FAPAS® Proficiency Test Report 07166

Metallic Contaminants in Soya Flour

December 2011–January 2012

Prepared and authorised on behalf of FAPAS by

Laura Prenton, Round Co-ordinator

0009 PT

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® proficiency test 07166 was dispatched in December 2011. Each participant received a soya flour test material to be analysed for aluminium, total arsenic, cadmium, lead and total mercury.
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	number of scores, |z| ≤2	total number of scores	% |z| ≤2
aluminium	12.3 mg/kg	34	53	64
total arsenic	711 µg/kg	55	78	71
cadmium	319 µg/kg	74	84	88
lead	503 µg/kg	65	85	76
total mercury	240 µg/kg	56	68	82

4. Surplus test materials are available for sale, see APPENDIX II.
5. Whereas this report has been produced in good faith and in accordance with best industry practice, neither The Food and Environment Research Agency nor the Secretary of State for Environment, Food and Rural Affairs accepts any liability whatsoever as to the application or use of the information contained therein.

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® proficiency testing scheme are available in our protocols [2, 3].

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 to ISO 17043.

The test material was prepared from commercially available soya flour.

Appropriate quantities of arsenic, cadmium, mercury and lead were spiked into the test material. The natural level of aluminium was sufficiently high, so no spiking was required for this element.

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 to ISO 17043.

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

2.3. Dispatch

The start date was 1 December 2011. Test materials were sent to 92 participants.

3. RESULTS

The instructions for reporting results were as follows:

- Determine the levels of aluminium (Al) in mg/kg, as received and total arsenic (As), cadmium (Cd), lead (Pb) and total mercury (Hg) present in the test material, in µg/kg, as received.

Results were submitted by 86 participants (93%) before the closing date for this test, 11 January 2012.

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 for each result. The procedure follows that recommended in the IUPAC International Harmonised Protocol for the Proficiency Testing of Analytical Chemistry Laboratories [4].

Further details on the procedure followed can be found in the relevant protocols [2, 3].

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) 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),

ii) non numerical results i.e. qualitative or semi-quantitative results.

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 [5].

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. ASSESSMENT 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 [4].

6. REFERENCES

1 Adobe Certified Document Services http://www.adobe.com/security/partners_cds.html, accessed 24/05/2011

2 FAPAS, 2010, Protocol for Proficiency Testing Schemes, Part 1 – Common Principles, Version 2, Issued December 2010.

3 FAPAS, 2009, Protocol for Proficiency Testing Schemes, Part 2 – FAPAS®, Revision 2009, Version 1, Issued November 2009.

4 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.

5 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									
	aluminium assigned value 12.3 mg/kg		total arsenic assigned value 711 µg/kg		cadmium assigned value 319 µg/kg		lead assigned value 503 µg/kg		total mercury assigned value 240 µg/kg	
	result mg/kg	z-score	result µg/kg	z-score	result µg/kg	z-score	result µg/kg	z-score	result µg/kg	z-score
001			728	0.1	326	0.1	489	-0.2	235	-0.1
002	8.04	**-3.2**	946	2.0	373	0.9	558	0.6	242	0.0
003			698	-0.1	283	-0.6	435	-0.8	236	-0.1
004			754	0.4	373	0.9	508	0.1	270	0.6
005	13.6	0.9	651	-0.5	307	-0.2	536	0.4	244	0.1
006	14.0	1.2	706	0.0	319	0.0	482	-0.2	230	-0.2
007			860.5	1.3						
008	10.4	-1.4			291	-0.5	448	-0.6		
009			823	0.9	305	-0.2	390	-1.3		
010			470	-2.0	350	0.5	780	**3.1**		
011	12.6	0.2	758	0.4	342	0.4	502	0.0	249	0.2
012			782.28	0.6	414.31	1.6	507.82	0.1		
013	5.61	**-5.0**	882	1.4	418	1.6	449	-0.6	143	-2.0
014	13.56	0.9	763	0.4	364	0.7	493	-0.1	247	0.2
015	8.79	**-2.6**	631.645	-0.7	207.55	-1.8	201.10	**-3.4**	242.05	0.0

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

Table 1 (continued): Results and z-Scores

laboratory number	analyte									
	aluminium assigned value 12.3 mg/kg		total arsenic assigned value 711 µg/kg		cadmium assigned value 319 µg/kg		lead assigned value 503 µg/kg		total mercury assigned value 240 µg/kg	
	result mg/kg	z-score	result µg/kg	z-score	result µg/kg	z-score	result µg/kg	z-score	result µg/kg	z-score
016	14.92	1.9	736.92	0.2	340.68	0.4	523.38	0.2	246.13	0.1
017	6.29	**-4.5**	818	0.9	283	-0.6	466	-0.4	17.5	**-4.7**
018	9.88	-1.8	796	0.7	366	0.8	528	0.3	247	0.2
019	6.1	**-4.6**	300	**-3.4**	4	**-5.2**	500	0.0	280	0.8
020	10.459	-1.4	971.344	**2.2**	367.034	0.8	459.684	-0.5	285.692	1.0
021	12.7	0.3	820	0.9	330	0.2	510	0.1	280	0.8
022			621	-0.7	438	2.0	630	1.4		
023	14.9	1.9	868	1.3	333	0.2	544	0.5	108	**-2.8**
024	16.11	**2.8**	680.85	-0.2	360.48	0.7	1875.72	**15.4**	327.32	1.8
025	11.23	-0.8	799	0.7	349	0.5	586	0.9	247	0.2
026	7.02	**-3.9**	587	-1.0	236	-1.4	568	0.7	351	**2.3**
027			566	-1.2	298	-0.3	486	-0.2	260	0.4
028	13	0.5	430	**-2.3**	340	0.4	500	0.0	240	0.0
029					243	-1.2	462	-0.5		
030					303	-0.3	457	-0.5		

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

Table 1 (continued): Results and z-Scores

laboratory number	analyte									
	aluminium assigned value 12.3 mg/kg		total arsenic assigned value 711 µg/kg		cadmium assigned value 319 µg/kg		lead assigned value 503 µg/kg		total mercury assigned value 240 µg/kg	
	result mg/kg	z-score	result µg/kg	z-score	result µg/kg	z-score	result µg/kg	z-score	result µg/kg	z-score
031	12.3	0.0	767	0.5	288	-0.5	516	0.1	246	0.1
032	9.99	-1.7	1117	**3.4**	336	0.3	562	0.7	256	0.3
033			632.81	-0.6	329.17	0.2	642.80	1.6	245.14	0.1
034	27.64	**11.3**	677.0	-0.3	299.9	-0.3	456.3	-0.5	229.4	-0.2
035			505	-1.7	321	0.0	503	0.0	339	**2.1**
036			765.4	0.5	312.0	-0.1	464.1	-0.4	225.4	-0.3
037			338	**-3.1**	232	-1.4	283	**-2.5**	230	-0.2
038					259	-1.0	438	-0.7	230	-0.2
039	12.16	-0.1	1.13	**-5.9**	0.390	**-5.3**	0.588	**-5.6**	0.276	**-5.0**
040	8.88	**-2.6**	215	**-4.1**	240	-1.3	130	**-4.2**	240	0.0
041	65.9	**39.6**	624	-0.7	314	-0.1	1402	**10.1**	289	1.0
042			400	**-2.6**	345	0.4	465	-0.4	227	-0.3
043			677	-0.3	266	-0.9	489	-0.2		
044	12.5	0.1	736	0.2	374	0.9	493	-0.1	264	0.5
045	14.62	1.7	555	-1.3	240	-1.3	495	-0.1		

