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Proficiency test for deoxynivalenol (DON), acetyl-DONs and DON-3G in cereals

EURL-PT-MP01 (2018)


Wageningen, April 2019
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Summary

A proficiency test (PT) for quantitative deoxynivalenol (DON), 3-acetyl-DON (3-Ac-DON), 15-acetyl-deoxynivalenol (15-Ac-DON), and deoxynivalenol-3-glucoside (DON-3G) in wheat and maize was organised by the European Union Reference Laboratory for mycotoxins & plant toxins between March-June 2018. DON is a regulated mycotoxin in the EU. Acetyl-DONs and DON-3G were included in this PT because data collection and monitoring is recommended by EFSA, and insight in analytical performance is needed also for these substances. The primary goal was to assess the proficiency of National Reference Laboratories (NRLs).

In total 50 participants from 29 countries registered (Annex 1). This included NRLs from all EU member states, and a number of official laboratories.

Two food/feed materials, wheat (A) and maize (B), were prepared containing DON, 3-Ac-DON, 15-Ac-DON, and DON-3G. The starting materials were naturally contaminated with low levels of DON, and in case of maize also with 15-acetyl-DON and DON-3G. Levels were artificially increased by spiking with DON, 3-Ac-DON and 15-Ac-DON, and wheat also with DON-3G. Both materials were sufficiently homogeneous and stable during the course of the PT. Each participant received one test sample per material.

The assigned values were derived from the consensus of the results submitted by the participants and ranged from 35 to 750 µg/kg for the different mycotoxins. The proficiency of the participants was assessed through z-scores, calculated using the assigned value and a relative target standard deviation of 25%.

All participants submitted results for DON and satisfactory z-scores were obtained by all participants except 2. Acetyl-DONs and DON-3G were covered by less than half and less than one third of the laboratories, respectively. The laboratories that did have these mycotoxins in their scope had adequate performance in most cases (≥79%). In this PT, four false positives and two false negatives were reported, all related to 15-acetyl-DON. In some cases, the limits of quantification (LOQ) were high in relation to typical occurrence data.

Approximately two third of the laboratories used methods based on LC-MS/MS. The others mainly used methods based on LC-UV involving an IAC clean-up. The interlaboratory reproducibility (RSDr) ranged from 14% to 28% without clear dependency regarding the mycotoxin or concentration.

Characteristics of the PT materials and the outcome of this PT are summarised in Table 1.
Table 1  Summary of proficiency test parameters and participants’ performance.

<table>
<thead>
<tr>
<th>Mycotoxin</th>
<th>Matrix</th>
<th>Assigned value (µg/kg)</th>
<th>Uncert. (µg/kg)</th>
<th>Robust RSDs(^1)</th>
<th>Included in scope of labs</th>
<th>No of labs reporting:</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>DON</td>
<td>A</td>
<td>572</td>
<td>15.5</td>
<td>15%</td>
<td>50</td>
<td>100%</td>
</tr>
<tr>
<td></td>
<td>B</td>
<td>753</td>
<td>21.5</td>
<td>16%</td>
<td>50</td>
<td>0</td>
</tr>
<tr>
<td>3-Ac-DON</td>
<td>A</td>
<td>34.5</td>
<td>2.16</td>
<td>21%</td>
<td></td>
<td>19</td>
</tr>
<tr>
<td></td>
<td>B</td>
<td>93.4</td>
<td>4.53</td>
<td>18%</td>
<td></td>
<td>22</td>
</tr>
<tr>
<td>15-Ac-DON</td>
<td>A</td>
<td>&lt;20</td>
<td>-</td>
<td>-</td>
<td></td>
<td>9(^2)</td>
</tr>
<tr>
<td></td>
<td>B</td>
<td>154</td>
<td>11.6</td>
<td>26%</td>
<td></td>
<td>20</td>
</tr>
<tr>
<td>DON-3G</td>
<td>A</td>
<td>209</td>
<td>19.0</td>
<td>28%</td>
<td></td>
<td>16</td>
</tr>
<tr>
<td></td>
<td>B</td>
<td>35.1</td>
<td>1.91</td>
<td>14%</td>
<td></td>
<td>11</td>
</tr>
</tbody>
</table>

Matrix: A= Wheat, B= Maize

1) robust relative standard deviation (interlaboratory RSD based on participants' results)
2) of which four results were false positives
3) calculated using a fit-for-purpose target RSD for proficiency of 25%. False negatives were counted here as unsatisfactory z-score.
4) the number and percentage here means: mycotoxin determined, at sufficiently low LOQ to be quantified, and obtaining a satisfactory z-score.
1 Introduction

