Fapas® – Food Chemistry Proficiency Test Report 19268

Pesticide Residues in Tomato Purée

May-June 2019

PARTICIPANT LABORATORY NUMBER

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SUMMARY

1. The test materials for Fapas® – Food Chemistry proficiency test 19268 were dispatched in May 2019. Each participant received a tomato purée 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
Chloridazon	107	46	57	81
Cymoxanil	92.2	80	90	89
Dimethomorph (sum of isomers)	44.4	84	96	88
Endosulfan-sulfate	98.2	72	89	81
Etrimfos	49.7	53	67	79
Fluopyram	41.5	76	83	92
Methoxyfenozide	50.4	85	94	90
Oxadixyl	60.6	82	91	90
Pirimicarb	32.1	89	98	91
Pyridalyl	44.0	40	54	74
Teflubenzuron	80.8	56	76	74

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1. INTRODUCTION

1.1. Proficiency Testing

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

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

2. TEST MATERIAL

2.1. Preparation

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

The test material was prepared from fresh tomatos. 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.

Chloridazon, Cymoxanil, Dimethomorph, Endosulfan-sulfate, Etrimfos, Fluopyram, Methoxyfenozide, Oxadixyl, Pirimicarb, Pyridalyl and Teflubenzuron 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 May 2019. Test materials were sent to 131 participants.

3. RESULTS

The instructions for reporting results were as follows:

- Determine the concentration of **pesticide residues** present in the test material, in µg/kg, as received together with the percentage recovery and limit of quantification (LoQ).
- All pesticide residues are to be reported as the **parent compound only**, unless specified otherwise.
- For each residue, select either "Not Detected", "Not Tested" or "Provide Result".

- For residues “Not Detected”, please enter an LOQ via the ‘add/edit results’ link on your list of tests. This has to be done after you have submitted results for all pesticide residues detected.
- For residues identified but not quantified, please select “Provide Result” and enter your result as BQL, i.e. below quantification limit.

Results were submitted by 124 participants (95%) before the closing date for this test, 21 June 2019.

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 Chloridazon, Cymoxanil, Dimethomorph, Endosulfan-sulfate, Etrimfos, Fluopyram, Methoxyfenozide, Oxadixyl, Pirimicarb, Pyridalyl and Teflubenzuron 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 where no percentage recovery was reported,

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

iv) results where no limit of quantification was reported.

For Chloridazon, Cymoxanil, Dimethomorph, Endosulfan-sulfate, Etrimfos, Fluopyram, Methoxyfenozide, Oxadixyl, Pirimicarb and Pyridalyl, this procedure was straightforward and the robust mean was chosen as the assigned value.

For Teflubenzuron, the mode was chosen as the assigned value because the distribution of results was skewed.

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
and σ_p = the standard deviation for proficiency.

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/11813/2017.

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 Chloridazon, Cymoxanil and Dimethomorph

laboratory number	analyte											
	Chloridazon assigned value: 107 µg/kg				Cymoxanil assigned value: 92.2 µg/kg				Dimethomorph assigned value: 44.4 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
001	#				73	125.6	10	-0.9	42	109.4	10	-0.2
002	119.3	98	10	0.5	90.7	96	10	-0.1	39.5	118	10	-0.5
003	89.66		10	-0.7	79.17		10	-0.6	33.59		10	-1.1
004	93.5		0.01 mg/kg	-0.6	76.8		0.01 mg/kg	-0.8	97.0		0.01 mg/kg	**5.4**
005	0			**-4.5**	92.6	109.8	1	0.0	#			
006	121.5	98.8	10.0	0.6	105.5	98.5	10.0	0.7	46.0	99.4	10.0	0.2
007	#				83.20	86.00	50.00	-0.4	40.20	95.00	20.00	-0.4
008	#				114	103	5	1.1	47	106	3.3	0.3
009	#				110	82	10	0.9	40	80	10	-0.5
010	105.56	70-120	0.005 mg/kg	-0.1	85.44	70-120	0.005 mg/kg	-0.3	34.87	70-120	0.005 mg/kg	-1.0
011	116.48	105	5	0.4	89.88	103	5	-0.1	45.51	105	5	0.1
012	120.50	110.62	10	0.6	95.22	117.94	10	0.1	41.08	104.85	10	-0.3
013	0		10	**-4.5**	#				43.23		10	-0.1
014	112	98	1.25	0.2	88.9	93	10.0	-0.2	42.2	102	2.50	-0.2
015	81	106	10	-1.1	88	70	10	-0.2	28	101	10	-1.7

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 Chloridazon, Cymoxanil and Dimethomorph

laboratory number	analyte											
	Chloridazon assigned value: 107 µg/kg				Cymoxanil assigned value: 92.2 µg/kg				Dimethomorph assigned value: 44.4 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
016	#				102	70-120	10	0.5	56	70-120	10	1.2
017	#				115.8	100	10	1.2	42.3	113	10	-0.2
018	119.8	117	10	0.5	106.4	109	10	0.7	43.2	106	10	-0.1
019	#				89	121	10	-0.2	44	87.5	10	0.0
020	#				92	90	10	0.0	41	87	10	-0.4
021	0	94~99	10	**-4.5**	0	94~99	10	**-4.5**	0	94~99	10	**-4.5**
022	#				83.3		0.010	-0.4	31.7	85	0.010	-1.3
023	120	96	10	0.5	97	103	10	0.2	43	91	10	-0.1
024	#				#				#			
025	#				#				#			
026	#				#				#			
027	#				#				#			
028	#				#				#			
029	#				99.7		10	0.4	48.2		10	0.4
030	118	102	10	0.5	93	101	10	0.0	50	101	10	0.6

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 Chloridazon, Cymoxanil and Dimethomorph

laboratory number	analyte											
	Chloridazon assigned value: 107 µg/kg				Cymoxanil assigned value: 92.2 µg/kg				Dimethomorph assigned value: 44.4 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
031	119.98	90.30	0.01mg/kg	0.5	89.08	103.80	0.01mg/kg	-0.2	43.23	103.60	0.01mg/kg	-0.1
032	0			**-4.5**	0			**-4.5**	0			**-4.5**
033	#				#				#			
034	#				95.5	99.8	10	0.2	48.3	100.0	10	0.4
035	#				#				#			
036	#				#				#			
037	#				94	79	5	0.1	37	94	5	-0.8
038	#				#				#			
039	#				#				#			
040	#				#				#			
041	#				#				#			
042	#				#				#			
043	131	80	10	1.0	102	80	10	0.5	41	80	10	-0.4
044	155	95	10	2.0	79	93	10	-0.7	85	98	10	**4.1**
045	#				80.5	87.2	10	-0.6	34.9	88.9	10	-1.0

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 Chloridazon, Cymoxanil and Dimethomorph

laboratory number	analyte											
	Chloridazon assigned value: 107 µg/kg				Cymoxanil assigned value: 92.2 µg/kg				Dimethomorph assigned value: 44.4 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
046	71	120	10	-1.5	96	110	10	0.2	41	100	10	-0.4
047	112		30	0.2	0			**-4.5**	37		10	-0.8
048	#				#				#			
049	85.91	88.04	8	-0.9	#				30.75	86.95	20	-1.4
050	#				#				#			
051	#				13.4	85	10	**-3.9**	35.2	95	10	-0.9
052	#				#				#			
053	#				#				47	118	20	0.3
054	#				#				#			
055	#				#				#			
056	#				#				0	70-120	10	**-4.5**
057	0	80-119.5	0.97-3.9	**-4.5**	132.1	103.68	1.9	2.0	61.2	98.13	0.97	1.7
058	0			**-4.5**	90.2	78	10	-0.1	87.5	75	10	**4.4**
059	0	100	10	**-4.5**	103	100	10	0.5	57	100	10	1.3
060	#				#				#			

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 Chloridazon, Cymoxanil and Dimethomorph

laboratory number	analyte											
	Chloridazon assigned value: 107 µg/kg				Cymoxanil assigned value: 92.2 µg/kg				Dimethomorph assigned value: 44.4 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
061	#				94.9		13.90	0.1	40.9		0.09	-0.4
062	#				#				#			
063	58	97.4	10	**-2.1**	63.	93.6	10	-1.4	31	107.1	10	-1.4
064	119	70-120	10	0.5	99	70-120	10	0.3	41	70-120	10	-0.4
065	150	88	10	1.8	98	93	10	0.3	43	98	10	-0.1
066	107	88	10	0.0	109.2	88	10	0.8	39.6	91	10	-0.5
067	81.24	75	10	-1.1	0	60-140	10	**-4.5**	44.01	96	10	0.0
068	112	90	10	0.2	85	90	10	-0.4	43	93	10	-0.1
069	126.8	97	10	0.8	102	97	10	0.5	51.6	108	10	0.7
070	#				87.01	90.0	5.0	-0.3	43.10	90.0	5.0	-0.1
071	#				0	70-120	10	**-4.5**	43.37	100	10	-0.1
072	#				*	105.00	133.20		52.00	88.00	16.65	0.8
073	128	86	10	0.9	117	90	10	1.2	59	84	10	1.5
074	#				#				#			
075	116.5	99.8	10	0.4	98.2	91.1	10	0.3	46.7	103.9	10	0.2

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

Table 1 (continued): Results and z-Scores for Chloridazon, Cymoxanil and Dimethomorph

laboratory number	analyte											
	Chloridazon assigned value: 107 µg/kg				Cymoxanil assigned value: 92.2 µg/kg				Dimethomorph assigned value: 44.4 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
076	#				#				#			
077	76	80-120	10	-1.3	63	80-120	10	-1.4	26	80-120	10	-1.9
078	114.40	92.38	10	0.3	97.67	94.65	10	0.3	49.05	95.11	10	0.5
079	#				118.06	70-120	10	1.3	51.51	70-120	10	0.7
080	#				103	> 90	50	0.5	33	> 90	50	-1.2
081	115	90	10	0.3	91	90	10	-0.1	45	90	10	0.1
082	#				77.7	85.1	10	-0.7	34.6	81.9	10	-1.0
083	75.7	90	5	-1.3	95.0	101	5	0.1	49.7	111	5	0.5
084	#				89.35	101	10	-0.1	40.55	88	10	-0.4
085	112	93	10	0.2	100	99	10	0.4	46	99	10	0.2
086	#				90.1		10	-0.1	46.6		10	0.2
087	#				91.8	100	10	0.0	46.6	100	10	0.2
088	113.00	90	10	0.3	87.00	91	10	-0.3	45.00	94	10	0.1
089	#				94.02	90.0	5.0	0.1	47.05	90.0	5.0	0.3
090	0		Varied	**-4.5**	92		50	0.0	22		10	**-2.3**

