FAPAS® Proficiency Test Report 17107

Ochratoxin A in Wheat Flour

January-February 2012

Prepared and authorised on behalf of FAPAS by

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0009 PT

PARTICIPANT LABORATORY NUMBER

Participants can log in to FAPAS SecureWeb at any time to obtain their laboratory number for this proficiency test.

Laboratory numbers are displayed in SecureWeb next to the download link for this report.

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SUMMARY

1. The test material for FAPAS® proficiency test 17107 was dispatched in January 2012. Each participant received a wheat flour test material to be analysed for ochratoxin A (OTA).
2. An assigned value (x_a) was determined for OTA and in conjunction with the standard deviation for proficiency (σ_p) was used to calculate a z-score for each result.
3. Results for this proficiency test are summarised as follows:

analyte	assigned value, x_a µg/kg	number of scores, |z| ≤2	total number of scores	% |z| ≤2
OTA	3.67	67	74	91

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

Proficiency testing aims to provide an independent assessment of the competence of participating laboratories. Together with the use of validated methods, proficiency testing is an essential element of laboratory quality assurance.

Further details of the FAPAS® proficiency testing scheme are available in our protocols [2, 3].

2. TEST MATERIAL

2.1. Preparation

Preparation of the samples for this proficiency test was sub-contracted to a laboratory meeting the quality requirements of the scheme's accreditation to ISO 17043.

The wheat flour was procured from a retail source and was found to contain no detectable OTA. The sample was then spiked and mixed.

Samples were stored at -20°C until dispatch.

2.2. Homogeneity

To test for homogeneity, randomly selected test materials were analysed in duplicate. Testing was sub-contracted to a laboratory meeting the quality requirements of the scheme's accreditation to ISO 17043.

These data showed sufficient homogeneity and were not included in the subsequent calculation of the assigned value.

2.3. Dispatch

The start date was 10 January 2012. Test materials were sent to 82 participants.

3. RESULTS

The instructions for reporting results were as follows:

- Determine the level of ochratoxin A, present in the test material, in **µg/kg, as received, corrected for recovery.**

Results were submitted by 75 participants (91%) before the closing date for this test, 21 February 2012.

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

Participants' comments are given in Table 2.

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

4. STATISTICAL EVALUATION OF RESULTS

The results submitted by participants were statistically analysed in order to provide an assigned value for OTA. The assigned value was then used in combination with the standard deviation for proficiency, σ_p, to calculate a z-score for each result. The procedure follows that recommended in the IUPAC International Harmonised Protocol for the Proficiency Testing of Analytical Chemistry Laboratories [4].

Further details on the procedure followed can be found in the relevant protocols [2, 3].

4.1. Calculation of the Assigned Value, x_a

The assigned value, x_a, for OTA was derived from the consensus of the results submitted by participants.

The following results were excluded from the calculation of the assigned value:

i) non numerical results i.e. qualitative or semi-quantitative results,

ii) results uncorrected for recovery.

For OTA, this procedure was straightforward and the robust mean was chosen as the assigned value.

The assigned value for OTA are shown in Table 3.

4.2. Standard Deviation for Proficiency, σ_p

The standard deviation for proficiency, σ_p, was set at a value that reflects best practice for the analyses in question.

For OTA, σ_p was derived from the appropriate form of the Horwitz equation [5].

The values for σ_p used to calculate z-scores from the reported results of this test are given in Table 3.

4.3. Individual z-Scores

Participants' z-scores were calculated as:

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

where x = the participant's reported result,
x_a = the assigned value
and σ_p = the standard deviation for proficiency.

Participants' z-scores for OTA are given in Table 1 and are 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 value and standard deviation for proficiency prior to their publication in Table 3.

The number and percentage of z-scores in the range $-2 \leq z \leq 2$ for OTA are given in Table 4.

5. ASSESSMENT OF SCORES

In normal circumstances, over time, about 95% of z-scores will lie in the range $-2 \leq z \leq 2$. Occasional scores in the range $2 < |z| < 3$ are to be expected, at a rate of 1 in 20. Whether or not such scores are of importance can only be decided by considering them in the context of the other scores obtained by that laboratory.

