2)]			Labs out of 45 with Acceptable z-score	
			satisfactory (% of z-scores)	questionable (% of z-scores)	unsatisfactory (% of z-scores)	No[3)]	%[3)]
DON	A	3694	98	0	2	41	91
	B	692	90	5	5	38	84
FB1	B	3863	90	2	7	38	84
FB2	B	222	88	8	5	35	78
T-2 toxins	A	17.8	94	3	3	31	69
HT-2 toxin	A	45.2	84	3	14	31	69
	B	104	85	3	13	34	76
ZEN	A	240	88	12	0	38	84
	B	88.6	86	5	9	38	84
Mycotoxins (voluntary)							
3-Ac-DON	A	484	90	0	10	19	42
15-Ac-DON	B	66.0	79	0	21	11	24
DON-3-G	A	853	71	6	24	12	27
Enn-A1	A	16.7	80	0	20	8	18
Enn-B	A	96.0	64	18	18	7	16
Enn-B1	A	60.7	82	0	18	9	20
NIV	A	60.5	70	20	10	7	16
	B	121	79	14	7	11	24

Matrix: A= Buckwheat flour, B= Maize flour.

1) robust relative standard deviation (interlaboratory RSD based on participants' results).

2) calculated using a fit-for-purpose target RSD for proficiency of 25%. False negatives were counted here as unsatisfactory z-score.

3) the number and percentage here means: analyte determined, method with a sufficiently low LOQ to allow quantification, and obtaining a satisfactory z-score.

1 Introduction

Mycotoxins chosen for quantification in this this PT were deoxynivalenol (DON) and zearalenone (ZEN) based on their occurrence in both matrices, fumonisin B1 (FB1) and fumonisin B2 (FB2) based on their occurrence in the matrix maize, and T-2 toxin (T-2) and HT-2 toxin (HT-2) based on upcoming regulation. All these six mycotoxins are already regulated or up for regulation in both food and feed (Regulation (EC) No 1881/2006; Directive 2002/32/EC; Recommendation 2006/576/EC).

In addition, the NRLs were encouraged on a voluntary basis to also analyse the samples for 3-acetyl-deoxynivalenol (3-Ac-DON), 15-acetyl-deoxinivalenol (15-Ac-DON), deoxynivalenol-3-glucoside (DON-3-G), nivalenol (NIV), the *Alternaria* toxins: alternariol (AOH) and alternariol monomethyl ether (AME), and the enniatins: enniatin A (Enn-A), enniatin A1 (Enn-A1), enniatin B (Enn-B) and enniatin B1 (Enn-B1). Monitoring of *Alternaria* toxins is currently required by Recommendation (EU) 2022/553 which became in place on April 2022. The acetyl-DONs, DON-3-G have a monitoring recommendation by EFSA and insights in analytical performance is needed for these substances.

Proficiency testing is conducted to provide participants with a powerful tool to evaluate and demonstrate the reliability of the data that are produced by the laboratory. Proficiency testing is an important requirement and is demanded by the ISO/IEC 17025:2017 [6]. Organisation of proficiency tests (PT) is one of the tasks of the European Union Reference Laboratories (EURLs) [7]. Here the primary goal is to assess the proficiency of the National Reference Laboratories (NRLs). To facilitate NRLs in their task, official laboratories (OLs) can also participate, in consultation with their NRL.

2 PT Material

2.1 Scope of the PT

This proficiency test focused on multiple mycotoxins in cereal matrices (oats flour and maize as representative matrices for food and feed), of which DON, FB1, FB2, T-2, HT-2 and ZEN were mandatory to be analysed, and 3-Ac-DON, 15-Ac-DON, DON-3-G, NIV, AOH, AME, Enn-A, Enn-A1, Enn-B and Enn-B1 were voluntary. The oats material (A) was a naturally contaminated material used as such. The maize material contained low levels of several mycotoxins and was spiked to reach target concentrations (see Table 2) taking the regulatory limits and commonly found concentrations into account.

Table 2 *Target concentrations µg/kg of the mycotoxins spiked to material B, maize.*

Mycotoxins	**Material B** **Target concentrations (µg/kg)**
DON	750
FB1	750
FB2	250
T-2 toxin	50
HT-2 toxin	50
ZEN	75

2.2 Material preparation

For preparation of the two PT materials A and B, respectively, oats flour and maize flour were used. The materials were milled using a centrifugal mill (ZM 200, Retsch, Haan) to obtain a particle size of 500 µm. The starting materials were naturally contaminated with several mycotoxins. The oats flour (material A) was naturally contaminated with all the mandatory and voluntary mycotoxins except the fumonisins. In case of the maize (material B), the material was naturally contaminated with DON, fumonisins and HT-2 toxin and ZEN, and the acetyl DONs, DON-3-G, NIV and Enn-B. For material B, the concentrations for DON, FB1, FB2, HT-2, ZEN were artificially increased by spiking.

For material A, 4.5 kilograms were used as such. For material B, 4.5 kilograms were first fortified by adding a solution of the mycotoxins in acetonitrile/water (1:1), aiming at the levels as presented in Table 2. The oats flour and maize flour were respectively mixed with 6.9 and 6.5 litres water and homogenised using an industrial mixer (brand Topcraft) according to in-house standard operating procedure [9]. The fortified slurry was freeze-dried and the resulting material was homogenised in a Stephan cutter UM 12, and stored in the freezer until use.

2.3 Sample identification

After homogenisation, materials A and B were divided into sub-portions of approximately 50 grams and stored in polypropylene, airtight closed containers at <-18 °C until use.

The samples for the participants were randomly selected and coded using a web application designed for proficiency tests. The code used was "2021/EURLPT MP/mycotoxins/xxx", in which the three-digit number of the code was automatically generated by the WFSR Laboratory Quality Services web application. One sample set was prepared for each participant. Each sample set consisted of one randomly selected sample of material A and one of material B. The codes of the samples for each sample set are shown in Annex 2. The samples for homogeneity and stability testing were also randomly selected out of materials A and B.

2.4 Homogeneity study

To verify the homogeneity of the PT materials, the content of ten containers material A and ten containers material B were analysed in duplicate for the mycotoxins.

Method in brief, the mycotoxins were extracted from the homogenised sample material after addition of water, by shaking with acidified acetonitrile. After a salt-induced phase partitioning step with magnesium sulphate followed by centrifugation, an aliquot of the acetonitrile phase was diluted with water. Analysis was done by high performance liquid chromatography coupled with tandem mass spectrometry (LC-MS/MS).

The homogeneity of both materials was evaluated according to the International Harmonized Protocol for Proficiency Testing of Analytical Laboratories [11] and ISO 13528:2015 [12]. Both materials proved to be sufficiently homogeneous for this PT. The results of the homogeneity study, grand means with the corresponding RSD_r, are presented in Table 3. The statistical evaluation of materials A and B is presented in Annex 3.

Table 3 *Concentrations of the mycotoxins in material A and B as obtained during the homogeneity testing [1)].*

	Material A		**Material B**	
Compound	**Conc. (µg/kg)**	**RSD_r (%)**	**Conc. (µg/kg)**	**RSD_r (%)**
DON	3897	3.3	660	2.9
FB1			3928	3.3
FB2			209	3.4
T-2	21.4	5.5	2.22	6.3
HT-2	54.5	5.6	139	3.1
ZEN	128	7.7	66.0	2.6
3-Ac-DON	434	2.4	15.7	8.9
15-Ac-DON	17.1	11	81.0	4.5
DON-3-G	818	4.8	30.4	20.0 [1)]
NIV	51.8	5.2	228	23 [1)]
AOH	49.0	17[2)]		
AME	18.4	5.8		
Enn-A	2.48	4.2		
Enn-A1	16.0	3.6		
Enn-B	95.2	3.5	5.64	4.9
Enn-B1	53.1	3.9		

1) Quantification based on solvent standards, concentrations are therefore estimates.

2) Method RSD_r too high to be suited for homogeneity assessment. Based on data for other/related mycotoxins, the materials were nevertheless considered homogeneous also in these cases.

2.5 Stability of the materials

The stability of the mycotoxins in the PT materials was assessed according to [11, 12]. On June 21st, 2021, the day of distribution of the PT samples, six randomly selected containers of material A and B were stored at <-70 °C. Under these conditions it is assumed that the mycotoxins are stable in the materials. In addition, six samples of each material were stored at <-20 °C.

On September 7th, 2021, 78 days after distribution of the samples, six samples of materials A and B, stored at <-70 °C and <-20 °C, were analysed in one batch. For each set of test samples, the average of the results and the standard deviation were calculated.

It was determined whether a consequential instability of the analytes had occurred [11, 12] in the materials stored at <-20 °C. A consequential instability is observed when the average value of an analyte in the samples stored at <-20 °C is more than $0.3\sigma_P$ below the average value of the analyte in the samples stored at <-70 °C. If so, the instability has a significant influence on the calculated z-scores.

The results of the stability of materials A and B are presented in Annex 4. None of the tested storage conditions caused a consequential difference for the analytes in both materials, except for AME. The average concentration at <-20 °C was higher than the average concentration of the samples stored at <-70 °. The concentration showed an increase of 14% (from 35.5 to 40.5 µg/kg). This increase in concentration was not taken into account in the calculation of the z-scores.

Note: the approach for quantification used in the homogeneity and stability analyses differed. While this did not affect the outcome of the assessments as such, it makes the data less suited for direct comparison of concentrations reported in the homogeneity and stability tables.

3 Organisational details

3.1 Participants

This proficiency test focused on the mandatory mycotoxins DON, FB1, FB2, T-2, HT-2 and ZEN and voluntary mycotoxins 3-Ac-DON, 15-Ac-DON, DON-3-G, NIV, AOH, AME, Enn-A, Enn-A1, Enn-B and Enn-B1 in food and feed, using oats flour and maize flour as representative matrices. Invitations to the NRL network were sent out on May 31th, 2021 (Annex 5). Forty-five participants registered for the PT (Annex 1) and reported their results. This included 38 NRLs from 24 EU Member States, plus Iceland, Norway, Serbia, Switzerland, and 7 Official Laboratories (all from 5 EU Member States). Each participant was free to use their method of choice reflecting their routine procedures. The participants were asked to report results through a web application designed for proficiency tests as well as to fill in a questionnaire, where it was asked to provide detailed information on the analytical method used.

3.2 Material distribution and instructions

Each participant received a randomly assigned laboratory code, generated by the web application. The sets of samples with the corresponding number, consisting of two coded samples (Annex 2) were sent to the PT participants on June 21st, 2021. The sets of samples were dispatched by courier to the participants in insulation boxes containing dry ice. The participants were asked to store the samples at <-20 °C and to analyse the samples according to their routine practice. As reported by participants, most of the parcels (30) were received within 48 hours after dispatch. Six participants received the parcel after 2 days. All samples were received in good order.

The samples were accompanied by a letter with instructions for the requested analysis (Annex 6) and an acknowledgement of the receipt form. In addition, by e-mail, each participant received instructions on how to use the web application to report the results. The questionnaire was intended to gather additional information on limits of quantification (LOQs), method recovery estimates (%) and other method-related aspects (e.g. extraction and clean-up, chromatographic and detection conditions, calibration approach) to investigate individual and/or general patterns on the submitted results.

A single analysis result for the mycotoxins in each sample was requested. The deadline for submitting the quantitative results was September 6th, 2021, allowing the participants 11 weeks for analysis of the test samples. All results, except six, were submitted within the deadline.

4 Evaluation of results

The statistical evaluation of the submitted results was carried out according to the International Harmonized Protocol for the Proficiency Testing of Analytical Laboratories [11], elaborated by ISO, IUPAC and AOAC, and ISO 13528:2015 [12] in combination with the insights published by the Analytical Methods Committee [4,5] regarding robust statistics.

The evaluation of results was based on assigned values and the standard deviation for proficiency assessment (σ_P). From this, z-scores were calculated to classify the participants' performance. Detailed information on the methods used for the statistical evaluation can be found in the background document 'EURL-MP-background doc_001 (v1) 'Performance assessment in proficiency tests organised by the EURL mycotoxins & plant toxins in food and feed' available from the EURL mycotoxins & plant toxins website[1].

4.1 Calculation of the assigned value

The robust mean was used as consensus value in this PT. The consensus value based on the participants' results (all participants, both NRLs and OLs) was used as the assigned value. The values and their uncertainties are summarised in Table 1 in the Summary section.

Assigned values were calculated when 7 or more results were submitted by the participants and when uncertainty was below 0.7σ_p. For the obligatory mycotoxins, assigned values were established for 4 of the 6 analytes in material A (DON, T2, HT2, ZEN) and 5 of the 6 analytes in material B (DON, FB1, FB2, HT2, ZEN). For the voluntary mycotoxins (see Table 3), assigned values could be established for 6 of the 10 mycotoxins in material A (3-Ac-DON, DON-3-G, NIV, ENNA1, ENN-B1, Enn-B2) and 2 of the 5 mycotoxins in material B (15-Ac-DON and NIV).

4.2 Standard deviation for proficiency assessment (σ_P)

A fixed relative target standard deviation for proficiency assessment (σ_P) of 25% was used, irrespective the analyte, matrix or concentration. This generic fit-for-purpose value is considered to reflect current analytical capabilities and the best practises for mycotoxin and plant toxin determination in food and feed. The rationale behind this is provided in the before mentioned EURL-MP-background doc_001.

4.3 Quantitative performance (z-scores)

For evaluation of numerical results submitted by each participant, z-scores were calculated based on the assigned value, its uncertainty, and the standard deviation for proficiency assessment (σ_P). In cases when the uncertainty of the assigned value was negligible and no instability of the analytes in the PT material was observed, z-scores were calculated using the following equation:

$$z = \frac{x - C}{\sigma_p} \qquad \text{Equation 1}$$

where:

z = z-score;
x = the result of the laboratory;
C = assigned value, here the consensus value;
σ_P = standard deviation for proficiency assessment.

1 Website EURLMP

The z-score compares the participants' deviation from the assigned value, taking the target standard deviation accepted for the proficiency test into account, and is interpreted as indicated in Table 4.

Table 4 *Classification of z-scores.*

$\lvert z_a \rvert \leq 2$	Satisfactory
$2 < \lvert z_a \rvert < 3$	Questionable
$\lvert z_a \rvert \geq 3$	Unsatisfactory

If the uncertainty of the assigned value and, if applicable, instability of the analyte in the PT material, is not negligible, then this is taken into account in the determination of the z-score. If applicable, this is indicated by assigning a z'-, z_i-or z_i'-score. For details see the background document 'EURL-MP PT performance assessment' on the EURL-MP website.

In this PT, the uncertainty of the assigned value for DON-3-G, NIV, Enn-A1 and Enn-B in material A and DON-3_G and NIV in material B were not negligible (but still $<0.7\sigma_P$) and, therefore, this was taken into account in the assignment of the z-score (z'). In five cases, 15-Ac-DON, AOH and AME in material A, and T-2 and DON-3-G in material B, no statistical evaluation was possible because of the high dispersion of the results (uncertainty exceeded 0.7 σ_P).

4.4 Evaluation of non-quantified results

In cases, where participant(s) reported '<[value]' or 'not detected' (nd) (i.e. below their limit of quantification (LOQ)), 'proxy-z-scores' were calculated to assess possible false negatives.

A proxy-z-score was calculated by using equation IV and equation V of the background document 'EURL-MP-background doc_001' (for details see the EURL-MP website), using the reported LOQ value as a result. This was only done when the LOQ was equal to or lower than the assigned value. Proxy-z-scores are for information only and indicated as a value between brackets.

Other types of reported results, e.g. 'detected' or 'not detected', without specification of LOQ, were excluded from the evaluation. In these cases, the participant was considered to have no quantitative method available for the applicable analyte/matrix.

4.5 False positive and false negative results

When an analyte is present in the material, i.e. an assigned value has been established, and the participant reports the analyte as below a specified LOQ, an assessment is made to judge whether such results should be classified as a false negative. This is the case when the proxy-z-score is <-2. False negatives are indicated as 'FN'. False negatives are to be interpreted as unsatisfactory performance.

A false positive is a quantitative result reported by the participant while the toxin is: i) not detected in the PT material by the organiser, and/or ii) not detected by most of the other participants. A threshold may apply, below which results are not considered as false positives, i.e. when the analyte concentration is below the LOQ of the organiser and/or most of the participants. This will be decided on a case-to-case basis. Since there is no assigned value, no z-score can be calculated. False positives will be indicated in the report as 'FP'. False positives should be interpreted as unsatisfactory performance.

5 Performance assessment

5.1 Scope and LOQ

This PT was dedicated to six mycotoxins that are regulated or up for regulation in food and feed (to be analysed mandatory) and ten not yet regulated mycotoxins (voluntary analysis). Annex 7 summarises the quantitative scopes of each participant, with an indication of the LOQs for each of the mycotoxins.

The scope of the laboratories is summarised in Figure 1. In most cases, the six mandatory mycotoxins were covered by the participants. Six participants (1x NRL food, 3x NRL feed, 2x NRL food & feed of which one EFTA NRL) did not include 2-4 of the mandatory mycotoxins. Eighteen participants did not measure any mycotoxin from the voluntary scope, while for several of these mycotoxins EFSA monitoring recommendations are in place, and/or maximum limits or guidance values are foreseen. Only four included all mycotoxins in the scope of their analysis. From this it is concluded that pro-active inclusion of 'new' mycotoxins in the scope of the participants' laboratories is challenging and/or does not have a high priority.

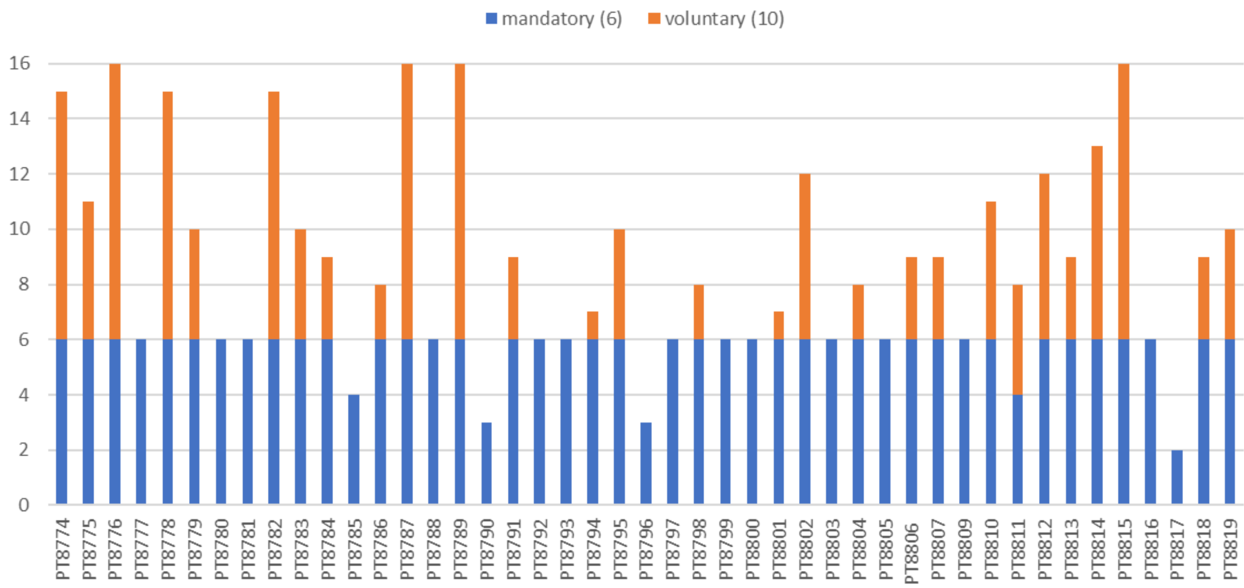

Figure 1 *Mandatory and voluntary mycotoxins included in the scope of the participants.*

Figure 2 illustrates the coverage per mycotoxin. As already indicated above, the regulated mycotoxins are well covered and measured by >90% of the participants. The DON-derivatives are measured by 40-50% of the participants, nivalenol by one-third. The *Alternaria* toxins and enniatins only by 25%.

