Fapas® – Food Chemistry Proficiency Test Report 07279

Metals in Canned Crab Meat

February-March 2017

PARTICIPANT LABORATORY NUMBER

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0009

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SUMMARY

1. The test material for Fapas® – Food Chemistry proficiency test 07279 was dispatched in February 2017. Each participant received a canned crab meat test material to be analysed for arsenic (As) (total), arsenic (As) (inorganic), cadmium (Cd), lead (Pb) and mercury (Hg) (total).
2. An assigned value (x_a) was determined for each analyte and in conjunction with the standard deviation for proficiency (σ_p) was used to calculate a z-score for each result. However, an assigned value could not be set for arsenic (As) (inorganic).
3. Results for this proficiency test are summarised as follows:

analyte	assigned value, x_a	units	number of scores, |z| ≤2	total number of scores	% |z|≤2
Arsenic (total)	16.3	mg/kg	45	52	87
Arsenic (inorganic)	*not set*				
Cadmium	7.55	mg/kg	63	64	98
Lead	48.1	µg/kg	45	53	85
Mercury (total)	106	µg/kg	59	63	94

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 test material was prepared from crab meat with a ratio of 3:1 dark:light meat.

All analytes were present at natural levels.

Samples were stored at ambient 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 values.

2.3. Dispatch

The start date was 3 February 2017. Test materials were sent to 75 participants.

3. RESULTS

The instructions for reporting results were as follows:

- Determine the level of total arsenic (As) and cadmium (Cd) present in the test material, in **mg/kg, as received** and the level of inorganic arsenic (As), lead (Pb) and total mercury (Hg) present in the test material, in **µg/kg, as received**.
- Results were submitted by 72 participants (96%) before the closing date for this test, 9 March 2017.

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 each analyte. The assigned values were then used in combination with the standard deviation for proficiency, σ_p, to calculate a z-score [5] for each result. The procedure is detailed in the relevant protocols [3, 4].

Further background on the procedure followed can be found in the IUPAC International Harmonised Protocol for the Proficiency Testing of Analytical Chemistry Laboratories [6].

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:

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).

For arsenic (As) (total), cadmium (Cd), lead (Pb) and mercury (Hg) (total), this procedure was straightforward and the robust mean was chosen as the assigned value.

It was not possible to set an assigned value for the inorganic Arsenic due to the significant uncertainty associated with it. A distribution of results can be seen in Figure 2.

The assigned values for arsenic (As) (total), cadmium (Cd), lead (Pb) and mercury (Hg) (total) 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 all analytes, σ_p was derived from the appropriate form of the Horwitz equation [7].

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 all analytes are given in Table 1 and shown as histograms in Figure 1 and Figures 3 to 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 3.

The number and percentage of z-scores in the range $-2 \leq z \leq 2$ for all analytes 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 [6].

6. REFERENCES

1 Adobe Certified Document Services, https://helpx.adobe.com/acrobat/kb/certificate-signatures.html, accessed 10/03/2017.

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

3 Fapas®, 2016, Protocol for Proficiency Testing Schemes, Part 1 – Common Principles, Version 5, Issued September 2016.

4 Fapas®, 2016, Protocol for Proficiency Testing Schemes, Part 2 – Fapas® – Food Chemistry Version 4, Issued September 2016.

5 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.

6 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.

