



Fapas[®] – Food Chemistry Proficiency Test Report 07314

Metallic Contaminants in Powdered Brown Rice

July-August 2018

PARTICIPANT LABORATORY NUMBER

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SUMMARY

1. The test material for Fapas[®] – Food Chemistry proficiency test 07314 was dispatched in July 2018. Each participant received a powdered brown rice test material to be analysed for arsenic (total), arsenic (inorganic), cadmium, lead and mercury (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.
3. Results for this proficiency test are summarised as follows:

analyte	assigned value, x_a $\mu\text{g}/\text{kg}$	number of scores, $ z \leq 2$	total number of scores	% $ z \leq 2$
Arsenic (total)	240	87	91	96
Arsenic (inorganic)	189	63	66	95
Cadmium	34.0	95	98	97
Lead	45.6	86	93	92
Mercury (total)	26.2	79	84	94

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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[®] – 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 material was prepared from brown rice flour procured from a retail source.

Arsenic (total), arsenic (inorganic), cadmium and lead were present at natural levels in the test material. Cadmium, lead and mercury were spiked into the test material.

Samples were stored at ambient 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 06 July 2018. Test materials were sent to 118 participants.

3. RESULTS

The instructions for reporting results were as follows:

- Determine the level of arsenic (total), arsenic (inorganic), cadmium, lead and mercury (total) present in the test material, in $\mu\text{g}/\text{kg}$, as received, uncorrected for recovery.

Results were submitted by 112 participants (95%) before the closing date for this test, 16 August 2018.

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 [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 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 all analytes, this procedure was straightforward and the robust mean was chosen as the assigned value.

The assigned values for all analytes 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 [8].

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 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 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 [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[®], 2017, Protocol for Proficiency Testing Schemes, Version 6, April 2017, Part 1 – Common Principles.
- 5 Fapas[®], 2017, Protocol for Proficiency Testing Schemes, Version 5, April 2017, Part 2 – Fapas[®] 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

laboratory number	analyte									
	Arsenic (total) assigned value: 240 µg/kg		Arsenic (inorganic) assigned value: 189 µg/kg		Cadmium assigned value: 34.0 µg/kg		Lead assigned value: 45.6 µg/kg		Mercury (total) assigned value: 26.2 µg/kg	
	result	z-score	result	z-score	result	z-score	result	z-score	result	z-score
001	246.3	0.1	179.2	-0.2	32.9	-0.1	44.6	-0.1		
002	208	-0.7			35	0.1	<50		22	-0.7
003	240	0.0	190	0.0	34	0.0	42	-0.4	27	0.1
004	259	0.4	179	-0.3	32.1	-0.3	44.0	-0.2	28.4	0.4
005	234	-0.1	187.4	0.0	33	-0.1	37.3	-0.8	31.7	0.9
006	246	0.1	239	1.3	33	-0.1	42	-0.4	26	0.0
007					33.4	-0.1	43.8	-0.2		
008	247	0.1	174	-0.4	36	0.3	50	0.4	26	0.0
009	237	-0.1			35	0.1	31	-1.5	25	-0.2
010	206	-0.7			33	-0.1	95	4.9	19	-1.3
011							47.8	0.2		
012	232.2	-0.2			33.52	-0.1			21.6	-0.8
013					32.8	-0.2	52.9	0.7		
014	285	0.9	207	0.5	38	0.5	54	0.8	30	0.7
015	240	0.0	212	0.6	36	0.3	48	0.2	28	0.3

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: 240 µg/kg		Arsenic (inorganic) assigned value: 189 µg/kg		Cadmium assigned value: 34.0 µg/kg		Lead assigned value: 45.6 µg/kg		Mercury (total) assigned value: 26.2 µg/kg	
	result	z-score	result	z-score	result	z-score	result	z-score	result	z-score
016	223	-0.4	185	-0.1	36	0.3	41	-0.5	27	0.1
017	238.86	0.0	165.36	-0.6	34.40	0.1	43.03	-0.3	25.27	-0.2
018					33.4	-0.1	42.0	-0.4		
019	217	-0.5			35	0.1	44	-0.2	27	0.1
020									25.5	-0.1
021	250	0.2	239.60	1.3	34.68	0.1	44	-0.2	25.630	-0.1
022	242.79	0.1			36.01	0.3	52.16	0.6	20.52	-1.0
023					31.4	-0.3	49.0	0.3		
024					32.5	-0.2	41.5	-0.4		
025	224.6	-0.3			34.2	0.0	48.2	0.3	25.2	-0.2
026			185	-0.1						
027					33.1	-0.1	42.6	-0.3		
028	249.00	0.2					49.15	0.3		
029	230	-0.2								
030					31.67	-0.3	41.29	-0.4		

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: 240 µg/kg		Arsenic (inorganic) assigned value: 189 µg/kg		Cadmium assigned value: 34.0 µg/kg		Lead assigned value: 45.6 µg/kg		Mercury (total) assigned value: 26.2 µg/kg	
	result	z-score	result	z-score	result	z-score	result	z-score	result	z-score
031	210	-0.6	180	-0.2	31.8	-0.3	46.0	0.0	25.6	-0.1
032	277	0.8	208	0.5	32.60	-0.2	43.70	-0.2	27.7	0.3
033	276	0.7	164	-0.6	< 30		31	-1.5	0.02	-4.5
034					37.39	0.5	49.85	0.4		
035	228.74	-0.2			46.52	1.7	54.36	0.9	45.58	3.4
036	176	-1.4			32	-0.3	46	0.0	23	-0.6
037	268	0.6	187	0.0	33.9	0.0	43.9	-0.2	25.4	-0.1
038	255.17	0.3			34.11	0.0	42.38	-0.3		
039	260	0.4	205	0.4	37.0	0.4	43.7	-0.2	27.5	0.2
040	238	-0.1	180	-0.2	34.1	0.0	47.1	0.1	26.6	0.1
041	238	-0.1	209	0.5	33.9	0.0	44.5	-0.1	26.9	0.1
042	215	-0.5			30	-0.5	42	-0.4	27.6	0.2
043	267.9	0.6	203.6	0.4	30.7	-0.4	35.8	-1.0	16.9	-1.6
044	253.38	0.3	168.20	-0.5	34.60	0.1	67.55	2.2	26.29	0.0
045	252.04	0.2	202.29	0.4	35.04	0.1	48.17	0.3	28.41	0.4

