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Report on the 2007 Proficiency Test for the Determination of Ochratoxin A in *Capsicum ssp* (Paprika Powder).

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Summary

A proficiency test was conducted with 68 laboratories from 17 EU Member States and four Third Countries. Test materials were a naturally contaminated "Ochratoxin A positive" and a "Ochratoxin A blank" capsicum material. The majority of laboratories chose to determine the ochratoxin A content by reverse-phase high-performance liquid-chromatography (RP-HPLC) with fluorescence detection against their own standard solutions as reference.

Applying the modified Horwitz equation according to Thompson[1] as a basis for the target standard deviation (22% in the case of this proficiency test), 79% of the laboratories achieved z-scores of less than $|2|$. The results were evaluated further on the basis of the returned questionnaire that each participant received. The questions asked were designed having in mind that future method development, if necessary, could profit from a comparison of the methodologies and method procedures applied by a comparatively large number of participating laboratories.

[1] M. Thompson (2000) *Analyst*, **125**, 385-386

Introduction

Ochratoxin A (OTA) is found in a variety of food products ranging from barley, to coffee, wine grapes and spices. At the moment several standards for the determination of OTA are available or under discussion at the European Standardization Committee (CEN), however none of these OTA related standards is developed for the analysis of spices. During discussions in international fora on future legislative limits for OTA in spices (in particular in paprika) the concern was expressed that the validity of currently available data on OTA in paprika strongly depends on the capability of laboratories to perform accurate OTA determinations and it appears uncertain whether this might be the case. As a result the European Commission's Directorate-General for Health and Consumer Protection (DG SANCO) asked the JRC to conduct a proficiency test (PT) on that matter to benchmark the OTA measurement capabilities in the Member and invited participation of laboratories in the Member States.

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 immunosorbent assays (ELISA). The most common principle in EU Member States is however HPLC-FLD, which is the basis for all CEN standards. All methodologies, irrespective of their detection principle, depend on the extraction of OTA from the matrix with a solvent. All invited laboratories were free to use their method of choice, but upon request a method that has been previously validated by the JRC was supplied.

Methodology

Each laboratory was supplied with one naturally contaminated "Ochratoxin A positive" and one "Ochratoxin A blank" capsicum material and a questionnaire that was used to evaluate the results. Laboratories were asked to report results within four weeks after dispatch and deadline extensions were granted upon requests.

For the evaluation of the results[2] we refer to the IUPAC Harmonised Protocol for the Proficiency Testing of Analytical Chemistry Laboratories[3] and used the frequently used ranking plot (laboratory number vs. reported results) to visualize z-scores and the location of each laboratory in the overall population. In addition, the results from the evaluation of the questionnaire were plotted. This was done by the use of box-and-whisker plots. In these plots the rectangular part of the plot extends from the lower quartile to the upper quartile, covering the centre half of reported results. The centre lines within each box show the location of the medians of all results within the plot. The whiskers extend from the box to the minimum and maximum values in each plot population, except for outliers. Outliers are points which lie more than 1.5 times the interquartile range above or below the box and are shown as small squares. Far outside points are points which lie more than 3.0 times the interquartile range above or below the box and are shown as small squares with plus signs through them. The presence of far outside points may indicate outliers or a highly skewed distribution.

[2] Individual results (as reported) are listed in the Tables in the Annex.

[3] M. Thompson, S. L. R. Ellison and R. Wood (2006) *Pure Appl. Chem.* **78**, 145–196

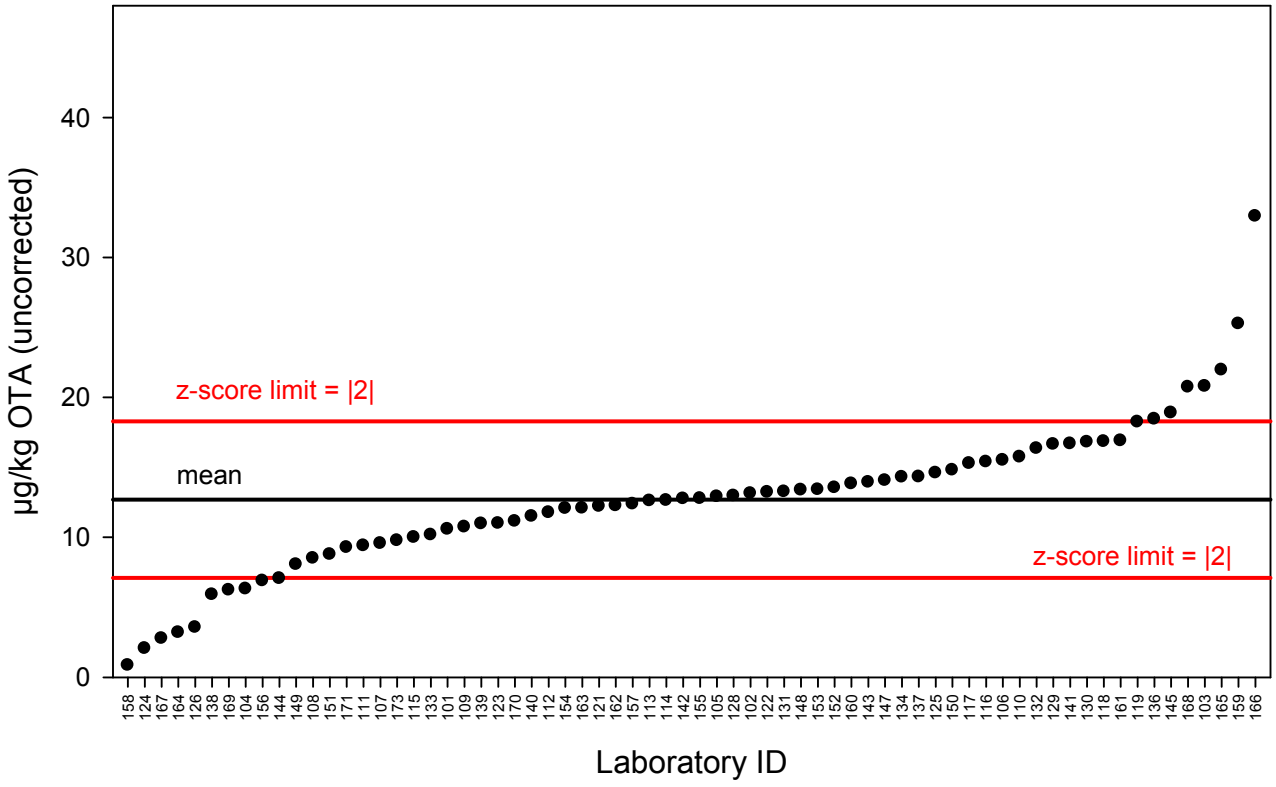
Results and Discussion:

Current EU legislation on mycotoxins in food[4] requires the reporting of analytical results in combination with recovery values, and decisions on rejection/acceptance of goods must take into account recovery information. Therefore both the uncorrected values and the recovery corrected values are plotted in Figures 1 and 2. The arithmetic mean did not differ significantly from various robust estimates of location as shown in Table 1. Therefore it was considered to use the arithmetic mean as the consensus value.

Table 1: Comparison of mean and median values.

	Uncorrected value [µg/kg]	Recovery [%]	Corrected value [µg/kg]
(arithmetic) mean	12.7	84.9	15.3
median	12.8	86.3	14.4
A15 mean	12.6	85.7	14.6
H15 mean	12.6	85.6	14.7

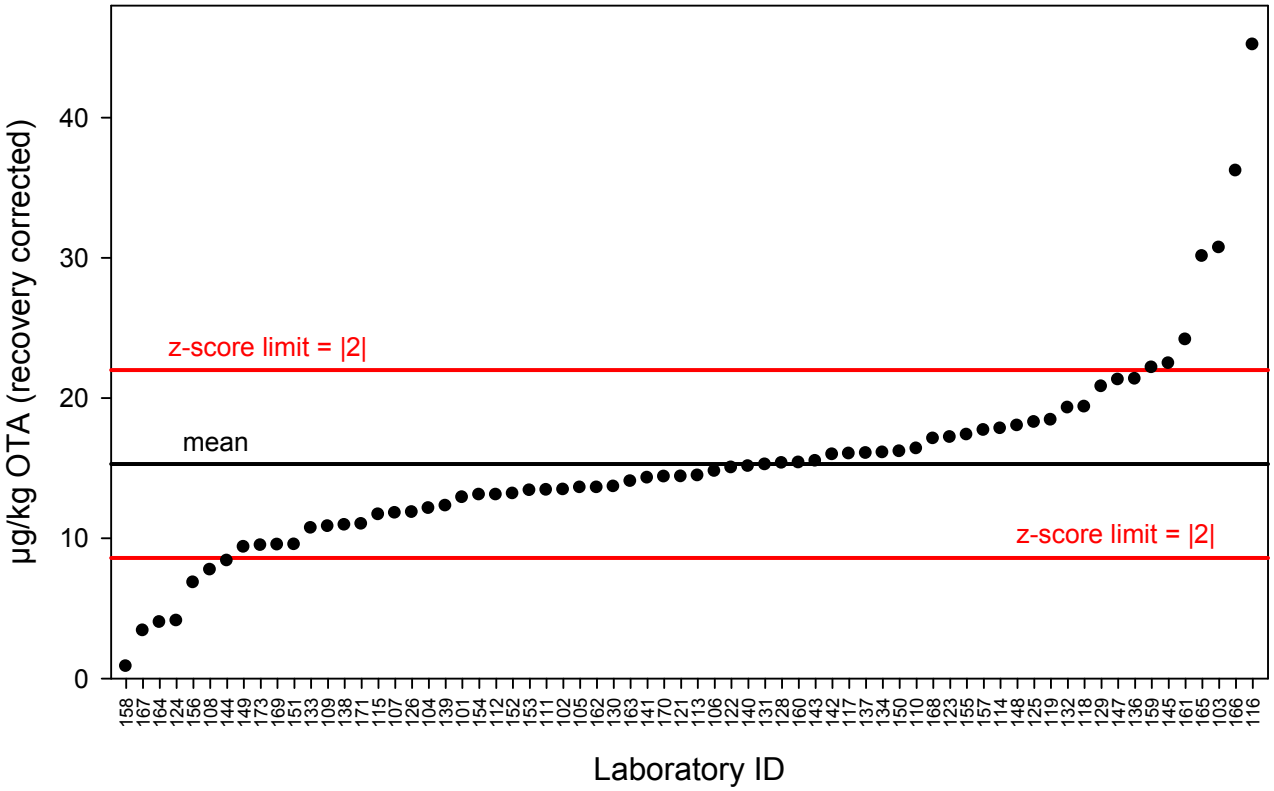
Figure 1: Recovery uncorrected results for OTA in paprika



The mean value calculated from the submitted results was adopted as the consensus value. The upper and lower z-score limits are the 44% (2 x 22%) deviation of the mean, indicating a deviation derived from a HorRat of 2.