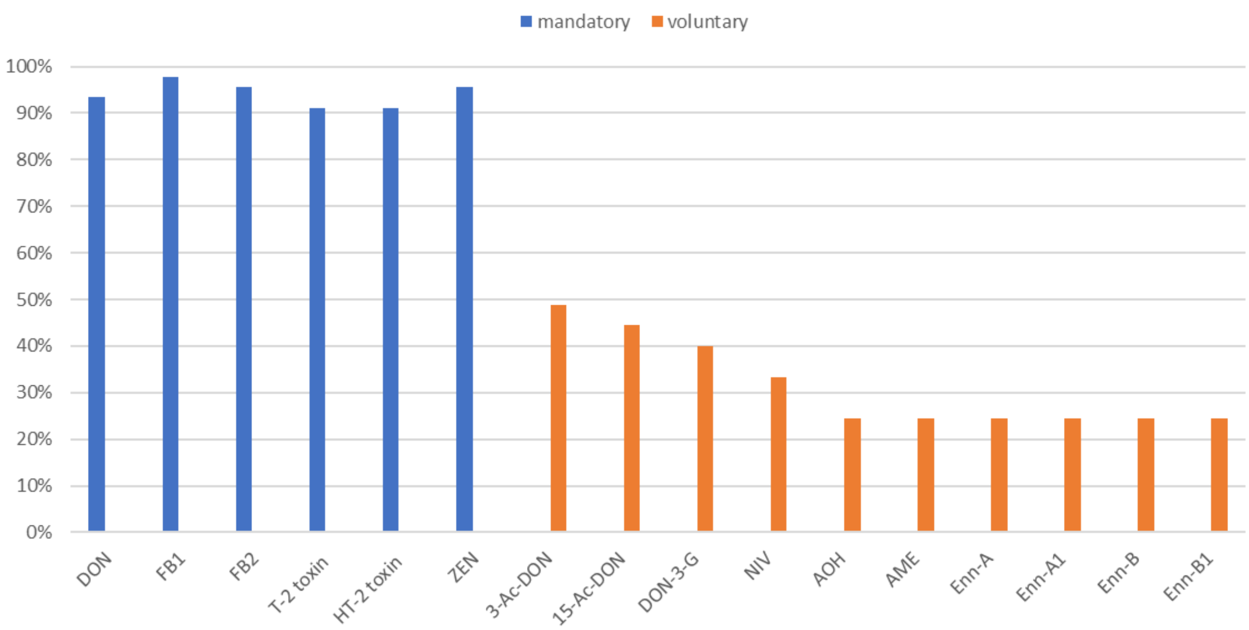

Figure 2 *Percentage of participants measuring the mycotoxins from this PT.*

The LOQs provided by the participants varied widely, both between mycotoxins, and for the individual mycotoxins between the participants (see Table 5a/b). The reasons for this include: intrinsic sensitivity of the different mycotoxins in instrumental detection (MS, fluorescence, UV), differences in sensitivity of the instruments and conditions used, and the definition and way of determination of the LOQ used by the participant. For the mandatory mycotoxins, the LOQs are adequate (≤0.5*ML) for compliance testing in almost all cases (here MLs for baby food are not considered). For data generation for risk assessment, the median LOQs appear adequate in most cases, meaning that data from half of the participants would be useful, and from the other half less useful (high probability of lot of left censored data) for risk assessment purposes.

Table 5a *LOQs as reported by the laboratories for the mandatory mycotoxins.*

	LOQ reported by participants (µg/kg)			Maximum level, or Recommended level, or Guidance level (µg/kg)	
Mycotoxin	**min**	**max**	**median**	**food (oats/maize)**	**feed (oats/maize)**
DON	8.5	450	50	750-1,750/750-1,750	8,000/12,000
FB1	0.88	1000	42.5	-/1,000-4,000	-/60,000
FB2	0.88	1000	30		
T-2 toxin	0.88	83	8.5	200-1,000/100-200	2,000/500
HT-2 toxin	0.88	125	10		
ZEN	1.75	80	10	75-100/100-350	2,000/3,000

Table 5b *LOQs as reported by the laboratories for the voluntary mycotoxins.*

	LOQ reported by participants (µg/kg)		
	min	**max**	**median**
3-Ac-DON	8	200	50
15-Ac-DON	8	200	50
DON-3-G	8	200	27
NIV	7	100	47
AOH	0.1	30	4
AME	0.1	10	3.5
Enn-A	0.1	25	4
Enn-A1	0.1	25	4
Enn-B	0.1	25	4
Enn-B1	0.1	25	4

5.2 Analytical methods

All participating laboratories were asked to fill in a questionnaire addressing their accreditation, the conditions used for sample preparation, chromatographic separation, detection, quantification and calibration (Annex 8). Fifteen participants did not complete the questionnaire. Five of these participants provided very limited information about the analysis and analytical method via the web application.

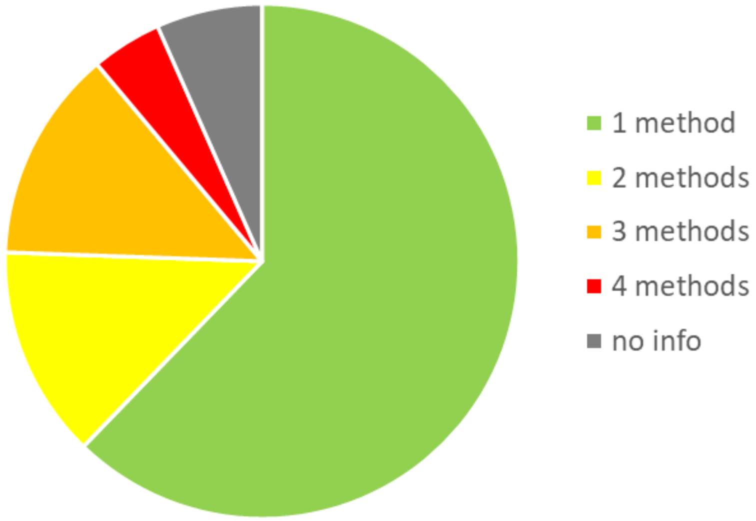

Figure 3 *Number of methods used for determination of the mandatory mycotoxins.*

The methods used were mostly (73%) in-house developed and validated methods. 65% of the methods were accredited, 21% not (for the remaining the status was not specified).

Almost two-third of the participants used one multi-method to cover the mandatory mycotoxins. In all these cases MS/MS (one participant HRMS) was used as detection method. The other third of the participants used two, three or even four different methods. In case of two methods, it were often the fumonisins that were measured separately. In other cases, multiple methods were used as a consequence of using dedicated immunoaffinity clean-up columns (IAC) for individual or subgroups of mycotoxins, or dedicated detection (fluorescence). Instrumental measurements were based on LC (only one exception: GC-MS, after derivatisation). Three participants used ELISA for analysis (ZEN, fumonisins).

The voluntary mycotoxins were typically included in the LC-MS/MS-based multi-methods used for the mandatory mycotoxins. In a few cases, either a dedicated extraction, clean-up or LC-MS/MS method was applied.

The sample amount extracted varied from 1-25 g, median 5 g. Acetonitrile, often acidified and with 10-25% of water, was mostly used for extraction. When a separate method for fumonisins was used, mixtures of acetonitrile/water/methanol were typically used. Extraction was done using blenders or by shaking, times varied from 1 min to 2 hours (median 40 min).

In roughly 45% of the methods (typically when LC-MS/MS was used for analysis of the extracts), no clean-up (other than dilution or filtration) was used, or only a liquid-liquid partitioning (QuEChERS, or defatting with hexane). In another 40% (typically when using LC-UV or LC-fluorescence) an IAC clean-up was used. In the remaining methods, an SPE step was used for clean-up.

For the participants using LC-MS/MS-based methods, more than half (63%) used isotope labelled internal standards for quantification, added to the final extract (46%), before clean-up (6%) or before extraction (10%). Quantification was then done based on standards prepared in solvent or matrix. For other LC-MS/MS based methods, various approaches for quantification were used, including standard addition to either sample or extract, and matrix-matched calibration. For LC-UV and LC-fluorescence based methods, quantification was usually done using solvent standards.

5.3 Performance

The quantitative performance was assessed through z-scores when 7 or more results were submitted by the participants and when uncertainty did not exceed $0.7\sigma_p$. The individual z-scores obtained by each participant, including their graphical representation, for the mycotoxins in material A (oats) and B (maize) are summarised in Annex 9 and 10, respectively. A summary of the performance of the participants in this PT for the mandatory mycotoxins is provided in Annex 11.

A summary of the statistical evaluation of the PT results is presented in Table 6. The table includes all relevant parameters: the assigned value (A), the uncertainty of the assigned value (u), the standard deviation for proficiency assessment (σ_p) and the robust (relative) standard deviation, based on participants' results.

Table 6 *Parameters of the mycotoxins and summary for material A (oats)*.*

	DON	T-2	HT-2	ZEN	3-Ac-DON	DON-3-G	NIV	Enn-A1	Enn-B	Enn-B1
A (µg/kg)	3694	17.8	45.2	240	484	853	60.5	16.7	96.0	60.7
u (µg/kg)	129	0.762	1.91	10.4	26.6	78.0	9.43	1.76	12.9	2.82
σ_p (µg/kg) (25%)	924	4.46	11.3	60.0	121	213	15.1	4.18	24.0	15.2
$u>0.3\sigma_p$	No	No	No	No	No	Yes	Yes	Yes	Yes	No
robust σ (µg/kg)	667	3.50	9.18	54.4	95.1	257	23.9	4.45	32.7	7.13
robust σ (%)	18.1	19.6	20.3	22.6	19.6	30.2	39.5	26.6	34.0	11.8
# reported	42	40	40	43	21	17	15	11	11	11
"<", nd		7	4		1		5	1	1	1
# quantitative results	42	33	36	43	20	17	10	10	10	10
$\|z\|\leq 2$	41	31	31	38	19	12	7	8	7	9
$2<\|z\|<3$	0	1	1	5	0	1	2		2	
$\|z\|\geq 3$	1	1	4		1	4	1	2	1	1
FN			1		1				1	1
satisfactory z-scores (%)	98	94	84	88	90	71	70	80	64	82

* Fumonisins were not present in oats. For 15-Ac-DON, alternariol and alternariol-monomethyl ether no consensus value could be obtained (the uncertainty of the robust mean was too high). For Enniatin A the number of participant's results was too low.

FN = False negative.

nd = not detected.

Table 7 *Parameters of the mycotoxins and summary for material B (maize).*

	DON	**FB1**	**FB2**	**HT-2**	**ZEN**	**15-Ac-DON**	**NIV**
A (µg/kg)	692	3863	222	104	88.6	66.0	121
u (µg/kg)	19.1	217	12.8	5.29	4.65	4.15	8.04
σ_p (µg/kg) (25%)	173	966	55.6	26.0	22.2	16.5	30.1
u>0.3σ_p	No	No	No	No	No	No	Yes
robust σ (µg/kg)	99.3	1114	64.0	26.1	24.7	12.0	24.1
robust σ (%)	14.3	28.8	28.8	25.0	27.8	18.1	20.0
# reported	42	42	42	40	44	19	15
“<“, nd		1	3	2		6	1
# quantitative results	42	41	39	38	44	13	14
$\lvert z\rvert \leq 2$	38	38	35	34	38	11	11
$2<\lvert z\rvert<3$	2	1	3	1	2		2
$\lvert z\rvert \geq 3$	2	2	1	3	4	2	1
FN		1	1	2		1	
satisfactory z-scores (%)	90	90	88	85	86	79	79

* Alternariol, alternariol-monomethyl ether, enniatins A, A1 and B1 were not present (<LOQ) in the maize material. For T2, and DON-3-G no consensus value could be obtained (the uncertainty of the robust mean was too high). For 15-Ac-DON the number of participant’s results was too low.

FN = False negative.

nd = not detected.

In the two materials, consensus values with acceptable uncertainty were obtained for 17 mycotoxin/matrix combinations. Other mycotoxins were either not present in the material, or the number of submitted results was too low, or the variability within the results was too high to establish a meaningful consensus value.

For the mandatory mycotoxins, the percentage of satisfactory z-scores were high (≥84%). It should be noted that in material A (oats), nine participants reported false positives for the fumonisins. The cause of this will be inquired in the follow up of this PT (causes may include carry-over in injection in instrumental analysis). For the voluntary mycotoxins, the percentage of satisfactory z-scores was (slightly) worse (64-82%).

5.4 Robust relative standard deviation

The robust relative standard deviation (RSD_R) was calculated according to ISO13528:2015 [12] (Algorithm A) for informative purposes only. In this study it was used as a good estimation of the interlaboratory variability. The RSD_R values are included in Annex 10 and 11, in Table 6 (Section 5.3) and in Table 1 (Summary section).

The robust standard deviation (RSD_R) of the reported results was below or around the target standard deviation for proficiency (25%). For the mandatory mycotoxins, one very high RSDR (86%) was observed for T2. This concerned a low concentration (approx. 7 µg/kg in maize) which was close or below the LOQ of most of the participants. For the voluntary mycotoxins, high RSD_Rs were observed for 15-Acetyl-DON, DON-3-G, Nivalenol, and the *Alternaria* toxins (concentrations ranging from 24-160 µg/kg). In all cases, the number of quantitative results reported was low (8-11). An overview of the RSD_R obtained in this PT is shown in Figure 4.

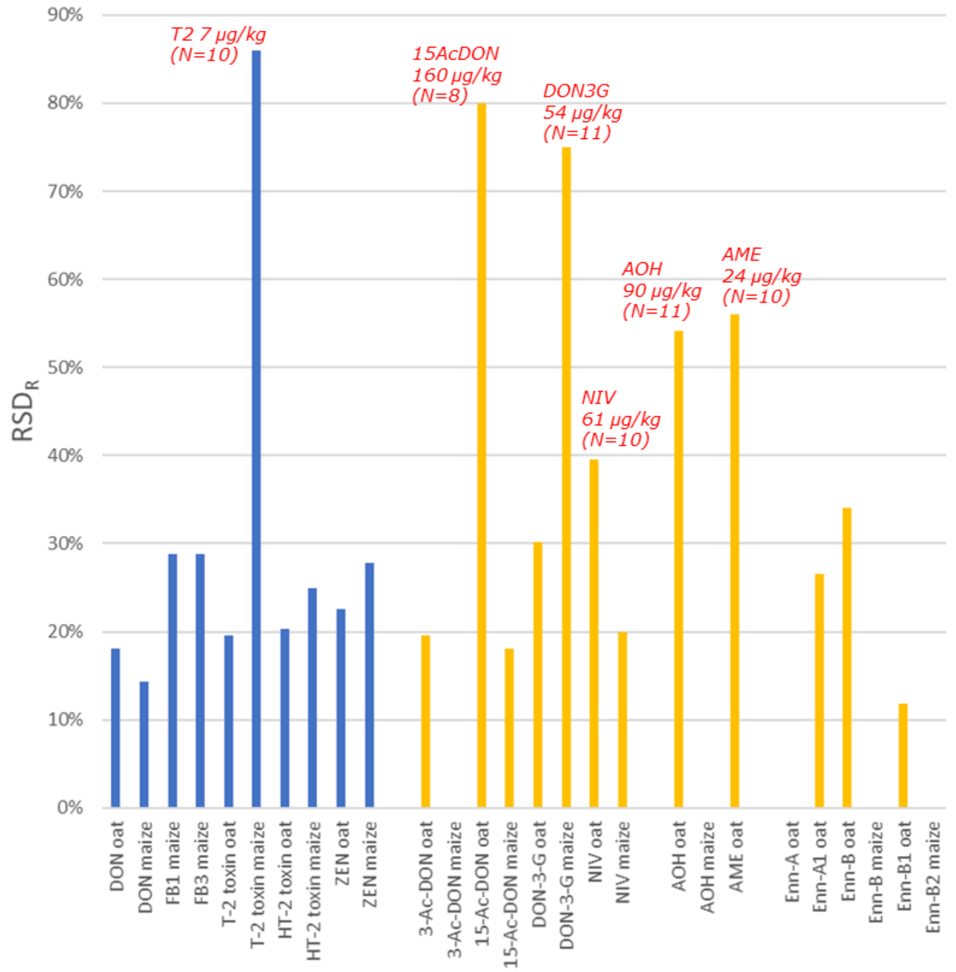

Figure 4 *Robust standard deviation (interlaboratory reproducibility) for the mycotoxins in oats and maize from the current PT.*

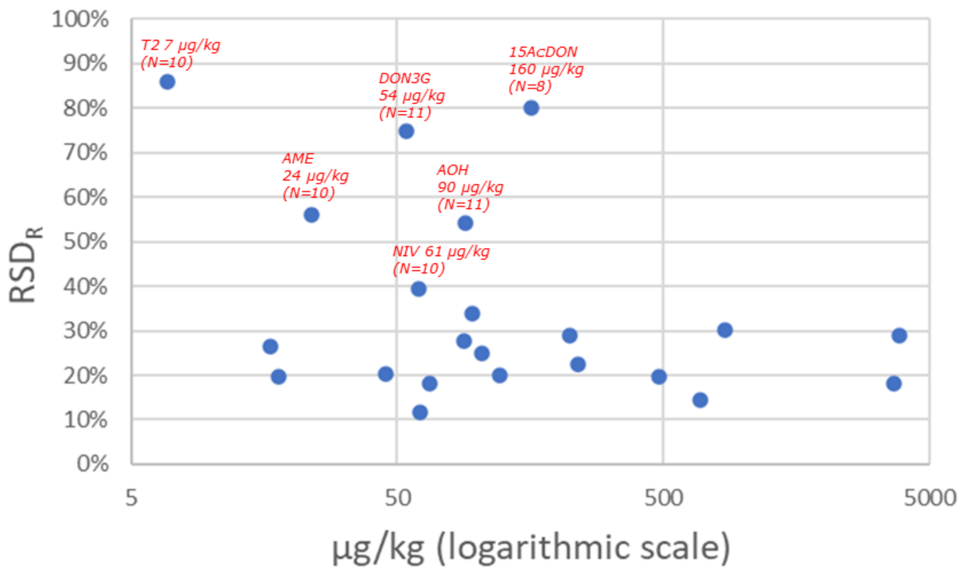

Figure 5 *RSD_R versus concentration of mycotoxins in the samples.*

In Figure 5 the RSD_R is plotted as function of the concentration of the mycotoxins in the sample. In line with earlier observations, no clear relationship can be observed. It is postulated that for T-2 toxin the concentration was close to the actual detection limits of the participants' methods (confirmed by the high number of participants reporting <LOQ). In the other cases, the mycotoxins were analysed by only a limited number of participants. In this case, a few deviating results result in a high RSD_R. A wider implementation and routine application may improve this.

6 Conclusions

Forty-five participants, 38 NRLs (from 24 EU Member States plus Iceland, Norway, Switzerland and Serbia) and 7 OLs participated in the EURLPT-06 on the quantitative determination of six mandatory (regulated) and 10 voluntary (not yet regulated) mycotoxins in oats and maize.

While the mandatory mycotoxins were determined by most of the participants (>90%), the voluntary mycotoxins were only reported by 25-50% of the participants.

A range of analytical methods were used. LC-MS/MS based multi-methods were dominating, but multiple single-mycotoxin or group-specific methods were also used.

For the mandatory mycotoxins satisfactory results were reported by most of the participants (84-98%), although a relatively high incidence of false positives was observed for fumonisins in oats. A total of 18 (40%) participants achieved optimal performance by detecting the mandatory mycotoxins with correct quantification and absence of false negative or false positive results in the two materials.

For the voluntary mycotoxins, satisfactory results were reported by 64-82% of the participants.

The interlaboratory reproducibility (RSD_R) was below or around the target relative standard deviation for proficiency in most cases. Exceptions were observed mainly for the voluntary mycotoxins.

Overall, it can be concluded that the regulated mycotoxins are well covered and determined with satisfactory performance in most cases. Not yet regulated mycotoxins are not well covered by the participants, but if included, also here satisfactory performance is obtained in most cases.

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[12] ISO 13528:2015. Statistical methods for use in proficiency testing by inter-laboratory comparison, 1st edition.

Annex 1 List of participants

Country	Organisation
AUSTRIA*	AGES Austrian Agency for Health and Food Safety
AUSTRIA***	Romer Labs Diagnostic GmbH
BELGIUM*	CER Groupe
BULGARIA*	Bulgarian Food Safety Agency
BULGARIA***	RVS RUSE LTD
CROATIA*	A. Stampar Teaching Institute of Public Health
CYPRUS*	Feeding Stuffs Quality Control Laboratory - Analytical Laboratories Section
CYPRUS*	STATE GENERAL LABORATORY
CZECH REPUBLIC*	UKZUZ (Central Institute for Supervising and Testing in Agriculture
CZECH REPUBLIC*	Czech Agriculture and Food Inspection Authority (CAFIA)
DENMARK*	Danish Veterinary and Food Administration
ESTONIA*	AGRICULTURAL RESEARCH CENTRE
FINLAND*	Finnish Customs Laboratory
FINLAND*	Finnish Food Authority
FRANCE*	SCL
FRANCE***	LABOCEA
GERMANY***	State Institute for Chemical and Veterinary Analysis of Food (CVUA) Sigmarignen
GERMANY**	Eurofins WEJ Contaminants representing NRL Iceland
GERMANY***	Lower Saxony State Office for Consumer Protection and Food Safety (LAVES)
GERMANY***	Chemisches und Veterinaruntersuchungsamt Rheinland
GERMANY*	Federal Institute fur Risk Assessment (BfR)
GREECE*	General Chemical State Laboratory
HUNGARY*	National Food Chain Safety Office
HUNGARY*	National Food Chain Safety Office
IRELAND*	The Public Analyst’s Laboratory
IRELAND*	The State Laboratory
ITALY***	IZSLER
ITALY*	Istituto Superiore di Sanita
LATVIA*	Institute of Food Safety, Animal Health and Environment “BIOR”
LITHUANIA*	National Food and Veterinary Risk Assessment Institute
LUXEMBOURG*	Laboratoire National de Santé
NORWAY**	Norwegian Veterinary Institute
POLAND*	National Institute of Public Health - National Institute of Hygiene
POLAND*	National Veterinary Research Institute
PORTUGAL*	National Institute of Agrarian and Veterinary Research
ROMANIA*	Institute for Hygiene and Veterinary Public Health
ROMANIA*	Directia Sanitara Veterinara si pentru Siguranta Alimentelor (DSVSA) Bucuresti
SERBIA*	SP LABORATORIJA A.D.
SLOVAKIA*	State veterinary and food institute Dolny Kubin Veterinary and food institute in Kosice
SLOVAKIA*	Regional Public Health Authority in Poprad (RUVZ)
SLOVENIA*	National Laboratory of Health, Environment and Food (NLZOH, Slovenia)
SLOVENIA*	University of Ljubljana, Veterinary Faculty, National Veterinary Institute
SWEDEN*	National Food Agency
SWEDEN*	National Veterinary Institute, SVA
SWITZERLAND**	Kantonales Laboratorium Bern

* National Reference Laboratory (NRL) of EU Member State.

