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Report on the 2010 Proficiency Test of the European Union Reference Laboratory for Mycotoxins, for the Network of National Reference Laboratories

Determination of Ochratoxin A in Cereals, Green Coffee, Paprika and Test Solution

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The mission of the JRC-IRMM is to promote a common and reliable European measurement system in support of EU policies.

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1. Summary

The Institute for Reference Materials and Measurements (IRMM) of the Joint Research Centre (JRC), a Directorate-General of the European Commission, operates the European Union Reference Laboratory (EU-RL) for Mycotoxins. One of its core tasks is to organise interlaboratory comparisons (ILCs) among appointed National Reference Laboratories (NRLs). This report presents the results of a ILC of the EU-RL for Mycotoxins which focused on the determination of ochratoxin A in food and feed samples.

The test materials were naturally contaminated cereals, green coffee and paprika samples and an ampouled ochratoxin A solution. The materials were labelled at IRMM and dispatched to the participants in May 2010. Each participant received two ampoules of solution and seven sachets containing approximately 30 g of test material each. Thirty-seven participants from 32 countries registered for the exercise. Thirty-six sets of results were reported for the solution, 37 for the cereals, 35 for the green coffee and 35 for the paprika.

The assigned values were 13.2 µg/mL for the test solution, 191 µg/kg for the cereals, 8.0 µg/kg for the green coffee and 13.0 µg/kg for the paprika. The uncertainties of the respective assigned values were 0.9 µg/mL, 9 µg/kg, 0.6 µg/kg and 0.9 µg/kg.

Participants were invited to report the uncertainty of their measurements. This was done by the majority of laboratories.

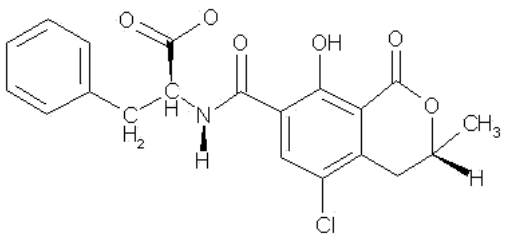
Laboratory results were rated with z-scores and zeta-scores in accordance with ISO 13528 and the International Harmonized Protocol for the Proficiency Testing of Analytical Chemistry Laboratories.

In total about 90% of the attributed z-scores were below an absolute value of two, which indicated that most of the participants performed satisfactory or better than minimal performance criteria required.

2. Introduction

Ochratoxins are pentaketides made up of dihydro-isocoumarin linked to ß-phenylalanine. Ochratoxin A (OTA) *(Figure 1)* is mainly produced by *Aspergillus ochraceus*, *A. carbonarius* and *A. niger* in tropical regions and by *Penicillium verrucosum* in temperate climates. It has been classified as a substance of Group 2B by the International Agency for Research on Cancer (IARC), meaning the existence of sufficient evidence of its renal carcinogenicity to animals and possibly to humans.

Figure 1: Structure of ochratoxin A



Cereals and their derivatives are the major contributor for ingestion of OTA but it is also found in a variety of food products ranging from coffee to nuts, wine, beer, dried fruits and spices.

The methodologies used for the determination of OTA in almost all food and feed matrices range from high-performance liquid-chromatography (HPLC) with various detection systems such as fluorescence (FLD) or mass selective detection (MSD), over thin-layer chromatography (TLC) to enzyme linked immunosorbant assays (ELISA). The most common principle in EU Member States is however HPLC-FLD, which is the basis for all CEN standards for OTA. All methodologies, irrespective of their detection principle, depend on the extraction of OTA from the matrix with a solvent.

Regulations (EC) No 1881/2006[1] and (EC) No 105/2010[2] (EC) lay down maximum limits for OTA in certain foods and methods for sampling and analysis. For feed the guidance values are set in (EC) No 576/2006[3]. *(Table 1)*

Table 1: Regulations and recommendations in the EU regarding the tested matrices in the proficiency test

Matrix	Legislative reference	Maximum limit
cereals (food)	Commission Regulation (EC) No 1881/2006 of 19 December 2006 setting maximum levels for certain contaminants in foodstuffs	5 µg/kg
cereals (feed)	Commission Recommendation of 17 August 2006 on the presence of deoxynivalenol, zearalenone, ochratoxin A, T-2 and HT-2 and fumonisins in products intended for animal feeding (2006/576/EC)	0.25 mg/kg * * guidance value
green coffee		
paprika	Commission Regulation (EU) No 105/2010 of 5 February 2010 amending regulation (EC) No 1881/2006 setting maximum levels for certain contaminants in foodstuffs as regards Ochratoxin A	30 µg/kg (as from 1.7.2010 until 30.6.2012) 15 µg/kg (as from 1.7.2012)

The EU-RL for Mycotoxins has organised a proficiency test (PT) for the network of appointed National Reference Laboratories (NRLs) to determine ochratoxin A in food and feed test samples. Three years ago, in 2007, another proficiency test was conducted for OTA analysis in paprika.[4]

All invited laboratories were free to use their method of choice. Upon request by some NRLs a method that has been previously validated by the JRC was supplied.

3. Scope

As stated in Article 32 of Regulation (EC) No 882/2004[5], one of the core duties of the EU-RL is to organise interlaboratory comparison tests (ILCs) for the benefit of staff from NRLs. The scope of this ILC was to test the competence of the appointed NRLs to determine the concentration of ochratoxin A in food and feed samples.

The ILC was designed and the reported data were processed along the lines of the International Harmonized Protocol for the Proficiency Testing of Analytical Chemical Laboratories (Thompson *et al.* 2006)[6].

The assessment of the measurement results was undertaken on the basis of requirements laid down in legislation and followed administrative and logistic procedures of ISO Guide 43[7].

4. Time frame

The ILC was agreed upon by the NRL network at the fourth EU-RL Mycotoxins workshop held on 26-27 March 2009. Specific details of the exercise were refined during the fifth EU-RL Mycotoxins workshop held on 10-11 March 2010. Invitation letters were sent to the participants on 22 April 2010 *(Annex 13.2)* and the planned ILC was published on the IRMM web page[8]. The samples were dispatched to the participants on 18 May 2010. Reporting deadline was 21 June 2010 which was postponed by a month.

5. Material

5.1. Preparation

The test materials were naturally contaminated cereals, green coffee and paprika test samples from various sources and ampouled ochratoxin A solution in a solvent of 99 parts per volume of toluene and 1 part per volume of glacial acetic acid.

5.2. Homogeneity

Sufficient homogeneity was assumed for the test solution after mixing.

Homogeneities of the contaminated cereals, green coffee and paprika test materials were evaluated according to chapter 3.11.2 of the Harmonized Protocol[6]. The contents of 10 randomly selected test sample sachets were analysed in duplicate by liquid chromatography with fluorescent detection (HPLC-FLD).

All analyses complied with the provisions given by the Harmonized Protocol. Hence it was concluded that the test materials were sufficiently homogeneous. *(Annex 13.1)*

5.3. Stability

The amount of OTA in the test materials was monitored at the beginning of the study, during the study as well as after receipt of the results of the participants as it is suggested in the Harmonized Protocol. Statistically significant differences of the results of analysis obtained on the three mentioned dates were not found.

5.4. Distribution

All samples were packed in polystyrene boxes and sent via express mail. One set of material was sent to every participant. The test materials were dispatched to the participants by IRMM on 18 May 2010. The samples were mostly received within 24 hours after dispatch.

Each participant received: *a)* seven packages containing approximately 30 g of test materials, *b)* two ampoules containing the OTA solution, *c)* an accompanying letter with instructions on sample handling and reporting *(Annex 13.3)*, *d)* a sample receipt form *(Annex 13.4)* and *e)* a registration key for the reporting interface.

The materials were shipped at room temperature; storage however was required to be at -18° C until the analysis was performed. Based on previous experience a short period of 1-2 days without cooling imposes no harm for the material, but a longer period of storage above -18° C had to be avoided.

6. Instructions to participants

The PT aimed to assess the content in three naturally contaminated test samples (marked as "Cereals - Contaminated", "Paprika - Contaminated", "Green Coffee - Contaminated"). The laboratories were asked to report the recovery corrected value in µg/kg, including the recovery in % and measurement uncertainty plus coverage factor. For recovery experiments they had the chance to use the material containers marked as "Blanks". Regarding green coffee two sachets of the same blank material were sent to them. One was for the recovery experiment and the other blank was to assess the original (rather low) amount of ochratoxin A since no completely blank material was available for this PT.

Another aim was to assess the content of ochratoxin A in solution by spectrophotometer and use this solution as basis for their calibration curve. For this, two identical ampoules of solution containing ochratoxin A in 99 parts per volume of toluene and 1 part per volume of glacial acetic acid were supplied. The laboratories were asked to report the value in µg/mL.

The results were to be reported in a special on-line form for which each participant received an individual access code. A specific questionnaire was attached to this on-line form. The questionnaire was intended to provide further information on the measurements and the laboratories. A copy of the questionnaire is presented in *Annex 13.5*.

7. Reference values and their uncertainties

Assigned values and their uncertainties for the OTA content of the test materials were established by "Exact-matching Double Isotope Dilution Mass Spectrometry". This methodology is considered to be a primary ratio method with a direct link to SI units.[9]

Due to time constraints this was only done for the test solution, cereals and the paprika material. For the raw coffee the consensus value of the reported results was used.

The standard uncertainty of the assigned value for the green coffee was determined in accordance with the Harmonised Protocol[6]. They correspond to the standard error of the consensus value, which is given by *Equation 1*:

$$u = \frac{\hat{\sigma}}{\sqrt{n}}$$ **Equation 1.**

where
$\hat{\sigma}$ is robust standard deviation (obtained by AMC algorithm); n is number of results

8. Evaluation of results

8.1. General observations

Thirty-seven participants from thirty-two countries registered to the PT. Thirty-six sets of results were reported for the OTA solution, 37 for cereals, 35 for green coffee and 35 for paprika. Twenty-two laboratories reported uncertainties for OTA in solution, 35 for cereals, 33 for green coffee and 34 for paprika.

8.2. Scores and evaluation criteria

Individual laboratory performance is expressed in terms of z and zeta (ζ) scores in accordance with ISO 13528[10] and the International Harmonised Protocol[6].

$$z = \frac{x_{lab} - X_{ref}}{\sigma_p}$$ **Equation 2.**

$$\zeta = \frac{x_{lab} - X_{ref}}{\sqrt{u^2_{lab} + u^2_{ref}}}$$ **Equation 3.**

where:

x_{lab} is the measurement result reported by a participant
X_{ref} is the reference value (assigned value)
u_{ref} is the standard uncertainty of the reference value
u_{lab} is the standard uncertainty reported by a participant
σ_p is the standard deviation for proficiency assessment (target standard deviation)

σ_p was calculated by the Horwitz equation:

- for analyte concentrations < 120 ppb *(green coffee and paprika)*

$$\sigma_p = 0.22 \cdot c$$ **Equation 4.**

- for analyte concentrations ≥ 120 ppb ≤ 13.8% *(test solution, cereals)*

$$\sigma_p = 0.02 \cdot c^{0.8495}$$ **Equation 5.**

where:
c = concentration of the assigned value, X_{ref}, expressed as a dimensionless mass ratio, e.g. 1 ppb = 10^{-9}, 1 ppm = 10^{-6}

The z score compares the participant's deviation from the reference value with the target standard deviation accepted for the proficiency test, σ_p. The z-score is interpreted as:

$\|z\| \leq 2$	satisfactory result
$2 < \|z\| \leq 3$	questionable result
$\|z\| > 3$	unsatisfactory result

The zeta (ζ) score provides an indication of whether the participant's estimate of uncertainty is consistent with the observed deviation from the assigned value. The ζ-score is the most relevant evaluation parameter, as it includes all parts of a measurement result, namely the expected value, its uncertainty as well as the uncertainty of the assigned values.

The interpretation of the zeta score is similar to the interpretation of the z-score:

$\|\zeta\| \leq 2$	satisfactory result
$2 < \|\zeta\| \leq 3$	questionable result
$\|\zeta\| > 3$	unsatisfactory result

An unsatisfactory |ζ|-score might be due to an underestimation of the uncertainty, or to a large error causing a large deviation from the reference value, or to a combination of the two factors. A laboratory with an unsatisfactory |ζ|-score has an estimation of the uncertainty of its measurements which is not consistent with the laboratory's deviation from the reference value.

8.3. Laboratory results and scoring

Assigned values for the OTA content of the test materials were established by the "Isotope Dilution LC-MS/MS" technique. For the green coffee the consensus value of the reported results was used.

Statistical evaluation of the results was performed using the ProLab software[11]. Kernel density plots were computed from the analytical results by representing the individual numeric values each as a normalised Gaussian distribution centred on the respective analytical value. The sum of these normal distributions forms then the Kernel density distribution.

The robust mean values and robust standard deviations were computed according to Algorithm A of ISO 13528[10] by application of a MS Excel macro that was written by the Analytical Methods Committee of The Royal Society of Chemistry (AMC)[12]. The representative figures are tabulated for each test sample in the following sections of the report.

8.3.1. Test solution

Table 2: Summary statistics for the test solution

Number of results		36
Range of results	µg/mL	11.55 - 26.69
Median	µg/mL	13.6
Mean of results of participants	µg/mL	14.1
Robust mean of results of participants	µg/mL	13.8
Assigned value (isotope dilution LC-MS/MS)	µg/mL	**13.2**
Expanded uncertainty (k=2) of the assigned value	µg/mL	0.9
Robust standard deviation ($\hat{\sigma}$)	µg/mL	0.7
Target standard deviation (fitness for purpose,RSD_R=10.8%)	µg/mL	1.4
Number (percentage) of results of |z| > 2.0		3 (8.3%)

Table 3: Results of analysis and z-scores for the test solution
(The meaning of colours: green – satisfactory, red - unsatisfactory result)

Lab Code	Result [µg/mL]	z-score	Lab Code	Result [µg/mL]	z-score
AA871	**13.806**	0.4	**JP176**	**14.103**	0.6
AF590	**13.5**	0.2	**KF608**	**13.3**	0.1
AN410	**13.58**	0.3	**KN355**	**12.92**	-0.2
AN745	**13.48**	0.2	**MA259**	**14.43**	0.9
BU793	**14.25**	0.7	**MC259**	**12.8**	-0.3
CI716	**18.6312**	3.8	**MC798**	**13.6579**	0.3
CI863	**12.248**	-0.7	**ML947**	**13.29**	0.1
DN411	**12.114**	-0.8	MN644	*No result*	-
DP133	**13.88**	0.5	**NS332**	**26.69**	9.4
ES408	**13.955**	0.5	**OS720**	**17.71**	3.1
GC998	**12.6**	-0.4	**PC100**	**13.3**	0.1
GI812	**14.137**	0.7	**PC105**	**13.313**	0.1
GL869	**12.73**	-0.3	**PG489**	**13.9**	0.5
GU330	**13.138**	0.0	**SB871**	**14.133**	0.7
HN798	**12.2**	-0.7	**SS486**	**15.3652**	1.5
HR099	**13.732**	0.4	**ST117**	**13.51**	0.2
JC489	**13.58**	0.3	**ST638**	**11.55**	-1.2
JK285	**13.95**	0.5	**YM410**	**14.58**	1.0
JN259	**13.95**	0.5			

The results are written as reported by the laboratories.

Figure 2: Individual results of OTA in test solution including the extreme values (E)
The red line corresponds to the reference value (X_{ref}) and the yellow area reflects the reference interval (X_{ref} ± $2u_{ref}$). The green line shows the median value of the results reported by the laboratories.