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

Table 1 (continued): Results and z-Scores

laboratory number	analyte									
	aluminium assigned value 12.3 mg/kg		total arsenic assigned value 711 µg/kg		cadmium assigned value 319 µg/kg		lead assigned value 503 µg/kg		total mercury assigned value 240 µg/kg	
	result mg/kg	z-score	result µg/kg	z-score	result µg/kg	z-score	result µg/kg	z-score	result µg/kg	z-score
046			0.2	**-5.9**	0.26	**-5.3**	0.40	**-5.6**	0.003	**-5.0**
047	9.63	-2.0	864	1.3	336	0.3	420	-0.9	198	-0.9
048	13.4	0.8	879	1.4	368	0.8	536	0.4	263	0.5
049	14.48	1.6	940.00	1.9	325.00	0.1	530.00	0.3	229.00	-0.2
050			682.55	-0.2	333.91	0.3	475.76	-0.3		
051	13.56	0.9	5357	**38.8**	409.7	1.5	1674	**13.1**	309	1.5
052			837	1.1	307	-0.2	230	**-3.1**	258	0.4
053			526.31	-1.5	295.03	-0.4	447.54	-0.6	193.97	-1.0
054	12.1	-0.2	820	0.9	350	0.5	520	0.2	174	-1.4
055			460.1	**-2.1**	284.1	-0.6	293.0	**-2.4**	232.1	-0.2
056	19.2	**5.1**	9.28	**-5.9**	179.5	**-2.3**	636	1.5		
057	16.86	**3.3**	1076.7	**3.1**	349.0	0.5	548.4	0.5	207.1	-0.7
058					346	0.5	472	-0.3	241	0.0
059			582.28	-1.1	390.36	1.2	1601.51	**12.3**	132.02	**-2.3**
060	10.82	-1.1	919.90	1.7	387.90	1.1	153.85	**-3.9**	156.00	-1.8

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

Table 1 (continued): Results and z-Scores

laboratory number	analyte									
	aluminium assigned value 12.3 mg/kg		total arsenic assigned value 711 µg/kg		cadmium assigned value 319 µg/kg		lead assigned value 503 µg/kg		total mercury assigned value 240 µg/kg	
	result mg/kg	z-score	result µg/kg	z-score	result µg/kg	z-score	result µg/kg	z-score	result µg/kg	z-score
061	11.6	-0.5	653	-0.5	323	0.1	502	0.0	251	0.2
062			217.2	**-4.1**	274.4	-0.7	430.3	-0.8	258.6	0.4
063	13.9	1.2	826	1.0	304	-0.2	583	0.9	275	0.7
064	15.43	**2.3**	ND		460	**2.3**	236	**-3.0**	360	**2.5**
065			507.93	-1.7	208.89	-1.8	1053.50	**6.2**	198.01	-0.9
066			645	-0.5			413	-1.0	275	0.7
067	15.21	**2.1**	0.790	**-5.9**	0.349	**-5.3**	0.510	**-5.6**	0.273	**-5.0**
068			84	**-5.2**	33	**-4.7**	48	**-5.1**	23	**-4.6**
069	13.6	0.9	638	-0.6	283	-0.6	552	0.5	217	-0.5
070	10.5	-1.4	1500	**6.6**	300	-0.3	500	0.0		
071	10.6	-1.3	838	1.1	300	-0.3	593	1.0	231	-0.2
072	9.80	-1.9			318	0.0	449	-0.6	239	0.0
073	9.7	-2.0	342	**-3.1**	369	0.8	450	-0.6	228	-0.2
074	14.00	1.2			290	-0.5	423	-0.9		
075			670	-0.3	314	-0.1	506	0.0	178	-1.3

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

Table 1 (continued): Results and z-Scores

laboratory number	analyte									
	aluminium assigned value 12.3 mg/kg		total arsenic assigned value 711 µg/kg		cadmium assigned value 319 µg/kg		lead assigned value 503 µg/kg		total mercury assigned value 240 µg/kg	
	result mg/kg	z-score	result µg/kg	z-score	result µg/kg	z-score	result µg/kg	z-score	result µg/kg	z-score
076	13.81	1.1	648.87	-0.5	253.13	-1.1	592.21	1.0	246.17	0.1
077	14.65	1.7	624.7	-0.7	264.39	-0.9	606.35	1.2	254.33	0.3
078	19.4	**5.2**	1204	**4.1**	565	**4.1**	1142	**7.2**	288	1.0
079			940	1.9	390	1.2	600	1.1		
080	9.382	**-2.2**	1.183	**-5.9**	0.234	**-5.3**	0.388	**-5.6**	0.390	**-5.0**
081			1161.67	**3.8**	640.43	**5.3**	4599.33	**45.9**		
082	10.3	-1.5	420.8	**-2.4**	241.9	-1.3	462.7	-0.5		
083	8.49	**-2.8**	548	-1.4	306	-0.2	406	-1.1	167	-1.5
084	0.90	**-8.5**	791	0.7	261	-1.0	366	-1.5	70.4	**-3.6**
085			632	-0.7	315	-0.1	517	0.2		
086	16.50	**3.1**	961.00	**2.1**	297.00	-0.4	482.00	-0.2	181.00	-1.2

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

Table 2: Participants' Comments

participant number	comments
008	Results are not corrected for recovery.
064	MDL: Al: 0.5 mg/kg, As: 500 ?g/kg, Cd:500 ?g/kg, Pb:500 ?g/kg, , Hg:500 ?g/kg (The results of As, Cd, Pb, Hg are less than MDL)

comments are as submitted by participants

Table 3: Assigned Values and Standard Deviations for Proficiency

analyte	data points, n	assigned value, x_a	uncertainty, u	standard deviation for proficiency, σ_p	
aluminium	52	12.3 mg/kg	0.459	Horwitz [5]	1.35
total arsenic	73	711 µg/kg	24.5	Horwitz [5]	120
cadmium	79	319 µg/kg	6.32	Horwitz [5]	60.6
lead	80	503 µg/kg	10.2	Horwitz [5]	89.2
total mercury	62	240 µg/kg	5.03	Horwitz [5]	47.6

Table 4: Number and Percentage of z-Scores where |z| ≤2

analyte	number of scores where |z| ≤2	total number of scores	% |z| ≤2
aluminium	34	53	64
total arsenic	55	78	71
cadmium	74	84	88
lead	65	85	76
total mercury	56	68	82

