Fapas® – Food Chemistry Proficiency Test Report 19284

Pesticide Residues in Sweet Bell Pepper

January-March 2020

PARTICIPANT LABORATORY NUMBER

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SUMMARY

1. The test materials for Fapas® – Food Chemistry proficiency test 19284 were dispatched in January 2020. Each participant received a sweet bell pepper test material to be analysed for a selection of pesticide residues.

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 µg/kg	number of scores, |z| ≤2	total number of scores	% |z|≤2
Azinphos-methyl	66.9	32	37	86
Boscalid	39.2	39	41	95
Chlordane (cis)	48.3	23	31	74
Cypermethrin (sum of constituent isomers)	104	36	39	92
Endosulfan (alpha)	48.7	29	34	85
Fluopyram	64.9	33	33	100
Flutriafol	43.5	31	35	89
Kresoxim-methyl	23.0	34	37	92
Oxadiazon	68.6	28	30	93
Pirimiphos-ethyl	67.1	33	36	92
Triadimenol	40.4	33	37	89

CONTENTS

1. INTRODUCTION 6
1.1. Proficiency Testing 6
2. TEST MATERIAL 6
2.1. Preparation 6
2.2. Homogeneity 6
2.3. Dispatch 6
3. RESULTS 6
4. STATISTICAL EVALUATION OF RESULTS 7
4.1. Calculation of the Assigned Value, x_a 7
4.2. Standard Deviation for Proficiency, σ_p 8
4.3. Individual z-Scores 8
5. INTERPRETATION OF SCORES 8
6. REFERENCES 9

TABLES

Table 1: Results and z-Scores for Azinphos-methyl, Boscalid and Chlordane (cis) 10
Table 2: Results and z-Scores for Cypermethrin, Endosulfan I and Fluopyram 14
Table 3: Results and z-Scores for Flutriafol, Kresoxim-methyl and Oxadiazon 18
Table 4: Results and z-Scores for Pirimiphos-ethyl and Triadimenol 22
Table 5: Additional Pesticide Residues Reported 26
Table 6: Participants' Comments 26
Table 7: Assigned Values and Standard Deviations for Proficiency 27
Table 8: Number and Percentage of z-Scores where $|z| \leq 2$ 27

FIGURES

Figure 1: z-Scores for Azinphos-methyl 28
Figure 2: z-Scores for Boscalid 29
Figure 3: z-Scores for Chlordane (cis) 30
Figure 4: z-Scores for Cypermethrin 31
Figure 5: z-Scores for Endosulfan I 32
Figure 6: z-Scores for Fluopyram 33
Figure 7: z-Scores for Flutriafol 34
Figure 8: z-Scores for Kresoxim-methyl 35
Figure 9: z-Scores for Oxadiazon 36
Figure 10: z-Scores for Pirimiphos-ethyl 37
Figure 11: z-Scores for Triadimenol 38

APPENDICES

APPENDIX I: Analytical Methods Used by Participants 39

APPENDIX II: Fapas® SecureWeb, Protocol and Contact Details 51

1. INTRODUCTION

1.1. Proficiency Testing

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

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

2. TEST MATERIAL

2.1. Preparation

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

The test material was prepared from Sweet (Bell) Peppers. The bulk sample was split into two batches: one for spiking and one for the blank test material.

Sub-samples were taken to screen for the possible presence of incurred residues and the remainder was stored at -20°C. No residues were detected at, or above, 15 µg/kg.

Azinphos-methyl, Boscalid, Chlordane (cis), Cypermethrin, Endosulfan, Fluopyram, Flutriafol, Kresoxim-methyl, Oxadiazon, Pirimiphos-ethyl and Triadimenol were 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 [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 13 January 2020. Test materials were sent to 50 participants.

3. RESULTS

The instructions for reporting results were as follows:

1) Determine the level of residues present in the test material, **in µg/kg,** in the material in the form it is received (no further correction for wet weight or dry weight or reconstitution), together with **percentage (%) recovery** and **limit of quantification (LoQ)**.
    - All residues are to be reported **as specified**. If this is not possible, use the comments box to note any residues that are *not* reported in the form specified.

- For each residue, select either “Not Detected”, “Not Tested” or “Provide Result”.
- Enter a default value for “% recovery” and “limit of quantification (LOQ) µg/kg”.
- AFTER you have entered your results for each residue you MUST review and if necessary, edit the values for “% recovery” and “limit of quantification (LOQ) µg/kg” that differ from the default value you gave.

Results were submitted by 46 participants (92%) before the closing date for this test, 6 March 2020.

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

If a participant analysed for a pesticide residue that was in the test material, but did not identify it, and their limit of quantification was below the level needed for a z-score of -3.0, they were assessed as if their result was zero.

If a participant analysed for a pesticide residue that was in the test material, but did not identify it, and their limit of quantification was above the level needed for a z-score of -3.0, the result was recorded as <LoQ.

Any participant identifying pesticide residues other than Azinphos-methyl, Boscalid, Chlordane (cis), Cypermethrin, Endosulfan, Fluopyram, Flutriafol, Kresoxim-methyl, Oxadiazon, Pirimiphos-ethyl and Triadimenol at a level ≥15 µg/kg are listed in Table 5 together with the percentage recovery and LoQ.

Participants’ comments are given in Table 6.

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

4. STATISTICAL EVALUATION OF RESULTS

The results submitted by participants were statistically analysed in order to provide an assigned value for each analyte. The assigned values were then used in combination with the standard deviation for proficiency, σ_p, to calculate a z-score [6] for each result. The procedure is detailed in the relevant protocols [4, 5].

Further background on the procedure followed can be found in the IUPAC International Harmonised Protocol for the Proficiency Testing of Analytical Chemistry Laboratories [7].

4.1. Calculation of the Assigned Value, x_a

The assigned value, x_a, for each analyte was derived from the consensus of the results submitted by participants.

The following results were excluded from the calculation of the assigned value:

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

ii) results reported as approximately 10, 100 or 1000 × greater or smaller than the majority of submitted results (as these were considered to be reporting errors),

iii) results whose recovery was outside the range 60-140% [8],

iv) results where no recovery was reported,

v) results where no limit of quantification was reported.

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 7.

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 [9].

The values for σ_p used to calculate z-scores from the reported results of this test are given in Table 7.

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 7,
and σ_p = the standard deviation for proficiency, see Table 7.

Participants' z-scores for all analytes are given in Table 1-Table 4 and shown as histograms in Figures 1–11. 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 7.

The number and percentage of z-scores in the range $-2 \leq z \leq 2$ for all analytes are given in Table 8.

5. INTERPRETATION OF SCORES

In normal circumstances, over time, about 95% of z-scores will lie in the range $-2 \leq z \leq 2$. Occasional scores in the range $2 < |z| < 3$ are to be expected, at a rate of 1 in 20. Whether or not such scores are of importance can only be decided by considering them in the context of the other scores obtained by that laboratory.

Scores where $|z| > 3$ are to be expected at a rate of about 1 in 300. Given this rarity, such z-scores very strongly indicate that the result is not fit-for-purpose and almost certainly requires investigation.

The consideration of a set or sequence of z-scores over time provides more useful information than a single z-score. Examples of suitable methods of comparison are provided in the IUPAC International Harmonised Protocol for the Proficiency Testing of Analytical Chemistry Laboratories [7].

6. REFERENCES

1 Adobe Approved Trust List, https://helpx.adobe.com/acrobat/kb/approved-trust-list2.html#Whatisit accessed 13/11/2018.

2 GlobalSign PDF Signing Tool, https://www.globalsign.com/en/digital-signatures/ accessed 13/11/2018.

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

4 Fapas®, 2017, Protocol for Proficiency Testing Schemes, Version 6, April 2017, Part 1 – Common Principles.

5 Fapas®, 2017, Protocol for Proficiency Testing Schemes, Version 5, April 2017, Part 2 – Fapas® Food Chemistry scheme (FAPAS).

6 AMC Tech Brief No. 74, z-Scores and other scores in chemical proficiency testing – their meanings, and some common misconceptions, *Anal. Methods*, 2016, **8**, 5553.

7 Thompson, M., Ellison, S.L.R. and Wood, R., 2006, The International Harmonised Protocol for the Proficiency Testing of Analytical Chemistry Laboratories, *Pure Appl. Chem.*, **78**, No. 1, 145–196.

8 Guidance document on analytical quality control and method validation procedures for pesticide residues and analysis in food and feed, Document No. SANTE/12682/2019.

9 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 for Azinphos-methyl, Boscalid and Chlordane (cis)

laboratory number	analyte											
	Azinphos-methyl assigned value: 66.9 µg/kg				Boscalid assigned value: 39.2 µg/kg				Chlordane (cis) assigned value: 48.3 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
001	58.53			-0.6	42.00			0.3	#			
002	78	80-120	10	0.8	47	80-120	10	0.9	47	80-120	10	-0.1
003	72.2	111	5	0.4	41.5	104	5	0.3	115.9	81	10	**6.4**
004	#				#				#			
005	77.8	100	10	0.7	44.0	100	10	0.6	46.5	100	10	-0.2
006	45	100	10	-1.5	26	116	10	-1.5	0			**-4.5**
007	60	108	10	-0.5	40	112	10	0.1	51	110	10	0.3
008	85	95	10ppb	1.2	#				#			
009	#				#				#			
010	68.37	95	10	0.1	41.64	90.3	10	0.3	51.02	90.9	10	0.3
011	46.3	86.4	5	-1.4	28.0	84.4	5	-1.3	43.3	88.1	5	-0.5
012	#				51.14	70-120	10	1.4	#			

z-scores outside |z| >2 are shown in **bold,** see Section 5 # = not tested % Rec = % recovery LoQ = limit of quantification

Table 1 (continued): Results and z-Scores for Azinphos-methyl, Boscalid and Chlordane (cis)

laboratory number	analyte											
	Azinphos-methyl assigned value: 66.9 µg/kg				Boscalid assigned value: 39.2 µg/kg				Chlordane (cis) assigned value: 48.3 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
013	59.6	87.9	5.5	-0.5	40.2	91.2	4.3	0.1	45.9	92.8	6.2	-0.2
014	61.5	75	30	-0.4	81.4	70-110	30	**4.9**	34.0	70-110	30	-1.3
015	0	98%	2ppb	**-4.5**	41.585	98%	2ppb	0.3	0	98%	2ppb	**-4.5**
016	63.4	98.9	6.95	-0.2	38.8	92.3	6.31	0.0	47.9	100.3	3.09	0.0
017	#				45.8	101	10	0.8	#			
018	59.86	100	10	-0.5	46.3	100	10	0.8	50.8	100	10	0.2
019	0	80	10	**-4.5**	33	80	10	-0.7	#			
020	61.5	104	10ppb	-0.4	30.7	102	10ppb	-1.0	42.1	100	10ppb	-0.6
021	54.2	101	10	-0.9	29.6	101	10	-1.1	50	108	10	0.2
022	106.8	103.1	0.01mg/kg	**2.7**	56.5	98.1	0.01mg/kg	2.0	0	101.3	0.01mg/kg	**-4.5**
023	72	82	10	0.3	36	90	10	-0.4	#			
024	67.49		10	0.0	41.03		10	0.2	370.69		10	**30.4**

z-scores outside |z| >2 are shown in **bold,** see Section 5 # = not tested % Rec = % recovery LoQ = limit of quantification