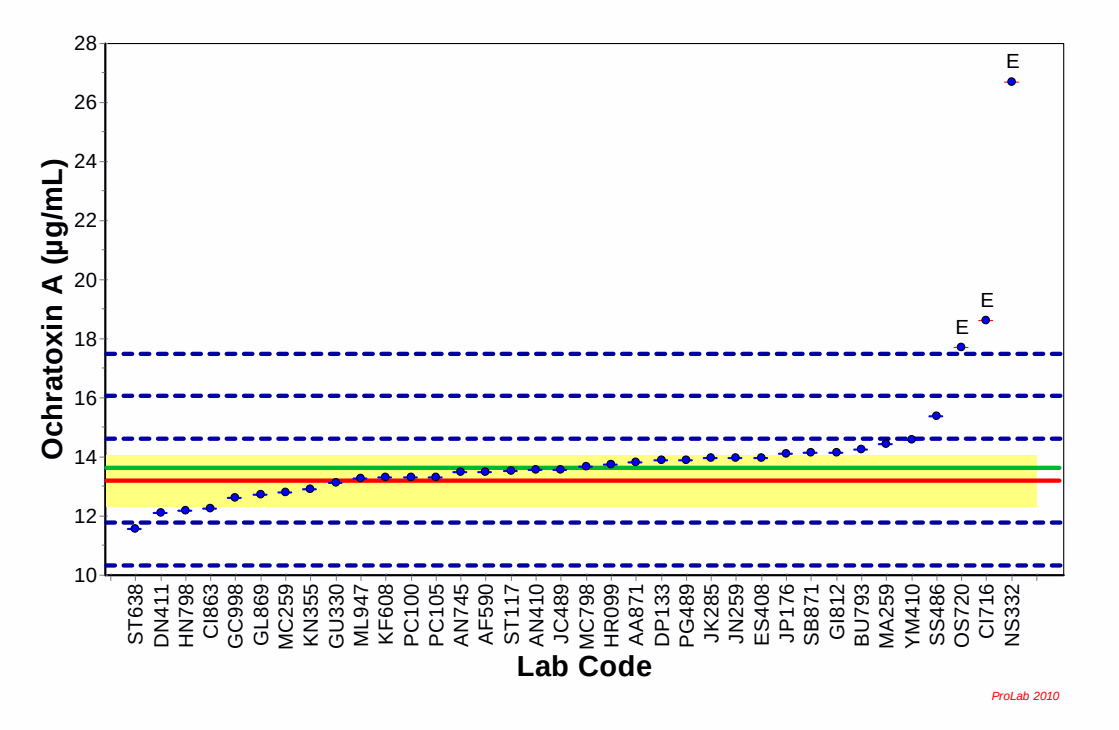
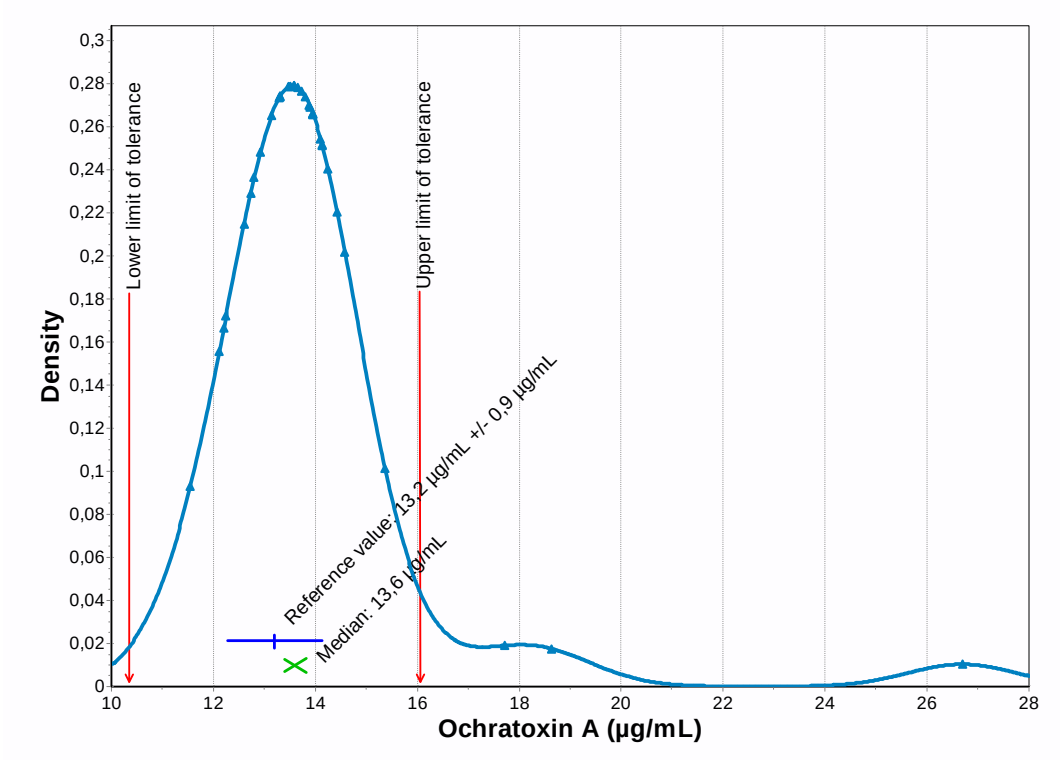


Figure 3: Kernel density plot (test solution)



8.3.2. Cereals

Table 4: Summary statistics for the cereals test sample

Number of results		37
Range of results	µg/kg	5 - 510
Median	µg/kg	191
Mean of results of participants	µg/kg	189
Robust mean of results of participants	µg/kg	190.1
Assigned value (isotope dilution LC-MS/MS)	µg/kg	**191**
Expanded uncertainty (k=2) of the assigned value	µg/kg	9
Robust standard deviation ($\hat{\sigma}$)	µg/kg	41
Target standard deviation (fitness for purpose,RSD_R=20.5%)	µg/kg	39
Number (percentage) of results of |z| > 2.0		3 (8.1%)
Number (percentage) of results of |ζ| > 2.0		7 (18.9%)

Table 5: Results of analysis and z-scores for the cereals test sample
(The meaning of colours: green – satisfactory, yellow – questionable, red - unsatisfactory result)

Lab Code	Result [µg/kg]	z-score	ζ-score	Lab Code	Result [µg/kg]	z-score	ζ-score
AA871	**135**	-1.4	-2.5	**JP176**	**200.8**	0.3	0.6
AF590	**237**	1.2	0.6	**KF608**	**179**	-0.3	-0.2
AN410	**198**	0.2	0.2	**KN355**	**168**	-0.6	-0.5
AN745	**203.9**	0.3	0.4	**MA259**	**222**	0.8	0.8
BU793	**166.5**	-0.6	-1.2	**MC259**	**184.6**	-0.2	-0.5
CI716	**156**	-0.9	-3.8	**MC798**	**193.6**	0.1	0.0
CI863	**163.22**	-0.7	-0.8	**ML947**	**200.22**	0.2	0.2
DN411	**190.5**	0.0	0.0	**MN644**	**5**	-4.7	-20.0
DP133	**138.7**	-1.3	-1.7	**NS332**	**20.78**	-4.3	-16.8
ES408	**136.3**	-1.4	-3.2	**OS720**	**243**	1.3	1.8
GC998	**233.42**	1.1	1.5	**PC100**	**195**	0.1	0.1
GI812	**183**	-0.2	-0.3	**PC105**	**186**	-0.1	-0.3
GL869	**162.1**	-0.7	-1.8	**PG489**	**270.3**	2.0	1.0
GU330	**215**	0.6	0.5	**SB871**	**202.3**	0.3	0.4
HN798	**130.4**	-1.5	-1.6	**SS486**	**141.12**	-1.3	-1.7
HR099	**231.4**	1.0	1.1	**ST117**	**181.4**	-0.2	-0.2
JC489	**202.7**	0.3	0.4	**ST638**	**139.94**	-1.3	-5.5
JK285	**510**	8.1	6.3	**YM410**	**214.8**	0.6	1.1
JN259	**258**	1.7	2.0				

The results are written as reported by the laboratories.

Figure 4: Individual results of OTA in cereals including the extreme values (E)
The red line corresponds to the reference value (X_{ref}) and the yellow area reflects the reference interval (X_{ref} ± $2u_{ref}$). The green line shows the median value of the results reported by the laboratories.

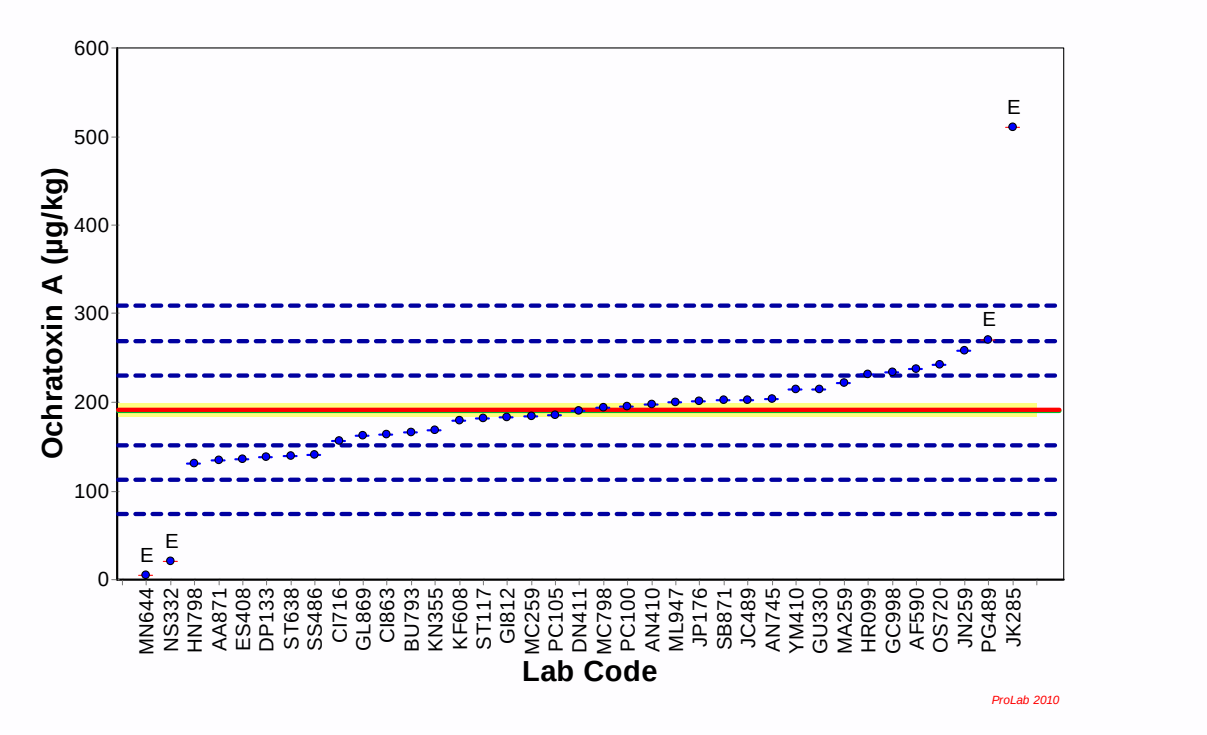
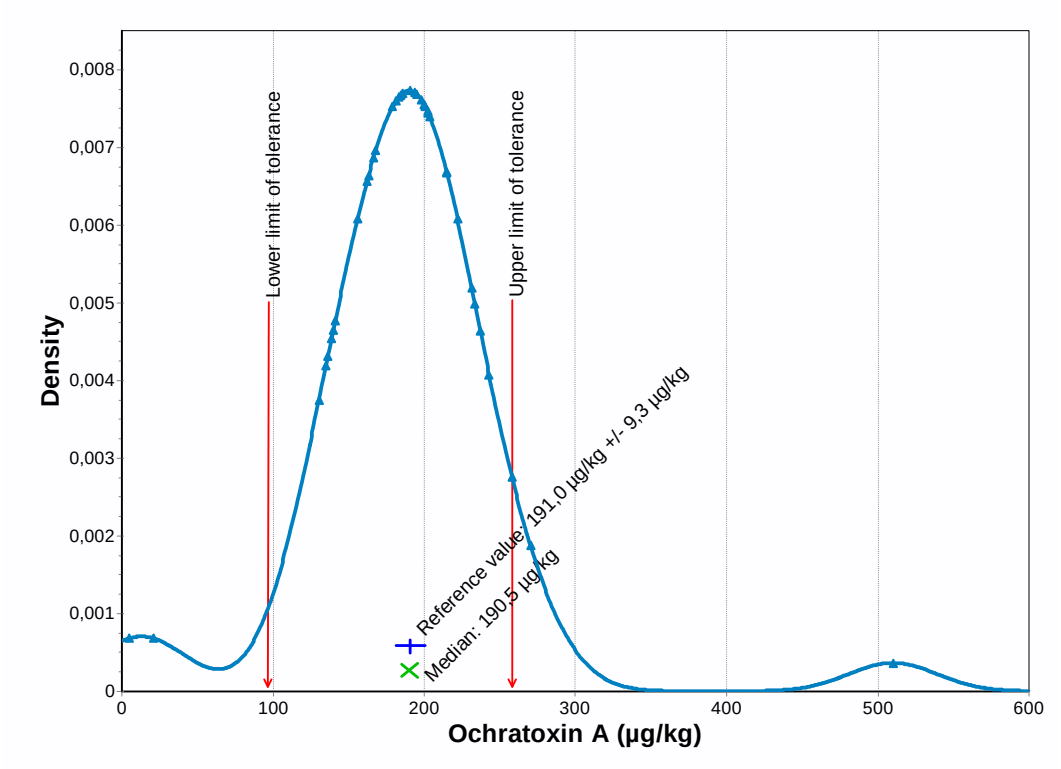


Figure 5: Kernel density plot (cereals)



8.3.3. Green coffee

Table 6: Summary statistics for the green coffee test sample

Number of results		35
Range of results	µg/kg	4.48 - 23.3
Median	µg/kg	8.2
Mean of results of participants	µg/kg	9.1
Robust mean of results of participants	µg/kg	8.0
Assigned value (consensus value of participants' results)	µg/kg	**8.0**
Expanded uncertainty (k=2) of the assigned value	µg/kg	0.6
Robust standard deviation ($\hat{\sigma}$)	µg/kg	1.7
Target standard deviation (fitness for purpose, RSD_R=22 %)	µg/kg	1.8
Number (percentage) of results of |z| > 2.0		5 (14.3%)
Number (percentage) of results of |ζ| > 2.0		5 (14.3%)

Table 7: Results of analysis and z-scores for the green coffee test sample
(The meaning of colours: green – satisfactory, yellow – questionable, red - unsatisfactory result)

Lab Code	Result [µg/kg]	z-score	ζ-score	Lab Code	Result [µg/kg]	z-score	ζ-score
AA871	**6.98**	-0.6	-0.9	**JP176**	**6.5**	-0.9	-1.7
AF590	*No result*	-	-	**KF608**	**7.2**	-0.5	-0.3
AN410	**7.96**	0.0	0.0	**KN355**	**9**	0.6	0.4
AN745	**8.7**	0.4	0.3	**MA259**	**8.2**	0.1	0.1
BU793	**7.16**	-0.5	-1.2	**MC259**	**9.8**	1.0	1.5
CI716	**13.7**	3.2	9.9	**MC798**	**9.5**	0.8	0.4
CI863	**6.84**	-0.7	-0.4	**ML947**	**7.82**	-0.1	-0.1
DN411	**7.7**	-0.2	-0.5	**MN644**	**23.28**	8.6	26.5
DP133	**7**	-0.6	-0.6	**NS332**	**14.67**	3.8	2.3
ES408	**7.85**	-0.1	-0.1	**OS720**	**14.2**	3.5	2.8
GC998	**9.366**	0.8	1.1	**PC100**	**8.15**	0.1	0.0
GI812	**10.5**	1.4	4.1	**PC105**	**7.84**	-0.1	-0.2
GL869	**6.05**	-1.1	-1.1	**PG489**	**8.4**	0.2	0.3
GU330	**12.7**	2.6	1.5	**SB871**	**10.22**	1.2	0.7
HN798	**7.9**	-0.1	-0.1	**SS486**	**7.23**	-0.5	-0.5
HR099	**7.6**	-0.2	-0.3	**ST117**	**6.6**	-0.8	-0.5
JC489	**9.22**	0.7	0.8	**ST638**	**4.48**	-2.0	-3.7
JK285	*No result*	-	-	**YM410**	**8.315**	0.2	0.3
JN259	**10.9**	1.6	1.5				

The results are written as reported by the laboratories.