Deoxynivalenol (DON) is a secondary fungal metabolite produced by *Fusarium* species growing on the cereals in the field, especially at temperate climates. It is one of the most frequently occurring mycotoxins in food and feed. Mainly cereals and cereal-based products like pasta, bread and beer are affected. Chemically, DON is classified as type-B trichothecene. In addition to DON, the structurally related acetylated DON and modified forms of DON (e.g. plant-conjugates) have been found in the same type of matrices, of which 3-acetyl-DON (3-Ac-DON), 15-acetyl-DON (15-Ac-DON), and DON-3-glucoside (DON-3G) are the most relevant ones. In a scientific opinion by EFSA [1], the relative concentrations of 3-Ac-DON, 15-Ac-DON and DON-3G to DON were estimated as 10%, 15% and 20%, respectively. In the EFSA opinion, a group-TDI of 1 µg/kg bw per day for the sum of the four DON forms has been set, and a group-ARfD of 8 µg/kg bw per eating occasion. In current EU legislation maximum levels have been set for DON in food [2] ranging from 200 to 1750 µg/kg. In feed guidance values have been set at 0.9 to 12 mg/kg [3]. Although the acetyl-DONs and DON-3G are not yet included in legislation, their monitoring is recommended [1,4] and therefore the DON-derivatives were included in this proficiency test.

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 of the EU Additional Measures Directive 93/99/EEC [5] and is demanded by ISO/IEC 17025:2017 [6]. Organisation of proficiency tests (PT) is one of the tasks of 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 the mycotoxins DON, 3-Ac-DON, 15-Ac-DON and DON-3G in food and feed, using wheat and maize as representative matrices. The target concentrations aimed for (see Table 2) took regulatory limits and commonly found concentrations into account. Levels for the acetyl-DONs and DON-3G included enhanced levels because this was the first time these derivatives were included in an EURL-PT for mycotoxins. The proficiency test was carried out according to ISO/IEC 17043:2010 [8]. At the time of conduct not all of these analyte/matrix combinations were yet part of the accreditation scope, this was achieved in July 2018.

Table 2  Target concentrations µg/kg of mycotoxins in the PT materials.

<table>
<thead>
<tr>
<th>Material</th>
<th>DON</th>
<th>3-Ac-DON</th>
<th>15-Ac-DON</th>
<th>DON-3G</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>400</td>
<td>100</td>
<td>100</td>
<td>200</td>
</tr>
<tr>
<td>B</td>
<td>750</td>
<td>100</td>
<td>150</td>
<td></td>
</tr>
</tbody>
</table>

2.2 Material preparation

For preparation of the two PT materials A and B, wheat flour and maize flour were used. The starting materials were naturally contaminated with low levels of DON, and in case of maize also 15-acetyl-DON and DON-3G. Levels were artificially increased by spiking with DON, 3-Ac-DON and 15-Ac-DON, and wheat also with DON-3G. For each material, four kilograms were first fortified by adding a solution of a mycotoxin mix in acetonitrile, aiming at the levels as presented in Table 2. The materials were mixed with approximately six litres of water, homogenized using an industrial mixer according to an in-house standard operating procedure [9]. The fortified slurries were freeze-dried, homogenized in a Stephan cutter, and stored in the freezer until use.

2.3 Sample identification

After homogenization, materials A and B were divided into sub-portions of approximately 35 grams and stored in polypropylene, airtight closed containers of 125 ml. After preparation the containers were stored in the freezer until use.

The samples for the participants were randomly selected and coded using a web application designed for proficiency tests. The code used was EURPLT-MP 01/xxx, in which the three-digit number of the code was automatically generated by the web application. One sample set was prepared for each laboratory consisting of one randomly selected sample of each material A and B. The codes of the samples for each sample set are presented in Annex 2. For homogeneity and stability testing, randomly selected containers of materials A and B were used.

2.4 Homogeneity study

To verify the homogeneity of the PT materials, ten containers of materials A and B were analysed in duplicate for DON, 3-Ac-DON, 15-Ac-DON and DON-3G. The method of analysis is described in detail
In brief, DON and related mycotoxins were extracted from the homogenised sample material after addition of water, by shaking with acidified acetonitrile. After a salt-induced phase partitioning step and centrifugation, an aliquot of the acetonitrile phase was dried with magnesium sulfate. After addition of isotopically labelled internal standards for each of the four mycotoxins, an aliquot of extract was taken, evaporated to dryness, and reconstituted in methanol/water. Analysis was then done by high performance liquid chromatography coupled with tandem mass spectrometry (LC-MS/MS). The homogeneity of the materials was assessed according to the International Harmonized Protocol for Proficiency Testing of Analytical Laboratories [11] and ISO 13528:2015 [12]. The results of the homogeneity study, grand mean with the corresponding RSDr, are presented in Table 3, and the statistical evaluation of materials A and B is presented in Annex 3. Both materials proved to be sufficiently homogeneous for this PT.