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: 240 µg/kg		Arsenic (inorganic) assigned value: 189 µg/kg		Cadmium assigned value: 34.0 µg/kg		Lead assigned value: 45.6 µg/kg		Mercury (total) assigned value: 26.2 µg/kg	
	result	z-score	result	z-score	result	z-score	result	z-score	result	z-score
046	236.45	-0.1			34.34	0.0	44.69	-0.1	27.97	0.3
047	245	0.1			42	1.1	98	5.2	62	6.2
048	459.50	4.6			17.03	-2.3	181.27	13.5	27.67	0.3
049	255	0.3	207.4	0.5	35.1	0.2	47	0.1	24.9	-0.2
050	244	0.1			39	0.7	46	0.0	24	-0.4
051	198	-0.9	174	-0.4	38	0.5	92	4.6	31	0.8
052	236.69	-0.1	205.75	0.4	33.34	-0.1	44.28	-0.1	20.21	-1.0
053	229.00	-0.2			30.80	-0.4	46.00	0.0	33.40	1.2
054	235	-0.1	177	-0.3	34.7	0.1	46.5	0.1	22.8	-0.6
055	225.45	-0.3								
056	228	-0.3			33.4	-0.1	52.1	0.6	26.1	0.0
057	243	0.1	196	0.2	34.6	0.1	48.5	0.3	26.3	0.0
058	325.00	1.8	212.50	0.6	35.60	0.2	24.11	-2.1	5.61	-3.6
059			217	0.7						
060	240	0.0			34.5	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: 240 µg/kg		Arsenic (inorganic) assigned value: 189 µg/kg		Cadmium assigned value: 34.0 µg/kg		Lead assigned value: 45.6 µg/kg		Mercury (total) assigned value: 26.2 µg/kg	
	result	z-score	result	z-score	result	z-score	result	z-score	result	z-score
061	250	0.2	174	-0.4	35.6	0.2	53.8	0.8	27.0	0.1
062	256.2	0.3			33.3	-0.1	37.09	-0.9	28.8	0.4
063	253.3	0.3	206.7	0.5	32.2	-0.2	47.7	0.2	26.9	0.1
064	234	-0.1	176	-0.3	34.4	0.1	46.0	0.0	25.4	-0.1
065	242.7	0.0	167	-0.6	34.3	0.0	45.4	0.0	26.5	0.0
066	235	-0.1	180	-0.2	31.4	-0.3	41.4	-0.4	27.4	0.2
067	220	-0.4	184	-0.1	30	-0.5	35	-1.1	27	0.1
068	235	-0.1	193	0.1						
069					35	0.1				
070	189.7	-1.1			27.9	-0.8	58.9	1.3		
071	<2500		179.4	-0.2	43.8	1.3	<500		<2500	
072	233	-0.2	201	0.3	<50		<50		23.5	-0.5
073	226	-0.3	181	-0.2	32.0	-0.3	42.4	-0.3	28.1	0.3
074	230.4	-0.2	202.8	0.4	33.8	0.0	43.2	-0.2	32.6	1.1
075	247	0.1	175	-0.4	35.1	0.2	50.1	0.4	25.7	-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: 240 µg/kg		Arsenic (inorganic) assigned value: 189 µg/kg		Cadmium assigned value: 34.0 µg/kg		Lead assigned value: 45.6 µg/kg		Mercury (total) assigned value: 26.2 µg/kg	
	result	z-score	result	z-score	result	z-score	result	z-score	result	z-score
076	251	0.2					33.6	-1.2	28.4	0.4
077	216	-0.5	193	0.1	32.5	-0.2	42.8	-0.3	22.0	-0.7
078	229.29	-0.2	208.97	0.5	32.35	-0.2	47.49	0.2	25.03	-0.2
079			0.166	-4.9						
080	225.37	-0.3	186.75	-0.1	31.82	-0.3	67.69	2.2	87.00	10.5
081	251	0.2	192	0.1	36.0	0.3	49.0	0.3	26.1	0.0
082	251	0.2			37.5	0.5	45.6	0.0	22.4	-0.7
083	246	0.1	181	-0.2	35.5	0.2	52.0	0.6	27.1	0.2
084	238	-0.1	176	-0.3	33.6	-0.1	47.0	0.1	26.0	0.0
085	231	-0.2	215	0.7	38.4	0.6	42.4	-0.3	21.8	-0.8
086	238	-0.1	174	-0.4	35.6	0.2	50.0	0.4	25.0	-0.2
087	318.55	1.6	190.16	0.0	34.88	0.1	33.41	-1.2	27.66	0.2
088	233	-0.2	167	-0.6	35.3	0.2	41.4	-0.4	24.9	-0.2
089	249.50	0.2			35.75	0.2	49.25	0.4	26.75	0.1
090	234.7	-0.1	180	-0.2	26	-1.1	65	1.9	27	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: 240 µg/kg		Arsenic (inorganic) assigned value: 189 µg/kg		Cadmium assigned value: 34.0 µg/kg		Lead assigned value: 45.6 µg/kg		Mercury (total) assigned value: 26.2 µg/kg	
	result	z-score	result	z-score	result	z-score	result	z-score	result	z-score
091					30	-0.5				
092			172	-0.4						
093			161.92	-0.7	36.37	0.3	42.52	-0.3	27.21	0.2
094	222.07	-0.4			30.6	-0.5	44.2	-0.1	24.8	-0.2
095	243	0.1	204	0.4	32.1	-0.3	53.2	0.8	23.7	-0.4
096	235.7	-0.1	179.8	-0.2	36.0	0.3	48.2	0.3	26.5	0.0
097	245	0.1	185	-0.1	35	0.1	47	0.1	26	0.0
098	226.73	-0.3	183.19	-0.1	34.69	0.1	41.09	-0.5	26.12	0.0
099	41.56	-4.2			28.73	-0.7	36.37	-0.9		
100	249	0.2			34	0.0	48	0.2	29	0.5
101			94.13	-2.4						
102	179	-1.3	175	-0.4	15	-2.5	not detect		27	0.1
103	250	0.2	160	-0.7	37.2	0.4	41.7	-0.4	24.3	-0.3
104	264	0.5			34	0.0	43	-0.3	25	-0.2
105	370	2.7			49	2.0				