Figure 6: Individual results of OTA in green coffee including the extreme values (E)
The red line corresponds to the reference value (X_{ref}) and the yellow area reflects the reference interval ($X_{ref} \pm 2u_{ref}$).

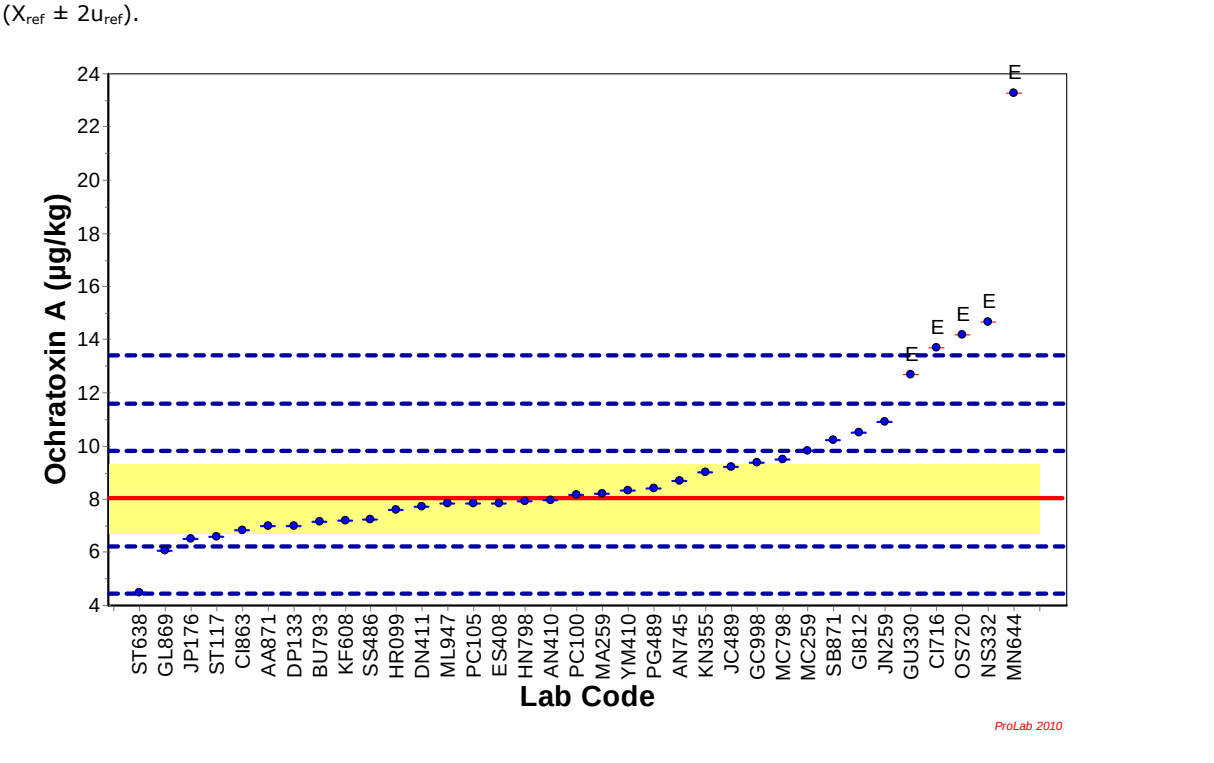
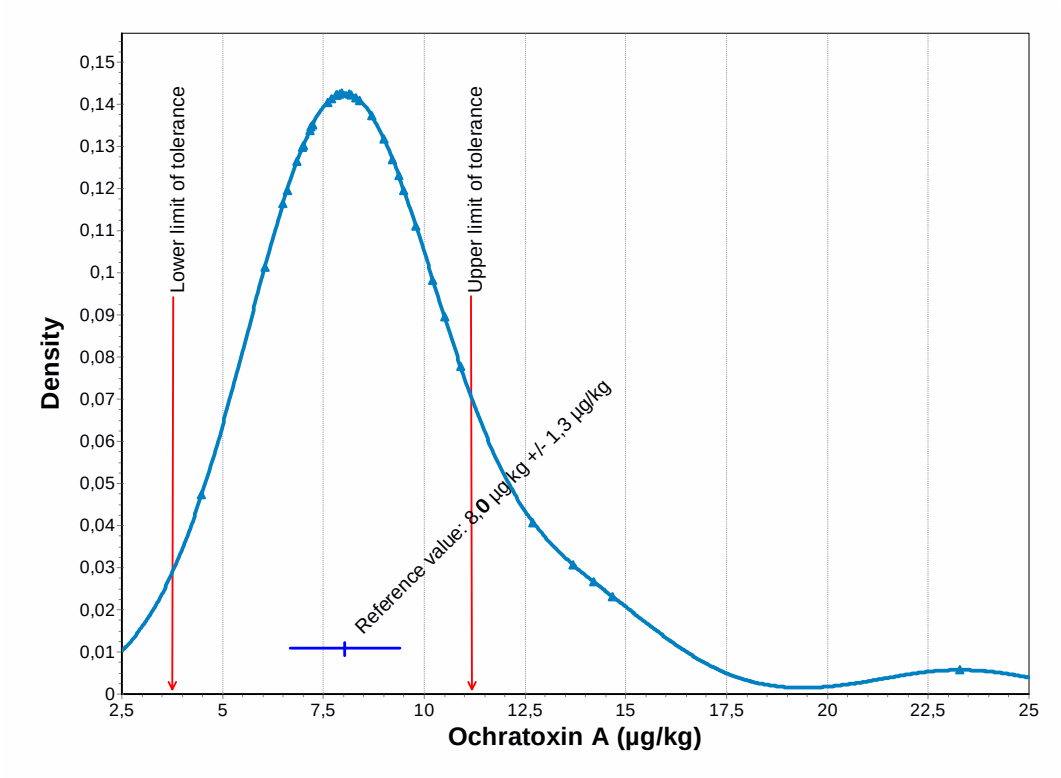


Figure 7: Kernel density plot (green coffee)



8.3.4. Paprika

Table 8: Summary statistics for the paprika test sample

Number of results		35
Range of results	µg/kg	3.62 - 21.39
Median	µg/kg	13.6
Mean of results of participants	µg/kg	13.7
Robust mean of results of participants	µg/kg	14.6
Assigned value (isotope dilution LC-MS/MS)	µg/kg	**13.0**
Expanded uncertainty (k=2) of the assigned value	µg/kg	0.9
Robust standard deviation ($\hat{\sigma}$)	µg/kg	2.8
Target standard deviation (fitness for purpose, RSD_R= 22 %)	µg/kg	2.9
Number (percentage) of results of |z| > 2.0		4 (11.4%)
Number (percentage) of results of |ζ| > 2.0		5 (14.3%)

Table 9: Results of analysis and z-scores for the paprika test sample
(The meaning of colours: green – satisfactory, yellow – questionable, red unsatisfactory result)

Lab Code	Result [µg/kg]	z-score	ζ-score	Lab Code	Result [µg/kg]	z-score	ζ-score
AA871	**12.89**	0.0	-0.1	**JP176**	**14.13**	0.4	0.9
AF590	*No result*	-	-	**KF608**	**13.8**	0.3	0.1
AN410	**12.8**	-0.1	-0.1	**KN355**	**9.1**	-1.4	-1.4
AN745	**13.9**	0.3	0.4	**MA259**	**13.63**	0.2	0.1
BU793	**21.39**	2.9	4.8	**MC259**	**11.8**	-0.4	-0.9
CI716	*No result*	-	-	**MC798**	**20.1**	2.5	1.0
CI863	**11.15**	-0.6	-0.4	**ML947**	**12.79**	-0.1	-0.1
DN411	**17.5**	1.6	1.2	**MN644**	**40**	9.4	29.3
DP133	**9.26**	-1.3	-1.7	**NS332**	**3.62**	-3.3	-8.2
ES408	**14.93**	0.7	0.6	**OS720**	**15.1**	0.7	0.9
GC998	**17.317**	1.5	2.0	**PC100**	**13.2**	0.1	0.0
GI812	**9.92**	-1.1	-1.7	**PC105**	**13.21**	0.1	0.1
GL869	**11.47**	-0.5	-0.4	**PG489**	**14.4**	0.5	0.4
GU330	**14.7**	0.6	0.5	**SB871**	**18.12**	1.8	3.9
HN798	**14.25**	0.4	0.3	**SS486**	**11.73**	-0.4	-0.5
HR099	**13.6**	0.2	0.3	**ST117**	**10.7**	-0.8	-1.1
JC489	**15.7**	0.9	1.1	**ST638**	**10.77**	-0.8	-1.4
JK285	**17.5**	1.6	1.1	**YM410**	**17.6**	1.6	2.4
JN259	**13.4**	0.1	0.2				

The results are written as reported by the laboratories.

Figure 8: Individual results of OTA in paprika including the extreme values (E)
The red line corresponds to the reference value (X_{ref}) and the yellow area reflects the reference interval (X_{ref} ± $2u_{ref}$). The green line shows the median value of the results reported by the laboratories.

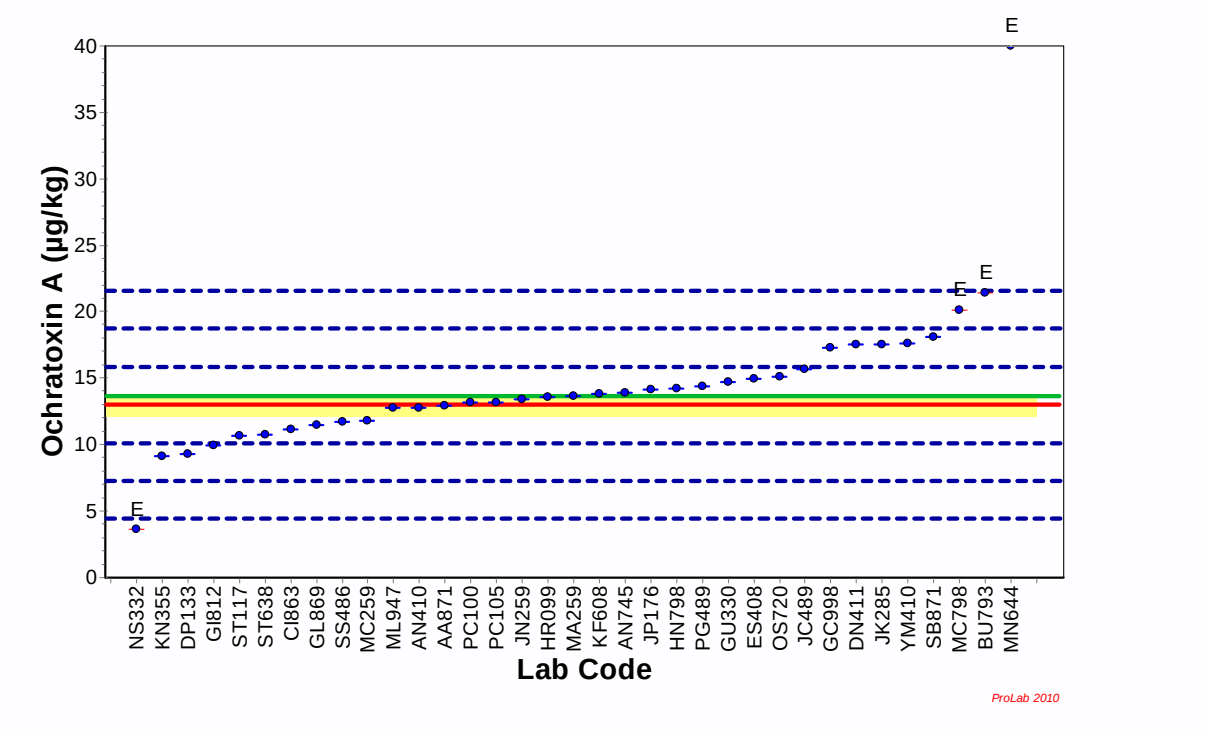
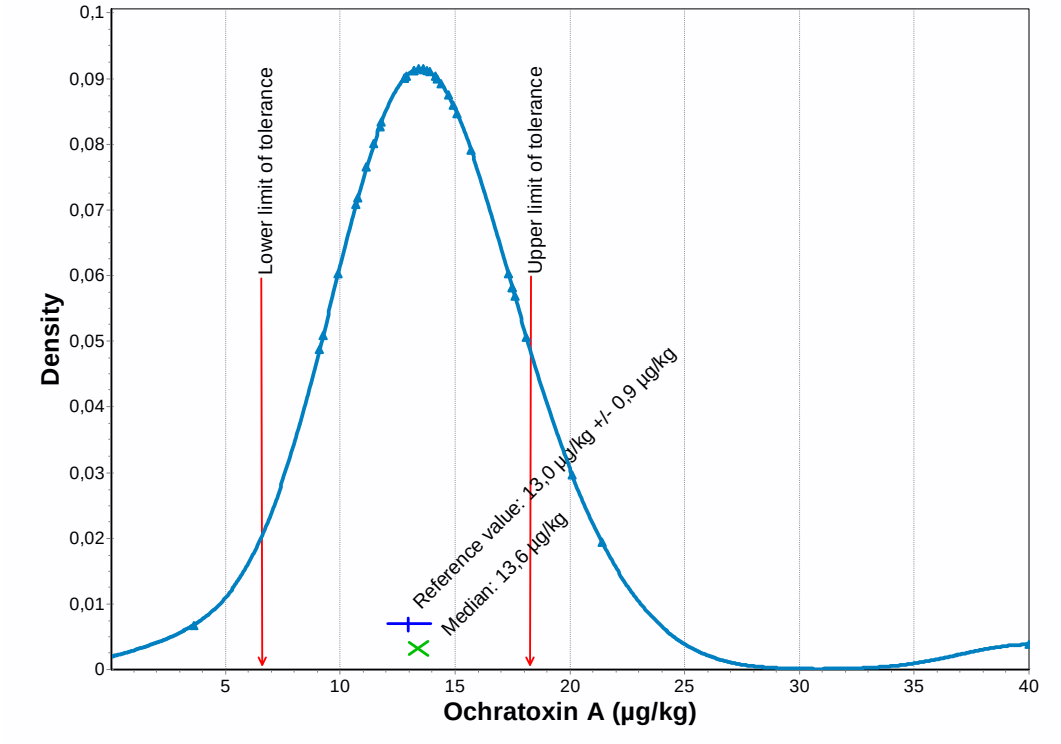


Figure 9: Kernel density plot (paprika)



8.4. Evaluation of the questionnaire

Thirty-six laboratories analyzed the ochratoxin A solution. Even though the EU-RL asked in the accompanying letter to do the analysis with a spectrophotometer, one laboratory analyzed the test solution with ELISA and one with HPLC-FLD technique.

For the recovery estimation nearly all of the participants used a "standard spiked to blank" method. Seven had an overnight stop during the analysis but it didn't have an effect of the results.

All of the laboratories who made the analysis by HPLC-FLD technique used immunoaffinity columns (IAC) as a clean up methodology. The manufacturers and the number of the labs using them are the following: R-Biopharm (20), Vicam (7), Romer Labs (4), Neogen (2), LC Tech (1).

Eighty-nine percent of the participants found the instructions distributed of this PT adequate and regarding the registering-reporting interface the EU-RL received mostly good reviews.

Details on the spectrophotometer conditions, samples preparation and HPLC analyses etc can be found in *Annex 13.6*.