Scores where $|z| > 3$ are to be expected at a rate of about 1 in 300. Given this rarity, such z-scores very strongly indicate that the result is not fit-for-purpose and almost certainly requires investigation.

The consideration of a set or sequence of z-scores over time provides more useful information than a single z-score. Examples of suitable methods of comparison are provided in the IUPAC International Harmonised Protocol for the Proficiency Testing of Analytical Chemistry Laboratories [4].

6. REFERENCES

1 Adobe Certified Document Services http://www.adobe.com/security/partners_cds.html, accessed 24/05/2011

2 FAPAS, 2012, Protocol for Proficiency Testing Schemes, Part 1 – Common Principles, Version 3, Issued January 2012.

3 FAPAS, 2012, Protocol for Proficiency Testing Schemes, Part 2 – FAPAS®, Version 2, Issued January 2012.

4 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.

5 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.

Table 1: Results and z-Scores

laboratory number	analyte			laboratory number	analyte		
	OTA				OTA		
	assigned value	3.67	µg/kg		assigned value	3.67	µg/kg
	result µg/kg	recovery %	z-score		result µg/kg	recovery %	z-score
001	3.6	72	-0.1	029	3.8	100	0.2
002	2.92	100	-0.9	030	3.0	83	-0.8
003	3.02	79.02	-0.8	031	4.74	95.6	1.3
004	3.46	100	-0.3	032	2.98	85	-0.9
005	3.3	91.1	-0.5	033	1.62	60	**-2.5**
006	4.17	77	0.6	034	2.53	85	-1.4
007	3.32	98	-0.4	035	3.46	100	-0.3
008	2.717	100	-1.2	036	3.4	87.9	-0.3
009	4.64	70	1.2	037	3.62	90	-0.1
010	3.85	80	0.2	038	4.66	73	1.2
011	3.40	106	-0.3	039	4.13	87	0.6
012	3.37	76	-0.4	040	3.95	79.3	0.3
013	4.0	92	0.4	041	2.842	86.3	-1.0
014	4.02	98	0.4	042	3.6	85.5	-0.1
015	3.87	79.6	0.2	043	4.77	87.0	1.4
016	3.9	83	0.3	044	6.9	110	**4.0**
017	1.65	95.7	**-2.5**	045	3.99	80	0.4
018	5.36	80	**2.1**	046	2.95	100	-0.9
019	3.636	149.6	0.0	047	3.45	80.0	-0.3
020	3.70	100	0.0	048	5.07	68	1.7
021	4.51	90.4	1.0	049	1.9	106	**-2.2**
022	3.9	90	0.3	050	3.82	80	0.2
023	3.5	84	-0.2	051	3.894	85	0.3
024	3.95	89.4	0.3	052	3.62	90	-0.1
025	4.1	98.8	0.5	053	3.27	88	-0.5
026	5.2	100	1.9	054	3.39	uncorr	-0.3
027	2.88	88.26	-1.0	055	3.75	uncorr	0.1
028	2.93	97.64	-0.9	056	5.04	uncorr	1.7

uncorr = participant did not state recovery %
z-scores outside |z| >2 are shown in **bold,** see Section 5

Table 1 (continued): Results and z-Scores

laboratory number	analyte			laboratory number	analyte		
	OTA				OTA		
	assigned value	3.67	µg/kg		assigned value	3.67	µg/kg
	result µg/kg	recovery %	z-score		result µg/kg	recovery %	z-score
057	3.5	97	-0.2	067	3.87	83.9	0.2
058	1.92	60	**-2.2**	068	3.19	91	-0.6
059	3.6	80	-0.1	069	4.86	76	1.5
060	3.40	90.4	-0.3	070	2.0545	uncorr	-2.0
061	2.75	104.4	-1.1	071	0.95	uncorr	**-3.4**
062	4.2	95	0.7	072	3.32	77.30	-0.4
063	5.16	95	1.8	073	4.81	100	1.4
064	3.40	90	-0.3	074	3.8	99	0.2
065	3.3	100	-0.5	075	<2	>90	
066	4.16	93	0.6				

uncorr = participant did not state recovery %
z-scores outside |z| >2 are shown in **bold,** see Section 5