** National Reference Laboratory (NRL) of the European Free Trade Association (Eurofins WEJ Contaminants = Iceland).

*** Official Laboratory (OL).

Annex 2 Codification of the samples

Participants code	Material A*	Material B*
PT8774	482	674
PT8775	664	291
PT8776	142	632
PT8777	660	507
PT8778	662	502
PT8779	982	698
PT8780	809	331
PT8781	439	886
PT8782	160	968
PT8783	821	217
PT8784	688	598
PT8785	430	537
PT8786	956	182
PT8787	414	949
PT8788	936	819
PT8789	497	481
PT8790	948	628
PT8791	979	485
PT8792	710	607
PT8793	204	996
PT8794	249	512
PT8795	138	113
PT8796	236	346
PT8797	432	841
PT8798	683	272
PT8799	436	242
PT8800	350	981
PT8801	618	654
PT8802	772	863
PT8803	891	644
PT8804	894	908
PT8805	566	711
PT8806	986	545
PT8807	910	646
PT8809	726	787
PT8810	356	170
PT8811	677	812
PT8812	523	330
PT8813	496	535
PT8814	998	251
PT8815	815	601
PT8816	254	725
PT8817	732	790
PT8818	472	115
PT8819	919	736

* All sample codes start with 2021/EURLPT MP/mycotoxins/.

Annex 3 Statistical evaluation of the homogeneity data

Sample No.	DON in A (µg/kg)	
	Replicate 1	Replicate 2
Hom/A001	3913	3919
Hom/A002	3778	3785
Hom/A003	4234	3893
Hom/A004	3907	3811
Hom/A005	3793	3784
Hom/A006	4104	3865
Hom/A007	4027	3825
Hom/A008	3919	4101
Hom/A009	3850	3816
Hom/A010	3888	3729
Grand mean	3897	
Cochran's test		
C	0.410	
Ccrit	0.602	
C < Ccrit?	NO OUTLIERS	
Target s = σ_P	974	
s_x	98.8	
s_w	119	
s_s	51.7	
Critical= 0.3 σ_P	292	
s_s < critical?	ACCEPTED	
s_w < 0.5 σ_P?	ACCEPTED	

s_x = Standard deviation of the sample averages.

s_w = Within-sample standard deviation.

s_s = Between-sample standard deviation.

Sample No.	T-2 in A (µg/kg)	
	Replicate 1	Replicate 2
Hom/A001	20.9	20.8
Hom/A002	23.7	20.3
Hom/A003	20.6	21.2
Hom/A004	21.9	24.4
Hom/A005	20.3	20.9
Hom/A006	21.6	20.1
Hom/A007	22.3	21.0
Hom/A008	21.4	21.5
Hom/A009	21.1	22.7
Hom/A010	20.5	20.2
Grand mean	21.4	
Cochran's test		
C	0.457	
Ccrit	0.602	
C < Ccrit?	NO OUTLIERS	
Target s = σ_P	5.34	
s_x	0.850	
s_w	1.13	
s_s	0.286	
Critical= 0.3 σ_P	1.60	
s_s < critical?	ACCEPTED	
s_w < 0.5 σ_P?	ACCEPTED	

s_x = Standard deviation of the sample averages.

s_w = Within-sample standard deviation.

s_s = Between-sample standard deviation.

Sample No.	HT-2 in A (µg/kg)	
	Replicate 1	Replicate 2
Hom/A001	54.5	52.3
Hom/A002	54.9	56.1
Hom/A003	55.5	52.5
Hom/A004	56.1	54.3
Hom/A005	49.8	49.9
Hom/A006	55.0	53.7
Hom/A007	58.9	55.4
Hom/A008	61.0	54.2
Hom/A009	58.6	55.5
Hom/A010	54.1	48.5
Grand mean	54.5	
Cochran's test		
C	0.384	
Ccrit	0.602	
C < Ccrit?	NO OUTLIERS	
Target s = σ_P	13.6	
s_x	2.54	
s_w	2.47	
s_s	1.84	
Critical= 0.3 σ_P	4.09	
s_s < critical?	ACCEPTED	
s_w < 0.5 σ_P?	ACCEPTED	

s_x = Standard deviation of the sample averages.

s_w = Within-sample standard deviation.

s_s = Between-sample standard deviation.

Sample No.	ZEN in A (µg/kg)	
	Replicate 1	Replicate 2
Hom/A001	121	133
Hom/A002	113	122
Hom/A003	139	121
Hom/A004	126	134
Hom/A005	154	129
Hom/A006	127	123
Hom/A007	136	113
Hom/A008	127	122
Hom/A009	131	122
Hom/A010	139	120
Grand mean	128	
Cochran's test		
C	0.273	
Ccrit	0.602	
C < Ccrit?	NO OUTLIERS	
Target s = σ_P	31.9	
s_x	6.21	
s_w	10.7	
s_s	0.000	
Critical= 0.3 σ_P	9.58	
s_s < critical?	ACCEPTED	
s_w < 0.5 σ_P?	ACCEPTED	

s_x = Standard deviation of the sample averages.

s_w = Within-sample standard deviation.

s_s = Between-sample standard deviation.

Sample No.	3-Ac-DON in A (µg/kg)	
	Replicate 1	Replicate 2
Hom/A001	433	441
Hom/A002	425	412
Hom/A003	431	436
Hom/A004	446	436
Hom/A005	431	427
Hom/A006	455	443
Hom/A007	448	442
Hom/A008	434	443
Hom/A009	424	429
Hom/A010	427	420
Grand mean	434	
Cochran's test		
C	0.219	
Ccrit	0.602	
C < Ccrit?	NO OUTLIERS	
Target s = σ_P	109	
s_x	9.80	
s_w	6.04	
s_s	8.82	
Critical= 0.3 σ_P	32.6	
s_s < critical?	ACCEPTED	
s_w < 0.5 σ_P?	ACCEPTED	

s_x = Standard deviation of the sample averages.

s_w = Within-sample standard deviation.

s_s = Between-sample standard deviation.

Sample No.	15-Ac-DON in A (µg/kg)	
	Replicate 1	Replicate 2
Hom/A001	17.5	20.9
Hom/A002	15.3	15.0
Hom/A003	17.4	15.9
Hom/A004	18.0	15.8
Hom/A005	15.5	14.3
Hom/A006	17.3	19.3
Hom/A007	19.1	16.8
Hom/A008	17.3	16.4
Hom/A009	19.1	15.5
Hom/A010	20.0	15.4
Grand mean	17.1	
Cochran's test		
C	0.323	
Ccrit	0.602	
C < Ccrit?	NO OUTLIERS	
Target s = σ_P	4.27	
s_x	1.32	
s_w	1.79	
s_s	0.391	
Critical= 0.3 σ_P	1.28	
s_s < critical?	ACCEPTED	
s_w < 0.5 σ_P?	ACCEPTED	

s_x = Standard deviation of the sample averages.
s_w = Within-sample standard deviation.
s_s = Between-sample standard deviation.

Sample No.	DON-3-G in A (µg/kg)	
	Replicate 1	Replicate 2
Hom/A001	841	811
Hom/A002	821	750
Hom/A003	842	851
Hom/A004	798	893
Hom/A005	795	826
Hom/A006	872	838
Hom/A007	814	799
Hom/A008	816	810
Hom/A009	721	854
Hom/A010	817	788
Grand mean	818	
Cochran's test		
C	0.490	
Ccrit	0.602	
C < Ccrit?	NO OUTLIERS	
Target s = σ_P	204	
s_x	24.7	
s_w	42.5	
s_s	0.000	
Critical= 0.3 σ_P	61.3	
s_s < critical?	ACCEPTED	
s_w < 0.5 σ_P?	ACCEPTED	

s_x = Standard deviation of the sample averages.

s_w = Within-sample standard deviation.

s_s = Between-sample standard deviation.

Sample No.	NIV in A (µg/kg)	
	Replicate 1	Replicate 2
Hom/A001	49.2	56.0
Hom/A002	54.3	49.4
Hom/A003	54.9	52.9
Hom/A004	52.3	50.6
Hom/A005	49.5	47.0
Hom/A006	53.9	51.5
Hom/A007	56.4	53.0
Hom/A008	51.9	53.0
Hom/A009	46.9	50.5
Hom/A010	53.5	49.5
Grand mean	51.8	
Cochran's test		
C	0.357	
Ccrit	0.602	
C < Ccrit?	NO OUTLIERS	
Target s = σ_P	13.0	
s_x	2.02	
s_w	2.55	
s_s	0.914	
Critical= 0.3 σ_P	3.89	
s_s < critical?	ACCEPTED	
s_w < 0.5 σ_P?	ACCEPTED	

s_x = Standard deviation of the sample averages.

s_w = Within-sample standard deviation.

s_s = Between-sample standard deviation.

Sample No.	AOH in A (µg/kg)	
	Replicate 1	Replicate 2
Hom/A001	39.1	57.7
Hom/A002	55.9	56.5
Hom/A003	70.1	44.9
Hom/A004	44.1	39.2
Hom/A005	50.6	49.6
Hom/A006	45.3	38.0
Hom/A007	49.0	42.3
Hom/A008	42.7	56.3
Hom/A009	60.2	49.1
Hom/A010	49.7	40.5
Grand mean	49.0	
Cochran's test		
C	0.425	
Ccrit	0.602	
C < Ccrit?	NO OUTLIERS	
Target s = σ_P	12.3	
s_x	5.71	
s_w	8.65	
s_s	0.000	
Critical= 0.3 σ_P	3.68	
s_s < critical?	ACCEPTED	
s_w < 0.5 σ_P?	NOT ACCEPTED	

s_x = Standard deviation of the sample averages.

s_w = Within-sample standard deviation.

s_s = Between-sample standard deviation.

Sample No.	AME in A (µg/kg)	
	Replicate 1	Replicate 2
Hom/A001	19.4	19.4
Hom/A002	20.2	19.1
Hom/A003	19.6	16.7
Hom/A004	16.5	17.8
Hom/A005	18.5	17.4
Hom/A006	17.2	17.5
Hom/A007	18.9	18.2
Hom/A008		
Hom/A009	19.7	18.4
Hom/A010	18.3	18.4
Grand mean	18.4	
Cochran's test		
C	0.556	
Ccrit	0.638	
C < Ccrit?	NO OUTLIERS	
Target s = σ_P	4.60	
s_x	0.863	
s_w	0.905	
s_s	0.579	
Critical= 0.3 σ_P	1.38	
s_s < critical?	ACCEPTED	
s_w < 0.5 σ_P?	ACCEPTED	

s_x = Standard deviation of the sample averages.

s_w = Within-sample standard deviation.

s_s = Between-sample standard deviation.

Sample No.	Enn-A in A (µg/kg)	
	Replicate 1	Replicate 2
Hom/A001	2.48	2.34
Hom/A002	2.38	2.46
Hom/A003	2.59	2.41
Hom/A004	2.56	2.48
Hom/A005	2.42	2.31
Hom/A006	2.69	2.43
Hom/A007	2.53	2.53
Hom/A008	2.45	2.52
Hom/A009	2.53	2.64
Hom/A010	2.45	2.3
Grand mean	2.48	
Cochran's test		
C	0.383	
Ccrit	0.602	
C < Ccrit?	NO OUTLIERS	
Target s = σ_P	0.619	
s_x	0.078	
s_w	0.096	
s_s	0.037	
Critical= 0.3 σ_P	0.186	
s_s < critical?	ACCEPTED	
s_w < 0.5 σ_P?	ACCEPTED	

s_x = Standard deviation of the sample averages.

s_w = Within-sample standard deviation.

s_s = Between-sample standard deviation.

Sample No.	Enn-A1 in A (µg/kg)	
	Replicate 1	Replicate 2
Hom/A001	16.0	15.8
Hom/A002	14.8	15.9
Hom/A003	16.0	15.8
Hom/A004	15.5	16.5
Hom/A005	15.5	15.6
Hom/A006	16.9	15.9
Hom/A007	15.9	16.0
Hom/A008	16.0	17.0
Hom/A009	16.7	16.8
Hom/A010	15.9	15.2
Grand mean	16.0	
Cochran's test		
C	0.223	
Ccrit	0.602	
C < Ccrit?	NO OUTLIERS	
Target s = σ_P	4.00	
s_x	0.456	
s_w	0.486	
s_s	0.301	
Critical= 0.3 σ_P	1.20	
s_s < critical?	ACCEPTED	
s_w < 0.5 σ_P?	ACCEPTED	

s_x = Standard deviation of the sample averages.

s_w = Within-sample standard deviation.

s_s = Between-sample standard deviation.

Sample No.	Enn-B in A (µg/kg)	
	Replicate 1	Replicate 2
Hom/A001	92.9	93.2
Hom/A002	91.3	94.6
Hom/A003	96.0	94.5
Hom/A004	98.5	98.4
Hom/A005	90.0	89.9
Hom/A006		
Hom/A007	97.7	99.1
Hom/A008	99.4	98.9
Hom/A009	97.3	97.4
Hom/A010	93.1	90.7
Grand mean	95.2	
Cochran's test		
C	0.526	
Ccrit	0.638	
C < Ccrit?	NO OUTLIERS	
Target s = σ_P	23.8	
s_x	3.36	
s_w	1.10	
s_s	3.26	
Critical= 0.3 σ_P	7.14	
s_s < critical?	ACCEPTED	
s_w < 0.5 σ_P?	ACCEPTED	

s_x = Standard deviation of the sample averages.

s_w = Within-sample standard deviation.

s_s = Between-sample standard deviation.

Sample No.	Enn-B1 in A (µg/kg)	
	Replicate 1	Replicate 2
Hom/A001	51.0	51.8
Hom/A002	51.0	55.2
Hom/A003	52.8	53.2
Hom/A004	53.6	54.3
Hom/A005	48.3	50.9
Hom/A006	55.7	53.1
Hom/A007	52.9	54.0
Hom/A008	53.5	55.2
Hom/A009	55.2	56.8
Hom/A010	52.1	51.2
Grand mean	53.1	
Cochran's test		
C	0.444	
Ccrit	0.602	
C < Ccrit?	NO OUTLIERS	
Target s = σ_P	13.3	
s_x	1.81	
s_w	1.42	
s_s	1.51	
Critical= 0.3 σ_P	3.98	
s_s < critical?	ACCEPTED	
s_w < 0.5 σ_P?	ACCEPTED	

s_x = Standard deviation of the sample averages.

s_w = Within-sample standard deviation.

s_s = Between-sample standard deviation.

Sample No.	DON in B (µg/kg)	
	Replicate 1	Replicate 2
Hom/B001	668	667
Hom/B002	654	669
Hom/B003	657	636
Hom/B004	689	669
Hom/B005	639	665
Hom/B006	683	630
Hom/B007	650	693
Hom/B008	681	671
Hom/B009	662	629
Hom/B010	649	641
Grand mean	660	
Cochran's test		
C	0.361	
Ccrit	0.602	
C < Ccrit?	NO OUTLIERS	
Target s = σ_P	165	
s_x	13.0	
s_w	19.5	
s_s	0.000	
Critical= 0.3 σ_P	0.907	
s_s < critical?	ACCEPTED	
s_w < 0.5 σ_P?	ACCEPTED	

s_x = Standard deviation of the sample averages.

s_w = Within-sample standard deviation.

s_s = Between-sample standard deviation.

Sample No.	FB1 in B (µg/kg)	
	Replicate 1	Replicate 2
Hom/B001	3759	4128
Hom/B002	4070	3938
Hom/B003	3701	3995
Hom/B004	4055	4172
Hom/B005	3903	3925
Hom/B006	3767	3998
Hom/B007	3903	3926
Hom/B008	4005	3989
Hom/B009	3766	3808
Hom/B010	3820	3921
Grand mean	3928	
Cochran's test		
C	0.423	
Ccrit	0.602	
C < Ccrit?	NO OUTLIERS	
Target s = σ_P	982	
s_x	92.7	
s_w	127	
s_s	24.3	
Critical= 0.3 σ_P	295	
s_s < critical?	ACCEPTED	
s_w < 0.5 σ_P?	ACCEPTED	

s_x = Standard deviation of the sample averages.

s_w = Within-sample standard deviation.

s_s = Between-sample standard deviation.

Sample No.	FB2 in B (µg/kg)	
	Replicate 1	Replicate 2
Hom/B001	201	216
Hom/B002	216	206
Hom/B003	198	212
Hom/B004	214	229
Hom/B005	207	209
Hom/B006	200	209
Hom/B007	206	211
Hom/B008	210	212
Hom/B009	202	201
Hom/B010	204	210
Grand mean	209	
Cochran's test		
C	0.271	
Ccrit	0.602	
C < Ccrit?	NO OUTLIERS	
Target s = σ_P	52.2	
s_x	5.39	
s_w	6.73	
s_s	2.54	
Critical= 0.3 σ_P	15.7	
s_s < critical?	ACCEPTED	
s_w < 0.5 σ_P?	ACCEPTED	

s_x = Standard deviation of the sample averages.

s_w = Within-sample standard deviation.

s_s = Between-sample standard deviation.

Sample No.	T-2 in B (µg/kg)	
	Replicate 1	Replicate 2
Hom/B001	2.18	2.03
Hom/B002	2.03	2.40
Hom/B003	2.04	2.18
Hom/B004	2.43	2.17
Hom/B005	2.34	2.16
Hom/B006	2.35	2.12
Hom/B007	2.21	2.14
Hom/B008	2.37	2.41
Hom/B009	2.18	2.12
Hom/B010	2.45	2.1
Grand mean	2.22	
Cochran's test	0.308	
C	0.602	
Ccrit		
C < Ccrit?	NO OUTLIERS	
Target s = σ_P	0.555	
s_x	0.089	
s_w	0.151	
s_s	0.000	
Critical= 0.3 σ_P	0.166	
s_s < critical?	ACCEPTED	
s_w < 0.5 σ_P?	ACCEPTED	

s_x = Standard deviation of the sample averages.

s_w = Within-sample standard deviation.

s_s = Between-sample standard deviation.

Sample No.	HT-2 in B (µg/kg)	
	Replicate 1	Replicate 2
Hom/B001	144	139
Hom/B002	137	137
Hom/B003	140	130
Hom/B004	140	136
Hom/B005	132	139
Hom/B006	137	138
Hom/B007	143	147
Hom/B008	145	144
Hom/B009	137	137
Hom/B010	143	140
Grand mean	139	
Cochran's test		
C	0.502	
Ccrit	0.602	
C < Ccrit?	NO OUTLIERS	
Target s = σ_P	34.8	
s_x	3.69	
s_w	3.36	
s_s	2.82	
Critical= 0.3 σ_P	10.4	
s_s < critical?	ACCEPTED	
s_w < 0.5 σ_P?	ACCEPTED	

s_x = Standard deviation of the sample averages.

s_w = Within-sample standard deviation.

s_s = Between-sample standard deviation.

Sample No.	ZEN in B (µg/kg)	
	Replicate 1	Replicate 2
Hom/B001	66.9	67.3
Hom/B002	66.9	66.0
Hom/B003	64.3	64.8
Hom/B004	67.5	68.3
Hom/B005	64.5	65.2
Hom/B006	68.5	65.6
Hom/B007	66.2	68.2
Hom/B008	65.7	67.2
Hom/B009	64.4	61.4
Hom/B010	64.9	66.8
Grand mean	66.0	
Cochran's test		
C	0.304	
Ccrit	0.602	
C < Ccrit?	NO OUTLIERS	
Target s = σ_P	16.5	
s_x	1.51	
s_w	1.21	
s_s	1.25	
Critical= 0.3 σ_P	4.95	
s_s < critical?	ACCEPTED	
s_w < 0.5 σ_P?	ACCEPTED	

s_x = Standard deviation of the sample averages.

s_w = Within-sample standard deviation.

s_s = Between-sample standard deviation.

Sample No.	3-Ac-DON in B (µg/kg)	
	Replicate 1	Replicate 2
Hom/B001	18.4	16.3
Hom/B002	15.4	14.9
Hom/B003	14.7	13.7
Hom/B004	15.7	16.5
Hom/B005	13.2	16.3
Hom/B006	16.6	14.6
Hom/B007	15.4	15.2
Hom/B008	16.7	18.1
Hom/B009	15.0	15.7
Hom/B010	18.0	14.3
Grand mean	15.7	
Cochran's test		
C	0.386	
Ccrit	0.602	
C < Ccrit?	NO OUTLIERS	
Target s = σ_P	3.94	
s_x	1.04	
s_w	1.32	
s_s	0.451	
Critical= 0.3 σ_P	1.18	
s_s < critical?	ACCEPTED	
s_w < 0.5 σ_P?	ACCEPTED	

s_x = Standard deviation of the sample averages.

s_w = Within-sample standard deviation.

s_s = Between-sample standard deviation.