9. Conclusions

Thirty-seven participants from thirty-two countries registered to the interlaboratory comparison for ochratoxin A of which 36 sets of results were reported for the test solution, 37 for cereals, 35 for green coffee and 35 for paprika.

The performance of most participants was very good, particularly when taking into account that several matrices had to be analysed and the green coffee was new to most. In total about 90% of the attributed z-scores were below an absolute value of two, which indicated that most of the participants performed satisfactory or better than minimal performance criteria required. The analysis of all data sorted either by analytical technique or sample preparation method showed no general tendencies. The great majority of NRLs in this interlaboratory comparison applied analytical methods which, with the regard to performance characteristics, were compliant with EU legislation.

Only z-scores are used for benchmarking and laboratories with unsatisfactory z-scores will be invited for a corrective action.

10. Acknowledgements

The organisers of the study would like to thank *Franz Ulberth*, *Anne-Mette Jensen*, *Lubomir Karasek* and *Donata Lerda* for their support and the Reference Materials Unit at IRMM, in particular *Håkan Emteborg*, for ampouling of the test samples.

The authors also would like thank to the colleagues at EU-RL Mycotoxins group for their help at daily work and revising this report: *Katrien Bouten, Helena Ernst*, *Marta Herrera Sanchez*, *Anna Kolossova*, *Katy Kroeger-Negoita* and *Vytautas Tamosiunas*.

The laboratories participating in this exercise, listed in *Table 10*, are also kindly acknowledged.

Table 10: Participating laboratories (Countries shown with italic letters are laboratories outside of the European Union.)

Organisation	Country
AGES GmbH	AUSTRIA
CODA-CERVA	BELGIUM
NDRVMI	BULGARIA
State General Laboratory	CYPRUS
Czech Agricultural and Food Inspection Authority	CZECH REPUBLIC
National Food Institute	DENMARK
Ministry of Food, Agriculture and Fisheries	DENMARK
Agricultural Research Centre	ESTONIA
Finnish Customs Laboratory	FINLAND
Finnish Food Safety Authority Evira	FINLAND
Laboratoire SCL de Rennes	FRANCE
Bundesinstitut für Risikobewertung	GERMANY
General Chemical State Laboratory	GREECE
Central Agricultural Office, Food and Feed Safety Directorate (Feed NRL)	HUNGARY
Central Agricultural Office, Food and Feed Safety Directorate (Food NRL)	HUNGARY
Central Food Laboratory	*INDIA*
Public Analyst's Laboratory	IRELAND
Istituto Superiore Di Sanita'	ITALY
Institute of Food Safety, Animal Health and Environment „BIOR"	LATVIA
National food and veterinary risk assessment institute	LITHUANIA
Laboratoire National de santé	LUXEMBOURG
Public Health Laboratory Malta	MALTA
RIKILT	NETHERLANDS
Servicio Nacional De Sanidad Agraria-Senasa	*PERU*
National Veterinary Research Institute	POLAND
National Institute of Hygiene	POLAND
Instituto Nacional de Investigação Agrária (INIA)	PORTUGAL
Sanitary Veterinary and Food Safety Directorate Bucharest	ROMANIA
University of Novi Sad	*SERBIA*
State Veterinary and Food Institute	SLOVAKIA
University in Ljubljana, Veterinary Faculty-National Veterinary Institute	SLOVENIA
Centro Nacional De Alimentacion (Spanish Food Safety and Nutrition Agency)	SPAIN
National Food Administration	SWEDEN
National Veterinary Institute (SVA)	SWEDEN
Kantonales Laboratorium Basel-Landschaft	*SWITZERLAND*
Tubitak	*TURKEY*
The Food & Environment Research Agency	UNITED KINGDOM

11. Abbreviations

ANOVA	Analysis of variance
CEN	European Committee for Standardisation
EC	European Commission
ELISA	Enzyme linked immunosorbant assays
EU	European Union
EU-RL	European Reference Laboratory
FLD	Fluorescent detection
HPLC	High-performance liquid chromatography
IAC	Immunoaffinity column
ILC	Interlaboratory Comparison
IRMM	Institute for Reference Materials and Measurements
ISO	International Organisation for Standardisation

IUPAC	International Union for Pure and Applied Chemistry
JRC	Joint Research Centre
NRL	National Reference Laboratory
OTA	Ochratoxin A
PT	Proficiency Test
TLC	Thin-layer chromatography

12. References

[1] EU, Commission Regulation (EC) No 1881/2006 of 19 December 2006 setting maximum levels for certain contaminants in foodstuffs. Official Journal of the European Union, 2006. L 364: p. 5-24.
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http://www.fsai.ie/uploadedFiles/Legislation/FSAI_-_Legislation/2010/02_feb2010/Reg105_2010.pdf

[3] Commission Recommendation of 17 August 2006 on the presence of deoxynivalenol, zearalenone, ochratoxin A, T-2 and HT-2 and fumonisins in products intended for animal feeding
http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2006:229:0007:0009:EN:PDF

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http://www.irmm.jrc.be/html/CRLs/crl_mycotoxins/interlaboratory_comparisons/EUR23382EN.pdf

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http://www.rsc.org/pdf/amc/brief6.pdf

13. Annexes

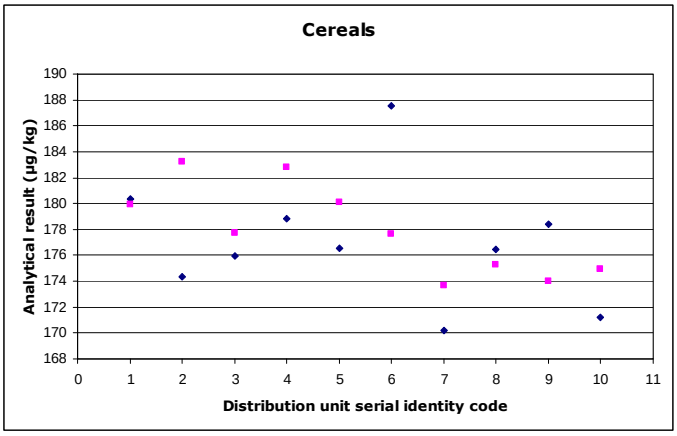
13.1. Homogeneity tests

Homogeneities of the contaminated cereals, green coffee and paprika test materials were evaluated according to chapter 3.11.2 of the Harmonized Protocol(5).

Table 11: Duplicated results for 10 distribution units of cereal flour analysed for OTA (µg/kg), together with some intermediate stages of the ANOVA calculation

Sample	Result *a*	Result *b*	$D = a - b$	$S = a + b$	$D^2 = (a - b)^2$
1	180,4	179,9	0,4	360,3	0,20
2	174,3	183,2	-8,9	357,5	78,57
3	175,9	177,8	-1,8	353,7	3,36
4	178,8	182,8	-4,0	361,7	16,09
5	176,5	180,1	-3,5	356,6	12,46
6	187,5	177,7	9,8	365,2	96,55
7	170,2	173,6	-3,5	343,8	11,95
8	176,4	175,3	1,1	351,7	1,31
9	178,4	174,0	4,4	352,5	19,33
10	171,2	174,9	-3,8	346,1	14,13

Figure 14: Analytical results of the homogeneity study of *cereal flour* test material



The data are presented visually above, and show no suspect features such as discordant duplicated results, outlying samples, trends, discontinuities, or any other systematic effects.

Cochran's test: The largest value of D^2 is 96.55 and the sum of D^2 is 253.94, so the Cochran test statistic is 96.55/253.94=0.380. This is less than the critical value of 0.602 for this type of test, so there is no evidence for analytical outliers and we proceed with the complete data set.

Homogeneity test

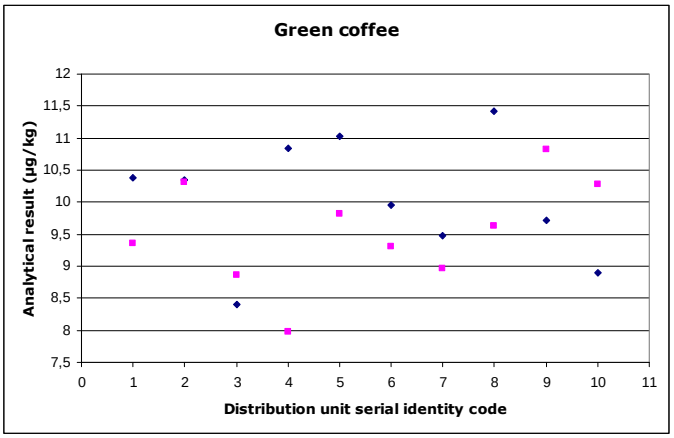
- Analytical variance: $s_{an}^2 = \Sigma D^2/2m = 253.94/20 = 12.70$
- Between-sample variance: the variance of the sums S = a + b is 45.34, so $s_{sam}^2 = (V_s/2 - s_{an}^2)/2 = (45.34/2 - 12.70)/2 = 4.99$
- Acceptable between-sample variance: the target standard deviation is 36.38 µg/kg, so the allowable between-sample variance is $\sigma_{all}^2 = (0.3\sigma_p)^2 = (0.3 \text{ X } 36.38)^2 = 119.10$
- Critical value: The critical value for the test is $1.88\ \sigma_{all}^2 + 1.01\ s_{an}^2 = 1.88 \text{ X } 119.10 + 1.01 \text{ X } 12.70 = 236.74$

Since $s_{sam}^2 = 4.99 < 236.74$, passed and the cereal material is sufficiently homogeneous.

Table 12: Duplicated results for 10 distribution units of green coffee analysed for OTA (µg/kg), together with some intermediate stages of the ANOVA calculation

Sample	Result *a*	Result *b*	*D = a - b*	*S = a + b*	$D^2 = (a - b)^2$
1	10,4	9,4	1,0	19,7	1,02
2	10,4	10,3	0,0	20,7	0,00
3	8,4	8,9	-0,5	17,3	0,22
4	10,8	8,0	2,9	18,8	8,27
5	11,0	9,8	1,2	20,8	1,45
6	10,0	9,3	0,7	19,3	0,42
7	9,5	9,0	0,5	18,4	0,25
8	11,4	9,6	1,8	21,1	3,20
9	9,7	10,8	-1,1	20,5	1,24
10	8,9	10,3	-1,4	19,2	1,93

Figure 15: Analytical results of the homogeneity study of *green coffee* test material



The data are presented visually above, and show no suspect features such as discordant duplicated results, outlying samples, trends, discontinuities, or any other systematic effects.

Cochran's test

The largest value of D^2 is 8.27 and the sum of D^2 is 18.01, so the Cochran test statistic is 8.27/18.01=0.459. This is less than the critical value of 0.602 for this type of test, so there is no evidence for analytical outliers and we proceed with the complete data set.

Homogeneity test

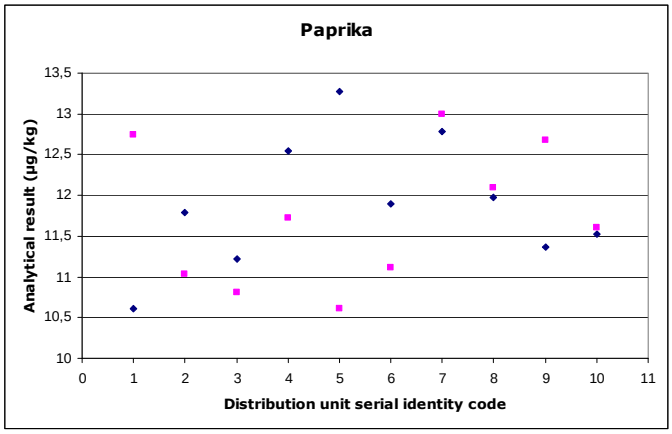
- Analytical variance: $s_{an}^2 = \sum D^2/2m = 18.01/20 = 0.90$
- Between-sample variance: the variance of the sums S = a + b is 1.50, so $s_{sam}^2 = (V_s/2 - s_{an}^2)/2 = (1.50/2 - 0.90)/2 = -0.076$
- Acceptable between-sample variance: the target standard deviation is 2.15 µg/kg, so the allowable between-sample variance is $\sigma_{all}^2 = (0.3\sigma_p)^2 = (0.3 \text{ X } 2.15)^2 = 0.418$
- Critical value: The critical value for the test is $1.88\ \sigma_{all}^2 + 1.01\ s_{an}^2 = 1.88 \text{ X } 0.418 + 1.01 \text{ X } 0.90 = 1.695$

Since $s_{sam}^2 = -0.076 < 1.695$, passed and the green coffee material is sufficiently homogeneous.

Table 13: Duplicated results for 10 distribution units of paprika analysed for OTA (µg/kg), together with some intermediate stages of the ANOVA calculation

Sample	Result *a*	Result *b*	$D = a - b$	$S = a + b$	$D^2 = (a - b)^2$
1	10,6	12,7	-2,1	23,4	4,54
2	11,8	11,0	0,8	22,8	0,56
3	11,2	10,8	0,4	22,0	0,17
4	12,6	11,7	0,8	24,3	0,67
5	13,3	10,6	2,7	23,9	7,13
6	11,9	11,1	0,8	23,0	0,62
7	12,8	13,0	-0,2	25,8	0,04
8	12,0	12,1	-0,1	24,1	0,01
9	11,4	12,7	-1,3	24,1	1,72
10	11,5	11,6	-0,1	23,1	0,00

Figure 16: Analytical results of the homogeneity study of *paprika* test material



The data are presented visually above, and show no suspect features such as discordant duplicated results, outlying samples, trends, discontinuities, or any other systematic effects.

Cochran's test

The largest value of D^2 is 7.13 and the sum of D^2 is 15.47, so the Cochran test statistic is 7.13/15.47=0.461. This is less than the critical value of 0.602 for this type of test, so there is no evidence for analytical outliers and we proceed with the complete data set.

Homogeneity test

- Analytical variance: $s_{an}^2 = \sum D^2/2m = 15.47/20 = 0.774$
- Between-sample variance: the variance of the sums S = a + b is 1.04, so $s_{sam}^2 = (V_s/2 - s_{an}^2)/2 = (1.04/2 - 0.774)/2 = -0.126$
- Acceptable between-sample variance: the target standard deviation is 2.60 µg/kg, so the allowable between-sample variance is $\sigma_{all}^2 = (0.3\sigma_p)^2 = (0.3 \text{ X } 2.60)^2 = 0.609$
- Critical value: The critical value for the test is $1.88\ \sigma_{all}^2 + 1.01\ s_{an}^2 = 1.88 \text{ X } 0.609 + 1.01 \text{ X } 0.774 = 1.926$

Since $s_{sam}^2 = -0.126 < 1.926$, passed and the paprika material is sufficiently homogeneous.

13.2. Invitation letter to laboratories

EUROPEAN COMMISSION
JOINT RESEARCH CENTRE

Institute for reference materials and measurements
European Reference Laboratory for Mycotoxins

CRL Mycotoxins

Geel, 22 April 2010
JRC.DDG.D.6/JS/bk/ARES(2010)209398

Interlaboratory Comparison of the EU-RL for Mycotoxins

Dear Madame/Sir,

On behalf of the EU-RL for Mycotoxins, I announce the opening of the interlaboratory comparison for the determination of Ochratoxin A in cereal flour, paprika powder and green coffee and in a solvent solution.

This proficiency test (PT) was announced during the last EU-RL Mycotoxins workshop. The details on the PT design will be communicated upon sample dispatch.

The EU-RL Mycotoxins would like to inform you that, according to Regulation (EC) No 882/2004, the participation of activities organised by the EU-RL is mandatory for the NRLs.

Participation is free of charge.

Confidentiality of the participants and their results are granted.

Registration of participants is open from 00:00 of 23/04/2010 to midnight of 03/05/2010.

Dispatch of the PT materials is foreseen to be on the 18th May and will be announced in advance.

