

Fapas<sup>®</sup> – Bespoke Food Chemistry Proficiency Test Report BFC0701 – Data Redacted Version

Metallic Contaminants in Baby Food

March - April 2020

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### SUMMARY

- This version of the report for proficiency test BFC0701 was created at the request of the initiating organisation, the Baby Food Council. This version has redacted the data corresponding to the assigned values for all analytes and samples, so as to be blind to laboratories that did not take part in the original proficiency test but would otherwise need to see the outcome of the test. The original non-redacted report remains the definitive accredited record of this proficiency test.
- The test materials for Fapas<sup>®</sup> Food Chemistry proficiency test BFC0701 were dispatched in March 2020. Each participant received four vegetable purée-based baby food test materials (A, B, C and D) to be analysed for arsenic (total), cadmium and lead.
- 3. An assigned value ( $x_a$ ) was determined for each analyte and in conjunction with the standard deviation for proficiency ( $\sigma_p$ ) was used to calculate a z-score for each result.

analyte	assigned value, x <sub>a</sub> µg/kg	number of scores,  z  ≤2	total number of scores	%  z ≤2
Arsenic (total)_A	Х	23	24	96
Cadmium_A	Х	20	22	91
Lead_A	Х	22	25	88
Arsenic (total)_B	Х	26	27	96
Cadmium_B	Х	26	27	96
Lead_B	Х	25	27	93
Arsenic (total)_C	Х	10	12	83
Cadmium_C	Х	9	13	69
Lead_C	Х	10	16	63
Arsenic (total)_D	Х	19	21	90
Cadmium_D	Х	20	21	95
Lead_D	Х	19	25	76

4. Results for this proficiency test are summarised as follows:

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### 1. INTRODUCTION

### **1.1. Proficiency Testing**

Proficiency testing aims to provide an independent assessment of the competence of participating laboratories. Together with the use of validated methods, proficiency testing is an essential element of laboratory quality assurance.

Further details of the Fapas<sup>®</sup> – Food Chemistry proficiency testing scheme are available in our protocols [4, 5].

This proficiency test was initiated by Baby Food Council, supported by the Environmental Defense Fund (US).

### 2. TEST MATERIAL

#### 2.1. Preparation

Preparation of the samples for this proficiency test was sub-contracted to a laboratory meeting the quality requirements of the scheme's accreditation [3].

The test materials were prepared from banana, butternut squash, cucumber and shelled peas; all were purchased from a retail source. The choice of ingredients reflected the expected low incurred levels of each ingredient, according to a survey of different foods undertaken previously by the sub-contracted laboratory. The butternut squash was cooked briefly to soften it to a consistency comparable to the other ingredients for ease of blending.

Each individual ingredient was screened for the presence of incurred analytes. The low level incurred analytes measured then determined the proportion of each ingredient in the final puréed mixture in order to keep the total incurred levels as low as practically possible. Although desirable, it was not possible to produce a completely blank test material (defined as non-measurable incurred levels).

For one of the four samples, arsenic (total), cadmium and lead were present at the natural (non-spiked) levels.

For the remaining three samples, arsenic (total), cadmium and lead were spiked into the test materials to give the following theoretical total levels (spike plus incurred):

	Arsenic (total), µg/kg	Cadmium, µg/kg	Lead, µg/kg
Sample A	Х	Х	Х
Sample B	Х	Х	Х
Sample D	Х	Х	Х

The levels of one sample (mid-point spiked level) were chosen to be in keeping with the LOQs of the FDA laboratory's experience in using the EAM 4.7 method. The remainder of this text is redacted from the original report.

Samples were stored at -20 °C until dispatch.

### 2.2. Homogeneity

To test for homogeneity, randomly selected test materials were analysed in duplicate. Testing was sub-contracted to a laboratory meeting the quality requirements of the scheme's accreditation [3].

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

### 2.3. Dispatch

The start date was 09 March 2020. Test materials were sent to 28 participants.

### 3. RESULTS

The instructions for reporting results were as follows:

1) Determine the level of analyte(s) present in the test materials as follows for each test materials:

analyte	units	comment	
Arsenic (total)	µg/kg	as received	
Cadmium	µg/kg	as received	
Lead	µg/kg	as received	

Please Note: It is important that you report the results in this way so that we can include as many results as possible in the statistical analysis.

- 2) The four sample types comprise the following: blank, low level, medium level, high level. These levels do not correspond to A, B, C, D, do not make any assumptions about the order of increasing or decreasing concentration.
- 3) The spiking levels are approximately in keeping with the US FDA's method EAM 4.7. This means that the method of analysis employed must be capable of quantification at approximately unit values of µg/kg.

You are encouraged to also include details of your analytical method; this will help to interpret the proficiency test data at these challenging levels.

Results were submitted by 27 participants (96%) before the closing date for this test, 20 April 2020.

Each participant was given a laboratory number, assigned in order of receipt of results. The reported analyte concentrations are given in Table 1 to Table 4.

Participants' comments are given in Table 5.

