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LABORATORY NUMBER: Available to participants from FAPAS SecureWeb

FAPAS® Proficiency Test 2260

Zearalenone in Animal Feed

October - December 2009

Report

Prepared on behalf of FAPAS® by

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0009 PT

SUMMARY

1. The test material for FAPAS® Proficiency Test 2260 was dispatched in October 2009. Each participant received an animal feed test material to be analysed for Zearalenone (ZON). In total, 83 sets of test material were distributed to participants in 30 countries. Of these, 77 participants, i.e. 93%, returned results for this analyte within the time-scale demanded by the Scheme.

2. The assigned value ($\hat{X}$) was calculated from the most appropriate measure of central tendency of participants' results [1, 2].

3. The target standard deviation (σ_p) was derived from the appropriate form of the Horwitz equation [3] and in conjunction with the assigned value ($\hat{X}$), was used to derive z-scores for participants' results. z-Scores are normally considered satisfactory if $|z| \leq 2$.

4. Results for this test are summarised as follows:

analyte	assigned value, $\hat{X}$ µg/kg	number of satisfactory scores $\|z\| \leq 2$	total number of scores	satisfactory %
ZON	129	59	76	78

5. Surplus test materials are available for sale, see APPENDIX III.

6. Whereas this Report has been produced in good faith and in accordance with best industry practice, neither the Food and Environment Research Agency nor the Secretary of State for Environment, Food and Rural Affairs accepts any liability whatsoever as to the application or use of the information contained therein.

CONTENTS

1. INTRODUCTION

1.1. Proficiency Testing

The demand for independent proof of competence from regulatory bodies and customers means that proficiency testing is relevant to all laboratories testing food and feed for quality and safety in every country. Hence, it is a requirement of accreditation to ISO 17025 [4] that the laboratory takes part in a proficiency testing scheme, if a suitable scheme exists. Further, for laboratories entrusted with the official control of food and feeds, Article 12 of EU Regulation (EC) 882/2004 [5] requires such laboratories to be assessed and accredited in accordance with ISO 17025, i.e. proficiency testing is a legal requirement for these laboratories. Thus, together with the use of validated methods, proficiency testing is an essential element of laboratory quality assurance.

The analysis of an external quality check sample as part of a laboratory's routine procedures provides objective standards for individual laboratories to perform against and permits them to compare their analytical results with those from other laboratories. Such standards and comparisons can go beyond the actual chemical analysis. For example, the ability to report results in specified units and within a given time scale are important aspects of quality. Hence, participants in FAPAS® who submit results after the closing date of a test are only included in the statistical evaluation if there are extenuating circumstances.

It is important to understand the statistical limitations of this external means of quality assessment when gauging the competence of a laboratory. The results of a typical chemical analysis will be normally distributed. That is to say, the majority of results will be centred on a mean value but, inevitably, some results will lie at the extremes of the distribution. The statistics of a normal distribution mean that about 95% of data points will lie between a z-score of –2 and +2. Performance in a FAPAS® proficiency test, therefore, is considered 'satisfactory' if a participant's z-score lies within this range. It follows that if a participant's z-score lies outside $|z| > 2$ there is about a 1 in 20 chance that their result is in fact an acceptable result from the extreme of the distribution. If a participant's z-score lies outside $|z| > 3$ the chance that their result is actually acceptable is only about 1 in 300.

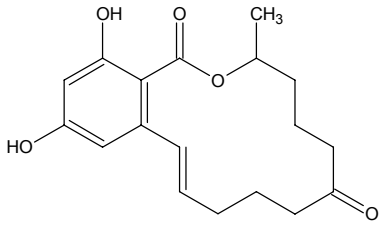
Full details of the FAPAS® proficiency testing scheme are available via our protocols [6, 7].

1.2. Fusarium Toxins: Zearalenone

Fusarium toxins, produced by moulds, are examples of toxic substances collectively known as mycotoxins. There are a huge number of mycotoxins, with a wide range of toxic effects. Since foodstuffs such as cereal crops, dried fruit, nuts, spices and fruit juices can be infected by moulds during their growth or storage, mycotoxins can be found as contaminants in these foods. They may be present in food without any obvious sign of fungal growth.