7 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									
	arsenic (total) assigned value 16.3 mg/kg		arsenic (inorganic) assigned value not set			cadmium assigned value 7.55 mg/kg		lead assigned value 48.1 µg/kg		mercury (total) assigned value 106 µg/kg	
	result	z-score	result µg/kg	As III + As V?	z-score	result	z-score	result	z-score	result	z-score
001						6.4	-1.3	< 80		119	0.6
002						8.10	0.6	48.0	0.0	97.4	-0.4
003	16.6	0.2	158	yes		7.7	0.2	45	-0.3	108	0.1
004	15.05	-0.7				6.95	-0.7	<200		<200	
005	13.13	-1.9				9.24	1.9	42.83	-0.5		
006	13.79	-1.5				5.85	-1.9	<60		123.7	0.8
007	18.594	1.3				7.6171	0.1	50.7	0.2	94	-0.5
008	16.42	0.1				7.34	-0.2	0.044	**-4.5**	0.110	**-4.5**
009	16.5	0.1				8.07	0.6	45	-0.3	105	0.0
010	18.32	1.2				7.77	0.3	41.93	-0.6	103.83	-0.1
011	16.9	0.3	30.5	Yes		7.72	0.2	43.9	-0.4	107.6	0.1
012	16.25	0.0				7.28	-0.3			82.3	-1.0
013	24.02	**4.5**				7.48	-0.1	67.69	1.8	118.38	0.5
014	15.99	-0.2	< 50	Yes		6.918	-0.7	33	-1.4	90	-0.7
015	17.3	0.6	366	yes		7.57	0.0	44.6	-0.3	109	0.1

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

Table 1 (continued): Results and z-Scores

laboratory number	analyte									
	arsenic (total) assigned value 16.3 mg/kg		arsenic (inorganic) assigned value not set			cadmium assigned value 7.55 mg/kg		lead assigned value 48.1 µg/kg		mercury (total) assigned value 106 µg/kg	
	result	z-score	result µg/kg	As III + As V?	z-score	result	z-score	result	z-score	result	z-score
016						7.68	0.2	57.2	0.9	98.2	-0.3
017	15.425	-0.5				7.489	-0.1	27.819	-1.9	84.110	-0.9
018						7.5	-0.1	28	-1.9	110	0.2
019	17.43	0.6				7.84	0.3	39.42	-0.8	86.65	-0.8
020						7.04	-0.6	49.8	0.2	104	-0.1
021	16.9	0.3				7.4	-0.2	47.6	-0.1	100.	-0.2
022	> 1.5					> 2.5		< 60		100	-0.2
023	17.3	0.6	103	Yes		7.62	0.1	49.8	0.2	109	0.1
024			37	Yes							
025	19.22	1.7				8.01	0.5	68.5	1.9	64.0	-1.8
026						7.84	0.3			131	1.1
027	17.331	0.6	3863	yes		7.352	-0.2	0.050	**-4.5**	0.098	**-4.5**
028	17.1	0.5	36	YES		7.9	0.4	45	-0.3	111	0.2
029	9.61	**-3.9**				6.11	-1.6				
030	15.7	-0.4	54.0	No		7.69	0.2	45.1	-0.3	104	-0.1

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

Table 1 (continued): Results and z-Scores

laboratory number	analyte									
	arsenic (total) assigned value 16.3 mg/kg		arsenic (inorganic) assigned value not set			cadmium assigned value 7.55 mg/kg		lead assigned value 48.1 µg/kg		mercury (total) assigned value 106 µg/kg	
	result	z-score	result µg/kg	As III + As V?	z-score	result	z-score	result	z-score	result	z-score
031	15.74	-0.3				7.69	0.2	46.68	-0.1	109.50	0.2
032	>4.00					>4.00		<0.12		0.11	**-4.5**
033						8.15	0.7	50.96	0.3		
034						8.27	0.8			128.16	1.0
035	17.36	0.6				8.11	0.6	46.9	-0.1	104.2	-0.1
036	19.8	2.0				7.33	-0.2	49.5	0.1	109	0.1
037	15.4	-0.5				7.88	0.4	60	1.1	100	-0.2
038	17.907	0.9	449	Yes		7.819	0.3	44	-0.4		
039						11.2	**4.1**	40.2	-0.7	134	1.2
040	12.57	**-2.2**	808.8	YES		6.13	-1.6	79.4	**3.0**	99.6	-0.3
041	16.67	0.2				7.86	0.4	46	-0.2	122	0.7
042	7.59	**-5.1**				7.25	-0.3	140	**8.7**	79.19	-1.1
043						8.94	1.6	48.9	0.1	129	1.0
044	18.038	1.0	80	yes		7.357	-0.2	58	0.9	140	1.5
045	17.44	0.7				7.72	0.2	43.1	-0.5	111	0.2