Table 2: Participants' Comments

participant number	comments
070	Result is not corrected for recovery.
071	VICAM OchraA

comments are as submitted by participants

Table 3: Assigned Values and Standard Deviations for Proficiency

analyte	data points, n	assigned value, x_a, µg/kg	uncertainty, u	standard deviation for proficiency, σ_p, µg/kg	
OTA	69	3.67	0.0901	Horwitz [5]	0.808

Table 4: Number and Percentage of z-Scores where |z| ≤2

analyte	number of scores where |z| ≤2	total number of scores	% |z| ≤2
OTA	67	74	91

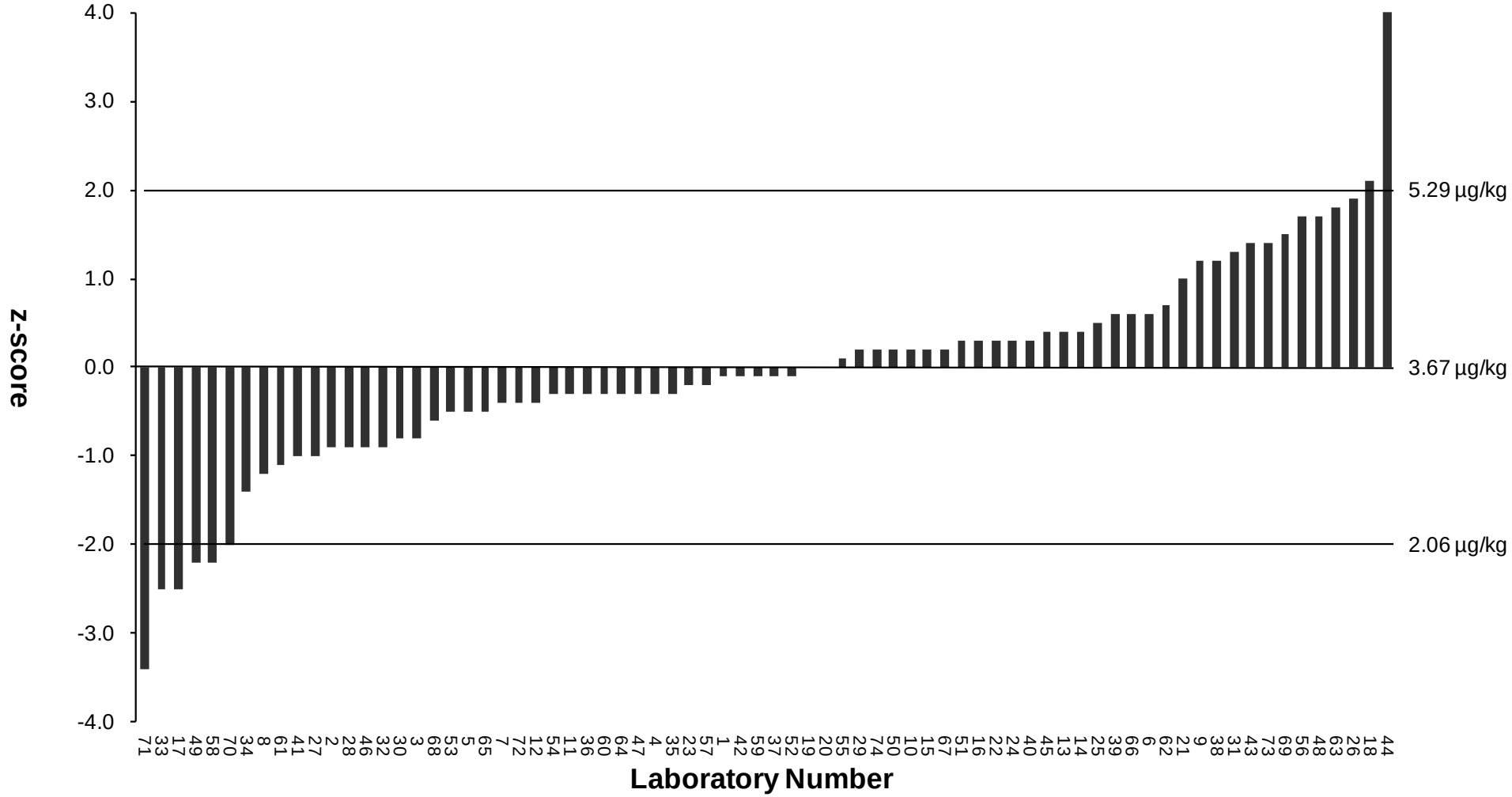

Figure 1: z-Scores for OTA

APPENDIX I: Analytical Methods Used by Participants

Methods are tabulated according to the information supplied by participants, but some responses may have been combined or edited for clarity.

Accredited Method Used	laboratory number
yes	001 002 004 005 006 007 009 013 017 019 020 021 022 024 025 026 027 029 032 033 034 035 036 037 038 039 042 045 046 048 050 051 052 053 054 057 058 059 060 064 066 067 068 072 074
no	008 011 012 015 016 028 031 040 043 044 047 049 055 056 061 069 070 075

Reference	laboratory number
AOAC Official Methods	006
AOAC Official Methods 2000.03	059
AOAC Official Methods 2005	043
AOAC Official Methods 2010 OMA 49.6.04	039
CCFRA Methods Manual	001
CEN 2005	017
Cereal–Ochratoxin A Extraction Method, Ref. No. A9–P14.V3, July 2005, R-Biopharm Rhône Ltd. 2005 A9 14	027
EN	002
EN 2003 14132: HPLC-FL	068
EN 2003 14132	013
EN 2009	021
EN 2009 14132	024
