Fapas® – Food Chemistry Proficiency Test Report 07280

Metallic contaminants in Tomato Paste

February-March 2017

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® – Food Chemistry proficiency test 07280 was dispatched in February 2017. Each participant received a tomato paste test material to be analysed for cadmium (Cd), iron (Fe), lead (Pb) and tin (Sn).

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	units	number of scores, $\|z\| \leq 2$	total number of scores	% $\|z\| \leq 2$
Cadmium	150	µg/kg	48	52	92
Iron	42.6	mg/kg	39	44	89
Lead	199	µg/kg	44	51	86
Tin	111	mg/kg	33	43	77

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 tomato paste from a retail source.

Iron was present at natural levels. Cadmium (Cd) lead (Pb) and tin (Sn) were subsequently 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 [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 14 February 2017. Test materials were sent to 59 participants.

3. RESULTS

The instructions for reporting results were as follows:

- Determine the level of cadmium (Cd) and lead (Pb) present in the test material, in **µg/kg, as received**. Determine the level of iron (Fe) and tin (Sn) present in the test material in **mg/kg, as received.**

Results were submitted by 56 participants (95%) before the closing date for this test, 21 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.

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

No additional participants' comments were submitted.

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 cadmium (Cd), iron (Fe) and lead (Pb), this procedure was straightforward and the robust mean was chosen as the assigned value.

For tin (Sn), the major mode was chosen as the assigned value because the distribution of results was skewed. As seen in previous Fapas® tests, we often see underreporting of tin as seen in Figure 4.

The assigned values for all analytes are shown in Table 2.

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 2.

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 1–4. 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 2.

The number and percentage of z-scores in the range $-2 \leq z \leq 2$ for all analytes are given in Table 3.

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							
	Cadmium assigned value: 150 µg/kg		Iron assigned value: 42.6 mg/kg		Lead assigned value: 199 µg/kg		Tin assigned value: 111 mg/kg	
	result	z-score	result	z-score	result	z-score	result	z-score
001	145.5	-0.1	44.73	0.6	159	-1.0	105.5	-0.6
002	67.47	**-2.6**	39.38	-0.8	135.46	-1.6	94.49	-1.9
003	176	0.8	47	1.1	376	**4.4**	144	**3.8**
004	155	0.2	39.1	-0.9	213	0.3	123	1.4
005	169	0.6			197	0.0		
006	140	-0.3	33.854	**-2.2**	41	**-3.9**	2.468	**-12.4**
007	663.64	**16.1**	34.75	-2.0	648.87	**11.1**	104.12	-0.8
008	153	0.1	47.5	1.3	184	-0.4	66.6	**-5.1**
009	180	0.9	23	**-5.1**	320	**3.0**	108	-0.3
010	147	-0.1	40.6	-0.5	190	-0.2	89.3	**-2.5**
011	150.5	0.0	44.4	0.5	198.1	0.0	106.0	-0.5
012	153	0.1	39.4	-0.8	228	0.7		
013	156.50	0.2	41.35	-0.3	199.00	0.0	118.43	0.9
014	146	-0.1	42.5	0.0	197	0.0	109	-0.2
015					320	**3.0**		
016	136.58	-0.4			194.16	-0.1	76.11	**-4.0**

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

Table 1 (continued): Results and z-Scores

laboratory number	analyte							
	Cadmium assigned value: 150 µg/kg		Iron assigned value: 42.6 mg/kg		Lead assigned value: 199 µg/kg		Tin assigned value: 111 mg/kg	
	result	z-score	result	z-score	result	z-score	result	z-score
017	153.45	0.1	40.02	-0.7	192.84	-0.1	135.79	**2.9**
018	187	1.2	48.4	1.5	186	-0.3	127.8	2.0
019	50.25	**-3.1**	49.83	1.9	136.57	-1.5	98.8	-1.4
020	150	0.0	39.7	-0.7	212	0.3		
021	170	0.6	40.4	-0.6				
022	152.34	0.1	42.48	0.0	189.89	-0.2	113.51	0.3
023	151.98	0.1	46.43	1.0	216.48	0.4	118.96	0.9
024	137	-0.4	40	-0.7	191	-0.2	93	-2.0
025	178	0.9	39.8	-0.7	205	0.2	101	-1.1
026	112.71	-1.2			191.81	-0.2	110.00	-0.1
027	134	-0.5						
028	149.7	0.0	47.0	1.1	215.6	0.4	99.5	-1.3
029	154.50	0.1	44.96	0.6	201.42	0.1	114.36	0.4
030	153	0.1	45	0.6	196	-0.1	110	-0.1
031	175	0.8	42.0	-0.1	191	-0.2	96.6	-1.6
032			77.5	**9.0**			107.2	-0.4

