



**Fapas<sup>®</sup> – Food Chemistry Proficiency Test Report 04334**

**Aflatoxins in Animal Feed (cereal based)**

**April-May 2018**

## PARTICIPANT LABORATORY NUMBER

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## SUMMARY

1. The test materials for Fapas<sup>®</sup> – Food Chemistry proficiency test 04334 were dispatched in April 2018. Each participant received an animal feed (cereal based) test material to be analysed for aflatoxin B<sub>1</sub>, aflatoxin B<sub>2</sub>, aflatoxin G<sub>1</sub>, aflatoxin G<sub>2</sub> and aflatoxins (total).
2. An assigned value ( $x_a$ ) was determined for aflatoxin B<sub>1</sub>, aflatoxin B<sub>2</sub>, aflatoxin G<sub>1</sub>, aflatoxin G<sub>2</sub> and aflatoxins (total) and in conjunction with the standard deviation for proficiency ( $\sigma_p$ ) 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$
Aflatoxin B <sub>1</sub>	6.59	41	46	89
Aflatoxin B <sub>2</sub>	3.65	34	37	92
Aflatoxin G <sub>1</sub>	2.45	32	35	91
Aflatoxin G <sub>2</sub>	1.54	27	33	82
Aflatoxins (total)	13.9	40	44	91

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## 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<sup>®</sup> – Food Chemistry proficiency testing scheme are available in our protocols [4, 5].

## 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 [3].

The test materials were prepared from pig feed.

Aflatoxin B<sub>1</sub>, aflatoxin B<sub>2</sub>, aflatoxin G<sub>1</sub> and aflatoxin G<sub>2</sub> were spiked into the test material.

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 [3].

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

### 2.3. Dispatch

The start date was 05 April 2018. Test materials were sent to 58 participants.

## 3. RESULTS

The instructions for reporting results were as follows:

Determine the level of aflatoxin B<sub>1</sub>, aflatoxin B<sub>2</sub>, aflatoxin G<sub>1</sub>, aflatoxin G<sub>2</sub> and total aflatoxins 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 % recovery was used, please make a note of this in the comments box.

Results were submitted by 53 participants (91%) before the closing date for this test, 17 May 2018.

Each participant was given a laboratory number, assigned in order of receipt of results. The reported analyte concentrations are given in Tables 1-3.

Participants' comments are given in Table 4.

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 aflatoxin B<sub>1</sub>, aflatoxin B<sub>2</sub>, aflatoxin G<sub>1</sub>, aflatoxin G<sub>2</sub> and aflatoxins (total). The assigned values were then used in combination with the standard deviation for proficiency,  $\sigma_p$ , to calculate a z-score [6] for each result. The procedure is detailed in the relevant protocols [4, 5].

Further background on the procedure followed can be found in the IUPAC International Harmonised Protocol for the Proficiency Testing of Analytical Chemistry Laboratories [7].

### 4.1. Calculation of the Assigned Value, $x_a$

The assigned value,  $x_a$ , for aflatoxin B<sub>1</sub>, aflatoxin B<sub>2</sub>, aflatoxin G<sub>1</sub>, aflatoxin G<sub>2</sub> and aflatoxins (total) 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 reported as approximately 10, 100 or 1000 × greater or smaller than the majority of submitted results (as these were considered to be reporting errors),
- iii) results uncorrected for recovery, except for aflatoxins (total), where participants submitted results that were the sum of their recovery corrected individual aflatoxins.

For aflatoxin B<sub>1</sub>, aflatoxin B<sub>2</sub>, aflatoxin G<sub>1</sub>, aflatoxin G<sub>2</sub> and aflatoxins (total), this procedure was straightforward and the robust mean was chosen as the assigned value.

The assigned values for aflatoxin B<sub>1</sub>, aflatoxin B<sub>2</sub>, aflatoxin G<sub>1</sub>, aflatoxin G<sub>2</sub> and aflatoxins (total) are shown in Table 5.

### 4.2. Standard Deviation for Proficiency, $\sigma_p$

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

For aflatoxin B<sub>1</sub>, aflatoxin B<sub>2</sub>, aflatoxin G<sub>1</sub>, aflatoxin G<sub>2</sub> and aflatoxins (total),  $\sigma_p$  was derived from the appropriate form of the Horwitz equation [8].

The values for  $\sigma_p$  used to calculate z-scores from the reported results of this test are given in Table 5.

### 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  $\sigma_p$  = the standard deviation for proficiency.

Participants' z-scores for aflatoxin B<sub>1</sub>, aflatoxin B<sub>2</sub>, aflatoxin G<sub>1</sub>, aflatoxin G<sub>2</sub> and aflatoxins (total) are given in Tables 1-3 and shown as histograms in Figures 1-5. 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 standard deviations for proficiency prior to their publication in Table 5.

The number and percentage of z-scores in the range  $-2 \leq z \leq 2$  for aflatoxin B<sub>1</sub>, aflatoxin B<sub>2</sub>, aflatoxin G<sub>1</sub>, aflatoxin G<sub>2</sub> and aflatoxins (total) are given in Table 6.