Table 1 (continued): Results and z-Scores for Azinphos-methyl, Boscalid and Chlordane (cis)

laboratory number	analyte											
	Azinphos-methyl assigned value: 66.9 µg/kg				Boscalid assigned value: 39.2 µg/kg				Chlordane (cis) assigned value: 48.3 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
025	47	101	0.01mg/kg	-1.4	27	97	0.01mg/kg	-1.4	0		0.01mg/kg	**-4.5**
026	51	91	10	-1.1	31	94	10	-1.0	49	107	10	0.1
027	#				51	97	10	1.4	#			
028	57.9	92	10	-0.6	35.9	103.5	10	-0.4	38.0	92	10	-1.0
029	79	95	10	0.8	46	94	10	0.8	54	97	10	0.5
030	52.6	70-120	10ppb	-1.0	0	70-120	10ppb	**-4.5**	#			
031	#				37.39	85	10	-0.2	#			
032	87.7	107	10	1.4	43.5	81	10	0.5	46.0	73	10	-0.2
033	137.5	105	10	**4.8**	23.2	100	10	-1.9	#			
034	75.31	96.60	10	0.6	23.41	95.80	10	-1.8	#			
035	78.96		0.010	0.8	44.98		0.010	0.7	46.98		0.010	-0.1
036	#				#				0	90	10	**-4.5**

z-scores outside |z| >2 are shown in **bold,** see Section 5 # = not tested % Rec = % recovery LoQ = limit of quantification

Table 1 (continued): Results and z-Scores for Azinphos-methyl, Boscalid and Chlordane (cis)

laboratory number	analyte											
	Azinphos-methyl assigned value: 66.9 µg/kg				Boscalid assigned value: 39.2 µg/kg				Chlordane (cis) assigned value: 48.3 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
037	71.01	107.61%		0.3	#				43.91	87.17%		-0.4
038	75	100	10	0.5	46	100	10	0.8	44	100	10	-0.4
039	63.2	90	10	-0.3	37.9	90	10	-0.2	45.6	90	10	-0.3
040	65.476	95.1	10	-0.1	37.202	94.6	10	-0.2	54.127	91.1	10	0.6
041	#				41.4	90~110	10	0.3	#			
042	#				45	100	10	0.7	#			
043	0	70-120	10	**-4.5**	41	95	10	0.2	41.7	86	10	-0.6
044	67.3	104	10	0.0	39.8	103	10	0.1	52.1	106	10	0.4
045	76	100	10	0.6	23.5	100	10	-1.8	77	100	10	**2.7**
046	69	100	10	0.1	48	90	10	1.0	55	100	10	0.6

z-scores outside |z| >2 are shown in **bold,** see Section 5 # = not tested % Rec = % recovery LoQ = limit of quantification

Table 2: Results and z-Scores for Cypermethrin, Endosulfan I and Fluopyram

laboratory number	analyte											
	Cypermethrin assigned value: 104 µg/kg				Endosulfan I assigned value: 48.7 µg/kg				Fluopyram assigned value: 64.9 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
001	#				#				#			
002	78	80-120	10	-1.1	44	80-120	10	-0.4	69	80-120	10	0.3
003	78.2	85	20	-1.1	26.3	103	10	**-2.1**	77.6	115	5	0.9
004	96	98	40	-0.3	43	95	40	-0.5	#			
005	122.0	100	10	0.8	0	100	10	**-4.5**	68.3	100	10	0.2
006	79	108	20	-1.1	40	97	10	-0.8	63	98	10	-0.1
007	115	121	10	0.5	62	86	10	1.2	66	83	10	0.1
008	30	90	10ppb	**-3.2**	#				#			
009	#				#				#			
010	125.8	112.1	10	1.0	51.07	84.7	10	0.2	#			
011	102.2	92.6	5	-0.1	46.5	87.5	5	-0.2	64.7	89.8	5	0.0
012	#				0	70-120	10	**-4.5**	86.22	70-120	10	1.5

z-scores outside |z| >2 are shown in **bold,** see Section 5 # = not tested % Rec = % recovery LoQ = limit of quantification

Table 2 (continued): Results and z-Scores for Cypermethrin, Endosulfan I and Fluopyram

laboratory number	analyte											
	Cypermethrin assigned value: 104 µg/kg				Endosulfan I assigned value: 48.7 µg/kg				Fluopyram assigned value: 64.9 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
013	112	90.8	2.6	0.4	#				63.2	101.1	4.6	-0.1
014	137.0	110	30	1.4	40.0	90	30	-0.8	#			
015	0	98%	2ppb	**-4.5**	0	98%	2ppb	**-4.5**	55.88	98%	2ppb	-0.6
016	106.2	101.2	3.93	0.1	#				63.1	99.8	3.31	-0.1
017	#				#				#			
018	135.3	100	10	1.4	71.1	100	10	**2.1**	78.6	100	10	1.0
019	109	80	10	0.2	31	80	10	-1.7	65	80	10	0.0
020	112.3	105	10ppb	0.4	45.5	100	10ppb	-0.3	56.8	99	10ppb	-0.6
021	118	114	10	0.6	53	103	10	0.4	57.7	97	10	-0.5
022	105.9	97.7	0.01mg/kg	0.1	63.1	104.3	0.01mg/kg	1.3	#			
023	90	94	10	-0.6	54	77	10	0.5	61	95	10	-0.3
024	137.60		10	1.5	44.03		10	-0.4	89.80		10	1.7

z-scores outside |z| >2 are shown in **bold,** see Section 5 # = not tested % Rec = % recovery LoQ = limit of quantification

Table 2 (continued): Results and z-Scores for Cypermethrin, Endosulfan I and Fluopyram

laboratory number	analyte											
	Cypermethrin assigned value: 104 µg/kg				Endosulfan I assigned value: 48.7 µg/kg				Fluopyram assigned value: 64.9 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
025	71	109	0.01mg/kg	-1.4	45	98	0.01mg/kg	-0.3	49	100	0.01mg/kg	-1.1
026	130	95	10	1.1	49	103	10	0.0	51	94	10	-1.0
027	148	111	10	1.9	49	97	10	0.0	#			
028	98.7	82	10	-0.2	45.3	127	10	-0.3	61.8	99	10	-0.2
029	110	101	10	0.3	58	110	10	0.9	71	105	10	0.4
030	#				#				53.6	70-120	10ppb	-0.8
031	#				#				60.77	85	10	-0.3
032	109.6	76	30	0.2	47.4	78	20	-0.1	66.5	87	10	0.1
033	140.9	91	10	1.6	58.6	99	10	0.9	#			
034	82.80	95.80	10	-0.9	52.83	95.60	10	0.4	73.09	102.50	10	0.6
035	112.94		0.010	0.4	43.98		0.010	-0.4	66.97		0.010	0.1
036	204.6	90	100	**4.4**	#				#			

z-scores outside |z| >2 are shown in **bold,** see Section 5 # = not tested % Rec = % recovery LoQ = limit of quantification

Table 2 (continued): Results and z-Scores for Cypermethrin, Endosulfan I and Fluopyram

laboratory number	analyte											
	Cypermethrin assigned value: 104 µg/kg				Endosulfan I assigned value: 48.7 µg/kg				Fluopyram assigned value: 64.9 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
037	78.98	84.17%		-1.1	49.46	91.03%		0.1	#			
038	87	100	10	-0.7	31	100	10	-1.7	67	100	10	0.1
039	101.9	90	10	-0.1	#				66.3	90	10	0.1
040	100.913	93.5	10	-0.1	53.056	94.8	10	0.4	67.56	100.8	10	0.2
041	#				#				#			
042	63	100	10	-1.8	48	100	10	-0.1	70	100	10	0.4
043	66.5	98	10	-1.6	38.4	86	10	-1.0	46.6	87	10	-1.3
044	103	89	10	0.0	#				71.3	104	10	0.4
045	104	100	10	0.0	65	100	10	1.5	74	100	10	0.6
046	93	100	10	-0.5	48	110	10	-0.1	67	110	10	0.1

z-scores outside |z| >2 are shown in **bold,** see Section 5 # = not tested % Rec = % recovery LoQ = limit of quantification

Table 3: Results and z-Scores for Flutriafol, Kresoxim-methyl and Oxadiazon

laboratory number	analyte											
	Flutriafol assigned value: 43.5 µg/kg				Kresoxim-methyl assigned value: 23.0 µg/kg				Oxadiazon assigned value: 68.6 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
001	#				#				62.34			-0.4
002	54	80-120	10	1.1	24	80-120	10	0.2	76	80-120	10	0.5
003	50.9	100	5	0.8	29.6	94	5	1.3	#			
004	#				#				#			
005	46.2	100	10	0.3	25.6	100	10	0.5	#			
006	36	99	10	-0.8	20	96	10	-0.6	62	106	10	-0.4
007	44	102	10	0.0	23	108	10	0.0	71	94	10	0.2
008	#				#				43	85	10ppb	-1.7
009	#				#				#			
010	43.25	90.7	10	0.0	25.36	88.3	10	0.5	69.18	91.7	10	0.0
011	0			**-4.5**	15.2	84.1	5	-1.5	48.3	82.9	5	-1.3
012	34.12	70-120	10	-1.0	21.62	70-120	10	-0.3	#			

z-scores outside |z| >2 are shown in **bold,** see Section 5 # = not tested % Rec = % recovery LoQ = limit of quantification

Table 3 (continued): Results and z-Scores for Flutriafol, Kresoxim-methyl and Oxadiazon

laboratory number	analyte											
	Flutriafol assigned value: 43.5 µg/kg				Kresoxim-methyl assigned value: 23.0 µg/kg				Oxadiazon assigned value: 68.6 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
013	#				21.7	103.4	7.4	-0.3	62.5	100.7	7.3	-0.4
014	61.4	70-110	30	1.9	#				#			
015	43.635	98%	2ppb	0.0	0	98%	2ppb	**-4.5**	0	98%	2ppb	**-4.5**
016	#				21.3	95.5	6.20	-0.3	65.6	92.8	7.22	-0.2
017	51.7	101	10ppb	0.9	26.0	95.2	10ppb	0.6	#			
018	48.5	100	10	0.5	27.1	100	10	0.8	#			
019	25	80	10	-1.9	26	80	10	0.6	74	80	10	0.4
020	#				20.2	103	10ppb	-0.6	58.7	105	10ppb	-0.7
021	36.9	96	10	-0.7	20	99	10	-0.6	71.9	99	10	0.2
022	43.9	103	0.01mg/kg	0.0	39.3	134.7	0.01mg/kg	**3.2**	63.8	102.8	0.01mg/kg	-0.3
023	38	85	10	-0.6	24	100	10	0.2	#			
024	20.78		10	**-2.4**	20.43		10	-0.5	46.79		10	-1.4

z-scores outside |z| >2 are shown in **bold,** see Section 5 # = not tested % Rec = % recovery LoQ = limit of quantification