In order to register, laboratories must:

1. Enter the details online:

   https://irmm.jrc.ec.europa.eu/ilc/ilcRegistration.do?selComparison=439

   When accessing this page you might be confronted with a Certificate Error page, please press the continue button to proceed with the registration.

2. Print the completed form (approved and confirmed version) when the system asks to do so, sign it and stamp it with your company stamp

3. Send it to the EU-RL Mycotoxins members indicated below

Retieseweg 111, B-2440 Geel - Belgium. Telephone: (32-14) 571 211. http://irmm.jrc.ec.europa.eu
Telephone: direct line (32-14) 571 229. Fax: (32-14) 571 783.

E-mail: jrc-irmm-crl-mycotox@ec.europa.eu

The PT coordinator is:

Zoltan KUNSAGI
Fax: +32 14 571 783
Email: JRC-IRMM-CRL-MYCOTOX@ec.europa.eu

Deadline for reporting will be the 21st June. You will receive the link for entering the results upon reception of the PT samples.

A detailed outline of the PT will accompany the PT sample parcel; anyhow we would like to encourage you to contact us in case you seek further clarification.

Please contact us at the mail address:

JRC-IRMM-CRL-MYCOTOX@ec.europa.eu

With kind regards,

Joerg Stroka
(Operating Manager of the EU-RL Mycotoxins)

Cc: Franz Verstraete, Franz Ulberth, Anne-Mette Jensen, Zoltan Kunsagi, Donata Lerda

13.3. Accompanying letter

EUROPEAN COMMISSION
JOINT RESEARCH CENTRE

Institute for reference materials and measurements
European Reference Laboratory for Mycotoxins

CRL Mycotoxins

Geel, 18 May 2010
JRC.DDG.D.6/JS/hn/ARES(2010)-263289

Ref: 2010 Proficiency Testing of National Reference Laboratories (NRLs) on Ochratoxin-A toxin in cereals, green coffee, paprika powder and solution.

Dear Participant,

Please read the following information carefully before starting any analysis. In case of questions, do not hesitate to contact us by either phone or email (see details below).

Please report by fax or e-mail immediately the parcel's receipt, by using the "**Receipt form**". If any material has been received damaged, immediately request a new material.

The materials are shipped at room temperature; storage however should be at -18° C until the analysis is performed. A short period of 1-2 days without cooling is no harm for the material, but a longer period of storage above -18° C shall be avoided.

The 2010 PT aims to:

1. Assess the content in three contaminated test samples (marked as "Cereals - Contaminated", "Paprika - Contaminated", "Green Coffee - Contaminated"). You will be asked to report the **recovery corrected value** in µg/kg, including your **recovery** in % and **measurement uncertainty** plus **coverage factor**.

For recovery experiments you have the chance to use the material containers marked as "Blanks".

Regarding **green coffee we sent you two blank materials.** One is for the recovery experiment and the other blank is to assess the original (rather low) amount of Ochratoxin A since no completely blank material is available for this PT.

2. Assess **by spectrophotometer** the content of Ochratoxin A in solution and use this solution as basis for your calibration curve. For this, two **IDENTICAL** ampoules of solution containing Ochratoxin A in 99 parts per volume of toluene and 1 part per volume of glacial acetic acid are supplied. The OTA content is in the range of 10-30 µg/mL. Report the value in exactly this unit [µg/mL].

Please calculate the concentration of Ochratoxin A according to the below mentioned procedure shaded in gray background. This procedure refers to CEN working document N434 (*Foodstuffs — Determination of Ochratoxin A in currants, raisins, sultanas, mixed dried fruit and dried figs — HPLC method with immunoaffinity column cleanup and fluorescence detection*):

Retieseweg 111, B-2440 Geel - Belgium. Telephone: (32-14) 571 211. http://irmm.jrc.ec.europa.eu
Telephone: direct line (32-14) 571 229. Fax: (32-14) 571 783.

E-mail: jrc-irmm-crl-mycotox@ec.europa.eu

WARNING – Ochratoxin A is a potent nephrotoxin and liver toxin and has been reported to have immuno-suppressant properties. Gloves and safety glasses should be worn at all times and all standard and sample preparation stages should be carried out in a fume cupboard.

To determine the exact concentration, record the absorption curve between a wavelength of 300 nm and 370 nm in a 1 cm quartz cell with solvent mixture (99 parts per volume of toluene and 1 part per volume of glacial acetic acid) as reference using spectrometer. Identify the wavelength for maximum absorption. Calculate the mass concentration of Ochratoxin A, ρ_{OTA} in micrograms per millilitre using this equation:

$$\rho_{OTA} = \frac{A_{max} \times M \times 100}{\varepsilon \times b}$$

where

A_{max} is the absorption determined at the maximum of the absorption curve (here: at 333 nm)

M is the molar mass, in grams per mol, of Ochratoxin A (M = 403.8 g/mol)

ε is the molar absorption coefficient, in square meters per mol, of ochratoxin A in the solvent mixture (here: 544 m^2/mol)

b is the optical path length, in centimeters, of the quartz cell

Please report all requested results and answer the questionnaire at

https://irmm.jrc.ec.europa.eu/ilc/ilcReporting.do

The password key for this interface is included in the parcel with the test materials. When you enter the code please pay attention to the capital letters!

Print out the pdf and return the signed and stamped Report sheet NOT later than **21st June 2010** to:

Zoltan Kunsagi
JRC-IRMM-FSQ
CRL Mycotoxins
Retieseweg 111
B-2440 Geel, Belgium
Tel: +32-14-571 313
FAX: +32-14-571 783
E-mail: Jrc-irmm-crl-mycotox@ec.europa.eu

In case of questions please do not hesitate to contact us.

Jörg Stroka
(Operating Manager of the Community Reference Laboratory for Mycotoxins)

Cc: Frans Verstraete, Anne-Mette Jensen, Franz Ulberth, Zoltan Kunsagi, Donata Lerda

13.4. Acknowledgment of receipt form

EUROPEAN COMMISSION
JOINT RESEARCH CENTRE

Institute for reference materials and measurements
Community reference laboratory for mycotoxins

CRL Mycotoxins

Geel, 18th May 2010

PROFICIENCY TESTING MATERIALS RECEIPT FORM

Name, Institute and Member State of the NRL:

NOTE: UPON RECEIPT STORE ALL MATERIALS IN A FREEZER AT -18 °C!

Please ensure that the items listed below have been received undamaged, and then check the relevant statement:

Date of the receipt	
All items have been received undamaged	Yes □/ No □
Items are missing/damaged. I require an additional series of samples	Yes □/ No □

Contents of parcel

a) Two ampoules containing a solution of Ochratoxin-A
b) Seven test materials for analysis:
- 1 cereal - contaminated
- 1 paprika - contaminated
- 1 green coffee - contaminated
- 1 cereal - blank
- 1 paprika - blank
- 2 green coffee - blank

Please fax or e-mail the completed form to:

Zoltan Kunsagi
JRC-IRMM-FSQ
CRL Mycotoxins
Retieseweg 111
B-2440 Geel, Belgium
Tel: +32-14-571 313
FAX: +32-14-571 783
E-mail: zoltan.kunsagi@ec.europa.eu

13.5. Questionnaire

Milc questionnaire

Comparison for PT 2010 OTA

Please fill in your results and answers to the questions. Print the final pdf and return the signed and stamped copy by fax +32 14 571 783 or by e-mail to JRC-IRMM-CRL-MYCOTOX@ec.europa.eu

Submission Form

1. **How many samples does your laboratory analyse for Ochratoxin A per year?** *

- ○ a) 0-50 samples per year
- ○ b) 50-250 samples per year
- ○ c) 250-1000 samples per year
- ○ d) more than 1000 samples per year

2. **Which of the following matrices does your laboratory analyse for Ochratoxin A on a routine basis?** *

- ☐ a) cereal flour
- ☐ b) green coffee
- ☐ c) paprika powder

2.1. Please specify the other matrices your laboratory analyse for OTA

3. **Are you accredited for the determination of Ochratoxin A?** *

- ○ a) Yes
- ○ b) No

3.1. If YES, please specify the matrices *

4. **Proficiency test samples: CEREALS CONTAMINATED**

4.1. What was the recovery (%) ? *

4.2. Please indicate the LOD for Ochratoxin A of the method used (µg/kg). *

4.3. Please indicate the LOQ for Ochratoxin A of the method used (µg/kg). *

5. **Proficiency test samples: PAPRIKA CONTAMINATED**

5.1. What was the recovery (%) ? *

5.2. Please indicate the LOD for Ochratoxin A of the method used (µg/kg). *

5.3. Please indicate the LOQ for Ochratoxin A of the method used (µg/kg). *

6. **Proficiency test samples: GREEN COFFEE CONTAMINATED**

6.1. What was the recovery (%) ? *

6.2. Please indicate the LOD for Ochratoxin A of the method used (µg/kg). *

6.3. Please indicate the LOQ for Ochratoxin A of the method used (µg/kg). *

7. **Spectrophotometric analysis**

7.1. What is the brand and model of your UV-spectrophotometer? *

7.2. Is this instrument a single or a two channel photometer? *

7.3. What type of light-source was used during the spectrophotometric measurement? *

- a) Tungsten
- b) Hg lamp
- c) Deuterium
- d) other

7.3.1. If other, please specify! *

7.4. Did you calibrate your UV-spectrophotometer? *

- a) Yes
- b) No

7.4.1. If YES, what procedure in short did you use (e.g. K2Cr2O7 solution or calibrated filter, give refer *

7.5. At which wavelength did you identify the maximum for Ochratoxin A (nm)? *

7.6. Optical path length of the cuvette (cm): *

7.7. What was the absorbance reading you obtained with the spectrophotometer? *

7.8. Do you normally check your calibrants by UV-spectrophotometry? *

- a) Yes
- b) No

8. **How did you perform the recovery estimate?** *

- a) C13 Standard to Extract
- b) C13 Standard to Sample
- c) Internal Standard to Extract
- d) Internal Standard to Sample
- e) Standard to Blank
- f) other

8.1. If other please specify *

9. **During the analysis did you need to include any over night stop?** *

- a) Yes
- b) No

9.1. If YES, please state for which samples and at what stage of the analysis. *

10. **What was the extraction solvent used?** *

11. **What was the extraction time?** *

12. **What was the extraction mode (e.g. blending or shaking)?** *

13. **What was the extraction solvent to sample ratio used during extraction (in mL/g)?** *

14. **What type of clean up methodology was used (e.g. immunoaffinity column)?** *

15. **If you used immunoaffinity columns...**

15.1. ... please specify the manufacturer of the immunoaffinity columns you used during the analysis

15.2. ... did you follow the instructions by the manufacturer ?

- a) Yes
- b) No

15.2.1. If NO, please give some brief details on the modification: *

15.3. ... please specify the capacity of the immunoaffinity column (ng)

16. **What type of detection method did you use?** *

- a) HPLC-FLD
- b) LC-MS/MS
- c) GC-MS
- d) ELISA
- e) TLC
- f) other

16.1. If HPLC-FLD, please specify your method (type of column, injection volume, mobile phase etc.)! *

16.2. If LC-MS/MS, please specify your method! *

16.3. If GC-MS, please specify your method! *

16.4. If ELISA, please specify the method: self-developed or commercial kits (from which producer), direc *

16.5. If TLC, please specify your method! *

16.6. If other, please specify the type of your method! *

17. **How did you integrate the signals?**

- Automatic
- Manual

17.1. If automatic, did you confirm the integration correctness visually? *

- a) Yes
- b) No

18. **Did you encounter any problems during the analysis?** *

- a) Yes
- b) No

18.1. If YES, what were the specific problems and to which samples do they apply? *

19. **Did you notice any unusual observations which, however, did not seem to have any effect on the resu** *

- a) Yes
- b) No

19.1. If YES, what were these observations and to which samples do they apply? *

20. **Did you find the instructions distributed for this PT adequate?** *

- a) Yes
- b) No

20.1. If NO, which parts do you think can improve? *

21. **What is your opinion about the registering / reporting format by this interface?** *

22. **Any other comments you wish to address?**

13.6. Experimental details

Table 14: Results and method performance characteristics (ochratoxin A solution)

Lab Code	Technique	Result [µg/mL]	Uncertainty value [µg/mL]	Coverage factor
AA871	Spectrophotometer	**13.806**		
AF590	Spectrophotometer	**13.5**		
AN410	Spectrophotometer	**13.58**		
AN745	Spectrophotometer	**13.48**		
BU793	Spectrophotometer	**14.25**	0.143	2
CI716	Spectrophotometer	**18.6312**		
CI863	Spectrophotometer	**12.248**	0.353	2
DN411	Spectrophotometer	**12.114**		
DP133	Spectrophotometer	**13.88**	0.62	2
ES408	Spectrophotometer	**13.955**	0.3	2
GC998	Spectrophotometer	**12.6**	0.57	2
GI812	Spectrophotometer	**14.137**		
GL869	Spectrophotometer	**12.73**		
GU330	Spectrophotometer	**13.138**	0.257	2
HN798	Spectrophotometer	**12.2**	0.59	2
HR099	Spectrophotometer	**13.732**		
JC489	Spectrophotometer	**13.58**	0.1	2
JK285	Spectrophotometer	**13.95**		
JN259	Spectrophotometer	**13.95**	0.2	2
JP176	Spectrophotometer	**14.103**	0.148	2
KF608	Spectrophotometer	**13.3**	0.3	2
KN355	Spectrophotometer	**12.92**	0.16	2
MA259	Spectrophotometer	**14.43**		
MC259	Spectrophotometer	**12.8**	0.5	2
MC798	Spectrophotometer	**13.6579**		
ML947	Spectrophotometer	**13.29**	0.66	2
MN644				
NS332	ELISA	**26.69**	5.1	2
OS720	Spectrophotometer	**17.71**	0.04	2
PC100	Spectrophotometer	**13.3**	1.5	2
PC105	Spectrophotometer	**13.313**	0.186	2
PG489	Spectrophotometer	**13.9**	0.3	4.303
SB871	Spectrophotometer	**14.133**	0.33	3
SS486	Spectrophotometer	**15.3652**		
ST117	Spectrophotometer	**13.51**	0.68	2
ST638	HPLC-FLD	**11.55**		
YM410	Spectrophotometer	**14.58**	0.37	2

Table 15: Results and method performance characteristics (cereal flour)

Lab Code	Technique	Result [µg/kg]	Uncertainty value [µg/kg]	Coverage factor	Recovery [%]	LOD [µg/kg]	LOQ [µg/kg]
AA871	HPLC-FLD	**135**	20.25	1	110	0.05	0.15
AF590	HPLC-FLD	**237**	71.61	2	75	0.1	0.2
AN410	HPLC-FLD	**198**	43.6	2	94	0.05	0.3
AN745	HPLC-FLD	**203.9**	32.6	2	94.9	0.03	0.1
BU793	LC-MS/MS	**166.5**	18.15	2	118.86	2.5	8.3
CI716	HPLC-FLD	**156**			94.5	0.05	0.1
CI863	HPLC-FLD	**163.22**	31.42	2	108	0.1	0.3
DN411	HPLC-FLD	**190.5**	16	2	98.3	0.1	0.3
DP133	HPLC-FLD	**138.7**	29.96	2	97.2	0.2	1
ES408	HPLC-FLD	**136.3**	14.2	2	83	0.035	0.115
GC998	HPLC-FLD	**233.42**	27.07	2	78.27	0.2	1
GI812	HPLC-FLD	**183**	22	2	96	1	3
GL869	HPLC-FLD	**162.1**	13.24	2	99.1	0.02	0.2
GU330	HPLC-FLD	**215**	51	2	87	10	30
HN798	HPLC-FLD	**130.4**	37.82	2	91.4	0.1	0.3
HR099	LC-MS/MS	**231.4**	37	2	102	1	2
JC489	HPLC-FLD	**202.7**	30.4	2	96.67	0.2	0.5
JK285	HPLC-FLD	**510**	50	2	40	0.5	2
JN259	HPLC-FLD	**258**	33	2	109.5	0.05	0.1
JP176	HPLC-FLD	**200.8**	12.05	2	88.26	0.3	1
KF608	HPLC-FLD	**179**	72	2	102	0.1	0.2
KN355	HPLC-FLD	**168**	50	2	76	0.05	0.1
MA259	HPLC-FLD	**222**	35.52	2	99.3	0.9	0.9
MC259	HPLC-FLD	**184.6**	10	2	90	0.1	0.3
MC798	HPLC-FLD	**193.6**	67.76	2	98	0.125	0.25
ML947	HPLC-FLD	**200.22**	60.07	2	99.6	0.2	0.6
MN644	ELISA	**5**			10	5	
NS332	ELISA	**20.78**	3.97	2	85	1	2
OS720	LC-MS/MS	**243**	28	2	97.5	0.6	2
PC100	HPLC-FLD	**195**	56	2	77	0.15	0.5
PC105	HPLC-FLD	**186**	13.45	2	99.4	0.004	0.15
PG489	HPLC-FLD	**270.3**	78.7	2	83.6	0.3	0.9
SB871	HPLC-FLD	**202.3**	24.5	3	84.3	0.2	1
SS486	HPLC-FLD	**141.12**	28.22	2	82.2	0.15	0.43
ST117	HPLC-FLD	**181.4**	57.1	2	88	0.1	0.2
ST638	HPLC-FLD	**139.94**	0.37	2	95	0.05	0.15
YM410	HPLC-FLD	**214.8**	20	2	98.7	0.5	0.5