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: 240 µg/kg		Arsenic (inorganic) assigned value: 189 µg/kg		Cadmium assigned value: 34.0 µg/kg		Lead assigned value: 45.6 µg/kg		Mercury (total) assigned value: 26.2 µg/kg	
	result	z-score	result	z-score	result	z-score	result	z-score	result	z-score
106					36	0.3	43	-0.3	32	1.0
107	253	0.3	206	0.4	39	0.7	51	0.5	38	2.0
108	355	2.4	265	2.0	13	-2.8	44	-0.2	35	1.5
109	256	0.3	195	0.2	33.3	-0.1	44.0	-0.2	24.5	-0.3
110	220	-0.4			32	-0.3	42	-0.4	23	-0.6
111	258	0.4	7.6	-4.7	33.3	-0.1	27.4	-1.8	23.6	-0.5
112					27.5	-0.9	<LOQ			

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

Table 2: Participants' Comments

laboratory number	comments
006	DMA: 42 µg/kg
036	The test technique is AAS and the determination is uncorrected for recovery.
041	Inorganic Arsenic is determined by In-house method FG023
043	The testing equipment is ICP, and the sample is treated by wet digestion.
047	results tested by Microwave digestion / ICP-MS
077	Arsenic (inorganic):GB5009.11-2014

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

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
Arsenic (total)	91	240	2	Horwitz [8] 47.7
Arsenic (inorganic)	64	189	2	Horwitz [8] 38.8
Cadmium	98	34.0	0.3	Horwitz [8] 7.48
Lead	93	45.6	0.6	Horwitz [8] 10.0
Mercury (total)	83	26.2	0.3	Horwitz [8] 5.77

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)	87	91	96
Arsenic (inorganic)	63	66	95
Cadmium	95	98	97
Lead	86	93	92
Mercury (total)	79	84	94

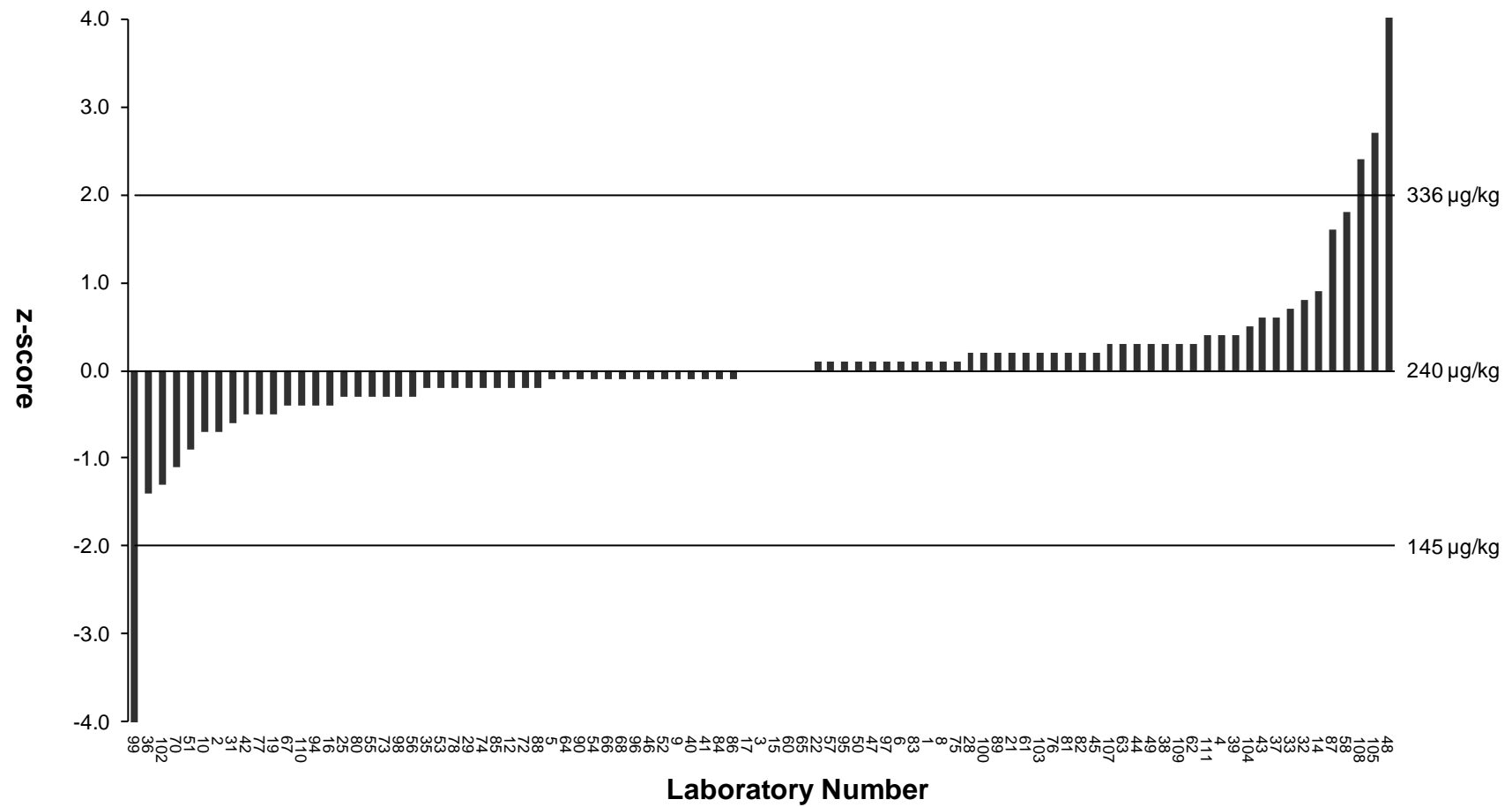


Figure 1: z-Scores for Arsenic (total)