Sample No.	15-Ac-DON in B (µg/kg)	
	Replicate 1	Replicate 2
Hom/B001	83.3	76.6
Hom/B002	82.4	76.6
Hom/B003	84.6	71.9
Hom/B004	79.7	86.2
Hom/B005	80.8	77.7
Hom/B006	80.9	82.1
Hom/B007	86.5	79.9
Hom/B008	84.7	81.0
Hom/B009	77.6	84.7
Hom/B010	80.9	81.6
Grand mean	81.0	
Cochran's test		
C	0.402	
Ccrit	0.602	
C < Ccrit?	NO OUTLIERS	
Target s = σ_P	20.3	
s_x	1.72	
s_w	4.47	
s_s	0.000	
Critical= 0.3 σ_P	6.07	
s_s < critical?	ACCEPTED	
s_w < 0.5 σ_P?	ACCEPTED	

s_x = Standard deviation of the sample averages.

s_w = Within-sample standard deviation.

s_s = Between-sample standard deviation.

Sample No.	DON-3-G in B (µg/kg)	
	Replicate 1	Replicate 2
Hom/B001	41.2	24.7
Hom/B002	26.4	30.0
Hom/B003	44.4	26.9
Hom/B004	34.6	28.0
Hom/B005	26.7	28.5
Hom/B006	33.4	25.3
Hom/B007	26.3	27.6
Hom/B008		
Hom/B009	25.7	36.1
Hom/B010	37.5	23.6
Grand mean	30.4	
Cochran's test		
C	0.306	
Ccrit	0.638	
C < Ccrit?	NO OUTLIERS	
Target s = σ_P	7.60	
s_x	2.76	
s_w	7.49	
s_s	0.000	
Critical= 0.3 σ_P	2.28	
s_s < critical?	ACCEPTED	
s_w < 0.5 σ_P?	NOT ACCEPTED	

s_x = Standard deviation of the sample averages.

s_w = Within-sample standard deviation.

s_s = Between-sample standard deviation.

Sample No.	NIV in B (µg/kg)	
	Replicate 1	**Replicate 2**
Hom/B001	335	188
Hom/B002	177	249
Hom/B003	222	192
Hom/B004	260	185
Hom/B005	184	207
Hom/B006	257	173
Hom/B007	185	228
Hom/B008	208	324
Hom/B009	200	290
Hom/B010	308	179
Grand mean	228	
Cochran's test		
C	0.267	
Ccrit	0.602	
C < Ccrit?	NO OUTLIERS	
Target s = σ_P	56.9	
s_x	24.7	
s_w	63.6	
s_s	0.000	
Critical= 0.3 σ_P	17.1	
s_s < critical?	ACCEPTED	
s_w < 0.5 σ_P?	NOT ACCEPTED	

s_x = Standard deviation of the sample averages.

s_w = Within-sample standard deviation.

s_s = Between-sample standard deviation.

Sample No.	Enn-B in B (µg/kg)	
	Replicate 1	**Replicate 2**
Hom/B001	5.57	5.64
Hom/B002	6.11	5.42
Hom/B003	5.66	5.43
Hom/B004	5.51	5.52
Hom/B005	5.64	5.42
Hom/B006	5.97	5.34
Hom/B007	5.43	6.24
Hom/B008	5.52	5.85
Hom/B009	5.48	5.38
Hom/B010	6.20	5.55
Grand mean	5.64	
Cochran's test		
C	0.305	
Ccrit	0.602	
C < Ccrit?	NO OUTLIERS	
Target s = σ_P	1.41	
s_x	0.146	
s_w	0.330	
s_s	0.000	
Critical= 0.3 σ_P	0.423	
s_s < critical?	ACCEPTED	
s_w < 0.5 σ_P?	ACCEPTED	

s_x = Standard deviation of the sample averages.

s_w = Within-sample standard deviation.

s_s = Between-sample standard deviation.

Annex 4 Statistical evaluation of the stability data

Stability evaluation for ***DON in material A***

Storage temperature	<-70 °C	<-18 °C
Time (days)	0	78
Calculated amounts (µg/kg)	3445	3475
	3592	3546
	3436	3471
	3385	3449
	3464	3425
	3589	3500
Average amount (µg/kg)		
n	6	6
st. dev (µg/kg)	85.6	42.1
Difference		7.67
0.3*σ_P		261
Consequential difference? Diff < 0.3*σ_P		No

Stability evaluation for ***T-2 in material A***

Storage temperature	<-70 °C	<-18 °C
Time (days)	0	78
Calculated amounts (µg/kg)	19.3	19.3
	20.5	18.6
	19.4	19.9
	17.9	20.4
	18.9	19.6
	20.9	18.8
Average amount (µg/kg)	19.5	19.4
n	6	6
st. dev (µg/kg)	1.10	0.670
Difference		0.051
0.3*σ_P		1.46
Consequential difference? Diff < 0.3*σ_P		No

Stability evaluation for ***HT-2 in material A***

Storage temperature	<-70 °C	<-18 °C
Time (days)	0	78
Calculated amounts (µg/kg)	39.6	44.4
	46.6	42.1
	46.6	45.2
	42.0	41.6
	39.3	40.8
	44.7	45.7
Average amount (µg/kg)	43.1	43.3
n	6	6
st. dev (µg/kg)	3.32	2.05
Difference		-0.159
0.3*σ_P		3.24
Consequential difference? Diff < 0.3*σ_P		No

Stability evaluation for ***ZEN in material A***

Storage temperature	<-70 °C	<-18 °C
Time (days)	0	78
Calculated amounts (µg/kg)	280	278
	349	285
	337	287
	260	330
	148	262
	325	336
Average amount (µg/kg)	283	296
n	6	6
st. dev (µg/kg)	74.4	29.6
Difference		-13.1
0.3*σ_P		21.2
Consequential difference? Diff < 0.3*σ_P		No

Stability evaluation for ***3-Ac-DON in material A***

Storage temperature	<-70 °C	<-18 °C
Time (days)	0	78
Calculated amounts (µg/kg)	622	555
	530	544
	530	631
	508	602
	727	525
	599	575
Average amount (µg/kg)	586	572
n	6	6
st. dev (µg/kg)	82.0	39.5
Difference		14.4
$0.3*\sigma_P$		44.0
Consequential difference? Diff < $0.3*\sigma_P$		No

Stability evaluation for ***DON-3-G in material A***

Storage temperature	<-70 °C	<-18 °C
Time (days)	0	78
Calculated amounts (µg/kg)	1108	1003
	956	945
	1116	1058
	1101	1090
	1154	1015
	1093	1060
Average amount (µg/kg)	1088	1029
n	6	6
st. dev (µg/kg)	68.1	51.9
Difference		59.4
$0.3*\sigma_P$		81.6
Consequential difference? Diff < $0.3*\sigma_P$		No

Stability evaluation for ***NIV in material A***

Storage temperature	<-70 °C	<-18 °C
Time (days)	0	78
Calculated amounts (µg/kg)	29.6	27.5
	31.1	30.4
	28.9	29.3
	30.4	25.6
	30.4	28.0
	31.9	28.9
Average amount (µg/kg)	30.4	28.3
n	6	6
st. dev (µg/kg)	1.05	1.66
Difference		2.09
$0.3*\sigma_P$		2.28
Consequential difference? Diff < $0.3*\sigma_P$		No

Stability evaluation for ***AOH in material A***

Storage temperature	<-70 °C	<-18 °C
Time (days)	0	78
Calculated amounts (µg/kg)	81	74
	166	133
	178	177
	129	226
	154	103
	193	232
Average amount (µg/kg)	150	158
n	6	6
st. dev (µg/kg)	40.3	64.9
Difference		-7.51
$0.3*\sigma_P$		11.3
Consequential difference? Diff < $0.3*\sigma_P$		No

Stability evaluation for ***AME in material A***

Storage temperature	<-70 °C	<-18 °C
Time (days)	0	78
Calculated amounts (µg/kg)	39.1	36.5
	36.6	33.5
	36.6	45.0
	36.2	44.5
	34.9	36.6
	29.3	47.1
Average amount (µg/kg)	35.5	40.5
n	6	6
st. dev (µg/kg)	3.31	5.68
Difference		-5.05
$0.3*\sigma_P$		2.66
Consequential difference? Diff < $0.3*\sigma_P$		Yes

Stability evaluation for ***Enn-A in material A***

Storage temperature	<-70 °C	<-18 °C
Time (days)	0	78
Calculated amounts (µg/kg)	3.23	3.57
	4.22	3.47
	3.75	3.69
	3.08	3.48
	3.63	3.63
	3.52	3.70
Average amount (µg/kg)	3.57	3.59
n	6	6
st. dev (µg/kg)	0.403	0.099
Difference		-0.018
$0.3*\sigma_P$		0.268
Consequential difference? Diff < $0.3*\sigma_P$		No

Stability evaluation for ***Enn-A1 in material A***

Storage temperature	<-70 °C	<-18 °C
Time (days)	0	78
Calculated amounts (µg/kg)	16.2	16.0
	19.6	16.7
	18.0	18.5
	16.0	16.9
	18.7	19.5
	17.1	17.5
Average amount (µg/kg)	17.6	17.5
n	6	6
st. dev (µg/kg)	1.42	1.28
Difference		0.103
$0.3*\sigma_P$		1.32
Consequential difference? Diff < $0.3*\sigma_P$		No

Stability evaluation for ***Enn-B in material A***

Storage temperature	<-70 °C	<-18 °C
Time (days)	0	78
Calculated amounts (µg/kg)	50.1	48.3
	53.1	48.8
	52.4	50.3
	47.8	48.7
	52.8	51.9
	48.7	50.7
Average amount (µg/kg)	50.8	49.8
n	6	6
st. dev (µg/kg)	2.26	1.40
Difference		1.06
$0.3*\sigma_P$		3.81
Consequential difference? Diff < $0.3*\sigma_P$		No

*Stability evaluation for **Enn-B1 in material A***

Storage temperature	<-70 °C	<-18 °C
Time (days)	0	78
Calculated amounts (µg/kg)	31.0	30.1
	34.2	31.1
	32.7	32.3
	27.9	30.8
	34.5	33.1
	30.0	32.3
Average amount (µg/kg)	31.7	31.6
n	6	6
st. dev (µg/kg)	2.59	1.10
Difference		0.103
0.3*σ_P		2.38
Consequential difference? Diff < 0.3*σ_P		No

*Stability evaluation for **DON in material B***

Storage temperature	<-70 °C	<-18 °C
Time (days)	0	78
Calculated amounts (µg/kg)	622	555
	530	544
	530	631
	508	602
	727	525
	599	575
Average amount (µg/kg)	586	572
n	6	6
st. dev (µg/kg)	82.0	39.5
Difference		14.4
0.3*σ_P		44.0
Consequential difference? Diff < 0.3*σ_P		No

*Stability evaluation for **FB1 in material B***

Storage temperature	<-70 °C	<-18 °C
Time (days)	0	78
Calculated amounts (µg/kg)	4288	4154
	4002	4024
	3791	3996
	3989	3933
	4054	4158
	3845	3924
Average amount (µg/kg)	3995	4032
n	6	6
st. dev (µg/kg)	175	103
Difference		-37.0
0.3*σ_P		300
Consequential difference? Diff < 0.3*σ_P		No

*Stability evaluation for **FB2 in material B***

Storage temperature	<-70 °C	<-18 °C
Time (days)	0	78
Calculated amounts (µg/kg)	221	209
	217	204
	211	204
	206	219
	208	222
	206	228
Average amount (µg/kg)	211	215
n	6	6
st. dev (µg/kg)	5.98	10.1
Difference		-3.04
0.3*σ_P		15.9
Consequential difference? Diff < 0.3*σ_P		No

Stability evaluation for ***T-2 in material B***

Storage temperature	<-70 °C	<-18 °C
Time (days)	0	78
Calculated amounts (µg/kg)	1.59	1.72
	1.65	1.73
	1.50	1.58
	1.72	1.94
	1.75	1.77
	1.84	1.98
Average amount (µg/kg)	1.68	1.79
n	6	6
st. dev (µg/kg)	0.120	0.147
Difference		-0.109
0.3*σ_P		0.126
Consequential difference? Diff < 0.3*σ_P		No

Stability evaluation for ***HT-2 in material B***

Storage temperature	<-70 °C	<-18 °C
Time (days)	0	78
Calculated amounts (µg/kg)	133	136
	119	123
	125	125
	119	127
	128	126
	123	133
Average amount (µg/kg)	125	128
n	6	6
st. dev (µg/kg)	5.38	4.74
Difference		-3.79
0.3*σ_P		9.35
Consequential difference? Diff < 0.3*σ_P		No

Stability evaluation for ***ZEN in material B***

Storage temperature	<-70 °C	<-18 °C
Time (days)	0	78
Calculated amounts (µg/kg)	95.7	98.8
	101	98.0
	97.9	101
	119	101
	106	101
	102	107
Average amount (µg/kg)	104	101
n	6	6
st. dev (µg/kg)	8.14	2.96
Difference		2.64
0.3*σ_P		7.77
Consequential difference? Diff < 0.3*σ_P		No

Stability evaluation for ***3-Ac-DON in material B***

Storage temperature	<-70 °C	<-18 °C
Time (days)	0	78
Calculated amounts (µg/kg)	14.5	14.3
	14.0	13.9
	13.0	12.9
	20.8	15.7
	15.0	13.5
	14.4	16.9
Average amount (µg/kg)	15.3	14.5
n	6	6
st. dev (µg/kg)	2.77	1.47
Difference		0.753
0.3*σ_P		1.15
Consequential difference? Diff < 0.3*σ_P		No

Stability evaluation for ***15-Ac-****DON* ***in material B***

Storage temperature	<-70 °C	<-18 °C
Time (days)	0	78
Calculated amounts (µg/kg)	82.8	82.2
	77.1	81.7
	76.0	73.9
	78.8	79.4
	76.3	79.7
	78.6	85.2
Average amount (µg/kg)	78.3	80.3
n	6	6
st. dev (µg/kg)	2.51	3.78
Difference		-2.08
0.3*σ_P		5.87
Consequential difference? Diff < 0.3*σ_P		No

Stability evaluation for ***DON-3-G in material B***

Storage temperature	<-70 °C	<-18 °C
Time (days)	0	78
Calculated amounts (µg/kg)		
Average amount (µg/kg)		
n		
st. dev (µg/kg)		
Difference		
0.3*σ_P		
Consequential difference? Diff < 0.3*σ_P		No

Stability evaluation for ***NIV in material B***

Storage temperature	<-70 °C	<-18 °C
Time (days)	0	78
Calculated amounts (µg/kg)	52.1	50.7
	59.0	59.5
	58.9	50.1
	62.8	61.6
	65.4	55.5
	58.8	53.6
Average amount (µg/kg)	59.5	55.2
n	6	6
st. dev (µg/kg)	4.51	4.66
Difference		4.32
0.3*σ_P		4.46
Consequential difference? Diff < 0.3*σ_P		No

Stability evaluation for ***Enn-B in material B***

Storage temperature	<-70 °C	<-18 °C
Time (days)	0	78
Calculated amounts (µg/kg)	3.56	3.43
	3.40	2.87
	2.95	3.17
	3.00	3.32
	3.33	3.08
	3.09	4.39
Average amount (µg/kg)	3.22	3.38
n	6	6
st. dev (µg/kg)	0.247	0.534
Difference		-0.159
0.3*σ_P		0.242
Consequential difference? Diff < 0.3*σ_P		No

Annex 5 Invitation letter

WAGENINGEN
UNIVERSITY & RESEARCH

European
Union
Reference
Laboratory

P.O. Box 230 | 6700 AE WAGENINGEN | The Netherlands

NRLs mycotoxins & plant toxins

Dear colleague,

The EURL mycotoxins & plant toxins, at Wageningen Food Safety Research (WFSR), will organize a proficiency test (PT) regarding multiple mycotoxins in food and feed matrices (EURLPT-MP06). The target mycotoxins include regulated mycotoxins for which quantitative determination is mandatory, and not yet regulated mycotoxins for which quantitative analysis is voluntary (see Table 1 below). This proficiency test will be organised under accreditation according to ISO 17043 (General requirements for proficiency testing - R013).

Harmonised EU regulation for deoxynivalenol, zearalenone, fumonisin B1 and fumonisin B2 is in place for food matrices (Regulation (EC) No 1881/2006 amended). Indicative levels have been set for T-2 toxin and HT-2 toxin in food and feed (Recommandation 2013/165/EU, maximum levels are under discussion). Guidance values apply for deoxynivalenol, zearalenone, fumonisin B1 and fumonisin B2 in feed (Recommendation 2006/576/EC, maximum levels are under discussion).

The primary goal of this proficiency test is to give laboratories the opportunity to evaluate or demonstrate their performance regarding the analysis of multiple mycotoxins in food and feed matrices. For the mandatory mycotoxins, laboratories are requested to use the methods as used for their enforcement, monitoring or control tasks, which may either be several dedicated single mycotoxin methods or multi-mycotoxins method(s).

According to Regulation (EU) 2017/625 all EU National Reference Laboratories (NRLs) mycotoxins & plant toxins in food and/or feed are mandatory to participate. I would like to invite you to participate in this PT.

1. Test materials
   One test sample of oatmeal and one test sample of maize flour will be provided. The test materials oatmeal and maize flour are representatives for food and feed. The test amount sent will be approximately 50 g.

2. Shipment of test materials
   Test materials will be sent in May 2021. The distribution of the test materials will be announced by e-mail. The deadline for reporting is strict and will be in early September.
3. Scope of analysis

Wageningen Food Safety Research

DATE
May 31, 2021

SUBJECT
Invitation EURL mycotoxins & plant toxins proficiency test mycotoxins in food and feed matrices (EURLPT-MP06)

POSTAL ADDRESS
P.O. Box 230
6700 AE WAGENINGEN
The Netherlands

VISITORS' ADDRESS
Wageningen Campus
Building 123
Akkermaalsbos 2
6708 WB WAGENINGEN

INTERNET
www.wur.nl/wfsr

C₀C NUMBER
09098104

HANDLED BY
Diana Pereboom

TELEPHONE
+31 (0) 614323017

EMAIL
pt.wfsr@wur.nl

Both materials are to be analysed for the indicated regulated (maximum limits, indicative levels, or guidance values) mycotoxins (mandatory), and optionally for an additional set of mycotoxins.

Table 1. Mycotoxins to be analysed in EURL-PT MP06.

Mycotoxins (mandatory)
deoxynivalenol (DON)
fumonisin B1 (FB1)
fumonisin B2 (FB2)
T-2 toxins (T-2)
HT-2 toxin (HT-2)
zearalenone (ZEN)
Mycotoxins (voluntary)
alternariol (ALT)
alternariol-monomethyl ether (AME)
3-acetyl-deoxynivalenol (3-Ac-DON)
15-acetyl-deoxynivalenol (15-Ac-DON)
deoxynivalenol-3-glucoside (DON-3G)
enniatin A (ENN-A)
enniatin A1 (ENN-A1)
enniatin B (ENN-B)
enniatin B1 (ENN-B1)
nivalenol (NIV)

The voluntary mycotoxins will not be benchmarked but will be evaluated for information purposes when reported by more than 7 particpants.

4. Questionnaire
A questionnaire will be sent electronically. In this questionnaire the particants will be asked to provide information about the laboratory method(s) used. It is not obligatory to use one multi-method. This information is necessary to conduct a more in depth analysis of the results obtained in this proficiency test.

5. Report
   - A report of the proficiency test will be dispatched early in 2022.
   - Results of the proficiency test will be presented anonymously
   - The follow-up protocol on proficiency test from DG Santé will be applied (only for the mandatory mycotoxins)

6. Additional information
   - WFSR is allowed to use the anonymous results of the proficiency test in presentations, seminars and publications
   - WFSR will never inform third parties (e.g. accreditation bodies) on specific laboratory results without informing the laboratory first

7. Costs
   - Participation is free of charge for the NRLs.
   - Official laboratories (OLs) can participate as long as sufficient test material is available, at a first come first serve basis. The participation fee for OLs is €270,- (ex. VAT) as a compensation for the preparation and transportation of the samples.
   - If an extra batch of samples is needed after the first shipping, the courier costs will be charged.

If you would like to participate, please fill out the accompanying participation form (preferably digitally) and send it back before the 14th of June 2021 to: pt.wfsr@wur.nl.

Looking forward to welcome you for this test,

Diana Pereboom-de Fauw
Proficiency tests

EURL mycotoxins & plant toxins in food and feed
Wageningen Food Safety Research
Wageningen
the Netherlands

Annex 6 Instruction letter

WAGENINGEN UNIVERSITY & RESEARCH

European Union Reference Laboratory

P.O. Box 230 | 6700 AE WAGENINGEN | The Netherlands

Wageningen Food Safety Research

DATE
June 21, 2021

SUBJECT
Instructions proficiency test mycotoxins in food and feed matrices (EURLPT-MP06)

WFSR/EURLPT-MP06/2021

OUR REFERENCE
2121710/WFSR

POSTAL ADDRESS
P.O. Box 230
6700 AE WAGENINGEN
The Netherlands

VISITORS' ADDRESS
Wageningen Campus
Building 123
Akkermaalsbos 2
6708 WB WAGENINGEN

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COC NUMBER
09098104

HANDLED BY
Diana Pereboom

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+31 (0) 614323017

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pt.wfsr@wur.nl

Dear Madam/Sir,

Thank you very much for your participation in the proficiency test for the analysis of multiple mycotoxins in food and feed matrices (EURLPT-MP06).

The parcel shipped to you should contain:

- One feed material consisting of maize flour and one food material consisting of oat flour. Each test material unit contains approximately 50 grams of the homogenised test material.