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,  $\sigma_{p_1}$  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*<sub>a</sub>

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  $1000 \times \text{greater}$  or smaller than the majority of submitted results (as these were considered to be reporting errors).

#### Sample A (BFC0701\_A)

For arsenic (total) and cadmium, this procedure was straightforward and the robust mean was chosen as the assigned value.

For Lead, the major mode was chosen as the assigned value because the distribution of results was slightly skewed. A plot of the distribution of results is shown in Figure 3 [redacted].

#### Sample B (BFC0701\_B)

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

#### Sample C (BFC0701\_C)

For arsenic (total), cadmium and lead, the median was chosen as the assigned value because of the low number of participants' valid results.

#### Sample D (BFC0701\_D)

For arsenic (total) and cadmium, this procedure was straightforward and the robust mean was chosen as the assigned value.

For Lead, the major mode was chosen as the assigned value because the distribution of results was slightly skewed. A plot of the distribution of results is shown in Figure 12 [redacted].

The assigned values for all analytes are shown in Table 6.

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

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

For all analytes,  $\sigma_p$  was derived from the appropriate form of the Horwitz equation [8].

The values for  $\sigma_p$  used to calculate z-scores from the reported results of this test are given in Table 6.

#### 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, see Table 6, and  $\sigma_p =$  the standard deviation for proficiency, see Table 6.

Participants' z-scores for all analytes are given in Table 1 to Table 4 and shown as histograms in Figures 1–12. 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 6.

The number and percentage of z-scores in the range  $-2 \le z \le 2$  for all analytes are given in Table 7.

### 4.4. Comments on Statistical Evaluation

The assigned values for the spiked samples are the consensus of participants' results. These values are remarkably close to the target spiked levels (which also match very closely the mean values determined during homogeneity testing, data not included). This is a strong indication of accuracy of the assigned values.

Sample [redacted] was the highest spiked level and was included so that there would be at least one sample within normal measurement range of the laboratories taking part. This was intended to provide some assessment of performance, regardless of difficulty of the other samples at challenging low levels. The tight distributions of results and the low uncertainty of the assigned values demonstrate a high degree of reproducibility precision among the participants.

Results for arsenic (total) in the spiked samples were always in good agreement, with low uncertainty of the assigned values and symmetrical distributions. Laboratory 025 consistently has over-estimated arsenic in three of the samples (but not cadmium or lead).

Results for cadmium in the spiked samples were always in good agreement, with low uncertainty of the assigned values and symmetrical distributions.

Results for lead in the lower spiked samples show asymmetry in the distributions with a curve towards the higher concentration in one sample and apparent bimodality in another sample (a small population of results that are over-estimates). The highest spiked level sample is not affected in the same way. Lead is a ubiquitous contaminant, even in laboratory environments. It would appear that some of the participants' laboratories have a low level background presence of lead which becomes apparent in the results for the lowest concentration samples. The same absolute level of background lead would not be so obvious in the highest concentration sample and the same asymmetry is not observed.

Sample [redacted] was the unspiked sample, with incurred levels of all three analytes. From the screening and preparation data, the anticipated incurred levels were arsenic X  $\mu$ g/kg, cadmium X  $\mu$ g/kg and lead X  $\mu$ g/kg. These agree remarkably well with the assigned values of arsenic X  $\mu$ g/kg, cadmium X  $\mu$ g/kg and lead X  $\mu$ g/kg. It is also worth noting that about half the laboratories taking part were sufficiently confident in their analyses to report results for this sample.

Sample (unspiked) arsenic and lead assigned values had low uncertainties. The uncertainty for cadmium is somewhat higher, in fact it technically fails the acceptance criterion for issuing z-scores in a proficiency test. (The uncertainty of the assigned value must be sufficiently low compared to the value of the standard deviation for proficiency,  $\sigma_{p_i}$  that z-scores would not be affected.) However, given the closeness of agreement between the median of participants' results and the screened level, and the importance of this proficiency test in its objectives, the decision has been made to issue z-scores. Readers of this report are asked to bear in mind the above commentary in the proper interpretation of this outcome.

Sample (unspiked) lead assigned value was determined as the median of results that were in good agreement. Normally, outliers would be flagged as more than about 10x the median of results. In this case, however, there is a distinct population of results (laboratories 013, 015, 018, 019, 027) that do not fit with the consensus, even though their results are generally within 10x the median. There are too few results to operate a mode calculation so the same effect has been created by excluding these results and taking the median of the remainder.

This report and its commentary has been discussed by the Baby Food Council members and approved for publication to all the participants.