## 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 [7].

## 6. REFERENCES

- 1 Adobe Approved Trust List, <https://helpx.adobe.com/acrobat/kb/approved-trust-list2.html#Whatisit> accessed 01/06/2017.
- 2 GlobalSign PDF Signing Tool, <https://www.globalsign.com/en/pdf-signing/> accessed 01/06/2017.
- 3 ISO/IEC 17043:2010, Conformity assessment – General requirements for proficiency testing.
- 4 Fapas<sup>®</sup>, 2017, Protocol for Proficiency Testing Schemes, Version 6, April 2017, Part 1 – Common Principles.
- 5 Fapas<sup>®</sup>, 2017, Protocol for Proficiency Testing Schemes, Version 5, April 2017, Part 2 – Fapas<sup>®</sup> Food Chemistry scheme (FAPAS).
- 6 AMC Tech Brief No. 74, z-Scores and other scores in chemical proficiency testing – their meanings, and some common misconceptions, *Anal. Methods*, 2016, **8**, 5553.
- 7 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.
- 8 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 for Aflatoxin B<sub>1</sub> and B<sub>2</sub>**

laboratory number	analyte					
	Aflatoxin B <sub>1</sub> assigned value: 6.59 µg/kg			Aflatoxin B <sub>2</sub> assigned value: 3.65 µg/kg		
	result	Recovery, %	z-score	result	Recovery, %	z-score
001						
002						
003	5.99	62.17	-0.4	3.55	77.66	-0.1
004	3.71	98.9	-2.0	2.70	99.9	-1.2
005	8.0	87	1.0	4.6	90	1.2
006	1.613	0.83	<b>-3.4</b>	0.832	0.86	<b>-3.5</b>
007	6.87	100	0.2	3.78	100	0.2
008	8.8	85	1.5	4.0	94	0.4
009						
010	9.32	74	1.9			
011						
012	7.0	80	0.3	4.4	84	0.9
013	6.72	121.62	0.1	3.56	123.30	-0.1
014	6.9	113.8	0.2			
015	6.6	90	0.0			
016	5.58	90	-0.7	3.36	95	-0.4
017	7.75	93	0.8	4.80	92	1.4
018	5.2	83	-1.0	2.9	85	-0.9
019	5.38	81.63	-0.8	3.24	80.00	-0.5
020	6.8	85	0.1			
021	4.86	74.65	-1.2	3.28	79.10	-0.5
022	6	70-110	-0.4	< 5	70-110	
023	7.43	100	0.6	4.55	100	1.1
024	6.21	88	-0.3			
025	7.27	104	0.5	3.65	111	0.0

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



**Table 1 (continued): Results and z-Scores for Aflatoxin B<sub>1</sub> and B<sub>2</sub>**

laboratory number	analyte					
	Aflatoxin B <sub>1</sub> assigned value: 6.59 µg/kg			Aflatoxin B <sub>2</sub> assigned value: 3.65 µg/kg		
	result	Recovery, %	z-score	result	Recovery, %	z-score
026	6.0		-0.4	3.2		-0.6
027	7.46	97.05	0.6			
028	7.5	96	0.6	4.1	99	0.6
029	4.16		-1.7	2.19		-1.8
030						
031	7.03		0.3	3.60		-0.1
032	7.30	91	0.5	4.72	78	1.3
033	5.716	99%	-0.6	3.558	96%	-0.1
034	10.6	91	<b>2.8</b>			
035	6.0	100	-0.4	4.0	100	0.4
036	4.795	87	-1.2	3.56	100	-0.1
037	7.17	80.66	0.4	4.15	87.70	0.6
038	11.7	82.6	<b>3.5</b>	5.32	91.6	<b>2.1</b>
039	6.61	90.00	0.0	3.71	70.00	0.1
040	7.41		0.6	4.43		1.0
041						
042	7.53	103.33	0.6	4.96	107.60	1.6
043	8.50	95.32	1.3	4.70	97.34	1.3
044						
045	7.38	100	0.5	0.74	100	<b>-3.6</b>
046	8.546	95	1.3			
047	4.88	90.0	-1.2	3.42	95.0	-0.3
048	7.37	88	0.5	3.92	95	0.3
049	7.73		0.8	3.40		-0.3
050	5.35	69.4	-0.9	2.86	92.4	-1.0

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

**Table 1 (continued): Results and z-Scores for Aflatoxin B<sub>1</sub> and B<sub>2</sub>**

laboratory number	analyte					
	Aflatoxin B <sub>1</sub> assigned value: 6.59 µg/kg			Aflatoxin B <sub>2</sub> assigned value: 3.65 µg/kg		
	result	Recovery, %	z-score	result	Recovery, %	z-score
051	5.5	90	-0.8	3.01	91	-0.8
052	3.10	85	<b>-2.4</b>	2.46	83	-1.5
053	3.23	83	<b>-2.3</b>	2.69	87	-1.2

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

**Table 2: Results and z-Scores for Aflatoxin G<sub>1</sub> and G<sub>2</sub>**

laboratory number	analyte					
	Aflatoxin G <sub>1</sub> assigned value: 2.45 µg/kg			Aflatoxin G <sub>2</sub> assigned value: 1.54 µg/kg		
	result	Recovery, %	z-score	result	Recovery, %	z-score
001						
002						
003	3.72	71.38	<b>2.3</b>	2.71	81.70	<b>3.4</b>
004	2.28	86.0	-0.3	1.06	95.4	-1.4
005	3.1	82	1.2	2.1	82	1.6
006	0.523	0.96	<b>-3.6</b>	0.184	0.95	<b>-4.0</b>
007	3.06	100	1.1	1.50	100	-0.1
008	2.8	87	0.6	1.3	90	-0.7
009						
010						
011						
012	3.0	70	1.0	2.0	80	1.3
013	2.53	104.76	0.1	1.81	97.42	0.8
014						
015						
016	2.16	102	-0.5	1.51	104	-0.1
017	2.80	93	0.6	1.77	77	0.7
018	1.9	95	-1.0	1.3	95	-0.7
019	1.75	72.00	-1.3	2.07	82.50	1.6
020						
021	<2.50 (1.13)	73.30		<1.50 (0.85)	68.50	
022	< 5	70-110		< 5	70-110	
023	2.70	100	0.5	1.86	100	0.9
024						
025	2.53	97	0.1	1.44	97	-0.3