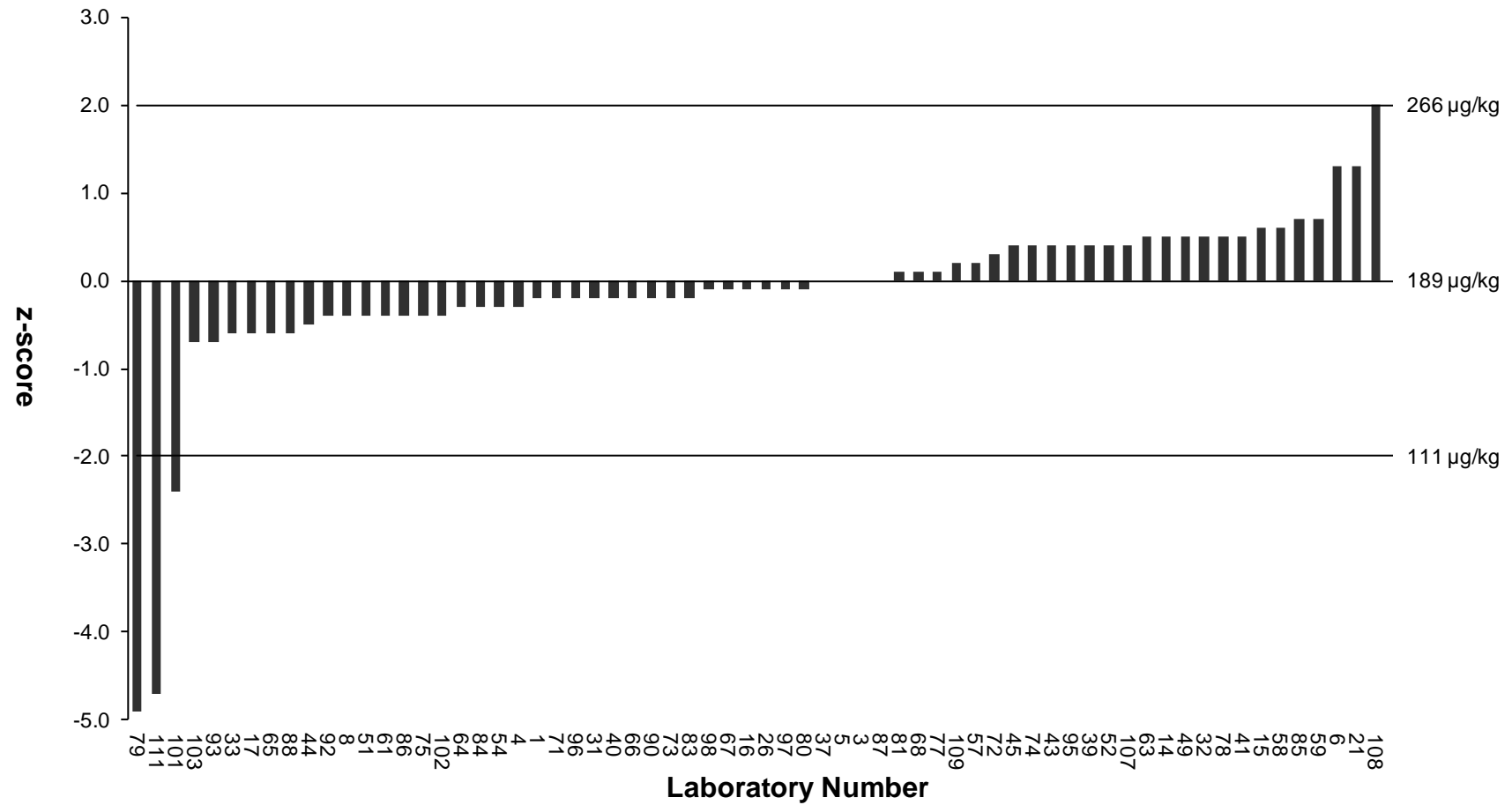


Figure 2: z-Scores for Arsenic (inorganic)