Table 3  Concentrations of mycotoxins in material A and B obtained during homogeneity testing.

<table>
<thead>
<tr>
<th>Material code</th>
<th>DON</th>
<th>3-Ac-DON</th>
<th>15-Ac-DON</th>
<th>DON-3G</th>
</tr>
</thead>
<tbody>
<tr>
<td>Concentration (µg/kg)</td>
<td>RSDr (%)</td>
<td>Concentration (µg/kg)</td>
<td>RSDr (%)</td>
<td>Concentration (µg/kg)</td>
</tr>
<tr>
<td>A</td>
<td>536</td>
<td>5.2</td>
<td>31.8</td>
<td>2.9</td>
</tr>
<tr>
<td>B</td>
<td>730</td>
<td>5.7</td>
<td>98.8</td>
<td>4.0</td>
</tr>
</tbody>
</table>

* below lowest validated level, indicative concentration 6 µg/kg with RSDr of 27%.

In material A (wheat), the concentrations of the acetyl-DONs were much lower than the anticipated target concentrations. During preparation of this material, the slurry mixing with water at ambient temperature took relatively long and it was hypothesized that acetyl-DON might be (enzymatically) de-acetylated. A follow up experiment in which the wheat and maize starting materials were spiked individually with the acetylated DONs, slurried with water, and left for 4 and 24 hours confirmed conversion of the acetylated DONs into DON in the wheat flour. In maize flour, no 15-Ac-DON and only very minor 3-Ac-DON conversion occurred.

2.5 Stability of the materials

The stability of the mycotoxins in the PT materials was assessed according to [11,12]. At the day of distribution of the PT samples, six randomly selected containers of each material A and B were stored at <-70°C. Under these conditions it is assumed that the mycotoxins are stable in the materials. Another twelve containers remained stored in the freezer. In addition, to mimic a possible thaw situation during transport, six containers were stored at room temperature for one day and then stored again in the freezer.

On June 5th, 2017, 43 days after distribution of the samples, for each of the storage conditions (<-70°C, freezer, one-day room temperature) six samples of materials A and B 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 occurred [11,12] in the materials stored in the freezer or stored at room temperature for one day. A consequential instability is observed when the average value of an analyte in the samples stored in the freezer or stored at room temperature for one day is more than 0.3σ 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. In none of the mycotoxin/storage condition combinations, a consequential difference was observed. The mycotoxins in the materials were therefore considered stable for the duration of the PT.
3 Organisational details

3.1 Participants

This proficiency test focused on the mycotoxins DON, 3-Ac-DON, 15-Ac-DON and DON-3G in food and feed, using wheat. Invitations to the NRL network were sent out on 7th of March 2018 (Annex 5). Fifty laboratories registered for the PT (Annex 1). This included 39 NRLs (38 from EU countries and one from Serbia), ten OLs, and one external laboratory. Each participant was asked a priori to indicate which compounds were included in the scope of their method. The participants were asked to report the results through an existing web application designed for proficiency tests organised by RIKILT.

3.2 Material distribution and instructions

Each of the participants received a randomly assigned laboratory code, generated by the web application. The sample sets with the corresponding number, consisting of two coded samples (Annex 2) were sent to the participants on April 23rd 2018. The sample sets were packed in an insulation box containing dry ice and were dispatched to the participants immediately by courier. The samples were accompanied by a letter describing the requested analysis (Annex 6) and an acknowledgement of receipt form. By e-mail the participants received instructions on how to use the web application to report the results.

The participants were asked to store the samples in the freezer and to analyse the samples according to their routine method. A single analysis result for the mycotoxins in each sample was requested. The deadline for submitting the quantitative results was June 4th, 2018, allowing the participants six weeks for the analysis.

All samples were received in good order by the participants. Results were submitted within the deadline with two exceptions. Participants PT052 and PT065 were unable to report results in time (a.o. due to instrument problems).

Participants were asked to provide information on their analysis method (extraction solvent/procedure, clean-up procedure, internal standards used, detection technique, limit of detection, limit of quantification).
4 Evaluation of results


The evaluation is based on assigned values and the standard deviation for proficiency assessment ($\sigma_P$). From this, z-scores are calculated to classify the participants’ performance. Details on the methods used for the statistical evaluation can be found in the background document ‘EURL-MP PT performance assessment’ on the EURL-MP website.