Table 3 (continued): Results and z-Scores for Flutriafol, Kresoxim-methyl and Oxadiazon

laboratory number	analyte											
	Flutriafol assigned value: 43.5 µg/kg				Kresoxim-methyl assigned value: 23.0 µg/kg				Oxadiazon assigned value: 68.6 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
025	35	94	0.01mg/kg	-0.9	19	89	0.01mg/kg	-0.8	#			
026	32	86	10	-1.2	18	92	10	-1.0	63	89	10	-0.4
027	0			**-4.5**	25	91	10	0.4	72	100	10	0.2
028	32.5	103.5	10	-1.2	18	96	10	-1.0	64.7	105	10	-0.3
029	49	101	10	0.6	29	101	10	1.2	81	102	10	0.8
030	0	70-120	10ppb	**-4.5**	14.0	70-120	10ppb	-1.8	#			
031	39.85	85	10	-0.4	#				58.16	85	10	-0.7
032	#				#				#			
033	41.0	95	10	-0.3	29.2	106	10	1.2	#			
034	36.34	95.60	10	-0.8	23.21	95.70	10	0.0	#			
035	45.98		0.010	0.3	23.99		0.010	0.2	80.96		0.010	0.8
036	#				50.5	96.7	10	**5.4**	#			

z-scores outside |z| >2 are shown in **bold,** see Section 5 # = not tested % Rec = % recovery LoQ = limit of quantification

Table 3 (continued): Results and z-Scores for Flutriafol, Kresoxim-methyl and Oxadiazon

laboratory number	analyte											
	Flutriafol assigned value: 43.5 µg/kg				Kresoxim-methyl assigned value: 23.0 µg/kg				Oxadiazon assigned value: 68.6 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
037	41.17	90.38%		-0.2	24.86	95.79%		0.4	65.71	92.54%		-0.2
038	53	100	10	1.0	23	100	10	0.0	92	100	10	1.5
039	#				20.0	90	10	-0.6	62.3	90	10	-0.4
040	38.988	93.7	10	-0.5	23.115	91.2	10	0.0	62.302	89.9	10	-0.4
041	#				20.7	90~110	10	-0.5	#			
042	44	100	10	0.0	<LOQ	100	10		73	100	10	0.3
043	52.9	90	10	1.0	16.1	81	10	-1.4	93.5	94	10	1.6
044	42.3	98	10	-0.1	25.0	102	10	0.4	68.5	102	10	0.0
045	50.5	100	10	0.7	<LOQ	100	10		138.5	100	10	**4.6**
046	57	100	10	1.4	23	100	10	0.0	77	100	10	0.6

z-scores outside |z| >2 are shown in **bold,** see Section 5 # = not tested % Rec = % recovery LoQ = limit of quantification

Table 4: Results and z-Scores for Pirimiphos-ethyl and Triadimenol

laboratory number	analyte							
	Pirimiphos-ethyl assigned value: 67.1 µg/kg				Triadimenol assigned value: 40.4 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
001	#				39.66			-0.1
002	0	80-120		**-4.5**	40	80-120	10	0.0
003	#				49.2	94	5	1.0
004	#				#			
005	#				37.9	100	10	-0.3
006	63	105	10	-0.3	25	96	20	-1.7
007	71	109	10	0.3	32	114	10	-0.9
008	49	80	10ppb	-1.2	0	70-120	10ppb	**-4.5**
009	#				#			
010	71.37	92.4	10	0.3	41.5	87.4	10	0.1
011	60.7	88.7	5	-0.4	37.6	91.6	5	-0.3
012	#				44.17	70-120	10	0.4

z-scores outside |z| >2 are shown in **bold,** see Section 5 # = not tested % Rec = % recovery LoQ = limit of quantification

Table 4 (continued): Results and z-Scores for Pirimiphos-ethyl and Triadimenol

laboratory number	analyte							
	Pirimiphos-ethyl assigned value: 67.1 µg/kg				Triadimenol assigned value: 40.4 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
013	62	108.9	9.9	-0.3	41.2	100.4	8.7	0.1
014	#				#			
015	0	98%	2ppb	**-4.5**	0	98%	2ppb	**-4.5**
016	67.4	105.6	1.64	0.0	42	101.9	6.86	0.2
017	#				35.9	92	10ppb	-0.5
018	84.5	100	10	1.2	50.9	100	10	1.2
019	59	80	10	-0.6	37	80	10	-0.4
020	64.3	104	10ppb	-0.2	#			
021	69	108	10	0.1	36.3	98	10	-0.5
022	77.9	115.4	0.01mg/kg	0.7	54.1	105	0.01mg/kg	1.5
023	70	100	10	0.2	35	90	10	-0.6
024	60.75		10	-0.4	39.84		10	-0.1

z-scores outside |z| >2 are shown in **bold,** see Section 5 # = not tested % Rec = % recovery LoQ = limit of quantification

Table 4 (continued): Results and z-Scores for Pirimiphos-ethyl and Triadimenol

laboratory number	analyte							
	Pirimiphos-ethyl assigned value: 67.1 µg/kg				Triadimenol assigned value: 40.4 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
025	61	100	0.01mg/kg	-0.4	40	84	0.01mg/kg	0.0
026	72	108	10	0.3	42	93	10	0.2
027	72	105	10	0.3	30	94	10	-1.2
028	47.9	72.5	10	-1.3	39.7	89	10	-0.1
029	79	96	10	0.8	51	100	10	1.2
030	#				59.6	70-120	10ppb	**2.2**
031	61.71	85	10	-0.4	#			
032	65.2	75	10	-0.1	#			
033	88.5	109	10	1.4	42.9	82	10	0.3
034	#				39.11	95.00	10	-0.1
035	63.97		0.010	-0.2	42.98		0.010	0.3
036	115.4	94	10	**3.3**	#			

z-scores outside |z| >2 are shown in **bold,** see Section 5 # = not tested % Rec = % recovery LoQ = limit of quantification

Table 4 (continued): Results and z-Scores for Pirimiphos-ethyl and Triadimenol

laboratory number	analyte							
	Pirimiphos-ethyl assigned value: 67.1 µg/kg				Triadimenol assigned value: 40.4 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
037	73.82	86.93%		0.5	#			
038	54	100	10	-0.9	33	100	10	-0.8
039	64.2	90	10	-0.2	42	90	10	0.2
040	61.329	86.0	10	-0.4	34.196	94.4	10	-0.7
041	64.7	90~110	10	-0.2	44.8	90~110	10	0.5
042	70	100	10	0.2	#			
043	50	80	10	-1.2	32.5	91	10	-0.9
044	73.8	105	10	0.5	43.0	104	10	0.3
045	75	100	10	0.5	0	100	10	**-4.5**
046	69	110	10	0.1	46	100	10	0.6

z-scores outside |z| >2 are shown in **bold,** see Section 5 # = not tested % Rec = % recovery LoQ = limit of quantification

Table 5: Additional Pesticide Residues Reported

laboratory number	pesticide residue ≥15 µg/kg	result µg/kg	recovery %	LoQ µg/kg
002	Pirimiphos-methyl	58	80 - 120	10
006	Heptachlor	59	94	10
008	Azinphos-ethyl	86	96	10 ppb
009	Prothiofos	120	95.8	900
012	Endosulfan II (beta)	43.38	70-120%	10
022	Chlordane (trans)	61.9	98.4	0.01mg/kg
025	Dieldrin - µg/kg	67	85	0,01 mg/kg
036	Chlordane (trans)	80.2	103	10
043	Azinphos-ethyl	80.5	94	10

Table 6: Participants' Comments

laboratory number	comments
028	The sample arrived room temperature, the bottle had deformity at the base and sides, which indicated the presence of gas inside, this gas can come from the decomposition of the sample.

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

Table 7: Assigned Values and Standard Deviations for Proficiency

analyte	data points, *n*	assigned value, x_a µg/kg	uncertainty, *u*	standard deviation for proficiency, σ_p	
Azinphos-methyl	30	66.9	2.6	Horwitz [8]	14.7
Boscalid	37	39.2	1.5	Horwitz [8]	8.63
Chlordane (cis)	23	48.3	1.3	Horwitz [8]	10.6
Cypermethrin	35	104	4	Horwitz [8]	22.9
Endosulfan I	28	48.7	1.9	Horwitz [8]	10.7
Fluopyram	31	64.9	1.5	Horwitz [8]	14.3
Flutriafol	29	43.5	1.6	Horwitz [8]	9.58
Kresoxim-methyl	33	23.0	0.8	Horwitz [8]	5.07
Oxadiazon	25	68.6	2.1	Horwitz [8]	15.1
Pirimiphos-ethyl	31	67.1	1.7	Horwitz [8]	14.8
Triadimenol	31	40.4	1.3	Horwitz [8]	8.89

Table 8: Number and Percentage of z-Scores where $|z| \leq 2$

analyte	number of scores where $\|z\| \leq 2$	total number of scores	% $\|z\| \leq 2$
Azinphos-methyl	32	37	86
Boscalid	39	41	95
Chlordane (cis)	23	31	74
Cypermethrin	36	39	92
Endosulfan I	29	34	85
Fluopyram	33	33	100
Flutriafol	31	35	89
Kresoxim-methyl	34	37	92
Oxadiazon	28	30	93
Pirimiphos-ethyl	33	36	92
Triadimenol	33	37	89

