FAPAS – Food Chemistry Proficiency Test Report 17165

Ochratoxin A in Paprika

August-September 2016

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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Fera is an ISO 9001 certified organisation.

UKAS PROFICIENCY TESTING
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SUMMARY

1. The test material for FAPAS – Food Chemistry proficiency test 17165 was dispatched in August 2016. Each participant received a paprika 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, $\|z\| \leq 2$	total number of scores	% $\|z\| \leq 2$
ochratoxin A	10.8	22	23	96

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 – Food Chemistry proficiency testing scheme are available in our protocols [3, 4].

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 [2].

The paprika was procured from a retail source and was found to naturally contain ochratoxin A (OTA). The paprika was mixed for 24 hours and packaged into foil sachets.

Samples were stored at -20°C temperature 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 [2].

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 August 2016. Test materials were sent to 25 participants.

3. RESULTS

The instructions for reporting results were as follows:

Determine the level of Ochratoxin A (OTA) present in the test material, in µg/kg, as received, corrected for recovery. Please note:

- Please state your % recovery when submitting your results.
- If a different correction factor to a % is used, please make a note of this in the comments section when submitting your results.
- It is important that you report the results in this way so that we can include as many results as possible in the statistical analysis.

Results were submitted by 24 participants (96%) before the closing date for this test, 21 September 2016.

Each participant was given a laboratory number, assigned in order of receipt of results. The reported OTA 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 values were 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 [5].

Further details on the procedure followed can be found in the relevant protocols [3, 4].

4.1. Calculation of the Assigned Value, x_a

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

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

- non numerical results i.e. qualitative or semi-quantitative results,
- results reported as approximately 10, 100 or 1000 × greater or smaller than the majority of submitted results (as these were considered to be reporting errors),
- results not corrected for recovery.

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

The assigned value for OTA is 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 ochratoxin A, σ_p was derived from the appropriate form of the Horwitz equation [6].

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 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. INTERPRETATION 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 [5].

6. REFERENCES

1 Adobe Certified Document Services, http://www.adobe.com/misc/pki/cds_cp.html, accessed 12/05/2016.

2 ISO/IEC 17043:2010, Conformity assessment – General requirements for proficiency testing.

3 FAPAS, 2014, Protocol for Proficiency Testing Schemes, Part 1 – Common Principles, Version 4, Issued May 2014.

4 FAPAS, 2014, Protocol for Proficiency Testing Schemes, Part 2 – FAPAS®, Version 3, Issued May 2014.

5 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.

6 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		
	ochratoxin A assigned value 10.8 µg/kg				ochratoxin A assigned value 10.8 µg/kg		
	result µg/kg	recovery (%)	z-score		result µg/kg	recovery (%)	z-score
001	13.0	73	0.9	013	9.0	95	-0.7
002	15.86	84.79	**2.1**	014	11.3	78	0.2
003	9.3	88	-0.6	015	9.326	100	-0.6
004	13.09	80.7	1.0	016	8.00		-1.2
005	13.2	108.0%	1.0	017	8.20	86	-1.1
006	9.5	103	-0.5	018	9.69	105.0	-0.5
007	10.8		0.0	019	12.0	73	0.5
008	10.87	103	0.0	020	9.14	112	-0.7
009	11.7	78.00	0.4	021	10.4	69	-0.2
				022	< LOQ (LOQ=2 µg/kg)		
010	12.9	90.9	0.9	023	9.82	90.87	-0.4
011	8.9	80-120	-0.8	024	9.70	83	-0.5
012	11.5	75	0.3				

z-scores outside |z| >2 are shown in **bold,** see Section 5

Table 2: Participants' Comments

participant number	comments
003	result corrected for recovery
022	LOQ=2 µg/kg

comments are as submitted by participants

Table 3: Assigned Value and Standard Deviation for Proficiency

analyte	data points, *n*	assigned value, X_a µg/kg	uncertainty, *u*	standard deviation for proficiency, σ_p, µg/kg	
ochratoxin A	21	10.8	0.419	Horwitz [6]	2.37