### 5. INTERPRETATION OF SCORES

In normal circumstances, over time, about 95% of z-scores will lie in the range  $-2 \le z \le 2$ . 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 06/04/2020.
- 2 GlobalSign PDF Signing Tool, https://www.globalsign.com/en/digital-signatures/ accessed 06/04/2020.
- 3 ISO/IEC 17043:2010, Conformity assessment General requirements for proficiency testing.
- 4 Fapas<sup>®</sup>, 2017, Protocol for Proficiency Testing Schemes, Version 6, April 2017, Part 1 Common Principles.
- 5 Fapas<sup>®</sup>, 2017, Protocol for Proficiency Testing Schemes, Version 5, April 2017, Part 2 Fapas<sup>®</sup> Food Chemistry scheme (FAPAS).
- 6 AMC Tech Brief No. 74, z-Scores and other scores in chemical proficiency testing their meanings, and some common misconceptions, *Anal. Methods*, 2016, **8**, 5553.
- 7 Thompson, M., Ellison, S.L.R. and Wood, R., 2006, The International Harmonised Protocol for the Proficiency Testing of Analytical Chemistry Laboratories, *Pure Appl. Chem.*, **78**, No. 1, 145–196.
- 8 Thompson, M., 2000, Recent trends in inter-laboratory precision at ppb and sub-ppb concentrations in relation to fitness for purpose criteria in proficiency testing, *Analyst*, **125**, 385-386.

laboratory number –	analyte					
	Arsenic assigned va	Arsenic (total)_A igned value: X μg/kg assiç		nium_A alue: X μg/kg	Lead_A assigned value: X µg/kg	
-	result	z-score	result	z-score	result	z-score
001	Х	-0.4	Х	-1.0	Х	-0.9
002	Х	0.3	Х	0.8	Х	0.6
003	Х	1.0	Х		Х	-0.2
004	Х	0.7	Х	0.1	Х	-0.2
005	Х	-0.2	Х	-4.5	Х	-0.2
006	Х	-0.1	Х	-0.1	Х	-0.2
007	Х	-0.1	Х	-0.2	Х	-0.2
008	Х	-0.1	Х		Х	-0.5
009	Х	-0.4	Х	-0.1	Х	0.6
010	Х	0.8	Х	-0.1	Х	-0.1
011	Х	-0.4	Х	-0.1	Х	-0.2
012	Х	-0.3	Х	-0.3	Х	0.0
013	Х		Х		Х	
014	Х	0.2	Х	0.1	Х	0.1
015	Х	-0.2	Х	-0.1	Х	1.5

# Table 1: Results and z-Scores for Sample A (BFC0701\_A)

laboratory number —	analyte					
	Arsenic (total)_A assigned value: X μg/kg		Cadmium_A assigned value: X μg/kg		Lead_A assigned value: X µg/kg	
-	result	z-score	result	z-score	result	z-score
016	Х	-0.5	х	-0.1	X	0.0
017	Х	0.8	Х	0.2	Х	0.0
018	Х	-0.5	Х	3.8	Х	17.9
019	Х	-0.8	Х	0.1	Х	5.3
020	Х	-0.5	Х		Х	-0.1
021	Х	0.1	Х	-0.1	Х	-0.4
022	Х		Х		Х	
023	Х	0.1	Х	0.2	Х	0.0
024	Х	0.4	х	-0.1	Х	0.1
025	Х	2.7	х	0.2	Х	0.1
026	Х		Х	0.6	Х	0.4
027	Х	-0.5	Х	-0.3	Х	2.5

# Table 1 (continued): Results and z-Scores for Sample A (BFC0701\_A)

laboratory number			ana	alyte		
-	Arsenic assigned va	Arsenic (total)_B assigned value: X μg/kg		Cadmium_B assigned value: X µg/kg		ad_B alue: X µg/kg
-	result	z-score	result	z-score	result	z-score
001	Х	-0.1	Х	-0.5	Х	-0.3
002	Х	0.9	Х	0.8	Х	0.0
003	Х	-0.1	Х	0.0	Х	0.2
004	Х	0.2	Х	0.1	Х	0.1
005	Х	0.0	Х	0.1	Х	0.2
006	Х	0.1	Х	0.0	Х	0.1
007	Х	0.0	Х	0.0	Х	-0.3
008	Х	0.3	Х	-0.1	Х	-0.1
009	Х	-0.3	Х	0.3	Х	-4.1
010	Х	0.3	х	0.3	Х	-0.1
011	Х	0.2	Х	0.2	Х	-0.2
012	Х	-0.1	х	-0.1	Х	0.1
013	Х	-0.6	Х	-0.9	Х	-0.1
014	Х	0.3	Х	0.1	Х	0.1
015	Х	-0.5	Х	-0.4	Х	0.1

# Table 2: Results and z-Scores for Sample B (BFC0701\_B)

laboratory number	analyte					
	Arsenic (total)_B assigned value: X μg/kg		Cadmium_B assigned value: X μg/kg		Lead_B assigned value: X µg/kg	
-	result	z-score	result	z-score	result	z-score
016	Х	-0.4	Х	0.0	X	-0.4
017	Х	0.2	Х	0.0	Х	-0.2
018	Х	0.1	Х	-0.2	Х	0.4
019	Х	-0.3	Х	0.1	Х	0.3
020	Х	-0.1	Х	0.0	Х	-0.1
021	Х	0.1	х	0.1	Х	0.1
022	Х	-0.3	х	-0.9	Х	-0.6
023	Х	0.4	х	0.2	Х	0.2
024	Х	0.2	х	0.2	Х	0.2
025	Х	-4.5	Х	-4.5	Х	-4.6
026	Х	0.1	х	0.1	Х	0.5
027	Х	-0.5	Х	-0.3	Х	0.0