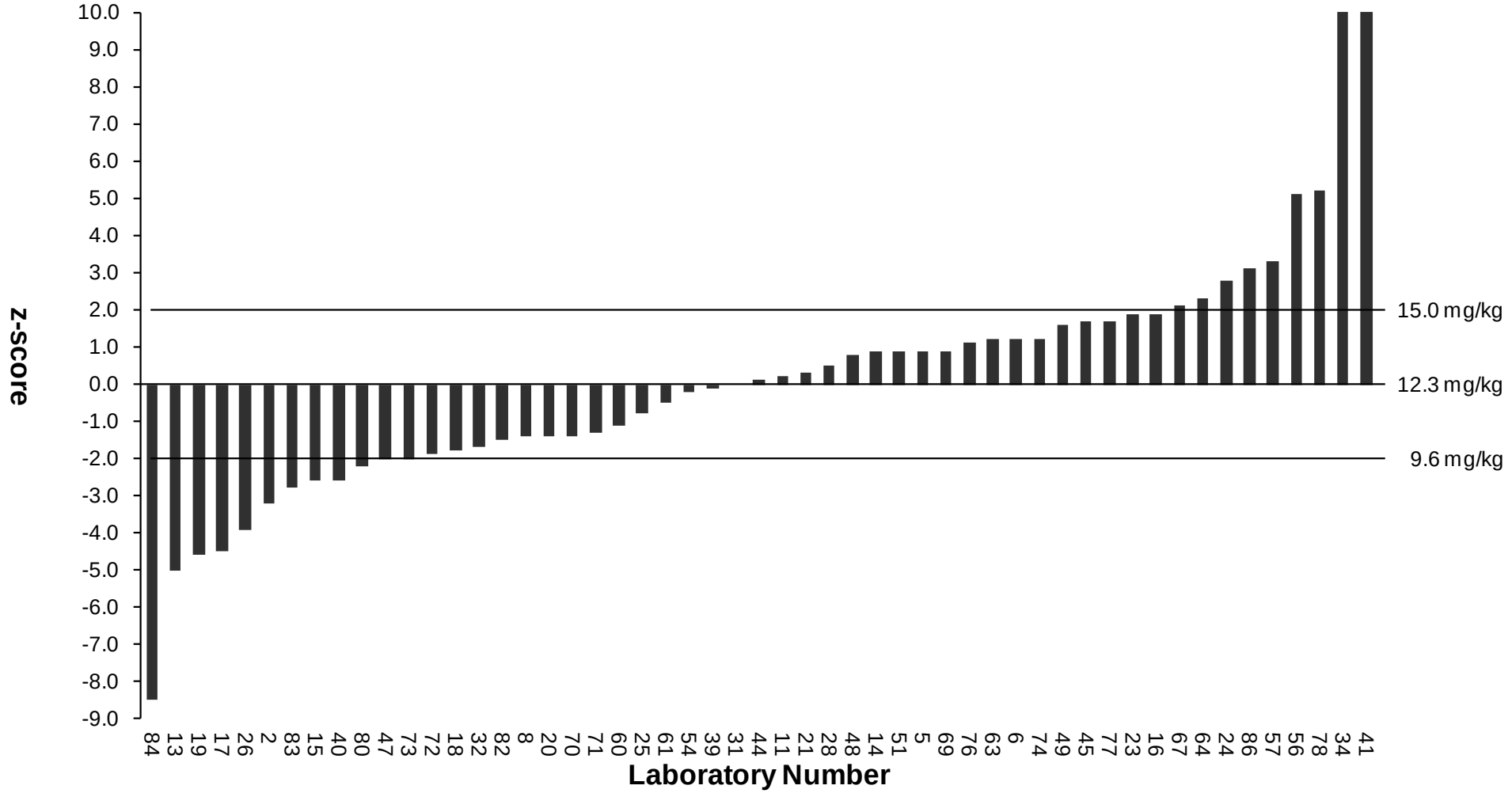

Figure 1: z-Scores for Aluminium

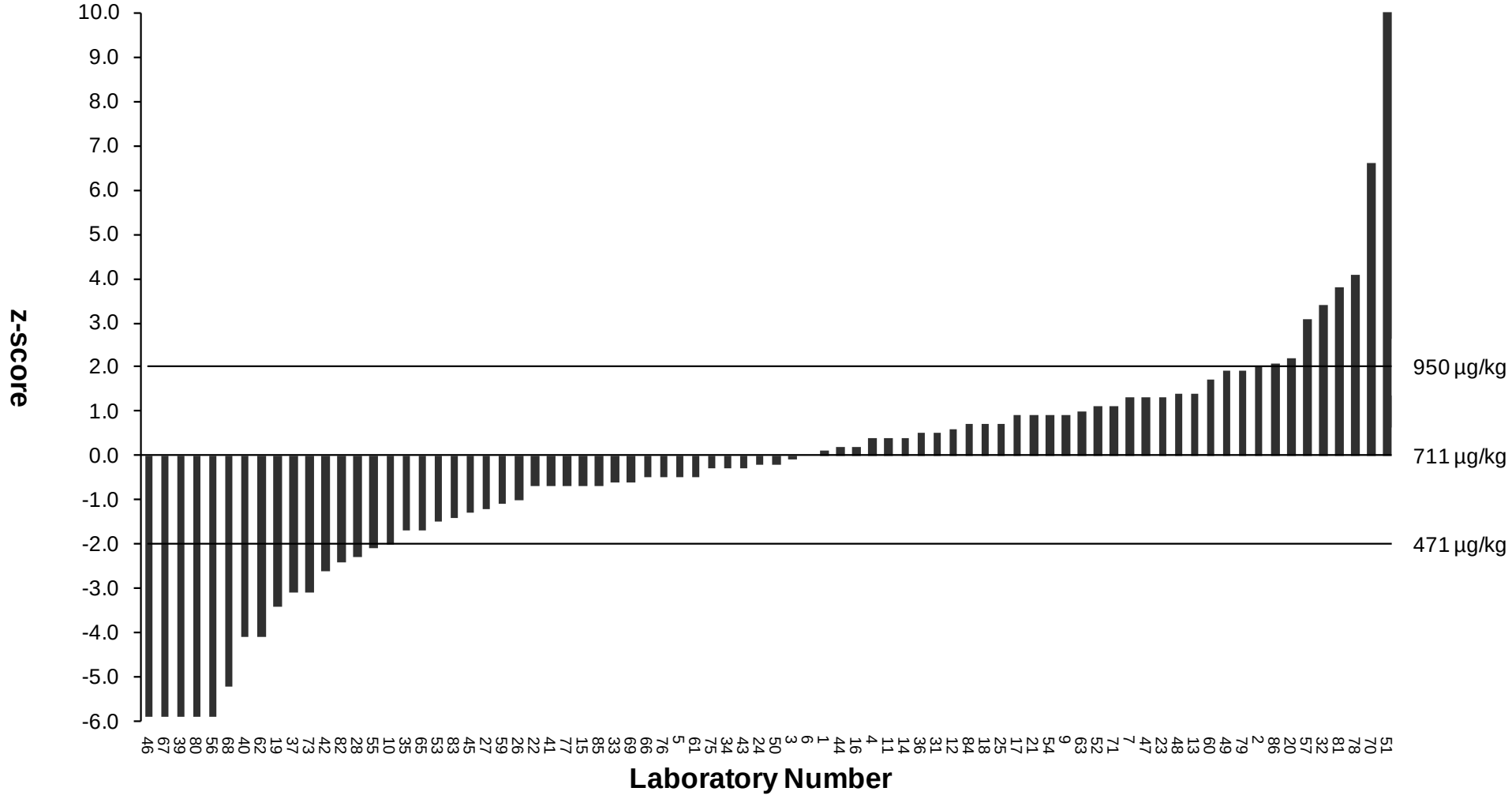

Figure 2: z-Scores for Total Arsenic

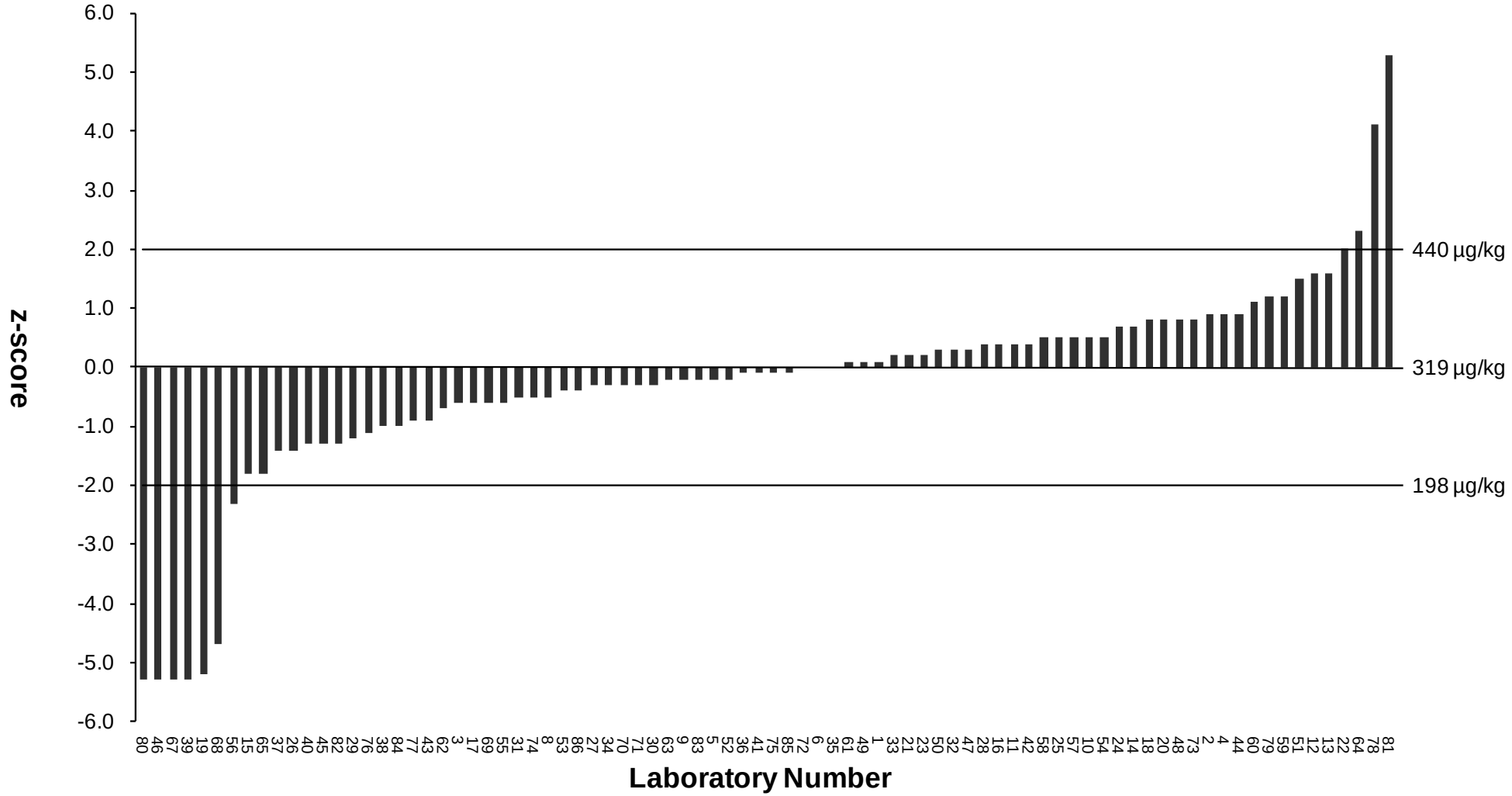

Figure 3: z-Scores for Cadmium

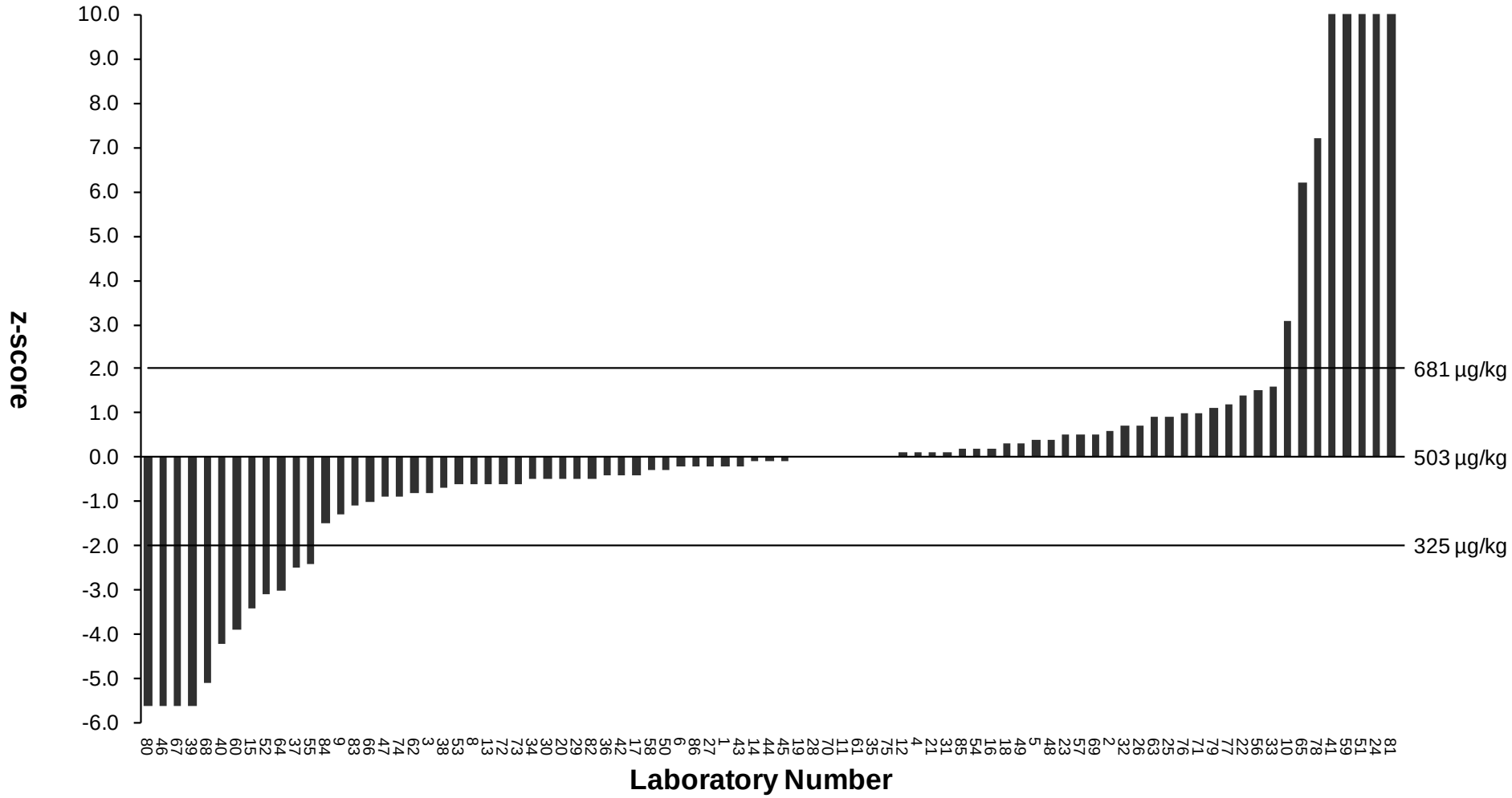

Figure 4: z-Scores for Lead

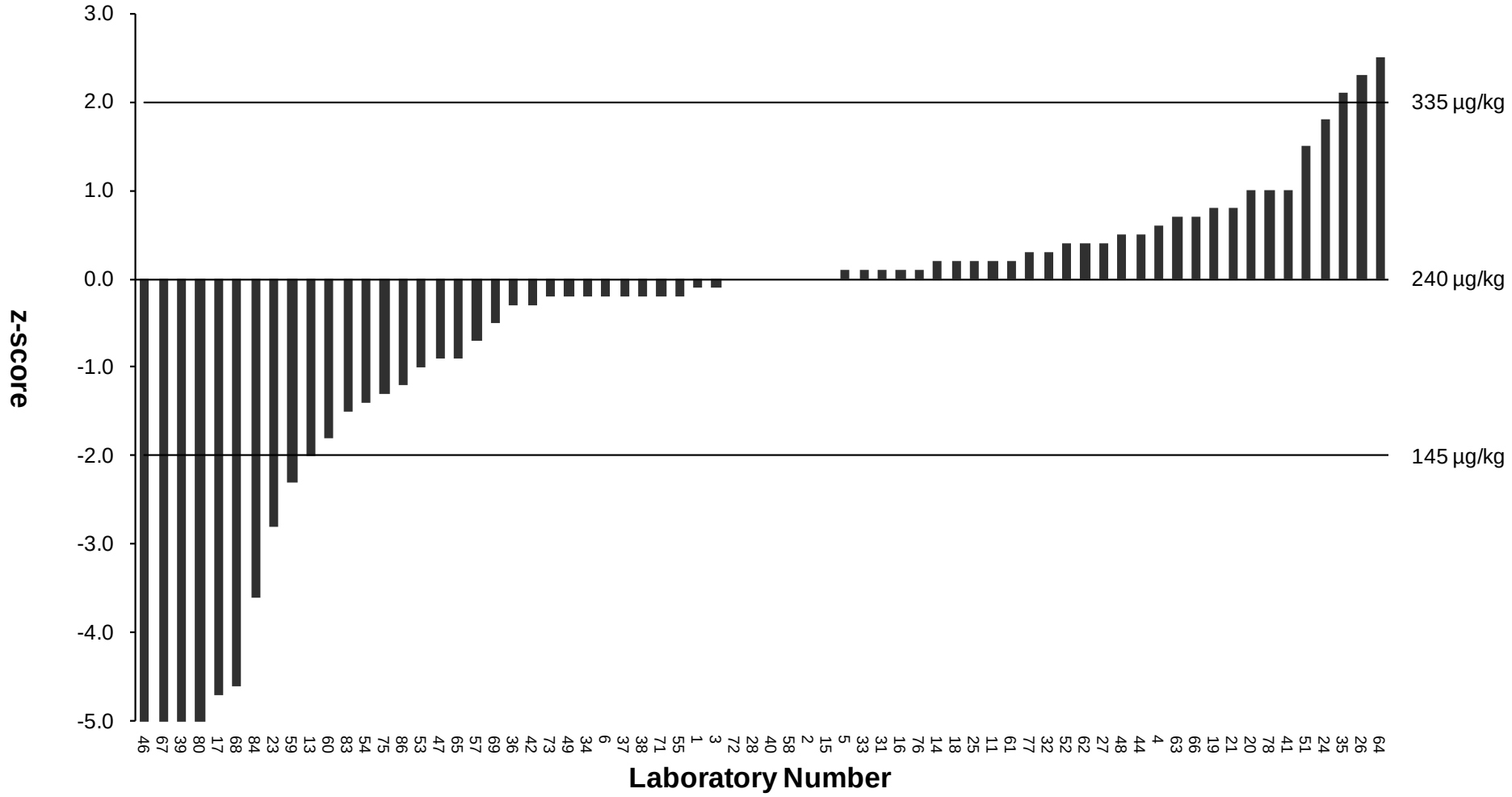

Figure 5: z-Scores for Total Mercury

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.

Aluminium

Accredited Method Used	laboratory number
yes	002 011 013 017 021 028 034 039 044 048 049 051 054 063 072
no	005 008 015 016 019 020 023 024 025 026 031 040 041 045 047 057 061 067 069 080 082 083

Reference	laboratory number
AOAC Official Methods	045
AOAC Official Methods 1999	067
AOAC Official Methods 2005	083
AOAC Official Methods 2005 18th	051
AOAC Official Methods 2005 Edn18 16 -19	020
AOAC Official Methods 999.10	026
AOAC Official Methods 999.11	015
APHA 2006 3111D	031
Atomic Spectroscopy	041
EN 2002 EN 13805, AAS-GF 10	057
EN 2003 UNI EN 14084:2003	023
EN 2010	072
EPA Method	049
GB/T 23374-2009 1 1	054
ICP-MS	047
ISO	039
ISO 2000 AKS-PL-21123	063
ISO 2005	021
ISO 2011	040