Table 4: Number and Percentage of z-Scores where $|z| \leq 2$

analyte	number of scores, $\|z\| \leq 2$	total number of scores	% $\|z\| \leq 2$
ochratoxin A	22	23	96

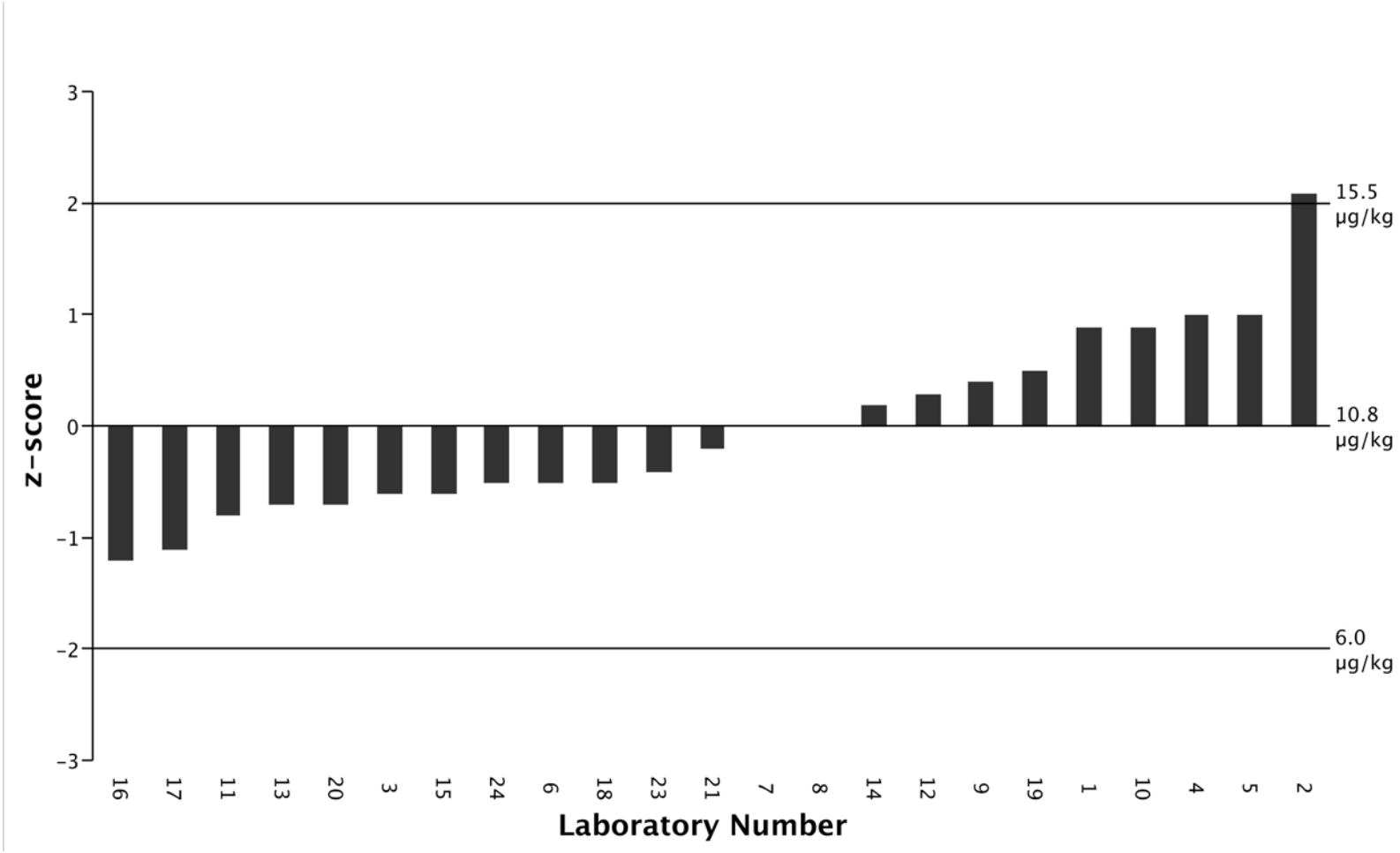

Figure 1: z-Scores for ochratoxin A

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	002 003 004 007 008 009 017 022 023
no	005 011 013 014