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

Table 1 (continued): Results and z-Scores

laboratory number	analyte									
	arsenic (total) assigned value 16.3 mg/kg		arsenic (inorganic) assigned value not set			cadmium assigned value 7.55 mg/kg		lead assigned value 48.1 µg/kg		mercury (total) assigned value 106 µg/kg	
	result	z-score	result µg/kg	As III + As V?	z-score	result	z-score	result	z-score	result	z-score
046										97.8	-0.3
047			174.0912	yes		7.057	-0.5	48.9573	0.1	111.1	0.2
048	17.17	0.5				6.428	-1.3	188.3	**13.2**	78.455	-1.2
049	14.71	-0.9	37.14	Yes, it is		8.15	0.7	63.40	1.4	120.92	0.7
050	16.44	0.1				7.70	0.2	49.8	0.2	95.8	-0.4
051	14.62	-1.0				6.152	-1.6	34.4	-1.3	63.5	-1.8
052	11.27	**-2.9**	101.00	Yes		7.29	-0.3	17.72	**-2.9**	118.55	0.6
053	16.8	0.3				7.46	-0.1	46.3	-0.2	95.9	-0.4
054	17.43	0.6				7.57	0.0	45.7	-0.2	132.4	1.1
055	19.4	1.8				7.68	0.2	44	-0.4	109	0.1
056	15.9	-0.2	386	yes		7.90	0.4	34.4	-1.3	83.2	-1.0
057						7.03	-0.6	≤100		67.71	-1.6
058	16.9	0.3	72.4	Yes		7.83	0.3	54.2	0.6	104	-0.1
059	15.03	-0.8				7.83	0.3			112	0.3
060	16.2	-0.1				7.5	-0.1	<180		88.7	-0.7

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

Table 1 (continued): Results and z-Scores

laboratory number	analyte									
	arsenic (total) assigned value 16.3 mg/kg		arsenic (inorganic) assigned value not set			cadmium assigned value 7.55 mg/kg		lead assigned value 48.1 µg/kg		mercury (total) assigned value 106 µg/kg	
	result	z-score	result µg/kg	As III + As V?	z-score	result	z-score	result	z-score	result	z-score
061								40.415	-0.7	109.033	0.1
062	17.64	0.8				8.83	1.4	151	**9.7**	114	0.4
063	16.1	-0.1				7.9	0.4	53	0.5	140	1.5
064	11.5	**-2.8**	<1	yes							
065						5.97	-1.8	211.29	**15.4**		
066										1500.04	**60.0**
067	16.7	0.2				7.75	0.2	48	0.0	112	0.3
068	16.5	0.1				7.73	0.2	41.2	-0.7	125	0.8
069	16.2	-0.1				6.68	-1.0	45.1	-0.3	87.5	-0.8
070	15.4	-0.5				7.19	-0.4			114.0	0.4
071	11.79	**-2.6**				6.08	-1.6				
072										101.9	-0.2

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

Table 2: Participants' Comments

laboratory number	comments
014	The result of Inorganic Arsenic less thank LOQ
036	The digestion is done according to our accredited method containing HF.
054	PT 07279 was tested by CTS-015 (Hg=133ppb) & by CTS-013 (pb less than LOQ , Cd=8.03ppm, As= 17.85ppm) & by CTS-012 (pb=47.72ppb, Cd=8.41ppm and As=15.39ppm).
057	LQ lead = 100 µg/kg
064	Arsenic (total) is the calculated sum of As(V), As(lll), AsC, AsB, DMA, and MMA. Arsenic (inorganic) is the calculated sum of As(V) and As(lll). ALSE has accreditation for the individual As species.
066	The result is an average of 6 measurement and its 1500.04 +/- 102.35
070	Lead is not tested because the amount of sample is insufficient.

comments are as submitted by participants

Table 3: Assigned Values and Standard Deviations for Proficiency

analyte	data points, *n*	assigned value, x_a	units	uncertainty, *u*	standard deviation for proficiency, σ_p	
Arsenic (total)	52	16.3	mg/kg	0.200	Horwitz [7]	1.72
Arsenic (inorganic)		*not set*				
Cadmium	64	7.55	mg/kg	0.070	Horwitz [7]	0.891
Lead	51	48.1	µg/kg	1.40	Horwitz [7]	10.6
Mercury (total)	59	106	µg/kg	2.00	Horwitz [7]	23.2