Microwave Digestion and AAS 2006 (Instruction Manual)	024

Sample Weight (g)	laboratory number
<1	005 008 011 016 017 019 020 021 023 026 034 039 044 048 049 054 057 061 067 069 072 080
≥1 - <2	002 015 024 025 028 040 041 045 047 051 063
≥2 - <5	013 031
≥5 - <10	083

Sample Preparation	laboratory number
dry ashing	015 045 083
extract into MIBK with APDC/DDDC	031
microwave digestion	002 005 008 011 016 017 020 021 023 024 025 026 028 034 039 041 044 045 047 048 049 051 054 057 061 063 067 069 072 080
pressure bomb digestion	040 072
wet digestion	013 019

Sample Preparation Reagents Used	laboratory number
hydrochloric acid	002 031 041 067 072 083
hydrogen peroxide	005 008 020 023 025 041 045 047 048 051 054 057 069
nitric acid	002 005 008 011 013 015 016 017 019 020 021 023 024 025 026 034 039 040 041 044 045 047 048 049 051 054 057 061 063 067 069 072 080

Modifier	laboratory number
lanthanum (La)	031
magnesium nitrate (Mg(NO3)2)	057
none	025 054 069

Determination	laboratory number
flame AAS	024 041 083
graphite furnace AAS	015 031 057
ICP-MS	005 008 011 016 017 020 021 034 044 047 048 049 054 061 067 069 072
ICP-OES	002 013 019 023 025 026 028 039 040 045 051 063 080