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

Table 1 (continued): Results and z-Scores

laboratory number	analyte							
	Cadmium assigned value: 150 µg/kg		Iron assigned value: 42.6 mg/kg		Lead assigned value: 199 µg/kg		Tin assigned value: 111 mg/kg	
	result	z-score	result	z-score	result	z-score	result	z-score
033	158	0.3	43.2	0.2	212	0.3	110	-0.1
034							113	0.3
035	112.4	-1.2	30.3	**-3.2**	125.3	-1.8	17.2	**-10.7**
036	151	0.0	43.3	0.2	184	-0.4	124	1.5
037	107	-1.3			174	-0.6	113.1	0.3
038	125.91	-0.8	40.9	-0.4	178.9	-0.5	95.65	-1.7
039	152	0.1	46	0.9	192	-0.2	110	-0.1
040	195	1.4			244	1.1		
041	155	0.2			294	**2.3**	118	0.8
042	148.15	-0.1	41.51	-0.3	168.46	-0.8	38.10	**-8.3**
043	133	-0.5	42	-0.1	194	-0.1		
044	149.9	0.0	43.7	0.3	232.6	0.8		
045	147.488	-0.1	36.827	-1.5	171.868	-0.7	85.053	**-2.9**
046	133	-0.5	43	0.1	197	0.0	113	0.3
047	147	-0.1			189	-0.2		
048	110	-1.2			210	0.3		

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

Table 1 (continued): Results and z-Scores

laboratory number	analyte							
	Cadmium assigned value: 150 µg/kg		Iron assigned value: 42.6 mg/kg		Lead assigned value: 199 µg/kg		Tin assigned value: 111 mg/kg	
	result	z-score	result	z-score	result	z-score	result	z-score
049	156.5	0.2	38.91	-0.9	199.5	0.0	93.8	-1.9
050	137	-0.4			144	-1.4		
051	138	-0.4	46.7	1.1	204	0.1	95.6	-1.7
052	148.9	0.0	42.2	-0.1	154.2	-1.1	114.5	0.4
053	158.15	0.3	42.40	0.0	265.47	1.6	113.55	0.3
054	147.0	-0.1	44.09	0.4	205.0	0.2	111.33	0.1
055	712.2	**17.6**	277.05	**60.6**	1073.6	**21.6**	469.1	**41.1**
056			43.960	0.4				

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

Table 2: Assigned Values and Standard Deviations for Proficiency

analyte	data points, *n*	assigned value, x_a	units	uncertainty, *u*	standard deviation for proficiency, σ_p	
Cadmium	52	150	µg/kg	3	Horwitz [7]	31.9
Iron	44	42.6	mg/kg	0.6	Horwitz [7]	3.87
Lead	51	199	µg/kg	5	Horwitz [7]	40.6
Tin	42	111	mg/kg	2	Horwitz [7]	8.72

Table 3: Number and Percentage of z-Scores where $|z| \leq 2$

analyte	number of scores where $\|z\| \leq 2$	total number of scores	% $\|z\| \leq 2$
Cadmium	48	52	92
Iron	39	44	89
Lead	44	51	86
Tin	33	43	77