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

analyte	number of scores where $\|z\| \leq 2$	total number of scores	% $\|z\| \leq 2$
Arsenic (total)	45	52	87
Cadmium	63	64	98
Lead	45	53	85
Mercury (total)	59	63	94

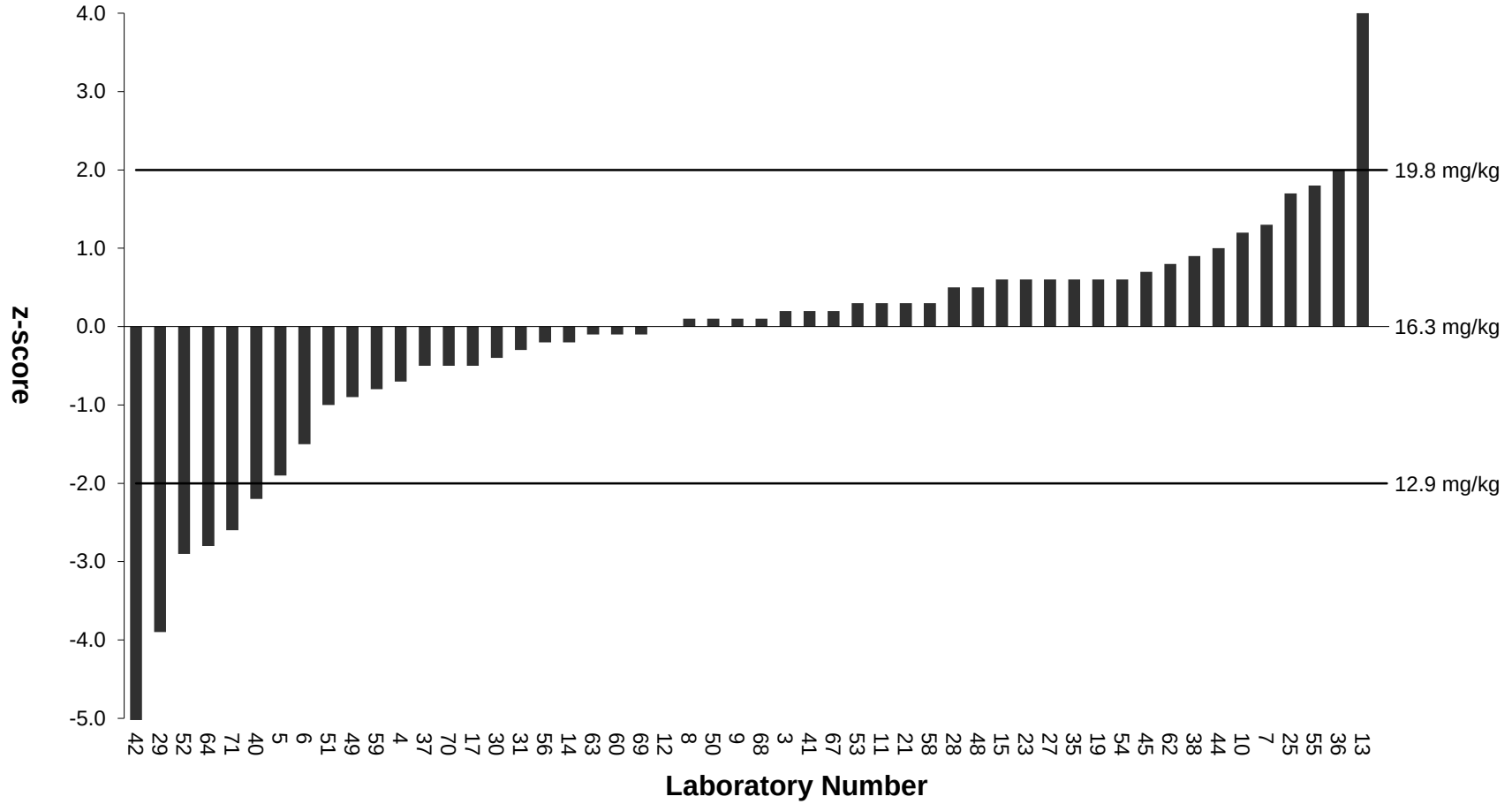

Figure 1: z-Scores for Arsenic (total)