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 Chloridazon, Cymoxanil and Dimethomorph

laboratory number	analyte											
	Chloridazon assigned value: 107 µg/kg				Cymoxanil assigned value: 92.2 µg/kg				Dimethomorph assigned value: 44.4 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
091	113.2	98	10	0.3	84.5	97	55.6	-0.4	41.2	95	2	-0.3
092	#				0	80-120	0.010 mg/kg	**-4.5**	108	80-120	0.010 mg/kg	**6.5**
093	100		10	-0.3	92		10	0.0	47		10	0.3
094	112.2	96.7	5	0.2	82.7	99.0	5	-0.5	44.1	98.1	5	0.0
095	#				#				#			
096	#				#				50	104	10	0.6
097	#				52.6	116	10	-2.0	43.7	101	10	-0.1
098	0		10	**-4.5**	98.65		10	0.3	45.68		10	0.1
099	#				75.21	90.87	10	-0.8	40.11	99.05	10	-0.4
100	#				69	86	10	-1.1	17	98	10	**-2.8**
101	#				105	111	10	0.6	39	102	5	-0.6
102	#				#				#			
103	#				87.43	100	10	-0.2	44.05	100	10	0.0
104	#				#				#			
105	89	92.7	10	-0.8	99	102	10	0.3	45	94.8	10	0.1

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 Chloridazon, Cymoxanil and Dimethomorph

laboratory number	analyte											
	Chloridazon assigned value: 107 µg/kg				Cymoxanil assigned value: 92.2 µg/kg				Dimethomorph assigned value: 44.4 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
106	#				<LOQ		50		47.85	104	10	0.3
107	114		10	0.3	16		10	**-3.8**	87		10	**4.4**
108	106.5	99.1	10.0	0.0	94.9	111.1	10.0	0.1	46.9	97.4	10.0	0.3
109	#				#				50	111	10	0.6
110	127.9	94	10	0.9	108.5	95	10	0.8	50.7	94	10	0.6
111	#				0	70-120	0.01 mg/kg	**-4.5**	34.97	98.2	10.00	-1.0
112	0		0.05	**-4.5**	0		0.05	**-4.5**	0		0.05	**-4.5**
113	62	96	10	-1.9	84	95	10	-0.4	39	99	10	-0.6
114	106	120	10	0.0	97	104	10	0.2	50	108	10	0.6
115	90	100	10	-0.7	105	100	10	0.6	51	100	10	0.7
116	#				90	99	20	-0.1	91	101	10	**4.8**
117	#				87.5	65	10	-0.2	53.5	83	3	0.9
118	94.81	72	10	-0.5	88.42	87	10	-0.2	46.57	99	10	0.2
119	144.520	98	6	1.6	114.151	102	6	1.1	52.850	104	6	0.9
120	#				113.72		10	1.1	#			

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 Chloridazon, Cymoxanil and Dimethomorph

laboratory number	analyte											
	Chloridazon assigned value: 107 µg/kg				Cymoxanil assigned value: 92.2 µg/kg				Dimethomorph assigned value: 44.4 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
121	#				71	91	10	-1.0	43	98	10	-0.1
122	72.96	70-130	10	-1.4	72.96	70-130	10	-0.9	44.98	70-130	10	0.1
123	75	80	10	-1.4	66	80	10	-1.3	41.5	100	10	-0.3
124	#				#				#			

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 Endosulfan-Sulfate, Etrimfos and Fluopyram

laboratory number	analyte											
	Endosulfan-sulfate assigned value: 98.2 µg/kg				Etrimfos assigned value: 49.7 µg/kg				Fluopyram assigned value: 41.5 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
001	81	82.2	10	-0.8	27	90.8	10	**-2.1**	31	109.9	10	-1.1
002	116.0	102	10	0.8	#				#			
003	105.25		2	0.3	35.47		10	-1.3	32.48		10	-1.0
004	0		0.01 mg/kg	**-4.5**	57.5		0.01 mg/kg	0.7	46.6		0.01 mg/kg	0.6
005	#				45.1	100.8	1	-0.4	#			
006	23.5	97.9	10.0	**-3.5**	61.6	98.6	10.0	1.1	47.9	98.1	10.0	0.7
007	89.60	100.00	15.00	-0.4	#				39.90	88.00	2.00	-0.2
008	98	106	5	0.0	#				42	106	5	0.1
009	120	86	10	1.0	#				30	100	10	-1.3
010	#				66.41	70-120	0.005 mg/kg	1.5	38.84	70-120	0.005 mg/kg	-0.3
011	90.31	91	5	-0.4	69.05	92	5	1.8	42.80	111	5	0.1
012	111.52	75	10	0.6	39.74	61	10	-0.9	37.23	109.20	10	-0.5
013	132.75		10	1.6	40.52		10	-0.8	36.57		10	-0.5
014	99.3	92	2.50	0.1	47.7	94	1.25	-0.2	40.6	100	1.25	-0.1
015	45	108	10	**-2.5**	30	96	10	-1.8	33	70	10	-0.9

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 Endosulfan-Sulfate, Etrimfos and Fluopyram

laboratory number	analyte											
	Endosulfan-sulfate assigned value: 98.2 µg/kg				Etrimfos assigned value: 49.7 µg/kg				Fluopyram assigned value: 41.5 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
016	#				#				#			
017	75	79	10	-1.1	0		10	**-4.5**	39	99	10	-0.3
018	98	112	10	0.0	51	105	10	0.1	35.6	110	10	-0.6
019	#				#				48	85.1	10	0.7
020	100	93	5	0.1	#				40	93	10	-0.2
021	0	94~99	10	**-4.5**	0	94~99	10	**-4.5**	41.4	99.8	10	0.0
022	67.8	90	0.010	-1.4	#				36.3	80	0.010	-0.6
023	108	96	10	0.5	64	102	10	1.3	46	100	10	0.5
024	#				#				39	104.8	5	-0.3
025	#				#				46.5	96	4.1	0.6
026	#				#				41	90	10	-0.1
027	#				#				38	100	10	-0.4
028	#				#				37	90	10	-0.5
029	106.2		10	0.4	65.1		10	1.4	38.5		10	-0.3
030	188	96	10	**4.2**	59	107	10	0.8	47	102	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 (continued): Results and z-Scores for Endosulfan-Sulfate, Etrimfos and Fluopyram

laboratory number	analyte											
	Endosulfan-sulfate assigned value: 98.2 µg/kg				Etrimfos assigned value: 49.7 µg/kg				Fluopyram assigned value: 41.5 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
031	157.23	101.30	0.01mg/kg	**2.7**	59.07	89.60	0.01mg/kg	0.9	42.41	105.90	0.01mg/kg	0.1
032	0			**-4.5**	0			**-4.5**	41.73	99.93	3.0	0.0
033	#				#				#			
034	124.5	88.3	10	1.2	31.8	110.1	10	-1.6	54.6	115	10	1.4
035	#				#				#			
036	#				#				39.8	97.0	2.6	-0.2
037	114	88	5	0.7	#				38	82	10	-0.4
038	#				#				#			
039	#				#				38.5	94.3	10	-0.3
040	#				#				#			
041	#				53.63	82.12	2	0.4	#			
042	#				#				#			
043	65	80	10	-1.5	69	80	10	1.8	32	80	10	-1.0
044	45.1	112	10	**-2.5**	54	95	10	0.4	0	100ppb/10 ppb	10	**-4.5**
045	99.4	92	10	0.1	39.2	102.3	10	-1.0	35.1	97.3	10	-0.7

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 Endosulfan-Sulfate, Etrimfos and Fluopyram

laboratory number	analyte											
	Endosulfan-sulfate assigned value: 98.2 µg/kg				Etrimfos assigned value: 49.7 µg/kg				Fluopyram assigned value: 41.5 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
046	82	66	10	-0.7	60	93	10	0.9	37	101	10	-0.5
047	182		30	**3.9**	#				30		10	-1.3
048	202	104	10.00	**4.8**	#				#			
049	#				25.73	95.15	5	**-2.2**	#			
050	#				#				#			
051	176.3	110	10	**3.6**	51.5	100	10	0.2	56.3	100	10	1.6
052	#				#				41.69	96.52	5	0.0
053	100	98	10	0.1	#				#			
054	#				19	96.2	0.9	**-2.8**	#			
055	70	83.5	0.001 mg/kg	-1.3	#				#			
056	#				#				#			
057	84.7	80	1.9	-0.6	0	80-119.5	0.97-3.9	**-4.5**	0	80-119.5	0.97-3.9	**-4.5**
058	139.1	78	10	1.9	71.8	70	10	2.0	0			**-4.5**
059	105	100	10	0.3	55	100	10	0.5	44	100	10	0.3
060	#				#				39	100	10	-0.3

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 Endosulfan-Sulfate, Etrimfos and Fluopyram

laboratory number	analyte											
	Endosulfan-sulfate assigned value: 98.2 µg/kg				Etrimfos assigned value: 49.7 µg/kg				Fluopyram assigned value: 41.5 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
061	142		1	2.0	41.9		3	-0.7	#			
062	115.708	85.66	10	0.8	#				#			
063	20.2	73	10	**-3.6**	0			**-4.5**	0			**-4.5**
064	116	70-120	10	0.8	50	70-120	10	0.0	44	70-120	10	0.3
065	0		0.01	**-4.5**	#				32	94	10	-1.0
066	32	93	10	**-3.1**	#				#			
067	101.61	85	10	0.2	40.86	102	10	-0.8	#			
068	93	85	10	-0.2	69	104	10	1.8	46	100	10	0.5
069	86.2	110	10	-0.6	48	95	10	-0.2	52.3	105	10	1.2
070	#				#				#			
071	#				#				31	99	10	-1.1
072	132.90	99.70	13.32	1.6	#				42.60	101.00	6.66	0.1
073	63	100	10	-1.6	43	100	10	-0.6	47	100	10	0.6
074	102.6	85	10	0.2	#				38.2	90	4	-0.4
075	#				47.8	105.3	10	-0.2	#			