[4] Commission (EC) No Regulation 401/2006 and Commission (EC) No Regulation 1881/2006 (http://europa.eu.int/eur-lex/lex/RECH_naturel.do)

Figure 2: Recovery corrected results for OTA in paprika



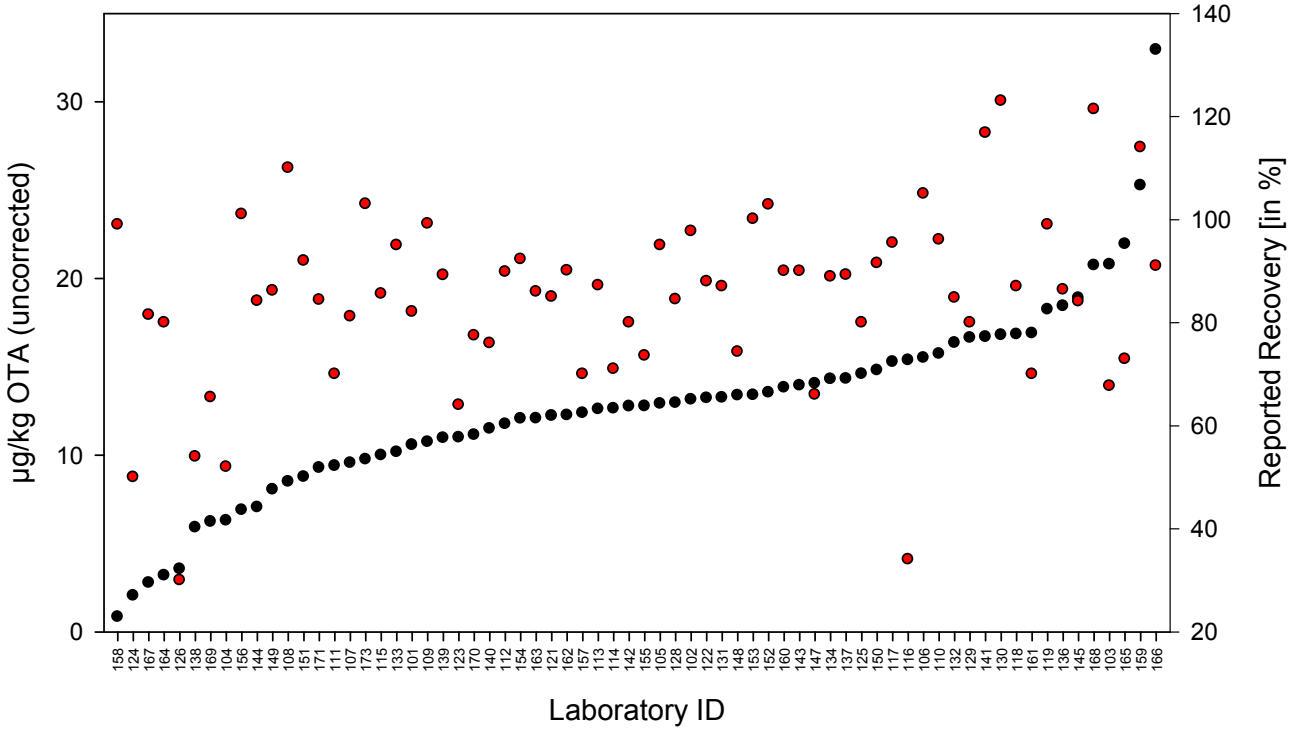
The mean value calculated from the submitted results was adopted as the consensus value. The upper and lower z-score limits are the 44% (2 x 22%) deviation of the mean, indicating a deviation derived from a HorRat of 2.

The number of laboratories that fell outside the z-score limit of 2 decreased from 17 to 14 after recovery correction which indicates a small improvement in the overall performance of the laboratory population. This supports the previous findings on recovery correction for aflatoxins reported by von Holst *et al.*[5]. Nevertheless in some particular cases a correction for recovery had a negative effect on the individual z-score. Such a negative effect is likely for cases where a laboratory's realistic recovery is different from the one stated in this study, as this has then a negative effect on the corrected value. To visualise the link between the reported result and the associated recovery, both values were plotted together in Figure 3.

An overall relation can be seen between the reported recovery and the value of the analytical result, which, in an ideal case, are influenced by the same analytical circumstances. In those cases where uncorrected analytical result and recovery are influenced by the same factors (in a particular laboratory), this has a beneficial effect on its overall performance, whereas where both analysis (uncorrected results and recovery experiment) do not match or are derived under different circumstances (influences), this can lead to severe bias.

[5] C. von Holst, J. Stroka, E. Anklam (2002), *Food Additives Contaminants*, **19**, 701-708

Figure 3: Recovery uncorrected results for OTA in paprika in combination with recovery data.



The black dots (●) show the uncorrected results (left legend) and the red dots (●) the reported recovery (right legend).

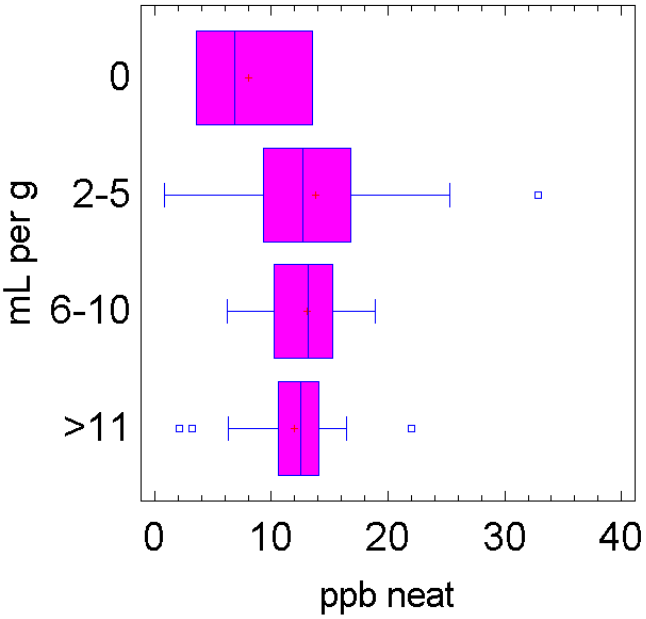
In addition to the z-score ranking the answers given by the laboratories in the accompanying questionnaire were evaluated and are discussed in the following. The reason behind this was that for the moment there is no standardised method for OTA in paprika available. The questions focussed on procedural details to help identifying critical parameters for future method standards, while also helping the laboratories to identify key parameters in their own methodology.

Parameters associated to the extraction procedure

It has been highlighted on several occasions in the past that the solvent-to-sample ratio (S2SR) in some cases can influence analytical results, as demonstrated for aflatoxins in paprika powder[6]. The reported S2SR were plotted as Box plots in Figure 4. The plot showed that no trend towards higher or lower values can be observed for increasing S2SR for the analytical results (uncorrected for recovery). Nevertheless the variability of results seems to decrease for higher S2SR, which is an indicator for better method robustness at higher S2SR values.

[6] J. Stroka *et.al.*, (1999) *Food Additives and Contaminants*, **16**, 331-338

Figure 4: Box plot for the S2SR for OTA in paprika.



Correlation between the obtained value for OTA in paprika and the S2SR reported. The top bar with the value of zero (0) represents those results for which no S2SR was reported. Other bars show values for a particular S2SR ranges.

Another parameter closely linked to extraction behaviour of OTA (similar as the S2SR) is the type of extraction solvent used. The results are shown in Figure 5. It can be seen that mixtures of MeCN/water results in slightly higher values (uncorrected for recovery) compared to all other solvents used. This can be due to an effect similar to that reported by Stroka *et al.* for aflatoxins in paprika. Besides, it can be seen that the addition of NaHCO3 to the extraction solvent seems to increase the robustness of the extraction efficiency independent of the nature of the solvent (pure water or MeOH mix). When comparing the influence of extraction solvent after recovery correction, it appears that a methanolic solution of NaHCO3 is a good compromise with respect to extraction robustness and extraction efficacy under the consideration of toxicological and waste management aspects. Nevertheless it is also the extraction solvent with the highest number of outliers (small squares) and the reasons for this should be evaluated prior to any final conclusion on the performance, as these outliers can also be due to other factors that need to be unscrambled first.

Figure 5: Box plot showing the effect of different extraction solvents used.