Table 16: Results and method performance characteristics (green coffee)

Lab Code	Technique	Result [µg/kg]	Uncertainty value [µg/kg]	Coverage factor	Recovery [%]	LOD [µg/kg]	LOQ [µg/kg]
AA871	HPLC-FLD	**6.98**	1.05	1	105	0.05	0.15
AF590							
AN410	HPLC-FLD	**7.96**	1.75	2	90	0.05	0.3
AN745	HPLC-FLD	**8.7**	2.5	2	106.4	0.05	0.1
BU793	LC-MS/MS	**7.16**	0.41	2	96.87	0.2	0.7
CI716	HPLC-FLD	**13.7**			63	0.2	0.5
CI863	HPLC-FLD	**6.84**	3	2	95	0.1	0.4
DN411	HPLC-FLD	**7.7**	0.4	2	76.3	0.1	0.3
DP133	HPLC-FLD	**7**	1.51	2	97	0.2	1
ES408	HPLC-FLD	**7.85**	1.56	2	82.6	0.087	0.287
GC998	HPLC-FLD	**9.366**	1.08	2	78.317	0.2	1
GI812	HPLC-FLD	**10.5**	0.2	2	69	1	3
GL869	HPLC-FLD	**6.05**	1.76	2	91	0.02	0.2
GU330	HPLC-FLD	**12.7**	3	2	78	1	3
HN798	HPLC-FLD	**7.9**	2.29	2	90.5	0.1	0.3
HR099	LC-MS/MS	**7.6**	1.2	2	88	1	2
JC489	HPLC-FLD	**9.22**	1.38	2	89.4	0.2	0.5
JK285							
JN259	HPLC-FLD	**10.9**	1.8	2	80.7	0.7	1.4
JP176	HPLC-FLD	**6.5**	0.71	2	87.31	0.3	1
KF608	HPLC-FLD	**7.2**	2.9	2	92	0.25	0.5
KN355	HPLC-FLD	**9**	2.7	2	57	0.05	0.1
MA259	HPLC-FLD	**8.2**	2.79	2	105	1.5	1.5
MC259	HPLC-FLD	**9.8**	1	2	80	0.1	0.3
MC798	HPLC-FLD	**9.5**	3.33	2	84	0.125	0.25
ML947	HPLC-FLD	**7.82**	2.34	2	98.6	0.2	0.6
MN644	ELISA	**23.28**			6	5	
NS332	ELISA	**14.67**	2.8	2	85	0.25	0.36
OS720	LC-MS/MS	**14.2**	2.1	2	77	0.6	2
PC100	HPLC-FLD	**8.15**	3.8	2	61	0.3	1
PC105	HPLC-FLD	**7.84**	0.72	2	93.2	0.0014	0.23
PG489	HPLC-FLD	**8.4**	1.3	2	100	0.7	2.1
SB871	HPLC-FLD	**10.22**	3.32	3	91.6	0.5	1.5
SS486	HPLC-FLD	**7.23**	1.56	2	100	0.12	0.36
ST117	HPLC-FLD	**6.6**	2.8	2	76	0.06	0.2
ST638	HPLC-FLD	**4.48**	0.76	2	83	0.08	0.24
YM410	HPLC-FLD	**8.315**	0.787	2	96.7	1	1

Table 17: Results and method performance characteristics (paprika)

Lab Code	Technique	Result [µg/kg]	Uncertainty value [µg/kg]	Coverage factor	Recovery [%]	LOD [µg/kg]	LOQ [µg/kg]
AA871	HPLC-FLD	**12.89**	1.93	1	100	0.05	0.15
AF590							
AN410	HPLC-FLD	**12.8**	2.82	2	98	0.05	0.3
AN745	HPLC-FLD	**13.9**	2.2	2	91.3	0.03	0.1
BU793	LC-MS/MS	**21.39**	1.5	2	110.83	1.7	5.7
CI716							
CI863	HPLC-FLD	**11.15**	4.9	2	95	0.1	0.4
DN411	HPLC-FLD	**17.5**	3.6	2	109.2	0.1	0.3
DP133	HPLC-FLD	**9.26**	2	2	84	0.2	0.5
ES408	HPLC-FLD	**14.93**	3	2	72.2	0.087	0.287
GC998	HPLC-FLD	**17.317**	2	2	87.63	0.2	1
GI812	HPLC-FLD	**9.92**	1.59	2	99	1	3
GL869	HPLC-FLD	**11.47**	3.36	2	81.1	0.02	0.2
GU330	HPLC-FLD	**14.7**	3.4	2	76	1	3
HN798	HPLC-FLD	**14.25**	4.13	2	90.5	0.2	0.8
HR099	LC-MS/MS	**13.6**	2.2	2	85	1	2
JC489	HPLC-FLD	**15.7**	2.35	2	101.7	0.2	0.5
JK285	HPLC-FLD	**17.5**	4	2	84	0.5	2
JN259	HPLC-FLD	**13.4**	2.3	2	86.4	0.7	1.4
JP176	HPLC-FLD	**14.13**	0.88	2	78	0.3	1
KF608	HPLC-FLD	**13.8**	5.5	2	95	0.5	1
KN355	HPLC-FLD	**9.1**	2.7	2	74	0.05	0.1
MA259	HPLC-FLD	**13.63**	4.1	2	87.8	0.2	0.2
MC259	HPLC-FLD	**11.8**	1	2	74	0.1	0.3
MC798	HPLC-FLD	**20.1**	7.04	2	78	0.125	0.25
ML947	HPLC-FLD	**12.79**	3.84	2	88.8	0.5	1.5
MN644	ELISA	**40**			5	5	
NS332	ELISA	**3.62**	0.69	2	90	0.25	0.36
OS720	LC-MS/MS	**15.1**	2.2	2	83.5	0.6	2
PC100	HPLC-FLD	**13.2**	5.7	2	84	0.15	0.5
PC105	HPLC-FLD	**13.21**	1.55	2	90.6	0.002	0.16
PG489	HPLC-FLD	**14.4**	3.5	4.303	100	0.5	1.5
SB871	HPLC-FLD	**18.12**	0.95	3	76.5	0.2	1
SS486	HPLC-FLD	**11.73**	2.42	2	100	0.12	0.36
ST117	HPLC-FLD	**10.7**	1.9	2	94	0.075	0.2
ST638	HPLC-FLD	**10.77**	1.29	2	88	0.03	0.09
YM410	HPLC-FLD	**17.6**	1.67	2	101.1	1	1

Table 18: Number of samples analysed by laboratories per year for ochratoxin A

Lab Code	Number of samples per year		
	< 50	50 – 250	251 – 1000
AA871			X
AF590	X		
AN410		X	
AN745		X	
BU793	X		
CI716	X		
CI863		X	
DN411			X
DP133	X		
ES408		X	
GC998		X	
GI812		X	
GL869		X	
GU330		X	
HN798		X	
HR099			X
JC489		X	
JK285		X	
JN259	X		
JP176		X	
KF608			X
KN355	X		
MA259		X	
MC259		X	
MC798		X	
ML947			X
MN644		X	
NS332		X	
OS720	X		
PC100		X	
PC105		X	
PG489			X
SB871		X	
SS486		X	
ST117			X
ST638	X		
YM410			X

Table 19: Matrices analysed on routine basis, accreditation

Lab Code	Which of the following matrices does your laboratory analyse for ochratoxin A on a routine basis?			Please specify the other matrices your laboratory analyse for OTA	Are you accredited for the determination of ochratoxin A?	If YES, please specify the matrices
	Cereal flour	Green coffee	Paprika			
AA871	X	X	X	animal feed, spices, coffee, meat, dried fruits, baby food, beverages	Yes	animal feed, spices, coffee, meat, dried fruits, baby food, beverages
AF590	X				No	
AN410	X	X	X	feed materials; feed	Yes	vegetable food, feed and respective raw materials
AN745	X			dried fruit, raisins	Yes	coffee (instant and roasted), raisins, cereals, beans, lenses, nuts, paprika
BU793	X			at the moment the lab is in the phase of method development on the newly purchased instruments	No	
CI716	X			roasted coffee, wine and animal feed	No	
CI863	X			wine, ham, baby food	Yes	cereal and cereal products
DN411	X			feedstuff, dried fruit, roasted coffee, instant coffee, wine, juice, baby food, beer	Yes	cereals, feedstuff, dried fruit, roasted coffee, instant coffee, wine, juice, baby food, beer
DP133	X		X	dried fruit	No	
ES408	X	X		raisins, roasted coffee	Yes	cereals
GC998	X			wheat, rye, barley	No	
GI812	X	X			Yes	ISO 170025
GL869	X	X		coffee, beer, wine, dried fruits	Yes	coffee, beer, vine, dried fruits, cereals
GU330	X			animal kidneys	Yes	feed
HN798	X			kidney, liver, muscles	Yes	cereals, animal tissues
HR099	X			Feed and feed ingredients	Yes	feed and feed ingredients
JC489	X			feeds	Yes	feeds, foods
JK285	X		X	chilli, pepper, breakfast cereals, maize, beer	Yes	spices, cereals, maize
JN259	X		X	dried fruits, beer, wine, baby food,	Yes	cereals, spices, beer and wine, dried fruits, baby food
JP176	X	X	X	Dried Vine Fruit, Wine, Beer, Liquorice, Chilli Powder, Chocolate, Grape Juice, Baby food, Ground Coffee, Instant coffee, Tumeric, Pepper	Yes	Cereal Flour, Coffee, Paprika, Dried vine Fruit, Wine, Beer, Liquorice, Chilli, Chocolate, Baby food
KF608	X	X	X	dried fruit, cereal products, roasted coffee, instant coffee, beverages, baby food etc, all type of food of non-animal origin	Yes	cereals, cereal products, dried vine fruit, green coffee, roasted coffee, soluble coffee, spices, non-alcoholic beverages, cereal

Lab Code	Which of the following matrices does your laboratory analyse for ochratoxin A on a routine basis?			Please specify the other matrices your laboratory analyse for OTA	Are you accredited for the determination of ochratoxin A?	If YES, please specify the matrices
	Cereal flour	Green coffee	Paprika			
						based baby food
KN355	X				Yes	cereals
MA259	X			feedingstuffs, dried fruits, coffee(excluding green coffee), cereals	Yes	dried fruits, coffee, cereals, cereals products, feedingstuffs
MC259	X		X	cocoa powder, roasted coffee, baby food	Yes	cereal flour, paprika powder, cocoa powder, roasted coffee, baby food
MC798	X			beer, feed, cereals,	Yes	flour, beer
ML947	X		X	animal feed, dried fruits, spices, roasted coffee, breakfast cereals cereal products	No	
MN644	X		X		No	
NS332	X			dried fruit, cereals	Yes	grain, corn, coffee
OS720	X	X	X		No	
PC100	X		X	dried fruit, beer, wine, roasted coffee, soluble coffee, baby and infant food	Yes	cereal flour, paprika powder, dried fruit, beer, wine, roasted coffee, soluble coffee, baby and infant food
PC105	X		X	cereal based foodstuffs, wine, beer, cocoa products, cheese, roasted coffee, edible oils, etc.	Yes	foodstuffs
PG489	X	X		baby food, soluble coffee, raisins, wine and cereal products	Yes	coffee, cereals and raisins
SB871	X	X	X	cocoa, liquorice, wine, beer, oils, figs, raisins, nuts, baby food, bread....	Yes	cocoa, liquorice, wine, beer, oils, figs, raisins, nuts, baby food, bread....
SS486	X	X	X	liver, feed, dried grapes	Yes	liver, feed, dried grapes, cereal flour, coffee, paprika powder
ST117	X	X	X	dried fruit, infant food, nuts, cocoa, other spices, wine, beer	Yes	Cereals, fruit, coffee, spices, infant food, nuts, pulses, cocoa, offal & blood, wine, beer
ST638	X			coffee, corn flakes, dried fig, flour	Yes	dried fig
YM410	X			feed samples	Yes	feed -cereals

Table 20: Spectrophotometric analysis I.

Lab Code	What is the brand and model of your UV-spectrophotometer?	Is this instrument a single or a two channel photometer?	What type of light-source was used during the spectrophotometric measurement?	At which wavelength did you identify the maximum for ochratoxin A (nm)?
AA871	Shimadzu UV-1602	two channel	Deuterium	334 nm
AF590	Shimadzu UV 2401 PC	two channel	Deuterium	333 nm
AN410	Amersham Biosciences - Ultrospec 2100 pro	single channel	Xenon	333 nm
AN745	Unicam UV2-100	two channel	Deuterium	334 nm
BU793	Cintra 303, GBC, Australia	two channel	Deuterium	333 nm
CI716	Unicam uv/vis - UV4	two channel	Tungsten	333 nm
CI863	Perkin-Elmer	two channel	Deuterium	333 nm
DN411	UV-1601 SHIMADZU	two channel	Tungsten	333 nm
DP133	GBC UV/VIS 911A	single channel	Deuterium	335,2 nm
ES408	UV-1700 SHIMADZU	two channel	Deuterium	333,2 nm
GC998	Systronics, 5204	two channel	Hg lamp	333 nm
GI812	Lamda 35 Perkin Elmer	two channel	Deuterium	333 nm
GL869	perkin-Elmer lambda 400	two channel	Tungsten	333 nm
GU330	Beckman DU-62	single channel	Deuterium	333 nm
HN798	ThermoSpectronic Helios Epsilon 9423UUE1000E	single channel	Tungsten	333 nm
HR099	Varian Cary 300 Bio	two channel	Deuterium	333 nm
JC489	Thermo Electron Corp., Nicolet Evolution 300	two channel	Xenon	333 nm
JK285	VARIAN Cary 3	two channel	Deuterium	334 nm
JN259	Hitachi UV-VIS 1800	single channel	Hg lamp	333,9 nm
JP176	Jenway UV -Vis Spectrophotometer, Model No. 6105	single channel	Deuterium	333 nm
KF608	Varian Cary 1E (100)	two channel	Deuterium	333,1 nm
KN355	Perkin Elmer Lambda 10	single channel	Deuterium	333 nm
MA259	UV-VIS Spectrometer, Agilent 8453	single channel	Deuterium	333 nm
MC259	Beckman DU-65	single channel	Deuterium	333 nm
MC798	Thermo Scientific, Genesys 6	two channel	Xenon	333 nm
ML947	UV 1800 Shimadzu	two channel	Deuterium	333,3 nm
OS720	Nicolet Evolution 300 thermo electron cooperation	two channel	Tungsten	334 nm
PC100	Analytic Jena, Specord 210	single channel	Deuterium	333 nm
PC105	SHIMADZU UV-160	two channel	Deuterium	331,5 nm
PG489	Shimadzu UV-1601	two channel	Deuterium	334 nm
SB871	Shimadzu UV 1700	two channel	Tungsten	334 nm
SS486	SPEKOL Carlzeiss Jena	single channel	Deuterium	333 nm
ST117	Hitachi U2000	two channel	Deuterium	334,4 nm
YM410	Perkin Elmer Lambda 12	two channel	Hg lamp	333 nm

Table 21: Spectrophotometric analysis II.