Wavelength (nm)	laboratory number
114	017
167.0	051
308.2	026
308.215	013
308.215 - 396.152 - 237.312	025
309.3	041 057 083
396	063
396.1	045
396.152	019 080
396.2	015 024
Not applicable	069

Mass (amu)	laboratory number
26.982	026
27	005 008 011 016 020 034 044 047 048 054 061 069

Units of Limit of Detection	laboratory number
micrograms per kilogram (µg/kg)	011 039 044 067 069 072
micrograms per litre (µg/L)	080
milligrams per kilogram (mg/kg)	002 005 008 013 015 016 017 019 021 023 024 025 026 028 031 034 045 047 048 049 051 054 057 061 063 083
milligrams per litre (mg/L)	041

Limit of Detection	laboratory number
≥0.001 - <0.01	047 072
≥0.01 - <0.1	005 015 016 023 031 049 054 080
≥0.1 - <1	008 013 019 021 045 048 051 057 061 063 083
≥1 - <10	017 024 025 026 028 034 041 067
≥10 - <100	002 011 069
≥100	044

Total Arsenic

Accredited Method Used	laboratory number
yes	002 003 004 007 009 010 011 012 013 017 020 021 025 028 031 034 036 037 039 043 044 046 048 049 050 051 053 054 057 060 063 069 079
no	005 014 015 016 019 023 024 026 027 033 035 040 041 045 047 052 055 059 061 066 067 073 075 080 081 082 083