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 Endosulfan-Sulfate, Etrimfos and Fluopyram

laboratory number	analyte											
	Endosulfan-sulfate assigned value: 98.2 µg/kg				Etrimfos assigned value: 49.7 µg/kg				Fluopyram assigned value: 41.5 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
076	116.81	105.2	10	0.9	Less than 50	99.8	50		#			
077	62	80-120	10	-1.7	60	80-120	10	0.9	30	80-120	10	-1.3
078	115.50	93.97	10	0.8	57.85	96.97	10	0.7	48.10	91.39	10	0.7
079	116.48	70-120	10	0.8	#				44.84	70-120	10	0.4
080	176	> 90	50	**3.6**	#				#			
081	120	90	10	1.0	#				#			
082	72.0	88.0	10	-1.2	52.9	92.9	10	0.3	35.1	82.4	10	-0.7
083	93.5	110	5	-0.2	53.9	109	5	0.4	45.9	107	5	0.5
084	97.46	97	10	0.0	55.80	101	10	0.6	#			
085	102	103	10	0.2	43	101	10	-0.6	42	102	10	0.1
086	125		10	1.2	62.8		10	1.2	#			
087	100.6	100	10	0.1	43.9	100	10	-0.5	50.7	100	10	1.0
088	128.00	90	10	1.4	#				#			
089	#				#				#			
090	130		10	1.5	0		Varied	**-4.5**	0		Varied	**-4.5**

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 Endosulfan-Sulfate, Etrimfos and Fluopyram

laboratory number	analyte											
	Endosulfan-sulfate assigned value: 98.2 µg/kg				Etrimfos assigned value: 49.7 µg/kg				Fluopyram assigned value: 41.5 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
091	122.6	98	10	1.1	42.5	96	37.52	-0.7	#			
092	#				#				#			
093	#				58		10	0.8	#			
094	126.0	102.3	5	1.3	46.0	99.0	5	-0.3	38.9	95.1	5	-0.3
095	#				#				#			
096	128	-	50	1.4	#				47	105	10	0.6
097	#				#				#			
098	97.01		10	-0.1	33.13		10	-1.5	47.03		10	0.6
099	75.72	100.99	10	-1.0	32.06	96.56	10	-1.6	31.62	97.18	10	-1.1
100	#				#				43		10	0.2
101	#				#				37	104	5	-0.5
102	#				#				#			
103	100.03	100	10	0.1	0	100	10	**-4.5**	49.11	100	10	0.8
104	87.71	98.9	5	-0.5	#				#			
105	100	93.0	10	0.1	34	94.0	10	-1.4	36	102	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 (continued): Results and z-Scores for Endosulfan-Sulfate, Etrimfos and Fluopyram

laboratory number	analyte											
	Endosulfan-sulfate assigned value: 98.2 µg/kg				Etrimfos assigned value: 49.7 µg/kg				Fluopyram assigned value: 41.5 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
106	108	88	10	0.5	283.32	101	10	**21.4**	49.0	101	10	0.8
107	170		10	**3.3**	30		10	-1.8	83		10	**4.6**
108	112.3	82.0	37.6	0.7	52.2	107.9	12.6	0.2	#			
109	91	89	10	-0.3	37	100	10	-1.2	47	111	10	0.6
110	121.3	83	10	1.1	55.7	133	10	0.5	48	95	10	0.7
111	44.26	102.1	10.00	**-2.5**	#				#			
112	114.170	106.526	0.05	0.7	0		0.05	**-4.5**	0		0.05	**-4.5**
113	83	94	10	-0.7	0			**-4.5**	43	95	10	0.2
114	117	117	10	0.9	50	100	10	0.0	53	118	10	1.3
115	80	100	10	-0.8	36	100	10	-1.3	32	100	10	-1.0
116	110	93	40	0.5	#				48	89	5.0	0.7
117	69.29	140	17.5	-1.3	#				58.5	117	3	1.9
118	107.04	118	5	0.4	93.48	95	10	**4.0**	43.38	97	10	0.2
119	#				#				#			
120	136.89	96.74	10	1.8	#				#			

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 Endosulfan-Sulfate, Etrimfos and Fluopyram

laboratory number	analyte											
	Endosulfan-sulfate assigned value: 98.2 µg/kg				Etrimfos assigned value: 49.7 µg/kg				Fluopyram assigned value: 41.5 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
121	73	85	10	-1.2	#				51	95	10	1.0
122	81.96	70-130	10	-0.8	44.98	70-130	10	-0.4	41.98	70-130	10	0.1
123	75.5	110	10	-1.1	35	110	10	-1.3	32	100	10	-1.0
124	103.6		5	0.3	#				#			

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 Methoxyfenozide, Oxadixyl and Pirimicarb

laboratory number	analyte											
	Methoxyfenozide assigned value: 50.4 µg/kg				Oxadixyl assigned value: 60.6 µg/kg				Pirimicarb assigned value: 32.1 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
001	38	113.4	10	-1.1	57	81.9	10	-0.3	26	105.2	10	-0.9
002	50.6	99	10	0.0	65.4	102	10	0.4	33.7	115	10	0.2
003	31.44		10	-1.7	52.01		10	-0.6	25.91		10	-0.9
004	52.5		0.01 mg/kg	0.2	48.9		0.01 mg/kg	-0.9	33.0		0.01 mg/kg	0.1
005	#				#				#			
006	67.9	99.4	10.0	1.6	65.5	99.2	10.0	0.4	35.0	98.8	10.0	0.4
007	40.80	104.00	2.00	-0.9	#				#			
008	52	101	3.3	0.1	#				#			
009	76	100	10	**2.3**	63	85	10	0.2	32	86	10	0.0
010	49.34	70-120	0.005 mg/kg	-0.1	59.58	70-120	0.005 mg/kg	-0.1	32.58	70-120	0.005 mg/kg	0.1
011	49.02	111	5	-0.1	75.51	115	5	1.1	35.49	106	5	0.5
012	42.54	106.11	10	-0.7	54.25	94	10	-0.5	30.35	103.84	10	-0.2
013	53.08		10	0.2	#				36.46		10	0.6
014	42.8	97	2.50	-0.7	64.2	95	2.50	0.3	31.9	101	1.25	0.0
015	30	95	10	-1.8	52	100	10	-0.6	31	105	10	-0.2

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 Methoxyfenozide, Oxadixyl and Pirimicarb

laboratory number	analyte											
	Methoxyfenozide assigned value: 50.4 µg/kg				Oxadixyl assigned value: 60.6 µg/kg				Pirimicarb assigned value: 32.1 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
016	51	70-120	10	0.1	#				37	70-120	10	0.7
017	34.9	103	10	-1.4	68	87	10	0.6	33	68	10	0.1
018	47.7	114	10	-0.2	63.3	114	10	0.2	30.9	108	10	-0.2
019	71	86.2	10	1.9	#				35	86.1	10	0.4
020	45	93	10	-0.5	64	92	10	0.3	31	89	5	-0.2
021	0	94~99	10	**-4.5**	59.4	94.7	10	-0.1	33.2	98.5	10	0.2
022	36.5	99	0.010	-1.3	56.3	75	0.010	-0.3	29.0	97	0.010	-0.4
023	67	100	10	1.5	63	80	10	0.2	32	92	10	0.0
024	#				62	93.7	5	0.1	33	99.0	5	0.1
025	42.1	96	5.0	-0.7	#				#			
026	#				62	90	10	0.1	#			
027	#				60	100	10	0.0	#			
028	#				59	90	10	-0.1	#			
029	42.2		10	-0.7	59.2		10	-0.1	36.2		10	0.6
030	70	106	10	1.8	66	101	10	0.4	35	106	10	0.4

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 Methoxyfenozide, Oxadixyl and Pirimicarb

laboratory number	analyte											
	Methoxyfenozide assigned value: 50.4 µg/kg				Oxadixyl assigned value: 60.6 µg/kg				Pirimicarb assigned value: 32.1 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
031	41.85	98.70	0.01 mg/kg	-0.8	57.30	102.50	0.01 mg/kg	-0.3	29.75	110.90	0.01 mg/kg	-0.3
032	0			**-4.5**	62.77	99.77	3.3	0.2	32.12	100.35	2.8	0.0
033	#				#				#			
034	54.7	106.6	10	0.4	56.8	93.0	10	-0.3	41.6	127.1	10	1.3
035	#				#				#			
036	#				61.3	103.9	1.1	0.0	32.8	96.7	1.1	0.1
037	40	82	10	-0.9	#				32	73	5	0.0
038	#				52.57	93.74	10	-0.6	#			
039	#				61.5	100.3	10	0.1	#			
040	#				#				31.62	85.54	25	-0.1
041	#				#				#			
042	#				25.36	84	10	**-2.6**	#			
043	32	80	10	-1.7	59	80	10	-0.1	34	80	10	0.3
044	84	89	10	**3.0**	85	87	10	1.8	39	113	10	1.0
045	34.6	88.7	10	-1.4	64.3	100.3	10	0.3	30.3	84.8	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 Methoxyfenozide, Oxadixyl and Pirimicarb

laboratory number	analyte											
	Methoxyfenozide assigned value: 50.4 µg/kg				Oxadixyl assigned value: 60.6 µg/kg				Pirimicarb assigned value: 32.1 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
046	38	81	10	-1.1	45	97	10	-1.2	27	91	10	-0.7
047	25		10	**-2.3**	72		30	0.9	29		10	-0.4
048	#				#				<LOQ	104	20.00	
049	31.00	91.82	5	-1.7	46.06	94.35	20	-1.1	28.85	86.88	5	-0.5
050	#				60.4	100.6	5	0.0	30.5	100.3	5	-0.2
051	33.5	95	10	-1.5	18.7	80	10	**-3.1**	10.1	80	10	**-3.1**
052	53.51	86.03	5	0.3	61.97	91.54	5	0.1	29.82	89.19	5	-0.3
053	#				#				35	103	10	0.4
054	#				#				#			
055	#				#				#			
056	44.60	70-120	10	-0.5	63.645	70-120	10	0.2	32.965	70-120	10	0.1
057	100.11	113.8	3.9	**4.5**	111.5	101.4	3.9	**3.8**	41.2	95.7	3.9	1.3
058	46.5	78	10	-0.3	0			**-4.5**	0			**-4.5**
059	64	100	10	1.2	69	100	10	0.6	35	100	10	0.4
060	#				54	100	10	-0.5	29	100	10	-0.4