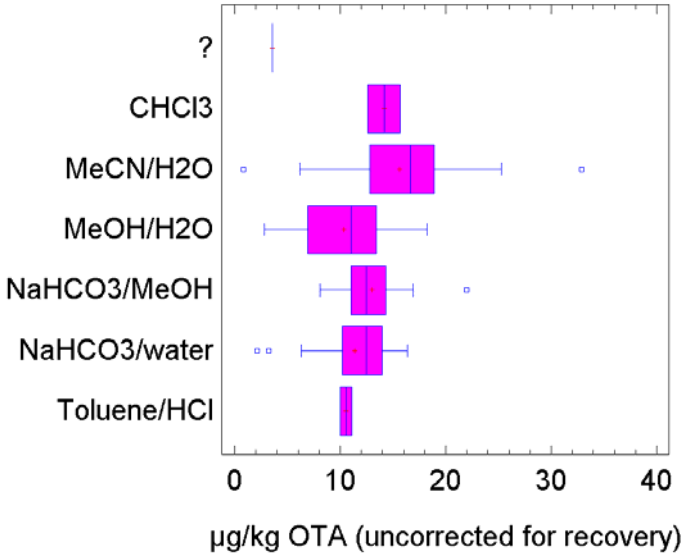
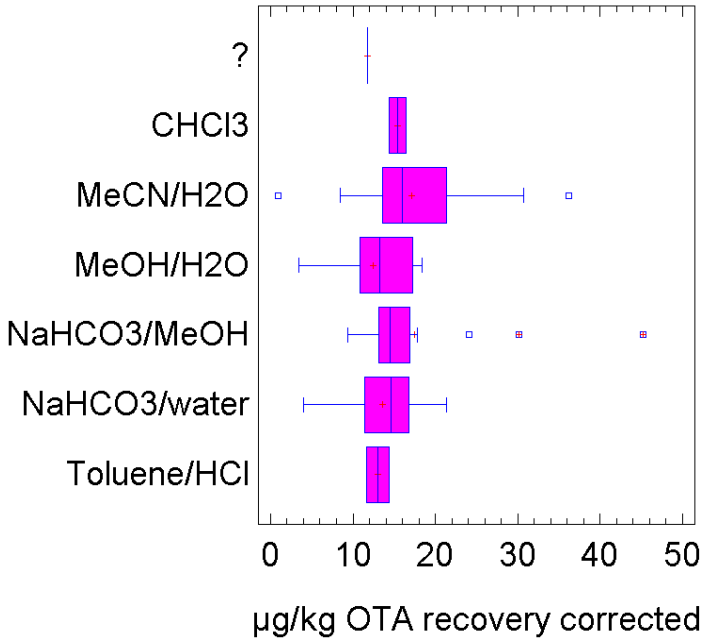
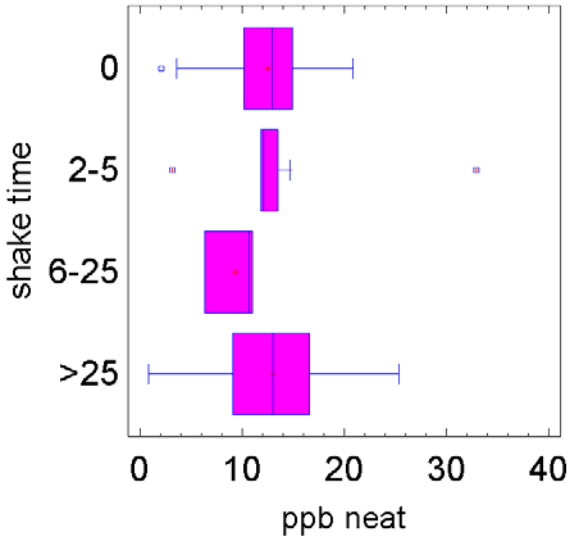


Figure 6: Box plot showing the effect of different extraction solvents used (after recovery correction)



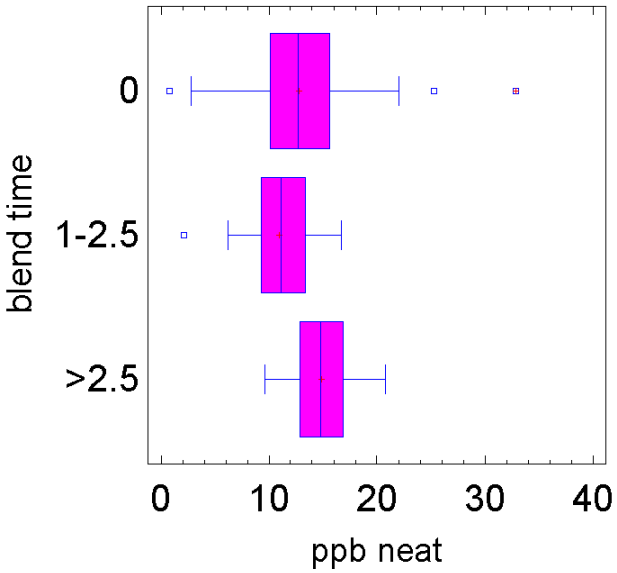
In addition to S2SR and the nature of the solvent, the physical extraction parameters were asked. Laboratories used shaking, high speed blending (such as Turrax) or sonification. In a few cases these parameters/procedures were used in combination for the extraction, and the respective values were considered as belonging to all groups of procedures applied (e.g. sonification and shaking) in the evaluation. Figure 7 shows the effect of shaking time on the analytical result, while Figure 8 the effects of blending time and Figure 9 the effect of the sonification duration.

Figure 7: Effects of the shaking time on the analytical result



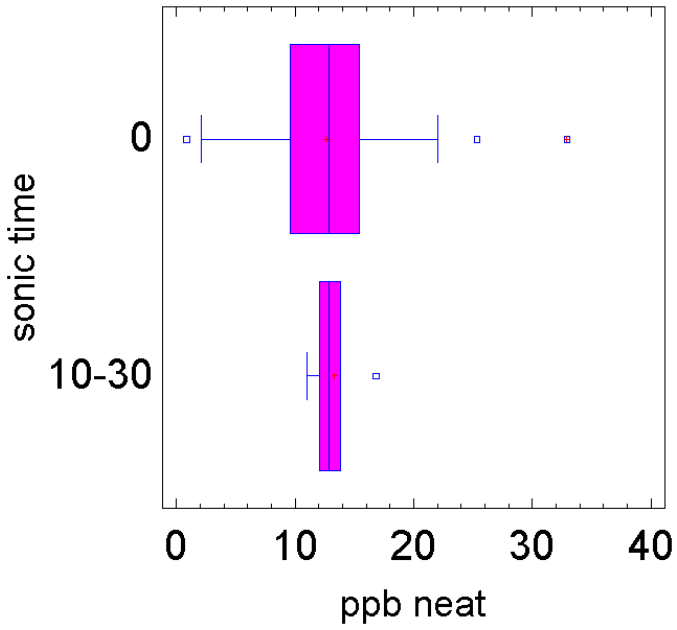
The upper bar with the value of zero (0) reflects all values other than shaking (→sonification and blending) the remaining bars the time in minutes for shaking.

Figure 8: Effects of the blending time on the analytical result



The upper bar with the value of zero (0) reflects all values other than blending (→sonification and shaking) the remaining bars the time in minutes for blending.

Figure 9: Effects of the sonification time on the analytical result



The upper bar with the value of zero (0) reflects all values other than sonification (→blending and shaking) the second all sonification extractions (here 10-30 minutes).

During the production of the test materials for this PT special care was taking regarding the milling of the material in order to achieve a fine and homogeneous powder (<0.5 mm in a centrifugal mill). This should benefit a fast migration of any OTA bound to the material to the surface during extraction and should be kept in mind for the hereafter discussed effects.
It appears from the data in Figures 7 - 9 that in case of shaking, the duration should be at least 30 minutes. For lower shaking time periods the number of data points is unfortunately too low. Furthermore for a short shaking period (2 minutes) the robustness of the procedures appears to suffer. For blending, an increase of OTA extraction efficacy found can be observed in the period of one to three minutes of blending time, while any further blending time seems to have no beneficial effect. The most robust procedure appears to be sonification for ten to thirty minutes. Nevertheless, the population

is rather small and for any further conclusions more data points would be necessary to prove the validity of this observation.

Calibration procedures

In addition to the extraction parameters emphasis was put on the influence of some aspects of the calibration. Laboratories were asked to indicate whether they checked their calibrants by spectrophotometry prior to analysis. The effect is shown in Figure 10. When comparing the source of the calibrant, it appears that there is a slight trend in the analytical results (Figure 11). The analysis of this trend must however also consider whether laboratories do a calibrant check or not, which has not been done in this case. Therefore caution should be exercised before drawing any conclusion on the basis of this result. Further information on the batch of the materials was evaluated, which can also be important. However, as a general recommendation, laboratories are strongly advised to perform calibrant checks. In the ideal case this relates to spectrophotometry (for content) and a general chromatographic check by LC-UV at the same wavelength that is used for spectrophotometry (purity assessment). The efficacy of the calibrant check as shown in Figure 10 relates not only to the generally smaller dispersion of the results (smaller boxes) but also to the fact that the uncontrolled calibrants give higher analytical results for OTA in test materials, which shows that the general tendency is that the apparent amount of OTA in the calibrant is overestimated, due to possible degradation.