Method Based On	laboratory number
International Standard	003 013
National Standard	007
Manufacturer/Kit Instructions/Technical Note	004 005 011 022 023
In house method	002 008 009 014 017

Sample Weight (g)	laboratory number
≥1 - <2	011
≥2 - <5	002
≥5 - <10	007 008 022
≥10 - <25	003 004 005 009 014 017 023
≥25 - <50	013

Extraction Solvent Components	laboratory number
acetonitrile	008 013
dichloromethane	011
methanol	003
sodium bicarbonate	003 005 009 014 022
water	004 008 009 013 014 023
sodium bicarbonate	007
sodium hydrogencarbonate	017

Extraction Procedure	laboratory number
blend / homogenise with solvent	003 009 023
shake with solvent	003 011 013 014 022
shaking	002 005
sonicate/ultrasonic bath	017
Ultra Turrax	004 007 008

Extraction Type	laboratory number
single	002 003 004 005 008 009 011 013 014 017 023
multiple	007

Sample Work Up	laboratory number
centrifuge	002 007 009 011
dilute	003 011
filter	003 004 008 009 013 014 017 022 023

Sample Clean-up by Immunoaffinity Column (Brand)	laboratory number
R-Biopharm Rhone	002 004 008 009 013 014 017 022 023
VICAM	003
Romer Labs	007

Mycotoxin Determination	laboratory number
ELISA	011
HPLC	002 003 004 005 007 008 009 013 014 017 022 023

HPLC Injection Volume (µl)	laboratory number
<5	008
≥10 - <25	002 014
≥50 - <100	003 007 013 017
≥100 - <150	004 005 009 022 023

HPLC Column Packing	laboratory number
C18	002 003 004 008 009 013 014 022 023
C8	005
endcapped	009

HPLC Column Temperature (°C)	laboratory number
ambient	002 003 007 013 022
>ambient - <50	004 005 008 009 014 017
≥50	023

Isocratic Mobile Phase	laboratory number
yes	002 003 004 007 009 013 017 023
no (gradient)	005 008 014

Mobile Phase Components	laboratory number
ethanoic acid (acetic acid)	003 008 013 017
acetonitrile	003 008 009 013 014 017
formic acid	014
water	003 004 005 008 009 013 014 017 022 023
water/acetonitrile/acetic acide	007

Mobile Phase Flow Rate (ml/min)	laboratory number
≥0.25 - <0.75	002 004 005 008
≥0.75 - <1.25	003 009 013 014 017 022 023
≥1.25 - <1.75	007

Post Column Mobile Phase Flow Rate (ml/min)	laboratory number
≥0.25 - <0.75	002 005 008
≥0.75	023

HPLC Post Column Derivatisation	laboratory number
aqueous ammonia	013
none	002 007 009

HPLC Detector Type	laboratory number
Diode Array Detector	005
fluorescence	002 003 004 007 008 009 013 014 017 022 023

Source of Standards	laboratory number
R-Biopharm Rhone	009 022 023
Romer Labs	005 017
Sigma/Aldrich	007 008 013
Supelco	003 014
Biopure	004
LGC	011

APPENDIX II: FAPAS SecureWeb, Protocol and Contact Details

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, reference materials and quality control materials.
- Freely download copies of reports (PDF file), of proficiency tests in which they have participated.
- View charts of their z-scores obtained in previous FAPAS – Food Chemistry proficiency tests.

2. PROTOCOL

The Protocols [3, 4] set out how FAPAS – Food Chemistry is organised. Copies can be downloaded from our website.

3. CONTACT DETAILS

This report was prepared and authorised on behalf of FAPAS by Rosemary A Smith (Round Coordinator). Participants with any comments or concerns about this proficiency test should contact:

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