Food Additives and Contaminant 2008 25 472-489	046
Health Canada 2008 BFCL-035	025
In-house Method	005 022 033 035 061 064 074
ISO	019 042
ISO 2006	045
J Agric Food Chem 2010 Jul 14;58(13) 7510-7519	044
J. Agric. Food Chem. 2005 53 8904-8910	060
J. Chromatography 1992 603 285-289	036
KFDA 2011 10.6.1.5 10-6-10	053
LC-Tech 2011	037

Reference (continued)	laboratory number
Manufacturer's Instructions	007 020 072
Ochraprep, R-Biopharm, Version: P14/V13/26.10.10 1-6: 2010	032
MIP Ager Ocra 2011 Rev6 2011	029
R-Biopharm Ref. No: A1-P14.V3 2005	028
R-Biopharm Rhone Ref N: A1-P14.V3 2005 84 1818-1827	067
R-Biopharm: ELISA procedure	056
RC Mass. Spect. 2006, (20) 2649-2659 2006 20 2649-2659	057
Research Report of Animal Feed 2006 31 109-117	016
Rhone Diag. Appl. Notes	054
Vicam Ochra Test 1999 39	050
Waters Company Application Note	011

Sample Weight (g)	laboratory number
≥1 - <2	058
≥2 - <5	008 011 056
≥5 - <10	004 016 020 022 025 035 040 042 043 044 045 046 047 049 055 057 060 070
≥10 - <25	001 006 007 012 013 021 024 027 028 032 033 036 037 051 052 061 063 064 068 069 075
≥25 - <50	002 005 015 019 031 038 039 050 053 054 066 067 074
≥50	009 017 029 034 059 072

Extraction Solvent Components	laboratory number
acetic acid	016 031 057
acetonitrile	002 004 005 006 011 012 016 017 019 021 022 024 025 029 031 032 034 035 038 039 040 043 044 045 046 047 048 049 050 051 052 053 057 058 059 063 066 069 072 074
acidic acid	046
chloroform	015
formic acid	022 040 049