# Table 2 (continued): Results and z-Scores for Sample B (BFC0701\_B)

laboratory number -			ana	alyte		
	Arsenic assigned va	Arsenic (total)_C signed value: X μg/kg a		Cadmium_C assigned value: X µg/kg		ad_C alue: X µg/kg
-	result	z-score	result	z-score	result	z-score
001	Х		X		X	
002	Х		Х		Х	
003	Х		Х		Х	
004	Х		Х		Х	
005	Х	-4.5	Х	-4.5	Х	-4.5
006	Х		Х		Х	
007	Х	-0.9	Х	-2.3	Х	-0.4
008	Х		Х		Х	
009	Х		Х		Х	
010	Х	-0.3	Х	0.6	Х	0.5
011	Х	0.3	Х	-1.1	Х	1.0
012	Х	0.0	Х	-0.4	Х	-0.2
013	Х		Х		Х	27.8
014	Х	0.5	Х	-1.1	Х	0.1
015	Х	-0.5	Х	1.1	Х	7.9

# Table 3: Results and z-Scores for Sample C (BFC0701\_C)

laboratory number	analyte					
-	Arsenic (total)_C assigned value: X μg/kg		Cadmium_C assigned value: X μg/kg		Lead_C assigned value: X µg/kg	
-	result	z-score	result	z-score	result	z-score
016	Х		Х		Х	
017	Х	1.8	Х	1.5	Х	1.0
018	Х		Х	6.9	Х	66.8
019	Х		Х		Х	24.9
020	Х		х		Х	
021	Х		х		Х	-0.7
022	Х		х		Х	
023	Х	-0.7	х	0.0	Х	-0.1
024	Х	-0.5	Х	-0.2	Х	-0.2
025	Х	8.6	Х	0.0	Х	0.1
026	Х		Х		Х	
027	Х	0.5	Х	7.7	Х	28.8

# Table 3 (continued): Results and z-Scores for Sample C (BFC0701\_C)

laboratory number –			ana	alyte		
	Arsenic assigned va	Arsenic (total)_D signed value: X μg/kg		Cadmium_D assigned value: X μg/kg		ad_D alue: X µg/kg
-	result	z-score	result	z-score	result	z-score
001	Х	-0.1	Х	-1.2	Х	-0.7
002	Х	0.5	Х		Х	-0.1
003	Х		Х		Х	0.4
004	Х	0.3	Х	0.5	Х	0.6
005	Х	-4.5	Х	-4.5	Х	-4.5
006	Х	-0.1	Х	0.0	Х	-0.1
007	Х	0.0	Х	-0.6	Х	-0.4
008	Х		Х		Х	
009	Х	-0.1	х	0.0	Х	0.6
010	Х	-0.3	х	-0.1	х	-0.3
011	Х	-1.4	х	0.0	Х	-0.7
012	Х	-0.2	х	-0.2	х	0.3
013	Х		Х		х	3.3
014	Х	0.0	х	0.0	х	-0.1
015	Х	0.2	Х	0.1	Х	2.9

# Table 4:Results and z-Scores for Sample D (BFC0701\_D)

laboratory number			ana	alyte		
	Arsenic assigned va	(total)_D alue: Χ μg/kg	tal)_D Cadmium_D e: X μg/kg assigned value: X μg/kg		Lead_D assigned value: X µg/kg	
-	result	z-score	result	z-score	result	z-score
016	Х	-1.0	X	-0.1	X	0.3
017	Х	0.8	Х	-0.1	Х	-0.1
018	Х	0.8	Х	1.2	Х	12.9
019	Х	-0.5	Х	1.5	Х	3.9
020	Х		Х		Х	-0.4
021	Х	0.0	Х	-0.2	Х	-0.1
022	Х		Х		Х	
023	Х	0.1	Х	0.1	Х	0.1
024	Х	0.1	Х	0.0	Х	0.1
025	Х	2.9	Х	0.0	Х	0.0
026	Х		Х	0.7	Х	0.3
027	Х	-0.6	Х	-0.2	Х	4.4

# Table 4 (continued): Results and z-Scores for Sample D (BFC0701\_D)

laboratory number	comments
016	We consider that the material [redacted] is the blank material.
024	Corrected for avg. spike recovery of fortified analytical portion, per Fapas instructions. As: 101.5% Cd: 104% Pb: 105% Reported values for sample TBFC0701_C are trace level between LOD (X, X, X ug/kg for As, Cd, Pb, respectfully) and LOQ (X, X, X ug/kg for As, Cd, Pb, respectfully).
026	no comments