Certain *Fusarium* spp., which are common soil fungi, are probably the most prevalent toxin-producing fungi of the northern temperate regions. They are commonly found on cereals grown in Europe, Asia and America. These *Fusarium* spp. produce a number of different mycotoxins, known as tricothecenes (e.g. DON) and some other toxins (ZON and fumonisins) and are regulated within the European Union (EU) [8]. This new regulation came into full effect March 2007, it consolidates and replaces the Commission Regulation EC No. 856/2005 [9].

ZON is a nonsteroidal estrogenic mycotoxin. It has been implicated in numerous mycotoxicoses in farm animals, especially in pigs, and has been linked with early puberty in young girls in Eastern European countries [10].



Zearalenone

2. TEST MATERIAL

2.1. Preparation

The test material was prepared by a laboratory contracted to do so by FAPAS®.

The test material was prepared using 13 kg of chicken feed. A 1 kg sample of the animal feed was spiked with a solution containing ZON and left overnight to allow the solvent to evaporate.

The spiked animal feed was then passed through a centrifugal mill fitted with a 1mm screen, this was then followed by the remaining unspiked animal feed.

The spiked animal feed sample was blended with a 1 kg portion of the unspiked animal feed using a bowl mixer, for 10 to 15 minutes. Further portions of the unspiked animal feed were added, stepwise, with mixing for 10 to 15 minutes after each addition. The final bulk sample was then mixed overnight.

The resulting test material was then dispensed into aluminium foil laminate sachets to create individual sub-samples (≈55 g) that were stored at -20°C prior to distribution.

2.2. Homogeneity

Ten randomly selected test materials were analysed in duplicate for ZON by a laboratory contracted to do so by FAPAS®. The results, together with their statistical evaluation [11], are given in APPENDIX I.

The statistical tests initially check the data for any widely discrepant pairs using Cochran's test. If found, such data are removed. Thereafter the remaining data are subject to analysis of variance (ANOVA) to estimate the sampling and analytical variances. These data show sufficient homogeneity and are not included in the subsequent calculation of the assigned value.

2.3. Distribution

Each participant received an individually numbered animal feed test material packed in a padded envelope together with a covering letter, electronic submission instructions and

results form (for participants without internet access). The dispatch date was 28 October 2009.

3. RESULTS

Participants were required to report their data in µg/kg, corrected for recovery, together with their percentage recovery or details of any other correction factor used. Results were submitted by 77 participants before the closing date for this test, 2 December 2009.

Each participant was given a laboratory number, assigned in order of receipt of results. The reported ZON concentrations are given in Table 1.

The analytical methods used by each participant are summarised in APPENDIX II.

4. STATISTICAL EVALUATION OF RESULTS

The object of the statistical procedure employed is to obtain a simple and transparent result, which the participant and other interested parties can readily appreciate. The procedure follows that recommended in the IUPAC/ISO/AOAC International Harmonised Protocol for the Proficiency Testing of Analytical Chemistry Laboratories [12].

4.1. Calculation of the Assigned Value, $\hat{X}$

The assigned value, $\hat{X}$, i.e. the best estimate of the true concentration of ZON, was set as the consensus of the results submitted by participants [1, 2]. The procedure used to derive this consensus involved where necessary:

- Removing non valid data, i.e.:
  - i) results from participants not quoting a percentage recovery or stating 'not corrected',
  - ii) semi-quantitative results,
  - iii) results reported as 10x greater or less than the majority of submitted results (as these were considered to be reporting errors).
- Considering the normality (Kolmogorov-Smirnov test), or otherwise, of the distribution of the selected results.
- Minimising the influence of outliers by the use of a robust statistical procedure to derive the mean [2].
- Assessing the standard uncertainty (u) of the robust mean:

$$u = \frac{\hat{\sigma}}{\sqrt{n}}$$

where $\hat{\sigma}$ = the robust standard deviation [2]
Note: this is NOT the target standard deviation for the test (σ_p)

and n = the number of data points used to calculate the robust mean.

This procedure was straightforward for ZON. The robust mean was deemed the best measure of central tendency and used to set the assigned value. The assigned value together with u, n and $\hat{\sigma}$ are shown in Table 2.