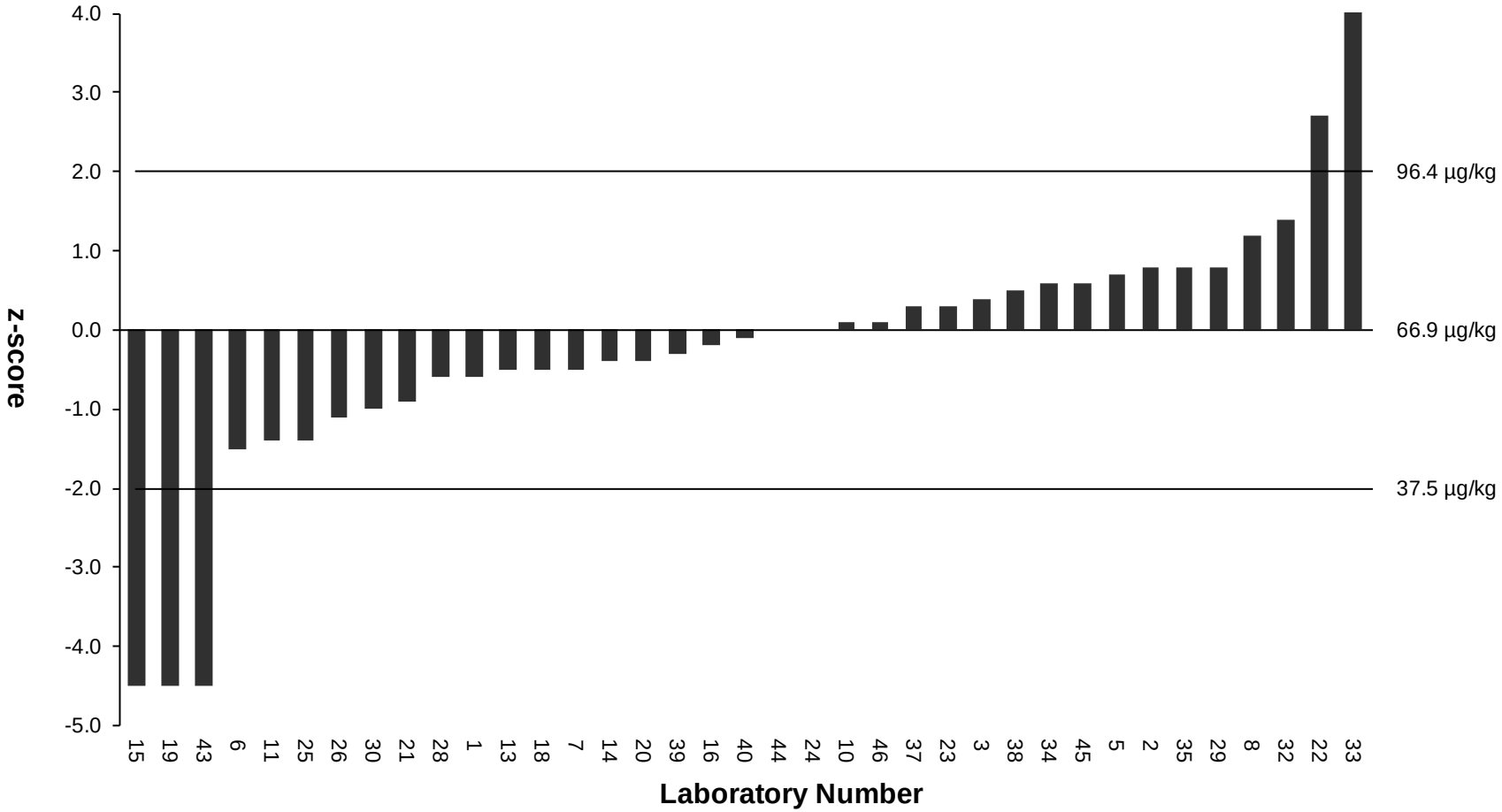

Figure 1: z-Scores for Azinphos-methyl

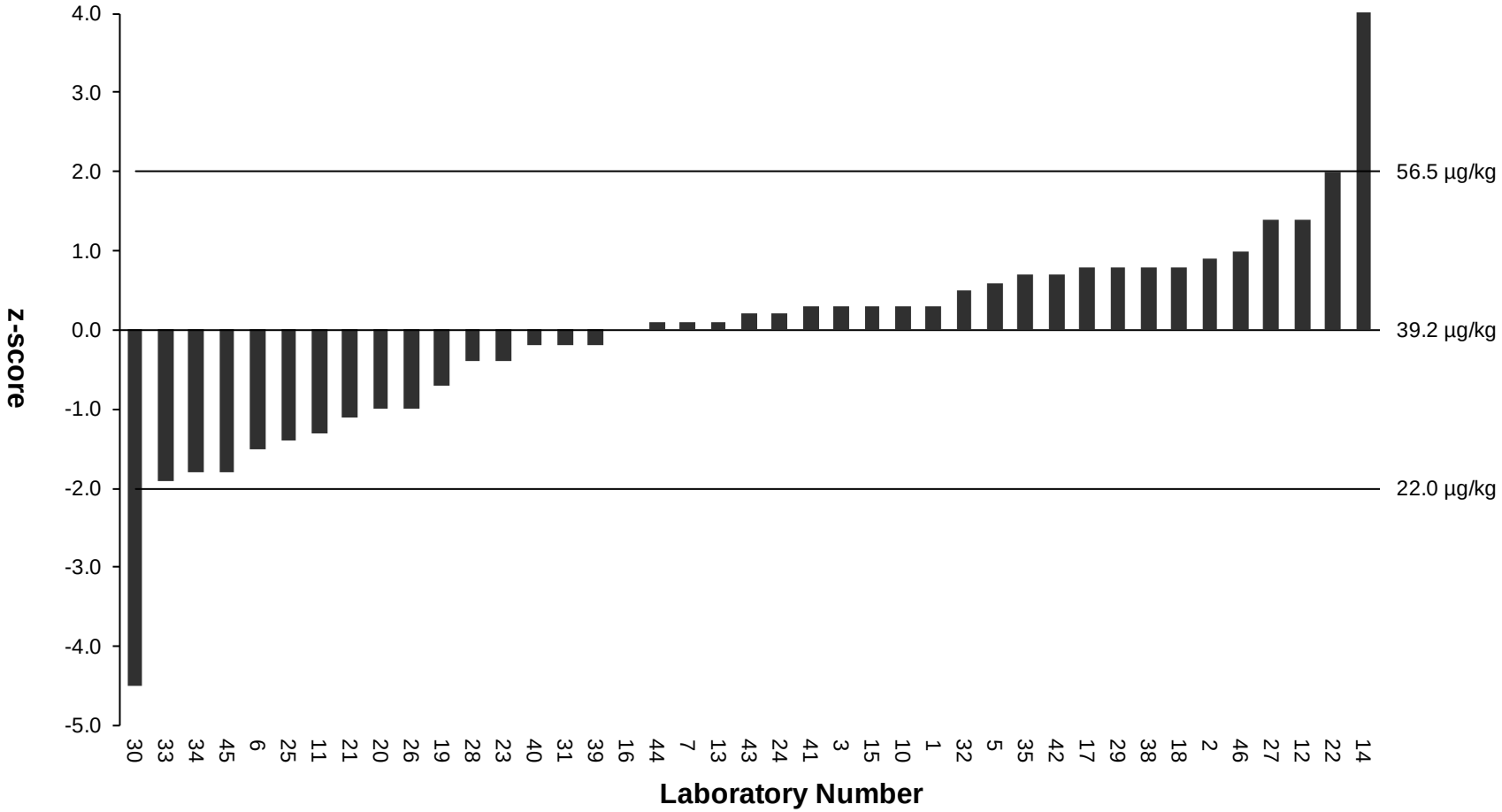

Figure 2: z-Scores for Boscalid

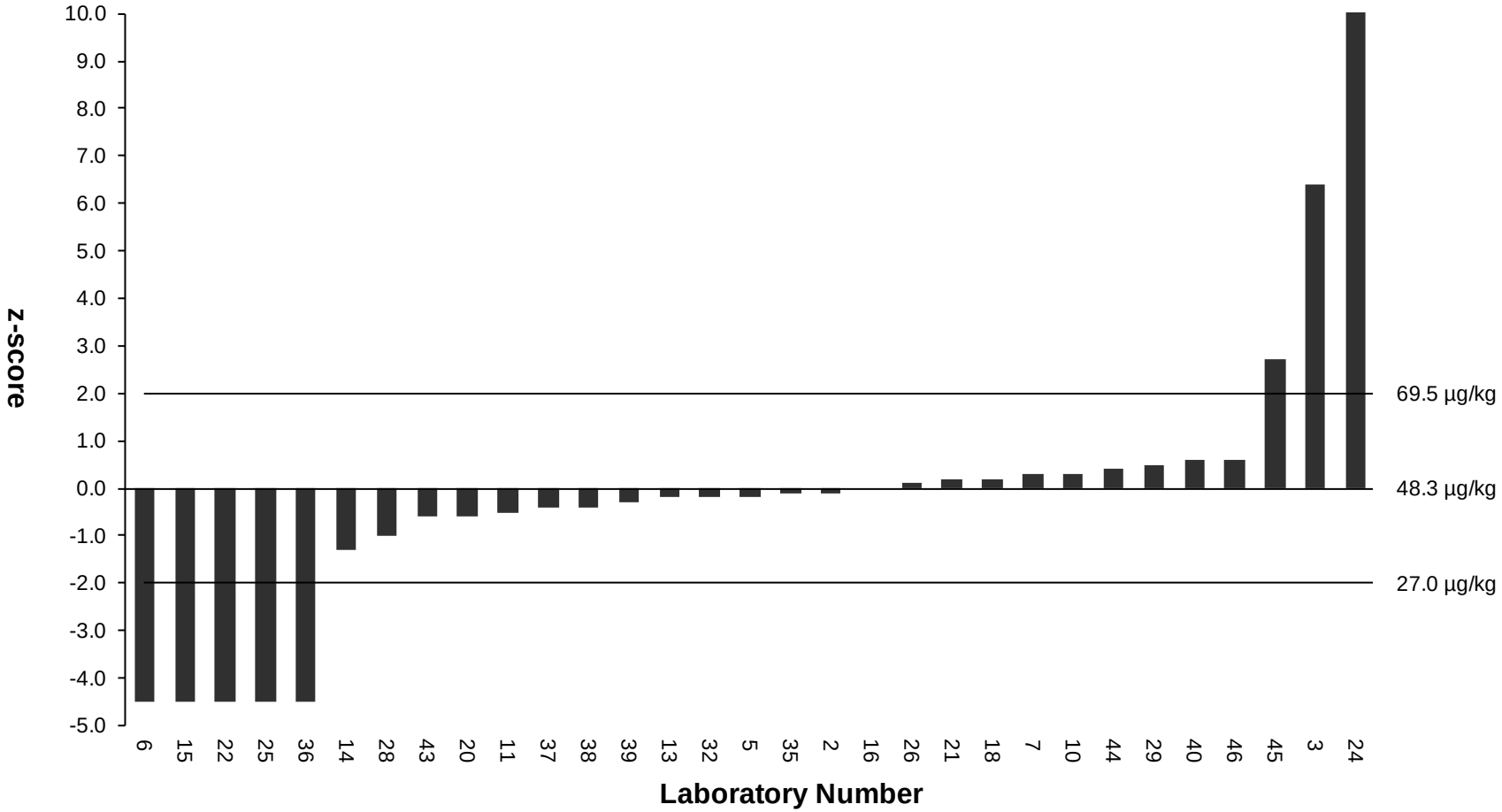

Figure 3: z-Scores for Chlordane (cis)