Extraction Solvent Components (continued)	laboratory number
methanol	009 013 025 033 036 037 042 048 059 060 069 070 075
NaHCO3	055 061
phosphate buffer	001 020 036
phosphoric acid	015
sodium bicarbonate	007 008 027 028 054 064 068
sodium carbonate	067
sodium chloride solution	013
Sodium hydrogen carbonate buffer pH 8.1	056
water	001 002 004 005 006 009 011 013 015 016 017 019 021 022 024 025 027 029 031 032 033 034 035 037 038 039 040 042 043 048 049 050 052 054 057 059 061 063 064 066 067 068 069 070 072 075

Extraction Procedure	laboratory number
add filter aid	015 032
add NaCl	052
blend/homogenise with solvent	001 009 017 027 028 029 032 033 034 039 043 047 048 050 052 054 064 066 067 069 072
citrate buffer	049
maceration/homogenisation	074
Quechers	044
shake with solvent	004 005 012 013 015 016 019 022 024 025 031 035 051 053 056 061 075
shaking	002 008 020 021 038 046 055 057 060 070
sonicate/ultrasonic bath	022 029 058 060
Ultra Turrax	006 007 037 042 068
ultrasonic extraction	057
vortex mix	040 059

Extraction Type	laboratory number
single	001 002 004 005 006 007 008 009 012 013 015 016 017 020 021 022 024 025 027 029 031 032 033 034 035 036 038 039 040 042 043 045 046 047 049 050 051 052 053 054 055 057 058 059 060 061 064 066 067 068 069 070 072 074 075
multiple	019 037 044 056

Sample Work Up	laboratory number
back-extraction	028
centrifuge	001 002 008 011 015 016 020 021 027 028 031 046 049 055 057 059 061 066 068 070 075
defatted with hexane	022 040
dilute	001 004 005 007 011 013 021 024 032 035 038 042 047 050 052 066 069
dry over Na2SO4	046
evaporate	015 049
filter	002 004 005 006 009 012 013 017 019 024 028 029 032 034 035 037 038 039 042 043 045 047 049 050 051 052 053 054 056 059 064 067 069 072 074
Ultra Turrax	017 033 067
none	025 044 060

Sample Clean-up by Immunoaffinity Column	laboratory number
Filter through 0.22 µm syringe tip filter	033
LC-Tech	037 042
Neogen	004 043
Quechers	044
R-Biopharm Rhone	001 002 005 006 007 017 021 025 027 029 032 034 038 048 050 054 055 064 066 067 072 074
Romer Labs	019 024 068
Vicam	009 013 028 036 039 045 051 052 053 059 061 069 070

Sample Clean-up by SPE	laboratory number
Baker	067
C18, PSA and NH2	049
IAC	013
Multi sep#229	016
Romer Labs	012 031 063
silica	015
Vicam	059

Mycotoxin Determination	laboratory number
ELISA	008 020 055 070 075
HPLC	001 002 004 005 006 007 009 011 012 013 015 016 017 019 021 022 024 025 027 028 029 031 032 034 035 036 037 038 039 040 042 043 044 045 046 047 048 050 051 052 053 054 057 058 059 060 061 063 064 066 067 068 069 072 074
UPLC-MS/MS	033 049

HPLC Injection Volume (µL)	laboratory number
<5	040
≥5 - <10	011 015 025 033 044 047 049 057
≥10 - <25	005 016 031 046 060 061 063
≥25 - <50	012 019 022 053 058
≥50 - <100	009 013 029 032 039 042 043 051 059
≥100 - <150	001 002 004 006 017 021 024 027 028 034 035 036 038 050 052 054 064 067 068 072 074
≥150	007 037 066 069

HPLC Column Packing	laboratory number
C12	069
C18	004 005 006 007 009 011 012 013 015 016 017 019 021 024 027 028 029 031 032 034 035 036 037 039 040 042 043 044 046 049 050 051 052 053 054 057 058 059 061 063 064 066 067 068 072 074

HPLC Column Packing (continued)	laboratory number
C8	022 025 047
endcapped	005 007 015 027 031 068
Hypersil ODS Thermo	045
lichrospher100rp18	038
non-endcapped	004
ODS(20)	001
PFP	060
XDB-C18	002