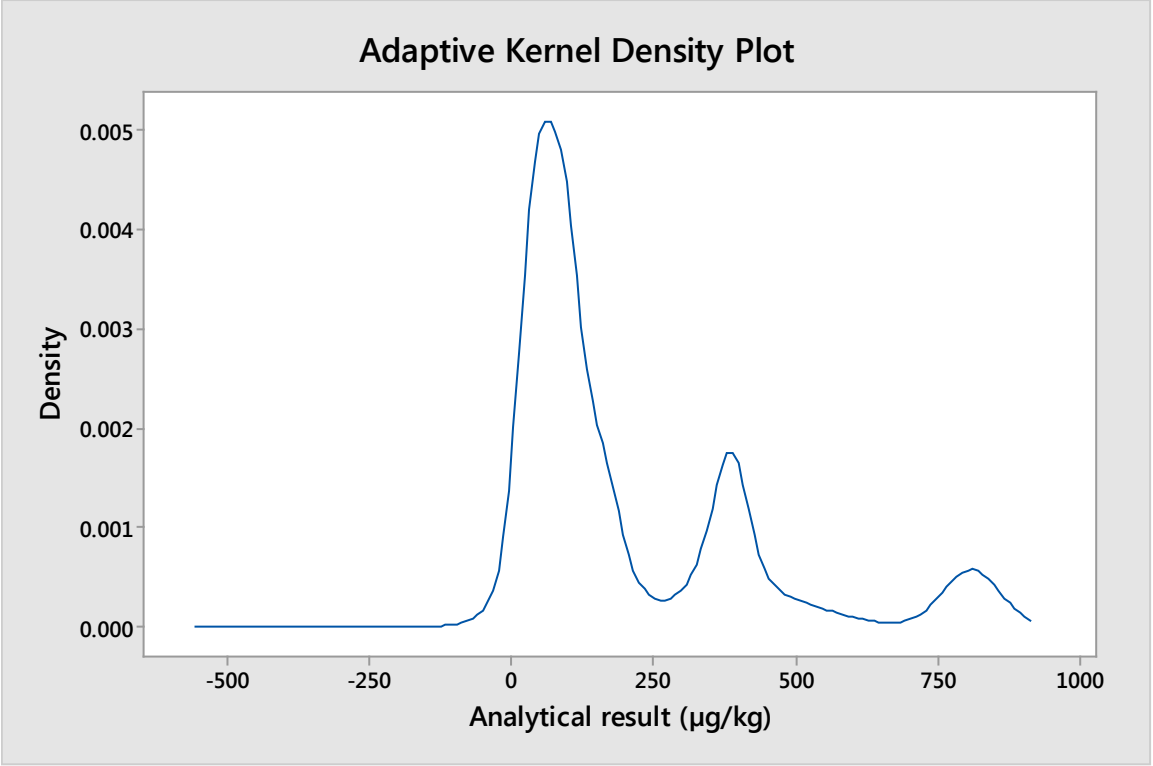

Figure 2: Distribution of Arsenic (inorganic) Data

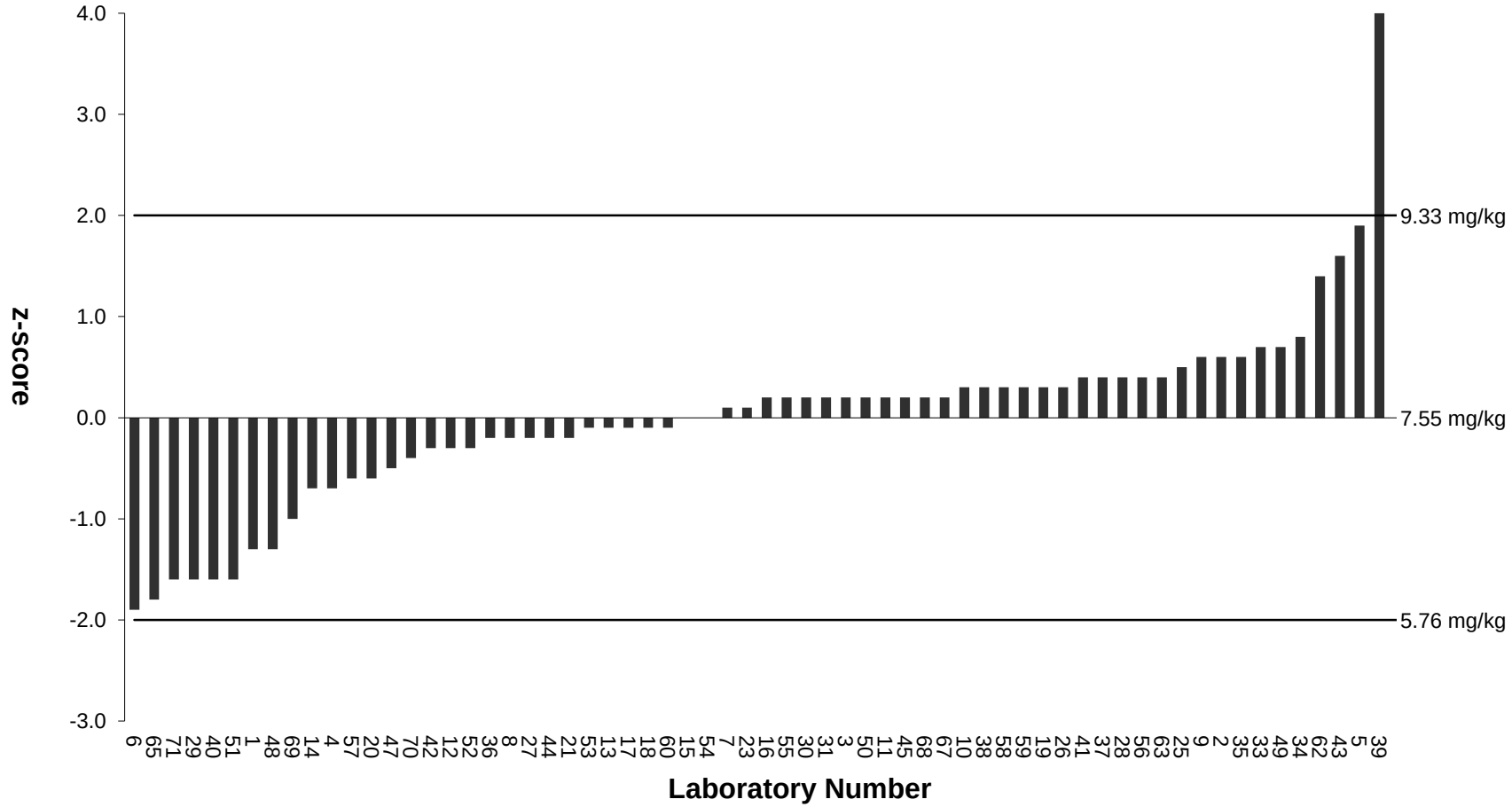

Figure 3: z-Scores for Cadmium

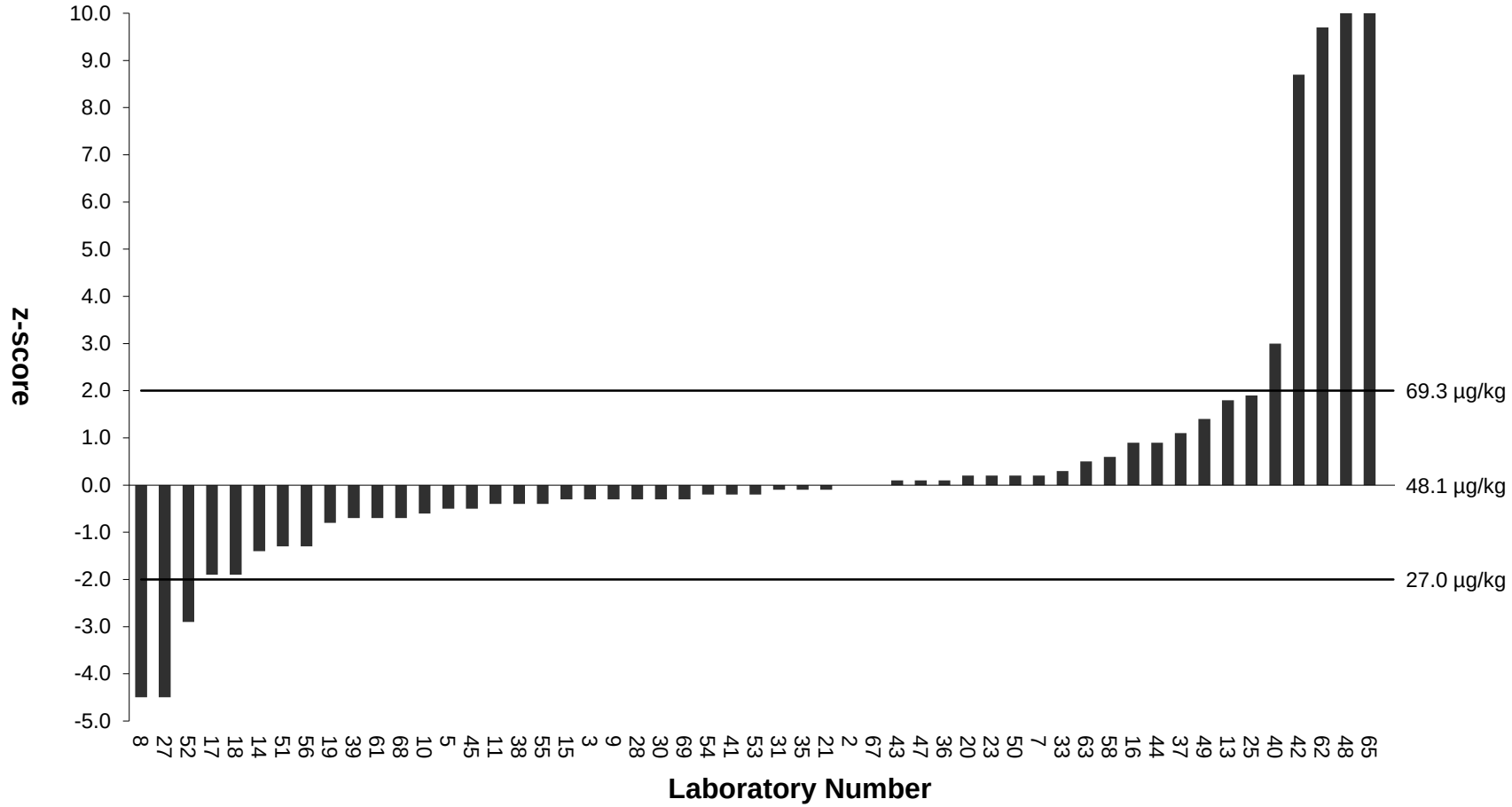

Figure 4: z-Scores for Lead

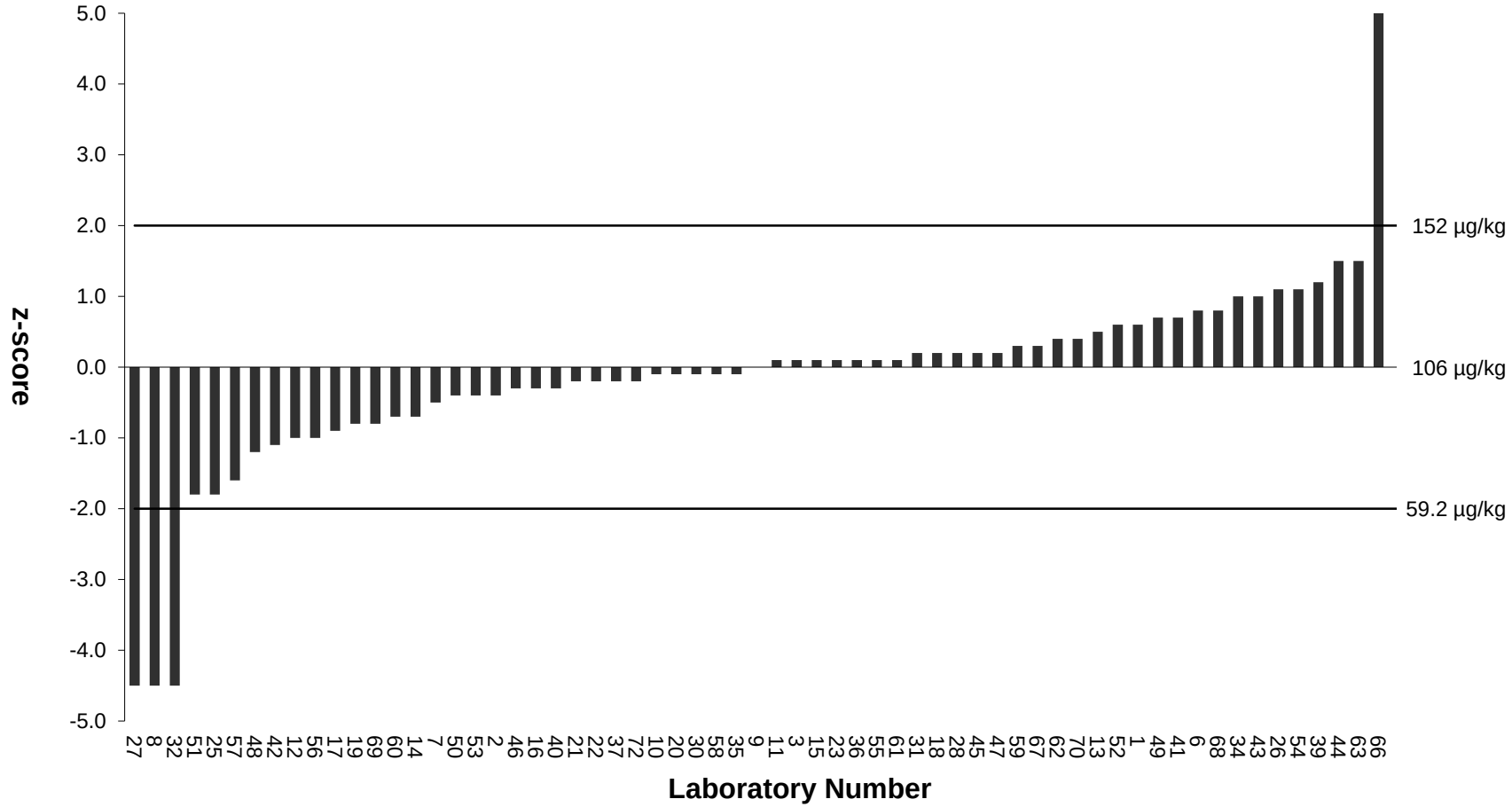

Figure 5: z-Scores for Mercury (total)

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.

Is The Method Used Accredited?	laboratory number
yes	001 002 003 004 006 007 009 010 011 015 016 019 020 027 028 030 033 035 036 037 038 039 040 043 046 048 049 050 052 053 056 057 058 059 060 061 063 066 067 069 070 071 072
no	017 022 024 031 047 051 054 062

What is Your Method Based On?	laboratory number
International Standard	007 010 016 024 028 031 035 036 051 056 058 059 060 066 070 072
National Standard	011 019 020 033 040 043 052 069
Paper Published In An International Journal	054 057
Manufacturer/Kit Instructions/Technical Note	017 039 047
In house method	001 002 003 004 006 009 015 022 027 030 037 038 048 049 050 053 061 062 063 067 071

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 Dominic Anderson (Round Coordinator). Participants with any comments or concerns about this proficiency test should contact:

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