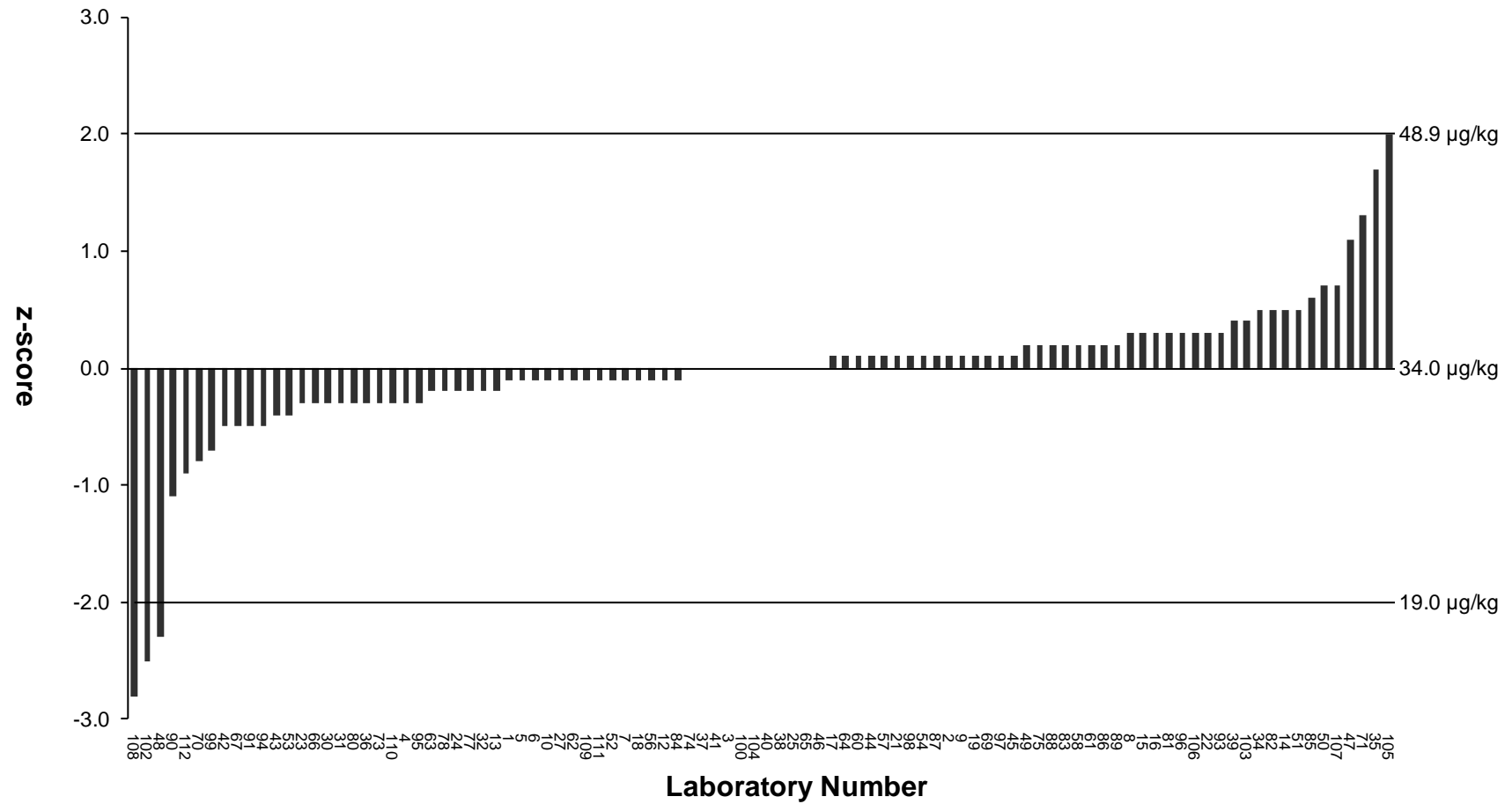


Figure 3: z-Scores for Cadmium

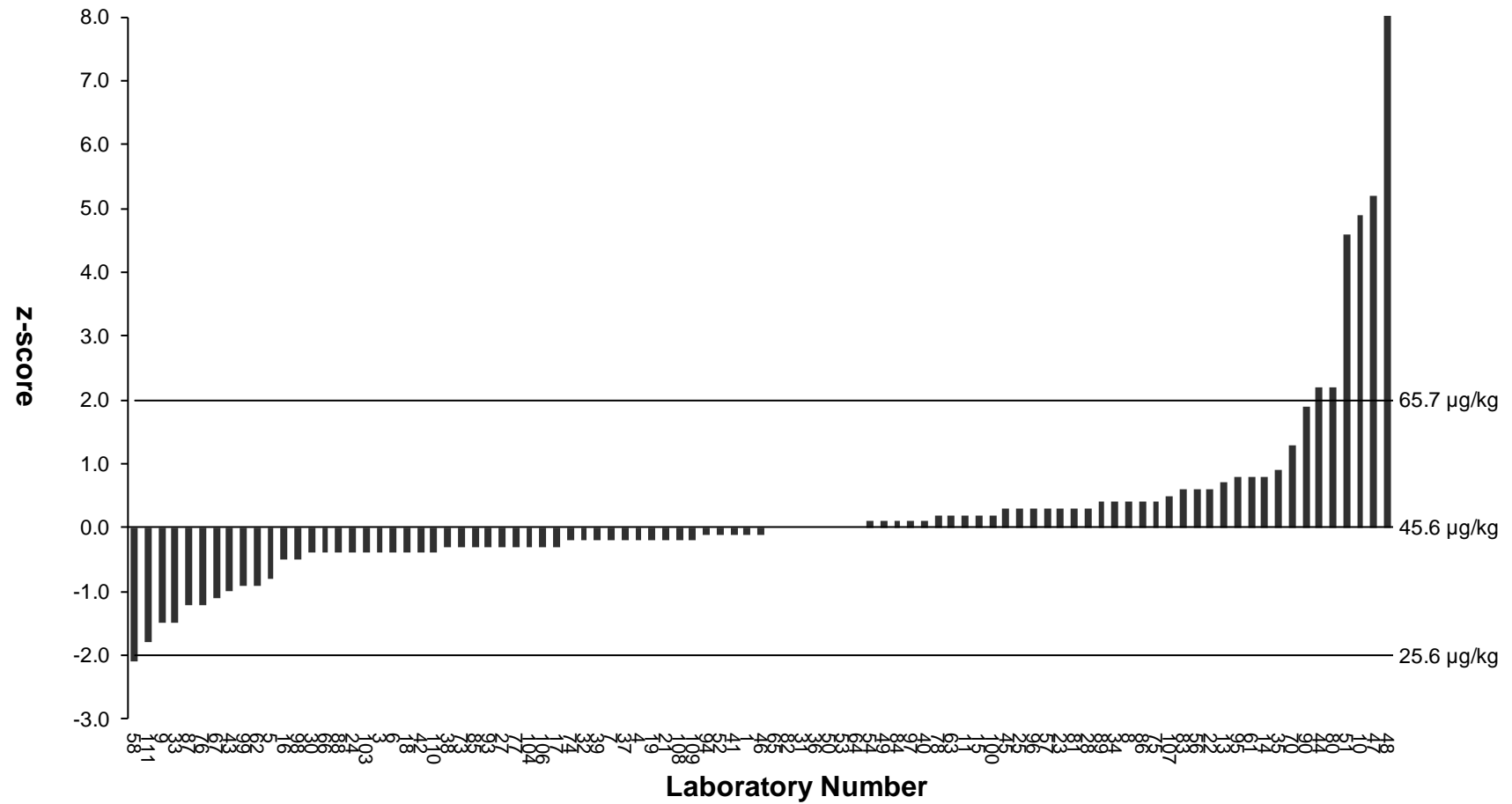


Figure 4: z-Scores for Lead

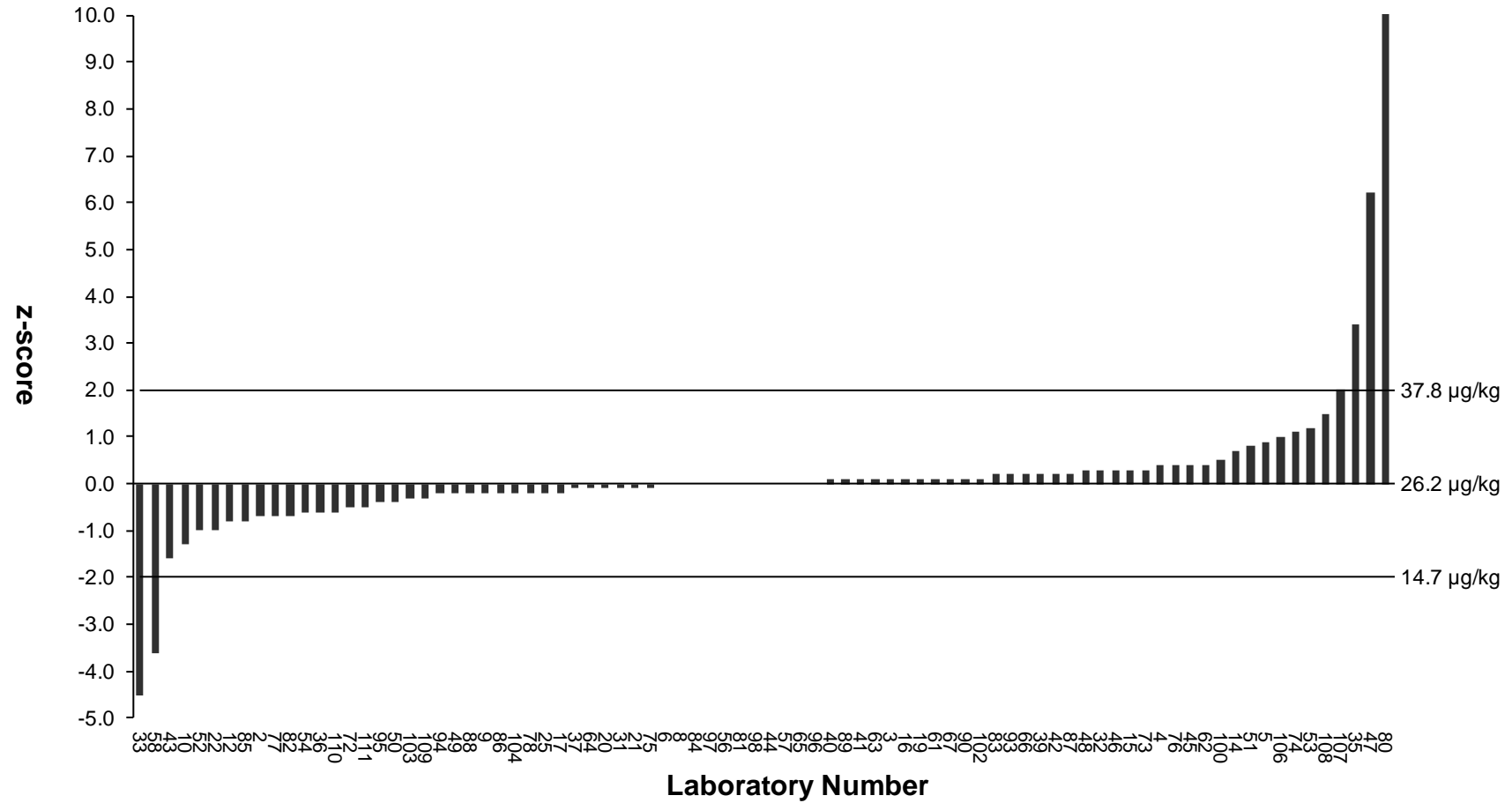


Figure 5: z-Scores for Mercury (total)

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.

Arsenic (total)

Sample Weight (g)	laboratory number
<1	002 003 004 008 014 016 022 028 029 032 035 036 037 039 040 044 045 046 052 053 055 056 057 058 063 065 066 067 070 072 073 074 076 078 081 086 088 089 094 096 097 103 108 111
≥1 - <2	001 017 038 042 043 047 050 051 064 071 080 083 085 087 095 098 105
≥2 - <5	010
≥10 - <25	102

Sample Preparation	laboratory number
dry ashing	010 099 108
microwave digestion	001 002 003 004 008 014 016 017 022 028 029 032 035 036 037 038 039 040 042 044 045 046 047 050 051 052 053 055 056 057 058 063 064 066 070 072 073 078 080 086 087 088 089 094 096 098 103 105 111
reduction As(V) - As(III)	043 076 108
wet digestion	043 067 071 074 076 081 083 085 095 097 102 108

Sample Preparation Reagents Used	laboratory number
HClO ₄	043 083
HF	096
hydrochloric acid	002 044 051 076 095 098 108
hydrogen peroxide	003 004 008 014 022 037 039 042 056 072 073 076 078 096 102 105
nitric acid	001 002 003 004 008 010 014 017 022 028 029 032 035 037 038 039 040 042 043 044 045 046 047 050 051 052 053 055 056 057 058 063 064 066 067 070 071 072 073 074 076 078 080 081 083 085 086 087 088 089 094 095 096 097 098 099 102 103 105 108 111
sulphuric acid	076 102
ascorbic acid/KI	108
hydrogen peroxide + nitric acid	036

Modifier	laboratory number
magnesium nitrate (Mg(NO ₃) ₂)	010 099 108
palladium (Pd)	058
potassium chloride	064
NO	097
none	045

Determination	laboratory number
AES	071