HPLC Column Temperature (°C)	laboratory number
ambient	001 007 009 011 017 024 027 028 029 034 036 038 052 067 069
>ambient - <50	002 004 005 006 012 013 015 016 019 021 022 025 031 032 033 035 037 039 040 042 043 044 045 046 047 049 050 051 053 054 057 058 059 061 063 064 066 072 074
≥50	060 068

Isocratic Mobile Phase	laboratory number
yes	001 002 004 005 006 007 009 012 013 016 017 021 024 027 029 031 032 034 036 038 039 042 043 045 047 050 051 052 053 054 059 064 066 067 068 069 072 074
no (gradient)	011 015 019 022 025 028 035 037 040 044 046 048 049 055 057 058 060 061

Mobile Phase Components	laboratory number
acetate	001 035 059
acetic acid	001 002 004 005 007 013 015 016 017 021 024 027 028 029 031 032 034 036 038 039 042 043 049 050 052 054 057 060 061 064 066 067 068 072
acetone	063
acetonitrile	001 002 004 005 006 007 012 013 015 016 017 019 021 024 027 028 029 031 032 033 034 036 038 039 042 043 045 046 047 050 051 052 053 054 059 060 061 064 066 067 068 069 072 074

Mobile Phase Components (continued)	laboratory number
ammonium acetate	060
ammonium formate	019
formic acid	011 019 025 033 040 044 046 047 049
KBr-HNO3	037
methanol	005 009 011 013 022 025 035 037 040 044 045 049 057 058 059 066
water	001 002 004 005 006 007 009 011 013 015 016 017 019 021 022 024 025 027 028 029 031 032 033 034 035 036 037 038 039 040 042 043 044 045 046 047 049 050 052 054 057 058 059 060 061 063 064 066 067 068 069 072

Mobile Phase Flow Rate (mL/min)	laboratory number
<0.25	016
≥0.25 - <0.75	011 012 015 022 025 033 035 040 044 045 046 047 049 057 058 060
≥0.75 - <1.25	001 004 005 006 007 009 013 017 019 021 024 027 028 029 031 032 034 036 037 038 039 042 043 050 051 052 053 054 059 061 063 064 066 067 068 069 072 074
≥1.25 - <1.75	002

Post Column Mobile Phase Flow Rate (mL/min)	laboratory number
<0.25	013
≥0.25 - <0.75	022 038 060
≥0.75	004 021 050 051 059 068

HPLC Post Column Derivatisation	laboratory number
none	001 005 006 009 022 032 034 038 044 047 048 050 052 053 054 059 060 061
NaOH	013

HPLC Detector Type	laboratory number
Diode Array Detector	070

HPLC Detector Type (continued)	laboratory number
fluorescence	001 002 004 005 006 007 009 012 013 015 017 019 021 024 027 028 029 031 032 034 036 037 038 039 042 043 045 050 051 052 053 054 059 061 064 066 067 068 069 072 074
MS-MS	011 016 022 025 033 035 040 044 046 047 048 049 057 058 060

Source of Standards	laboratory number
Biopure	002 017 019 066
Coring System	022
Fermentek	046
Included with ELISA kit	070
Industrial Analytical	033
Makor	005
R-Biopharm Rhone	008 020 021 027 034 035 051 054 056 064 072 074
Romer Labs	011 040
Sigma/Aldrich	001 007 012 013 015 022 025 032 036 037 038 043 044 046 047 049 052 057 058 059 060 067 068
Supelco	004 006 009 024 029 042 045 050 053 061 063 069
Trilogy	016
Wako	031 039

APPENDIX II: FAPAS SecureWeb, Reports and Protocol

1. FAPAS SECUREWEB

Access to the secure area of our website 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.
- 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.

3. PROTOCOL

The Protocols [2, 3] set out how FAPAS® is organised. Copies can be downloaded from our website.

4. QUALITY SYSTEMS

FAPAS® is accredited by UKAS as complying with the requirements of ISO/IEC 17043:2010

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

5. CONTACT DETAILS

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

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