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 Methoxyfenozide, Oxadixyl and Pirimicarb

laboratory number	analyte											
	Methoxyfenozide assigned value: 50.4 µg/kg				Oxadixyl assigned value: 60.6 µg/kg				Pirimicarb assigned value: 32.1 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
061	49.8		0.93	-0.1	61.2		2.50	0.0	36.0		0.04	0.6
062	#				#				#			
063	0			**-4.5**	32.1	96.4	10	**-2.1**	16.4	95.8	10	**-2.2**
064	56	70-120	10	0.5	72	70-120	10	0.9	33	70-120	10	0.1
065	36	92	10	-1.3	121	100	10	**4.5**	29	101	10	-0.4
066	51.8	85	10	0.1	#				34.2	95	10	0.3
067	50.78	107	10	0.0	47.80	89	10	-1.0	27.81	90	10	-0.6
068	47	94	10	-0.3	63	95	10	0.2	30	88	10	-0.3
069	54.1	101	10	0.3	56.7	98	10	-0.3	35.8	99	10	0.5
070	58.2	90.0	5.0	0.7	63.5	90.0	5.0	0.2	32.5	90.0	5.0	0.1
071	#				#				25.47	92	10	-0.9
072	52.10	106.00	9.99	0.2	#				#			
073	56	82	10	0.5	75	100	10	1.1	47	100	10	**2.1**
074	34.8	69	4	-1.4	#				#			
075	54.1	103.8	10	0.3	67.3	107.8	10	0.5	33.7	103.6	10	0.2

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 Methoxyfenozide, Oxadixyl and Pirimicarb

laboratory number	analyte											
	Methoxyfenozide assigned value: 50.4 µg/kg				Oxadixyl assigned value: 60.6 µg/kg				Pirimicarb assigned value: 32.1 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
076	#				#				#			
077	52	80-120	10	0.1	43	80-120	10	-1.3	21	80-120	10	-1.6
078	53.80	97.68	10	0.3	62.41	93.48	10	0.1	31.27	95.34	5	-0.1
079	47.08	70-120	10	-0.3	60.80	70-120	10	0.0	33.05	70-120	10	0.1
080	< 50	> 90	50		#				49	> 90	50	**2.4**
081	55	90	10	0.4	66	90	10	0.4	33	90	10	0.1
082	37.3	84.9	10	-1.2	49.9	94.4	10	-0.8	24.8	78.7	10	-1.0
083	55.7	100	5	0.5	68.6	109	5	0.6	37.0	99	5	0.7
084	52.55	80	10	0.2	63.22	109	10	0.2	29.41	96	10	-0.4
085	56	102	10	0.5	63	97	10	0.2	33	96	10	0.1
086	55.0		10	0.4	67.0		10	0.5	31.8		10	0.0
087	58.0	100	10	0.7	49.5	100	10	-0.8	26.1	100	10	-0.8
088	55.00	94	10	0.4	65.00	92	10	0.3	33.00	99	10	0.1
089	65.12	90.0	5.0	1.3	70.01	90.0	5.0	0.7	38.02	90.0	5.0	0.8
090	24		20	**-2.4**	0		Varied	**-4.5**	16		10	**-2.3**

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 Methoxyfenozide, Oxadixyl and Pirimicarb

laboratory number	analyte											
	Methoxyfenozide assigned value: 50.4 µg/kg				Oxadixyl assigned value: 60.6 µg/kg				Pirimicarb assigned value: 32.1 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
091	50.0	94	2	0.0	58.7	94	10	-0.1	30.9	95	2	-0.2
092	50	> 120	10	0.0	#				33	80-120	10	0.1
093	52		10	0.1	64		10	0.3	32		10	0.0
094	51.4	97.2	5	0.1	57.9	103.8	5	-0.2	32.6	105.0	5	0.1
095	#				#				#			
096	#				#				45	97	10	1.8
097	#				64.4	112	10	0.3	#			
098	50.36		10	0.0	63.08		10	0.2	35.51		10	0.5
099	36.80	102.72	10	-1.2	52.41	108.25	10	-0.6	27.83	89.50	10	-0.6
100	44	130	10	-0.6	42	110	10	-1.4	19	101	10	-1.9
101	42	107	5	-0.8	#				33	106	5	0.1
102	#				#				58.6	98	10	**3.8**
103	59.23	100	10	0.8	64.77	100	10	0.3	34.88	100	10	0.4
104	#				#				#			
105	43	94.8	10	-0.7	55	98.7	10	-0.4	29	99.2	10	-0.4

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 Methoxyfenozide, Oxadixyl and Pirimicarb

laboratory number	analyte											
	Methoxyfenozide assigned value: 50.4 µg/kg				Oxadixyl assigned value: 60.6 µg/kg				Pirimicarb assigned value: 32.1 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
106	58	108	10	0.7	64	95	10	0.3	31	97	10	-0.2
107	45		10	-0.5	98		10	**2.8**	53		10	**3.0**
108	52.3	102.7	10.0	0.2	65.2	114.6	12.6	0.3	33.2	108.0	25.0	0.2
109	56	111	10	0.5	#				31	102	10	-0.2
110	71.9	100	10	1.9	66.5	95	10	0.4	34.0	91	10	0.3
111	#				#				#			
112	0		0.05	**-4.5**	0		0.05	**-4.5**	0		0.05	**-4.5**
113	37	97	10	-1.2	39	93	10	-1.6	29	94	10	-0.4
114	60	98	10	0.9	73	120	10	0.9	34	102	10	0.3
115	59	100	10	0.8	53	100	10	-0.6	29	100	10	-0.4
116	51	87	20	0.1	#				31	89	5.0	-0.2
117	58	117	3	0.7	65.08	113	10	0.3	34.38	104	3	0.3
118	37.14	103	10	-1.2	61.10	94	10	0.0	30.94	114	10	-0.2
119	66.930	100	6	1.5	63.891	118	6	0.2	32.184	105	6	0.0
120	72.12	83.14	10	2.0	66.25		10	0.4	#			

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 Methoxyfenozide, Oxadixyl and Pirimicarb

laboratory number	analyte											
	Methoxyfenozide assigned value: 50.4 µg/kg				Oxadixyl assigned value: 60.6 µg/kg				Pirimicarb assigned value: 32.1 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
121	38	90	10	-1.1	68	90	10	0.6	36	80	10	0.6
122	50.97	70-130	10	0.1	58.97	70-130	10	-0.1	38.98	70-130	10	1.0
123	55	100	10	0.4	46	90	10	-1.1	27	100	10	-0.7
124	#				#				#			

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 Pyridalyl and Teflubenzuron

laboratory number	analyte							
	Pyridalyl assigned value: 44.0 µg/kg				Teflubenzuron assigned value: 80.8 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
001	#				83	114.5	10	0.1
002	#				85.5	96	10	0.3
003	#				42.09		10	**-2.2**
004	0		0.01 mg/kg	**-4.5**	93.0		0.01 mg/kg	0.7
005	40.5	98.0	5	-0.4	#			
006	0	98	10.0	**-4.5**	90.7	98.3	10.0	0.6
007	22.30	55.00	5.00	**-2.2**	88.80	114.00	10.00	0.5
008	#				#			
009	#				20	100	10	**-3.4**
010	46.94	70-120	0.005 mg/kg	0.3	59.04	70-120	0.005 mg/kg	-1.2
011	39.49	85	5	-0.5	73.00	104	5	-0.4
012	#				191.78	88.47	10	**6.2**
013	#				81.79		10	0.1
014	57.7	90	2.50	1.4	83.5	120	5.00	0.2
015	23	106	10	**-2.2**	38	70	10	**-2.4**

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 Pyridalyl and Teflubenzuron

laboratory number	analyte							
	Pyridalyl assigned value: 44.0 µg/kg				Teflubenzuron assigned value: 80.8 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
016	#				#			
017	43	104	10	-0.1	74.4	130	10	-0.4
018	44.1	122	10	0.0	64.8	110	10	-0.9
019	#				82	82.6	10	0.1
020	#				#			
021	47.7	99.1	10	0.4	0	94~99	10	**-4.5**
022	#				#			
023	49	93	10	0.5	79	91	10	-0.1
024	#				#			
025	46.4	96	5.2	0.2	#			
026	42	90	10	-0.2	#			
027	43	100	10	-0.1	#			
028	42	90	10	-0.2	#			
029	#				89.2		10	0.5
030	56	107	10	1.2	95	111	10	0.8

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 Pyridalyl and Teflubenzuron

laboratory number	analyte							
	Pyridalyl assigned value: 44.0 µg/kg				Teflubenzuron assigned value: 80.8 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
031	#				138.08	113.60	0.01 mg/kg	**3.2**
032	0			**-4.5**	0			**-4.5**
033	#				#			
034	#				89.9	140.4	10	0.5
035	#				#			
036	#				#			
037	#				57	76	5	-1.3
038	#				#			
039	41.2	92.7	10	-0.3	#			
040	#				#			
041	#				#			
042	#				#			
043	#				164	80	10	**4.7**
044	0	100ppb/10ppb	10	**-4.5**	0	100ppb/10ppb	10	**-4.5**
045	#				55.2	87.6	10	-1.4

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 Pyridalyl and Teflubenzuron

laboratory number	analyte							
	Pyridalyl assigned value: 44.0 µg/kg				Teflubenzuron assigned value: 80.8 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
046	0		10	**-4.5**	39	101	10	**-2.4**
047	67		30	**2.4**	34		10	**-2.6**
048	#				#			
049	#				44.82	77.74	20	-2.0
050	38.9	104.9	2	-0.5	#			
051	53.3	110	10	1.0	11.5	80	10	**-3.9**
052	45.05	94.71	5	0.1	#			
053	#				#			
054	#				#			
055	#				#			
056	#				#			
057	150.8	82.6	3.9	**11.0**	0	80-119.5	0.97-3.9	**-4.5**
058	41.6	89	10	-0.2	100.3	101	10	1.1
059	50	100	10	0.6	98	100	10	1.0
060	34	100	10	-1.0	#			