4.1 Calculation of the assigned value

The consensus value based on the participants’ results (NRLs and OLs) was used as the assigned value. The robust mean was used as consensus value in this PT. The values and their uncertainties are summarised in Table 1 in the summary section. Consensus values could be established for all analytes in both materials, except for 15-Ac-DON in material A (wheat) which was below the LOQ as used by the EURL-MP and below the LOQ of the majority of the participants.

4.2 Standard deviation for proficiency assessment ($\sigma_P$)

A fixed relative target standard deviation for proficiency assessment of 25% was used, irrespective the mycotoxin, matrix or concentration. This generic fit-for-purpose value is considered to reflect current analytical capabilities and best practises for mycotoxin and plant toxin determination in food and feed. The rationale behind this is provided in the background document ‘EURL-MP PT performance assessment’ on the EURL-MP website.

4.3 Quantitative performance (z-scores)

For evaluation of numerical results submitted by the participant, z-scores are calculated based on the assigned value, its uncertainty, and the standard deviation for proficiency assessment ($\sigma_P$). When the uncertainty of the assigned value is negligible and no instability of the analytes in the PT material is observed, z-scores are calculated by:

$$z = \frac{x - C}{\sigma_P}$$  

where:

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

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.
|z| ≤ 2 | Satisfactory |
| 2 < |z| < 3 | Questionable |
| |z| ≥ 3 | Unsatisfactory |

If not negligible, the uncertainty of the assigned value and, if applicable, instability of analytes in the PT material, are taken into account in the determination of the z-scores. If applicable, this is indicated by assigning a z’-, zr-, or zi’-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-3G in material A and 15-Ac-DON in material B were not negligible and taken into account in the assignment of the z-score (z’). In all other cases, the uncertainty of the assigned value was negligible. No instability of the analytes in the PT material was observed.

### 4.4 Evaluation of non-quantified results

In case the participant reported '<[value]>', i.e. below their limit of quantification (LOQ), ‘proxy-z-scores’ were calculated as a way to assess possible false negatives and to benchmark the LOQ relative to the assigned value and the LOQ of the other participants.

A proxy-z-score was calculated by Equation 1, using the LOQ value as result. Proxy-z-scores are for information only and indicated as a value between brackets. Values below -2 are considered as false negatives (see 4.5). Values above 2 indicate that the LOQ is high in relation to the assigned value and high in comparison to other participants.

Other types of results, e.g. ‘detected’, or ‘not detected’ without specification of an LOQ, were excluded from the evaluation. In these cases the participant was considered not to have a quantitative method available for the applicable mycotoxin/matrix.

### 4.5 False positives and false negatives

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 the majority of the other participants.

A threshold may apply, below which results are not considered false positives, e.g. when the analyte concentration is below the LOQ of the organiser and/or the majority of the participants. This is decided on a case-to-case basis. False positives are indicated as ‘FP’. False positives are to be interpreted as unsatisfactory performance.

When an analyte is present in the material, i.e. an assigned value has been established, and the participant reports the analyte as '<[value]>', and this value is well below the assigned value, then the result can be classified as a false negative. This is the case when the proxy-z-score (see 4.4) is < -2. False negatives are indicated as ‘FN’. False negatives are to be interpreted as unsatisfactory performance.
5 Assessment of participants’ performance

5.1 Scope and LOQ

This PT was dedicated to DON, 3-Ac-DON, 15-Ac-DON and DON-3G. In Annex 7 the quantitative scope for each participant is provided, with indication of the LOQ provided. It was noted that three participants did not report results for the acetyl-DONs or DON-3G, despite the fact that these compounds were indicated to be in their scope during the a priori survey at the time of registration for the PT. While all laboratories have methods for determination of DON, only 22 out of 50 reported quantitative results for the acetyl-DONs, and only 16 out of 50 for DON-3G. Fourteen laboratories determined all four mycotoxins requested. The LOQs as provided by the participants varied widely, from low µg/kg up to 500 µg/kg. The median LOQs were 50 µg/kg for DON and DON-3G and 25 µg/kg for the acetyl-DONs.

There can be several causes for the gap in the scope observed for many laboratories. A first reason is that only DON is currently regulated, i.e. for analysis in the frame of enforcement inclusion of acetyl-DONs and DON-3G is not yet required. This could be a reason to not (yet) including the other DON forms in the method. Another reason might be that a number of laboratories are using methods involving an immuno-affinity-based clean-up (see 5.2) which may not be suited for simultaneous determination of all four toxins due to poor cross-reactivity [15]. Insight in the reasons for not covering the full scope will be obtained through a follow up questionnaire from the EURL-MP.

The quite extreme differences in LOQs may have several causes. The first is due to differences in analysis methods, i.e. different degrees of concentration factors of the final extract, and differences in sensitivity of (MS) instruments. Another cause may lie in the different ways that LOQs are defined and calculated. Finally, it can also not be excluded that in some cases the LOQ actually is a reporting limit, i.e. a cut-off value below which no results are reported, and is a rather arbitrary value below the regulatory limit but above the actual method LOQ.