### Table 5: Participants' Comments

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

analyte	data points, <i>n</i>	assigned value, <sub>Xa</sub> µg/kg	uncertainty, <i>u</i>	standard de for proficien	viation icy, $\sigma_p$
Arsenic (total)_A	24	Х	Х	Horwitz [8]	Х
Cadmium_A	21	Х	Х	Horwitz [8]	Х
Lead_A	25	Х	Х	Horwitz [8]	Х
Arsenic (total)_B	26	Х	Х	Horwitz [8]	Х
Cadmium_B	26	Х	Х	Horwitz [8]	Х
Lead_B	26	Х	Х	Horwitz [8]	Х
Arsenic (total)_C	11	Х	Х	Horwitz [8]	Х
Cadmium_C	12	Х	Х	Horwitz [8]	Х
Lead_C	10	Х	Х	Horwitz [8]	Х
Arsenic (total)_D	20	Х	Х	Horwitz [8]	Х
Cadmium_D	20	Х	Х	Horwitz [8]	Х
Lead_D	24	Х	Х	Horwitz [8]	Х

### Table 6: Assigned Values and Standard Deviations for Proficiency

analyte	number of scores where  z  ≤2	total number of scores	%  z ≤2
Arsenic (total)_A	23	24	96
Cadmium_A	20	22	91
Lead_A	22	25	88
Arsenic (total)_B	26	27	96
Cadmium_B	26	27	96
Lead_B	25	27	93
Arsenic (total)_C	10	12	83
Cadmium_C	9	13	69
Lead_C	10	16	63
Arsenic (total)_D	19	21	90
Cadmium_D	20	21	95
Lead_D	19	25	76

# Table 7: Number and Percentage of z-Scores where $|z| \le 2$



Figure 1: z-Scores for Arsenic (total)\_A



Figure 2: z-Scores for Cadmium\_A



### Figure 3: z-Scores for Lead\_A

Insert shows a plot of the distribution of the results [redacted in this version of the report]



Figure 4: z-Scores for Arsenic (total)\_B



Figure 5: z-Scores for Cadmium\_B



Figure 6: z-Scores for Lead\_B



Figure 7: z-Scores for Arsenic (total)\_C



Figure 8: z-Scores for Cadmium\_C



Figure 9: z-Scores for Lead\_C



Figure 10: z-Scores for Arsenic (total)\_D



Figure 11: z-Scores for Cadmium\_D



### Figure 12: z-Scores for Lead\_D

Insert shows a plot of the distribution of the results [redacted in this version of the report]

### 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
no	015
yes	001 004 006 007 008 009 012 014 016 017 019 021 022 023 024 025 027
What is Your Method Based On?	laboratory number
International Standard	001 004 006 008 016 019
International Standard National Standard	001 004 006 008 016 019 007 012 024 027
International Standard National Standard Paper Published In An International Journal	001 004 006 008 016 019 007 012 024 027 015

# Baby Food (BFC0701\_A)

## Arsenic (total)

Sample Weight (g)	laboratory number
<1	001 002 004 008 009 014 016 017 019 027
≥1 - <2	006 007 012 023 024
≥2 - <5	015 021 022 025

Sample Preparation	laboratory number
microwave digestion	001 002 004 006 007 008 009 012 014 015 016 019 021 023 024 027
wet digestion	022 025

Sample Preparation Reagents Used	laboratory number
hydrochloric acid	006 012 015 016 023 025
hydrogen peroxide	001 004 007 009 014 019 024 025
nitric acid	001 002 004 006 007 008 009 014 015 016 017 019 021 022 023 024 025 027

Modifier	laboratory number
N/A	007
Determination	laboratory number
ICP-MS	001 002 004 006 008 009 012 014 015 016 017 019 021 022 023 024 025 027
ICP-QQQ-MS	007
Wavelength (nm)	laboratory number
N/A	007
Mass (amu)	laboratory number
75	001 002 004 009 012 014 015 016 017 019 021 022 023 025
91 (AsO reaction mode)	007
per EAM 4.7	024
Limit of Detection	laboratory number
≥0.001 - <0.01	015 017 021
≥0.01 - <0.1	014 016
≥0.1 - <1	007 023 024
≥1 - <10	001 002 004 006 009 012 019 025
≥100	022
Units of Limit of Detection	laboratory number
micrograms per kilogram (µg/kg)	001 002 004 006 007 009 012 019 022 023 024 025
milligrams per kilogram (mg/kg)	014 015 016 017 021

# Baby Food (BFC0701\_A)

# Cadmium

Sample Weight (g)	laboratory number
<1	001 002 004 008 009 014 016 017 019 027
≥1 - <2	007 012 023 024
≥2 - <5	015 021 022 025
Sample Preparation	laboratory number
microwave digestion	001 002 004 007 008 009 012 014 015 016 019 021 023 024 027
wet digestion	022 025
UtraClave	017
Sample Preparation Reagents Used	laboratory number
hydrochloric acid	012 015 016 023 025
hydrogen peroxide	001 004 007 009 014 019 024 025
nitric acid	001 002 004 007 008 009 014 015 017 019 021 022 023 024 025 027
Modifier	laboratory number
N/A	007
none	017
Determination	laboratory number
ICP-MS	001 002 004 008 009 012 014 015 016 017 019 021 022 023 024 025 027
ICP-QQQ-MS	007
Wavelength (nm)	laboratory number
N/A	007