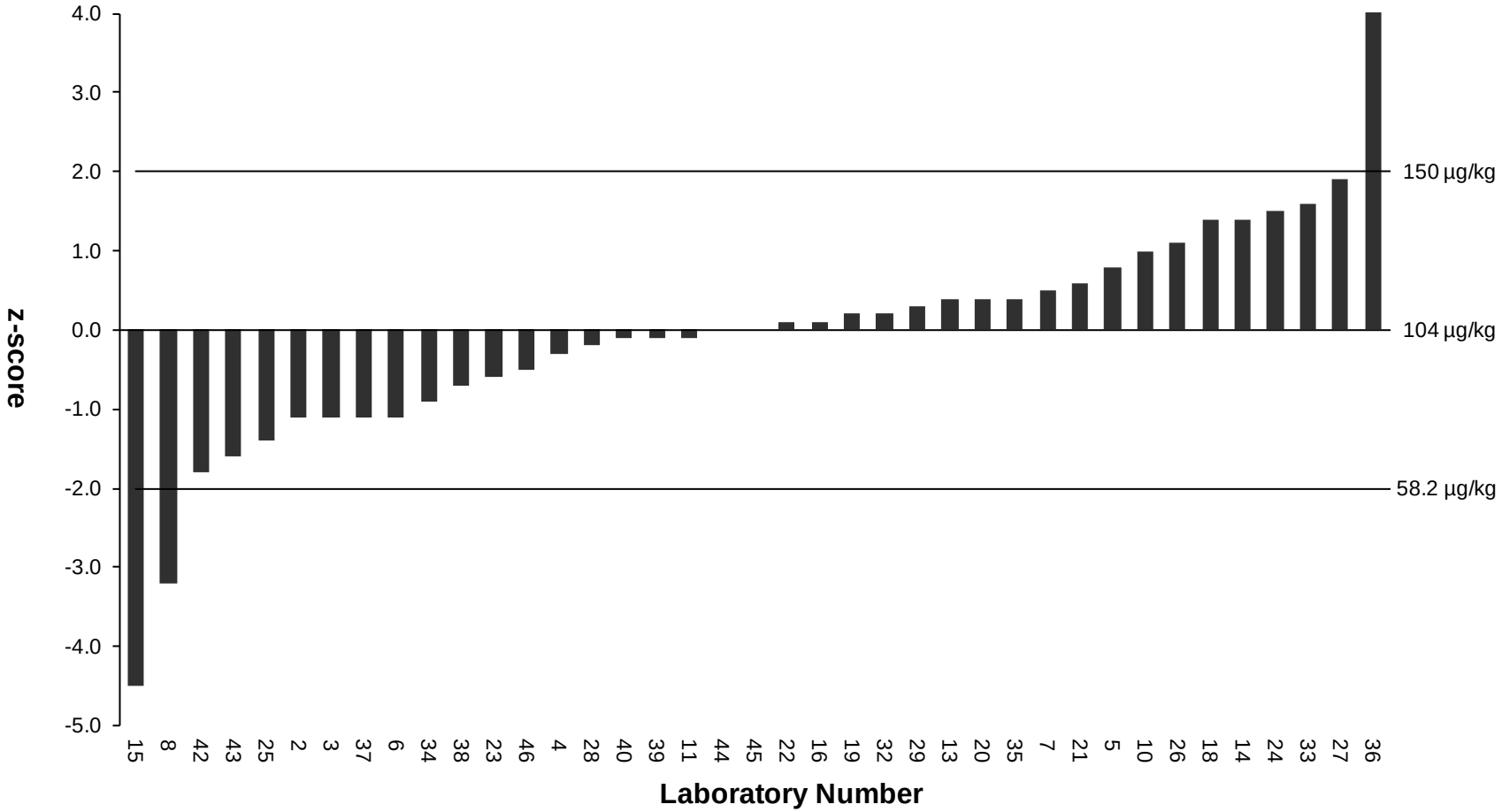

Figure 4: z-Scores for Cypermethrin

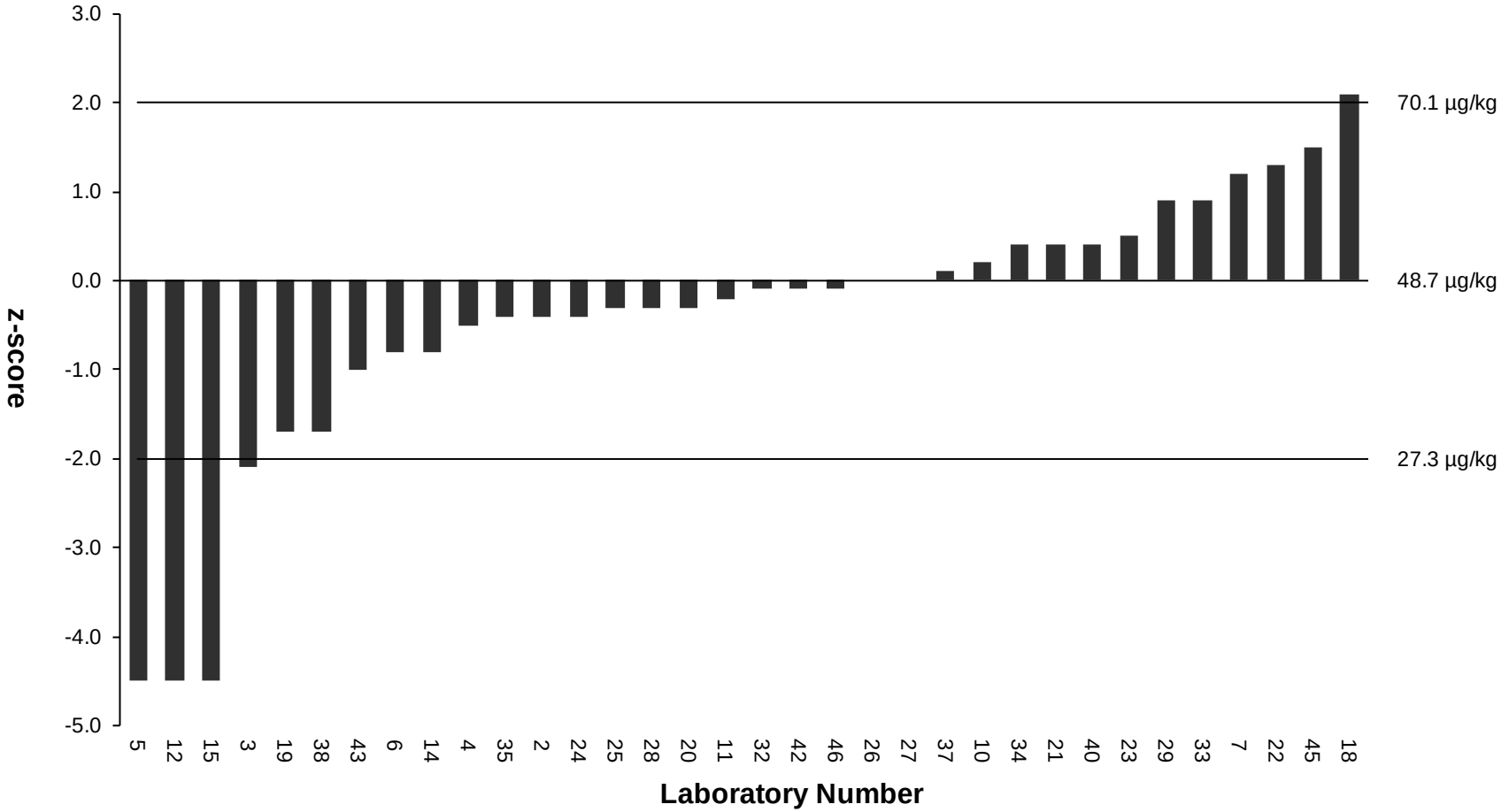

Figure 5: z-Scores for Endosulfan I

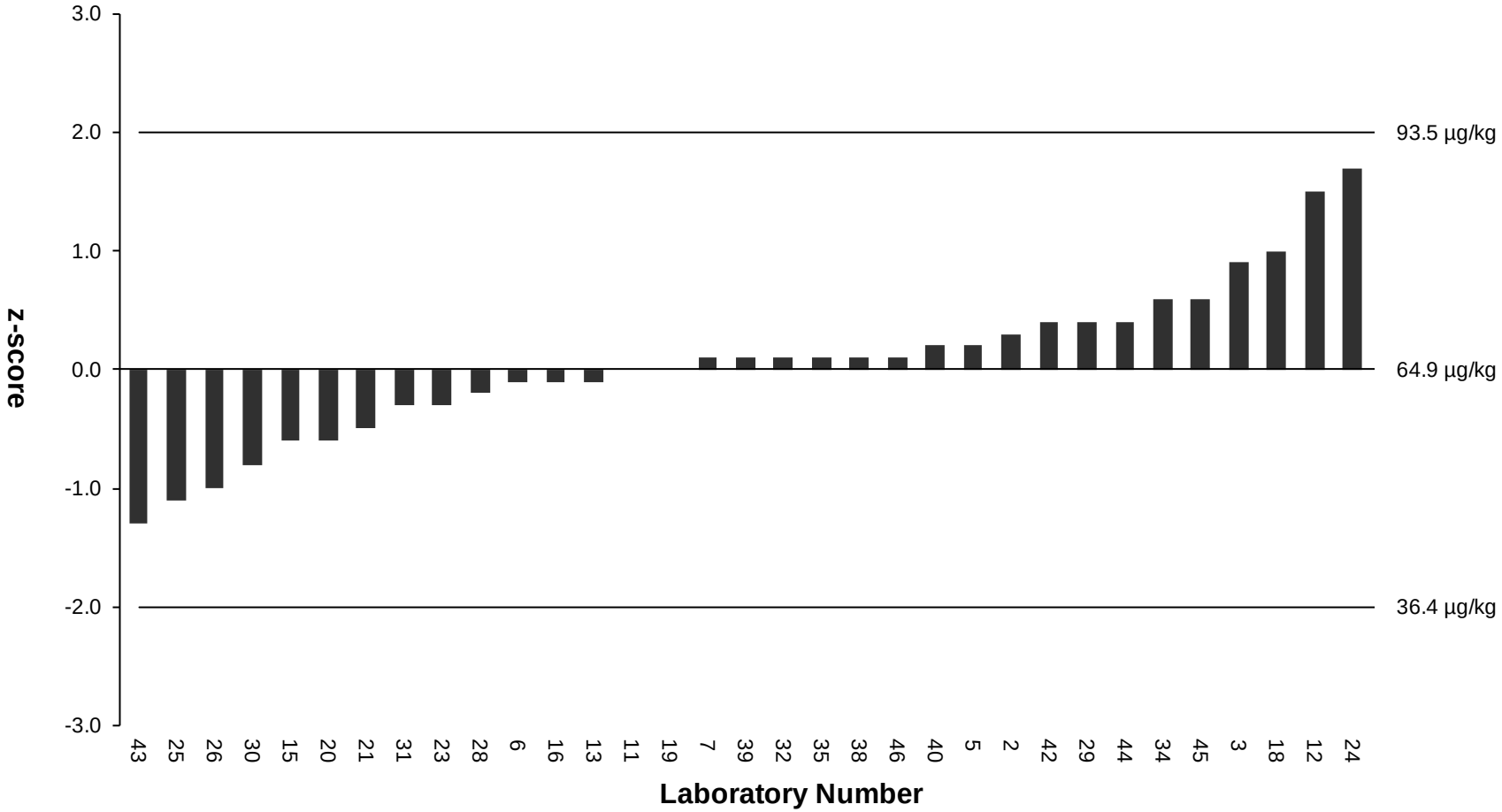

Figure 6: z-Scores for Fluopyram

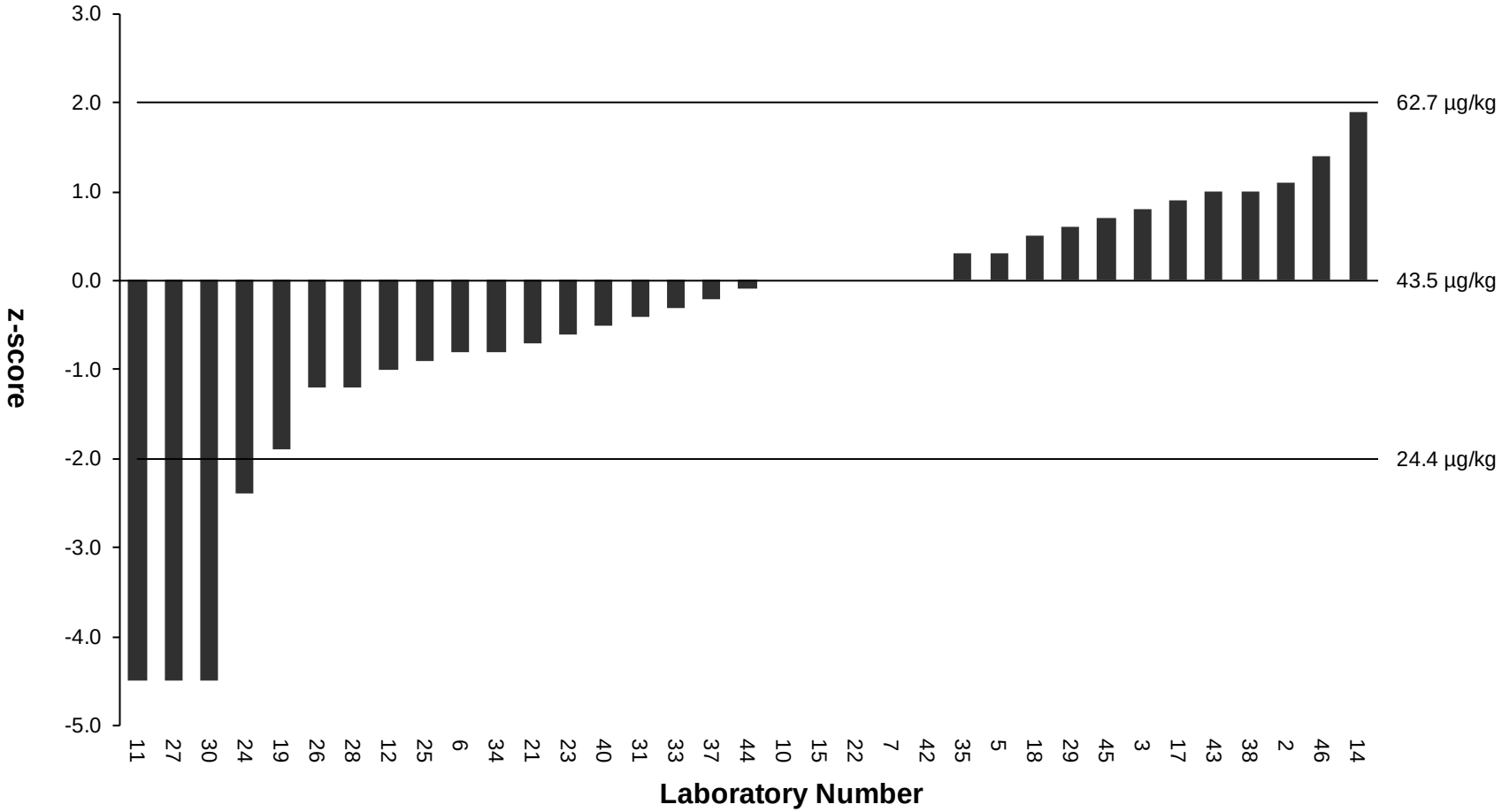

Figure 7: z-Scores for Flutriafol

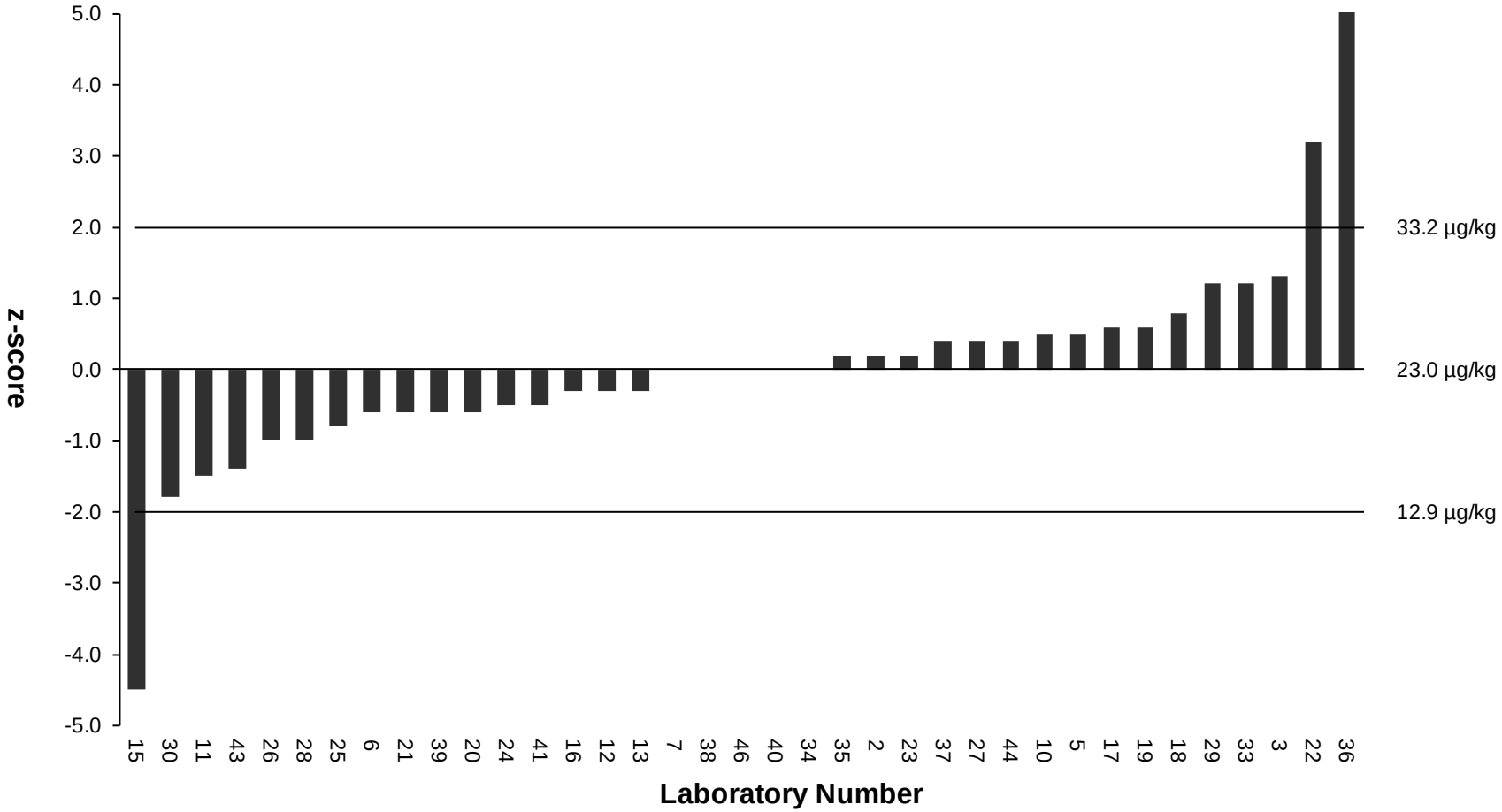

Figure 8: z-Scores for Kresoxim-methyl

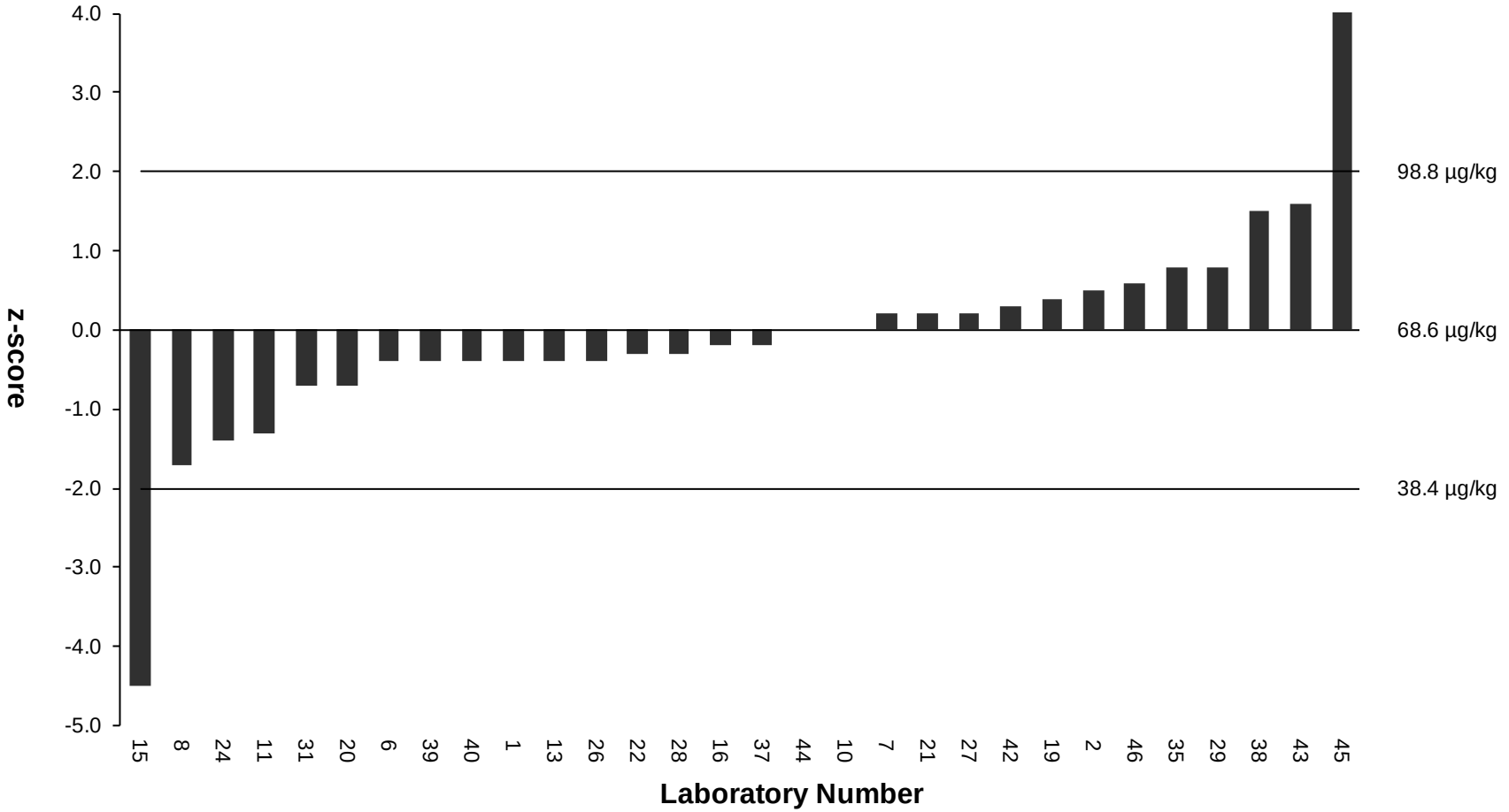

Figure 9: z-Scores for Oxadiazon

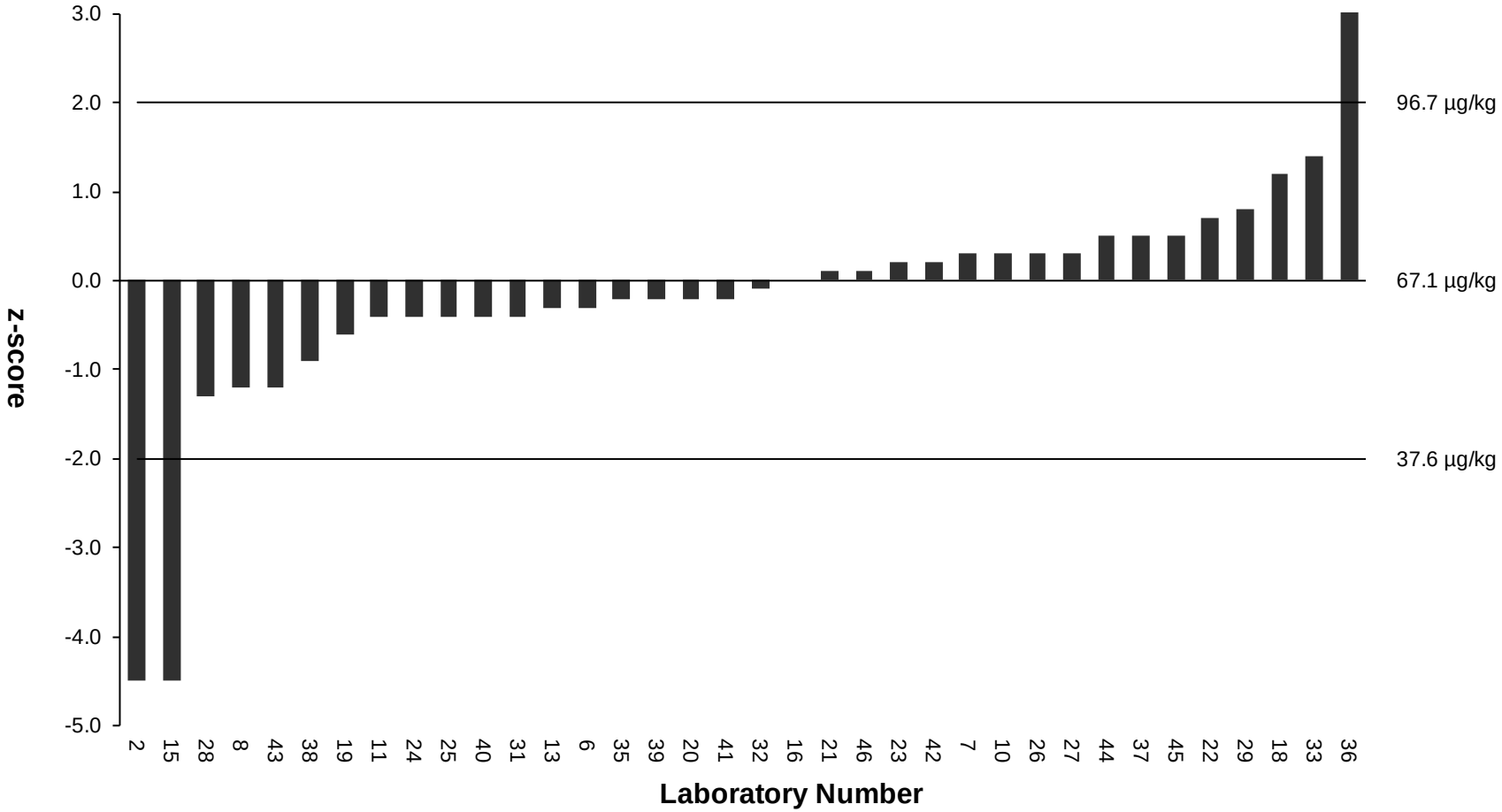

Figure 10: z-Scores for Pirimiphos-ethyl

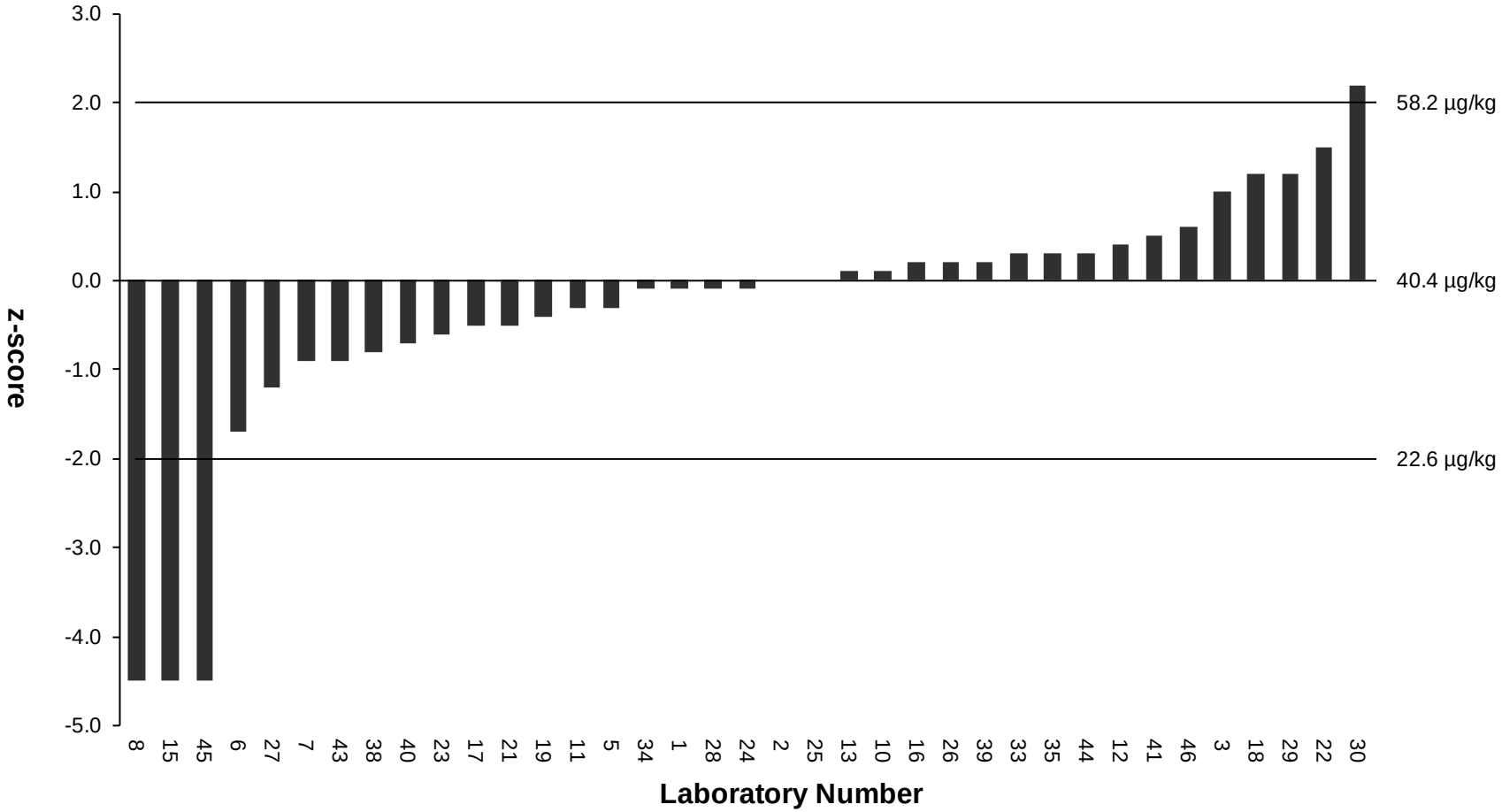

Figure 11: z-Scores for Triadimenol

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.

Accredited Method Used	laboratory number
no	001 010 017 028 030 031 032 034 036
yes	002 003 004 006 008 009 011 012 013 014 015 018 020 021 024 025 026 027 039 041 042 043 044

Method Based On	laboratory number