Figure 10: Influence of a photometric calibrant check on the result

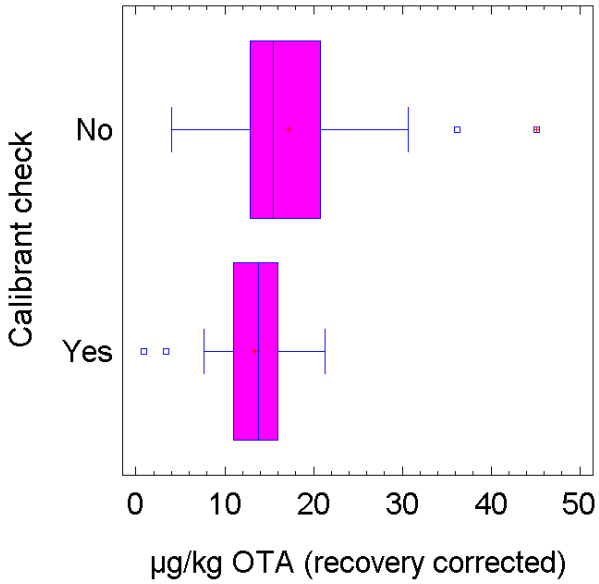
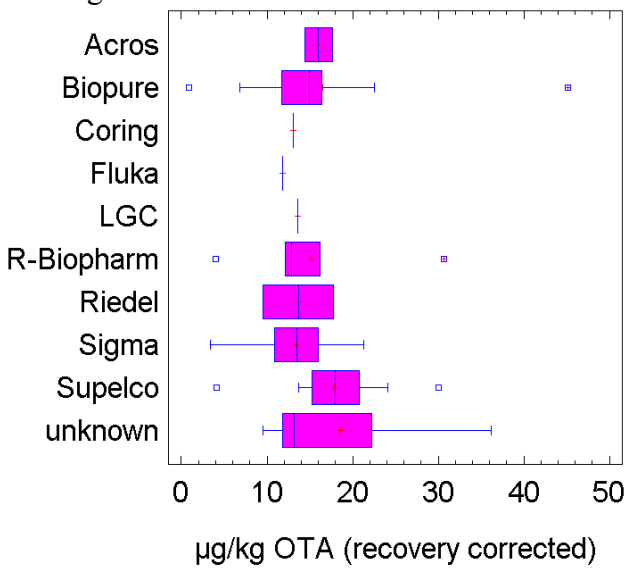


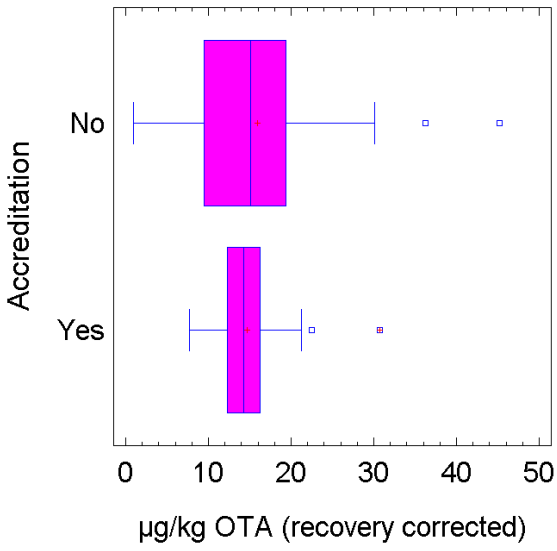
Figure 11: Influence of the source of the calibrant



Accreditation

An interesting effect was observed when results were plotted against the fact whether the laboratory stated that it is accredited for this type of analysis (Figure 12). This question must be seen under the aspect that in several cases laboratories were assigned (or identified) by their national competent authority in the EU Member States to participate in this PT. Therefore the question of accreditation could probably be also expressed as whether the laboratory has had previous experience with this type of analysis (OTA in paprika). At least it might be heavily influenced by this fact and as a result, experience appears to have a clear influence on the performance. Differently from the effect of calibrant checks, the median values are nearly the same (similar to the corresponding mean values of 14.9 vs. 15.7 µg/kg). The dispersion however is significantly smaller.

Figure 12: Previous experience proven by accreditation.



Evaluation of effects on overall performance

With respect to the initial request by DG SANCO and evaluation of the above described criteria/parameters the overall performance of the participating laboratories can be described in the following way. The reproducibility relative standard deviation (RSD_R) of the overall laboratory population without any statistical outlier removal (n=68) was 45.6%.
Taking into account only those laboratories that stated that they have proven their competence in this kind of methodology by accreditation (n=37) the RSD_R was 29.8%. This is a rather impressive figure, taking into account that this reproducibility reflects a HorRat[7] value of 1.0 (classical Horwitz equation) while the modified HorRat according to Thompson $HorRat_{Th}$ was 1.4.
For those laboratories that only performed a calibrant check (n=34) the RSD_R was 35.3% which can be translated to a HorRat and $HorRat_{Th}$ of 1.2 and 1.6 respectively.
For the laboratory population that performed a calibrant check and proved their competence in the methodology by accreditation (n=19) an even more impressive RSD_R of 28% was achieved reflecting a HorRat and $HorRat_{Th}$ of 0.9 and 1.3 respectively. The interpretation of this performance figures shall take into consideration that this study was not a method validation by collaborative trial but a PT and that only the methodology (immunoaffinity clean up followed by high performance liquid-chromatography with FL determination) was shared by all PT participants, while laboratory specific procedures, such as extraction mode, were not prescribed.
Nevertheless the obtained reproducibility figures indicate (under the condition of a calibrant check and proven experience in methodology) that the comparability of results between laboratories for the discussed methodology - *OTA in paprika by IAC clean-up with HPLC-FL* - can compare with minimum performance criteria as they apply to method validation studies by EU legislation[8].
An additional observation is that those laboratories that used LC coupled with mass selective detection (#156 and #159) reported values that seem to indicate that this methodology needs further optimisation prior any use in future collaborative trail activities.

Conclusion

During this PT not only the current situation on the performance of laboratories within the EU Member States and Third Countries was evaluated, but also methodological aspects that are useful for a possible development of a standard method have been elaborated.
The importance of proper calibration procedures was demonstrated in this PT. Extraction parameters were compared and the available data indicated that sonification is a valid alternative to the widely used high speed blending or shaking, as it can combine the positive practical aspects of the other two extraction procedures with regards to cross contamination (shaking) and time of extraction (blending). A positive, while only slight influence on the analytical result was shown by the correction for recovery.
The overall performance of the methods as used by this population of laboratories was comparable to reproducibility estimates that are required by EU legislation from collaboratively validated methods, under the premises that a calibration check is performed and that the laboratory has practical experience in this methodology.

[7] W. Horwitz (1982) *Analytical Chemistry* **54**, 67A-76A.
[8] Commission Regulation (EC) No 401/2006

Annex

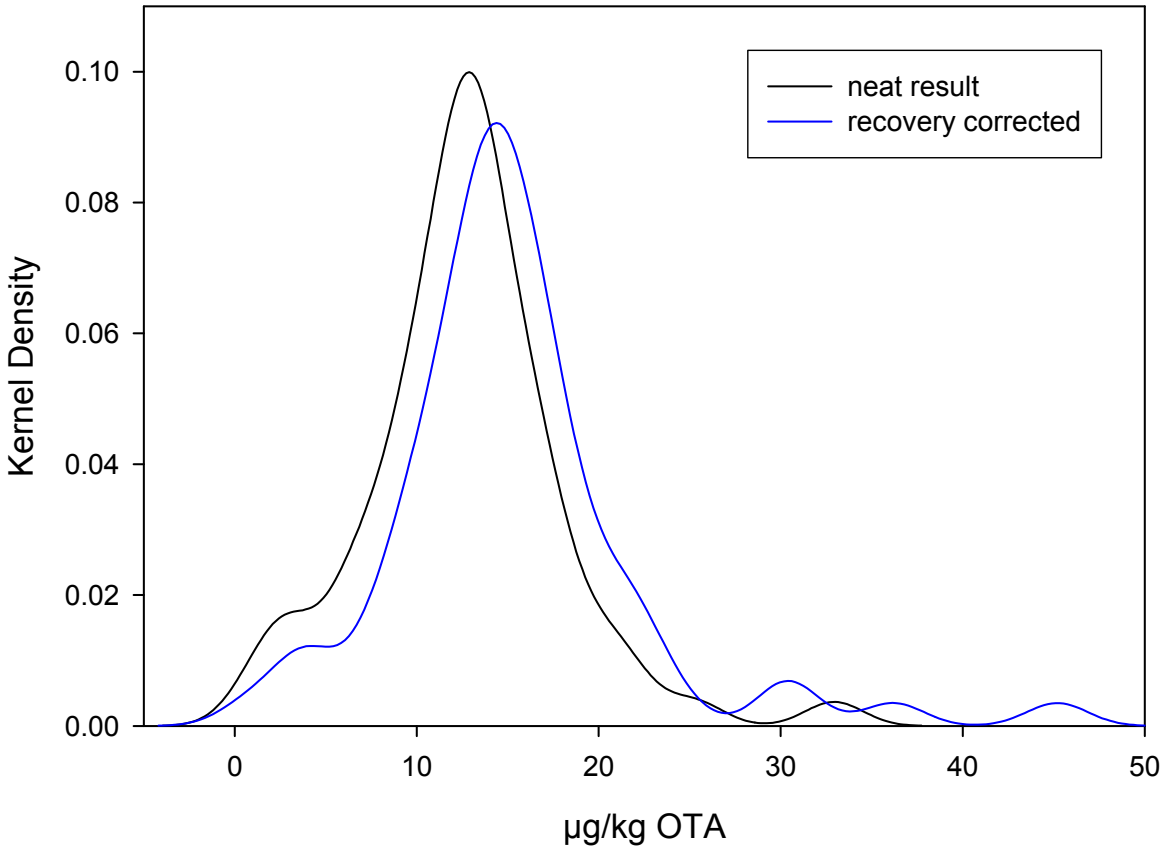
ID	OTA [µg/kg]	z-score[9]	Recovery [%]	OTA corrected [µg/kg]	z-score[10]
101	10.6	-0.8	82.1	12.9	-0.7
102	13.1	0.2	97.8	13.4	-0.5
103	20.8	2.9	67.7	30.7	4.6
104	6.3	-2.3	52.0	12.1	-0.9
105	12.9	0.1	95.0	13.6	-0.5
106	15.5	1.0	105.0	14.8	-0.2
107	9.6	-1.1	81.2	11.8	-1.0
108	8.5	-1.5	110.0	7.7	-2.2
109	10.7	-0.7	99.2	10.8	-1.3
110	15.7	1.1	96.1	16.4	0.3
111	9.4	-1.2	70.0	13.4	-0.6
112	11.8	-0.3	89.9	13.1	-0.7
113	12.6	0.0	87.2	14.4	-0.2
114	12.6	0.0	71.0	17.8	0.8
115	10.0	-1.0	85.6	11.7	-1.1
116	15.4	1.0	34.0	45.2	8.9
117	15.3	0.9	95.5	16.0	0.2
118	16.8	1.5	87.0	19.4	1.2
119	18.2	2.0	99.0	18.4	0.9
121	12.2	-0.2	85.0	14.4	-0.3
122	13.2	0.2	88.0	15.0	-0.1
123	11.0	-0.6	64.0	17.2	0.6
124	2.1	-3.8	50.0	4.1	-3.3
125	14.6	0.7	80.0	18.3	0.9
126	3.6	-3.3	30.0	11.8	-1.0
128	13.0	0.1	84.5	15.3	0.0
129	16.6	1.4	80.0	20.8	1.6
130	16.8	1.5	123.0	13.7	-0.5
131	13.3	0.2	87.0	15.2	0.0
132	16.4	1.3	84,8	19.3	1.2
133	10.2	-0.9	95.0	10.7	-1.4
134	14.3	0.6	88.9	16.1	0.2
136	18.4	2.1	86.4	21.3	1.8
137	14.3	0.6	89.3	16.0	0.2