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

**Table 2 (continued): Results and z-Scores for Aflatoxin G<sub>1</sub> and G<sub>2</sub>**

laboratory number	analyte					
	Aflatoxin G <sub>1</sub> assigned value: 2.45 µg/kg			Aflatoxin G <sub>2</sub> assigned value: 1.54 µg/kg		
	result	Recovery, %	z-score	result	Recovery, %	z-score
026	2.2		-0.5	1.5		-0.1
027						
028	2.6	99	0.3	1.7	97	0.5
029	1.69		-1.4	1.04		-1.5
030						
031	2.40		-0.1	0.48		<b>-3.1</b>
032	2.84	83	0.7			
033	2.262	88%	-0.4	1.388	105%	-0.5
034						
035	2.2	100	-0.5	1.8	100	0.8
036	1.5	90	-1.8	1.21	100	-1.0
037	2.29	92.52	-0.3	1.55	91.99	0.0
038	5.0	88.8	<b>4.7</b>	2.37	92.7	<b>2.4</b>
039	2.85	84.00	0.7	1.26	80.00	-0.8
040	2.63		0.3	1.72		0.5
041						
042	2.93	88.80	0.9	1.74	101.55	0.6
043	2.94	88.65	0.9	2.17	78.00	1.8
044						
045	2.04	100	-0.8			
046						
047	2.23	90.5	-0.4	1.09	71	-1.3
048	2.67	86	0.4	1.70	94	0.5
049	3.55		2.0	1.74		0.6
050						

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

**Table 2 (continued): Results and z-Scores for Aflatoxin G<sub>1</sub> and G<sub>2</sub>**

laboratory number	analyte					
	Aflatoxin G <sub>1</sub> assigned value: 2.45 µg/kg			Aflatoxin G <sub>2</sub> assigned value: 1.54 µg/kg		
	result	Recovery, %	z-score	result	Recovery, %	z-score
051	2.4	88	-0.1	1.2	89	-1.0
052	1.63	87	-1.5	0.74	88	<b>-2.4</b>
053	1.57	84	-1.6	0.81	89	<b>-2.2</b>

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

**Table 3: Results and z-Scores for Aflatoxins (total)**

laboratory number	analyte		
	Aflatoxins (total) assigned value: 13.9 µg/kg		
	result	Recovery, %	z-score
001	17		1.0
002	15	96	0.4
003	15.97	73.23	0.7
004	9.75		-1.4
005	17.8	85	1.3
006	3.6	3.6	<b>-3.4</b>
007	15.20		0.4
008			
009	13.57	108.96	-0.1
010	18.22	84	1.4
011	15.00	103.37	0.4
012	16.4	79	0.8
013	15.09	112.51	0.4
014			
015			
016	12.61	104	-0.4
017	17.12		1.0
018	11.3		-0.9
019	12.44		-0.5
020			
021	11.33	79.00	-0.8
022	6	70-110	<b>-2.6</b>
023	16.54	100	0.9
024			
025			

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

**Table 3 (continued): Results and z-Scores for Aflatoxins (total)**

laboratory number	analyte		
	Aflatoxins (total) assigned value: 13.9 µg/kg		
	result	Recovery, %	z-score
026	12.9		-0.3
027			
028	15.9		0.6
029	9.08		-1.6
030	10.9	94.25	-1.0
031	13.53		-0.1
032	17.55	81	1.2
033	12.924	97%	-0.3
034	13.8	95	0.0
035	14	100	0.0
036	11.065	94.25	-0.9
037	15.16	88.22	0.4
038	24.4	86.4	<b>3.4</b>
039	14.43	-	0.2
040	16.19		0.7
041	7.1	*	<b>-2.2</b>
042	17.2	/	1.1
043	18.31		1.4
044	11.85		-0.7
045	10.16	100	-1.2
046			
047	11.62		-0.8
048	15.66	89	0.6
049	16.42		0.8
050			

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

**Table 3 (continued): Results and z-Scores for Aflatoxins (total)**

laboratory number	analyte		
	Aflatoxins (total) assigned value: 13.9 µg/kg		
	result	Recovery, %	z-score
051	12.11	90	-0.6
052	7.93	86	-2.0
053	8.30	85	-1.8

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



**Table 4: Participants' Comments**

laboratory number	comments
002	the corrected result for recovery is 15.6 µg/kg
008	The results are corrected for recovery.
015	This method is accredited to ISO 17025.
017	Aflatoxins (total) is the total of already corrected results, thus no recovery % for Aflatoxins (total).
021	Aflatoxin G1 Lab Result 1.13 ug/kg, LOD = 1.0 ug/kg , LOQ =2.5 ug/kg, Aflatoxin G2 Lab Result 0.85 ug/kg, LOD = 0.70 ug/kg, LOQ=1.5 ug/kg
023	Submitted results not corrected for recovery.
025	Standard additions- used for quantitation. % Recovery based on quality control material analyzed at same time.
041	* Kit-System with no standards, so no recovery analysed, method not accredited
048	Expanded Uncertainty (k = 2,00; 95%): AFB1 = 2,4 µg/kg; AFB2 = 1,1 µg/kg; AFG1 = 1,0 µg/kg; AFG2 = 1,5 µg/kg; AFBG = 3,2 µg/kg.

comments are as submitted by participants but some may have been edited to maintain participant anonymity

**Table 5: Assigned Values and Standard Deviations for Proficiency**

analyte	data points, <i>n</i>	assigned value, $x_a$ µg/kg	uncertainty, <i>u</i>	standard deviation for proficiency, $\sigma_p$
Aflatoxin B <sub>1</sub>	39	6.59	0.27	Horwitz [8] 1.45
Aflatoxin B <sub>2</sub>	31	3.65	0.16	Horwitz [8] 0.804
Aflatoxin G <sub>1</sub>	29	2.45	0.11	Horwitz [8] 0.540
Aflatoxin G <sub>2</sub>	27	1.54	0.10	Horwitz [8] 0.339
Aflatoxins (total)	34	13.9	0.6	Horwitz [8] 3.06