Since NRLs are expected to have analytical capabilities not only in the frame of compliance testing of regulatory limits but also in the frame of data generation for risk assessment, efforts should be made toward inclusion of acetyl-DONs and DON-3G, and laboratories are recommended to aim for LOQs in the range of ≤50-100 µg/kg for DON, and ≤10-20 µg/kg for the three DON derivatives.

5.2 Analysis methods

Details on the analytical methods used by the participants are included in Annex 8. The methods used can roughly be categorised in methods based on LC-MS/MS (two thirds), often without clean-up, and methods based on LC-UV (one third) with immunoaffinity column (IAC) clean-up. GC-MS was used by one laboratory.

LC-UV–based methods always involved a clean-up using IAC, and an extraction with water. In most cases, only DON was determined, although one laboratory also reported on all four analytes. The inclusion of other DON derivatives besides DON itself with methods involving IAC clean-up might be difficult as IAC columns often have no or limited cross-reactivity for the DON derivatives [15].

In LC-MS/MS based methods extraction was mostly done using acetonitrile/water (23x), with or without acidification (acetic acid or formic acid). In six cases a salt-induced phase partitioning was done (QuEChERS type of extraction/clean-up). Methanol/water was used by three laboratories. In many cases no clean-up was performed, apart from a phase partitioning in case of QuEChERS-based approaches, or a dilution of the extract. When a clean-up was included, this was by solid phase
extraction (SPE, 8x) or by IAC (4x). Fourteen laboratories used isotopically labelled internal standards, in most cases only for DON. Despite the good possibilities to cover all forms of DON in LC-MS/MS-based methods, ten laboratories reported only DON.

Based on the results and method details provided by the laboratories, no obvious effects of extraction, clean-up or measurement methods on the results were observed.

5.3 Performance

The quantitative performance was assessed through z-scores. For each participant, the individual z-scores for the mycotoxins in material A (wheat) and B (maize) are provided in Annex 9 and 10, respectively. These annexes also show graphical representations of the z-scores.

For DON satisfactory z-scores were obtained by almost all participants in both materials. There were only two exceptions (one NRL and one OL). Combining the results for the two materials, 97% of the z-scores were satisfactory.

As indicated in 5.1, 22 out of the 50 laboratories determined the acetyl-DONs. For 3-Ac-DON in total five unacceptable z-scores were observed, mostly for wheat that contained the lower concentration. 15-Ac-DON was not present in material A (<RL [20 µ/kg] used by the EURL, indicative level 6 µg/kg). 15-Ac-DON was quantified in this material above 20 µg/kg by four laboratories. Those results were classified as false positives. In material B, 15-Ac-DON was present at 154 µg/kg but reported as below LOQ by two laboratories. As the assigned value was well above their LOQs, these results were classified as false negatives. Besides the false negatives, unsatisfactory performance was observed for one other laboratory (z-score >3). Combining the results for both acetyl-DONs in both materials, 81% of the z-scores were satisfactory (here the false positives were considered as unsatisfactory). The poorer performance for the acetyl-DONs may be due to difficulties in the chromatographic analysis. 3-Ac-DON and 15-Ac-DON are often co-eluting under generic chromatographic conditions which makes their determination less straightforward. However, it is possible to separate them chromatographically, and to a certain extend also mass spectrometrically (details see [10]).

DON-3G was included in the analysis by 16 out of the 50 laboratories. In general satisfactory z-scores were obtained in both materials, although due to the relatively low level in material B (maize, 35 µg/kg) only eleven laboratories could quantify this DON conjugate.

A summary of the characteristics and performance of the participants in this PT for each mycotoxin in each material is provided in Table 1 in the Summary.

In Annex 11 an overview is given of the overall performance for each participant in this PT. For the two materials combined, a maximum of seven satisfactory z-scores could be obtained, and ‘7 out of 7’ reflects optimal performance in terms of scope and capability for quantitative determination. The number of laboratories that analysed the materials for all four mycotoxins was fourteen. Of these, seven achieved optimal performance. For the other seven, either the LOQ was too high, false positives or false negatives were reported, or a non-satisfactory z-score was obtained.