Mass (amu)	laboratory number
111	012
111, 114	004
per EAM 4.7	024

Limit of Detection	laboratory number
<0.001	017
≥0.001 - <0.01	015 021
≥0.01 - <0.1	014 016 023 024
≥0.1 - <1	007
≥1 - <10	001 002 004 009 012 019 025
≥10 - <100	022

Units of Limit of Detection	laboratory number
micrograms per kilogram (µg/kg)	001 002 004 007 009 012 019 022 023 024 025
milligrams per kilogram (mg/kg)	014 015 016 017 021

# Baby Food (BFC0701\_A)

### Lead

Sample Weight (g)	laboratory number
<1	001 002 004 008 009 014 016 017 019 027
≥1 - <2	007 012 023 024
≥2 - <5	015 021 022 025

Sample Preparation	laboratory number
microwave digestion	001 002 004 007 008 009 012 014 015 016 019 021 023 024 027
wet digestion	022 025
UltraClave	017

Sample Preparation Reagents Used	laboratory number
hydrochloric acid	012 015 016 023 025
hydrogen peroxide	001 004 007 009 014 019 024 025
nitric acid	001 002 004 007 008 009 014 015 017 019 021 022 023 024 025 027
Modifier	laboratory number
N/A	007
none	017
Determination	laboratory number
ICP-MS	001 002 004 008 009 012 014 015 016 017 019 021 022 023 024 025 027
ICP-QQQ-MS	007
Wavelength (nm)	laboratory number
N/A	007
Mass (amu)	laboratory number
208	012
206, 207, 208	004
206+207+208	009
208	001 002 014 015 016 017 019 021 022 023 025
per EAM 4.7	024
Sum of 206, 207, 208	007
Limit of Detection	laboratory number
<0.001	017
≥0.001 - <0.01	015 021
≥0.01 - <0.1	014 016
≥0.1 - <1	007 023
≥1 - <10	001 002 004 009 012 019 024 025
≥10 - <100	022

Units of Limit of Detection	laboratory number
micrograms per kilogram (µg/kg)	001 002 004 007 009 012 019 022 023 024 025
milligrams per kilogram (mg/kg)	014 015 016 017 021

# Baby Food (BFC0701\_B)

# Arsenic (total)

Sample Weight (g)	laboratory number
<1	001 002 004 008 009 014 016 017 019
≥1 - <2	007 012 023
≥2 - <5	015 021 025
Sample Preparation	laboratory number
microwave digestion	001 002 004 007 008 009 012 014 015 016 019 021 023
wet digestion	025
UltraClave	017
Sample Preparation Reagents Used	laboratory number
hydrochloric acid	012 015 016 023 025
hydrogen peroxide	001 004 007 009 014 019 025
nitric acid	001 002 004 007 008 009 014 015 016 017 019 021 023 025
Modifier	laboratory number
N/A	007
none	017
Determination	laboratory number
ICP-MS	001 002 004 008 009 012 014 015 016 017 019 021 023 025
ICP-QQQ-MS	007

Wavelength (nm)	laboratory number
N/A	007
Mass (amu)	laboratory number
75	012
91 (AsO reaction mode)	007
Limit of Detection	laboratory number
≥0.001 - <0.01	015 021
≥0.01 - <0.1	014 016
≥0.1 - <1	007 023
≥1 - <10	001 002 004 009 012 019 025
Units of Limit of Detection	laboratory number
micrograms per kilogram (µg/kg)	001 002 004 007 009 012 019 023 025
milligrams per kilogram (mg/kg)	014 015 016 021

# Baby Food (BFC0701\_B)

## Cadmium

002 004 008 009 014 016 019
012 023
021 025

Sample Preparation	laboratory number
microwave digestion	001 002 004 007 008 009 012 014 015 016 019 021 023
wet digestion	025

Sample Preparation Reagents Used	laboratory number
hydrochloric acid	012 015 016 023 025
hydrogen peroxide	001 004 007 009 014 019 025
nitric acid	001 002 004 007 008 009 014 015 019 021 023 025
Modifier	laboratory number
N/A	007
Determination	laboratory number
ICP-MS	001 002 004 008 009 012 014 015 016 019 021 023 025
ICP-QQQ-MS	007
Wavelength (nm)	laboratory number
111	012
N/A	007
Mass (amu)	laboratory number
111	001 002 007 009 014 015 016 019 021 023 025
111, 114	004
Limit of Detection	laboratory number
≥0.001 - <0.01	015 021
≥0.01 - <0.1	014 016 023
≥0.1 - <1	007
≥1 - <10	001 002 004 009 012 019 025
Units of Limit of Detection	laboratory number
micrograms per kilogram (µg/kg)	001 002 004 007 009 012 019 023 025
milligrams per kilogram (mg/kg)	014 015 016 021