**Table 6: Number and Percentage of z-Scores where  $|z| \leq 2$** 

analyte	number of scores where $ z  \leq 2$	total number of scores	% $ z  \leq 2$
Aflatoxin B <sub>1</sub>	41	46	89
Aflatoxin B <sub>2</sub>	34	37	92
Aflatoxin G <sub>1</sub>	32	35	91
Aflatoxin G <sub>2</sub>	27	33	82
Aflatoxins (total)	40	44	91

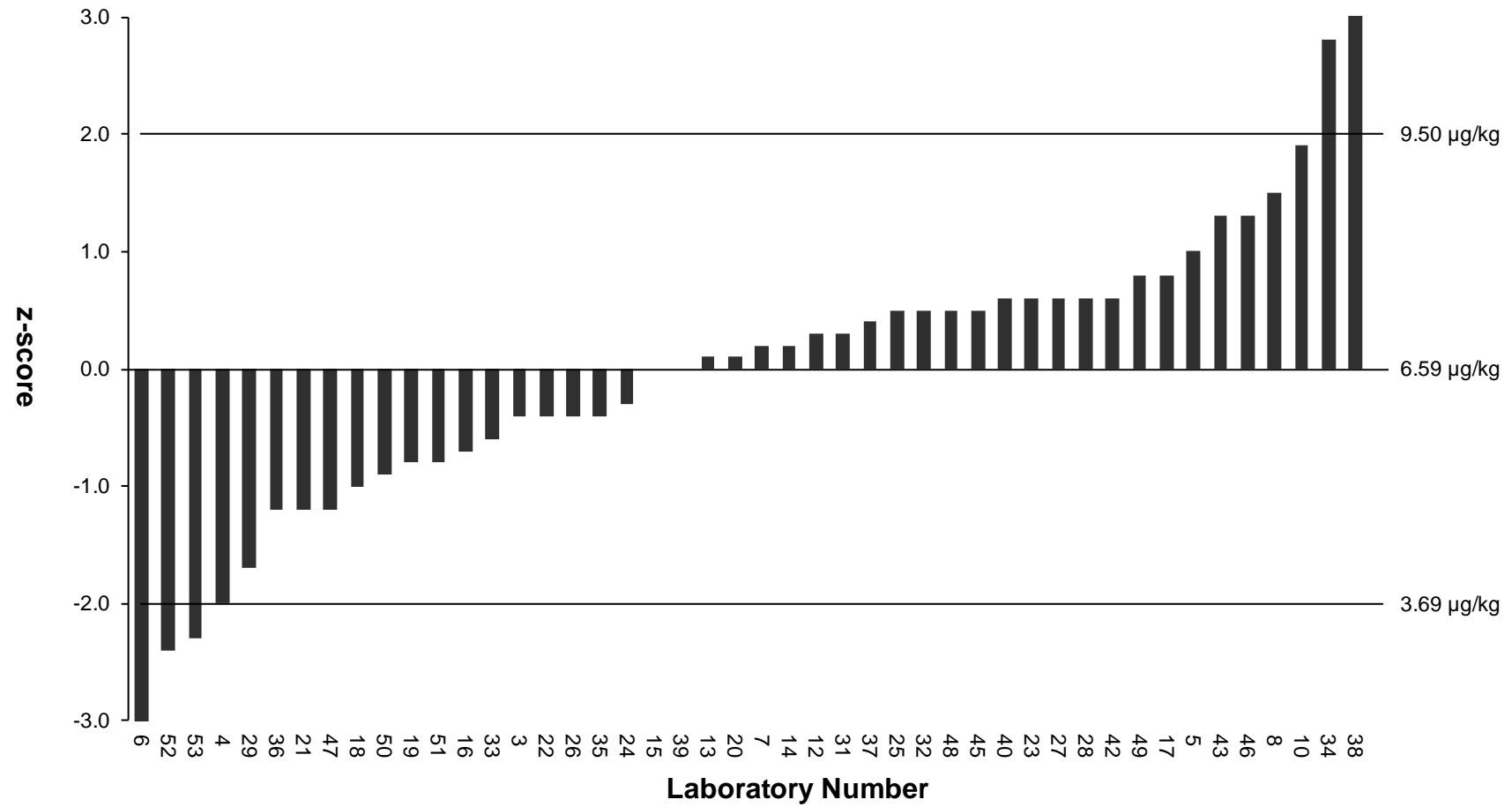


Figure 1: z-Scores for Aflatoxin B<sub>1</sub>

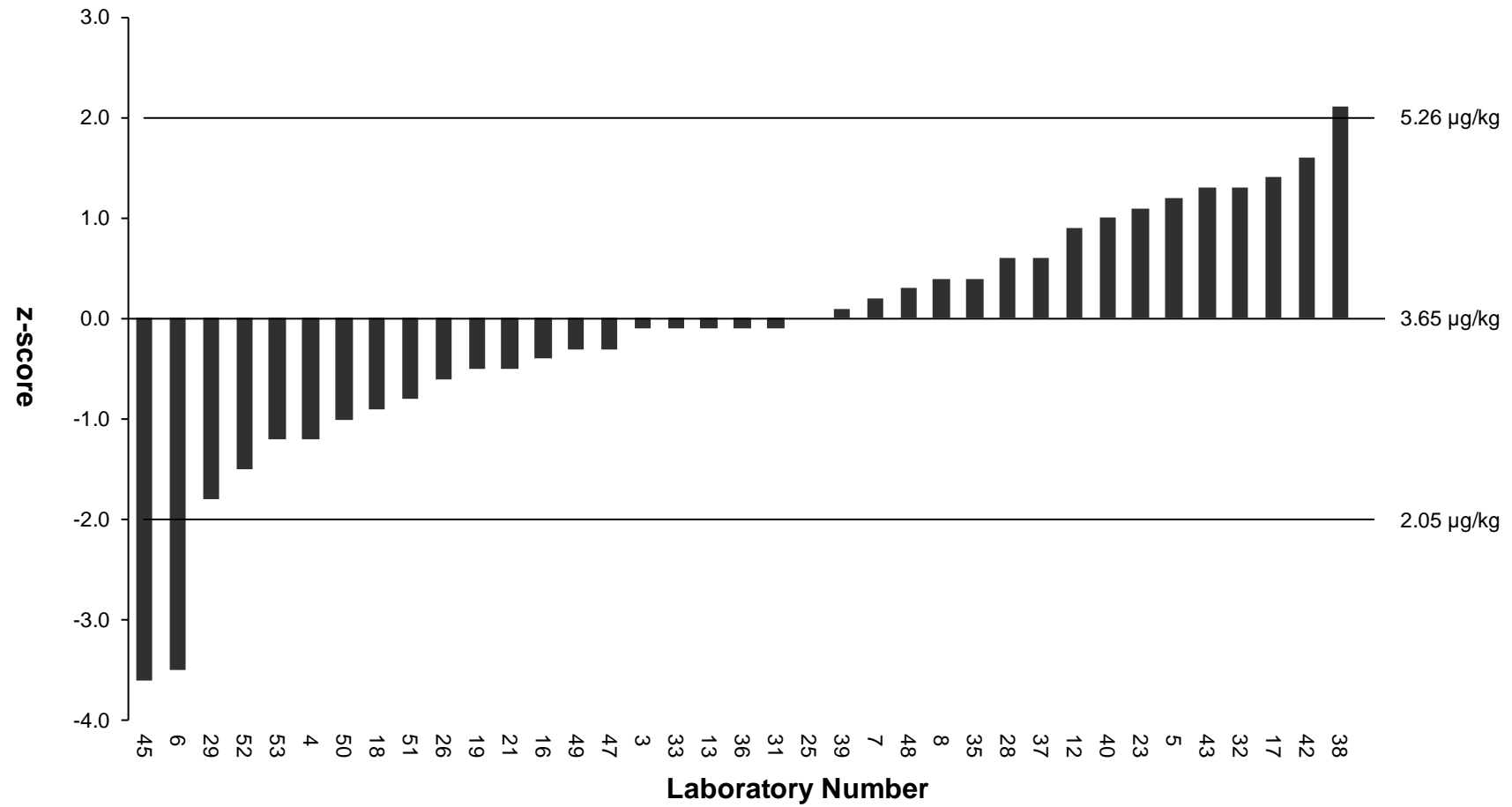


Figure 2: z-Scores for Aflatoxin B<sub>2</sub>

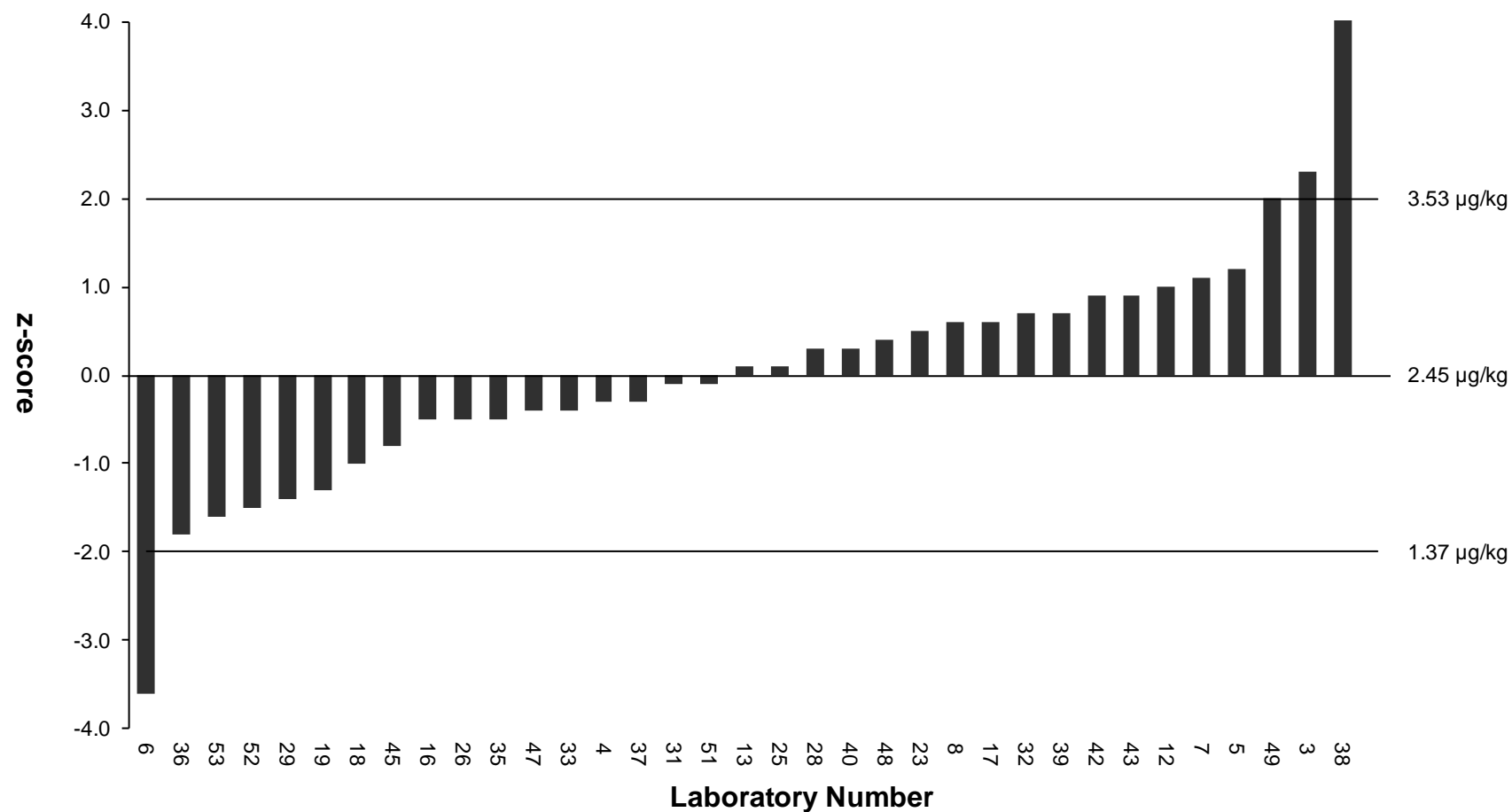


Figure 3: z-Scores for Aflatoxin G<sub>1</sub>

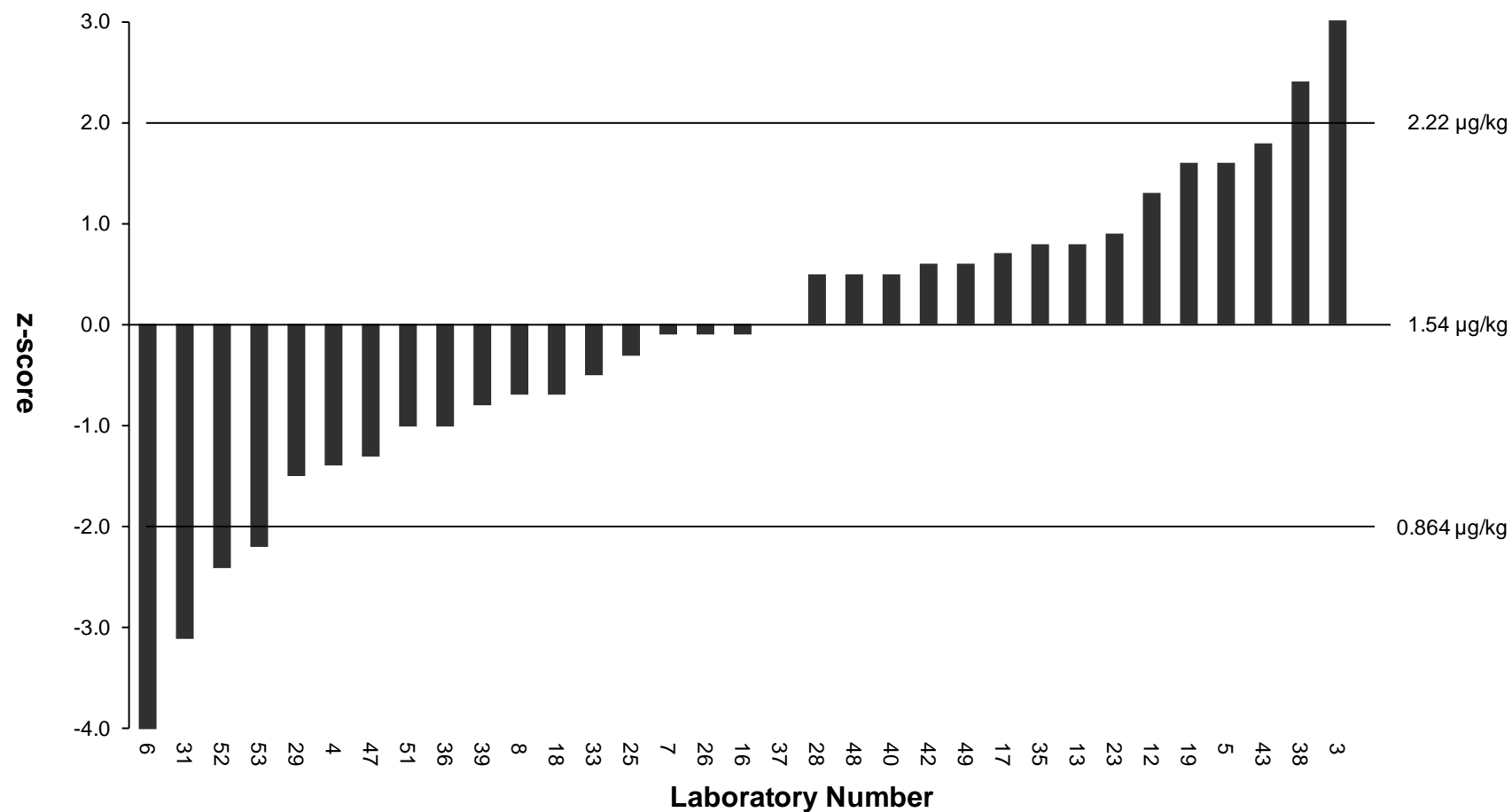


Figure 4: z-Scores for Aflatoxin G<sub>2</sub>

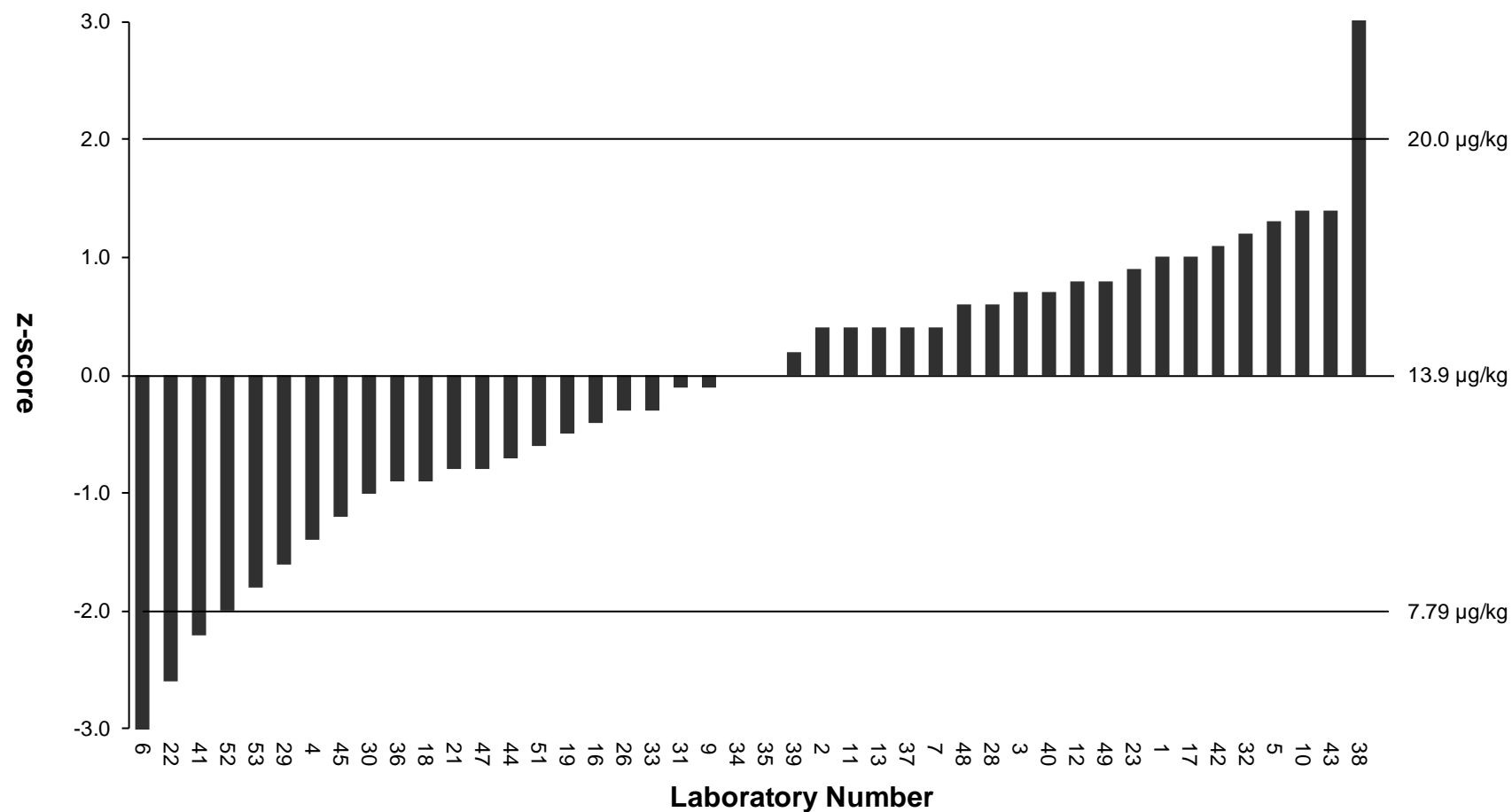


Figure 5: z-Scores for Aflatoxins (total)