cold vapour / hydride generation AAS	076 108
cold vapour / hydride generation AFS	036 040 067 081
fluorescence	064 083
graphite furnace AAS	058
hydride generation AAS	010 099 108
cold vapour / hydride generation ICP-MS	066
ICP	043
ICP-MS	001 002 003 004 008 014 017 022 028 029 032 035 037 038 039 042 044 045 046 047 050 051 052 053 055 056 057 063 070 072 073 074 078 080 085 086 087 088 089 094 095 096 097 098 103 111
ICP-OES	105
spectrophotometry	102

Wavelength (nm)	laboratory number
189.042	105
193.7	010 058 067 076 083 099 108
510nm	102
As 193.696	071
N/A	111
NA	042
NO	097

Mass (amu)	laboratory number
25 ml	099
74.12	028
74.9216	045
75	001 002 004 008 014 017 022 032 035 037 039 042 047 050 051 052 053 056 057 066 071 073 074 078 080 085 086 088 094 095 096 098 103 111
91	003
NO	097

Limit of Detection	laboratory number
<0.001	053
≥0.001 - <0.01	028 029 051 052 055 074 086 095 097
≥0.01 - <0.1	003 008 010 017 032 035 036 039 040 046 056 064 066 067 076 081 088 089 094 096
≥0.1 - <1	001 002 045 050 078 085 099 103 105
≥1 - <10	042 058 063 073 098 111
≥10 - <100	004 022 047 071 072 080 083 108
≥100	102

Units of Limit of Detection	laboratory number
micrograms per kilogram (µg/kg)	001 004 022 035 042 045 047 057 058 063 071 072 073 078 080 081 083 085 088 094 097 098 102 103 108 111
micrograms per litre (µg/l)	050 089 099
milligrams per kilogram (mg/kg)	002 003 008 010 017 028 029 032 036 039 040 046 051 052 053 055 056 064 066 067 074 076 086 095 096 105

Arsenic (inorganic)

Sample Weight (g)	laboratory number
<1	003 004 008 014 032 037 039 044 058 063 067 071 073 078 081 085 095 096 097 103 108 111
≥1 - <2	001 017 040 045 051 052 057 066 074 079 080 083 086 087 088 098 101 102
≥2 - <5	064 072