4.2. Target Standard Deviation for the Test, σ_p

The value of σ_p determines the limits of satisfactory performance in a FAPAS® proficiency test. It is set at a value that reflects best practice for the analyses in question. The standard deviation of reproducibility found in collaborative trials is generally considered an appropriate indicator of the best agreement that can be obtained between laboratories. However, not all analyses have been characterised in this manner. In such cases, the predictive models of the appropriate form of the Horwitz equation [3] are valuable indicators of best practice.

For ZON, σ_p was derived from the appropriate form of the Horwitz equation [3]. This equation predicts a standard deviation from a given concentration, c, and requires c to be expressed as a dimensionless mass ratio, e.g. 1 ppm ≡ 10^{-6} or % ≡ 10^{-2}. It follows therefore that to express the dimensionless standard deviation predicted by the equation in the original concentration units it must be divided by the relevant mass ratio.

i) for analyte concentrations <120 ppb

$$\sigma_p = \frac{0.22c}{mr}$$

ii) for analyte concentrations ≥120 ppb and ≤13.8%

$$\sigma_p = \frac{0.02c^{0.8495}}{mr}$$

iii) for analyte concentrations >13.8%

$$\sigma_p = \frac{0.01c^{0.5}}{mr}$$

where, in all three cases, c = concentration, i.e. the assigned value, $\hat{X}$, expressed as a dimensionless mass ratio, e.g. 1 ppm ≡ 10^{-6} or % ≡ 10^{-2}

and mr = dimensionless mass ratio, e.g. 1 ppm ≡ 10^{-6} or % ≡ 10^{-2}.

The value of σ_p used to calculate z-scores from the reported results in this test is given in Table 2.

4.3. Individual z-Scores

Participants' z-scores were calculated as:

$$z = \frac{(x - \hat{X})}{\sigma_p}$$

where x = the participant's reported result,
$\hat{X}$ = the assigned value,
and σ_p = the target standard deviation.

Participants' z-scores for ZON are given in Table 1 and shown as a histogram in Figure 1.

It is possible for the z-scores published in this report to differ slightly from the z-score that can be calculated using the formula given above. These differences arise from the necessary rounding of the actual assigned values and target standard deviations prior to their publication in Table 2.

The number and percentage of z-scores in the satisfactory range, $|z| \leq 2$, for ZON are given in Table 3.

5. REFERENCES

1 Lowthian, P.J. and Thompson, M., 2002, Bump-hunting for the proficiency tester – searching for multimodality, *Analyst*, **127**, 1359-1364.

2 Analytical Methods Committee, 1989, Robust Statistics – How not to reject outliers Part 1. Basic Concepts, *Analyst*, **114**, 1693-1697.

3 Thompson, M., 2000, Recent trends in inter-laboratory precision at ppb and sub-ppb concentrations in relation to fitness for purpose criteria in proficiency testing, *Analyst*, **125**, 385-386.

4 ISO/IEC 17025:2005, General requirements for the competence of testing and calibration laboratories.

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6 FAPAS®, 2009, Protocol for Proficiency Testing Schemes, Part 1 – Common Principles, Revision 2009, Version 1, Issued November 2009.

7 FAPAS®, 2009, Protocol for Proficiency Testing Schemes, Part 2 – FAPAS®, Revision 2009, Version 1, Issued November 2009.

8 Regulation (EC) No 1881/2006 of 19 December 2006 setting maximum levels for certain contaminants in foodstuffs, *Official Journal* **L 364**, 20/12/2006, 0005-0024.

9 Regulation (EC) 856/2005 of 6 June 2005 amending Regulation (EC) No. 466/2001 as regards *Fusarium* toxins, *Official Journal* **L 143**, 07/06/2005, 3-8.

10 Szuetz, P., Mesterhazy, A., Falkay, G.Y., Bartok. T., 1997, Early telearche symptoms in children and their relations to zearalenone contamination in foodstuffs, *Cereals Research Communications*, **25**, 429-436.