## 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.

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<b>Is the Method Used Accredited?</b>	<b>laboratory number</b>
no	003 008 009 016 023 031 033 041 045 046
yes	001 004 005 006 007 010 011 012 013 015 017 019 021 022 025 027 029 032 036 037 038 039 040 043 048 049 050 051

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<b>What is Your Method Based On?</b>	<b>laboratory number</b>
International Standard	006 012 013 016 019 027 031 032 036 038 045 046
National Standard	001 037 040
Paper Published In An International Journal	017 048
Manufacturer/Kit Instructions/Technical Note	009 010 011 022 023 025 028 030 041
In house method	003 004 005 007 008 015 021 029 033 039 043 049 050

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<b>Sample Weight (g)</b>	<b>laboratory number</b>
≥1 - <2	004
≥2 - <5	009 038
≥5 - <10	003 005 008 023 025 037 040 050
≥10 - <25	007 013 022 028 029 036 041 045 049
≥25 - <50	006 011 012 015 017 019 021 030 033 039 043
≥50	001 010 016 027 031 032 046 048

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<b>Extraction Solvent Components</b>	<b>laboratory number</b>
acetone	006 027 032 045 046
acetonitrile	005 007 008 009 013 017 023 025 028 038 043 049
chloroform	033
formic acid (methanoic acid)	007
methanol	001 003 010 012 015 016 019 021 029 030 031 036 037 039 040 050
water	001 003 005 007 009 010 012 013 015 016 017 023 025 028 029 031 032 033 036 037 039 043 045 046
65% ethanol solution	041
methanol and water	048
Methanol, Water	011
methanol/water 60:40 VV	004
Methanol/waters	022

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<b>Extraction Procedure</b>	<b>laboratory number</b>
add filter aid	015
add NaCl	001 003 004 012 015 016 019 021 039 040 048
blend / homogenise with solvent	001 010 011 015 016 030 032 039 045
maceration/homogenisation	012
shake with solvent	003 006 008 009 017 023 025 031 033 036 038 041 043 046 050
shaking	005 007 013 027 029
sonicate/ultrasonic bath	029 049
Ultra Turrax	028 037
Add NaCl and shaking	022
centrifuge	041

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<b>Extraction Type</b>	<b>laboratory number</b>
multiple	009 022 032 040 043 050
single	001 003 004 005 006 007 008 010 011 012 013 016 017 019 021 023 025 027 029 031 033 036 037 038 039 041 045 046 048 049