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 Pyridalyl and Teflubenzuron

laboratory number	analyte							
	Pyridalyl assigned value: 44.0 µg/kg				Teflubenzuron assigned value: 80.8 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
061	#				77.0		20.00	-0.2
062	#				#			
063	0			**-4.5**	0			**-4.5**
064	30	70-120	10	-1.4	51	70-120	10	-1.7
065	#				153	100	10	**4.1**
066	#				#			
067	#				74.67	103	10	-0.3
068	51	100	10	0.7	85	95	10	0.2
069	37.6	92	10	-0.7	101	81	10	1.1
070	#				92.2	90.0	5.0	0.6
071	#				#			
072	37.30	93.00	16.65	-0.7	#			
073	43	100	10	-0.1	125	98	10	**2.5**
074	#				#			
075	41.7	100.3	10	-0.2	70.4	99.5	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 (continued): Results and z-Scores for Pyridalyl and Teflubenzuron

laboratory number	analyte							
	Pyridalyl assigned value: 44.0 µg/kg				Teflubenzuron assigned value: 80.8 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
076	#				#			
077	39	80-120	10	-0.5	84	80-120	10	0.2
078	48.23	93.89	10	0.4	80.25	91.36	10	0.0
079	#				#			
080	#				141	> 90	50	**3.4**
081	#				79	90	10	-0.1
082	#				52.9	70.8	10	-1.6
083	#				83.9	109	5	0.2
084	#				#			
085	60	100	10	1.7	84	98	10	0.2
086	#				75.8		10	-0.3
087	#				#			
088	#				77.00	96	10	-0.2
089	#				118.0	90.0	5.0	**2.1**
090	0		Varied	**-4.5**	55		50	-1.5

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 Pyridalyl and Teflubenzuron

laboratory number	analyte							
	Pyridalyl assigned value: 44.0 µg/kg				Teflubenzuron assigned value: 80.8 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
091	#				75.2	95	20	-0.3
092	#				0	80-120	0.010 mg/kg	**-4.5**
093	34		10	-1.0	#			
094	34.1	104.2	5	-1.0	75.8	98.8	5	-0.3
095	#				#			
096	#				#			
097	#				#			
098	0		10	**-4.5**	89.85		10	0.5
099	#				75.77	91.41	10	-0.3
100	#				61	105	10	-1.1
101	#				82	103	10	0.1
102	#				#			
103	#				#			
104	#				#			
105	46	90.5	10	0.2	74	112	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 Pyridalyl and Teflubenzuron

laboratory number	analyte							
	Pyridalyl assigned value: 44.0 µg/kg				Teflubenzuron assigned value: 80.8 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
106	#				#			
107	77		10	**3.4**	#			
108	#				88.2	87.9	10.0	0.4
109	#				80	105	10	0.0
110	57.2	85	10	1.4	89.8	105	10	0.5
111	#				#			
112	0		0.05	**-4.5**	0		0.05	**-4.5**
113	46	92	10	0.2	55	96	10	-1.5
114	47	88	10	0.3	98	96	10	1.0
115	37	100	10	-0.7	63	100	10	-1.0
116	53	11	10	0.9	67	40	20	-0.8
117	#				#			
118	49.85	93	10	0.6	55.20	103	10	-1.4
119	#				82.823	80	6	0.1
120	#				#			

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 Pyridalyl and Teflubenzuron

laboratory number	analyte							
	Pyridalyl assigned value: 44.0 µg/kg				Teflubenzuron assigned value: 80.8 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
121	#				94	86	10	0.7
122	37.98	70-130	10	-0.6	49.98	70-130	10	-1.7
123	33	110	10	-1.1	45	90	10	-2.0
124	#				#			

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
044	Carbofuran	21	89	10 ppb
044	Methiocarb-sulfone	77	110	10 ppb
058	Pirimicarb (desmethyl)	42.5	80	10
068	DDT-pp	36	100	10
079	Phenthoate	114.27	70-120	10
097	Pirimicarb (desmethyl)	30.1	120	10

Table 6: Participants' Comments

laboratory number	comments
002	Spinosad detected in the blank sample in the same quantity as in test sample.
011	Not corrected for recovery.
034	There is about 2.3 Âµg/kg (< LoQ:10) Spinosad in the blank test material.
043	Please improve the list of pesticides to be searched (eg an excel sheet): searching for pesticides to be analyzed is impractical!!
047	Etrimfos was detected but not calculated and reported as not tested.
057	Choridazon and fluopyram were detected (no available standard for quantitation). Ethirimol, carbaryl, bupimirate, aldicarb were detected after sample was concentrated.
066	LC MS / MS & GC MS / MS
072	Cymoxanil was detected above our limit of detection (40 Âµg/kg), but below our limit of quanititation (133.20 Âµg/kg). Because of this we did not report a numerical value.
074	The limits of quantitation for each tested analyte ranged from 4 to 50 ppb.
090	All analytes used different internal method.
111	In-house method
116	Spike was in organic tomato matrix purchased locally.

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	
Chloridazon	42	107	4	Horwitz [9]	23.6
Cymoxanil	70	92.2	1.6	Horwitz [9]	20.3
Dimethomorph	80	44.4	0.8	Horwitz [9]	9.78
Endosulfan-sulfate	73	98.2	3.1	Horwitz [9]	21.6
Etrimfos	49	49.7	2.0	Horwitz [9]	10.9
Fluopyram	69	41.5	0.8	Horwitz [9]	9.12
Methoxyfenozide	78	50.4	1.4	Horwitz [9]	11.1
Oxadixyl	78	60.6	0.9	Horwitz [9]	13.3
Pirimicarb	84	32.1	0.4	Horwitz [9]	7.06
Pyridalyl	40	44.0	1.2	Horwitz [9]	9.68
Teflubenzuron	57	80.8	2.6	Horwitz [9]	17.8

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

analyte	number of scores where $\|z\| \leq 2$	total number of scores	% $\|z\| \leq 2$
Chloridazon	46	55	81
Cymoxanil	80	90	89
Dimethomorph	84	96	88
Endosulfan-sulfate	72	89	81
Etrimfos	53	67	79
Fluopyram	76	83	92
Methoxyfenozide	85	94	90
Oxadixyl	82	91	90
Pirimicarb	89	98	91
Pyridalyl	40	54	74
Teflubenzuron	56	76	74