5.4 Robust relative standard deviation

For informative purposes the robust standard deviation (RSD\textsubscript{r}) was calculated according to ISO13528:2015 [12]. This provides a good estimation of the interlaboratory variability. The individual RSD\textsubscript{r} values for each toxin in both materials are included in Annex 9 and 10, and also in Table 1. They ranged from 14% for DON-3G in material B (35 µg/kg) to 28% for DON-3G in material A (209 µg/kg).
6 Conclusions

Fifty laboratories, including NRLs from all member states, participated in EURL-PT-MP01 on the quantitative determination of DON, 3-Ac-DON, 15-Ac-DON and DON-3G in cereals (wheat and maize). All laboratories determined DON, but only 44% included the acetyl-DONs, and only 32% DON-3G. Fourteen laboratories analysed the materials for all four target toxins. LOQs varied widely from low µg/kg to 500 µg/kg (medians in the range 25-50 µg/kg). LOQs were generally adequate for compliance testing for DON, but not always for monitoring in the frame of risk assessment.

Two-thirds of the laboratories used methods based on LC-MS/MS, either with or without clean-up. One third used methods based on LC-UV with IAC as clean-up step.

For DON satisfactory results were obtained in almost all cases. For the other three mycotoxins satisfactory performance rates were lower, 81% for the acetyl-DONs and 89% for DON-3G. Only seven out of 50 laboratories obtained satisfactory performance for all four toxins.

The quantitative performance of the participants was generally good, but extension of the scope is needed in many cases (and lower LOQs in some) to align with EFSA monitoring recommendations. In a relatively limited number of cases, a follow up is needed regarding questionable or unsatisfactory z-scores and false positive/false negative results.
References


[10] EURL-MP-method_001 v1, 2018, Determination of deoxynivalenol and related compounds in cereals by LC-MS/MS, EURL mycotoxins and plant toxins, RIKILT Wageningen University & Research. https://www.wur.nl/upload_mm/3/a/2/89801fc3-9656-44b6-989c-61b0c7cc6ef9_EURL-MP-method_001%20DON%20and%20related%20compounds%20by%20LC-MS-MS%20v1.pdf


## Annex 1  List of participants

<table>
<thead>
<tr>
<th>Country</th>
<th>Organisation</th>
</tr>
</thead>
<tbody>
<tr>
<td>AUSTRIA*</td>
<td>AGES Austrian Agency for Health and Food Safety</td>
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<tr>
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* National Reference Laboratory of EU Member State
Annex 2  Codification of the samples

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* All sample codes start with EURLPT-MP 01/.
# Annex 3  Statistical evaluation of homogeneity data

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sx = Standard deviation of the sample averages.  
sw = Within-sample standard deviation.  
ss = Between-sample standard deviation.

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Grand mean 261

Cochran’s test
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Grand mean 730

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$s_w$ = Within-sample standard deviation.
$s_s$ = Between-sample standard deviation.
### 3-Ac-DON in B (µg/kg)

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Replicate 1</th>
<th>Replicate 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hom/B001</td>
<td>103</td>
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</tr>
<tr>
<td>Hom/B002</td>
<td>102</td>
<td>101</td>
</tr>
<tr>
<td>Hom/B003</td>
<td>100</td>
<td>105</td>
</tr>
<tr>
<td>Hom/B004</td>
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<td>Hom/B005</td>
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<td>Hom/B006</td>
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<td>98.4</td>
</tr>
<tr>
<td>Hom/B007</td>
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<td>101</td>
</tr>
<tr>
<td>Hom/B008</td>
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<tr>
<td>Hom/B009</td>
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<tr>
<td>Hom/B010</td>
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Grand mean: 98.8

**Cochran’s test**

<table>
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<tr>
<th>C</th>
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<tbody>
<tr>
<td>0.388</td>
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</tbody>
</table>

**Target s = σp**

<table>
<thead>
<tr>
<th>Cx</th>
<th>Sw</th>
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</thead>
<tbody>
<tr>
<td>3.15</td>
<td>3.43</td>
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**Critical= 0.3 σp**

<table>
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<tr>
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### 15-Ac-DON in B (µg/kg)

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<tr>
<td>Hom/B003</td>
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<td>147</td>
</tr>
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<td>Hom/B006</td>
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<td>143</td>
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<td>Hom/B007</td>
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<td>134</td>
</tr>
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<td>Hom/B008</td>
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<td>Hom/B010</td>
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Grand mean: 144

**Cochran’s test**

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**Target s = σp**

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**Critical= 0.3 σp**

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<tbody>
<tr>
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</table>

$s_x$ = Standard deviation of the sample averages.

$s_w$ = Within-sample standard deviation.