11 Fearn, T. and Thompson, M., 2001, A new test for sufficient homogeneity, *Analyst*, **126**, 1414-1417.

12 Thompson, M., Ellison, S.L.R. and Wood, R., 2006, The International Harmonised Protocol for the Proficiency Testing of Analytical Chemistry Laboratories, *Pure Appl. Chem.*, **78**, No. 1, 145-196.

Table 1: Results and z-Scores for ZON in Animal Feed Test Material

laboratory number	analyte		
	ZON		
	assigned value	129	µg/kg
	result µg/kg	recovery %	z-score
001	95.23	88	-1.2
002	158.1	101	1.0
003	99.8	100	-1.0
004	132.8	114	0.1
005	77.4	uncorr	-1.8
006	113	83	-0.6
007	71.9	95	-2.0
008	336.52	uncorr	**7.4**
009	116	85	-0.5
010	156.43	107.81	1.0
011	80.37	95.19	-1.7
012	175	100.0	1.6
013	>100(180)		
014	122	89	-0.3
015	155.6	98.6	0.9
016	157.03	uncorr	1.0
017	52	96	**-2.7**
018	151	100	0.8
019	125	104	-0.1
020	126	85	-0.1
021	183	96	1.9
022	109.46	99.4	-0.7
023	201	N.A	**2.6**
024	93.5	93.5	-1.3
025	139.9	75	0.4
026	133	74.5	0.1

uncorr = participant did not state recovery or stated not corrected for recovery
z-Scores outside the satisfactory range i.e. |z| >2 are shown in **bold.**

Table 1 (Continued): Results and z-Scores for ZON in Animal Feed Test Material

laboratory number	analyte		
	ZON		
	assigned value	129	µg/kg
	result µg/kg	recovery %	z-score
027	100.57	75	-1.0
028	177	100	1.7
029	152	104	0.8
030	81.33	100	-1.7
031	128	uncorr	0.0
032	112	83	-0.6
033	140	105	0.4
034	57	uncorr	**-2.6**
035	130.25	uncorr	0.0
036	150.0	100	0.7
037	210.8	63.2	**2.9**
038	101.4	83	-1.0
039	103	83	-0.9
040	186	104	2.0
041	128	90	0.0
042	73.3	97.6	-2.0
043	145.2	100	0.6
044	89.21	89.3	-1.4
045	276	uncorr	**5.2**
046	156	110	1.0
047	262.2	100	**4.7**
048	140.0	100	0.4
049	112	100	-0.6
050	57.17	>90	**-2.6**
051	115	90	-0.5
052	126.9	80	-0.1

uncorr = participant did not state recovery or stated not corrected for recovery
z-Scores outside the satisfactory range i.e. |z| >2 are shown in **bold.**

Table 1 (Continued): Results and z-Scores for ZON in Animal Feed Test Material

laboratory number	analyte		
	ZON		
	assigned value	129	µg/kg
	result µg/kg	recovery %	z-score
053	165.53	74.13	1.3
054	91.79	72.11	-1.3
055	286.89	uncorr	**5.6**
056	90.70	100	-1.4
057	162	91	1.2
058	61	78.2	**-2.4**
059	895	uncorr	**27.3**
060	132.9	uncorr	0.1
061	0.116	79.5	**-4.6**
062	50.3	92	**-2.8**
063	245	63	**4.1**
064	95.82	uncorr	-1.2
065	168.3	101.91%	1.4
066	114.7	78.8	-0.5
067	188	uncorr	**2.1**
068	187	uncorr	**2.1**
069	162	96	1.2
070	98	80	-1.1
071	183.2	105	1.9
072	211.6	86	**2.9**
073	122	80	-0.3
074	127.5	83.9	-0.1
075	159.8	92.3	1.1
076	158.5	80	1.0
077	110.4	79.0	-0.7

uncorr = participant did not state recovery or stated not corrected for recovery
z-Scores outside the satisfactory range i.e. |z| >2 are shown in **bold.**

Table 2: Assigned Values and Target Standard Deviations

analyte	assigned value, µg/kg				target standard deviation	
	data points, *n*	robust mean, $\hat{X}$	robust standard deviation, $\hat{\sigma}$	uncertainty, *u*	derived from	σ_p
ZON	61	129	41.6	5.33	Horwitz*	28.1

* See page 7 for the appropriate form of the Horwitz equation.