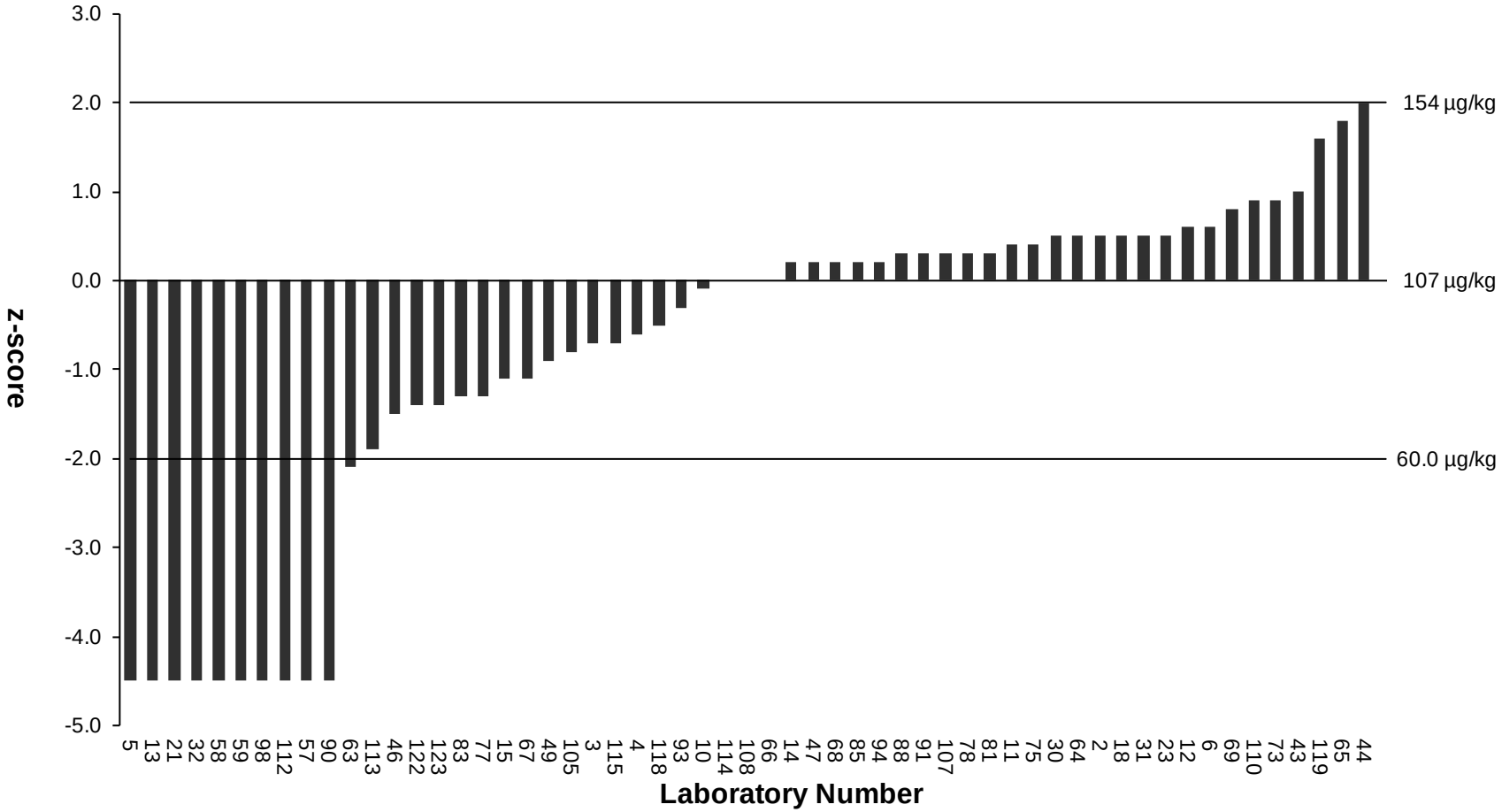

Figure 1: z-Scores for Chloridazon

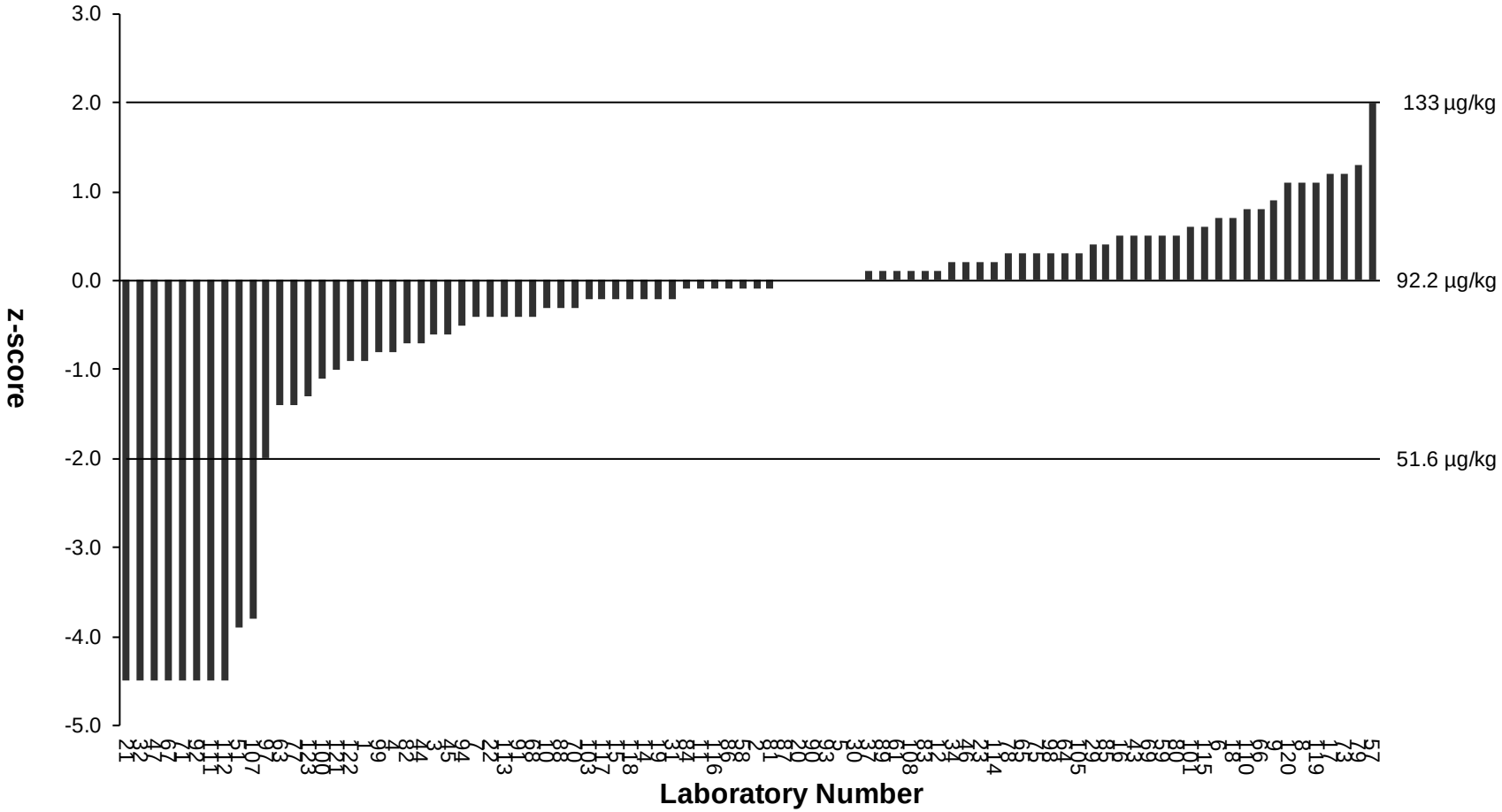

Figure 2: z-Scores for Cymoxanil

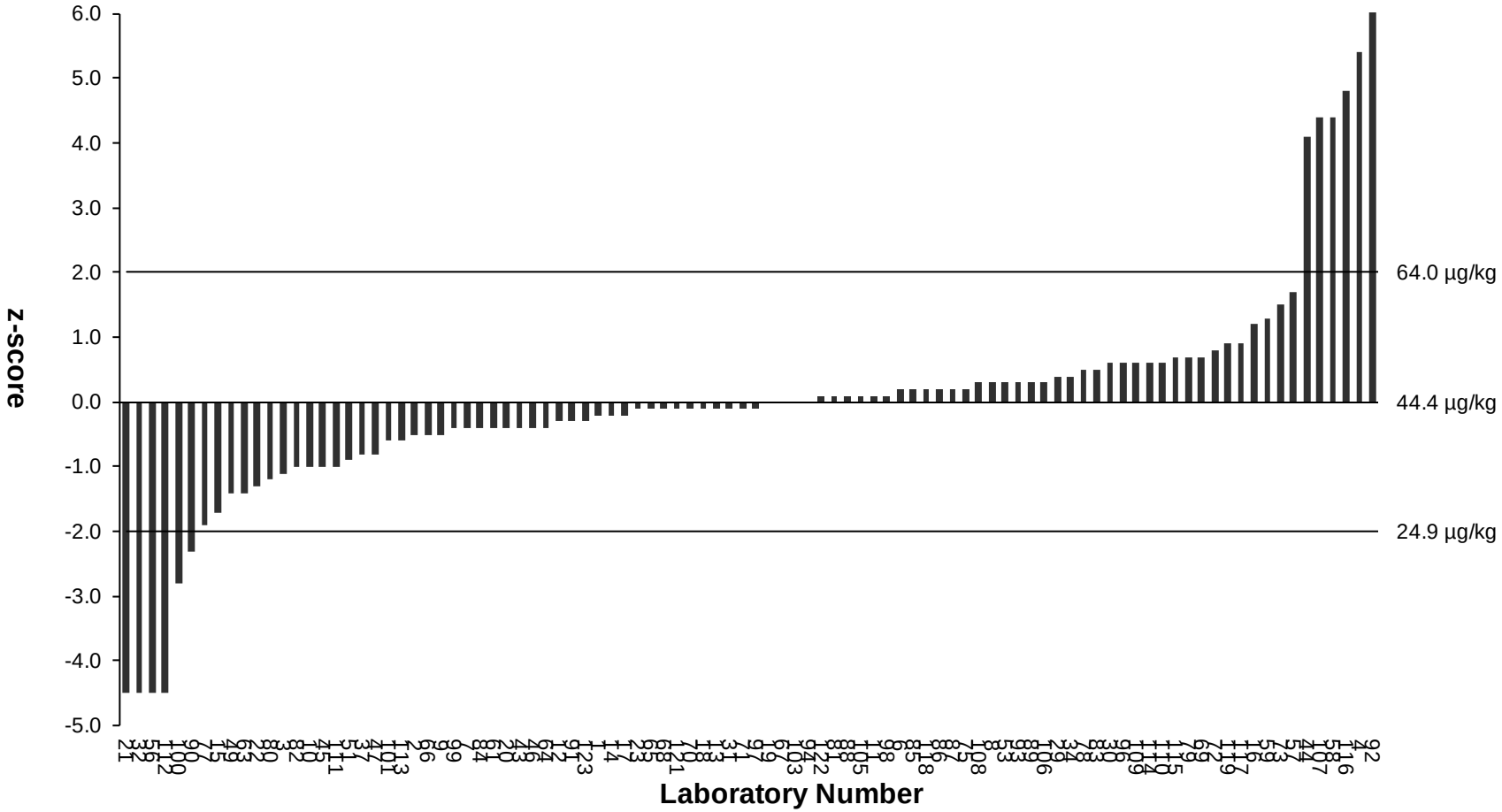

Figure 3: z-Scores for Dimethomorph