Instructions:

- After arrival the samples should be stored in the freezer.
- Please fill in the accompanied 'acknowledgement of receipt form' and return it immediately upon receipt of the samples by e-mail to pt.wfsr@wur.nl.
- Before analysis, homogenise the samples according to your laboratory's procedure.
- Treat the test material as a sample for routine analysis. Report one result and not an average of multiple measurements.
- Please report all analytical results in **µg/kg relative to a feed with a moisture content of 12% (assuming 0% moisture in the sample)**.
- Report the results as follows:
  If an analyte is not included in the scope of the method, please report **'nt (not tested)'**.
  When the result for an analyte is below the LOQ of your method, please report the result as **'<LOQ-value'** with the LOQ value specified (e.g. <10 µg/kg). Do not report any of the results as 'not detected' or some other code.
- Please use the following web application for entering your results for the test samples (https://crlwebshop.wur.nl/apex/f?p=107:LOGIN). Instructions for use of this web application were sent to you earlier by e-mail. If you didn't receive these instructions or you have a question, please contact us.

DATE
June 21, 2021

OUR REFERENCE
2121710/WFSR

PAGE
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- Provide detailed information in the questionnaire on the analysis of the multiple mycotoxins and the analytical method used and send it back to us by e-mail (pt.wfsr@wur.nl).
- The deadline for submitting test-results for this test is **the 6th of September, 2021.** Please note that this will be a strict deadline; results reported after the deadline will not be considered. The EURL should be contacted at least 2 weeks in advance, if for exceptional reasons the deadline cannot be met.

- Your username is:
- Your password is:
- Your lab code to enter this proficiency test is:

Please contact me in case you have any questions or need any assistance.

With kind regards,

Diana Pereboom
Proficiency tests

EURL mycotoxins & plant toxins
Wageningen Food Safety Research (WFSR)
The Netherlands

Annex 7 Scope and LOQ

Lab code	Mandatory mycotoxins						Voluntary mycotoxins									
	DON	FB1	FB2	T-2 toxin	HT-2 toxin	ZEN	3-Ac-DON	15-Ac-DON	DON-3-G	NIV	AOH	AME	Enn-A	Enn-A1	Enn-B	Enn-B1
	LOQ (µg/kg)															
PT8774	10	10	10	2	10	2	10		10	10	0.1	0.1	0.1	0.1	0.1	0.1
PT8775	50	10	20	5	5	20				80			5	5	5	5
PT8776	40	40	40	6	6	4	40	40	40	50	4	2	4	4	4	4
PT8777	50	50	50	10	10	10										
PT8778	20	20	20	10	10	10	10	10		20	2	2	10	10	10	10
PT8779	51	8.8	30	3.9	13	8.5	91	58	75	31						
PT8780	50	20	20	4	6	10										
PT8781	120	45	15	1.6	2	20										
PT8782	50	100	100	5	5	5	100	100		100	3	3	2	2	2	2
PT8783 LC-MS/FLD	80	1000/100	-/100	8	60	4	80	80	200	80						
PT8784	50	25	25	5	5	10	20	20	20							
PT8785		25	25	10	10											
PT8786	40	100	100	15	15	10					4	4				
PT8787	80	200	200	80	80	80	80	40	40	80	8	8	0.8	0.8	0.8	0.8
PT8788	225	62.5	62.5	12.5	12.5	5										
PT8789	10	10	10	2	10	10	15	15	15	20	3	1	0.8	0.8	0.4	0.4
PT8790		125	125			50										
PT8791	200	100	100	10	50	20	50	50	200							
PT8792	450	1000	1000	25	25	50										
PT8793_food/feed	120/300	55/250	45/250	10/50	10/50	25/50										
PT8794																
PT8795	10	10	10	10	10	10	10	10	10	10						
PT8796	106	75				23,8										
PT8797 LC-MS/FLD	54	-/19	-/21	6	6	13										
PT8798	60	90	30	10	10	10	50	50								
PT8799	100	50	50	10	10	10										
PT8800	8.5	20	20	8.5	8.5	8.5										

	Mandatory mycotoxins						**Voluntary mycotoxins**									
	DON	**FB1**	**FB2**	**T-2 toxin**	**HT-2 toxin**	**ZEN**	**3-Ac-DON**	**15-Ac-DON**	**DON-3-G**	**NIV**	**AOH**	**AME**	**Enn-A**	**Enn-A1**	**Enn-B**	**Enn-B1**
Lab code	**LOQ (µg/kg)**															
PT8801	10	51	45	4	3	20				7						
PT8802	10	50	50	10	10	10					10	10	20	20	20	20
PT8803	40	25	25	10	10	1.75										
PT8804	100	100	100	10	10	40	100			100						
PT8805 LC-MS/FLD	20	30	30	2	2	-/1.4										
PT8806	50	50	50	5	5	10	10	50	25							
PT8807	66	0.88	0.88	0.88	0.88	8.8	8.8	8.8	8.8							
PT8809	25	32	10	27	26	10										
PT8810	49	5	5	6	6	5	8	8	8		5	5				
PT8811	58			83	125	46	83	153	27	44						
PT8812	10	10	10	10	15	5					10	10	8	8	8	8
PT8813	200	200	200	5	5	20	200	200	200							
PT8814	100	25	25	5	10	25	25	100	/				25	25	25	25
PT8815	60	30	30	30	50	25	150	150	15	60	30	-	3	3	3	3
PT8816	12	12	12	6	6	5										
PT8817 FLD		80	24													
PT8818	150	375	125	50	50	20	150	150	150							
PT8819 LC-MS /FLD/ GC	-/-/100	50	50	-/-/100	-/-/100	-/50	-/-/100	-/-/100	40	-/-/100						

Annex 8 Method details

Lab code	Column	Column length (mm)	Total run time (min)	DON	FB1	FB2	T-2 toxin	HT-2 toxin	ZEN	3-Ac-DON	15-Ac-DON	DON-3G	NIV	AOH	AME	ENN-A	ENN-A1	ENN-B	ENN-B1
				Retention time (min)															
PT8774	Waters Aqcuity HSS T3, 100 x 2.1 mm, 1.8 µm	100	22	2.3	3	5.3	8.9	7.1	9.5	4.1	-	2.1	2	6.8	9.4	12.8	12.5	11.8	12.2
PT8775	Acquity UPLC BEH C18, 2.1 × 100mm, 1.7 µm	100	11	2.17	4.58	5.14	4.94	4.62	5.18				1.7			6.46	6.37	6.17	6.28
PT8776	Xbridge BEH; 75 mm x 3 mm;	75	23	5.6	6.3	7.5	7	6.3	7.4	10.7	10.5	6.1	4.2	5.3	8	11.5	11.1	10.5	10.8
PT8777	Inertsil ODS-3V, 4.6 x 150 mm, 5 um	150	25		7.8	18.4													
PT8777	Acquity BEH, C18, 2.1 x 100 mm, 1.7 um	100	10	1.78			5.22	4.81	4.99										
PT8778	Water ACQUITY UPLC HSS T3, 2.1 x 100 mm, 1.8 µm	100	12	1.37	1.57	2.08	4.4	4.05	4.69	2.7	2.7		4.08	5.28	6.7	7.18	6.94	6.53	6.73
PT8778	Macherey-Nagel Nucleoshell RP 18 plus, 2.7 µm		8																
PT8778	Macherey-Nagel Nucelodur 100-3 C8	100	6																
PT8778	Macherey-Nagel Nucelodur 100-5 C18	100	10.5																
PT8779	Phenomenex Kinetex Biphenyl, 2.1 x 100 mm, 2.6 µm	100	12	4.26	7.16	7.84	7.9	7.28	8.5	5.99	5.98	4.27	3.32						
PT8780																			
PT8781	Cortecs C18, 1.6, 2.1 x 100 mm	100	9	3.681	4.82	5.096	5.11	4.917	5.277										
PT8782	Zorbax Eclipse Plus C18 RRHD, 2.1 x 50 mm, 1.8 µm	50	27	2.85	10.2	11.4	10.6	9.85	11.1	6.1	6.1		1.8	9.6	11.4	14.5	14.3	13.8	14.1
PT8783 LC-MS	Phenomenex, Gemini, C18, 150 x 4.6 mm, 5 µm	150	15	6.53	6.53	6.53	6.53	6.53	6.53	9.18	9.15	6.47	4.91						
PT8783 FLD	Zorbax Eclipse XDB-C18, 250 x 4.6 mm, 5 µm	250	24		7.87	15.67													
PT8784	Waters ACQUITY UPLC BEH C18 2.1 x 100 mm, 1.7µm	100	10	1.2	1.1	1.2	2.7	2.5	1	10.4	10.6	5.4							
PT8785	Supelco Ascentis Express C18 7.5 x 2.1, 2.7 µm	7.5	16	4.38	7.06	9.94	8.54	7.3											
PT8786	Alltima HP C18 150 mm x 3.0 mm, 5 µm	100	30	4.4			9.9	9.5	10.4										
PT8786	Prodigy ODS-2 150 x3,2 mm, 5 µm	150			10	16													
PT8786	Xselect HSS T3 100x2,1 mm, 2.5 µm	100												8	10.5				
PT8787	YMC-Triart C18, 100 x 2.1 mm	100	20	3.6	3.9	4.4	5.6	5.3	5.7	4.5	4.9	3	1.9	8.1	10.5	16.5	16.1	15.1	15.7
PT8788	Kinetex XB-C18, 50x 2.1 mm”	50	16	4.51	7.87	8.84	9.09	8.35	9.89										
PT8789	Phenomenex Gemini-NX, C18 150 x 2.00 mm, 5 µm	150	20	4.5	9.2	13.0	10.2	8.5	12.1										
PT8789	Phenomenex Gemini-NX, C18 150 x 2.00 mm, 5 µm,	150	25							19.1	18.6	6.3	2.5			8.7	8.5	8.1	8.3

Lab code	Column	Column length (mm)	Total run time (min)	DON	FB1	FB2	T-2 toxin	HT-2 toxin	ZEN	3-Ac-DON	15-Ac-DON	DON-3G	NIV	AOH	AME	ENN-A	ENN-A1	ENN-B	ENN-B1
				Retention time (min)															
PT8789	Phenomenex Gemini-NX, C18 150 x 2.00 mm, 5 µm,	150	13											6.5	8.1				
PT8790	Zorbax Eclipse Plus, 2.1 x 50 mm, 1.8 µm	50	17		6.2	7.6			7.7										
PT8791	Waters CORTECS C18, 2.1 x 100 mm, 2.7 µm	100	14	3.44	8.91	9.49	9.18	8.69	9.38	7.75	7.99	4.47							
PT8792	Phenomenex Kinetex EVO C18 2.1 x1 50 mm, 2.6 µ	150	18	3.2	8.9	9.7	9.3	8.7	9.8										
PT8793_food/feed	Agilent, Zorbax Eclipse Plus C18, 4.6 x 150 mm, 5 µm	150	30	8.9															
PT8793_food/feed	Agilent, Zorbax Eclipse Plus C18, 4.6 x 150 mm, 5 µm	150	9						5.2										
PT8793_food/feed	Agilent, Zorbax Eclipse Plus C18, 4.6 x 150 mm, 5 µm	150	12		4.19	9.18													
PT8793_food/feed	Agilent, Zorbax Eclipse Plus C18, 4.6 x 150 mm, 5 µm	150	30				7.9	19											
PT8794	C18 150 mm	150	20		6.6	14.3													
PT8794	C18 250 mm	250	20						12.5										
PT8794	C18 250 mm	250	15	7															
PT8795																			
PT8796	Thermoscientific ODS Hypersil 250 x 4.6 mm	250																	
PT8797 LC-MS	YMC Triart C18, 100 × 2.1 mm, 12 nm, 1.9 µm	100	8.75	2.475			5.506	5.263	5.9										
PT8797 FLD	LiChrospher 100 RP-18, LichroCART 250 - 4, 5 µm	250	25		13.886	21.517													
PT8798	Hypersil GOLD, 2.1 x 50 mm, 1.9 µm	50	15	3.65	9.55	9.97	9.6	8.21	9.63	6.36	6.35								
PT8799																			
PT8800																			
PT8801																			
PT8802																			
PT8803	Kinetex C18 100 x4.6 mm	100	15		elisa	elisa	5	12	elisa										
PT8804	Ascenis Expres C18, 150 x 4,6 mm, 2.7 um	150	23	6.9	9.5	10.2	10.6	10.1	11.1				8.6						
PT8805 LC-MS	Waters BEH RP18,2.1 x 100 mm, 1.7 um	100	8		3.01	3.38													
PT8805 LC-MS	Acqiuty UPLC HSS C18, 2.1 x 100 mm, 1.8 um	100	5	1.53			4.45	4.09											
PT8805 FLD			10						1.2										
PT8806	Phenomenex, Kinetex C18, 50 x 3.00 mm, 1.7 µm	50	14	2.1	6.5	6.9	6.8	6.6	7.1	14.4	14.2	9.8							
PT8807	BEH C18 100 mm	100	18	1.8	6.8	7.6	7.5	8.5	8.7	14.2	15	5.2							
PT8808																			
PT8809																			
PT8810	Waters, Xselect HSS T3 C18, 2.1 x 100 mm, 2.5 µm	100	14		8.63	9.76	9.06	8.33	9.61					8.5	10				
PT8810	Cortes UPLC C 18;100 x 3 mm; 1.6 µm	100	20	4.5						10.8	11.1	4.8							

Lab code	Column	Column length (mm)	Total run time (min)	DON	FB1	FB2	T-2 toxin	HT-2 toxin	ZEN	3-Ac-DON	15-Ac-DON	DON-3G	NIV	AOH	AME	ENN-A	ENN-A1	ENN-B	ENN-B1
				Retention time (min)															
PT8811	Kinetex®F5, 100 x 2.1 mm, 1.7 µm	100		3.9			9.3	8.5	10.05	6.5	6.4	4.1	2.6						
PT8812	Zorbax Eclipse Plus C18 2.1 x 100 mm, 1.8-µm	100		1.76	6.55	7.43	7.11	6.52	4.46					4.98	6.37	8.42	8.27	7.9	8.1
PT8813	Zorbax SB-C18 2,1 x1 50 mm, 3.5 µm	150	12	3.774						4.406	4.408	3.752							
PT8813			15		4.707	5.374	6.683	5.679	7.101										
PT8814	HSS T3, 2.1 x 150 mm,1.8 µm	150	10	1.82	4.29	5.37	5.33	4.83	5.67			/				7.58	7.48	7.24	7.36
PT8814	HSS T3, 2.1 x150 mm, 1.8 µm	150	6.5							2.94	2.75								
PT8815	Phenomenex Gemini C18, 100 x 3 mm, 3 µm	100		5.6	11.1	12.3	11.8	11.2	12.5	8.9	7.7	6	2.9	11.7	-	14.3	14.2	13.9	14
PT8816																			
PT8817 FLD	GL Sciences, Inertsil ODS,-4.6 x 150 mm, 2 5 µm	150	14		5	12													
PT8818	Accucore, 100 mm x 2.1 mm, 2.6 µm	100	18	3.55	10.85	11.65	10.05	8.54	11.2	7.05	7.04	4.55							
PT8819 LC-MS	Agilent, Zorbax Eclipse, 100 × 2.1 mm; 1,7 µm	100	18		11.1	13.6						3.3							
PT8819 FLD	Thermo scientific, Hypersil ODS, 250 x 4.6 mm, 5 µm,	250	8						4.66										
PT8819 GC-MS	HP-5 MS 30 m, 0.25 mm, 0.25 µm		35	12.7			17.27	17.54		13.61	13.77		14.01						

Lab code	Sample weight (g)	Extraction solvent	Extraction solvent volume (ml)	Extraction conditions	Extraction time (min)	Sample clean-up	SPE cartridge	Volume extract Loaded on SPE (ml)	Matrix equivalent final extract (g/ml)	Mobile phase	Detection technique
PT8774	5	ACN+H_2O+acetic acid	20	mechanical shaking	10	Quechers + defatting with hexane, filtration			1	Two runs (acidic and basic): Acid A: H_2O+ammonium acetate+acetic acid; Acid B: ACN+acetic acid; Basic A: water+ammonium formate; Basic B: MeOH	
PT8775	2	ACN + 0.1% HCOOH in H_2O (1:1)	20	mechanical shaking	20	dilution			0.1	A: H_2O + 0.1% HCOOH B: MeOH + 0.1% HCOOH + 1mM $HCOONH_4$	
PT8776	4	ACN, MeOH, H_2O	15; 5; 5	mechanical shaking	120	none				A: NH_4HCO_3 1mM in MeOH+H_2O (5+95) B: MeOH	
PT8777	25	ACN:MeOH:H_2O (25:25:50) (FUMs)	125	ultraturrax	2	SPE	Rbiopharm Rhone Fumoniprep	2/3	0.4	MeOH:0.1 Msodium phosphate (75:25)	
	5	ACN:H_2O (84:16) (T2-HT2-DON-ZEN)	20	ultraturrax	1	SPE	MycoSep AFLAZON	8	1	A: 5mM ammonium acetate in H_2O B: 5mM ammonium acetate in MeOH	
PT8778	10	ACN / H_2O	40	mechanical shaking	10	SPE	OASIS HLB	10 mL/ 4 mL	1	H_2O buffered/MeOH	
	5	ACN / H_2O	20	mechanical shaking	30	SPE	Macherey-Nagel Chromabond Florisil/ Alox	4mL/ 4mL	0.083	H_2O buffered/MeOH	
	2.5	ACN/ MeOH / H_2O	80	mechanical shaking	20	none			0.03125	acidified H_2O/ACN	
	5	ACN / H_2O	20	mechanical shaking	30	none			0.083	H_2O buffered/MeOH	
PT8779	5	ACN: H_2O:formic acid (79:20:1; v:v:v)	25	mechanical shaking	60	none			0.2	A: 5 mM ammonium formate/0,1% formic acid in H_2O B: 5 mM ammonium formate/0,1% formic acid in MeOH	
PT8780		H_2O +ACN+AA 50/50/2 v/v/v									
PT8781	5	H_2O, ACN, acetic acid,	20	shaking (hand/vortex)	30	other			0.1	A:formic acid (1.5ml/l) in H_2O +10mM ammonium formate; B: formic acid (0.5ml/l) in MeOH	
PT8782	20	ACN:H_2O:acetic acid (79:20:1)	80	mechanical shaking	30	filtration			0.25	A: 10 mM ammonia formate, pH 3,0; B: MeOH with 0,2% formic acid	

Lab code	Sample weight (g)	Extraction solvent	Extraction solvent volume (ml)	Extraction conditions	Extraction time (min)	Sample clean-up	SPE cartridge	Volume extract Loaded on SPE (ml)	Matrix equivalent final extract (g/ml)	Mobile phase	Detection technique
PT8783	25	79% ACN +20% H_2O +1% acetic acid (multitox)	100	other	120	dilution			0.025	A: MeOH:H_2O:acetic acid – (10:89:1) B: MeOH:H_2O:acetic acid (97:2:1) (both with 5mM ammonium acetate)	
	10	25% ACN + 25% MeOH + 50% H_2O (FUMs)	50	mechanical shaking	40	other	IAC clean-up	10	0.2	A: 50% ACN + 50% acetic acid(2%) B: 60% ACN + 40% acetic acid(2%)	
PT8784	1	ACN/ H_2O or MeOH/H2O	20	mechanical shaking	30	SPE	IAC R-Biopharm	1 to 10	0.05 to 0.5		
PT8785	5	H_2O:ACN:formic acid (20:79:1,v/v/v)	25	mechanical shaking	30	none					
PT8786	10	ACN:H_2O:acetic acid (80/20/1) (DON, T-2, HT-2, ZEN)	100	mechanical shaking	60	none			0.2	A: H_2O with 1 mM ammonium acetate at 0,1% and acetic acid; B: MeOH with 1 mM ammonium acetate at 0,1% and acetic acid	
PT8786	20	MeOH/solution tampon PBS(50:50) (FUMs)	250	mechanical shaking	120	other			0.5	A: H_2O with 0,5% formic acid; B: MeOH with 0,5% formic acid	
PT8786	2	MeOH:H_2O /acetic acid (85:14:1) (AOH, AME)	15	mechanical shaking	45	SPE	type styurène-divinyle benzène	15	1	A: aqueous ammonium acetate 5mM; B: MeOH	
PT8787	10	ACN, H_2O, acid	40	mechanical shaking	60	none			0.25		
PT8788	1	ACN: H_2O: formic acid, (79:20:1, v/v/v)	4	mechanical shaking	30	dilution			0.25	A: MeOH with 0.1% acetic acid in the 10 mM ammonium acetate (5:95, v/v); B: MeOH with 0.1% acetic acid in the ammonium acetate (95:5,v/v)	
PT8789	5	ACN:H_2O:HCOOH (79:20:1) (DON, FB1,FB2, ZEA, T2, HT2)	25	mechanical shaking	45	none				A: H_2O, 5mM ammonium acetate, 1% acetic acid, B: MeOH, 5mM ammonium acetate, 1% acetic acid	
	5	ACN:H_2O:HCOOH (79:20:1) (NIV, 15-Ac-DON, 3-Ac-DON, DON-3-G, ENNs)	25	mechanical shaking	45	none				A: H_2O, 5mM ammonium acetate, 1% acetic acid, B: MeOH, 5mM ammonium acetate, 1% acetic acid	
	2	MeOH:H_2O:acetic acid (85:14:1) (AOH, AME)	15	mechanical shaking	45	SPE	STRATA XL	7.5/7	0.14	A: H_2O 5mM ammonium acetate; B: MeOH	
PT8790	2	ACN 80%	2 x 10 mL	mechanical shaking	2x60	none				A: H_2O with 0.1% formic acid; B: ACN	