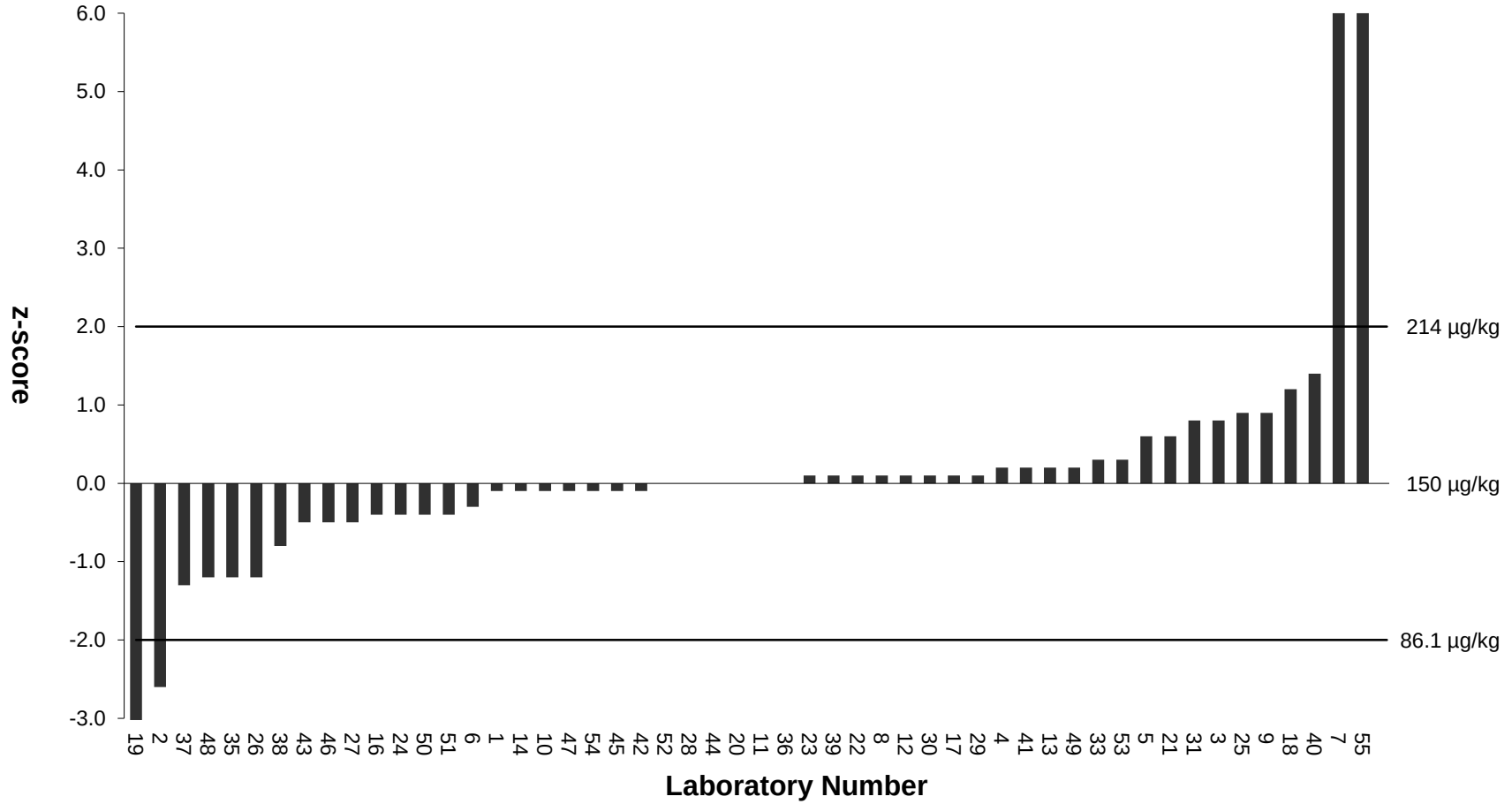

Figure 1: z-Scores for Cadmium

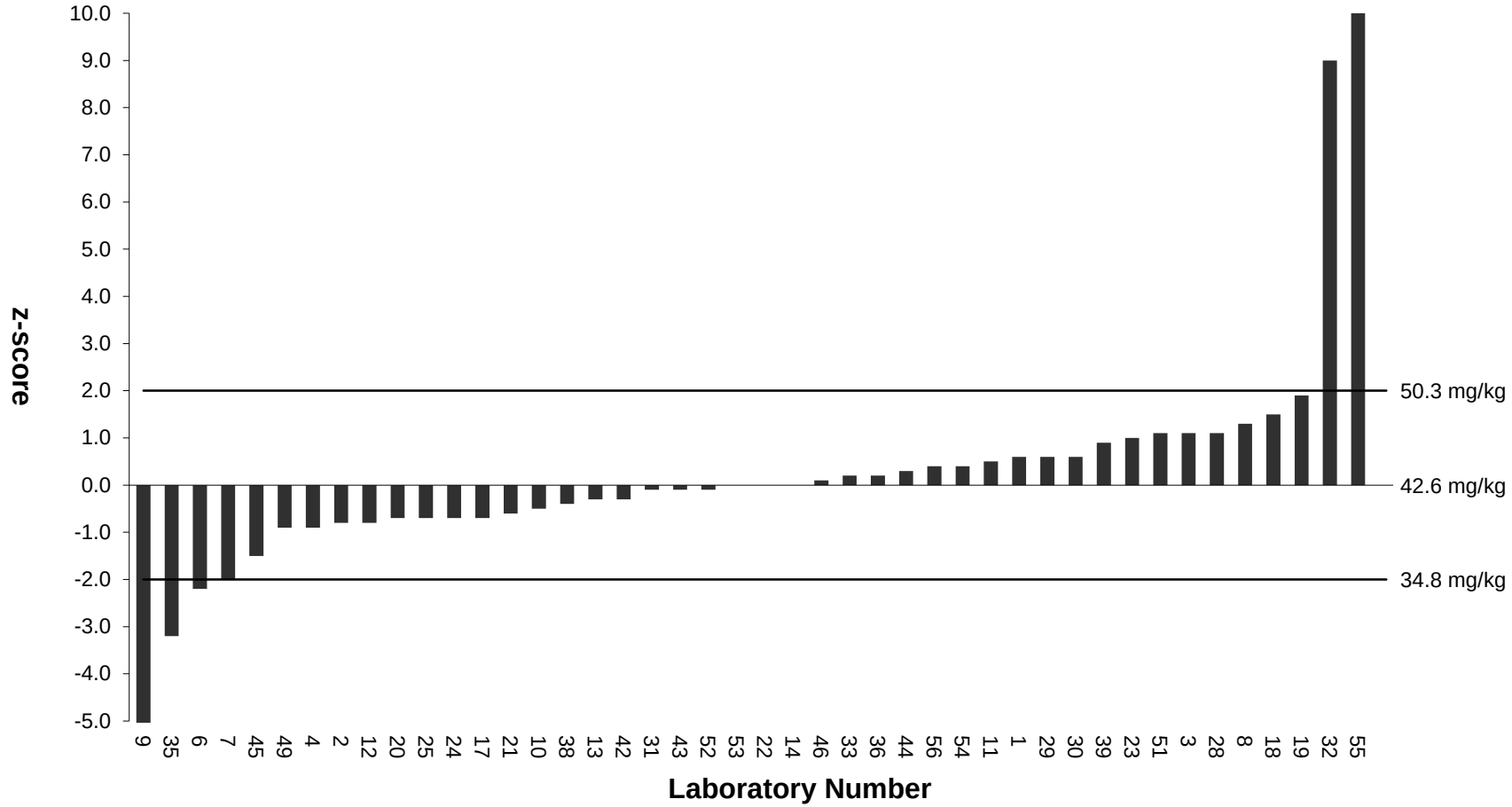

Figure 2: z-Scores for Iron

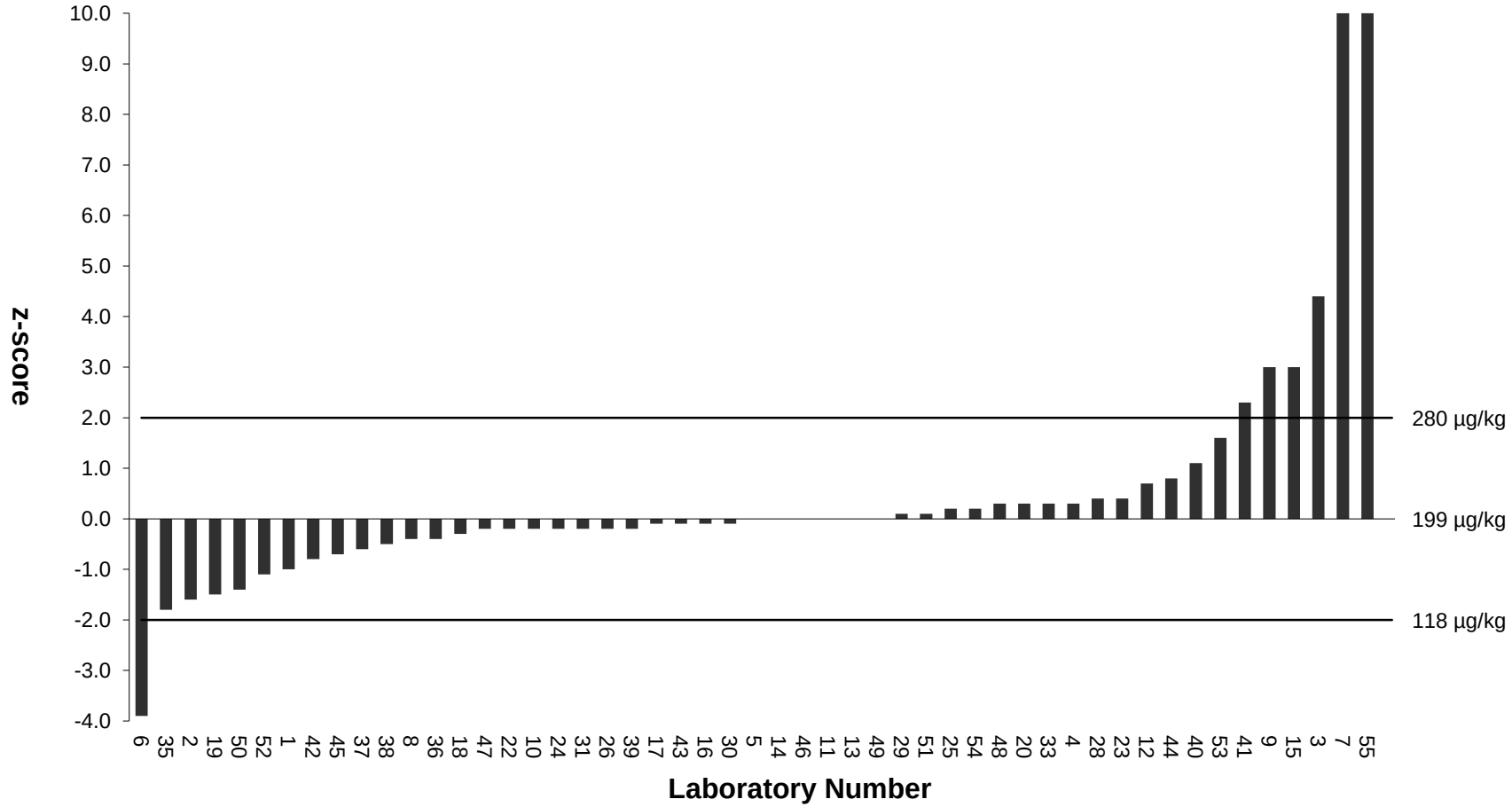

Figure 3: z-Scores for Lead

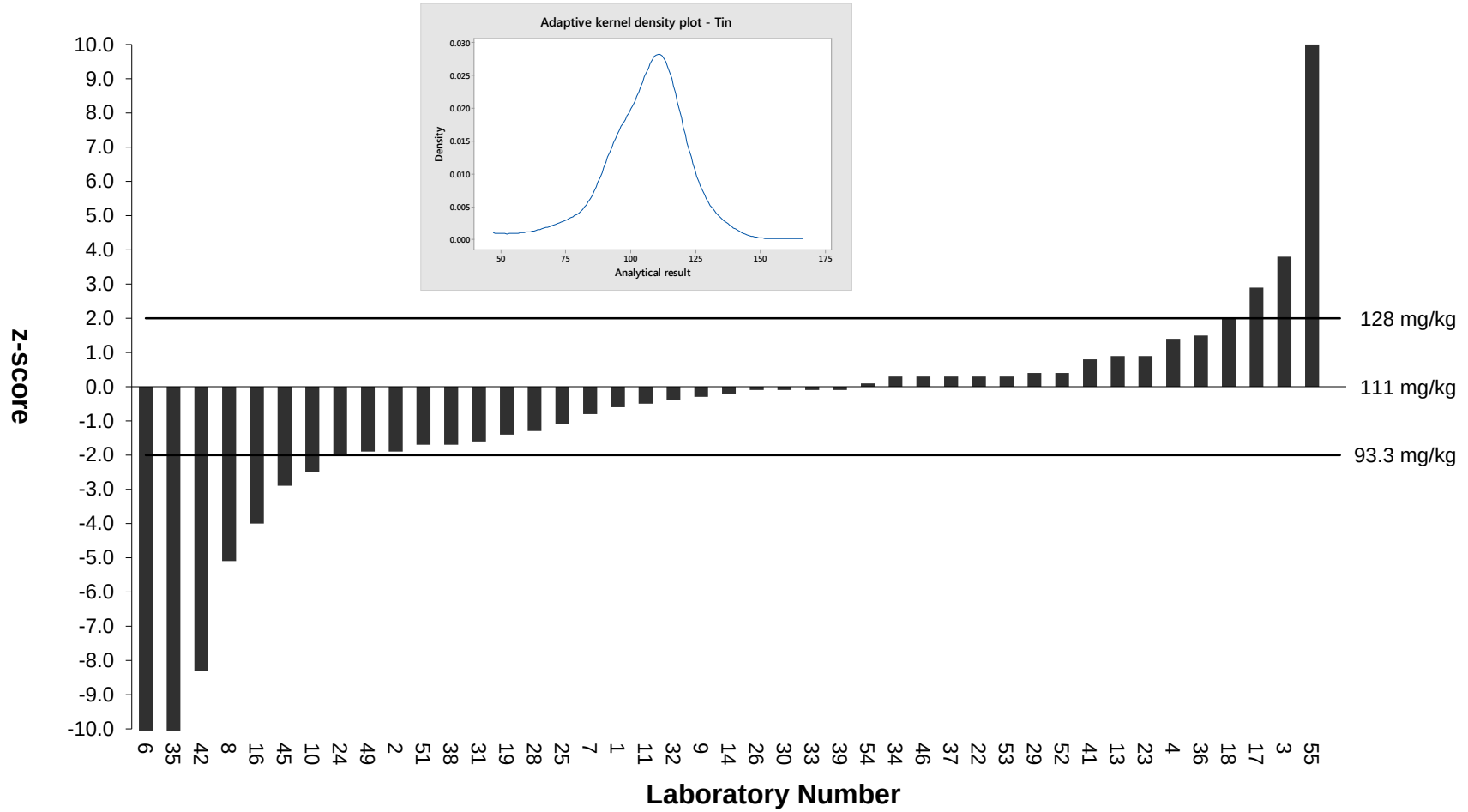

Figure 4: z-Scores for Tin

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	002 004 007 008 009 010 011 012 013 014 017 019 021 022 023 027 028 030 033 035 036 038 039 041 042 045 046 047 048 050
no	015 029 049 056

What is Your Method Based On?	laboratory number
International Standard	007 015 017 022 023 027 028 029 030 036 038 056
National Standard	002 008 021 035 042 048
Paper Published In An International Journal	012
In house method	004 009 010 011 013 014 019 033 039 041 045 046 047 049

Cadmium

Sample Weight (g)	laboratory number
<1	004 010 012 013 014 017 021 022 023 027 028 033 035 036 038 039 041 042 046 049
≥1 - <2	008 011 030 045 047 050
≥2 - <5	007 019
≥10 - <25	009 048