International Standard	002 004 008 011 020 021 024 025 026 031 034 039 042 043 044
National Standard	013 015 041
Manufacturer/Kit Instructions/Technical Note	012
In house method	003 006 009 010 014 017 018 027 028 030 032 036

Quoted percentage recovery measured in same analytical batch as test material	laboratory number
no	003 006 021 024 032
yes	002 004 008 009 010 011 012 013 014 015 017 018 020 025 026 027 028 030 031 034 036 039 041 042 043 044

If measured in this batch, stage spike added	laboratory number
prior to extraction	002 003 004 006 008 009 010 011 012 013 015 017 018 020 021 025 026 027 028 030 031 032 034 036 039 041 042 043 044
prior to clean up	014

Concentration of Spike (µg/kg)	laboratory number
≥1 - <5	039
≥5 - <10	018 021 024 030 034
≥10 - <25	002 004 012 015 025 027 032 036 043
≥25 - <50	026 031
≥50 - <100	003 006 008 010 013 014 017 028 041 042 044

Concentration of Spike (µg/kg) (continued)	laboratory number
≥100	009 011 020

Composition of Blank Commodity used for Spiking	laboratory number
blank provided	002 004 008 009 010 011 014 015 017 018 020 024 025 026 031 032 039 041 042 043 044
bellpepper	036
Cucumber	034
in house blank	021
Leafy Vegetable	030
tomato	012

Calibration	laboratory number
standard addition	012 015
matrix-matched	003 004 010 011 013 014 015 017 018 020 021 024 025 026 027 032 034 036 039 041 042 043 044
solvent	015 026 030
multi-level	002 006 008 009 025 026 028 030 031
single-level	026

Internal Standard Added	laboratory number
no	003 004 006 014 015 017 021 025 030 031 032 036 043 044
yes	002 008 009 010 011 012 013 018 020 024 026 027 028 034 039 041 042

Internal Standard Used	laboratory number
BNB	027
in House Standard	026
Carbaryl D7	018
Triphenylphosphate	002 009 010 011 012 013 018 020