$s_s$ = Between-sample standard deviation.
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<tr>
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<th>Replicate 2</th>
</tr>
</thead>
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<td>Hom/B003</td>
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<td>Hom/B004</td>
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<td>Hom/B005</td>
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<td>Hom/B007</td>
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<td>Hom/B008</td>
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<td>Hom/B009</td>
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<td>Hom/B010</td>
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Grand mean 24.9

Cochran’s test

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</thead>
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Target $s = \sigma_p$

<table>
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<th>$s_w$</th>
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Critical= 0.3 $\sigma_p$

$s_x <$ critical? ACCEPTED

$s_w < 0.5 \sigma_h$? 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 stability data

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

<table>
<thead>
<tr>
<th>Storage temperature</th>
<th>&lt;-70°C</th>
<th>&lt;-18 °C</th>
<th>1 day RT</th>
</tr>
</thead>
<tbody>
<tr>
<td>Time (days)</td>
<td>0</td>
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<tr>
<td>Calculated amounts (µg/kg)</td>
<td></td>
<td></td>
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</tr>
<tr>
<td></td>
<td>507</td>
<td>491</td>
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<td>506</td>
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<td>507</td>
</tr>
<tr>
<td></td>
<td>508</td>
<td>510</td>
<td>493</td>
</tr>
<tr>
<td>Average amount (µg/kg)</td>
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<td>514</td>
<td>511</td>
<td>506</td>
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<td>6</td>
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<td>st. dev (µg/kg)</td>
<td>14.1</td>
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</tbody>
</table>

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

<table>
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<th>Storage temperature</th>
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</thead>
<tbody>
<tr>
<td>Time (days)</td>
<td>0</td>
<td>43</td>
<td>43</td>
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<tr>
<td>Calculated amounts (µg/kg)</td>
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<td>Average amount (µg/kg)</td>
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</table>

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

<table>
<thead>
<tr>
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<th>&lt;-70 °C</th>
<th>&lt;-18 °C</th>
<th>1 day RT</th>
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</thead>
<tbody>
<tr>
<td>Time (days)</td>
<td>0</td>
<td>43</td>
<td>43</td>
</tr>
<tr>
<td>Calculated amounts (µg/kg)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
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### Stability evaluation for DON in material B.

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<td>688</td>
<td>680</td>
<td>722</td>
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<td>705</td>
<td>711</td>
<td>713</td>
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<td>714</td>
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<td>696</td>
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<td>707</td>
<td>712</td>
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<td>Calculated amounts (µg/kg)</td>
<td>723</td>
<td>699</td>
<td>734</td>
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<td>688</td>
<td>680</td>
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<td>660</td>
<td>707</td>
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### Statistical evaluation for 3-Ac-DON in material B.

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<tbody>
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### Statistical evaluation for 15-Ac-DON in material B.

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</tr>
<tr>
<td>0</td>
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<td>163</td>
<td>156</td>
</tr>
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<td></td>
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<td>168</td>
<td>167</td>
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<td></td>
<td>166</td>
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<td>159</td>
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<td></td>
<td>154</td>
<td>158</td>
<td>153</td>
</tr>
<tr>
<td>Calculated amounts (µg/kg)</td>
<td>169</td>
<td>155</td>
<td>162</td>
</tr>
<tr>
<td></td>
<td>156</td>
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<td>159</td>
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<tr>
<td>n</td>
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<td>6</td>
<td>6</td>
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<td>st. dev (µg/kg)</td>
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<td>12.3</td>
<td>12.3</td>
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</table>
### Statistical evaluation for DON-3G in material B.

<table>
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Annex 5  Invitation letter

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

Dear Madam/Sir,

The European Union Reference Laboratory for mycotoxins and plant toxins announces the first proficiency test on deoxynivalenol and related compounds in food and feed matrices, EURLPT-MP01.

Aim of the PT is to provide laboratories with an assessment of their analytical performance and the reliability of their data – in comparison to other laboratories.

Obliged and eligible laboratories

According to Regulation (EU) 2017/625 it is obligatory for EU National Reference Laboratories (NRLs) mycotoxins in food and/or feed to participate.

For NRLs the participation is free of charge. If an extra batch of test materials is needed after the first shipping, the courier costs will be charged.

Official laboratories (OLs) can also participate as long as sufficient test material is available, at a first come first serve basis. The participation fee for OLs is 270 EURO per participant. OLs will be contacted for payment details upon registration.

Deadline for registration is 1 April 2018

Test materials

The test materials will be wheat flour and corn flour. The participants will receive approximately 35 gram of each test material.

Target analytes
This PT will focus on the quantification of deoxynivalenol (DON) as included in Commission Regulation (EC) No 1881/2006 (food) and Commission Recommendation 2006/576/EC (feed).

It furthermore will include three related compounds of deoxynivalenol: 3-acetyl deoxynivalenol (3-Ac-DON), 15-acetyl deoxynivalenol (15-Ac-DON) and deoxynivalenol-3-glucoside (DON-3- Glc). EFSA recommends monitoring of these DON related compounds.