Sample Preparation	laboratory number
dry ashing	007 019 048
microwave digestion	004 008 010 011 012 013 014 017 021 022 023 027 028 030 033 035 036 038 039 041 042 045 046 047 049 050
wet digestion	009

Sample Preparation Reagents Used	laboratory number
hydrochloric acid	007 019 033
hydrogen peroxide	004 010 012 013 017 027 028 033 036 038 039 045 049
nitric acid	004 008 009 010 011 012 013 014 017 019 021 022 023 027 028 030 033 035 036 038 039 041 042 045 046 047 048 049 050

Modifier	laboratory number
magnesium nitrate (Mg(NO3)2)	049
NH4H2PO4	021 049 050
NH4NO3	045
palladium (Pd)	030
DHPA	039
Phosporic Acid	019

Determination	laboratory number
flame AAS	007 048
graphite furnace AAS	019 021 027 030 039 049 050
ICP-MS	004 008 010 011 012 013 014 017 022 023 028 033 036 038 041 045 046 047
ICP-OES	009 035 042

Wavelength (nm)	laboratory number
228,8	049
228.8	007 019 021 027 030 048 050
228.802	042
228.802nm	035

Mass (amu)	laboratory number
\	021
111	004 010 011 012 013 014 017 023 033 041 045
112	049
112.4	019

Limit of Detection	laboratory number
≥0.001 - <0.01	007 012 014 017 019 022 035 047 048
≥0.01 - <0.1	010 011 033 036 039 045
≥0.1 - <1	041
≥1 - <10	009 013 021 030 042
≥10 - <100	004 023 027 028 049

Units of Limit of Detection	laboratory number
micrograms per kilogram (µg/kg)	004 009 013 019 021 022 023 028 030 042 047 049
micrograms per litre (µg/l)	027
milligrams per kilogram (mg/kg)	007 010 011 012 014 017 033 036 039 041 045 048
milligrams per litre (mg/l)	035 046

Iron

Sample Weight (g)	laboratory number
<1	004 010 012 013 014 017 021 022 023 028 035 036 038 042 056
≥1 - <2	008 011 030 039 045 049
≥2 - <5	007 019
≥10 - <25	009 046

Sample Preparation	laboratory number
dry ashing	007 019 049
microwave digestion	004 008 010 011 012 013 014 017 021 022 023 028 030 033 035 036 038 039 042 045 056
solvent extraction	046
wet digestion	009

Sample Preparation Reagents Used	laboratory number
hydrochloric acid	007 011 014 019 033 046 049
hydrogen peroxide	004 010 012 013 017 028 033 036 038 039 045 056
nitric acid	004 008 009 010 011 012 013 014 017 021 022 023 028 030 033 035 036 038 039 042 045 056

Modifier	laboratory number
magnesium nitrate (Mg(NO3)2)	039
NO	021

Determination	laboratory number
flame AAS	007 019 030 046 049 056
graphite furnace AAS	039
ICP-MS	004 008 010 012 013 017 022 028 033 038
ICP-OES	009 011 014 021 023 035 036 042 045

Wavelength (nm)	laboratory number
238.204	011 021 042 045
238.204nm	035
248.3	007 019 030 046 049 056
259	014
259.939	023

Mass (amu)	laboratory number
\	021
55.845	019
56	004 010 012 017 033
57	013

Limit of Detection	laboratory number
≥0.001 - <0.01	017 022 035
≥0.01 - <0.1	009 010 019 021 039
≥0.1 - <1	011 012 014 030 045 046
≥1 - <10	004 013 023 033 036 056
≥10 - <100	042
≥100	028

Units of Limit of Detection	laboratory number
micrograms per kilogram (µg/kg)	013 022 028 042
milligrams per kilogram (mg/kg)	004 009 010 011 012 014 017 019 021 023 030 033 036 039 045 046 056
milligrams per litre (mg/l)	035