Reference	laboratory number
AFNOR	027
AOAC Official Methods	010 045
AOAC Official Methods 1999	067
AOAC Official Methods 2003 17th Edn Rev. 2	009
AOAC Official Methods 2005	083
AOAC Official Methods 2005 Edn18	020 051
AOAC Official Methods 2006	055
AOAC Official Methods 2007 Vol 90 No 3 844	069
AOAC Official Methods 986.15	015(based on) 031 057
AOAC Official Methods 997.15D (9.2.20A)	081
AOAC Official Methods 999.10	026
Atomic Spectroscopy	041
BOE	035
BS EN 2004 11 pages	037
EN 2003 UNI 14084	023
EPA Method	049
GB/T 5009.11 2003	053
GB/T 5009.11-2003 3 73	054
GB/T 5009.11-2003 vol 1 73-74	060
Hygienic Standard for Cosmetics 2007 - 166-168	007
ICP-MS	047
ICP-OES 1998 11969	014
ISO	039 040
ISO 2000 AKS-PL-21123	063
ISO 2002 EN14546	075
ISO 2005	021

Reference (continued)	laboratory number
LMBG Method	036
Manufacturer's Instructions	012 013
Microwave Digestion and AAS 2006 Instruction Manual	024
NEN_EN 15763 2010	002
NMKL	059

Sample Weight (g)	laboratory number
<1	002 004 005 009 010 011 012 013 015 016 017 020 021 023 024 025 026 027 034 035 036 039 040 043 045 048 049 050 052 053 057 059 061 066 067 069 071 079 080 081
≥1 - <2	003 014 019 028 031 033 041 044 047 051 054 060 063 075 083
≥5 - <10	037 055 073
≥10 - <25	007 046

Sample Preparation	laboratory number
dry ashing	003 033 037 043 046 055 075 083
high pressure asher	063
microwave digestion	002 004 005 007 009 010 011 012 013 014 015 016 017 020 021 023 024 025 026 028 034 035 036 039 041 044 045 047 048 049 050 051 057 059 066 067 069 071 079 080
pressure bomb digestion	027 040 046 071
reduction As(V) - As(III)	035 046 052
wet digestion	019 031 052 053 054 060 061 073 081

Sample Preparation Reagents Used	laboratory number
hydrochloric acid	002 003 009 010 014 033 041 045 046 050 055 067 075 083
hydrogen peroxide	004 005 009 015 020 023 025 035 041 045 046 047 048 051 052 057 066 069 071 079 081
magnesium nitrate, magnesium oxide	033
nitric acid	002 003 004 005 007 009 011 012 013 014 015 016 017 019 020 021 023 024 025 026 027 031 033 034 035 036 037 039 040 041 043 044 045 046 047 048 049 050 051 053 054 057 059 060 061 063 066 067 069 071 073 075 079 080 081
sulphuric acid	052 053 054 060

Modifier	laboratory number
ascorbics acid /potassium iodure	083
Ge	007
HCl	046
magnesium nitrate (Mg(NO3)2)	003 012 015 026 031 036 037 041 043 075
NABH4	002 046
NH4H2PO4	019
Ni(NO3)2	066
Nickel nitrate	024
niquel hexahidrate nitrate	033
palladium (Pd)	012 027 035 036 041 059 071
potassium chloride	080
SnCl2	046
none	025 054 060 069

Determination	laboratory number
AFS	060
Atomic Fluorescence Spectrophotometer	054
cold vapour / hydride generation AAS	003 052 055
cold vapour / hydride generation ICP-OES	040 080
cold vapour AFS	045
FIAS	031
flame AAS	046
graphite furnace AAS	012 019 024 027 028 033 035 036 037 041 059 066 071
hydride generation AAS	015 043 046 057 061 063 075 083
hydride generation ICP-MS	007
ICP	073
ICP-MS	002 004 005 011 016 017 020 021 023 025 034 047 048 049 050 067 069 079
ICP-MS with collision cell	044
ICP-OES	002 009 010 013 014 026 039 051 081
spectrophotometric	053

Wavelength (nm)	laboratory number
188.979	014 081
188.980	009 050
189.0	051

Wavelength (nm) (continued)	laboratory number
189.042	002
193.759	080
193.696	013
193.7	012 015 019 024 026 027 031 033 036 037 041 043 045 052 054 055 057 059 060 061 063 066 071 075 083
214.4	010
283.3	053
not applicable	069

Mass (amu)	laboratory number
2	031
74.9	079
74.922	026
75	004 005 007 011 016 020 023 025 034 044 047 048 050 069
91	002

Units of Limit of Detection	laboratory number
micrograms per kilogram (µg/kg)	002 005 010 011 014 015 020 023 024 025 026 031 033 034 035 036 039 043 044 045 047 048 050 051 055 059 060 061 066 067 069 071 081 083
micrograms per litre (µg/L)	007 019 027 037 041 080
milligrams per kilogram (mg/kg)	003 009 012 013 016 017 021 028 046 049 052 053 054 057 063 075 079

Limit of Detection	laboratory number
≥0.01 - <0.1	003 012 016 017 023 028 031 034 046 049 052 054 057 063 075 080 081
≥0.1 - <1	007 009 013 021 045 067 079 083
≥1 - <10	005 011 015 019 024 027 036 041 043 044 047 059
≥10 - <100	002 010 014 025 026 033 037 048 050 053 060 061 066 069 071
≥100	020 035 051

Cadmium

Accredited Method Used	laboratory number
yes	002 003 004 008 009 010 011 012 013 014 017 020 021 023 025 027 028 029 031 033 034 036 037 038 039 040 041 043 044 046 047 048 049 050 051 053 054 055 057 059 060 063 069 072 074 075 079 080
no	005 015 016 019 024 026 030 035 045 052 058 061 067 073 081 082 083

Reference	laboratory number
AFNOR	027
AFNOR 2002 NF EN 13804	080
AOAC Official Methods	010 045
AOAC Official Methods 1999	037 067
AOAC Official Methods 1999 18th Edn 2005 Ch 9 p17	029
AOAC Official Methods 2003 17th Edn Rev. 2	009
AOAC Official Methods 2005	055 083
AOAC Official Methods 2005 18th 16-19	047
AOAC Official Methods 2005 Edn18th	020 030 051
AOAC Official Methods 2007 Vol 90, No 3 844	069
AOAC Official Methods 985.01	031
AOAC Official Methods 997.15D (9.2.20A)	081
AOAC Official Methods 999.10	026 057
AOAC Official Methods 999.11	015
Atomic Spectroscopy 2007 NF EN 14084	019
BOE	035
EN 2003	038 041
EN 2003 14083	075
EN 2003 UNI 14084	023
EN 2004	025
EN 2009 CEN 15763	008
EN 2010	072
EPA Method 2007	049
Food Additives and Contaminant 2000 20 543	074
GB/T 5009.15 2003	053
GB/T 5009.15-2003 3 113	054

Reference (continued)	laboratory number
GB/T 5009.15-2003 vol 1 113-114	060
ISO	039
ISO 2000 AKS-PL-21123	063
ISO 2005	021
ISO 2005 14084	014
ISO 2011	040
LMBG Method	036
Manufacturer's Instructions	012 013
Microwave Digestion and AAS 2006 (Instruction Manual)	024
NEN_EN 15763 2010	002
NMKL	059

Sample Weight (g)	laboratory number
<1	002 004 005 008 009 010 011 012 013 016 017 020 021 023 024 025 026 027 029 033 034 035 036 039 044 048 049 050 053 057 058 059 060 067 069 071 072 079 081
≥1 - <2	014 019 028 030 037 040 041 045 047 051 052 054 063 074 080
≥2 - <5	015 031 043 075
≥5 - <10	046 055 061 073 083
≥10 - <25	003 038