[9] For the neat result
[10] For the recovery corrected result

ID	OTA [µg/kg]	z-score[11]	Recovery [%]	OTA corrected [µg/kg]	z-score[12]
138	5.9	-2.4	54.0	10.9	-1.3
139	11.0	-0.6	89.2	12.3	-0.9
140	11.5	-0.4	76.0	15.1	0.0
141	16.7	1.4	116.8	14.3	-0.3
142	12.8	0.0	80.0	16.0	0.2
143	13.9	0.4	90.0	15.5	0.1
144	7.1	-2.0	84.2	8.4	-2.1
145	18.9	2.2	84.1	22.5	2.1
147	14.1	0.5	66.0	21.3	1.8
148	13.4	0.2	74.3	18.0	0.8
149	8.1	-1.7	86.2	9.4	-1.8
150	14.8	0.8	91.5	16.2	0.3
151	8.8	-1.4	92.0	9.5	-1.7
152	13.5	0.3	102.9	13.2	-0.6
153	13.4	0.3	100.1	13.4	-0.6
154	12.1	-0.2	92.3	13.1	-0.7
155	12.8	0.0	73.6	17.4	0.6
156	6.9	-2.1	101.0	6.8	-2.5
157	12.4	-0.1	70.0	17.7	0.7
158	0.8	-4.2	99.0	0.9	-4.3
159	25.3	4.5	114.0	22.2	2.0
160	13.8	0.4	90.0	15.4	0.0
161	16.9	1.5	70.0	24.1	2.6
162	12.3	-0.2	90.1	13.6	-0.5
163	12.1	-0.2	86.0	14.0	-0.4
164	3.2	-3.4	80.0	4.0	-3.4
165	22.0	3.3	72.9	30.1	4.4
166	32.9	7.3	91.0	36.2	6.2
167	2.8	-3.6	81.5	3.4	-3.5
168	20.7	2.9	121.4	17.1	0.5
169	6.2	-2.3	65.5	9.5	-1.7
170	11.1	-0.6	77.5	14.4	-0.3
171	9.3	-1.2	84.4	11.0	-1.3
173	9.8	-1.0	103.0	9.5	-1.7

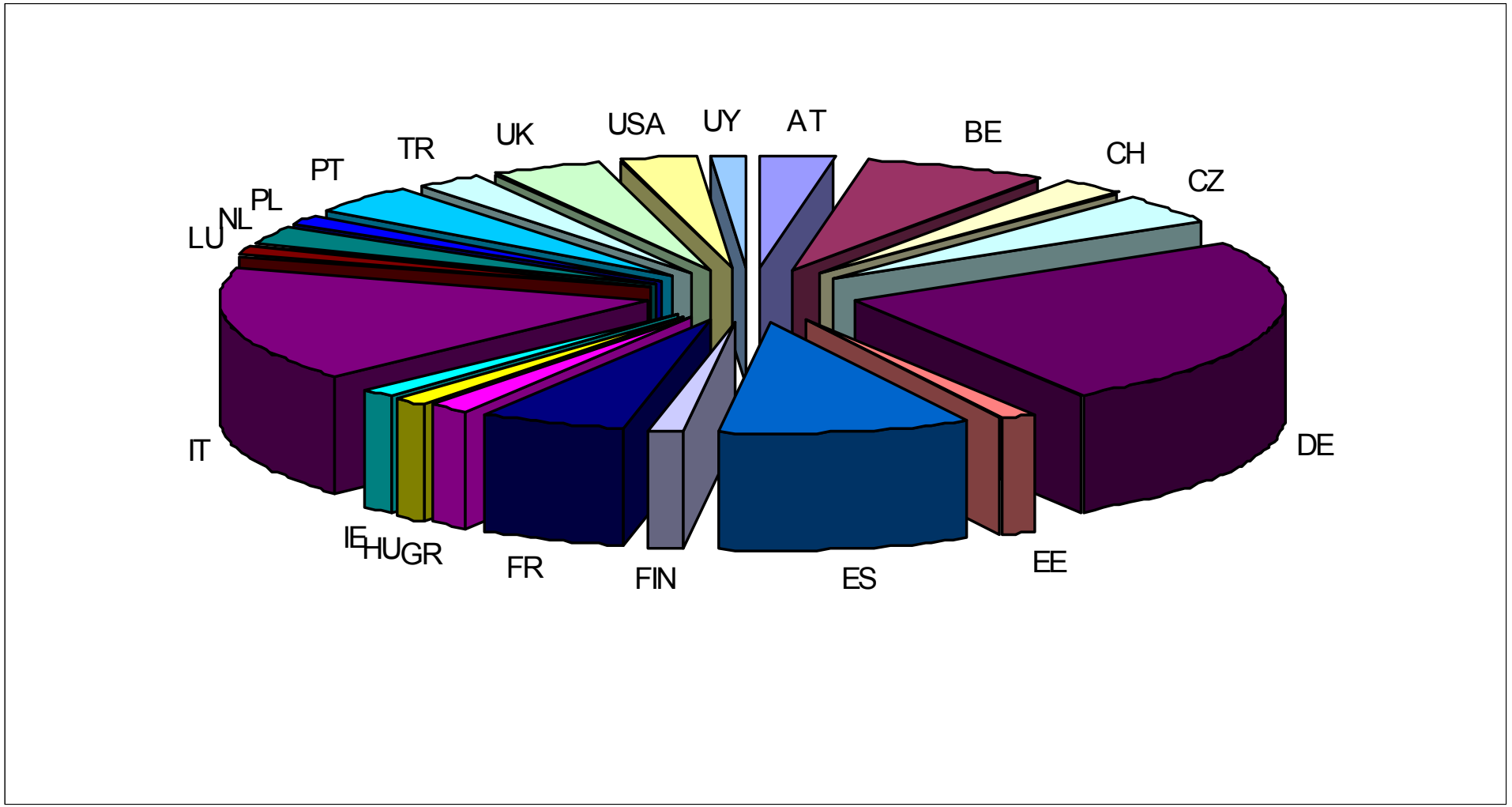
[11] For the neat result
[12] For the recovery corrected result

Kernel Density Plot of the result prior and after recovery correction.



A slight shift can be seen to higher values after recovery correction. The population of results appears normal distributed with some outliers at the higher end scale (overestimation of results).

Participation by Countries



Comparison of robust and conventional statistical data evaluation.

AMC Robust Statistics V1.0	Neat result				
		RobRSD	**ConvRSD**	z+2 mHor	z-2 mHor
ROBUST STATISTICS SUMMARY					
Estimate	*Estimate value*				
Median	12.765			18.3	7.1
A15 mean	12.581589				
H15 mean	12.565751	12.6	12.7		
MAD	2.675				
MADe	3.9659573				
sMAD	3.9659573				
H15 Std Dev	4.3571361	34.7	41.8		
AMC Robust Statistics V1.0	Recovery				
ROBUST STATISTICS SUMMARY					
Estimate	*Estimate value*				
Median	86.3				
A15 mean	85.681296				
H15 mean	85.635965	85.6	84.9		
MAD	8.75				
MADe	12.972758				
sMAD	12.972758				
H15 Std Dev	14.190129	16.6	20.5		
AMC Robust Statistics V1.0	Corrected				
ROBUST STATISTICS SUMMARY					
Estimate	*Estimate value*				
Median	14.413006			22.0	8.6
A15 mean	14.599415				
H15 mean	14.693314	14.7	15.3		
MAD	2.7584696				
MADe	4.0897095				
sMAD	4.0897095				
H15 Std Dev	4.7260978	32.2	45.6		

Comparison of conventional and robust statistics for the total population results for the uncorrected value (neat result), the recoveries reported (Recovery) and the recovery corrected result (Corrected). The difference in both results can be explained by the fact that no outlier test was applied for the conventional statistics approach.