Lab code	Sample weight (g)	Extraction solvent	Extraction solvent volume (ml)	Extraction conditions	Extraction time (min)	Sample clean-up	SPE cartridge	Volume extract Loaded on SPE (ml)	Matrix equivalent final extract (g/ml)	Mobile phase	Detection technique
PT8791	2	ACN:H_2O	16	mechanical shaking	40	dilution			0.05	A: 2mM ammonium acetate with 0.1% formic acid in H_2O, B: 2mM ammonium acetate with 0.1% formic acid in MeOH	
PT8792	5	ACN:H_2O:FA (79:20:1)	2x25	mechanical shaking	2x30	none					
PT8793	12.5	H_2O (DON)	200	blender	3	Other	immunoaffinity column Rbiopharm	2	0.125 / 1.0	A: H_2O; B: ACN	
	10	ACN:H_2O (9:1) (ZEN)	50	mechanical shaking	30	Other	immunoaffinity column Rbiopharm	20	0.8	MeOH 75% /H_2O 25%	
	10	MeOH:ACN:H_2O (1:1:2) (FUMs)	50	mechanical shaking	40	Other	immunoaffinity column Rbiopharm	10	0.4	MeOH 77% / NaH_2PO_4 in water 0.1M 23%	
	10	MeOH:H_2O (9 : 1) (T-2, HT-2)	50	blender	3	other	immunoaffinity column Rbiopharm	25	1.0	A:H_2O; B: ACN	
PT8794	3	ACN:MeOH:H_2O (25:25:50, v/v/v) (FUMs)	15	mechanical shaking	30	SPE	immunoaffinity columns FUMONIPREP / R-Biopharm Rhone LTD)	5ml extract diluted to 25 ml PBS and 5ml loaded on SPE / 3 ml eluted	1	MeOH(77%):0.1 NaH_2PO_4(23%)	
	2.5	ACN:H_2O (60:40, v/v) (ZEN)	10	mechanical shaking	60	SPE	immunoaffinity columns ZearaStar / ROMER	2ml extract diluted to 10 ml PBS and all 10ml loaded on SPE / 1,5 ml eluted	2.5	H_2O (46%):MeOH(8%):ACN(46%)	
	3	H_2O (DON)	30	mechanical shaking	30	SPE	immunoaffinity columns DONStar / ROMER	2ml / 1,5 ml	3	H_2O (80%):MeOH(10%):ACN(10%)	
PT8795											

Lab code	Sample weight (g)	Extraction solvent	Extraction solvent volume (ml)	Extraction conditions	Extraction time (min)	Sample clean-up	SPE cartridge	Volume extract Loaded on SPE (ml)	Matrix equivalent final extract (g/ml)	Mobile phase	Detection technique
PT8796	25	5 g PEG + 200 ml H_2O (DON)		mechanical shaking	120		immun. column				
	5	ACN: H_2O /84:16 (ZEN)		mechanical shaking	30	add PBS puffer to 4ml filtrate.	Immun. column				
PT8797	5	ACN, MeOH and H_2O (FUMS)	25	mechanical shaking	120	SPE	Immunoaffinity columns FUMONIPREP from R-BIOPHARM Rhone	10/2.5	5.0	A: MeOH; B: 0,1 M NaH_2PO_4	
	2	Ethyl acetate (DON, T-3, HT-2, ZEN)	16	mechanical shaking	30	none				A: MeOH + 0.1% formic acid + 5 mmol ammonium formate; B: H_2O + 0.1% formic acid + 5 mmol ammonium formate	
PT8798	5	58% ACN and 2% acetic acid in H_2O	20	mechanical shaking	60	dilution			0.125	A: 5mM NH_4COOH + 0,1% HCOOH in H_2O; B: 5mM $HCOONH_4$ + 0,1% HCOOH in MeOH	
PT8799		ACN/ H_2O /formic acid 74:25:1.		filtration		dilution water 1:1					
PT8800											
PT8801		(DON, NIV)					IAC				
		(T-2, HT-2, ZEN, FUMs)					IAC				
PT8802											
PT8803	5.0	H_2O (DON)	100	shaking (hand/vortex)	5	other	IAC DONprep (R-Biopharm)	10	0.5	MeOH: H_2O = 15:85	
		MeOH: H_2O (70:30) (FUMs, ZEN)					ELISA				
PT8804	10	ACN/H_2O/acetic acid	40	mechanical shaking	90	none				For ESI-: A: 5 mM ammonium formate; B: 5 mM ammonium formate/MeOH; For ESI+: A: 5 mM ammonium formate + 1% acetic	

Lab code	Sample weight (g)	Extraction solvent	Extraction solvent volume (ml)	Extraction conditions	Extraction time (min)	Sample clean-up	SPE cartridge	Volume extract Loaded on SPE (ml)	Matrix equivalent final extract (g/ml)	Mobile phase	Detection technique
										acid:B: 5 mM ammonium formate/MeOH + 1% acetic acid	
PT8805	10	MeOH/ACN/ H_2O: 1/1/2 (FUMs)	100	mechanical shaking	120	other	R-Biopharm Rhone	12.5/2	1.25	A: 0,1% formic acid; B: ACN	
	5	90% ACN (ZEN)	100	mechanical shaking	60	other	R Biopharm Rhone	5/2	0.25	A: 0.1% formic acid; B: MeOH:ACN (1:1)	
	25	84% ACN (DON, T-2, HT-2)	1	mechanical shaking	60	other	MycoSep 227 Trich+	5	3.125	A: H_2O; B: MeOH	
PT8806	5	50% ACN in H_2O with 0,1% formic acid	20	mechanical shaking	20	QuEChERS			6.67	A:0,5mM ammonium acetate and 0,1% formic acid in H_2O; B:0,5mM ammonium acetate and 0,1% formic acid in MeOH	
PT8807	10	ACN:H_2O (50:50)	80	ultraturrax	3	dilution			0.03	A H_2O + 0,1% FA; B: ACN + 0.1% FA	
PT8808											
PT8809		ACN: H_2O (70:30)		mechanical shaking		none					
PT8810	2.5	ACN:0.5% acetic acid and water (FUMs, T-2, HT-2, ZEN, AOH, AME)	10	shaking (hand/vortex)	1	LLE, n-hexane			0.5	A:H_2O/5 mM ammonium formate and 0.3% FA; B: MeOH/5 mM ammonium formate and 0,3% FA	
	10	ACN: H_2O (84:16, v/v) (DONs)	50	mechanical shaking	60	SPE	OASIS HLB; Waters	1	2.5	A: H_2O +1%HOAc+385mg AC-NH4; B: MeOH	
PT8811	2.5	ACN/ H_2O + formic acid	20	mechanical shaking	60	liquid/liquid extraction				A: CH_3COONH_4 + CH_3COOH in H_2O; B: MeOH	
PT8812	2	ACN + 1% HCOOH	10	mechanical shaking	30	dilution			0.2	A- H_2O, 5mM ammonium formate, 0,2% HCOOH: B: MeOH, 0,2% HCOOH	
PT8813	2	H_2O (DON, 15-AcDON, 3-Ac-DON, DON-3-G)	20	mechanical shaking	60	SPE	IAC Romerlabs	2	0.4	A: 0,1% acetic acid in H_2O; B MeOH	
PT8813	2	70% ACN (ZEN)	20	mechanical shaking	60	SPE	IAC Romerlabs	2	0.4	A: 0.1% formic acid in H_2O: B:0.1% formic acid in ACN	
PT8813	2	MeOH:ACN: H_2O (1:1:2); (FUMs)	20	mechanical shaking	60	SPE	IAC Romerlabs	2	0.4	A: 0.1% formic acid in H_2O; B: 0.1% formic acid in ACN	

Lab code	Sample weight (g)	Extraction solvent	Extraction solvent volume (ml)	Extraction conditions	Extraction time (min)	Sample clean-up	SPE cartridge	Volume extract Loaded on SPE (ml)	Matrix equivalent final extract (g/ml)	Mobile phase	Detection technique
PT8813	2	70% MeOH (T-2, HT-2)	20	mechanical shaking	60	SPE	IAC Neogen,	2	0.4	A: 0.1% formic acid in H_2O; B 0.1% formic acid in ACN	
PT8814	5	ACN:H_2O:formic Acid (80:18:2) (DON, FUMs, T-2, HT-2, ZEN, ENNs)	15	mechanical shaking	60	LLE			0.33	A: H_2O + 10 mM Ammonium format; B: MeOH + 10 mM Ammonium Format	
	5	ACN:H_2O:formic Acid (80/18/2) (3 and 15-Ac-DON	15							A: H_2O + 0.1% formic acid; B: ACN + 0.1% Formic acid	
PT8815	10	ACN: H_2O:HFo (69.5:29.5:1 v;v;v)	30	mechanical shaking	60	dilution				A: H_2O + 1% HOAc + 5 mM ammonium acetate B: MeOH 97% + H2O 2% + 1% HOAc + 5 mM ammonium acetate	
PT8816	5	10ml H_2O +10 ml ACN, Acetic Acid	20								
PT8817	25	MeOH:ACN:H2O(25:25:50)	100	blender	2	other	IAC R-Biopharm type	2	0.625	MeOH:H_2O 0.1M NaH_2PO_4 (77:23)	
PT8818	5	ACN:H_2O:formic acid(79:20:1)	25	shaking (hand/vortex)	30	none				A(0,1%FA IN H_2O),B(MeOH), for all mycotoxins except 3-Ac-DON, 15-Ac-DON and DON-3G: A: H_2O, 5 mM ammonium acetate, 0,1% acetic acid; B: MeOH, 5mM ammonium acetate, 0,1% acetic acid	
PT8819	5	ACN:H_2O:acetic acid: (84:15,5:0,5, v/v/v)	50	mechanical shaking	60	none			0.1	A: H_2O /2,5 mM ammonium acetate/0,5% acetic acid; B: MeOH/2,5 mM ammonium acetate/0,5% acetic acid	
	25	ACN: H_2O (75:25)	125	mechanical shaking	30	SPE	R-Biopharm Rhone, RBRRP90	10	0.2	ACN: H_2O:acetic acid=500:500:12	
	10	ACN: H_2O (84:16)	100	mechanical shaking	60	none	Romer, Trich 227+	8	0.125	helium	

ACN = acetonitrile; MeOH = methanol; H2O = water; FA (HCOOH) = formic acid; HOAc (CH3COOH) = acetic acid; HCOONH4 = ammonium formate; CH3COONH4 = ammonium acetate; NH4HCO3 = ammonium hydrogencarbonate; NaH2PO4 = sodium phosphate.

Annex 9 False positive and false negative results

False negative results.

Lab code	Material	Compound missed
PT8798	A	3-Ac-DON
PT8802	A	Enn-B
PT8802	A	Enn-B1
PT8806	A	HT-2
PT8782	B	HT-2
PT8795	B	FB1
PT8795	B	FB2
PT8795	B	15-Ac-DON
PT8804	B	HT-2

False positive results.

Lab code	Material	Compound reported while not present in the material
PT8774	A	FB1
PT8779	A	FB1
PT8780	A	FB1
PT8780	A	FB2
PT8781	A	FB1
PT8786	A	FB1
PT8786	A	FB2
PT8795	A	FB1
PT8795	A	FB2
PT8797	A	FB1
PT8797	A	FB2
PT8801	A	FB1
PT8807	A	FB1
PT8807	A	FB2
PT8802	B	Enn-A1

Annex 10 Results: Material A (oats flour)

	Material A					
	DON A: 3694 μg/kg u: 129 μg/kg σ_p: 924 μg/kg (25%) robust σ: 667 μg/kg (18.1%)		FB1		FB2	
Lab code	Result (μg/kg)	z-score	Result (μg/kg)	z-score	Result μg/kg	z-score
PT8774	4250	0.60	417 **FP**	In sample no FB1 present (cut-off level of 5 μg/kg)	<10	In sample no FB2 present (cut-off level of 5 μg/kg)
PT8775	4448.7	0.82	<10		<20	
PT8776	3850	0.17	<40		<40	
PT8777	3247.7	-0.48	<50		<50	
PT8778	2640	-1.14	<20		<20	
PT8779	3600	-0.10	90 **FP**		<30	
PT8780	2780	-0.99	1430 **FP**		3620 **FP**	
PT8781	4600	0.98	35 **FP**		<15	
PT8782	3778	0.09	<100		<100	
PT8783	3710	0.02	<100		<100	
PT8784	3791.7	0.11	<25		<25	
PT8785	3732	0.04	<25		<25	
PT8786	4500	0.87	550 **FP**		530 **FP**	
PT8787	3927	0.25	<200		<200	
PT8788	5173	1.60	<62.5		<62.5	
PT8789	3652	-0.05	<10		<10	
PT8790			<125		<125	
PT8791	3778	0.09	<100		<100	
PT8792	4620	1.00	<1000		<1000	
PT8793	3714	0.02	<55		<45	
PT8795	710	**-3.23**	3250 **FP**		180 **FP**	
PT8796	3740	0.05				
PT8797	3401	-0.32	38 **FP**		335 **FP**	
PT8798	3200	-0.54	<90		<30	
PT8799	2210	-1.61	<50		<50	
PT8800	2126	-1.70	<20		<20	
PT8801	2503.4	-1.29	355 **FP**		>51	
PT8802	4208	0.56	<50		<50	
PT8803	4673.3	1.06	<25.0		<25.0	
PT8804	3980	0.31	<100		<100	
PT8805	3800	0.11	<30		<30	
PT8806	3663	-0.03	<50		<50	
PT8807	3838.24	0.16	25.28 **FP**		33.66 **FP**	
PT8809	3712	0.02	<31		<10	
PT8810	3718	0.03	<5		<5	
PT8811	4699.8	1.09				
PT8812	4110	0.45	<10		<10	
PT8813	2978.4	-0.78	<200		<200	
PT8814	2638	-1.14	<25		<25	
PT8815	3740	0.05	<30		<30	
PT8816	3278	-0.45	<12		<12	
PT8817			<80		<24	
PT8818	4072	0.41	<375		<125	
PT8819	2460	-1.34	<50		<50	

A = assigned value (robust mean).

u = uncertainty of consensus value.

σp = target standard deviation for proficiency.

robust σ = robust (relative) standard deviation based on participants’ results.

	Material A					
	T-2 A: 17.8 µg/kg u: 0.762 µg/kg $σ_p$: 4.46 µg/kg (25%) robust σ: 3.50 µg/kg (19.6%)		HT-2 A: 45.2 µg/kg u: 1.91 µg/kg $σ_p$: 11.3 µg/kg (25%) robust σ: 9.18 µg/kg (20.3%)		ZEN A: 240 µg/kg u: 10.4 µg/kg $σ_p$: 60.0 µg/kg (25%) robust σ: 54.4 µg/kg (22.6%)	
Lab code	Result (µg/kg)	z-score	Result (µg/kg)	z-score	Result (µg/kg)	z-score
PT8774	19.6	0.40	25.5	-1.74	219	-0.35
PT8775	17.22	-0.14	44.35	-0.07	268.64	0.47
PT8776	18.5	0.15	45.1	-0.01	184.2	-0.93
PT8777	16.5	-0.30	33	-1.08	220.5	-0.33
PT8778	14.6	-0.73	40	-0.46	234	-0.10
PT8779	17	-0.19	42	-0.28	270	0.50
PT8780	15	-0.64	33	-1.08	67	**-2.88**
PT8781	13	-1.08	300	**22.57**	272	0.53
PT8782	16.2	-0.37	39.5	-0.50	378	**2.30**
PT8783	16.2	-0.37	45.7	0.05	191	-0.82
PT8784	21.4	0.80	43.7	-0.13	294	0.90
PT8785	18.4	0.13	40.6	-0.40	199	-0.69
PT8786	<15	(-0.64)	43	-0.19	250	0.16
PT8787	23.2	1.20	45.4	0.02	266.3	0.44
PT8788	12.3	-1.24	49.8	0.41	325	1.41
PT8789	21.2	0.75	50.7	0.49	229	-0.19
PT8790					209.5	-0.51
PT8791	17	-0.19	<50	(0.43)	236	-0.07
PT8792	17.8	-0.01	47	0.16	283	0.71
PT8793	16.3	-0.34	53.8	0.76	322	1.36
PT8795	<10	(-1.76)	105	**5.30**	75	**-2.75**
PT8796					199	-0.69
PT8797	20	0.49	43	-0.19	197	-0.72
PT8798	22	0.93	54	0.78	230	-0.17
PT8799	<10	(-1.76)	54	0.78	160	-1.33
PT8800	26.5	1.94	82	**3.26**	188	-0.87
PT8801	169.4	**33.99**	895.8	**75.33**	416.2	**2.93**
PT8802	17.1	-0.16	33	-1.08	366.7	**2.11**
PT8803					280.9	0.68
PT8804	20.4	0.58	49.7	0.40	177	-1.05
PT8805	14	-0.86	41	-0.37	230	-0.17
PT8806	14.8	-0.68	<5	**(-3.56)FN**	279	0.65
PT8807	12.38	-1.22	17.71	**-2.43**	256.17	0.27
PT8809	<27	**(2.06)**	<26	(-1.70)	146	-1.57
PT8810	18.7	0.19	43	-0.19	218	-0.37
PT8811	<83	**(14.61)**	58.9	1.22	263.6	0.39
PT8812	16.6	-0.28	51.7	0.58	237	-0.05
PT8813	17.2	-0.14	41.2	-0.35	284.7	0.74
PT8814	21.9	0.91	43.4	-0.16	223	-0.29
PT8815	<30	**(2.73)**	<50	(0.43)	263	0.38
PT8816	14.8	-0.68	32.7	-1.10	197.5	-0.71
PT8817						
PT8818	31	**2.95**	58	1.14	291	0.85
PT8819	<100	**(18.43)**	40	-0.46	260	0.33

A = assigned value (robust mean).

u = uncertainty of consensus value.

σp = target standard deviation for proficiency.

robust σ = robust (relative) standard deviation based on participants’ results.

	Material A							
	3-Ac-DON **A: 484 µg/kg** **u: 26.6 µg/kg** **σ_p: 121 µg/kg (25%)** **robust σ: 95.1 µg/kg (19.6%)**		**15-Ac-DON** **A: 160 µg/kg** **u: 56.5 µg/kg** **σ_p: 40.0 µg/kg (25%)** **robust σ: 128 µg/kg (79.9%)**		**DON-3-G** **A: 853 µg/kg** **u: 78.0 µg/kg** **σ_p: 213 µg/kg (25%)** **robust σ: 257 µg/kg (30.2%)**		**NIV** **A: 60.5 µg/kg** **u: 9.43 µg/kg** **σ_p: 15.1 µg/kg (25%)** **robust σ: 23.9 µg/kg (39.5%)**	
Lab code	**Result (µg/kg)**	**z-score**	**Result (µg/kg)**	**z-score**	**Result (µg/kg)**	**z'-score**	**Result (µg/kg)**	**z'-score**
PT8774					2370	**6.68**	22.7	**-2.12**
PT8775				No statistical evaluation possible Uncertainty exceeds 0.7σ_p			<80	
PT8776	484	0.00	<40		975	0.53	<50	(-0.59)
PT8777								
PT8778	376	-0.90	103				59	-0.08
PT8779	420	-0.53	110		2200	**5.93**	95	1.94
PT8780								
PT8781								
PT8782	509	0.20	397				<100	
PT8783	535	0.42	<80		979	0.55	51.1	-0.53
PT8784	478.5	-0.05	<20		735.1	-0.52		
PT8785								
PT8786								
PT8787	558.5	0.61	<40		770	-0.37	57.1	-0.19
PT8788								
PT8789	609	1.03	<15		406	-1.97	48.3	-0.68
PT8790								
PT8791	569	0.70	<50		864	0.05		
PT8792								
PT8793								
PT8795	10	**-3.92**	55		25	**-3.65**	105	**2.50**
PT8796								
PT8797								
PT8798	<50	**(-3.59)FN**	<50					
PT8799								
PT8800								
PT8801							448	**21.74**
PT8802								
PT8803								
PT8804	330	-1.28					<100	
PT8805								
PT8806	404	-0.66	<50		178	**-2.97**		
PT8807	523.13	0.32	<8.8		2590.42	**7.65**		
PT8809								
PT8810	507	0.19	<8		868	0.06		
PT8811	572	0.72	<153		819.1	-0.15	58.9	-0.09
PT8812								
PT8813	416.2	-0.56	567.2		687.6	-0.73		
PT8814	500	0.13	<100					
PT8815	696	1.75	219		849	-0.02	<60	(-0.03)
PT8816								
PT8817								
PT8818	508	0.19	163		1216	1.60		
PT8819	380	-0.86	16		702	-0.67	40	-1.15

A = assigned value (robust mean).

u = uncertainty of consensus value.

σp = target standard deviation for proficiency.

robust σ = robust (relative) standard deviation based on participants' results.