# Baby Food (BFC0701\_B)

# Lead

Sample Weight (g)	laboratory number
<1	001 002 004 008 009 014 016 019
≥1 - <2	007 012 023
≥2 - <5	015 021 025
Sample Preparation	laboratory number
microwave digestion	001 002 004 007 008 009 012 014 015 016 019 021 023
wet digestion	025
Sample Preparation Reagents Used	laboratory number
hydrochloric acid	012 015 016 023 025
hydrogen peroxide	001 004 007 009 014 019 025
nitric acid	001 002 004 007 008 009 014 015 019 021 023 025
Modifier	laboratory number
N/A	007
Determination	laboratory number
ICP-MS	001 002 004 008 009 012 014 015 016 019 021 023 025
ICP-QQQ-MS	007
Wavelength (nm)	laboratory number
N/A	007

Mass (amu)	laboratory number
208	012
206, 207, 208	004
206+207+208	009
208	001 002 014 015 016 019 021 023 025
Sum of 206, 207, 208	007

Limit of Detection	laboratory number
≥0.001 - <0.01	015 021
≥0.01 - <0.1	014 016
≥0.1 - <1	007 023
≥1 - <10	001 002 004 009 012 019 025

Units of Limit of Detection	laboratory number
micrograms per kilogram (μg/kg)	001 002 004 007 009 012 019 023 025
milligrams per kilogram (mg/kg)	014 015 016 021

# Baby Food (BFC0701\_C)

# Arsenic (total)

Sample Weight (g)	laboratory number
<1	001 002 004 008 009 014 019
≥1 - <2	007 012 023
≥2 - <5	015 021 025

Sample Preparation	laboratory number
microwave digestion	001 002 004 007 008 009 012 014 015 019 021 023
wet digestion	025

Sample Preparation Reagents Used	laboratory number
hydrochloric acid	012 015 023 025
hydrogen peroxide	001 004 007 009 014 019 025
nitric acid	001 002 004 007 008 009 014 015 019 021 023 025
Modifier	laboratory number
N/A	007
Determination	laboratory number
ICP-MS	001 002 004 008 009 012 014 015 019 021 023 025
ICP-QQQ-MS	007
Wavelength (nm)	laboratory number
N/A	007
Mass (amu)	laboratory number
75	012
91 (AsO reaction mode)	007
Limit of Detection	laboratory number
≥0.001 - <0.01	015 021
≥0.01 - <0.1	014
≥0.1 - <1	004 007 009 023
≥1 - <10	001 002 012 019 025
Units of Limit of Detection	laboratory number
micrograms per kilogram (μg/kg)	001 002 004 007 009 012 019 023 025
milligrams per kilogram (mg/kg)	014 015 021

# Baby Food (BFC0701\_C)

# Cadmium

Sample Weight (g)	laboratory number
<1	001 002 004 008 009 014 019
≥1 - <2	007 012 023
≥2 - <5	015 021 025
Sample Preparation	laboratory number
microwave digestion	001 002 004 007 008 009 012 014 015 019 021 023
wet digestion	025
Sample Preparation Reagents Used	laboratory number
hydrochloric acid	012 015 023 025
hydrogen peroxide	001 004 007 009 014 019 025
nitric acid	001 002 004 007 008 009 014 015 019 021 023 025
Modifier	laboratory number
N/A	007
Determination	laboratory number
ICP-MS	001 002 004 008 009 012 014 015 019 021 023 025
ICP-QQQ-MS	007
Wavelength (nm)	laboratory number
N/A	007
Mass (amu)	laboratory number
111	012
111, 114	004

Limit of Detection	laboratory number
≥0.001 - <0.01	015 021
≥0.01 - <0.1	014 023
≥0.1 - <1	007
≥1 - <10	001 002 004 009 012 019 025
Units of Limit of Detection	laboratory number
micrograms per kilogram (µg/kg)	001 002 004 007 009 012 019 023 025

014 015 021

# Baby Food (BFC0701\_C)

milligrams per kilogram (mg/kg)

### Lead

Sample Weight (g)	laboratory number
<1	001 002 004 008 009 014 019
≥1 - <2	007 012 023
≥2 - <5	015 021 025
Sample Preparation	laboratory number
microwave digestion	001 002 004 007 008 009 012 014 015 019 021 023
wet digestion	025
Sample Preparation Reagents Used	laboratory number
hydrochloric acid	012 015 023 025
hydrogen peroxide	001 004 007 009 014 019 025
nitric acid	001 002 004 007 008 009 014 015 019 021

Modifier	laboratory number
N/A	007

023 025

Determination	laboratory number
ICP-MS	001 002 004 008 009 012 014 015 019 021 023 025
ICP-QQQ-MS	007
Wavelength (nm)	laboratory number
N/A	007
Mass (amu)	laboratory number
208	012
206, 207, 208	004
206+207+208	009
208	001 002 014 015 019 021 023 025
Sum of 206, 207, 208	007
Limit of Detection	laboratory number
≥0.001 - <0.01	015 021
≥0.01 - <0.1	014
≥0.1 - <1	007 023
≥1 - <10	001 002 004 009 012 019 025
Units of Limit of Detection	laboratory number
micrograms per kilogram (µg/kg)	001 002 004 007 009 012 019 023 025
milligrams per kilogram (mg/kg)	014 015 021
Baby Food (BFC0701_D)	
Arsenic (total)	
Sample Weight (g)	laboratory number
<1	001 002 004 008 009 014 016 019
≥1 - <2	007 012 023
≥2 - <5	015 021 025