Lead

Sample Weight (g)	laboratory number
<1	004 010 012 013 014 017 022 023 028 033 035 036 038 041 042 046 049
≥1 - <2	008 011 015 030 039 045 047 050
≥2 - <5	007 019
≥10 - <25	009 048

Sample Preparation	laboratory number
dry ashing	007 019 048
microwave digestion	004 008 010 011 012 013 014 015 017 022 023 028 030 035 036 038 039 041 042 045 046 047 049 050
wet digestion	009 033
Block Digestion	033

Sample Preparation Reagents Used	laboratory number
hydrochloric acid	007 019 033
hydrogen peroxide	004 010 012 013 015 017 028 033 036 038 039 045 049
nitric acid	004 008 009 010 011 012 013 014 015 017 019 022 023 028 030 033 035 036 038 039 041 042 045 046 047 048 049 050

Modifier	laboratory number
magnesium nitrate (Mg(NO3)2)	039 049
NH4H2PO4	049 050
palladium (Pd)	030
DHPA	039
Phosporic Acid	019

Determination	laboratory number
flame AAS	015 048
graphite furnace AAS	007 019 030 039 049 050
ICP-MS	004 008 010 011 012 013 014 017 022 023 028 033 036 038 041 045 046 047
ICP-OES	009 035 042

Wavelength (nm)	laboratory number
217	048
220.353	042
220.353nm	035
283,3	049
283.3	007 019 030
283.8	050
283nm	015

Mass (amu)	laboratory number
206, 207, 208	004 014
207.2	019
208	010 012 013 017 023 033 041 045 049
208 (206+207)	011

Limit of Detection	laboratory number
≥0.001 - <0.01	007 012 014 017 019 022 035 047
≥0.01 - <0.1	010 011 033 036 039 045 048
≥0.1 - <1	041
≥1 - <10	013 042
≥10 - <100	004 009 023 028 030 049

Units of Limit of Detection	laboratory number
micrograms per kilogram (µg/kg)	004 009 013 019 022 023 028 030 042 047 049
milligrams per kilogram (mg/kg)	007 010 011 012 014 017 033 036 039 041 045 048
milligrams per litre (mg/l)	035

Tin

Sample Weight (g)	laboratory number
<1	004 010 013 014 017 022 023 028 033 035 038 039 041 042 049
≥1 - <2	008 011 030 036
≥2 - <5	007 019 045
≥10 - <25	009 046

Sample Preparation	laboratory number
dry ashing	007 019
microwave digestion	004 008 010 011 013 014 017 022 023 028 030 033 035 036 038 039 041 042 049
solvent extraction	046
wet digestion	009 045

Sample Preparation Reagents Used	laboratory number
hydrochloric acid	007 011 013 014 019 033 038 045 046
hydrogen peroxide	004 010 017 028 033 036 038 039 049
nitric acid	004 008 009 010 011 013 014 017 019 022 023 028 030 033 035 036 038 039 041 042 045 049

Modifier	laboratory number
magnesium nitrate (Mg(NO3)2)	049
palladium (Pd)	049
potassium chloride	019

Determination	laboratory number
flame AAS	007 019 030 046
graphite furnace AAS	049
ICP-MS	004 008 010 013 014 017 022 028 033 038 041
ICP-OES	009 011 023 035 036 039 042 045

Wavelength (nm)	laboratory number
189.927	023 039 042
189.927nm	035
189.989	011
224.6	046
235.5	007 019 030
283.998	045
286.3	049

Mass (amu)	laboratory number
118	004 010 017 033 041
118, 120	014
118.7	019
120	013

Limit of Detection	laboratory number
≥0.001 - <0.01	014 017 035
≥0.01 - <0.1	010 019
≥0.1 - <1	009 011 033 039 041
≥1 - <10	004 007 013 022 036 045
≥10 - <100	023 030 042 046
≥100	028

Units of Limit of Detection	laboratory number
micrograms per kilogram (µg/kg)	013 028 042
milligrams per kilogram (mg/kg)	004 007 009 010 011 014 017 019 022 023 030 033 036 039 041 045 046
milligrams per litre (mg/l)	035

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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