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<b>Sample Work Up</b>	<b>laboratory number</b>
centrifuge	008 023 036 038 041
defatted with hexane	004 029
dilute	001 003 005 007 009 012 015 016 021 023 031 032 039 045
evaporate	033 037
filter	001 003 005 006 010 011 013 015 017 019 022 023 025 027 031 032 033 036 037 040 043 046 048 049 050
pH adjustment	012 036
Ultra Turrax	037 048

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<b>Sample Clean-up by Immunoaffinity Column (Brand)</b>	<b>laboratory number</b>
R-Biopharm Rhone	003 004 005 009 012 016 017 022 023 027 028 031 032 036 037 038 039 043 045 046 048 050
VICAM	001 010 011 015 019 021 029
HLB PRIME	008
Reveal Q+ for Aflatoxin (Neogen)	041
Romer	013 025
Romer Labs /Austria	033

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<b>Sample Clean-up by SPE</b>	<b>laboratory number</b>
fluorescence	046
single-level	045
Aflatest column	011
HLB PRIME	008
immunoaffinity	036
NONE	012
silica	015

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<b>Mycotoxin Determination</b>	<b>laboratory number</b>
ELISA	009 030 041
fluorometric	001 011
HPLC	003 004 005 006 010 012 013 015 016 017 019 022 023 027 028 029 031 032 033 036 037 038 039 040 043 045 046 048 049
HPLC and Fluorometer	021
HPLC-HRMS	025
LC-MS	050
LC-MS/MS	007
UHPLC	008

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<b>HPLC Injection Volume (<math>\mu</math>l)</b>	<b>laboratory number</b>
<5	025
$\geq 5$ - <10	008 022 038
$\geq 10$ - <25	004 005 013 021 033 037 039 049
$\geq 25$ - <50	007 019 029 050
$\geq 50$ - <100	003 006 010 036 040 048
$\geq 100$ - <150	012 015 016 017 023 027 031 032 043 045 046
$\geq 150$	028

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<b>HPLC Column Packing</b>	<b>laboratory number</b>
C18	003 004 005 007 008 010 012 013 015 016 017 019 021 023 025 027 028 029 031 032 033 036 037 038 039 040 043 045 046 048 049 050
C18 X-Terra type	022
endcapped	003 023
Inertsil ODS-3	006

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<b>HPLC Column Temperature (<math>^{\circ}</math>C)</b>	<b>laboratory number</b>
ambient	003 004 017 019 021 028 032 036 050
>ambient - <50	005 006 007 008 010 012 013 015 016 022 023 025 027 029 031 033 037 038 039 040 043 045 046 048 049

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<b>Mobile Phase Components</b>	<b>laboratory number</b>
acetonitrile	003 005 013 016 017 028 029 031 032 033 036 037 038 039 045 046 048
formic acid (methanoic acid)	007 008 025
HNO <sub>3</sub> & KBr (for Kobra Cell)	004 005 006 012 016 017 023 028 031 032 036 037 043 045 046 048
methanol	003 005 007 010 012 013 015 016 017 019 023 025 027 028 029 031 032 033 036 037 039 040 043 045 046 048 049 050
THF	003
water	003 005 007 010 012 013 015 016 017 023 025 027 028 029 031 032 033 036 037 039 043 045 046 048
ACN:DI:MeOH	021
ammonium formate	025

<b>Mobile Phase Flow Rate (ml/min)</b>	<b>laboratory number</b>
<0.25	050
≥0.25 - <0.75	005 007 008 022 025 038 049
≥0.75 - <1.25	003 004 006 010 012 015 016 017 019 021 023 027 028 029 031 032 033 036 037 039 040 043 045 046 048
≥1.25 - <1.75	013

<b>Post Column Mobile Phase Flow Rate (ml/min)</b>	<b>laboratory number</b>
	008
<0.25	022
≥0.25 - <0.75	003 005 033 037 049
≥0.75	012 015 017 036 039 040 043 045 046

<b>HPLC Pre Column Derivatisation</b>	<b>laboratory number</b>
	008
none	012 015 017 019 022 036 039 040 043 045 049 050

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<b>HPLC Post Column Derivatisation</b>	<b>laboratory number</b>
Kobra cell	004 005 012 016 017 023 028 032 036 037 043 045 046 048
pyridinium bromide perbromide (C <sub>5</sub> H <sub>6</sub> Br <sub>3</sub> N)	003 010 038
none	008 022 039 040 049 050
HPLC-Fluorescence detection	021
Photochem	015
Romer	013
UV	033

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<b>Source of Standards</b>	<b>laboratory number</b>
R-Biopharm Rhone	009 012 022 023 031 038 043 045 046 050
Romer Labs	005 007 008 028 049
Sigma/Aldrich	003 006 013 017 025 032 036 037
Supelco	004 010 011 015 019 029 030 039 048
VICAM	001
n'TOX /Novakits France	033
Trilogy Mycotoxin Standard	016

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<b>HPLC Detector Type</b>	<b>laboratory number</b>
fluorescence	003 004 005 010 012 013 015 016 017 019 021 023 028 029 031 032 033 036 037 038 039 043 045 046 048
MS-MS	007 008 022 049 050
UV	006
HRMS	025

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## APPENDIX II: Fapas<sup>®</sup> SecureWeb, Protocol and Contact Details

### 1. Fapas<sup>®</sup> SECUREWEB

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- 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<sup>®</sup> – Food Chemistry proficiency tests.

### 2. PROTOCOL

The Protocols [4, 5] set out how Fapas<sup>®</sup> – Food Chemistry is organised. Copies can be downloaded from our website.

### 3. CONTACT DETAILS

This report was prepared and authorised on behalf of Fapas<sup>®</sup> by Jacob Darlow (Round Coordinator). Participants with any comments or concerns about this proficiency test should contact:

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