ID	Method Reference	Accredited	Concentration check
101	Application Note–Paprika–Ochratoxin A Extraction Method, Ref. No. A3–P14.V3, July 2005, R-Biopharm Rhône Ltd.	Yes	No
102	VICAM, Application Note, ref. A-3-P14.V1, March, 1998	Yes	Yes
103	R-Biopharm Rhone Poulenc recommended method	Yes	No
104	Internal method based in the report " Acerca de la possible contaminación por ocratoxina A en alimentos. C. Araguas, E. Gonzalez… Alimentaria, Mayo 03/23"	No	No
105		Yes	No
106	DIN ISO 3696 modified	Yes	Yes
107	ASU § 35 LMBG (§ 64 LFGB) L 15.00-1	Yes	Yes
108	Ochraprep®, Instructions for Use	Yes	Yes
109	DIN NA 057-05-07 AA N 254	No	Yes
110	Modified method of SOP ARO/430	Yes	No
111	VICAM Ochratest Instruction Manual, 1999	No	Yes
112	EN 14132	No	Yes
113	Methodology Publications of National Institute of Hygiene, 2005	Yes	Yes
114		Yes	No
115	§35 LMBG Untersuchung von Lebensmittel: Ochratoxin A in Getreide und Getreideprodukten (November 1999).	Yes	No
116	Adapted from Neogen, method for coffee	No	No
117	prEN 14132	No	Yes
118	in-house	No	Yes
119	in-house	No	Yes
121	in-house	Yes	Yes
122	Determination of Ochratoxin A in animal feed	No	No
123		No	No
124	METHOD R-BIOPHARM	No	No
125	Application note for analysis of ochratoxin A in paprika using OCRAPREP, R-Biopharm July 2005	No	No
126		Yes	Yes
128	UNI EN 14132:2003	Yes	No
129	OR SELL	No	No
130	MIP AGER OCRA 2007 Rev3	Yes	No
131	Instruction from IAC Supplier	No	No
132	Internal Method (R-Biopharm Rhone Application Note for analysis of Ochratoxin A in paprika using Ochraprep	No	No
133	A. Pittet 1996	Yes	Yes
134	SLMB Method 54.1.1.4/99	Yes	Yes

ID	Method Reference	Accredited	Concentration check
136	AOAC 2000.03	No	Yes
137	AOAC Official Method 2004.10 & AOAC Official Method 2000.03	No	Yes
138	Method 970.45 AOAC2005:ch. 49 & validated method M. J. Hernandez, M. V. García Moreno, E. Durán, et al.Analytica Chimica Acta, 566, 2006:117-121.	Yes	Yes
139	DIN EN 14133 Determination of OTA in wine and beer - HPLC method with immunoaffinity column clean-up; DIN EN 14132 Determination of OTA in barley and rosted coffee - HPLC method with immunoaffinity column clean-up;	Yes	Yes
140	in-house, based on IA column supplier application note	No	Yes
141	Rhone-Poulenc Method Sheet-Quantitative detection of Ochratoxin A Ref Ochraprep IFU (P1448)	Yes	Yes
142	EN 14132	Yes	Yes
143	in-house	Yes	No
144	J. AOAC Int Vol. 84, No. 6, 2001, 1818f	Yes	No
145	VDLUFA Methodenbuch	Yes	No
146	DIN EN ISO 15141 part 1 (modified) HPLC **with silica column clean-up**	Yes	Yes
147	internal reference PNTA0077	Yes	No
148	in-house method	Yes	No
149	Project SMT-CT96-2045, High performance liquid chromatographic method for the determination of ochratoxin A in barley	No	Yes
150	R-BioPharm application note of IAC	Yes	No
151		No	Yes
152	CEN/TC 275/WG 5	Yes	Yes
153	DIN EN 14132 mod.	Yes	No
154	DIN EN ISO 15141	Yes	No
155	Application note for analysis of ochratoxin A in paprika using Ochraprep (R-BIOPHARM RHONE LTD)	Yes	Yes
156	(HPLC-MS/MS), I. Yu. Goryacheva (Analytica Chimica acta 577 (2006) 38-45	No	No
157	in-house (described)	Yes	Yes
158	J AOAC Int 83:1377–1383.	No	Yes
159	inhouse method **LC-MS**	No	No
160	in-house	Yes	No
161	Method based on procedure provided by JRC	No	No
162	Application Coring-system, Immunoaffinity Column / HPLC (1996)	Yes	Yes
163	“mixture” of some official methods (German LFGB § 64 method, Swiss method 1387.1, German VDLUFA OTA draft method), some findings in theses, some papers and VICAM OchraTest Instruction Manual (March 25, 2003).	No	Yes
164	IAC clean-up & HPLC-FL	No	No
165	Ochratest (Vicam)	No	No
166	R-Biopharm	No	No
167	Food Additives, and Contaminantes 22(9):856-863,2005	No	Yes
168	VDLUFA-Methodenbuch Band III 3.Erg. 1993 - Methode 16.10.1	Yes	Yes
169		Yes	No
170	Instructions manufaturer	No	Yes
171	VICAM Procedure March 25, 2003	Yes	Yes
173	paper in Food Microbiology, 2007	No	No

ID	Method For Calibrant Check	OTA reference material			
		commercial solution	crystalline substance	provider	lot #
101		Yes		R-Biopharm Rhône Ltd	UG 366
102	Official Methods of Analysis, AOAC, 17th edition, (2000)		Yes	Sigma	51K4085
103		Yes		R-Biopharm Rhone LTD	UK419
104		Yes		R-Biopharm	
105			Yes	LGC	
106	DIN ISO 3696		Yes	Sigma	27H4031
107	6640 l mol-1 cm-1		Yes	Fluka	369626/1597
108			Yes	Sigma	50K4101
109	§ 64		Yes	Sigma	
110		Yes		Biopure	07164B
111	EN 15141-1:2003		Yes	Sigma-Aldrich	
112	EN 14132		Yes	Sigma	38H4120
113	PN EN 114132:2004		Yes	Biopure	03093Z
114		Yes		Riedel de Haen	6279x
115		Yes		Biopure	L07164B
116		Yes		Biopure	6225A
117	prEN 14132		Yes	Acros	A0211590001
118	EN 14132		Yes	Sigma	52K 4061
119	NE EN 14132		Yes	Sigma	
121	BIPEA source		Yes	Sigma-Aldrich	
122		Yes		Biopure	L07164B
123		Yes		Supelco	LB36062
124		Yes		Supelco	LB25609
125		Yes		Supelco	LB36062
126		Yes			
128		Yes		Supelco	LB36062
129		Yes		Supelco	
130		Yes		Supelco	LB25609
131		Yes		Supelco	
132		Yes		Supelco	LB36062
133	official method no.1387.1 of the Swiss “SLMB”		Yes	Sigma	
134	Official Method of Analysis AOAC INT.16th Ed.		Yes	Sigma	27H4031

ID	Method For Calibrant Check	OTA reference material			
		commercial solution	crystalline substance	provider	lot #
136	AOAC 2000.03		Yes	Sigma	126K4027
137	AOAC Official Method 2000.03		Yes	Sigma	126K4026
138	UV		Yes	Sigma	
139	§35 LMBG L 15.00-1,		Yes	Sigma	061 K 4038
140	EC Report EUR 16825 EN.1995		Yes	Sigma	
141	see 1.		Yes	Sigma	045K4132
142	EN 14132		Yes	Sigma	
143		Yes		Biopure	06452A
144		Yes		Sigma	85H4009
145		Yes		Biopure	52732
146	DIN EN ISO 15141 part 1		Yes		
147		Yes		Supelco	LB46579
148		Yes		Supelco	LB36062
149	Resolution Oeno 16/2001,		Yes	Sigma	
150		Yes		R-BioPharm	UK419
151			Yes	Sigma	51K4085
152	CEN/TC 275/WG 5		Yes		
153			Yes	Sigma	51K4085
154		Yes		Coring	R06225A
155	AOAC		Yes	SIGMA-ALDRICH	063K4060
156		Yes		Biopure	
157	Calibrant vs. Methanol @ 330 nm.	Yes		Arcos	A014130101
158	AOAC		Yes	Biopure	L07302A
159		Yes			
160		Yes		Supelco	
161		Yes		Supelco	LB36062
162			Yes	SIGMA-ALDRICH	
163	Lambert-Beer law		Yes	SIGMA-ALDRICH	126K4027
164		Yes		R-BioPharm	
165		Yes		Supelco	
166		Yes			
167	AOAC Method 973,37 Stoloff and Scott 1995, Adapted		Yes	Sigma	S1K4085
168	photometry		Yes	Sigma	50K4101
169		Yes		Riedel-de Haën	6279X
170	AOAC		Yes	acros	A009519501
171	IPH/FAVV Workshop – Brussel WIV - 12/6/2007		Yes	Sigma	045K4132
173		Yes			

ID	Concentration determination		
	gravimetrically	spectrophotometrically	both
101			
102		Yes	
103			
104			
105		Yes	
106		Yes	
107			Yes
108		Yes	
109		Yes	
110			
111		Yes	
112		Yes	
113		Yes	
114			
115			
116			
117		Yes	
118		Yes	
119		Yes	
121		Yes	
122			
123			
124			
125			
126			
128			
129			
130			
131			
132			
133		Yes	
134		Yes	
136			Yes
137		Yes	