GC Method: Sample Weight (g)	laboratory number
≥1 - <5	004 011 039 044
≥5 - <10	009 013 018 021 026 027 028 032 034 036 041
≥10 - <20	002 003 006 010 012 020 024 025 042 043
≥20 - <50	014

GC Method: Extraction Solvent Components	laboratory number
acetone	014
acetonitrile	002 003 004 009 010 011 012 013 018 020 021 024 025 026 027 028 032 034 036 039 041 042 044
ethyl acetate	006 043

GC Method: Extraction pH Adjusted	laboratory number
no	002 003 004 006 008 009 011 012 013 014 017 018 020 021 025 026 028 036 041 042 043 044
yes	010 027 032 034 039

GC Method: Extraction Techniques Used	laboratory number
QuEChERS	002 003 006 009 010 011 012 013 018 020 021 024 025 026 027 028 032 034 036 039 041 042 043 044
liquid-liquid partition	004 014

GC Method: Sample Clean-up Technique	laboratory number
liquid-liquid partition	004
none	002 006 020 021 044
solid phase extraction (SPE) (column/cartridge)	014
solid phase extraction (SPE) (dispersive)	003 009 010 012 013 018 024 025 026 027 028 032 034 039 041 042 043
QUECHERS	011

GC Method: SPE Sorbent Type	laboratory number
C18	025 032
PSA	003 010 012 013 018 025 026 027 028 034 039 041 042 043
aluminum oxide	014

GC Method: GC Column Packing	laboratory number
50% methyl 50% phenyl polysiloxane	002 021
65% methyl 35% phenyl polysiloxane	004 012
95% methyl 5% phenyl polysiloxane	003 006 010 013 014 018 026 027 028 032 034 039 041 042 043 044
5% Phenyl 95% dimethylarylene siloxane	011
5% phenyl methylpolysiloxane	036
capillary column	009

GC Method: GC Detector Type	laboratory number
ECD	004 014
FPD	004 014
MS	009 027
MS-MS	002 003 006 010 011 012 013 018 020 021 024 025 026 028 032 034 039 041 042 043 044
orbitrap	036

LC Method: Sample Weight (g)	laboratory number
≥1 - <5	011 039 044
≥5 - <10	008 013 018 021 026 028 032 034
≥10 - <20	002 003 006 010 012 014 015 017 020 025 027 030 031 041 042 043

LC Method: Extraction Solvent Components	laboratory number
acetonitrile	002 003 008 010 011 012 013 014 015 017 018 020 021 024 025 026 027 028 030 031 032 034 039 041 042 043 044
ethyl acetate	006
Acetic acid	015

LC Method: Extraction pH Adjusted	laboratory number
no	002 003 006 011 012 013 017 018 020 021 025 026 027 028 030 031 041 042 044
yes	008 010 014 015 032 034 039 043

LC Method: Extraction Techniques Used	laboratory number
QuEChERS	002 003 006 008 010 011 012 013 014 015 017 018 020 021 024 025 026 027 028 030 031 032 034 039 041 042 043 044

LC Method: Sample Clean-up Technique	laboratory number
liquid-liquid partition	041
none	002 006 013 020 021 028 030 039 044
solid phase extraction (SPE) (column/cartridge)	015
solid phase extraction (SPE) (dispersive)	003 010 012 014 015 017 018 024 025 026 027 031 032 034 042 043
QUECHERS	011

LC Method: SPE Sorbent Type	laboratory number
C18	013 021 027 044
Envicarb/GCB	008
PSA	003 010 012 014 015 017 018 026 031 032 034 041 042 043

LC Method: HPLC Column Packing	laboratory number
C18	002 003 006 008 010 011 012 013 014 015 017 018 020 021 025 026 027 028 032 034 039 041 042 043 044

LC method: Mobile Phase Components	laboratory number
acetonitrile	003
ammonium acetate	015 025 032
ammonium formate	008 011 012 017 025 027 030 031 043 044
formic acid (methanoic acid)	002 003 011 012 015 027 030 043 044

LC method: Mobile Phase Components (continued)	laboratory number
methanol	003 008 010 011 012 013 015 018 020 021 026 027 028 030 034 039 041 042 043 044
water	003 008 010 011 012 013 014 025 026 028 030 034 043 044
MEOH/2mM formiato amÃ³nico, 0.01% fórmico en agua	006

LC Method: Detector Type	laboratory number
HR-MS	030
MS-MS	002 003 006 010 011 012 013 014 015 017 018 020 021 025 026 027 028 031 032 034 039 041 042 043 044

Azinphos-methyl

Source of Standards	laboratory number
Accu Standard	002 011 013 020
Dr Ehrenstorfer	008 010 018 021 025 026 028 032
Merck	044
Sigma/Aldrich	014 028 034
lab instruments	038
Thermo Scientific	030

Method Principle	laboratory number
GC	014 018
LC	002 003 008 010 011 013 020 021 025 026 028 030 032 034 038 044

Identification by Mass Spectrometry	laboratory number
yes	002 003 008 010 011 013 014 018 020 021 025 026 028 030 032 034 038 044

Boscalid

Source of Standards	laboratory number
Accu Standard	002 011 013 020
Dr Ehrenstorfer	010 018 021 025 026 027 028 032 041
Merck	044
Sigma/Aldrich	014 028 034
Supelco	012
RESTEK	017

Method Principle	laboratory number
GC	010 027 032
LC	002 003 011 012 013 014 017 018 020 021 025 026 028 034 038 041 044

Identification by Mass Spectrometry	laboratory number
no	041
yes	002 003 010 011 012 013 014 017 018 020 021 025 026 027 028 032 034 038 044

Chlordane (cis)

Source of Standards	laboratory number
Accu Standard	011 014 020
Dr Ehrenstorfer	010 018 021 026 028 032
Merck	044
Sigma/Aldrich	028

Method Principle	laboratory number
GC	002 003 010 011 013 014 018 020 021 026 028 032 038 044

Identification by Mass Spectrometry	laboratory number
no	014
yes	002 003 010 011 013 018 020 021 026 028 032 038 044

Cypermethrin

Source of Standards	laboratory number
Accu Standard	002 011 020
Dr Ehrenstorfer	008 010 018 021 025 026 028 032 036
Merck	044
Sigma/Aldrich	014 034
Supelco	004

Method Principle	laboratory number
GC	002 003 004 010 011 013 014 018 020 021 025 026 028 032 034 036 038
LC	008 044

Identification by Mass Spectrometry	laboratory number
no	004 014
yes	002 003 008 010 011 013 018 020 021 025 026 028 032 034 036 038 044

Endosulfan I (alpha)

Source of Standards	laboratory number
Accu Standard	002 011 014 020
Dr Ehrenstorfer	010 018 021 025 026 027 028 032
Sigma/Aldrich	034
Supelco	004

Method Principle	laboratory number
GC	002 003 004 010 011 014 018 020 021 025 026 027 028 032 034 038

Identification by Mass Spectrometry	laboratory number
no	004 014
yes	002 003 010 011 018 020 021 025 026 027 028 032 034 038

Fluopyram

Source of Standards	laboratory number
Accu Standard	011 020
Dr Ehrenstorfer	018 021 025 026 028 032
Merck	044
Sigma/Aldrich	028 034
Supelco	012
Thermo Scientific	030

Method Principle	laboratory number
GC	002 011 013 020 032 034 038
LC	003 012 018 021 025 026 028 030 044

Identification by Mass Spectrometry	laboratory number
yes	002 003 011 012 013 018 020 021 025 026 028 030 032 034 038 044

Flutriafol

Source of Standards	laboratory number
Dr Ehrenstorfer	010 018 021 025 026 028
Merck	044
Sigma/Aldrich	014 028 034
Supelco	012

Method Principle	laboratory number
GC	010 018 025
LC	002 003 012 014 017 021 026 028 034 038 044

Identification by Mass Spectrometry	laboratory number
yes	002 003 010 012 014 017 018 021 025 026 028 034 038 044

Kresoxim-methyl

Source of Standards	laboratory number
Accu Standard	002 011 013 020
Dr Ehrenstorfer	010 018 021 025 026 027 028 036 041
Merck	044
Sigma/Aldrich	028 034
Supelco	012
RESTEK	017
Thermo Scientific	030

Method Principle	laboratory number
GC	002 010 012 018 025 027 034 036 038
LC	003 011 013 017 020 021 026 028 030 041 044

Identification by Mass Spectrometry	laboratory number
no	041
yes	002 003 010 011 012 013 017 018 020 021 025 026 027 028 030 034 036 038 044

Oxadiazon

Source of Standards	laboratory number
Accu Standard	011 013 020
Dr Ehrenstorfer	008 010 021 026 027 028
Merck	044
Sigma/Aldrich	028

Method Principle	laboratory number
GC	010 027 028
LC	002 008 011 013 020 021 026 038 044

Identification by Mass Spectrometry	laboratory number
yes	002 008 010 011 013 020 021 026 027 028 038 044

Pirimiphos-ethyl

Source of Standards	laboratory number
Accu Standard	011 013 020
Dr Ehrenstorfer	008 010 018 021 025 026 027 028 032 036 041
Merck	044
Sigma/Aldrich	028

Method Principle	laboratory number
GC	010 011 013 018 020 021 025 026 027 028 032 036 038 041
LC	008 044

Identification by Mass Spectrometry	laboratory number
no	041
yes	008 010 011 013 018 020 021 025 026 027 028 032 036 038 044

Triadimenol

Source of Standards	laboratory number
Accu Standard	002 011
Dr Ehrenstorfer	010 018 021 025 026 027 028 041
Merck	044
Sigma/Aldrich	028 034
Supelco	012
RESTEK	017
Thermo Scientific	030

Method Principle	laboratory number
GC	002 010 011 013 025 026 027 028 038 041

Method Principle (continued)	laboratory number
LC	003 012 017 018 021 030 034 044

Identification by Mass Spectrometry	laboratory number
no	041
yes	002 003 010 011 012 013 017 018 021 025 026 027 028 030 034 038 044

APPENDIX II: Fapas® SecureWeb, Protocol and Contact Details

1. Fapas® SECUREWEB

Access to the secure area of our website is only available to participants in our proficiency tests. Please contact us if you require a UserID and Password. Fapas® SecureWeb allows participants to:

- Obtain their laboratory numbers for the proficiency tests in which they have participated.
- View the results they submitted in past and current proficiency tests.
- Submit their results and methods for current tests.
- Review future tests they have ordered.
- Order proficiency tests, reference materials and quality control materials.
- Freely download copies of reports (PDF file), of proficiency tests in which they have participated.
- View charts of their z-scores obtained in previous Fapas® – Food Chemistry proficiency tests.

2. PROTOCOL

The Protocols [4, 5] set out how Fapas® – Food Chemistry is organised. Copies can be downloaded from our website.

3. CONTACT DETAILS

This report was prepared and authorised on behalf of Fapas® by Simon Hunter (Round Coordinator). Participants with any comments or concerns about this proficiency test should contact:

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