Table 3: Number and Percentage of Satisfactory z-Scores

analyte	number of satisfactory scores $\|z\| \leq 2$	total number of scores	satisfactory %
ZON	59	76	78

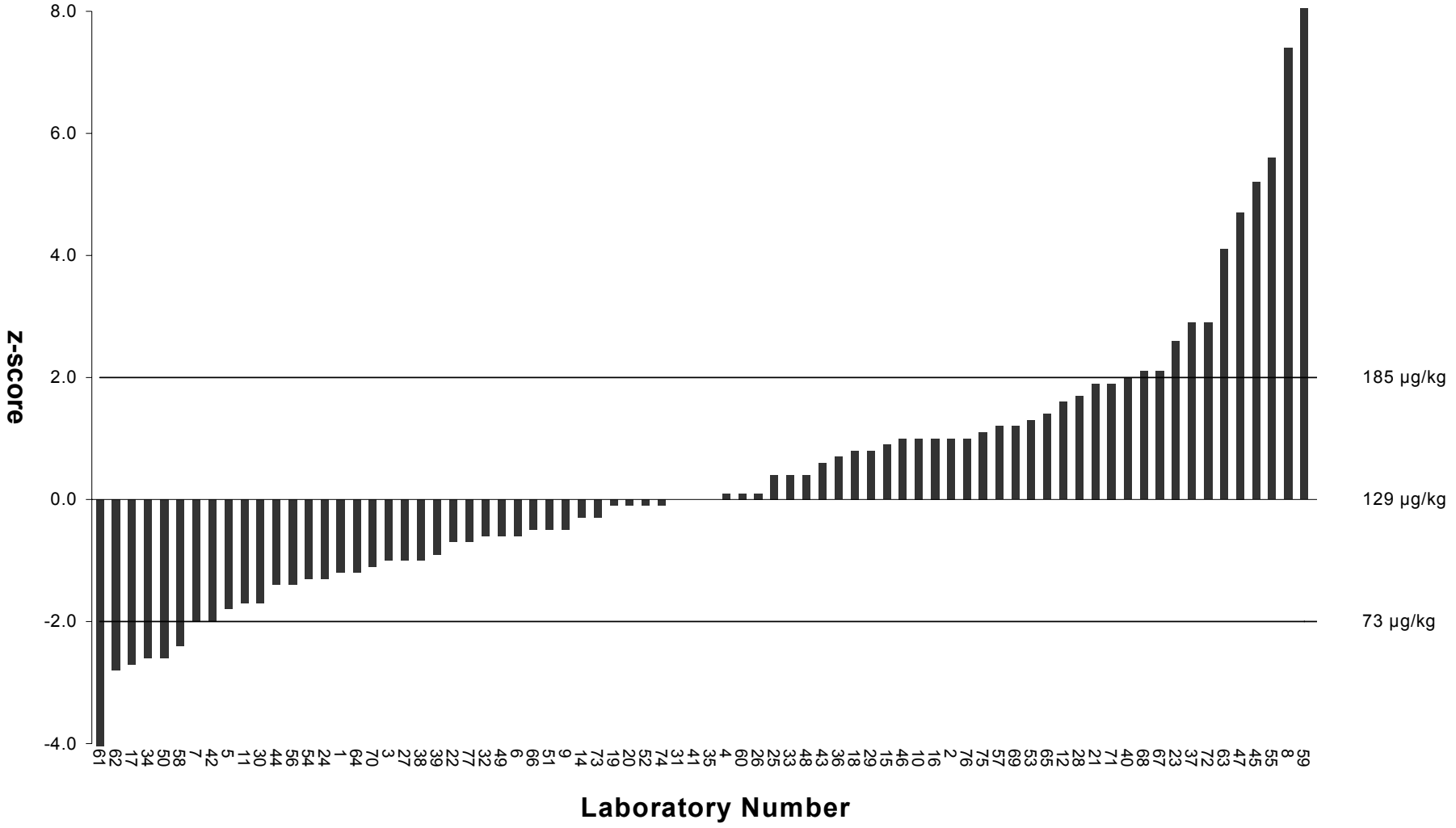

Figure 1: z-Scores for ZON (129 µg/kg) in Animal Feed Test Material

APPENDIX I: Homogeneity Data for Animal Feed Test Material

sample identity		analyte	
		ZON µg/kg	
		replicate 1	replicate 2
1		138	145
2	**c**	52.8	119
3		85.9	118
4		146	132
5		142	137
6		96.7	111
7		127	142
8		127	139
9		147	141
10		140	111
mean		129	
n		18	
origin of target sd (σ_p)		Horwitz *	
abs. target sd (σ_p)		28.1	
abs. target sd as RSD%		21.8	
s_{an}		12.2	
s_{sam}^2		177	
σ_{all}^2		71.1	
critical		303	
s_{sam}^2<*critical?*		**ACCEPT**	

c = pair removed as Cochrans outlier
* see page 7 for appropriate form of the Horwitz equation.

APPENDIX II: Analytical Methods Used by Participants

Notes

1) Participants' methods are tabulated according to the information submitted electronically, but some responses may have been combined or edited for clarity.
2) Participants with performance outside the satisfactory range, i.e. |z| >2, are no longer shown in bold in this section but their z-scores are now shown in bold in Table 1.

Accredited Method Used	laboratory number
yes	002 005 014 015 017 019 023 025 026 027 028 029 031 032 033 036 038 039 040 041 044 046 047 048 049 051 054 055 057 061 063 064 065 067 069 070 072 074 076
no	001 003 007 008 009 012 013 016 020 021 022 024 034 035 037 042 043 045 050 052 053 056 058 059 060 066 071 073 075

Reference	laboratory number
AOAC Official Methods	025
AOAC Official Method 970.45	072
AOCS 2002	044