Sample Preparation	laboratory number
dry ashing	003 015 031 038 043 045 046 055 073 083
extract into MIBK with APDC/DDDC	003
microwave digestion	002 004 005 008 009 010 011 012 013 014 016 017 020 021 023 024 025 026 028 033 034 035 036 037 039 041 044 045 047 048 049 050 051 057 058 059 060 063 067 069 071 072 074 075 079
pressure bomb digestion	027 029 040 046 071 072
wet digestion	019 030 052 053 054 061 080 081

Sample Preparation Reagents Used	laboratory number
HClO4	030 053 054
hydrochloric acid	002 003 009 010 014 031 055 067 072 073 080 083

Sample Preparation Reagents Used (continued)	laboratory number
hydrogen peroxide	004 005 008 009 020 023 025 029 033 035 037 045 046 047 048 051 057 058 069 071 074 075 079 081
nitric acid	002 004 005 008 009 010 011 012 013 014 015 016 017 019 020 021 023 024 025 026 027 029 030 033 034 035 036 037 038 039 040 041 043 044 045 046 047 048 049 050 051 052 053 054 057 058 059 060 061 063 067 069 071 072 073 074 075 079 080 081

Modifier	laboratory number
caesium (Cs)	079
magnesium nitrate (Mg(NO3)2)	012 023 024 029 031 036 037 041 047 074 075
NH4H2PO4	012 014 015 019 023 024 027 029 031 033 036 043 047 052 053 054 071 074 075
palladium (Pd)	041 058 063
none	025 060

Determination	laboratory number
cold vapour / hydride generation ICP-OES	031
flame AAS	003 038 046 055 061 083
graphite furnace AAS	012 014 015 023 024 027 028 029 033 035 036 037 041 043 047 052 054 057 058 059 060 063 071 073 074 075
ICP-MS	002 004 005 008 011 016 017 020 021 025 034 044 048 049 050 067 069 072 079
ICP-OES	009 010 013 019 026 030 039 040 045 051 080 081
spectrophotometric	053

Wavelength (nm)	laboratory number
214.4	010
214.439	013 019 050
226.502	009 081
228.8	003 012 014 015 023 024 026 029 033 036 037 038 041 043 047 051 052 053 054 055 057 058 059 060 061 063 071 074 075 079 083
228.802	002 030 031 080

Wavelength (nm) (continued)	laboratory number
283.3	027
317.9	045
not applicable	069

Mass (amu)	laboratory number
0.8g	080
111	004 005 008 011 016 020 025 034 044 048 049 050 069 079
112.41	026
114	002 017

Units of Limit of Detection	laboratory number
micrograms per kilogram (µg/kg)	002 005 010 011 014 015 020 023 024 025 026 031 033 034 035 036 039 043 044 047 048 051 053 054 055 058 059 060 061 067 069 071 072 074 075 081 083
micrograms per litre (µg/L)	019 027 029 037 041 049 080
milligrams per kilogram (mg/kg)	003 008 009 012 013 016 017 021 028 030 038 045 046 050 052 057 063 079

Limit of Detection	laboratory number
≥0.001 - <0.01	003 008 016 049 063 072
≥0.01 - <0.1	009 012 013 021 023 028 029 030 034 038 046 050 052 057 079 081
≥0.1 - <1	005 015 019 024 027 037 041 045 054 058 060 067 080 083
≥1 - <10	011 014 017 025 033 035 036 043 044 047 048 053 059 061 069 071 074
≥10 - <100	002 010 020 026 031 055 075
≥100	051

Lead

Accredited Method Used	laboratory number
yes	002 003 004 008 009 010 011 013 014 017 020 021 023 025 027 028 029 031 033 034 036 037 038 039 040 041 043 044 046 047 048 049 050 051 053 054 057 059 060 063 066 069 072 074 075 079 080
no	005 012 015 016 019 024 026 030 035 045 052 055 058 061 067 073 081 082 083

Reference	laboratory number
AFNOR	027
AFNOR 2002 NF EN 13804	080
AOAC Official Methods	010
AOAC Official Methods 1982	045
AOAC Official Methods 1999	037 067
AOAC Official Methods 1999 18th Edn 2005 Ch 9, p17	029
AOAC Official Methods 2003 17th Edn Rev. 2	009
AOAC Official Methods 2005	055 083
AOAC Official Methods 2005 18th 16-19	047
AOAC Official Methods 2005 18th Edn	020 030 051
AOAC Official Methods 2007 Vol 90, No 3 844	069
AOAC Official Methods 985.01	031
AOAC Official Methods 997.15D (9.2.20A)	081
AOAC Official Methods 999.10	026 057
AOAC Official Methods 999.11	015
Atomic Spectroscopy 2007 NF EN 14084	019
BOE	035
EN 2003	041 038
EN 2003 14083	075
EN 2003 UNI 14084	023
EN 2004	025
EN 2009 CEN 15763	008
EN 2010	072
EPA Method	049
Food Additives and Contaminant 2000 20 543	074
GB 5009.12-2010 4 1	054
GB/T 5009.12 2003	053

Reference (continued)	laboratory number
GB/T 5009.12-2003 vol 1 89-90	060
ISO	039
ISO 2000 AKS-PL-21123	063
ISO 2005	021
ISO 2005 14084	014
ISO 2011	040
LMBG Method	036
Manufacturer's Instructions	012 013
Microwave Digestion and AAS 2006 (Instruction Manual)	024
NEN_EN 15763 2010	002
NMKL	059
Pearson's Composition and Anal 2004 Method 4/7 1 to 12	079

Sample Weight (g)	laboratory number
<1	002 004 005 008 009 010 011 012 013 016 017 020 021 023 024 025 026 027 029 033 034 035 036 039 044 048 049 050 057 059 060 066 067 069 071 072 079 081
≥1 - <2	014 019 028 030 037 040 041 045 047 051 052 053 054 063 080
≥2 - <5	015 031 043 074 075 083
≥5 - <10	046 055 058 061 073
≥10 - <25	003 038

Sample Preparation	laboratory number
dry ashing	003 015 031 038 043 045 046 055 073 083
extract into MIBK with APDC/DDDC	003
microwave digestion	002 004 005 008 009 010 011 012 013 014 016 017 020 021 023 024 025 026 028 033 034 035 036 037 039 041 044 045 047 048 049 050 051 057 058 059 060 063 066 067 069 071 072 074 075 079
pressure bomb digestion	027 029 040 046 071 072
wet digestion	019 030 052 053 054 061 080 081

Sample Preparation Reagents Used	laboratory number
HClO4	030 053 054
hydrochloric acid	002 003 009 010 014 031 055 067 072 073 080 083
hydrogen peroxide	004 005 008 009 020 023 025 029 033 035 037 045 046 047 048 051 057 058 066 069 071 074 075 079 081
nitric acid	004 005 008 009 010 011 012 013 014 015 016 017 019 020 021 023 024 025 026 027 029 030 033 034 035 036 037 038 039 040 041 043 044 045 046 047 048 049 050 051 052 053 054 057 058 059 060 061 063 066 067 069 071 072 073 074 075 079 080 081

Modifier	laboratory number
magnesium nitrate (Mg(NO3)2)	012 023 024 029 031 036 037 041 047 071 074 075
NH4H2PO4	014 019 023 024 027 029 031 033 036 043 052 053 054 066 074 075
palladium (Pd)	012 041 047 058 063 071
none	025 060 069