Sample Preparation	laboratory number
microwave digestion	001 002 004 007 008 009 012 014 015 016 019 021 023
wet digestion	025
Sample Preparation Reagents Used	laboratory number
hydrochloric acid	012 015 016 023 025
hydrogen peroxide	001 004 007 009 014 019 025
nitric acid	001 002 004 007 008 009 014 015 016 019 021 023 025
Modifier	laboratory number
N/A	007
Determination	laboratory number
ICP-MS	001 002 004 008 009 012 014 015 016 019 021 023 025
ICP-QQQ-MS	007
Wavelength (nm)	laboratory number
N/A	007
Mass (amu)	laboratory number
75	012
91 (AsO reaction mode)	007
Limit of Detection	laboratory number
≥0.001 - <0.01	015 021
≥0.01 - <0.1	014 016
≥0.1 - <1	007 023
≥1 - <10	001 002 004 009 012 019 025

Units of Limit of Detection	laboratory number
micrograms per kilogram (μg/kg)	001 002 004 007 009 012 019 023 025
milligrams per kilogram (mg/kg)	014 015 016 021

# Baby Food ( TBFC0701\_D )

### Cadmium

Sample Weight (g)	laboratory number
<1	001 002 004 008 009 014 016 019
≥1 - <2	007 012 023
≥2 - <5	015 021 025
Sample Preparation	laboratory number
microwave digestion	001 002 004 007 008 009 012 014 015 016 019 021 023
wet digestion	025
Sample Preparation Reagents Used	laboratory number
hydrochloric acid	012 015 016 023 025
hydrogen peroxide	001 004 007 009 014 019 025
nitric acid	001 002 004 007 008 009 014 015 019 021 023 025
Modifier	laboratory number
N/A	007
Determination	laboratory number
ICP-MS	001 002 004 008 009 012 014 015 016 019 021 023 025
ICP-QQQ-MS	007

Wavelength (nm)	laboratory number
N/A	007
Mass (amu)	laboratory number
111	012
111, 114	004
Limit of Detection	laboratory number
≥0.001 - <0.01	015 021
≥0.01 - <0.1	014 016 023
≥0.1 - <1	007
≥1 - <10	001 002 004 009 012 019 025
Units of Limit of Detection	laboratory number
micrograms per kilogram (µg/kg)	001 002 004 007 009 012 019 023 025
milligrams per kilogram (mg/kg)	014 015 016 021

# Baby Food (BFC0701\_D)

### Lead

Sample Weight (g)	laboratory number
<1	001 002 004 008 009 014 016 019
≥1 - <2	007 012 023
≥2 - <5	015 021 025

Sample Preparation	laboratory number
microwave digestion	001 002 004 007 008 009 012 014 015 016 019 021 023
wet digestion	025

Sample Preparation Reagents Used	laboratory number
hydrochloric acid	012 015 016 023 025
hydrogen peroxide	001 004 007 009 014 019 025
nitric acid	001 002 004 007 008 009 014 015 019 021 023 025
Modifier	laboratory number
N/A	007
Determination	laboratory number
ICP-MS	001 002 004 008 009 012 014 015 016 019 021 023 025
ICP-QQQ-MS	007
Wavelength (nm)	laboratory number
N/A	007
Mass (amu)	laboratory number
208	012
206, 207, 208	004
206+207+208	009
208	001 002 014 015 016 019 021 023 025
Sum of 206, 207, 208	007
Limit of Detection	laboratory number
≥0.001 - <0.01	015 021
≥0.01 - <0.1	014 016
≥0.1 - <1	007 023
≥1 - <10	001 002 004 009 012 019 025
Units of Limit of Detection	laboratory number
micrograms per kilogram (µg/kg)	001 002 004 007 009 012 019 023 025
milligrams per kilogram (mg/kg)	014 015 016 021

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

### 1. Fapas<sup>®</sup> SECUREWEB

Access to the secure area of our website is only available to participants in our proficiency tests and the owner of this proficiency test, which in this case is Baby Food Council. Please contact us if you require a UserID and Password. Fapas<sup>®</sup> 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<sup>®</sup> Food Chemistry proficiency tests.

### 2. PROTOCOL

The Protocols [4, 5] set out how Fapas  $^{\mbox{\tiny B}}$  – Food Chemistry is organised. Copies can be downloaded from our website.

### 3. CONTACT DETAILS

This report was prepared and authorised on behalf of Fapas<sup>®</sup> by Mark Sykes (Head of Science, Fapas<sup>®</sup>) and Sri Sumathi (Round Coordinator). Participants with any comments or concerns about this proficiency test should contact:

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