Sample Preparation	laboratory number
dry ashing	108
microwave digestion	003 004 014 052 058 078 080 087
reduction As(V) - As(III)	003 008 032 063 083 097 108
solvent extraction	001 037 039 040 045 066 067 072 073 074 079 085 086 088 096 098 101 102 103 108 111
wet digestion	017 051 081 095 108
block digester	071
digestion by heating block	044
hot water immersion	064
SPE extractie	003

Sample Preparation Reagents Used	laboratory number
hydrochloric acid	003 004 058 071 072 108
hydrogen peroxide	014 017 032 096 079
nitric acid	014 037 039 040 044 045 051 052 057 063 064 066 067 073 074 078 080 081 083 085 086 087 095 096 097 101
0.15M nitric acid	103
25% methanol	111
hydrogen peroxide	014 017 032 096 079
malonic acid	001
water	102

Modifier	laboratory number
NH4NO3	039
palladium (Pd)	058
NO	097
none	045

Determination	laboratory number
cold vapour / hydride generation AAS	008 051
cold vapour / hydride generation AFS	040 073 081
fluorescence	083
graphite furnace AAS	058
HPLC	014 083 096 102
hydride generation AAS	097 108
ICP-MS	003 004 014 057 063 071 074 080 087 096 098 101 102
HPLC+FLUORESCENCE	064
HPLC-ICPMS	067 111
HPLC-ICP-MS	001 032 066 078 085 103
IC-ICPMS	017
IC-ICP-MS	039
LC	087
LC-ICP-MS	045 088

Wavelength (nm)	laboratory number
193.7	051 058 083 108
N/A	111
NO	097

Mass (amu)	laboratory number
75	001 004 014 032 037 039 045 057 066 067 071 074 078 080 085 088 095 096 102 103 111
91	003 098
NO	097

Limit of Detection	laboratory number
≥0.001 - <0.01	017 032 074 095 096 097
≥0.01 - <0.1	003 008 039 040 052 064 066 079 081 086 102
≥0.1 - <1	001 088
≥1 - <10	044 045 058 063 078 085 098 103 111
≥10 - <100	004 067 071 072 073 080 083 108

Units of Limit of Detection	laboratory number
micrograms per kilogram (µg/kg)	001 004 044 045 051 058 063 067 071 072 073 078 080 081 083 085 088 097 098 103 108 111
milligrams per kilogram (mg/kg)	003 008 017 039 040 052 064 066 074 079 086 095 096 102
milligrams per litre (mg/l)	032

Cadmium

Sample Weight (g)	laboratory number
<1	002 003 004 008 014 022 023 024 032 034 035 036 037 038 039 040 044 045 046 051 053 056 057 058 063 066 067 070 072 073 074 078 081 083 086 088 089 094 096 097 099 102 103 111 112
≥1 - <2	001 007 017 018 027 030 042 047 050 052 064 071 080 085 087 095 098 105
≥2 - <5	010
≥10 - <25	108

Sample Preparation	laboratory number
dry ashing	010 083 108
microwave digestion	001 002 003 004 007 008 014 017 018 022 023 024 027 030 032 034 035 036 037 038 039 040 042 044 045 046 047 050 051 052 053 056 057 058 063 064 066 067 070 071 072 073 078 080 086 087 088 089 094 096 098 099 102 103 105 111 112
solvent extraction	017
wet digestion	074 081 085 095 097

Sample Preparation Reagents Used	laboratory number
HF	096
hydrochloric acid	002 044 051 095 098 108
hydrogen peroxide	003 004 008 014 022 037 039 042 056 072 073 078 096 102 105
nitric acid	001 002 003 004 007 008 010 014 017 018 022 023 024 027 030 032 034 035 037 038 039 040 042 044 046 047 050 051 052 053 056 057 058 063 064 066 067 070 071 072 073 074 078 080 081 083 085 086 087 088 089 094 095 096 097 098 099 102 103 105 111 112
sulphuric acid	010
hydrogen peroxide + nitric acid	036
none	045

Modifier	laboratory number
NH ₄ H ₂ PO ₄	036 058 067 072 073
palladium (Pd)	010 072 099
potassium chloride	064
NO	097
none	045

Determination	laboratory number
flame AAS	108
fluorescence	064
graphite furnace AAS	010 036 040 058 067 072 073 083 099
ICP-MS	001 002 003 004 007 008 014 017 018 022 023 024 027 030 032 034 035 037 038 039 042 044 045 046 047 050 051 052 053 056 057 063 066 070 074 078 080 081 085 086 087 088 089 094 095 096 097 098 102 103 111
ICP-OES	071 105 112

Wavelength (nm)	laboratory number
226.502	071
228.8	010 058 067 073 083 099 108
228.802	105
NA	042
NO	097

Mass (amu)	laboratory number
110.9042	045
111	001 002 008 014 017 018 030 032 034 037 039 047 050 052 053 056 057 066 074 078 080 085 088 094 095 096 098 102 103
112	023
114	003 004 035 042 051 086
25ml	099
NO	097

Limit of Detection	laboratory number
<0.001	010 030 053
≥0.001 - <0.01	008 023 036 039 040 051 052 066 074 081 086 095 096 097
≥0.01 - <0.1	002 003 007 032 035 046 050 064 078 088 089 094 102 105 112
≥0.1 - <1	001 018 045 058 085 099 103
≥1 - <10	004 034 042 063 067 073 083 098 108
≥10 - <100	022 047 056 072 080 111
≥100	071

Units of Limit of Detection	laboratory number
micrograms per kilogram (µg/kg)	001 004 007 018 022 030 034 035 042 045 047 056 058 063 067 071 072 073 078 080 081 083 085 088 094 097 098 103 108 111
micrograms per litre (µg/l)	050 089 099
milligrams per kilogram (mg/kg)	002 003 008 010 023 032 036 039 040 046 051 052 053 064 066 074 086 095 096 102 105
milligrams per litre (mg/l)	112

Lead

Sample Weight (g)	laboratory number
<1	002 003 004 008 014 022 023 024 028 032 034 035 036 037 038 039 040 044 045 046 051 053 056 057 058 063 066 067 070 072 073 074 078 081 086 088 089 094 096 097 099 102 103 111 112
≥1 - <2	001 007 017 018 027 030 042 047 050 052 064 071 080 083 085 087 095 098
≥5 - <10	076
≥10 - <25	108