Lab Code	Did you calibrate your UV-spectrophotometer?	If YES, what procedure in short did you use (e.g. $K_2Cr_2O_7$ solution or calibrated filter, give reference)	Optical path length of the cuvette (cm):	What was the absorbance reading you obtained with the spectrophotometer?	Do you normally check your calibrants by UV-spectrophotometry?
AA871	Yes	blank reference	1 cm	0.093 (abs of 1:1 dilution)	No
AF590	No		1 cm	0.182	Yes
AN410	Yes	GLP-procedure according to the producer's instruction	1 cm	0.183	Yes
AN745	No	When calibrated earlier: $K_2Cr_2O_7$ solution	1 cm	0.185	Yes
BU793	Yes	$K_2Cr_2O_7$ solution	1 cm	0.1921	Yes
CI716	No		1 cm	0.251	Yes
CI863	Yes	$K_2Cr_2O_7$ solution	1 cm	0.165	No
DN411	Yes	Quality assurance principles for analytical laboratories. F.M.Garfield. 1996	1 cm	0.1632	Yes
DP133	Yes	$K_2Cr_2O_7$	1 cm	0.187	Yes
ES408	Yes	Starna RM-N1N35N	1 cm	0.188	Yes
GC998	Yes	Calibrated filter	1 cm	0.17	Yes
GI812	No		1 cm	0.1945	No
GL869	Yes	calibrated filter (holmium)	1 cm	0.171	No
GU330	No		1 cm	0.177	No
HN798	Yes	$K_2Cr_2O_7$	1 cm	0.164	No
HR099	No		1 cm	0.201	No
JC489	No		1 cm	0.183	Yes
JK285	No		1 cm	0.188	Yes
JN259	No		1 cm	333.9	Yes
JP176	Yes	Calibrated versus water as per manufacturer's instructions	1 cm	0.19	No
KF608	No		1 cm	0.179	Yes
KN355	Yes	$K_2Cr_2O_7$	1 cm	0.174	Yes
MA259	Yes	$K_2Cr_2O_7$ solution	1 cm	0.196044	Yes
MC259	No		1 cm	0.173	Yes
MC798	No		1 cm	0.184	Yes
ML947	Yes	oxyde d'holmium 4% in HClO4 10% ref RM-HL n°11989 and aqueuses solutions Co Ni ref NIST SRM931g-LGC Promochem	1 cm	0.179	Yes
OS720	Yes	$K_2Cr_2O_7$	1 cm	0.2386	Yes
PC100	Yes	$K_2Cr_2O_7$, calibrated filter	1 cm	0.1791	Yes
PC105	Yes	$K_2Cr_2O_7$	1 cm	0.18	Yes
PG489	Yes	$K_2Cr_2O_7$	1 cm	0.187	No
SB871	Yes	calibrated filters	1 cm	0.1995	Yes
SS486	Yes	holmium filter	1 cm	0.207	No
ST117	Yes	$K_2Cr_2O_7$ solutions & Holmium & Didymium filters	1 cm	0.182	Yes
YM410	No		1 cm	0.197	Yes

Table 22: Recovery estimate, overnight stop

Lab Code	How did you perform the recovery estimate?	If other please specify	During the analysis did you need to include any over night stop?	If YES, please state for which samples and at what stage of the analysis.
AA871	Standard to Blank		No	
AF590	Standard to Blank		No	
AN410	Standard to Blank		No	
AN745	Standard to Blank		Yes	Extraction, clean-up one day, HPLC next day
BU793	Standard to Blank		No	
CI716	Standard to Blank		Yes	All Samples
CI863	other	Spiking a blank sample with a known amount of standards solution	No	
DN411	other	according Certified Reference Materials in appropriate matrix	Yes	For all samples one day extraction and the second day dilution and passing through the column
DP133	Standard to Blank		No	
ES408	Standard to Blank		No	
GC998	Standard to Blank		No	
GI812	Standard to Blank		Yes	After evaporation of all samples.
GL869	Standard to Blank		Yes	after elution of IA columns
GU330	Standard to Blank		No	
HN798	Standard to Blank		No	
HR099	Standard to Blank		No	
JC489	Standard to Blank		No	
JK285	Standard to Blank		No	
JN259	Standard to Blank		No	
JP176	Standard to Blank		No	
KF608	Standard to Blank		No	
KN355	Standard to Blank		Yes	after eluting OTA from IAC, all samples, next day start with evaporation of eluting solvent
MA259	Standard to Blank		No	
MC259	Standard to Blank		No	
MC798	other	std spiked blank sample	No	
ML947	Standard to Blank		No	
MN644	Internal Standard to Extract		No	
NS332	Standard to Blank		No	
OS720	Standard to Blank		No	
PC100	Standard to Blank		Yes	green coffee - over night filtration
PC105	Standard to Blank		No	
PG489	Standard to Blank		No	
SB871	Standard to Blank		No	
SS486	Standard to Blank		No	
ST117	Standard to Blank	Also standard to extract before cleanup for wheat (to check high level recovery)	No	
ST638	other	test materials	No	
YM410	Standard to Blank		No	

Table 23: Extraction mode

Lab Code	What was the extraction solvent used?	What was the extraction time?	What was the extraction mode (e.g. blending or shaking)?	What was the extraction solvent to sample ratio used during extraction (in mL/g)?
AA871	cereals (acetonitrile/water; 60/40); green coffee, paprika (methanol/3%sodiumhydrogencarbonate; 50/50)	1 hour	shaking	6,67 mL/g
AF590	acetonitrile/ water 60:40	1 h	shaking	100 mL/20 g
AN410	coffee: 1% $NaHCO_3$; others: acetonitrile-water (4:1)	60 min	shaking	coffee: 20 mL/g; others: 5 mL/g
AN745	Cereal, paprika: acetonitrile:water 84:16, Green coffee: methanol: $NaHCO_3$ 3% 50:50	Cereal, paprika: 30 min, Green coffee 3 min	Cereal, paprika: shaking Green coffee: blending	Cereal, paprika: 100mL/25 g Green coffee: 250mL/12.5 g
BU793	acetonitrile/water 80:20	1 hour	shaking	4 mL/g
CI716	For coffee MeOH/$NaHCO_3$ 3%(50/50) and for cereals PBS/MeOH (50/50) (V/V)	30 min	Orbital shaking	for coffee 150mL/15g , for cereal 40mL/10g
CI863	Cereals (AcCN:H_2O 6:4); Paprika and Coffee (MeOH:$NaHCO_3$,3% 1:1)	Cereals (3 minutes); Paprika and Coffee (40 minutes)	Cereals (blending); Paprika and Coffee (shaking)	Cereals (100mL/25g); Paprika and Coffee (100ml/10g)
DN411	For paprika and cereals 60% acetonitrile/water; for coffee - 3% sodium bicarbonate in methanol/water	2 hours	shaking	Paprika, cereal:4 mL/g Coffee: 20 mL/g
DP133	Acetonitrile / water for cereals, methanol / water for spices and sodium hydrogen carbonate for green coffee	Cereals 3 min; Paprika 35 min; Green coffee 3 min	Cereals blending; Paprika shaking; Green coffee blending	Cereals 4 mL/g; Paprika 6 mL/g; Green coffee 5 mL/g
ES408	MeOH:$NaHCO_3$ 70:30	3 min	blending	5 mL/g
GC998	Methanol-3% aq. Sod. Carbonate (1:1) for Paprika, Green Coffee and AcN-H_2O (84:16) for Cereal	30 min	Shaking	1:8 (Paprika, Green Coffee), 1:4 (Cereal)
GI812	MeOH/Water	2 hours	shaking	coffee 15g/100mL; paprika and cereals 25g/100mL
GL869	ACN/water 60/40 for cereals; $NaHCO_3$ 1% for coffee and paprika	30 min	ultra-turrax + shaking	Cereals: 5 mL/g coffee, paprika: 20 mL/g
GU330	Acetonitril-water for cereal flour, sodium bicarbonate/water for coffee, methanol/sodium bicarbonate for paprika	30 min	shaking	20/5 with cereal flour, 100/5 with coffee and paprika
HN798	cereals: methanol+water (80+20, v/v); paprika: methanol+1%$NaHCO_3$ (50+50, v/v); coffee: methanol+3% $NaHCO_3$ (50+50,v/v)	45 min	shaking	cereals: 1/3; paprika: 1/20; coffee: 1/8
HR099	Cereal, Paprika: 60% acetonitrile-water; Coffee: 1% sodiumbicarbonate in water	Cereal, Paprika, Coffee: 2 h	shaking	Cereal: 4, Paprika, Coffee: 20
JC489	3% bicarbonate/methanol =50/50	1 hour	shaking	150/15 mL/g
JK285	Methanol/ Water = 80/20 volume parts	3 minutes	blending	4mL/g
JN259	AcN/water - cereals; $CHCl_3$ - paprika, coffee	3 min - cereals. 30 min - paprika, coffee	cereals: blending paprika, coffee: shaking	100 mL - cereals; 12,5 mL - paprika, coffee
JP176	1% sodium Bicarbonate	2 minutes	blending	20mL/g
KF608	acetonitrile-water (cereal), 2% sodiumbicarbonate in water (green coffee), methanol-3%-sodiumbicarbonate (paprika)	3 min	blending	20 mL/g (cereal), 10 mL/g (green coffee), 20 ml/g (paprika)
KN355	cereals: 60%ACN:40%H_2O; paprika: 50%MeOH:50%$NaHCO_3$ (1%); green coffee: 50%MeOH:50%$NaHCO_3$ (3%)	2 min	blending	cereals: 4 mL/g; paprika: 20 mL/g; green coffee: 8 mL/g
MA259	cereal flour- acetonitril:water; paprika-methanol+1%$NaHCO_3$; green coffee-1%$NaHCO_3$	3 min	blending	cereal flour-100/25; paprika-100/25; green coffee-100/5
MC259	sodium bicarbonate/deionised water, acetonitrile/water	30 min	shaking	20/5 mL/g

Lab Code	What was the extraction solvent used?	What was the extraction time?	What was the extraction mode (e.g. blending or shaking)?	What was the extraction solvent to sample ratio used during extraction (in mL/g)?
MC798	methanol (coffee and paprika) and acetonitrile(flour)	3 min	ultra-turrax	5 mL/g
ML947	CH_3CN 60% for cereal, green coffee and paprika	2 min	blending	cereal: 10 mL/g green coffee: 4 mL/g paprika: 10 mL/g
MN644	Methanol	approx. 3 min	shaking	12.5 mL/5g
NS332	Methanol:water	20 min cereals, 10 min green coffee, paprika	shaking	50 mL H_2O/5g;200mL $NaHCO_3$1%/10g;20mL ACN:H_2O/10g
OS720	paprika and green coffee in $NaHCO_3$ 1% , cereal in acetonitrile:water (60:40)	30 min	shaking	100/5 green coffee, 200/50 cereal 200/10 paprika
PC100	paprika, cereal flour - 60% acetonitrile, green coffee - 1 % $NaHCO_3$	2 min	blending (ultra-turrax)	cereal flour: 5 mL/g paprika: 10 mL/g green coffee: 20 mL/g
PC105	tert-butyl-methyl ether (after acidification of samples with phosphoric acid)	cereal : 2 x 3 minutes. Green coffee and paprika : 1 x 3 minutes	blending	5 mL/g
PG489	Acetonitrile-Water for cereals, 1% $NaHCO_3$ for coffee, Methanol-3% $NaHCO_3$ for paprika	3 min	blending	Cereals: 4 mL/g Coffee: 20 mL/g Paprika: 8 mL/g
SB871	coffee/paprika: 3%$NaHCO_3$/Methanol (1:1); cereals water/methanol (20:80 V/V)	40 min	shaking (turbular)	5 g sample per 50 mL = 10 mL extraction solvent per g
SS486	1% $NaHCO_3$	30 min	shaking	200mL extraction solution for 10 g of sample
ST117	Wheat acetonitrile/water, coffee sodium bicarbonate, paprika methanol/water	Coffee & wheat approx 3-5mins, spices approx 40 mins	Coffee & wheat blending, paprika shaking	paprika 150mL/25g, wheat 100mL/25g, coffee 100mL/20g
ST638	paprika: methanol:water , cereals: acetonitrile:water , green coffee:methanol:Na-bicarbonate	3 min	green coffee and cereal: blending, paprika: shaking	paprika - 1:4 cereal - 1:5 green coffee - 1:3
YM410	acetonitril/water 60/40 v/v	30 min	shaking	40mL/5 g

Table 24: Immunoaffinity column

Lab Code	What type of clean up methodology was used (e.g. immunoaffinity column)?	please specify the manufacturer of the immunoaffinity columns you used during the analysis	did you follow the instructions by the manufacturer ?	If NO, please give some brief details on the modification	please specify the capacity of the immunoaffinity column (ng)
AA871	immunoaffinity column	R-Biopharm	Yes		
AF590	immunoaffinity column	R-Biopharm	Yes		not specified
AN410	immunoaffinity column	R-Biopharm	No	slightly different extraction solvent to sample ratios used during extraction	
AN745	Filtration, immunoaffinity column	R-Biopharm	No	No back flashing of the column. Elution with 3-4 ml methanol/acetic acid, no water.	
BU793	No clean up				
CI716	For coffee: Sepack C18 and immunoaffinity column. For cereal: immunoaffinity column	Vicam	Yes		0.25-300 ppb
CI863	immunoaffinity column	R-Biopharm	No	Elution solvent and ml and kind of eluent	300 ng
DN411	immunoaffinity column	R-Biopharm	Yes		
DP133	immunoaffinity column	R-Biopharm	Yes		
ES408	immunoaffinity column	Vicam	Yes		
GC998	Immunoaffinity Column (Paprika, Green Coffee), Mycosep 229 Ochra Push-through Clean up column (Cereal)	Romer Labs	Yes		
GI812	immunoaffinity column	Romer Labs	Yes		4 ng
GL869	immunoaffinity column	R-Biopharm	Yes		>150 ng
GU330	immunoaffinity column	R-Biopharm	Yes		
HN798	immunoaffinity column	Vicam	Yes		100 ng
HR099	No clean up				
JC489	immunoaffinity column	Romer Labs	Yes		
JK285	immunoaffinity column	Vicam	Yes		1000 ng
JN259	immunoaffinity column	Vicam	Yes		100 ng
JP176	immunoaffinity column	R-Biopharm	Yes		1000 ng
KF608	immunoaffinity column	R-Biopharm	No	no water elution after elution with desorption	not known exactly, tested 30 ng