ID	Concentration determination		
	gravimetrically	spectrophotometrically	both
138		Yes	
139		Yes	
140		Yes	
141		Yes	
142		Yes	
143			
144			
145			
146		Yes	
147			
148			
149		Yes	
150			
151		Yes	
152		Yes	
153			
154			
155		Yes	
156			
157			
158		Yes	
159			
160			
161			
162		Yes	Yes
163			Yes
164			
165			
166			
167		Yes	
168		Yes	
169			
170		Yes	
171		Yes	
173			

ID	Extraction solvent	Extraction solvent to sample ratio	Extraction mode
101	1% Sodium Bicarbonate	20:1	Blend 2 min
102	1% Sodium Bicarbonate	25:1	Blend 2 min
103	Acetonitrile:water (60:40)	4:1	Blend 3 min
104	1% Sodium Bicarbonate	20:1	Shake 20 min
105	Acetonitrile/Water	10:1	Blend 3 min
106	MeOH/NaHCO3(50+50)	20:1	Shake
107	NaHCO3/Methanol	10:1	Blend 15 min
108	water/NaHCO3(99/1)	20:1	Blend 2 min
109	NaHCO3:H2O(80:20)	20:1	Blend 15 min & Shake 15 min
110	CHCl3	20:1	Shake 30 min
111	Methanol:1%NaHCO3(70:30)	0,167 g/ml	Blend 2 min
112	Methanol-3%Sodiumbicarbonate (1+1)	10:1	Shake 3 min
113	CHCl3	5:1	Shake 30 min
114	Methanol/3%NaHCO3(50/50)	5:1	Shake 60 min
115	2M HCl/0,4M MgCl2/Toluene(15/25/50)	9:1	Shake 60 min
116	MeOH/1%NaHCO3(50/50)	10:1	Blend 4 min
117	MeCN–H2O(60-40)	8:1	Blend 2 min
118	60% MeCN	10:1	Magnetic stirrer 30 min
119	MeOH/H2O(8:2)	10:1	Shake 30 min
121	1%bicarbonate aqueous solution	20:1	Blend
122	MeOH/3%NaHCO3(50+50)	10:1	Shake 40 min
123	CH3OH+Water+NaCl	4:1	30 min
124	0.1%NaHCO3	20:1	Blend 2 min
125	1% aqueous bicarbonate	20:1	Blend
126			
128	MeCN/ WATER 6:4	4:1	Blend 3 min
129	MeCN/H2O 60%	2.5:1	Blend 30 min
130	MeCN/H2O(60/40)	2:1	Sonication 15 min
131	NaHCO3 1% aqueous solution	20:1	Blend
132	NaHCO3 1% aqueous solution	20:1	Shake 30 min
133	MeOH/3%NaHCO3	10:1	Shake 60 min
134	Methanol/3% NaHCO3(50+50)	8:1	Blend 3 min
136	Acetonitrile/Water 60/40	4:1	Blend 3 min
137	MeOH-3% aqueous NaHCO3(1+1)	8:1	Shake 40 min

ID	Extraction solvent	Extraction solvent to sample ratio	Extraction mode
138	Methanol/ water/ NaCl	5:1	Shake 30 min
139	Solution of NaHCO3 (1%) : H2O (80:20, v/v)	20:1	Sonicate 15 min & Shake 15 min
140	1% sodium bicarbonate solution	20:1	Blend
141	60% Acetonitrile	4:1	Blend 1 min
142	Acetonitril/Water	4:1	Blend
143	methanol:bicarbonate 70:30	10:1	Blend 2 min
144	Acetonitril / Water (60+40, v/v)	4:1	Shake 30 min
145	MeCN/H2O(6:4)	8:1	Mag. stir 30 min
146	Toluene (in the presence of solutions of hydrochloric acid and magnesium chloride)	10:1	Shake 60 min
147	1% aqueous sodium bicarbonate	20:1	Blend 2 min
148	0,1% NaHCO3	40:1	Blend 2 min
149	MeOH/3% aqueous NaHCO3 solution 50:50	8:1	Shake 40 min
150	1% NaHCO3	10:1	Blend 3 min & Shake 30 min
151	Acetonitrile:Water(60:40)	4:1	Blend 2 min
152	Water and methanol	2+8	Shake 2 min
153	MeOH/Water	5:1	Shake 30 min
154	Methanol/Bicarbonat 1 %	4:1	Blend 2 min
155	1% aqueous NaHCO3	20:1	Shake 60 min + Sonicate 10 min
156	MeOH/H2O (80/20)	14:3	Shake 60 min
157	Methanol:Wasser (80:20)	4:1	Shake 60 min
158	Acetonitrile/water 60/40	5:1	Shake 60 min
159	Acetonitrile/water	4:1	Shake 30 min
160	3% NaHCO3 solution	50:1	Sonicate 30 min
161	Methanol–3% NaHCO3 50:50 (v/v)	8:1	Blend 3 min
162	methanol/3 % aqueous NaHCO3(50/50)	40:1	Blend 2 min
163	methanol and 3 % aqueous NaHCO3 solution (50/50, v/v)	10:1	Sonicate 25 min & overhead shake 5 min
164	1% aqueous NaHCO3	20:1	Shake 2 min
165	Methanol, H2O(3% NaHCO3) 50:50	14:1	Shake 60 min
166	Acetonitrile/Water	5:1	Shake 2 min
167	H20/MeOH(20/80)	4:1	Shake 30 min
168	acetonitrile+water=60+60	4:1	Shake 60 min
169	Acetonitrile/water (60/40)	6.7:1	Blend 1 min
170	Toluene/acetic acid (99/1)	10:1	Blend 2 min
171	Methanol/Water 4/1	4:1	Blend 1 min
173	1% aqueous NaHCO3	20:1	Blend 2 min

ID	Clean-up	Type of column	Injection volume, µL
101	IAC	Waters Spherisorb®, S5 ODS2 250 x 4.6 mm, 5 µm column (Part no. PSS831915)	100
102	IAC	C18 (25 x 4.6 mm, 5um)	20
103	IAC	Hypersil ODS 250*4.6mm 5µ with precol	100
104	IAC	ODS Hypersil 250x4,6; 5µ	100
105	IAC	Kromasil C18 100-5, 250 x 2 mm	10
106	IAC	Nova Pak C18, 3,9 x 150 mm	50
107	IAC	RP 8 250 x 4,6 mm, 5 µm	20
108	IAC	RP-18, 5 µm	100
109	IAC	Rp 18 Lichrospher100 250 * 4mm 5µm	100
110	Solvent-solvent extraction, including IMA clean-up	Phenomemex Prodigy 5u ODS(3) 100A 150 x 4.60 mm	800
111	IAC	Lichrosorb RP-18, 5um 4,6 x 200 mm	50
112	IAC	RP C18	20-50
113	IAC	C18, 5um 250x4,6 mm Waters Symmetry	100
114	IAC	Zorbax Eclipse XDB-C18; 5 µ; 250*4,6	100
115	IAC	Zorbax Eclipse XDB-C8 5µm, 150x4.6	80
116	IAC	RP-18, Lichrospher 250-4	100
117	IAC	Waters C18 Sun Fire 5 µm 4.6 X 150	100
118	IAC	C18	100
119	IAC	Kromasil C18	50
121	IAC	150x4.6 mm, 3 µm	100
122	IAC	150 x 4,6 mm	150
123	IAC	C18 RP	200
124	IAC	C18	200
125	IAC	C18 5µm length 25 cm i.d. 4.6mm	100
126			
128	IAC	ODS–INERTSIL (4,6mmX250mm–5 µm)	100
129	IAC	C18	20
130	IAC	DISCOVERY C18	50
131	IAC	C18	200
132	IAC	RP18 250x4 mm 5µm	100
133	IAC	RP-18	80
134	IAC	C18, endcapped	50
136	IAC	C18	125
137	IAC	250mmx4.0 mm reversed-phase C18, ODS-2, 5 µm particle, 11.5% carbon loading, endcapped, 80Å pore size	100

ID	Clean-up	Type of column	Injection volume, µL
138	Liquid/Liquid partition	C18 Columna C18 Gemini, 250mm x 4,60 mm, 5 mm, 110 A, Phenomenex lot 347810-1	20
139	IAC	Lichrospher 100 RP 18,5 µm, 250-4 and precolumn	20
140	IAC	SphereClone 5mm ODS 2 250mm x 4.6mm	100
141	IAC	Spherisorb ODS 2	100
142	IAC	C18	100
143	IAC	C18	50
144	IAC	LiChrosorb RP 18, 7 µm	50
145	IAC	Lichrocart 250-4 HPLC cartridge Purosphere RP-18 endcapped (5 µm)	100
146	Silica column clean-up	LiChrospher RP-C18 100, 5µm, 250x3mm	20
147	IAC	Licrospher 60 Rp-select B (5mm)	100
148	IAC	Nova-Pak C18, 25cm	100
149	IAC	Hichrom Lichrosorb RP 18-5(15cmx4,6 mm id.)	100
150	IAC	Lichrospher C18 250 mm 5microm.	100
151	IAC	Nucleosil C18, 5µm, 4.6mm, 250mm	100
152	IAC	Spherisorb S50 DS2	100
153	IAC	Phenomex Luna C18 250 x 3	100
154	IAC	C 18	30
155	IAC	Synergi 4m Hydro-RP,80A, 250x4,6nm	100
156	IAC	Alltima C18 Alltech	20
157	IAC	Lichrospher 100 RP 18	3-10
158	IAC	C18 Simmetry 150x4.6 mm 5 µm	100
159	dilution	Atlantis T5 (C18)	20
160	IAC	RP C18	20
161	IAC	Agilent ZORBAX Eclipse XDB-C18, 4.6x150 mm, 5 µ	100
162	IAC	RP-18, Spherisorb, 5 µm	100
163	IAC	Phenomenex Synergi 4 µ Fusion-RP80, 150 x 4.6 mm	100
164	IAC	C18	125
165	IAC	C18	10
166	IAC	Waters ODS 2 250 mm 4.6 µm	20
167	IAC	Sunfire, Waters,C18 5um 4,6*250 mmm	200
168	IAC	LiChrospher 100 RP-18e; 5µm; 250mm x 4mm	100
169	IAC	C18	100
170	IAC	C18 endcapped	200
171	IAC	C18	200
173	IAC	C18	100