Lab code	Material A AOH A: 90.4 µg/kg u: 18.4 µg/kg σ_p: 22.6 µg/kg (25%) robust σ: 48.9 µg/kg (54.1%) Results µg/kg	z-score	AME A: 23.8 µg/kg u: 5.29 µg/kg σ_p: 5.95 µg/kg (25%) robust σ: 13.4 µg/kg (56.2%) Results µg/kg	z-score
PT8774	19.7	No statistical evaluation Possible Uncertainty exceeds $0.7\sigma_p$	4.4	No statistical evaluation possible Uncertainty exceeds $0.7\sigma_p$
PT8775				
PT8776	129		29.7	
PT8777				
PT8778	76		28.5	
PT8779				
PT8780				
PT8781				
PT8782	50.3		15.4	
PT8783				
PT8784				
PT8785				
PT8786	88		28	
PT8787	9.7		4.2	
PT8788				
PT8789	126		38.4	
PT8790				
PT8791				
PT8792				
PT8793				
PT8795				
PT8796				
PT8797				
PT8798				
PT8799				
PT8800				
PT8801				
PT8802	99.8		29	
PT8803				
PT8804				
PT8805				
PT8806				
PT8807				
PT8809				
PT8810	114		38.2	
PT8811				
PT8812	94.6		16.9	
PT8813				
PT8814				
PT8815	193			
PT8816				
PT8817				
PT8818				
PT8819				

A = assigned value (robust mean).

u = uncertainty of consensus value.

σp = target standard deviation for proficiency.

robust σ = robust (relative) standard deviation based on participants' results.

	Material A							
	Enn-A		Enn-A1 A: 16.7 µg/kg u: 1.76 µg/kg σ_p: 4.18 µg/kg (25%) robust σ: 4.45 µg/kg (26.6%)		Enn-B A: 96.0 µg/kg u: 12.9 µg/kg σ_p: 24.0 µg/kg (25%) robust σ: 32.7 µg/kg (34.0%)		Enn-B1 A: 60.7 µg/kg u: 2.82 µg/kg σ_p: 15.2 µg/kg (25%) robust σ: 7.13 µg/kg (11.8%)	
Lab code	**Result (µg/kg)**	**z-score**	**Result (µg/kg)**	**z’-score**	**Result (µg/kg)**	**z’-score**	**Result (µg/kg)**	**z-score**
PT8774	0.53	No statistical Evaluation possible Too little results	0.81	**-3.51**	9.2	**-3.19**	3.24	**-3.79**
PT8775	< 5		15.67	-0.23	92.78	-0.12	65.06	0.29
PT8776	<4		17.9	0.26	101.3	0.19	62.1	0.09
PT8777								
PT8778	<10		21.5	1.05	110	0.51	63.4	0.18
PT8779								
PT8780								
PT8781								
PT8782	3.3		15.3	-0.32	78.2	-0.65	43.5	-1.13
PT8783								
PT8784								
PT8785								
PT8786								
PT8787	3.8		19	0.50	172	**2.79**	67.5	0.45
PT8788								
PT8789	4.02		18.9	0.48	111	0.55	61.8	0.07
PT8790								
PT8791								
PT8792								
PT8793								
PT8795								
PT8796								
PT8797								
PT8798								
PT8799								
PT8800								
PT8801								
PT8802	<20		<20		<20	**(-2.79)FN**	<20	**(-2.68)FN**
PT8803								
PT8804								
PT8805								
PT8806								
PT8807								
PT8809								
PT8810								
PT8811								
PT8812	8.14		13.7	-0.67	78.9	-0.63	51.4	-0.61
PT8813								
PT8814	<25		37.8	**4.64**	158	**2.27**	69.5	0.58
PT8815	<3		11.8	-1.09	55.9	-1.47	59.3	-0.09
PT8816								
PT8817								
PT8818								
PT8819								

A = assigned value (robust mean).
u = uncertainty of consensus value.
σp = target standard deviation for proficiency.
robust σ = robust (relative) standard deviation based on participants’ results.

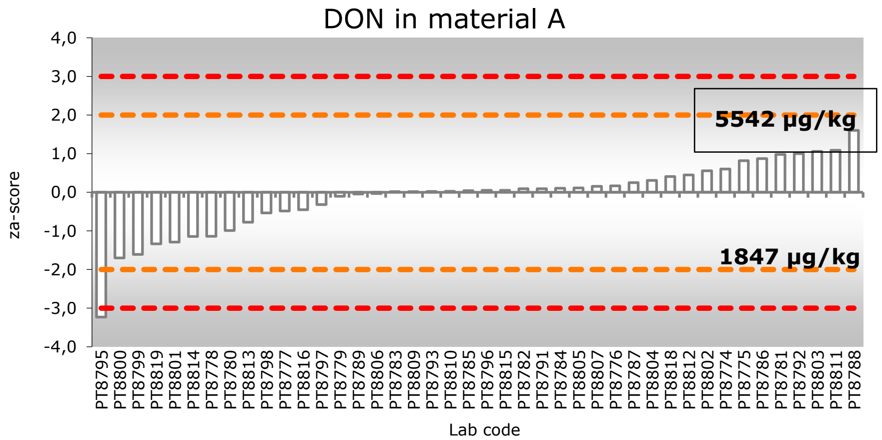

__Figure 6__ Graphical representation of the z-scores for DON in the material A. Dotted lines show PT performance boundaries ± 2 (also in µg/kg) and ± 3.

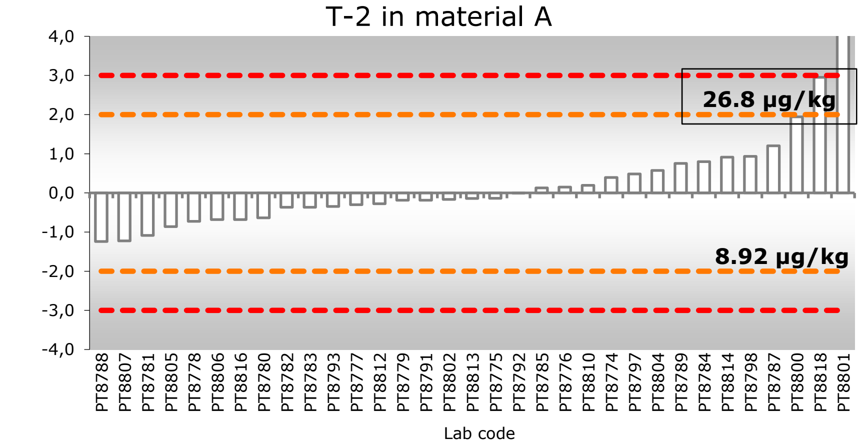

__Figure 7__ Graphical representation of the z-scores for T2 in the material A. Dotted lines show PT performance boundaries ± 2 (also in µg/kg) and ± 3.

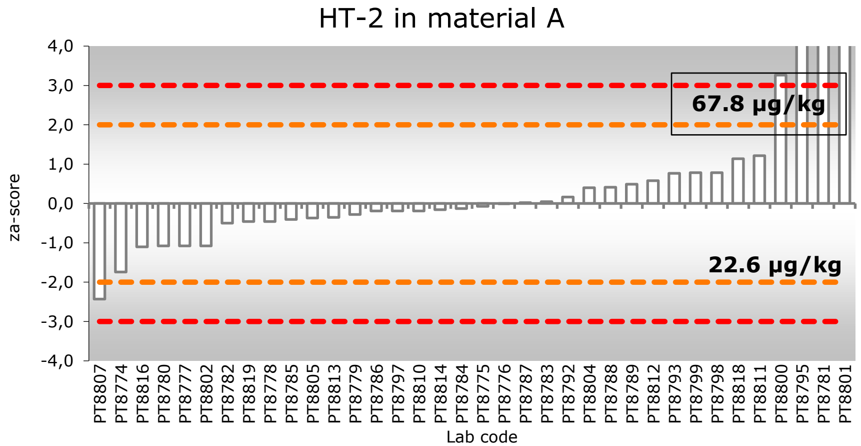

__Figure 8__ Graphical representation of the z-scores for HT2 in the material A. Dotted lines show PT performance boundaries ± 2 (also in µg/kg) and ± 3.

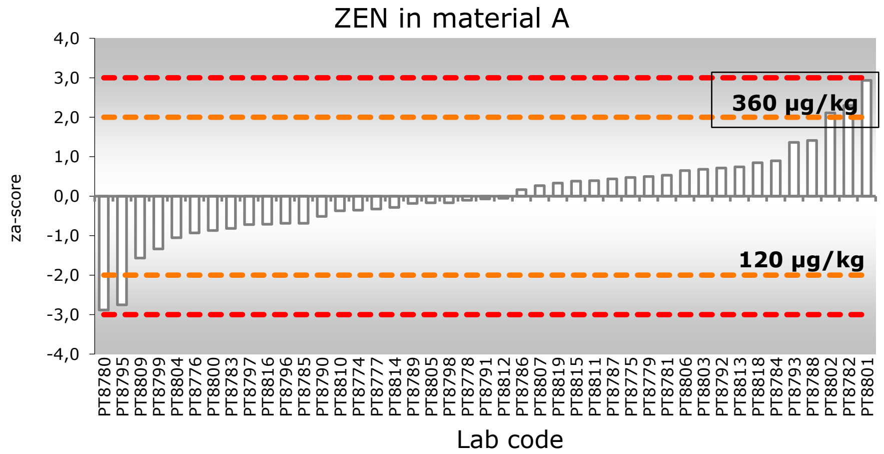

__Figure 9__ Graphical representation of the z-scores for ZEN in the material A. Dotted lines show PT performance boundaries ± 2 (also in µg/kg) and ± 3.

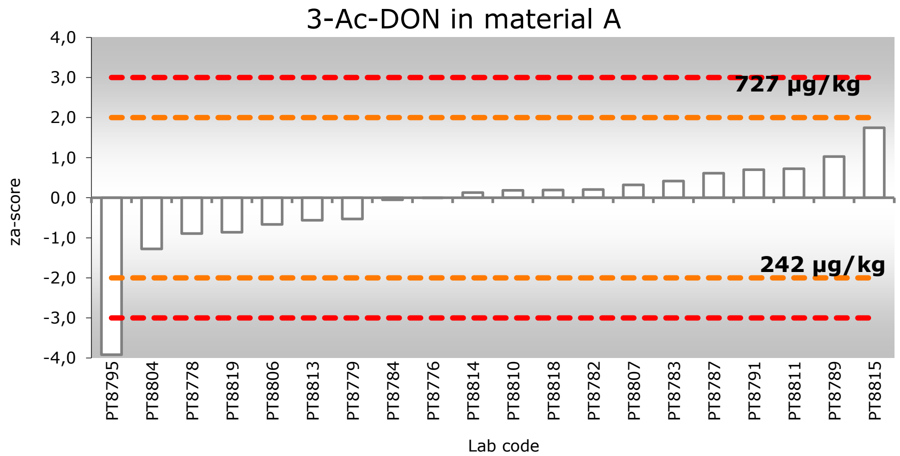

Figure 10 Graphical representation of the z-scores for 3-Ac_DON in the material A. Dotted lines show PT performance boundaries ± 2 (also in µg/kg) and ± 3

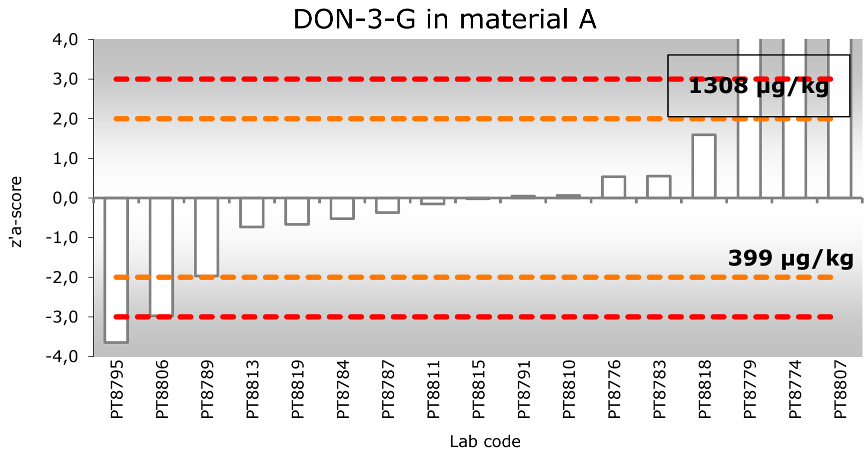

Figure 11 Graphical representation of the z'-scores for DON-3_G in the material A. Dotted lines show PT performance boundaries ± 2 (also in µg/kg) and ± 3

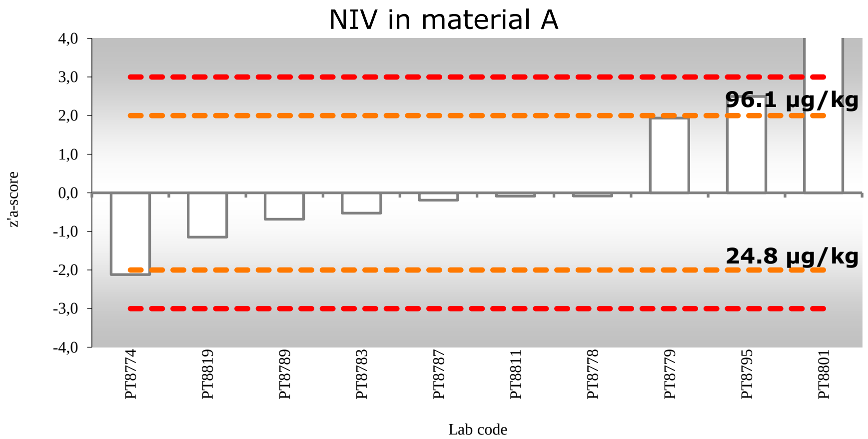

Figure 12 Graphical representation of the z'-scores for NIV in the material A. Dotted lines show PT performance boundaries ± 2 (also in µg/kg) and ± 3.

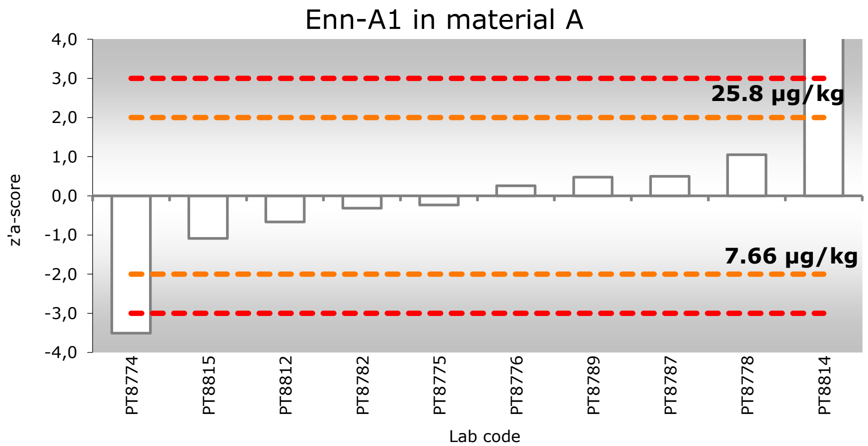

Figure 13 Graphical representation of the z'-scores for Enn-A1 in the material A. Dotted lines show PT performance boundaries ± 2 (also in µg/kg) and ± 3.

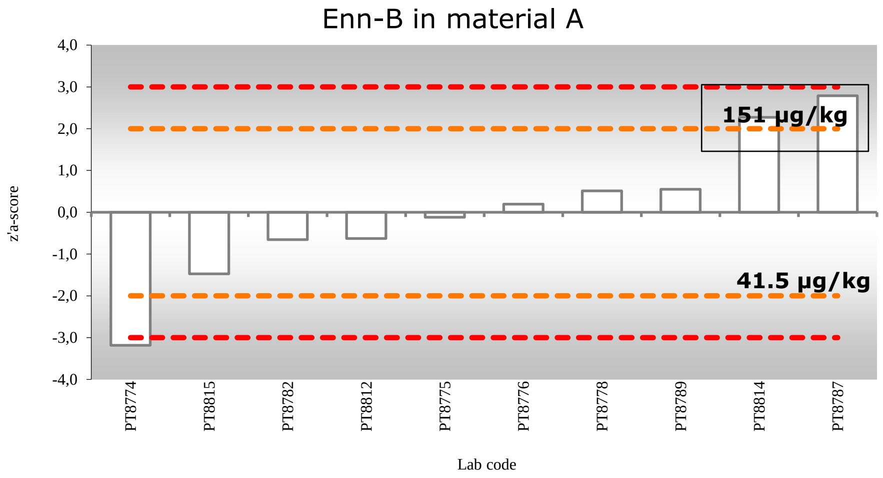

Figure 14 *Graphical representation of the z'-scores for Enn-B in the material A. Dotted lines show PT performance boundaries ± 2 (also in µg/kg) and ± 3*

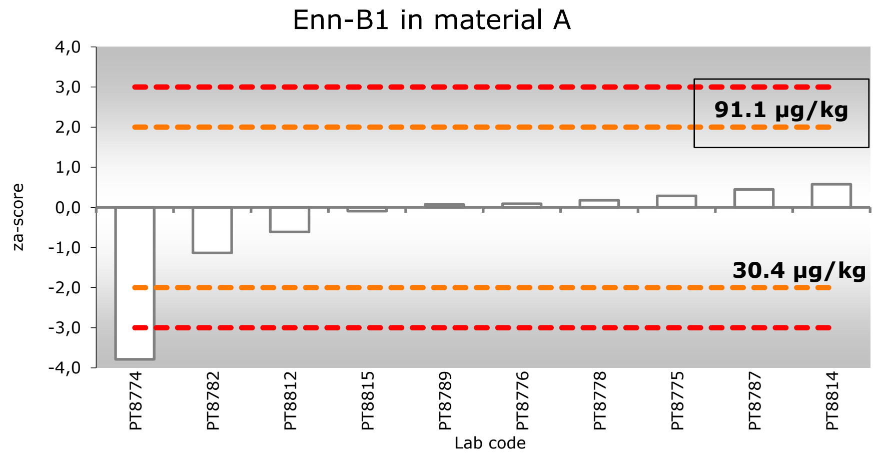

Figure 15 *Graphical representation of the z-scores for Enn-B1 in the material A. Dotted lines show PT performance boundaries ± 2 (also in µg/kg) and ± 3.*

Annex 11 Results: Material B (maize flour)

	Material B					
	DON A: 692 µg/kg u: 19.1 µg/kg σₚ: 173 µg/kg (25%) robust σ: 99.3 µg/kg (14.3%)		FB1 A: 3863 µg/kg u: 217 µg/kg σₚ: 966 µg/kg (25%) robust σ: 1114 µg/kg (28.8%)		FB2 A: 222 µg/kg u: 12.8 µg/kg σₚ: 55.6 µg/kg (25%) robust σ: 64.0 µg/kg (28.8%)	
Lab code	**Result (µg/kg)**	**z-score**	**Result (µg/kg)**	**z-score**	**Result (µg/kg)**	**z-score**
PT8774	1430	**4.27**	44560	**42.14**	348	**2.26**
PT8775	724.9	0.19	2617.6	-1.29	171.68	-0.91
PT8776	738.1	0.27	4370	0.52	215	-0.13
PT8777	645.8	-0.27	3530	-0.35	180.6	-0.75
PT8778	620	-0.42	7273	**3.53**	330	1.94
PT8779	590	-0.59	4800	0.97	180	-0.76
PT8780	666	-0.15	1074	**-2.89**	2599	**42.76**
PT8781	1060	**2.13**	5203	1.39	171.4	-0.92
PT8782	640	-0.30	2107	-1.82	159	-1.14
PT8783	641	-0.30	4100	0.24	187	-0.64
PT8784	743.5	0.30	5215.9	1.40	335.7	**2.04**
PT8785	825	0.77	2970	-0.92	315	1.67
PT8786	800	0.62	4000	0.14	240	0.32
PT8787	682	-0.06	4062	0.21	212.5	-0.18
PT8788	769	0.44	4930	1.10	273	0.91
PT8789	695	0.02	3649	-0.22	211	-0.20
PT8790			3776	-0.09	266.5	0.79
PT8791	683	-0.05	3365	-0.52	191	-0.56
PT8792	878	1.07	5260	1.45	221	-0.02
PT8793	599.5	-0.53	4058	0.20	<250	(-05)
PT8794	678	-0.08	3775	-0.09	163	-1.07
PT8795	3300	**15.07**	<10	**(-3.99)FN**	<10	**(-3.82)FN**
PT8796	665	-0.16				
PT8797	599	-0.54	3110	-0.78	199	-0.42
PT8798	695	0.02	3008	-0.89	120	-1.84
PT8799	300	**-2.27**	2270	-1.65	217	-0.10
PT8800	791	0.57	3140	-0.75	300	1.40
PT8801			3841	-0.02	>45	
PT8802	699.6	0.04	3326	-0.56	194	-0.51
PT8803	918.5	1.31				
PT8804	903	1.22	2567	-1.34	165	-1.03
PT8805	760	0.39	5030	1.21	260	0.68
PT8806	548	-0.83	5088	1.27	177	-0.82
PT8807	681.56	-0.06	4165.24	0.31	286.27	1.15
PT8809	631	-0.35	3853	-0.01	278	1.00
PT8810	709	0.10	4073	0.22	189	-0.60
PT8811	785.1	0.54				
PT8812	629	-0.36	2438	-1.48	151	-1.28
PT8813	649.4	-0.25	3604.3	-0.27	256.9	0.62
PT8814	461	-1.34	3240	-0.65	172	-0.91
PT8815	723	0.18	4650	0.81	280	1.04
PT8816	573	-0.69	4085	0.23	177	-0.82
PT8817			5371	1.56	245	0.41
PT8818	644	-0.28	4006	0.15	276	0.97
PT8819	530	-0.94	2440	-1.47	107	**-2.07**

A = assigned value (robust mean).

u = uncertainty of consensus value.