Participation
You can participate by completing the accompanying "EURLPT-MP01 Participation form" and return it before April 1, 2018 to: eurl.mycotoxins-planttoxins@wur.nl.

Shipment of test materials and deadline for submission
The shipment of test materials is scheduled in April week 10-17, 2018. The distribution of the test materials will be announced by email. If any laboratories have holidays during the shipment period, please inform us.

Results must be submitted via the electronic submission form for which each participant must register, as explained in the "EURLPT-MP01 Participation form". See calendar below for complete time schedule EURLPT-MP01.

Reporting
Laboratory proficiency will be determined through z-scores. Confidentiality of results is guaranteed. The results of the proficiency test will be presented anonymously in the report. The report will be published in the public domain of the EURL Mycotoxins & plant toxins website. The results of this PT will be discussed during the EURL workshop.

Kind regards,

Diana Pereboom

Proficiency tests

EURL mycotoxins & plant toxins
RIKILT Wageningen University & Research
the Netherlands

**Calendar EURLPT-MP01**  
(last update March 7 2018)

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<td>Deadline for registration</td>
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<td>Distribution of test materials</td>
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<td>Deadline for result submission</td>
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<td>Preliminary report (only compilation of results)</td>
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<td>Discussion on results</td>
<td>October 9-10, 2018</td>
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<td>Final Report published</td>
<td>November 2018</td>
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Annex 6  Instruction letter

Dear Madam, Sir,

Thank you very much for your interest in the proficiency test for the analysis of deoxynivalenol and related compounds in food and feed matrices. Hereby I send you a parcel containing two randomly coded samples. Each sample consists of approximately 35 grams of test material.

Please fill out the accompanying acknowledgement of receipt form and return it immediately upon receipt of the samples, preferably by e-mail (pt.rikilt@wur.nl)

Instructions:
- After arrival store the samples in the freezer.
- Before analysis, homogenize them according to your laboratory’s procedure.
- Treat the test material as if it was a sample for routine analysis.
- Report one result and not an average of multiple measurements.
- Report all results in µg/kg relative to a feed with a moisture content of 12% (assuming 0% moisture in the sample).
- Please use the web application for entering your results (https://crilwebshop.wur.nl/apex/f?p=107:LOGIN). Information about the use of this web application was sent to you earlier by e-mail.
- The deadline for submitting test results for this test is June 4th 2018.

- Your username is:
- Your password is:
- Your lab code to enter this proficiency test is:
- Please inform us about your applied method and detection technique (via the web application).

Please contact me if you have any questions or need any assistance.
With kind regards.

Diana Pereboom
Proficiency tests

EURL mycotoxins & plant toxins
RIKILT Wageningen University & Research, the Netherlands
## Annex 7  Scope and LOQ

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ACN = acetonitrile; EtOAc = ethyl acetate; FA = formic acid; HAc = acetic acid; MeOH = methanol; PEG = polyethylene glycol (PEG)

SPE = solid phase extraction; IAC = immunoaffinity column
Annex 9  Results material A (wheat)

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

u = uncertainty of consensus value

σ_p = target standard deviation for proficiency

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

**15-Ac-DON**

C: <20 µg/kg  
(≈6 µg/kg)

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**DON-3G**

C: 209 µg/kg  
u: 19.03 µg/kg  
σₚ: 52.2 µg/kg (25%)  
robust σ: 59.0 µg/kg (28%)

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C = consensus 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 a  Graphical representation of the z-scores for DON in material A (wheat). Dotted lines show PT performance boundaries ±2 (also in µg/kg) and ±3.

Figure b  Graphical representation of the z-scores for 3-Ac-DON in material A. Dotted lines show PT performance boundaries ±2 (also in µg/kg) and ±3.
Figure c  Graphical representation of the z*-scores for DON-3G in material B. Dotted lines show PT performance boundaries ±2 (also in µg/kg) and ±3.
## Annex 10  Results material B

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*σp* = target standard deviation for proficiency  
robust *σ* = robust (relative) standard deviation based on participants’ results
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σp = target standard deviation for proficiency  
robust σ = robust (relative) standard deviation based on participants’ results
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C = consensus 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 d  Graphical representation of the z-scores for DON in material B. Dotted lines show PT performance boundaries ±2 (also in µg/kg) and ±3.

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

**Figure g** Graphical representation of the $z$-scores for DON-3G in material B. Dotted lines show PT performance boundaries ±2 (also in µg/kg) and ±3.
Annex 11  Overview performance per laboratory

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

** reported too late
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, 5,000 employees and 10,000 students, 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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Proficiency test for deoxynivalenol (DON), acetyl-DONs and DON-3G in cereals

EURL-PT-MP01 (2018)