Lab Code	What type of clean up methodology was used (e.g. immunoaffinity column)?	please specify the manufacturer of the immunoaffinity columns you used during the analysis	did you follow the instructions by the manufacturer ?	If NO, please give some brief details on the modification	please specify the capacity of the immunoaffinity column (ng)
				solution	
KN355	immunoaffinity column	R-Biopharm	Yes		
MA259	immunoaffinity column	R-Biopharm	Yes		5 ng
MC259	immunoaffinity column	R-Biopharm	Yes		
MC798	immunoaffinity column	Vicam	Yes		
ML947	immunoaffinity column	Neogen	No	elution with 2mL MeOH evaporation and dissolution in 1 mL mobile phase	950 ng
MN644	No clean up				
NS332	immunoaffinity column	Romer Labs	Yes	-	2000 ng
OS720	immunoaffinity column	R-Biopharm	No	volumne final de extracción	
PC100	immunoaffinity column	R-Biopharm	Yes		2000 ng
PC105	immunoaffinity column	R-Biopharm	No	cereal : The extract is in methanol / PBS 15 %; washing with water, elution with methanol. The extracts of green coffee and paprika are in 1% sodium bicarbonate; washing with Tween 1 % in water, elution with Methanol	2000 ng
PG489	immunoaffinity column	R-Biopharm	Yes		100 ng
SB871	immunoaffinity column	LCTech	No	inhouse optimized method	200 ng
SS486	immunoaffinity column	Neogen	No	extraction solution (originally 60% methanol, amount of sample from 25 to 10)	
ST117	immunoaffinity column	R-Biopharm	No	Extraction & dilution slightly different from recommended	
ST638	immunoaffinity column	Vicam	Yes		
YM410	immunoaffinity column	R-Biopharm	Yes		

Table 25: Detection techniques, specifying the methods

Lab Code	Detection techniques	Please specify your method (type of column, injection volume, mobile phase etc.)!
AA871	HPLC-FLD	Zorbax Eclipse XDB-C18, 5 µ, 250*4.6 mm; 100 µL; acetonitril/2% aceticacid (45/55)
AF590	HPLC-FLD	Waters Symmetry C 18, 3,9 mmx150 mmx 5µm, 40 µL, acetonitrile/water/acetic acid 99:99:2 ex 333 nm. em 450 nm
AN410	HPLC-FLD	C18 column; inj vol 100-250 µL; mobile phase acetonitrile-water-acetic acid (99:99:2)
AN745	HPLC-FLD	Column: ACE3 C18 3 µm 50x4.6 mm, Injection volume: 20 µl, Mobil phase: water:acetonitrile:acetic acid (500:500:10)
BU793	LC-MS/MS	C18 50x2,1mmx1.9 µm, 10 µL inj vol, MPh: (A)Water+1% HAc + 5 mM NH_4Ac (B)Methanol +1% HAc + 5 mM NH_4Ac
CI716	HPLC-FLD	Vol inj: 200 µL; column Whaters Spherisorb 5µm 250x4.6 mm; Mobile phase Acetonitrile/Water/Acetic acid(99/99/2) Wavelength: Ex=333 nm, Em = 477 nm
CI863	HPLC-FLD	C18, simmetry column, 150*4.6 mm, 5micron; 150 µL injection; Mobil phase MeOH:AcCN:H_2O(2%ac) 25:35:40
DN411	HPLC-FLD	LiChroCART 250-4 LiChrospher 100 RP-18e (5 µm), 100 µL, acetonotrile:water:acetic acid (48:51:1)
DP133	HPLC-FLD	Column C18 25cm, 4.6mm i.d. ; Injection Volume 50µL ; Mobile Phase Acetonitrile / Water / Acetic Acid (99:99:2)
ES408	HPLC-FLD	SphereClone 5 µ ODS(2) 150X4.60 mm, 100µL , H_20:CH_3CN:CH_3COOH 113:85:2
GC998	HPLC-FLD	Reversed-phase C18 (Waters) 250x4.6 mm with 5 µm particle, 20 µL injection volume, Acetonitrile, Methanol, Water-Glacial Acetic Acid (35+35+29+1)
GI812	HPLC-FLD	Zorbax SB-C18; 10mkl;Water:ACN:CH_3COOH 60:40:1
GL869	HPLC-FLD	C18 250 mm 5µm mob. phase: ACN/water/acetic acid (51/47/2 for cereals; 45/53/2 for coffee and paprika)
GU330	HPLC-FLD	Hypersil ODS 5 µm, 250x4.6 mm (Agilent), inj vol. 20 µL, mob. phase water-acetonitrile-acetic acid (470+510+20), λex = 333 nm, λem = 443 nm.
HN798	HPLC-FLD	Inertsil ODS-3 100A, 150 x 4.6mm, 5 µm; 50 µL, acetonitril+water+ glacial acide acid (49.5+49.5+1, v/v/v); flow: 1.4 mL/min
HR099	LC-MS/MS	Restek, Ultra aqueous C18 3µm 100x2,1mm; LC-MS/MS system, consist of Shimadzu HPLC-system (degasser, gradient pump, autosampler en column oven) & Applied Biosystems QTRAP 5500 MS/MS
JC489	HPLC-FLD	RESTEK PINNACLE PAH 5µ 250 x 4.6 mm 100 µL 2% AcOH/AcN = 47/53
JK285	HPLC-FLD	Sperisorb 80-5 ODS2; 5µm, 250mm from MAchery and Nagel, injection volume 60µL; mobile phase: water acetate buffer and methanol
JN259	HPLC-FLD	C18, 100 µL, AcN/H_2O/CH_3COOH
JP176	HPLC-FLD	Column: Waters Spherisorb S5 ODS2 250x4.6 mm, 5 µm, Injection Volume: 100 µL, Mobile Phase: Acetonitrile:Water:Acetic Acid (51:47:2), Wavelength Excitation 333nm Emission 443nm, Flow 1mL/min, Column Temperature 25°C, Run Time 15 minutes
KF608	HPLC-FLD	acetonitrile+water+acetic acid (99+99+2)
KN355	HPLC-FLD	LiChrosorb RP-18, 5µm, 125x4 mm, 50 µl, MF: 50% ACN: 49 % H_2O, 1% CH_3COOH
MA259	HPLC-FLD	Lichrosorb RP-18 5µm, 250 mm x 4.6m; injection volume 50 µL, mobile phase - acetonitril:water: acetic acid(99:99:2,v/v/v);
MC259	HPLC-FLD	Thermo Column ODS Hypersil 250×4.6 mm, 5 µm; 20 µL; Acetoitrile/Water/Acetic acid
MC798	HPLC-FLD	Alltima C18, 3µm, 100x4.6mmID, flow 1 mL/min, acetonitrile/water/acetic acid = 45/55/1
ML947	HPLC-FLD	100 µL injected mobile phase : MeOH-CH_3CN-CH_3COOH 3% (35-35-30)
MN644	ELISA	Commercial kits from R-Biopharm
NS332	ELISA	EuroProxima
OS720	LC-MS/MS	Application Note-paprika- Ochratoxin A, Extraction method, Ref No. A-3-P14.V.4 May 2009, R. Biopharm Rhône Ltd.
PC100	HPLC-FLD	C18 column, 100 µL injection volume, mobile phase: 49% acetonitrile+49% H_2O+2% acetic acid
PC105	HPLC-FLD	TRACER Extrasil 5 µm, 250 x 4.6 mm; injection volume : 25 µL; mobile phase : Methanol / 9% Acetic acid (40:60)
PG489	HPLC-FLD	C18-RP Zorbax Eclipse, 100µl, Acetonitrile, Water, Acetic Acid
SB871	HPLC-FLD	Inertsil ODS 250 x 4.6 5µ column; 25 µL Injection volume; eluent acetonitrile:water:glacial acid (48:51:10V/V); flow 1 mL/min; column temp. 30°C
SS486	HPLC-FLD	column: LiChrospher RP-18e, 125x4mm, Merck,25 µL, MP:acetonitrile/water/acetic acid glacial 40/60/2
ST117	HPLC-FLD	Spherisorb ODS2 Excel (25cmx4.6mm id), 400 µL, mobile phase acetonitrile:water:acetic acid 99:99:2, 1mL/min
ST638	HPLC-FLD	C18 Column, 100 µL, acetonitrile:water:acetic acid=47:51:2
YM410	HPLC-FLD	Waters Nova-Pak C18 µm 3.6x150 mm, 100 µL injection volume, mobile phase Acetonitril/water/acetic acid 49/49/2

Table 26: Integration mode, problems during the analysis

Lab Code	How did you integrate the signals?	If automatic, did you confirm the integration correctness visually?	Did you encounter any problems during the analysis?	If YES, what were the specific problems and to which samples do they apply?	Did you notice any unusual observations which, however, did not seem to have any effect on the results?	If YES, what were these observations and to which samples do they apply?
AA871	Manual		No		No	
AF590	Manual		No		No	
AN410	Automatic	Yes	No		No	
AN745	Automatic	Yes	No		Yes	Very high concentration of OTA in the cereal sample, dilution needed
BU793	Automatic	Yes	No		No	
CI716	Automatic	Yes	No		Yes	The step of filtration of green coffee spent many hours(o.n.)
CI863	Automatic	Yes	No		No	
DN411	Automatic	Yes	No		No	
DP133	Manual		No		No	
ES408	Automatic	Yes	No		No	
GC998	Automatic	Yes	No		No	
GI812	Automatic	Yes	No		No	
GL869	Automatic	Yes	No		Yes	orange colour of IA eluate
GU330	Automatic	Yes	No		No	
HN798	Automatic	No	No		No	
HR099	Manual		No		No	
JC489	Automatic	Yes	No		No	
JK285	Automatic	Yes	Yes	cereals high content and low recovery	Yes	low recovery by spiking cereals flour
JN259	Automatic	Yes	No		No	
JP176	Automatic	Yes	No		No	
KF608	Manual		Yes	Cereal sample, contaminated, absorbed a lot of extraction solvent	No	
KN355	Automatic	Yes	No		No	
MA259	Manual		No		No	
MC259	Automatic	Yes	No		No	
MC798	Automatic	Yes	Yes	oily extracts for the paprika, needing addition of celite in top of the Immuno column	No	
ML947	Automatic	Yes	No		No	
OS720	Automatic	Yes	No		Yes	cereal sample was out of calibration curve, but I did need dilute the sample
PC100	Manual		Yes	high level of OTA in cereal flour therefore repeated clean-up with lower amount of sample and	No	

Lab Code	How did you integrate the signals?	If automatic, did you confirm the integration correctness visually?	Did you encounter any problems during the analysis?	If YES, what were the specific problems and to which samples do they apply?	Did you notice any unusual observations which, however, did not seem to have any effect on the results?	If YES, what were these observations and to which samples do they apply?
				different calibration curve		
PC105	Automatic	Yes	No		No	
PG489	Automatic	Yes	No		No	
SB871	Automatic	Yes	No		No	
SS486	Automatic	Yes	Yes	the concentration of Your standard in ampoule measured by spectrophotometer does not correspond to that, we usually use from manufacturer	No	
ST117	Automatic	Yes	Yes	Wheat sample much higher than calibration range. It was diluted, cleaned up and reanalysed with extract spiked @200ug/kg - also diluted before cleanup & analysis	No	
ST638	Manual		Yes	standard solvent didn't mix with my dilution solvent	No	
YM410	Automatic	Yes	No		No	

Table 27: Instructions for the proficiency test

Lab Code	Did you find the instructions distributed for this PT adequate?	If NO, which parts do you think can improve?
AA871	Yes	
AF590	Yes	
AN410	Yes	
AN745	Yes	
BU793	No	"it would be helpful if the range of expected concentration was indicated"
CI716	No	"I would like to know the work range for the recovery experiment"
CI863	Yes	
DN411	Yes	
DP133	Yes	
ES408	Yes	
GC998	Yes	
GI812	Yes	
GL869	Yes	
GU330	Yes	
HN798	Yes	
HR099	Yes	
JC489	Yes	
JK285	Yes	
JN259	Yes	
JP176	Yes	
KF608	Yes	
KN355	Yes	
MA259	Yes	
MC259	Yes	
MC798	Yes	
ML947	Yes	
MN644	Yes	
NS332	Yes	
OS720	No	"to include the range of concentration of the samples"
PC100	Yes	
PC105	Yes	
PG489	Yes	
SB871	Yes	
SS486	Yes	
ST117	Yes	
ST638	No	"result measurements can be online"
YM410	Yes	

Table 28: Opinions about the registering/reporting interface

Lab Code	What is your opinion about the registering / reporting format by this interface?
AA871	ok
AF590	easy and quick
AN410	Unreasonably time consuming. It'd be better to apply the FAPAS-style, ie menus with pre-chosen options, especially for chemical names etc.
AN745	OK, but different methods for the different matrices made the filling-in complicated.
BU793	it is practical but it is more convenient to be able to see the questionnaire before submitting the results
CI716	Good
CI863	Any problem for the result submission, a little bit long the filling of the questionnaire
DN411	It would be good to know the questions of the Questionnaire in advance
DP133	Very user-friendly
ES408	User friendly.
GC998	May be better to have different formats for each category of sample
GI812	Why so much questions are needed?
GL869	almost ok
GU330	Appropriate.
HN798	ok
HR099	-
JC489	Usable
JK285	The time from the announcement to the pt was really short
JN259	ok
JP176	Both rapid and convenient. I would prefer to report the % recovery on the same page as the result and uncertainty values
KF608	Easy to use, nice to have possibility to save intermediate results
KN355	Fine
MA259	ok
MC259	user friendly
MC798	Reporting format not adequate : too large; better if the four results are on the same page
ML947	ok
MN644	good
NS332	ok
OS720	I think it is excellent, because fast and easy communication
PC100	It would be very useful to have opportunity to see all reported results from the lab together in one web page
PC105	ok
PG489	It was satisfactory
SB871	best way
SS486	a lot of buttons to be pressed to confirm results
ST117	OK, but maybe could provide separate spaces for different method information for different matrices
ST638	good
YM410	good

Table 29: Other comments

Lab Code	Any other comments you wish to address?
AF590	Our expanded uncertainty is +/- 31 % for Ochratoxin A
AN745	Results of the samples were corrected with recoveries from control charts and not with the recoveries received in this study.
HR099	There is no specific room available for variations in the procedure (e.g. the extraction procedure).
JK285	Our lab is not a NRL.
PC105	Strictly speaking, we do not perform routine analysis for OTA, but survey studies on specific foodstuffs
SS486	Because of differences between the concentration of Your standard and ours, all measurements were calculated according our calibration standard, only the concentration of OTA solution is stated, like it was measured on spectrophotometer, not like HPLC response. If I calculated amount of OTA according Your standard the amount of OTA change to 207 ppb in cereals, 10.6 ppb in coffee, 17,2 in paprika.

European Commission

EUR 24621 EN – Joint Research Centre – Institute for Reference Materials and Measurements
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Abstract
The Institute for Reference Materials and Measurements (IRMM) of the Joint Research Centre (JRC), a Directorate-General of the European Commission, operates the European Union Reference Laboratory (EU-RL) for Mycotoxins. One of its core tasks is to organise interlaboratory comparisons (ILCs) among appointed National Reference Laboratories (NRLs). This report presents the results of a ILC of the EU-RL for Mycotoxins which focused on the determination of ochratoxin A in food and feed samples.

The test materials were naturally contaminated cereals, green coffee and paprika samples and an ampouled ochratoxin A solution. The materials were labelled at IRMM and dispatched to the participants in May 2010. Each participant received two ampoules of solution and seven sachets containing approximately 30 g of test material each. Thirty-seven participants from 32 countries registered for the exercise. Thirty-six sets of results were reported for the solution, 37 for the cereals, 35 for the green coffee and 35 for the paprika.

The assigned values were 13.2 µg/mL for the test solution, 191 µg/kg for the cereals, 8.0 µg/kg for the green coffee and 13.0 µg/kg for the paprika. The uncertainties of the respective assigned values were 0.9 µg/mL, 9 µg/kg, 0.6 µg/kg and 0.9 µg/kg.

Participants were invited to report the uncertainty of their measurements. This was done by the majority of laboratories.

Laboratory results were rated with z-scores and zeta-scores in accordance with ISO 13528 and of the International Harmonized Protocol for the Proficiency Testing of Analytical Chemistry Laboratories.

In total about 90% of the attributed z-scores were below an absolute value of two, which indicated that most of the participants performed satisfactory or better than minimal performance criteria required.

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