Determination	laboratory number
cold vapour / hydride generation ICP-OES	031
flame AAS	003 038 046 061 083
graphite furnace AAS	012 014 015 019 023 024 027 028 029 033 035 036 037 041 043 047 052 053 054 055 057 058 059 060 063 066 071 073 074 075
ICP-MS	002 004 005 008 011 016 017 020 021 025 034 044 048 049 050 067 069 072 079
ICP-OES	009 010 013 026 030 039 040 045 051 080 081

Wavelength (nm)	laboratory number
193.7	012
217	003 024 037 074 075
220.3	026 045 051
220.353	002 009 013 030 031 050 080 081
228.8	027 043 057 058
238.2	010

Wavelength (nm) (continued)	laboratory number
283.3	014 015 019 023 029 033 036 041 047 052 053 054 055 059 060 061 063 066 071
not applicable	069

Mass (amu)	laboratory number
0.8g	080
206+207+208	004
207.2	026
208	002 005 008 011 016 017 020 025 034 044 048 050 069 079

Units of Limit of Detection	laboratory number
micrograms per kilogram (µg/kg)	002 005 011 014 015 020 023 024 025 026 031 033 034 035 036 039 043 044 047 048 051 053 055 058 059 060 061 067 069 071 072 074 075 081 083
micrograms per litre (µg/L)	019 027 029 037 041 080
milligrams per kilogram (mg/kg)	003 008 009 010 012 013 016 017 021 028 030 038 045 046 049 050 052 054 057 063 066 079

Limit of Detection	laboratory number
≥0.001 - <0.01	008 010 054 072
≥0.01 - <0.1	009 012 013 016 017 021 028 029 030 034 046 049 057 063 066 081
≥0.1 - <1	005 015 023 037 038 041 045 050 052 067 079 080 083
≥1 - <10	003 011 014 019 024 027 043 044 047 053 055 058 059 060 061 069 071
≥10 - <100	002 020 025 026 031 033 035 036 048 074 075
≥100	051

Total Mercury

Accredited Method Used	laboratory number
yes	002 003 004 011 013 014 015 017 019 020 021 025 028 031 034 036 037 038 039 041 044 046 048 049 051 053 054 057 058 059 063 066 069 072 073
no	005 016 023 024 026 027 033 035 040 047 052 055 061 067 075 080 083

Reference	laboratory number
afgeleid van afvalstoffencompendium OVAM – CMA 2/I/A.6.1 – CMA 2/I/B.3	002
AFNOR	027
AFNOR 2002 NF EN 13804	080
AMA 254 Advanced Mercury Analyser Operating Manual 2002 Altec Ltd.,Prague, Czech Republic	041
AOAC Official Methods 1999	067
AOAC Official Methods 2005	055 083
AOAC Official Methods 2005 Edn 18th	020 051
AOAC Official Methods 2007 Vol 90, No 3 844	069
AOAC Official Methods 971.21	031 057
AOAC Official Methods 974.14 (based on)	015
AOAC Official Methods 999.10	026
Atomic Spectroscopy	019
BOE	035
BS EN 2002	021
EH 13806 2002	037
EN 2003 13806	075
EN 2010	072
EPA Method	049
GB/T 5009.17 2003	053
GB/T 5009.17-2003 3 131	054
ISO	039 040
ISO 1997 1483	014
ISO 2000 AKS-PL-21123	063
LMBG Method	036
Manufacturer's Instructions	013 047

Reference (continued)	laboratory number
Microwave Digestion and AAS 2006 (Instruction Manual)	024
NMKL	059
Pyrolysis method 2007	038

Sample Weight (g)	laboratory number
<1	003 004 005 011 013 016 017 019 020 021 023 024 025 026 027 033 034 035 036 038 039 041 044 047 048 049 053 054 057 058 059 061 066 067 069 071 072
≥1 - <2	002 014 015 028 031 037 051 052 063 080 083
≥5 - <10	055 073
≥10 - <25	046 075

Sample Preparation	laboratory number
direct determination	041
dry ashing	027 046
microwave digestion	002 005 011 013 014 016 017 020 021 023 024 026 028 033 034 035 036 037 039 044 048 049 051 054 057 059 067 069 071 072
pressure bomb digestion	040 046 071 072
wet digestion	003 015 031 052 053 055 061 073 075 080 083
none	004 019 025 047 066

Sample Preparation Reagents Used	laboratory number
hydrochloric acid	002 014 031 067 072 080 083
hydrogen peroxide	005 013 020 023 033 035 046 048 051 054 057 069 071
nitric acid	002 005 011 013 014 015 016 017 020 021 023 024 026 027 031 033 034 035 036 037 039 040 044 046 048 049 051 052 053 054 055 057 059 061 067 069 071 072 073 075 080
sulphuric acid	003 031 055 075
none	004 025 041 047 066

Modifier	laboratory number
lanthanum (La)	031
magnesium nitrate (Mg(NO3)2)	036
palladium (Pd)	027
SnCl2	002 003 037 046 052 061
vanadium pentoxide	035
none	004 025 041 047 054 066 069

Determination	laboratory number
automated mercury analyser	002 004 019 025 036 038 041 047 058 063 066
cold vapour / hydride generation AAS	003 015 021 023 024 028 033 035 036 037 039 040 046 052 059 061 073 075 083
cold vapour / hydride generation ICP-OES	026
cold vapour AFS	027 054
FIAS	031 071
hydride generation AAS	014 055 057
ICP-MS	005 011 016 017 020 034 044 048 049 067 069 072
ICP-OES	013 051 080
spectrophotometric	053

Wavelength (nm)	laboratory number
184.9	051
193.7	055
194.16	080
194.164	013
194.2	026
253.7	002 059 063
253.65	041
253.7	003 014 015 019 023 024 027 031 033 036 037 052 054 057 058 061 071 075 083
254	004 025
not applicable	069

Mass (amu)	laboratory number
1	031
200.59	026
201	011
202	005 016 020 034 044 048 069

Units of Limit of Detection	laboratory number
micrograms per kilogram (µg/kg)	005 011 014 015 019 020 023 024 025 026 031 033 034 035 036 038 039 041 044 047 048 051 053 054 055 058 059 061 067 069 071 072 075 083
micrograms per litre (µg/L)	027 037 049 080
milligrams per kilogram (mg/kg)	002 003 013 016 017 021 028 046 052 057 063 066

Limit of Detection	laboratory number
≥0.001 - <0.01	016 021 023 037 039 046 049 053 063 066 072
≥0.01 - <0.1	002 003 013 017 028 031 034 052 057 067
≥0.1 - <1	041 054 059 080 083
≥1 - <10	005 011 014 015 019 024 027 035 036 038 044 047 048 055 058 061 069 071 075
≥10 - <100	020 025 026 033 051

APPENDIX II: FAPAS SecureWeb, Reports and Protocol

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 and quality control materials.
- Freely download copies of reports, in Acrobat PDF format, of proficiency tests in which they have participated.

2. REPORTS

The Acrobat PDF version of this report is available to all participants as a free download from FAPAS SecureWeb.

3. PROTOCOL

The Protocols [2, 3] set out how FAPAS® is organised. Copies can be downloaded from our website.

4. QUALITY SYSTEMS

FAPAS® is accredited by UKAS as complying with the requirements of ISO/IEC 17043:2010

The Food and Environment Research Agency is an ISO 9001 certified organisation.

5. CONTACT DETAILS

Participants with any comments or concerns about this proficiency test should contact:

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