Application Note–Cereals–Acetonitrile Extraction Method for Zearalenone, Ref. No. A1–RP91.V3, July 2005, R-Biopharm Rhône Ltd.	065
DIN 32645	031
EN 2009 15792 1-15p	001
GIPSA	055
IFU Standard	061
J. Chromatography A 2005 1062 209-216	018
Manufacturer's Instructions	007 008 047
Practical Standards Colombia NTC 4881 Method of Analysis of Zearalenona 2000	076
PAFQ 287 2008	073
R-Biopharm 2006	048
R-Biopharm Rhone Application note	042

Reference (continued)	laboratory number
RIDASCREEN Zearalenon	053
Validation report No B5-1000/02/000566 by DG SANCO and JRC 2005	032
Varian Application Bond Elut Mycotoxin 2006 Application Note 000295 1-4	049
VDLUFA	051 057

Sample Weight (g)	laboratory number
≥1 - <2	029 069
≥2 - <5	005 031 035 058 059
≥5 - <10	012 021 025 028 033 040 044 045 046 047 048 053 057 064
≥10 - <25	001 002 007 008 009 014 016 018 023 026 032 034 038 043 049 051 061 063 066 067 070
≥25 - <50	003 013 015 019 022 024 036 039 041 050 052 054 065 071 072 074 075 076
≥50	027 042 073

Extraction Solvent	laboratory number
acetic acid	009 018
acetonitrile	002 003 005 007 009 011 012 014 015 017 019 022 025 028 031 033 036 038 039 041 043 047 049 051 056 057 058 061 063 064 065 067 069 070 071 073 074 075 076
hexane	072
methanol	001 008 013 016 020 021 023 024 026 027 029 032 035 037 040 041 044 045 046 048 050 052 053 054 055 059 063 066 072
phosphate buffer	042
sodium chloride solution	042 072
water	001 002 003 005 007 009 011 012 013 014 015 017 018 019 020 021 022 023 024 025 026 027 028 031 032 033 034 036 038 039 040 043 044 045 047 049 050 051 054 057 058 061 063 064 065 066 067 069 070 071 073 074 075 076