Sample Preparation	laboratory number
dry ashing	076 083 108
microwave digestion	001 002 003 004 007 008 014 017 018 022 023 024 027 028 030 032 034 035 036 037 038 039 040 042 044 045 046 047 050 051 052 053 056 057 058 063 064 066 067 072 073 078 080 086 087 088 089 094 096 098 099 102 103 111 112
wet digestion	074 081 085 095 097

Sample Preparation Reagents Used	laboratory number
HF	096
hydrochloric acid	002 044 051 095 098 108
hydrogen peroxide	003 004 008 014 022 037 039 042 056 072 078 096 102
nitric acid	001 002 003 004 007 008 014 017 018 022 023 024 027 028 030 032 034 035 037 038 039 040 042 044 045 046 047 050 051 052 053 056 057 058 063 064 066 067 070 072 073 074 076 078 080 081 083 085 086 087 088 089 094 095 096 097 098 099 102 103 111 112
sulphuric acid	076
hydrogen peroxide + nitric acid	036

Modifier	laboratory number
magnesium nitrate (Mg(NO ₃) ₂)	083
NH ₄ H ₂ PO ₄	036 058 072 076 083
palladium (Pd)	072 099
potassium chloride	064
NO	097
none	045

Determination	laboratory number
flame AAS	108
fluorescence	064
graphite furnace AAS	036 040 058 072 076 083 099
ICP-MS	001 002 003 004 007 008 014 018 022 023 024 027 028 030 032 034 035 037 038 039 042 044 045 046 047 050 051 052 053 056 057 063 066 067 070 073 074 078 080 081 085 086 087 088 089 094 095 096 097 098 102 103 111
ICP-OES	112

Wavelength (nm)	laboratory number
217	108
283.3	058 076 083 099
N/A	111
NA	042
NO	097

Mass (amu)	laboratory number
206	008 035 080
206 + 207 + 208	004
206, 207, 208	042
206/207/208	050
206+207+208	003 056 096
207.2	028
207.9766	045
207+208+209	014
208	001 002 017 018 023 030 032 034 037 047 051 052 053 057 066 067 073 074 078 085 086 088 094 095 098 102
25ml	099
NO	097
Sum of 206, 207, 208	103
Sum(206, 207, 208)	039

Limit of Detection	laboratory number
<0.001	030 053
≥0.001 - <0.01	008 039 051 074 095 097
≥0.01 - <0.1	002 003 007 023 028 032 035 036 040 046 052 064 066 081 086 088 089 094 096 102 112
≥0.1 - <1	001 018 042 045 050 076 085 099
≥1 - <10	034 058 063 078 098 111
≥10 - <100	004 022 047 056 067 072 073 080 083 108

Units of Limit of Detection	laboratory number
micrograms per kilogram (µg/kg)	001 004 007 018 022 030 034 035 042 045 047 056 058 063 067 072 073 078 080 083 085 088 094 097 098 108 111
micrograms per litre (µg/l)	050 089 099
milligrams per kilogram (mg/kg)	002 003 008 023 028 032 036 039 040 046 051 052 053 064 066 074 076 086 095 096 102
milligrams per litre (mg/l)	112

Mercury (total)

Sample Weight (g)	laboratory number
<1	002 003 004 008 014 020 022 032 035 036 037 039 040 042 044 045 046 052 053 056 057 058 063 066 067 072 073 074 078 081 085 086 088 089 094 096 097 102 103 111
≥1 - <2	010 017 047 050 051 064 076 080 083 087 095 098
≥2 - <5	108

Sample Preparation	laboratory number
microwave digestion	002 003 008 014 017 022 032 035 036 039 040 044 046 047 050 051 052 053 056 057 058 064 066 067 072 073 080 083 086 087 089 094 096 098 111
wet digestion	074 076 081 095 097 103 108
DIRECT MEASUREMENT	042
DMA	063
no sample preparation	004
none	045
without preparation	078

Sample Preparation Reagents Used	laboratory number
HClO ₄	103
HF	096
hydrochloric acid	002 042 044 051 095 098 108
hydrogen peroxide	003 008 014 022 039 056 072 073 096
nitric acid	002 003 008 010 014 017 022 032 035 039 040 044 046 047 050 051 052 053 056 057 058 064 067 072 073 076 080 081 083 085 086 087 089 094 095 096 097 098 103 108 111
sulphuric acid	010 103 108
-	004
hydrogen peroxide + nitric acid	036
none	045

Modifier	laboratory number
palladium (Pd)	010
potassium chloride	064
SnCl ₂	056 073 076
NO	097
none	045

Determination	laboratory number
automated mercury analyser	003 004 020 022 037 042 045 073 078 102
cold vapour / hydride generation AAS	056 058 076 089 096 103 108
cold vapour / hydride generation AFS	036 040 052 066 067 081
fluorescence	064 083
graphite furnace AAS	010
ICP-MS	002 014 017 032 035 039 044 046 047 050 051 053 057 074 080 086 087 094 097 098 111
spectrophotometry	085

Wavelength (nm)	laboratory number
228.8	010
253,7	056
253.1	067
253.625	045
253.65	004 042
253.7	003 052 058 066 073 076 083 085 095 096 103 108
253.7nm	102
254	020 022 078
N/A	111
NO	097

Mass (amu)	laboratory number
201	014 074 080 098
202	002 008 017 032 035 039 047 050 051 053 057 086 094
NO	097

Limit of Detection	laboratory number
<0.001	053 074
≥0.001 - <0.01	008 032 036 039 040 051 052 056 086 095 096 097
≥0.01 - <0.1	002 003 010 035 046 064 066 067 076 078 081 088 094 102
≥0.1 - <1	020 045 050 085 089 103
≥1 - <10	004 042 058 073 083 098 111
≥10 - <100	022 047 072 080 108

Units of Limit of Detection	laboratory number
micrograms per kilogram (µg/kg)	004 022 035 042 045 047 056 058 072 073 078 080 081 083 085 088 094 097 098 103 108 111
micrograms per litre (µg/l)	020 050 089
milligrams per kilogram (mg/kg)	002 003 008 010 032 036 039 040 046 051 052 053 064 066 067 074 076 086 095 096 102

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 [4, 5] 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 Abigail Burstein (Round Coordinator). Participants with any comments or concerns about this proficiency test should contact:

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