σp = target standard deviation for proficiency.

robust σ = robust (relative) standard deviation based on participants' results.

	Material B					
	T-2 A: 6.82 µg/kg u: 2.31 µg/kg $σ_p$: 1.70 µg/kg (25%) robust σ: 5.84 µg/kg (85.6%)		HT-2 A: 104 µg/kg u: 5.29 µg/kg $σ_p$: 26.0 µg/kg (25%) robust σ: 26.1 µg/kg (25.0%)		ZEN A: 88.6 µg/kg u: 4.65 µg/kg $σ_p$: 22.2 µg/kg (25%) robust σ: 24.7 µg/kg (27.8%)	
Lab code	**Result (µg/kg)**	**z-score**	**Result (µg/kg)**	**z-score**	**Result (µg/kg)**	**z-score**
PT8774	2.8	No statistical evaluation possible Uncertainty exceeds $0.7σ_p$	125	0.80	102	0.60
PT8775	<5		77.94	-1.01	76.02	-0.57
PT8776	<6		119	0.57	70	-0.84
PT8777	<10		102.4	-0.07	109.4	0.94
PT8778	<10		134	1.14	117	1.28
PT8779	<3.9		100	-0.16	85	-0.16
PT8780	<4		80	-0.93	49	-1.79
PT8781	6.4		190	**3.30**	126	1.69
PT8782	<5		<5	**(-3.81)FN**	69.2	-0.88
PT8783	<8		125	0.80	52.7	-1.62
PT8784	<5.0		114.3	0.39	97.4	0.40
PT8785	<10		137	1.26	68.2	-0.92
PT8786	<15		113	0.34	73	-0.71
PT8787	<80		113.1	0.34	91.5	0.13
PT8788	<12.5		114	0.38	90	0.06
PT8789	2.49		117	0.49	88.5	-0.01
PT8790					68.14	-0.93
PT8791	< 10		102	-0.08	70	-0.84
PT8792	1.59		130	0.99	109	0.92
PT8793	<50		86.8	-0.67	95.7	0.32
PT8794					29	**-2.69**
PT8795	25		30	**-2.85**	210	**5.48**
PT8796					89	0.02
PT8797	<6		112	0.30	67	-0.98
PT8798	16		92	-0.47	117	1.28
PT8799	<40		106	0.07	82	-0.30
PT8800	5.85		218	**4.37**	87.5	-0.05
PT8801	216.2		10.4	**-3.60**	155.3	**3.01**
PT8802	<10		75	-1.12	163.9	**3.40**
PT8803					213.4	**5.63**
PT8804	<10		<10	**(-3.62)FN**	72.7	-0.72
PT8805	2		121	0.65	116	1.23
PT8806	<5		97.4	-0.26	84	-0.21
PT8807	<0.88		58.56	-1.75	89.08	0.02
PT8809	<27		85	-0.74	28.2	**-2.73**
PT8810	<6		107	0.11	72.6	-0.72
PT8811	<83		118.4	0.55	84.7	-0.18
PT8812	<10		69.6	-1.33	94.2	0.25
PT8813	<5		111.8	0.29	82.8	-0.26
PT8814	<5		98.6	-0.21	68.7	-0.90
PT8815	<30		146	1.61	103	0.65
PT8816	6		83.3	-0.80	71.4	-0.78
PT8817						
PT8818	<50		81	-0.89	100	0.51
PT8819	<100		84	-0.77	105	0.74

A = assigned value (robust mean).

u = uncertainty of consensus value.

σp = target standard deviation for proficiency.

robust σ = robust (relative) standard deviation based on participants' results.

	Material B							
	3-Ac-DON		15-Ac-DON A: 66.0 µg/kg u: 4.15 µg/kg σ_p: 16.5 µg/kg (25%) robust σ: 12.0 µg/kg (18.1%)		DON-3-G A: 54.2 µg/kg u: 15.3 µg/kg σ_p: 13.6 µg/kg (25%) robust σ: 40.5 µg/kg (74.8%)		NIV A: 121 µg/kg u: 8.04 µg/kg σ_p: 30.1 µg/kg (25%) robust σ: 24.1 µg/kg (20.0%)	
Lab code	**Result (µg/kg)**	**z-score**	**Result (µg/kg)**	**z-score**	**Result (µg/kg)**	**z'-score**	**Result (µg/kg)**	**z'-score**
PT8774	<10	No statistical evaluation possible Too little results			93.1	No statistical Evaluation possible Uncertainty exceeds 0.7σ_p	46.9	**-2.44**
PT8775							128.7	0.27
PT8776	<40		72.7	0.41	<40		94.4	-0.87
PT8777								
PT8778	40		57.3	-0.53			128	0.25
PT8779	<91		<58	(-0.49)	350		130	0.31
PT8780								
PT8781								
PT8782	<100		<100				<100	(-0.68)
PT8783	<80		57.7	-0.50	<200		108	-0.42
PT8784	<20.0		120.2	**3.28**	<20			
PT8785								
PT8786								
PT8787	<80		59.5	-0.39	21.1		118.8	-0.06
PT8788								
PT8789	<15		162	**5.82**	<15		129	0.28
PT8790								
PT8791	<50		58	-0.49	<200			
PT8792								
PT8793								
PT8794								
PT8795	540		<10	**(-3.39)FN**	750		65	-1.84
PT8796								
PT8797								
PT8798	<50		66	0.00				
PT8799								
PT8800								
PT8801							220.2	**3.30**
PT8802								
PT8803								
PT8804	<100						192	**2.37**
PT8805								
PT8806	17.6		88.2	1.34	25.5			
PT8807	9.51		80.24	0.86	98.04			
PT8809								
PT8810	6.11		54.1	-0.72	21.2			
PT8811	<83		62.1	-0.24	13.3		121.4	0.03
PT8812								
PT8813	<200		<200		25.5			
PT8814	<25		<100					
PT8815	<150		<150		<15		136	0.51
PT8816								
PT8817								
PT8818	<150				276			
PT8819	<100		54	-0.73	38		91	-0.98

A = assigned value (robust mean).
u = uncertainty of consensus value.
σp = target standard deviation for proficiency.
robust σ = robust (relative) standard deviation based on participants' results.

Lab code	Material B			
	AOH		AME	
	Results µg/kg	z-score	Results µg/kg	z-score
PT8774	<0.1	In sample no AOH present (cut-off level of 5 µg/kg)	0.18	In sample no AME present (cut-off level of 5 µg/kg)
PT8775				
PT8776	<4		<2	
PT8777				
PT8778	<2		<2	
PT8779				
PT8780				
PT8781				
PT8782	<3		<3	
PT8783				
PT8784				
PT8785				
PT8786	<4		<4	
PT8787	<8		<8	
PT8788				
PT8789	<3		<1	
PT8790				
PT8791				
PT8792				
PT8793				
PT8794				
PT8795				
PT8796				
PT8797				
PT8798				
PT8799				
PT8800				
PT8801				
PT8802	<10		<10	
PT8803				
PT8804				
PT8805				
PT8806				
PT8807				
PT8809				
PT8810	<5		<5	
PT8811				
PT8812	<10		<10	
PT8813				
PT8814				
PT8815	<30			
PT8816				
PT8817				
PT8818				
PT8819				

A = assigned value (robust mean).

u = uncertainty of consensus value.

σp = target standard deviation for proficiency.

robust σ = robust (relative) standard deviation based on participants' results.

Lab code	Material B							
	Enn-A		Enn-A1		Enn-B		Enn-B1	
	Result (µg/kg)	z-score	Result (µg/kg)	z'-score	Result (µg/kg)	z'-score	Result (µg/kg)	z-score
PT8774	0.58	In sample no Enn-A present (cut-off level of 1 µg/kg)	0.33	In sample no Enn-A1 present (cut-off level of 1 µg/kg)	0.22	No statistical evaluation possible Too little results	0.29	No statistical evaluation possible Too little results
PT8775	<5		<5		<5		<5	
PT8776	<4		<4		<4		<4	
PT8777								
PT8778	<10		<10		<10		<10	
PT8779								
PT8780								
PT8781								
PT8782	<2		<2		4.3		<2	
PT8783								
PT8784								
PT8785								
PT8786								
PT8787	<0.8		<0.8		7.5		2	
PT8788								
PT8789	< 0.8		<0.8		4.05		1.55	
PT8790								
PT8791								
PT8792								
PT8793								
PT8794								
PT8795								
PT8796								
PT8797								
PT8798								
PT8799								
PT8800								
PT8801								
PT8802	<20		15.9 **FP**		81.4		45.5	
PT8803								
PT8804								
PT8805								
PT8806								
PT8807								
PT8809								
PT8810								
PT8811								
PT8812	<8		<8		<8		<8	
PT8813								
PT8814	<25		<25		<25		<25	
PT8815	<3		<3		2.95		<3	
PT8816								
PT8817								
PT8818								
PT8819								

A = assigned value (robust mean).

u = uncertainty of consensus value.

σp = target standard deviation for proficiency.

robust σ = robust (relative) standard deviation based on participants' results.

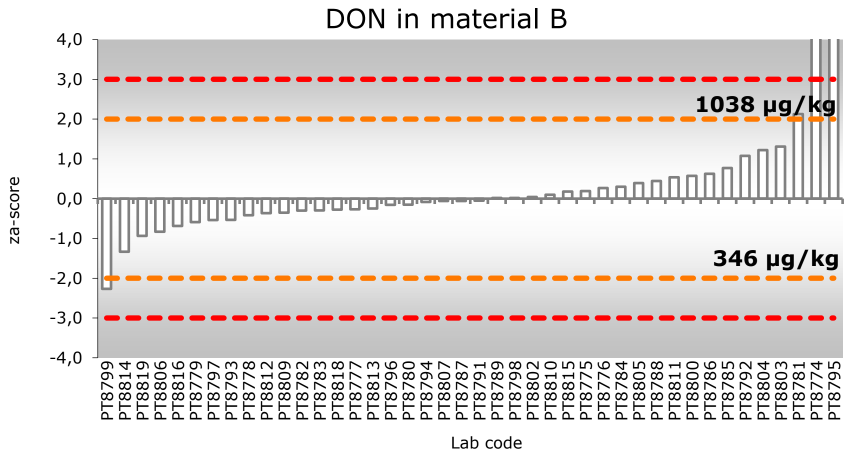

Figure 16 *Graphical representation of the z-scores for DON in the material B. Dotted lines show PT performance boundaries ± 2 (also in µg/kg) and ± 3.*

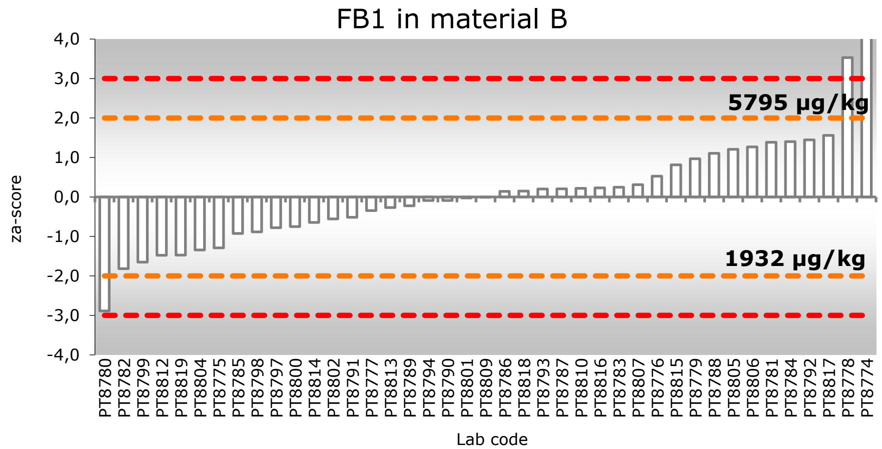

Figure 17 *Graphical representation of the z-scores for FB1 in the material B. Dotted lines show PT performance boundaries ± 2 (also in µg/kg) and ± 3.*

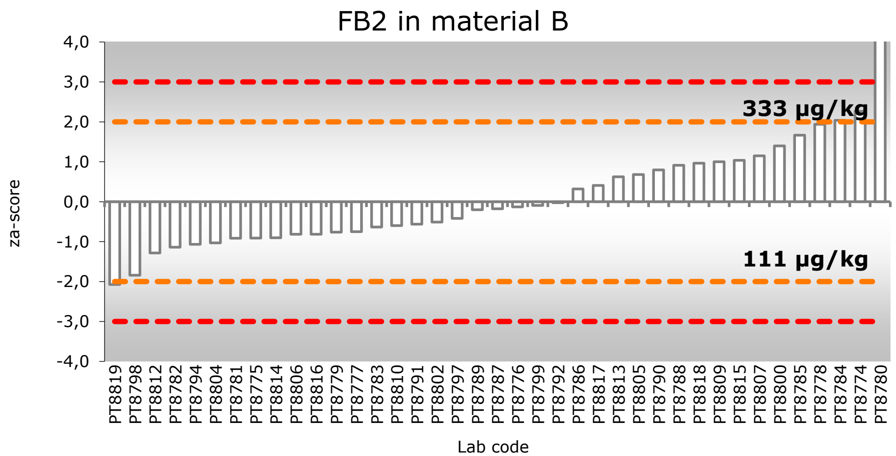

Figure 18 *Graphical representation of the z-scores for FB2 in the material B. Dotted lines show PT performance boundaries ± 2 (also in µg/kg) and ± 3.*

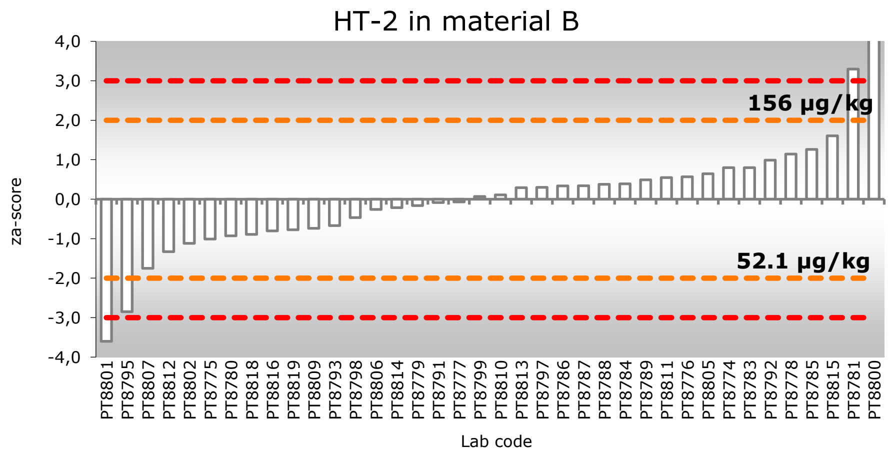

Figure 19 *Graphical representation of the z-scores for HT-2 in the material B. Dotted lines show PT performance boundaries ± 2 (also in µg/kg) and ± 3.*

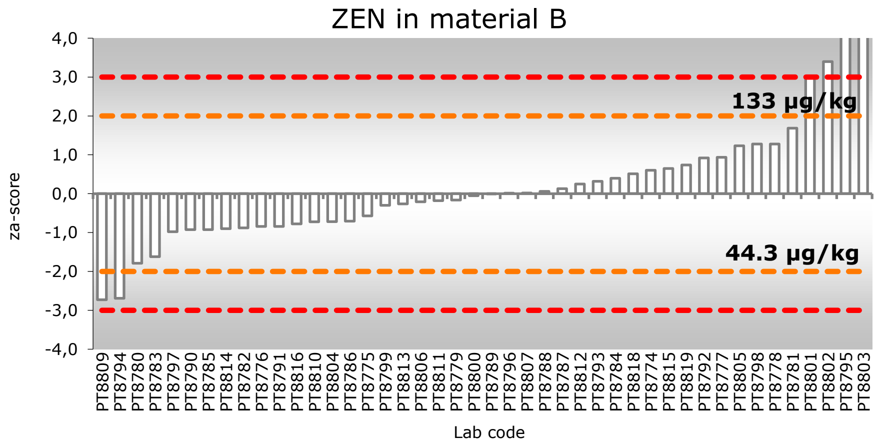

Figure 20 *Graphical representation of the z-scores for ZEN in the material B. Dotted lines show PT performance boundaries ± 2 (also in µg/kg) and ± 3.*

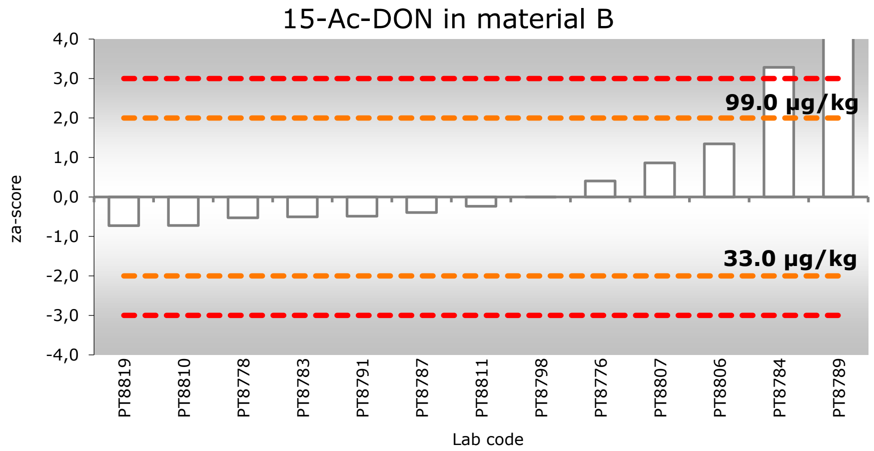

Figure 21 *Graphical representation of the z-scores for 15-Ac-DON in the material B. Dotted lines show PT performance boundaries ± 2 (also in µg/kg) and ± 3.*

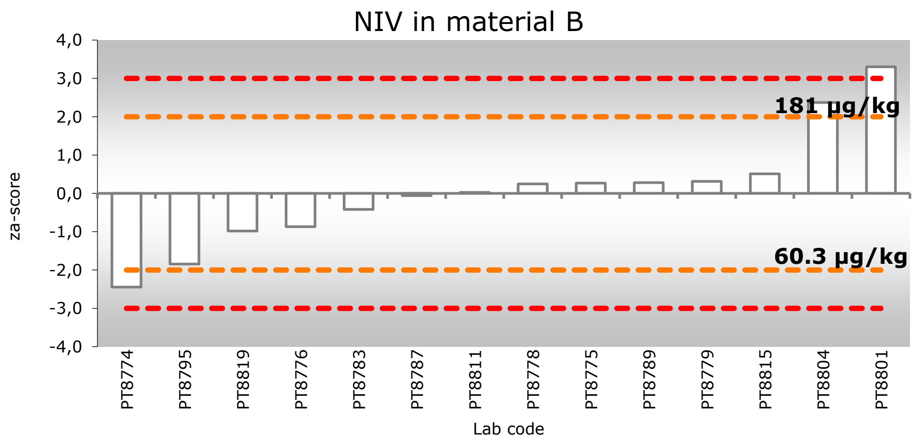

Figure 22 *Graphical representation of the z-scores for NIV in the material B. Dotted lines show PT performance boundaries ± 2 (also in µg/kg) and ± 3*

Annex 12 Overview performance per participant

Participant code	Satisfactory performance mandatory mycotoxins	FN	FP
PT8774	6 out of 9		1
PT8775	9 out of 9		
PT8776	9 out of 9		
PT8777	9 out of 9		
PT8778	8 out of 9		
PT8779	9 out of 9		1
PT8780	6 out of 9		2
PT8781	6 out of 9		1
PT8782	7 out of 9	1	
PT8783	9 out of 9		
PT8784	8 out of 9		
PT8785	9 out of 9		
PT8786	8 out of 9		2
PT8787	9 out of 9		
PT8788	9 out of 9		
PT8789	9 out of 9		
PT8790	4 out of 9		
PT8791	8 out of 9		
PT8792	9 out of 9		
PT8793	8 out of 9		
PT8794	2 out of 5		
PT8795	0 out of 9	2	2
PT8796	4 out of 9		
PT8797	9 out of 9		2
PT8798	9 out of 9		
PT8799	7 out of 9		
PT8800	7 out of 9		
PT8801	2 out of 9		1
PT8802	7 out of 9		1
PT8803	3 out of 9		
PT8804	8 out of 9	1	
PT8805	9 out of 9		
PT8806	8 out of 9	1	
PT8807	8 out of 9		2
PT8809	6 out of 9		
PT8810	9 out of 9		
PT8811	6 out of 9		
PT8812	9 out of 9		
PT8813	9 out of 9		
PT8814	9 out of 9		
PT8815	7 out of 9		
PT8816	9 out of 9		
PT8817	2 out of 9		
PT8818	8 out of 9		
PT8819	7 out of 9		

* Satisfactory performance means a satisfactory z-score was obtained for the mycotoxins present in material A and B.

** Participant PT9163 did not analyse material B.

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WFSR Report 2023.005

The mission of Wageningen University & Research is “To explore the potential of nature to improve the quality of life”. Under the banner Wageningen University & Research, Wageningen University and the specialised research institutes of the Wageningen Research Foundation have joined forces in contributing to finding solutions to important questions in the domain of healthy food and living environment. With its roughly 30 branches, 7,200 employees (6,400 fte) and 13,200 students and over 150,000 participants to WUR’s Life Long Learning, Wageningen University & Research is one of the leading organisations in its domain. The unique Wageningen approach lies in its integrated approach to issues and the collaboration between different disciplines.

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