Extraction Procedure	laboratory number
add NaCl	014 025 026 037 038 052 054 064 069 073
blend / homogenise with solvent	005 007 008 011 015 016 019 020 022 025 027 031 034 036 038 039 042 050 051 052 054 065 071 073 076
maceration / homogenisation	003
shake with solvent	002 008 009 012 017 021 023 024 028 029 032 033 035 040 041 043 045 046 047 048 049 051 053 058 059 063 064 066 067 069 070 072 074 075
shaking	001 018 055 057 061
sonicate/ultrasonic bath	013 036 067 070
Ultra Turrax	014 024 057
ultrasonic extraction	020
vortex mix	044

Extraction Type	laboratory number
single	001 002 003 005 008 009 011 012 013 014 015 016 017 018 019 020 021 022 023 024 025 026 027 028 029 031 032 033 034 036 037 038 039 040 042 043 045 046 047 048 049 050 052 053 054 055 056 057 058 059 061 063 064 065 066 069 070 071 072 073 076
multiple	007 035 041 044 051 074 075

Extraction Time	laboratory number
30 seconds	038
1 minute	055
2 minutes	003 042 050 057
3 minutes	007 015 016 023 039 040 048 051 053 054
5 minutes	021 044 045 066 071
6 minutes	073
10 minutes	034 035 070
15 minutes	026 059 067
20 minutes	036 063
30 minutes	002 020 029 037 041 061 072

Extraction Time (continued)	laboratory number
1 hour	001 008 009 018 024 032 033 047 049 056 058 069 075 076
90 minutes	028 074

Sample Work Up	laboratory number
centrifuge	012 029 035 044 047 051 056 057 058 065 067 069
defatted with hexane	072
dilute	002 003 008 022 023 026 028 031 035 038 039 040 043 050 054 055 063 070
dry over Na_2SO_4	072
evaporate	033 054 072 074 075
filter	001 002 007 008 009 011 014 015 016 017 018 019 020 021 022 023 024 025 026 027 028 031 032 033 036 037 038 040 041 042 043 045 046 048 049 050 051 052 053 054 061 063 064 066 067 069 070 071 074 075 076
pH adjustment	007 008 023 034 051
none	073

Sample Clean-up by Immunoaffinity Column (Brand)	laboratory number
NeoColumn Zearalenone	050
Neogene	024
R-Biopharm Rhone	002 007 011 013 017 022 028 034 036 042 043 047 048 051 052 057 061 065
Romer Labs	020 074 075
VICAM	001 003 005 014 025 026 027 031 032 038 054 063 064 070 073
ZearaStar (Romer Labs)	019

Sample Clean-up by SPE	laboratory number
Bond Elut Varian	015
Florisil	033
Micotox	076
Romer Labs	009 012 013 018 047 048 067 071 074 075
Varian Bond Elut Mycotoxin	049

Mycotoxin Determination	laboratory number
ELISA	012 013 021 023 035 037 040 044 045 046 048 053 055 056 066
fluorometric	052
GC	028 059
HPLC	001 002 003 007 009 014 015 017 018 019 020 022 024 025 026 027 029 031 032 033 034 036 038 039 041 042 043 049 050 051 057 058 061 063 064 065 067 069 070 071 073 074 076
LC-MS	054
LC-MS/MS	047 075
TLC/HPTLC	072

HPLC Pre Column Derivatisation	laboratory number
none	001 002 007 019 020 022 026 027 028 029 032 034 038 042 043 049 050 051 054 057 058 061 063 064 073 074 075

HPLC Injection Volume (µL)	laboratory number
<5	069
≥5 - <10	029 047 058 067
≥10 - <25	002 005 009 012 014 015 018 026 039 041 042 049 054 057 071
≥25 - <50	003 017 019 033 075
≥50 - <100	024 025 027 034 036 064 070 076
≥100 - <150	007 011 020 022 028 031 032 038 043 050 051 061 063 065
≥150	001 073 074

HPLC Column Packing	laboratory number
C 6-Phenyl 110 A°	047
C18	001 003 005 007 009 011 012 014 015 017 018 019 020 022 024 025 026 027 028 029 031 032 033 036 038 039 041 042 043 049 050 051 054 057 058 061 064 065 067 069 070 071 073 074 075 076
C8	002
endcapped	002 007 009 014 019 028 031 065
non-endcapped	026
ODS-3V	034
Synergi Polar (Phenomenex)	063