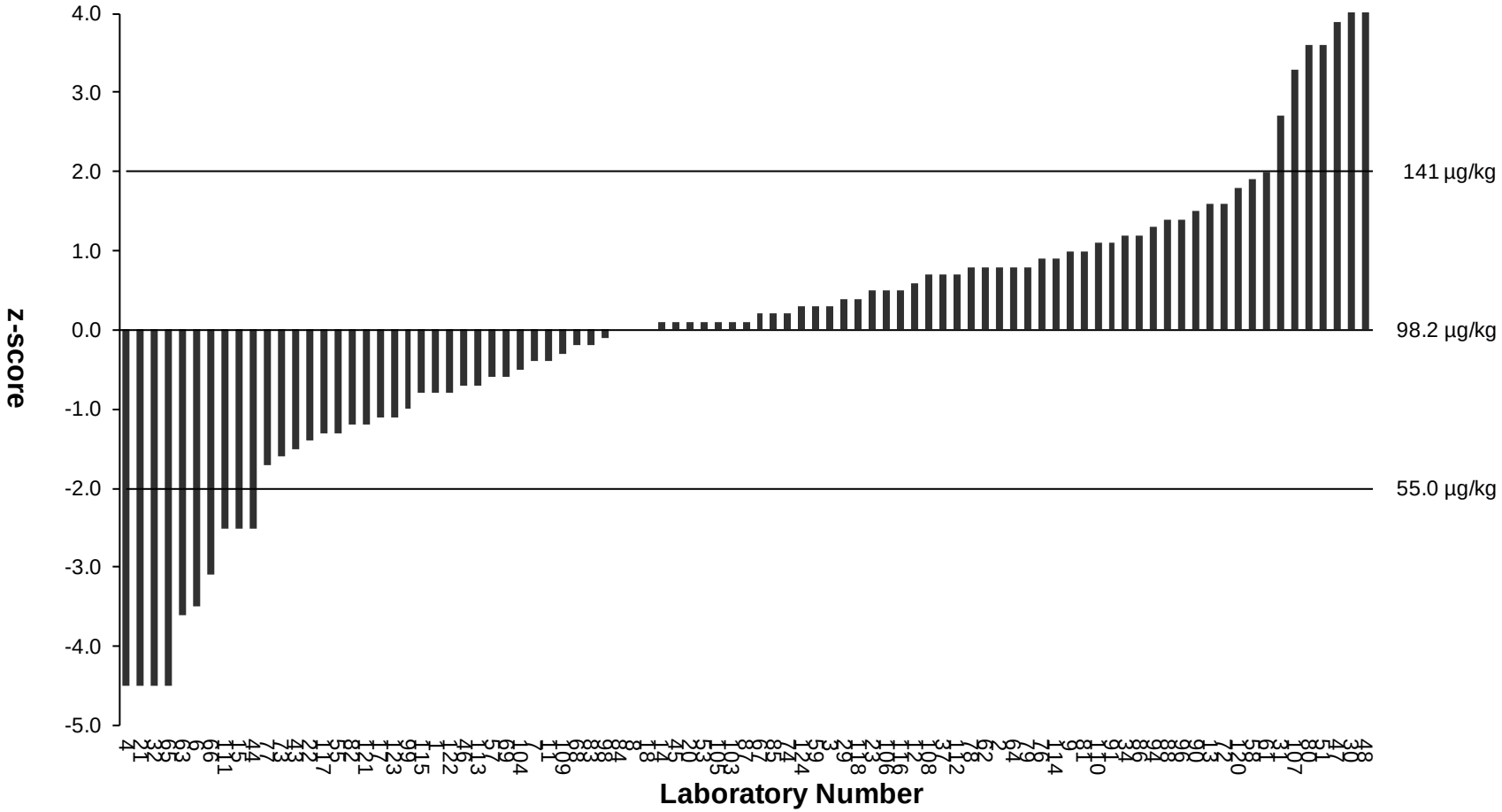

Figure 4: z-Scores for Endosulfan-Sulfate

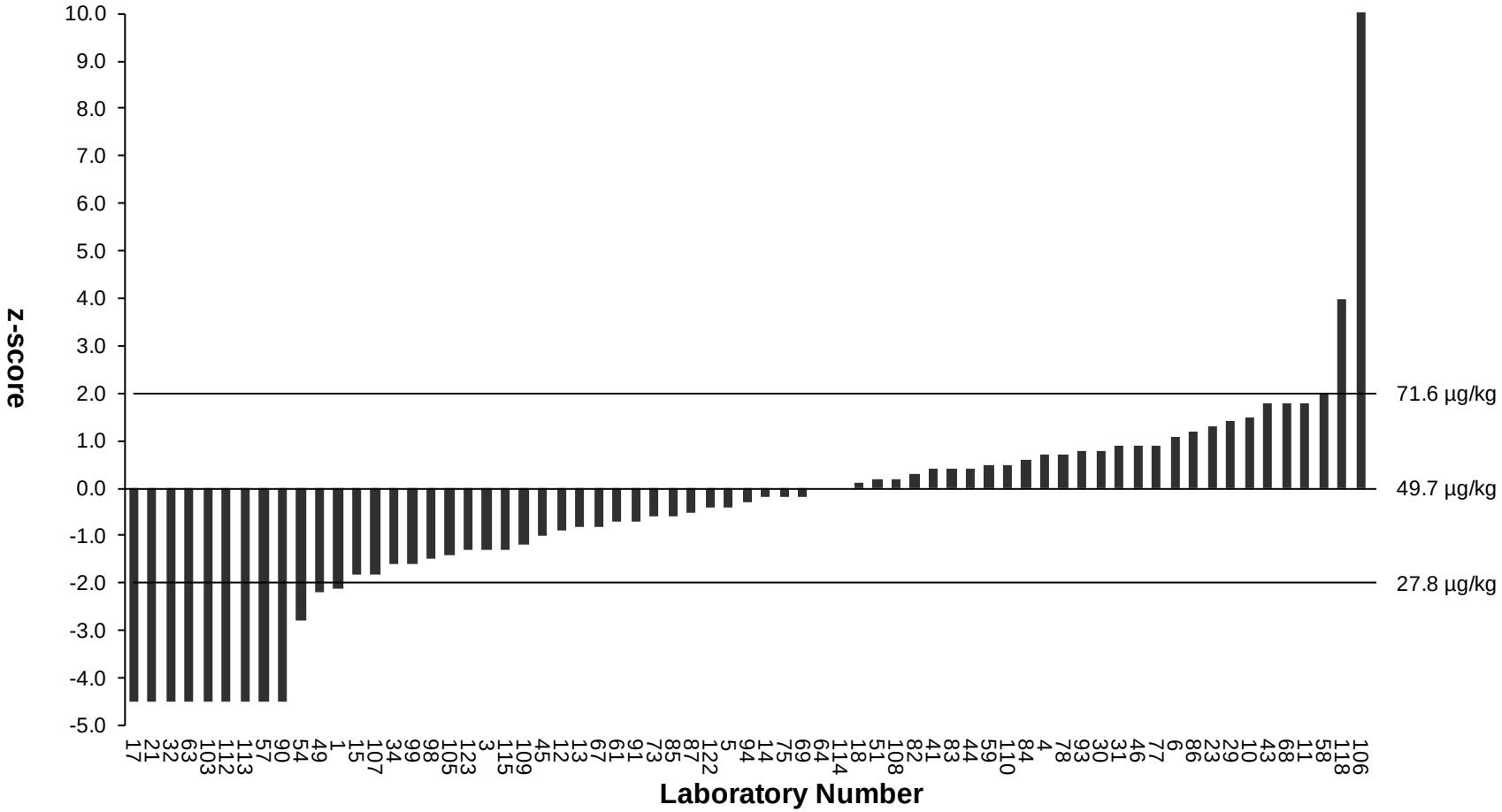

Figure 5: z-Scores for Etrimfos

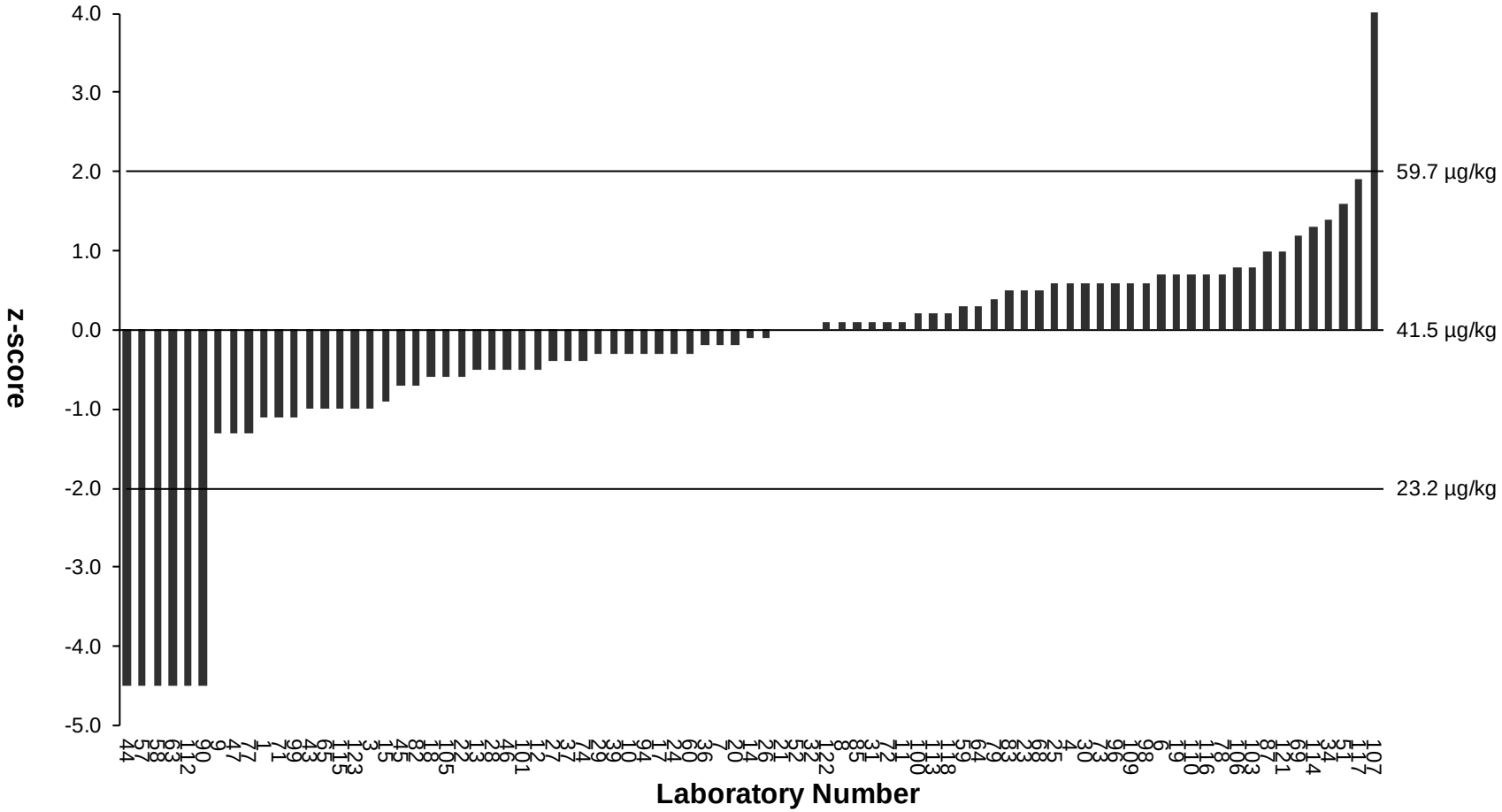

Figure 6: z-Scores for Fluopyram