ID	HPLC Details			
	Isocratic/Gradient (I/G)	Mobile phase	FL λex, nm	FL λem, nm
101	I	Acetonitrile:Water:Acetic Acid (51:47:2)	333	443
102	I	Methanol/9% Acetic Acid (60 : 40)	390	440
103	I	Acetonitrile:Water:Acetic acid (495:495:10)	333	460
104	I	acetonitrile/Water/Acetic acid(51/47/2)	333	460
105	I	Acetonitril/Water/Acetic Acid	333	460
106	I	MeCN/H2O(2 % acetic acid)(32 + 68)	331	471
107	I	Acetonitrile/Water/Acetic Acid =51:47:2	330	460
108	I	Acetonitrile/water/acetic acid (49/49/2; v/v/v)	330	460
109	I	Acetonitril/H2O/Acetic acid(49,5/49,5/1)	330	460
110	G	A:KBr(1.47MM)-MeOH-ACN-HAc (3300+930+780+100); B:KBr(1.47MM)-MeOH-ACN-HAc (140+1283+1073+50)	332	468
111	I	MeCN:H2O:CH3COOH(99:99:2)	333	477
112	I	MeCN-H2O-AceticAcid(495-495-10)	333	460
113	I	MeCN:2% CH3COOH(55:45)	333	460
114	I	acetonitrile/2%acetic acid(45/55)	330	460
115	I	500ml 2%acetic acid+500ml MeCN+134mg KBr+100 µl 69.5%HNO3	330	460
116	I	MeCN/H2O/acetic acid 55/50/1	333	460
117	I	H2O-MeCN-acetic acid(51-48-1)	333	460
118	I	H2O-CH3CN-CH3COOH 54-45-1	333	440
119	I	MeCN/H2O/Acetic acid9600/400/8)	335	475
121	I	water-MeOH-acetic acid	330	460
122	I	MeOH:H2O 3%glacial acetic acid: AcCN(45:35:25)	333	460
123	I	MeCN/Water/Acetic Acid (49.5/49.5/1)	334	460
124	I	CH3CN/4%CH3COOH 60/40	333	460
125	I	Acetonitrile:water:acetic acid (47:51:2)	333	443
126				
128	I	water/CH3CN/Glacial acetic acid(102/96/2)	333	460
129	I	MeCN/H2O/ACETHIC ACID	333	443
130	I	H2O/MeCN/Acetic acid(99/99/2)	333	460
131	I	MeCN:H2O:acetic acid(47:51:2)	333	443
132	I	H2O:MeCN:Acetic Acid (49.5:49.5:1)	333	460
133	I	MeCN/Na acetate/acetic acid	330	470
134	I	H2O/HOAC/MeCN/MeOH(570/30/300/100)	330	464
136	I	Acetonitrile/water/acetic acid (45/54/1)	333	460
137	I	H2O/MeCN/acetic acid(51+48+1)	333	460

ID	HPLC Details			
	Isocratic/Gradient (I/G)	Mobile phase	FL λex, nm	FL λem, nm
138	I	MeCN(5.1.1):H2O(5.1.3):HAc (5.1.4) (49.5/49.5/1)	333	460
139	I	55% water/acetic acid (490/10) + 45% acetonitrile	330	460
140	I	acetonitrile:water:acetic acid 99:99:2	333	477
141	I	water:acetonitrile: 495:495+10ml Acetic acid	333	477
142	I	MeOH/MeCN/H20	390	440
143	I	acetonitril:water:acetic acid 500:500:10	332	460
144	I	60% Acetonitril + 40% Water/Acetic acid (1000ml+20ml, v/v)	333	469
145	I	MeCN/H2O/Acetic acid(510+470+20)	330	465
146	I	Acetonitrile/demin.Water/glacial acetic acid (99/99/2)	330	460
147	I	H2O:MeCN:acetic acid (51:48:1)	333	460
148	I	acetonitril+water+acetic acid (99+99+2)	333	477
149	I	water–acetonitril-acetic acid, 99:99:2	333	460
150	I	water/acetonitrile/acetic acid:53/45/2	333	470
151	I	2% Acetic acid:Acetonitrile (55:45)	333	460
152	I	Water/acetonitrile/Acetic acid(99+99+2)	333	460
153	I	MeOH/H2O/MeCN/HAC(55/40/5/1)	390	460
154	I	2 % acetic acid / MeCN (1:1)	333	477
155	I	102H2O:96Acetonitril:2acetic acid	333	460
156	G	MeCN/H2O/FA		
157	I	0,006 m Natriumdihydrogenphosphat:	330	460
158	I	acetonitrile/water/1% acetic acid 99/99/2	333	460
159	G	Methanol/water + ammonium formate		
160	G	water pH 2,3 (adjusted with phosphoric acid/acetonitrile	330	460
161	I	Acetonitrile/Methanol/Acetic acid (99:99:2)	333	477
162	I	acetonitrile, water, acetic acid	333	460
163	I	Methanol/water/glacial acetic acid (70/30/1,5)	333	460
164	I	Acetic acid/Acetonitrile/Water (2/47/51)	333	443
165	I	Acetonitrile,Methanol,Acetic ac (35:35:29:1)	333	477
166	G	Acetonitrile/ Water/ acetic acid	330	460
167	I	H2O/MeCN/CH3C00H	333	477
168	I	acetonitrile+water+85%H3PO4 410+590+4 (v+v+v);pH 2,3	353	460
169	I	Acetonitrile/water/acetic acid (99/99/2)	333	477
170	I	water/acetonitil/acetic acid (50/50/1)	333	460
171	I	water/MeCN/Acetic acid 495/495/10	333	477
173	I	MeCN/H2O/acetic acid=51/47/2	333	443

ID	Calibrated range of the method	Comments
101	0.2-20µg/l	
102	0.1–23.5ug/kg	Blank contains small amounts of OTA
103	0.10-10 µg/L	
104	1-50µg/L	
105	0.5–10µg/kg	
106	0,2–18,6µg/mL	
107	0,05–6,5ng/ml	
108	3 - 184 ng/ml	
109	0,2–6 µg/kg	
110	32-320 ng	
111	0,0001 ng/ul - 0,1 ng/ul	
112	0,56-28 ng/ml	
113	1-40 ng/g	
114	0,3–3,7µg/kg	
115	2–20 µg/kg	
116	0-4 ng/ml	
117	0.5-10 µg/l	
118	1,25-50 µg/kg	
119		
121	2-125 ng/ml	
122	2-40 ng/g	
123	0.2-5 µg/kg	
124	0.1-5 ng/mL	
125	0.557-22.304ng/ml	
126		
128	0.5-10ng/ml	
129	0-40 µg/L	
130	0,36 - 11,46µg/kg	
131	1.0–60 ng/ml	
132	0.1–5ng/ml	
133	0.2–12ng/ml	
134	0.3-6.4ppb	
136	1-100 ppb	
137	2 ppb – 20 ppb	

ID	Calibrated range of the method	Comments
138	0,75-2,3 µg/kg	
139	1,0 - 40 ng/ml	
140	0-20ng/mL	
141	2.5-10µg/L	
142	0.01 - 50 µg/kg	
143	0,1 - 20 µg/kg	
144	0,9997	
145	0- 4.68 µg/kg	
146	0,05–116 µg/kg	
147	0.1-10 ug/L	
148	2,208 - 0,2208 ng/ml	
149	0,5-10 ng/mL	
150	1-20 ng/g	
151	0.5 - 50 µg/kg	
152	0,03 - 0,7 ng/ml	
153	0.1-100ng/ml	
154	0,2–50 ng/ml	
155	0,5–10 ng/ml	
156	0-10 ppb	
157	0,4 -10 ng/ml	
158	0.75-25.00 ng/g	
159	0,07 - 73 ug/kg	
160	up to 50 ng/mL	
161	0.5–10.0 µg/L	
162	0,03–2,5 ug/l	
163	2,37 µg/L–4,745 µg/L–9,492 µg/L–23,73 µg/L	
164	1-20 ppb	
165	0,5-2 ng	
166	0.5 -10 µg/kg	
167	0,17-4,68 ng/ml	
168	1,1, 5,4, 10,8, 27,0, 54,0ng/ml	
169	0,2-2,5µg/l	
170	1-10 µg/kg	
171	1 - 36 µg/kg	
173	0.25-20µg/kg	

European Commission

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Abstract

A proficiency test was conducted with 68 laboratories from 17 EU Member States and four Third Countries. Test materials were one naturally contaminated "Ochratoxin A positive" and one "Ochratoxin A blank" capsicum material. The majority of laboratories chose to determine the ochratoxin A content by reverse-phase high-performance liquid-chromatography (RP-HPLC) with fluorescence detection against their own standard solutions as reference.

Applying the modified Horwitz equation according to Thompson as a basis for the target standard deviation (22% in the case of this proficiency test), 79% of the laboratories achieved z-scores of less than $|2|$. The results were evaluated further on the basis of the returned questionnaire that each participant received. The questions asked were focussed on the fact that future method development, if necessary, could be supported by comparison of the methodologies and method procedures applied.

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