HPLC Column Temperature (°C)	laboratory number
ambient	007 015 025 027 029 031 032 034 036 038 043 051 057 061 063 065 070 073 074 075
>ambient - <50	001 002 009 011 012 014 017 018 019 020 022 024 026 028 033 039 041 042 047 049 050 058 064 067 069 071 076
≥50	003 054

Mobile Phase Components	laboratory number
acetate	028
acetic acid	002 003 007 011 027 029 038 039 050 051 061 065
acetonitrile	002 003 007 011 015 017 019 022 025 026 036 038 039 041 042 047 050 051 057 058 061 063 064 065 070 073 074 076
ammonium acetate	024 047
formic acid	041 058 067 075
methanol	001 005 009 012 014 018 022 024 025 026 027 028 031 032 033 043 049 054 063 064 067 069 071 073 074
phosphoric acid	076
water	001 002 003 005 007 009 011 012 014 015 017 018 019 020 022 025 026 027 028 031 032 033 034 036 038 042 043 049 050 051 054 057 061 063 064 065 067 069 070 071 073 074 076

Mobile Phase Flow Rate (mL/min)	laboratory number
<0.25	015 039
≥0.25 - <0.75	002 005 009 017 018 019 020 024 028 029 031 041 047 049 054 057 058 064 069
≥0.75 - <1.25	001 003 007 011 012 014 022 025 026 027 032 033 034 036 038 042 043 050 051 063 065 067 070 071 073 076
≥1.75 - <2.25	061 074 075

Post Column Mobile Phase Flow Rate (mL/min)	laboratory number
<0.25	015
≥0.25 - <0.75	014 024 029 047 071
≥0.75	022 051

HPLC Detector Type	laboratory number
fluorescence	001 002 003 007 011 012 014 017 019 020 022 025 026 027 031 032 036 038 042 043 050 051 057 061 063 064 065 070 071 073 074 075 076
MS	009 054
MS-MS	005 015 018 024 028 029 033 039 041 047 049 058 067 069
UV/Vis	034

GC Injection Volume (µL)	laboratory number
≥5 - <10	002
≥10	028 057

GC Column Packing	laboratory number
50% methyl 50% phenyl polysiloxane	028

Column Temp Programme	laboratory number
isothermal	033 054
gradient	028 044 050 073

Carrier Gas	laboratory number
helium	028

GC Flow Rate	laboratory number
≥0.75 - <1.25	028

GC Detector	laboratory number
MS	028

Source of Standards	laboratory number
BCR	046
Biopure	009 018
Micotox	076
Promochem	031
Provided by Manufacturer	021 035
R-Biopharm Rhone	011 013 022 029 040 042 044 047 048 053 065
Romer Labs	007 016 023 051 067
Sigma/Aldrich	001 002 003 012 014 017 020 025 026 032 033 038 039 041 049 057 063 065 071 072
Supelco	027 050 054 058 066 070

APPENDIX III: FAPAS® SecureWeb, Reports and Protocol

1. FAPAS® SECUREWEB

Access to the secure area of our web site is only available to participants in our proficiency tests. Please contact us if you require a UserID and Password. FAPAS® SecureWeb allows participants to:

- Obtain their laboratory numbers for the proficiency tests in which they have participated.
- View the results they submitted in past and current proficiency tests.
- Submit their results and methods for current tests.
- Review future tests they have ordered.
- Order proficiency tests and quality control materials, ***including surplus test materials from the batch used in this proficiency test***.
- Freely download copies of reports, in Acrobat PDF format, of proficiency tests in which they have participated.

2. REPORTS

The Acrobat PDF version of this report is available to all participants as a free download from FAPAS® SecureWeb.

A printed and bound version of this report is priced £35 if ordered at the same time as the proficiency test or £50 if ordered subsequently.

3. PROTOCOL

The Protocols [6,7] set out how FAPAS® is organised. They give full details of the statistical procedures used and includes worked examples. Copies can be downloaded from our website.

5. CONTACT DETAILS

Participants with any comments or concerns about this proficiency test should contact:

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The Food and Environment Research Agency is an ISO 9001 certified organisation.