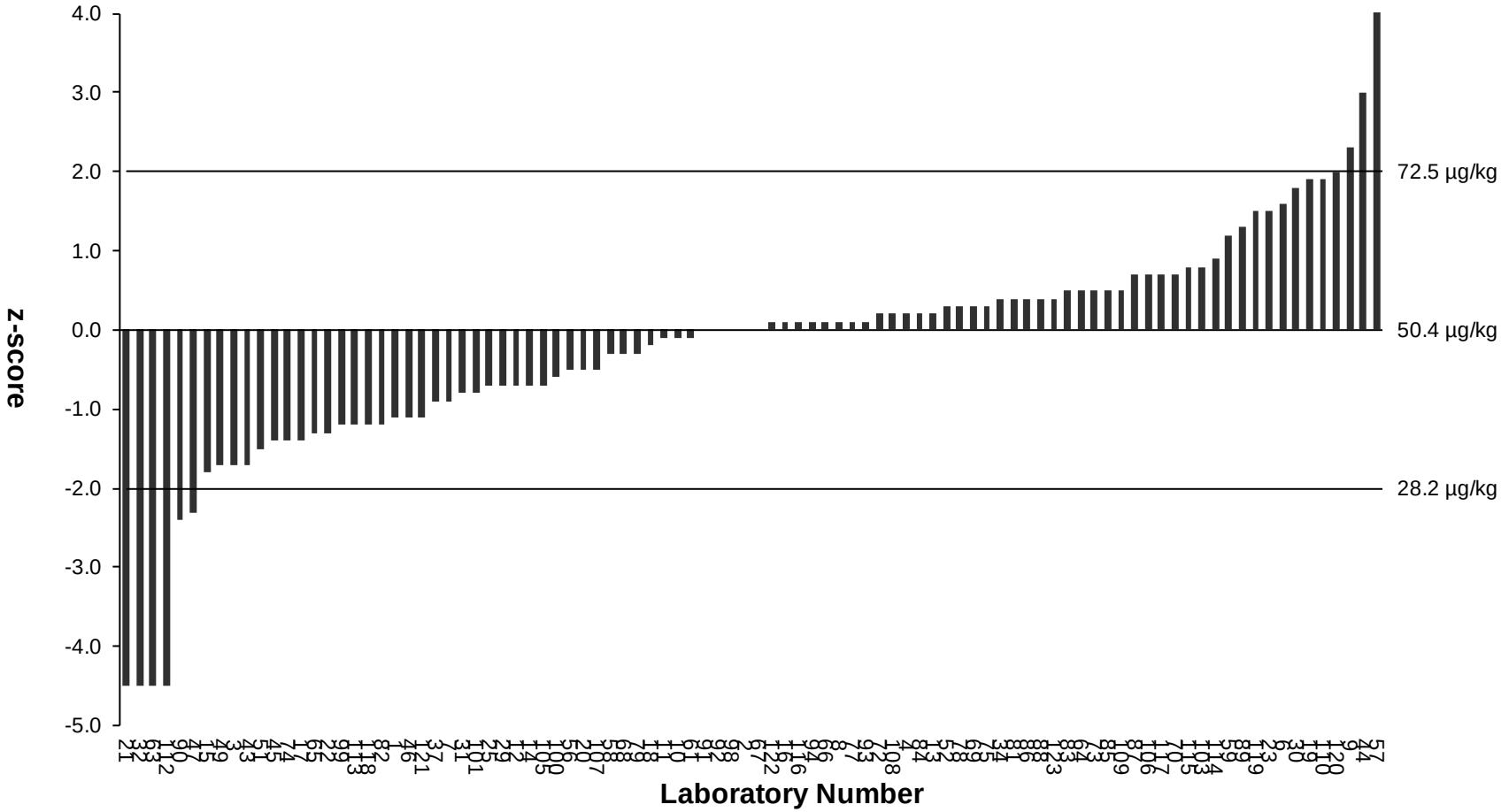

Figure 7: z-Scores for Methoxyfenozide

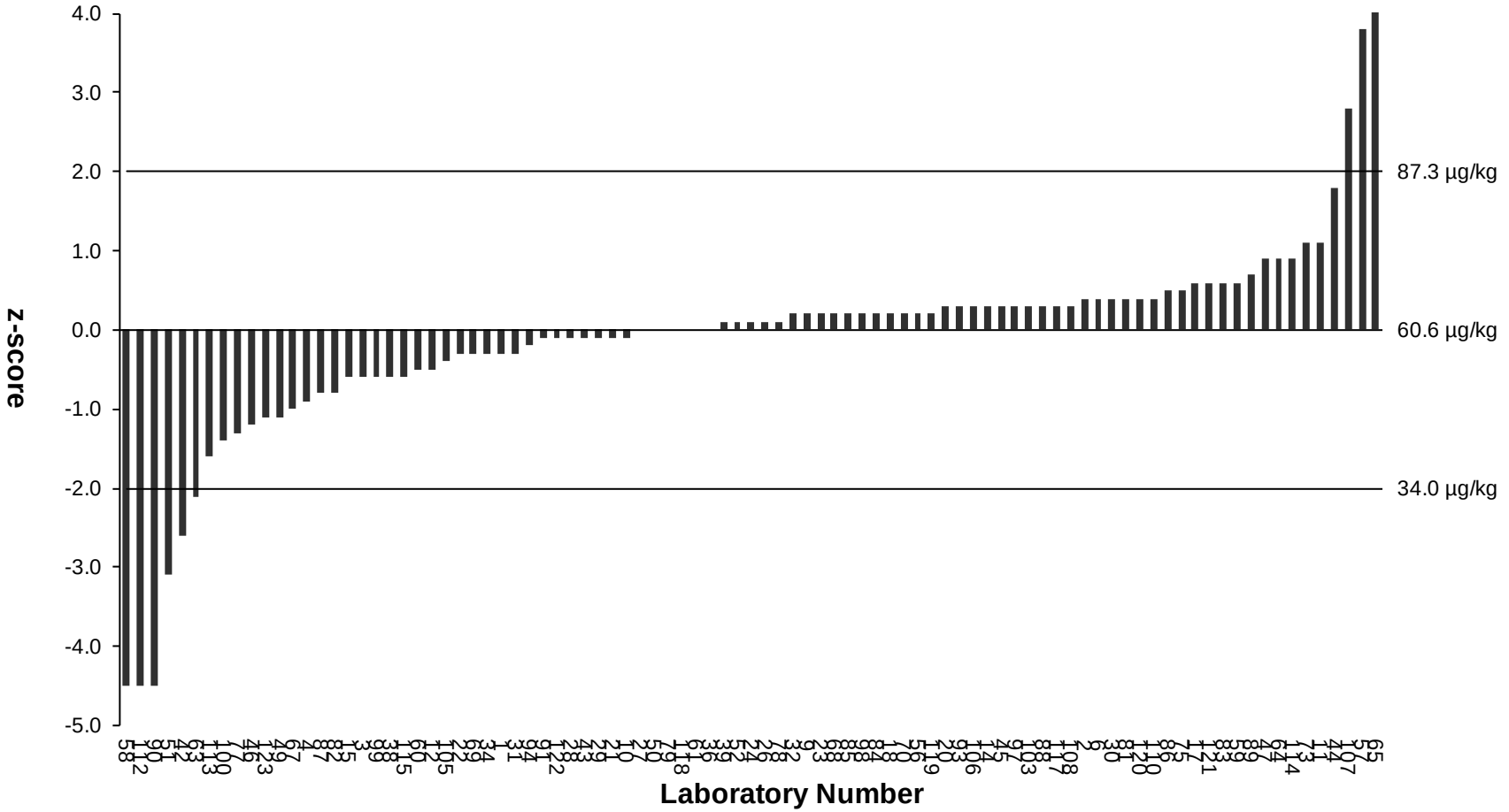

Figure 8: z-Scores for Oxadixyl

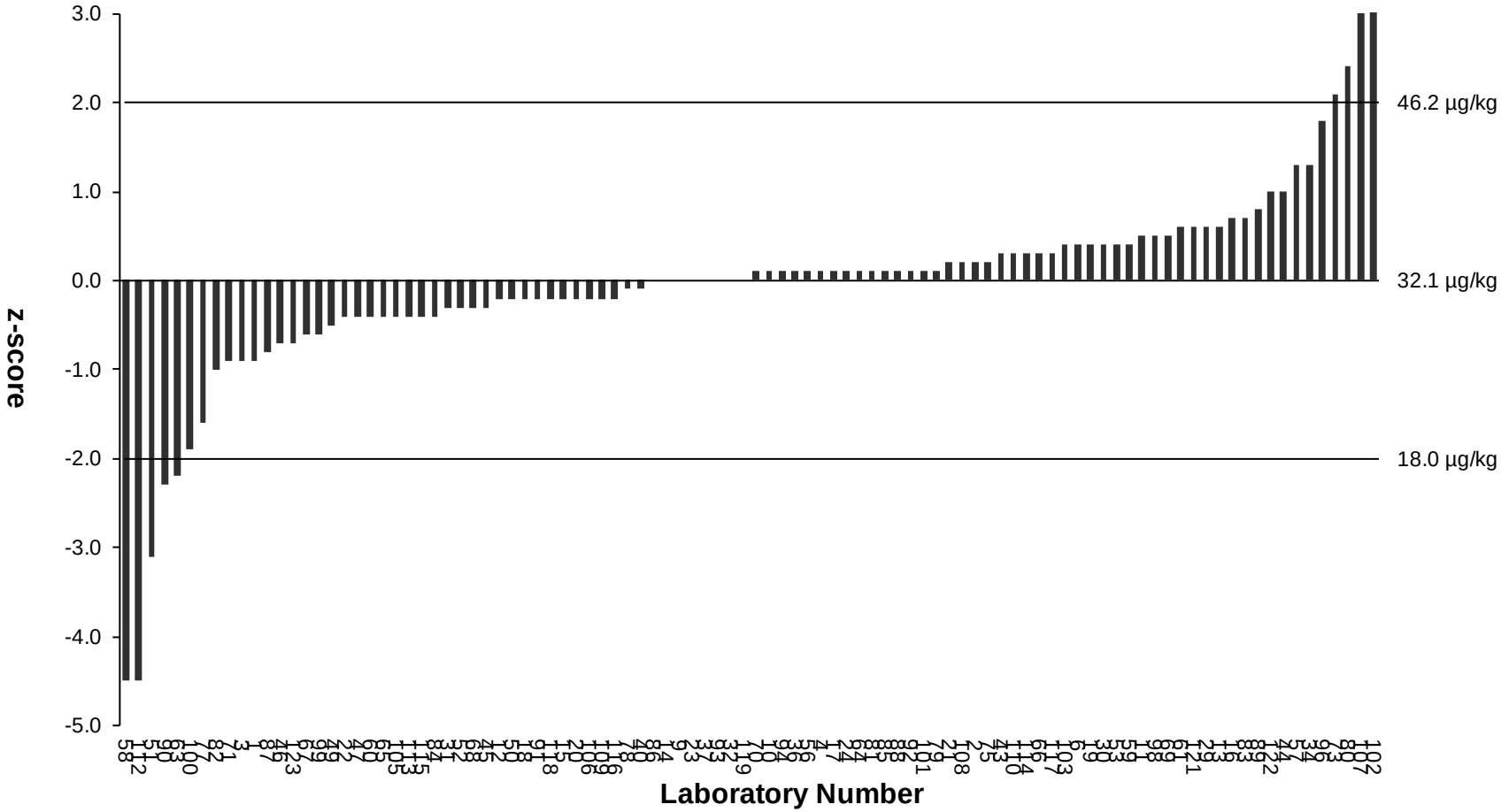

Figure 9: z-Scores for Pirimicarb

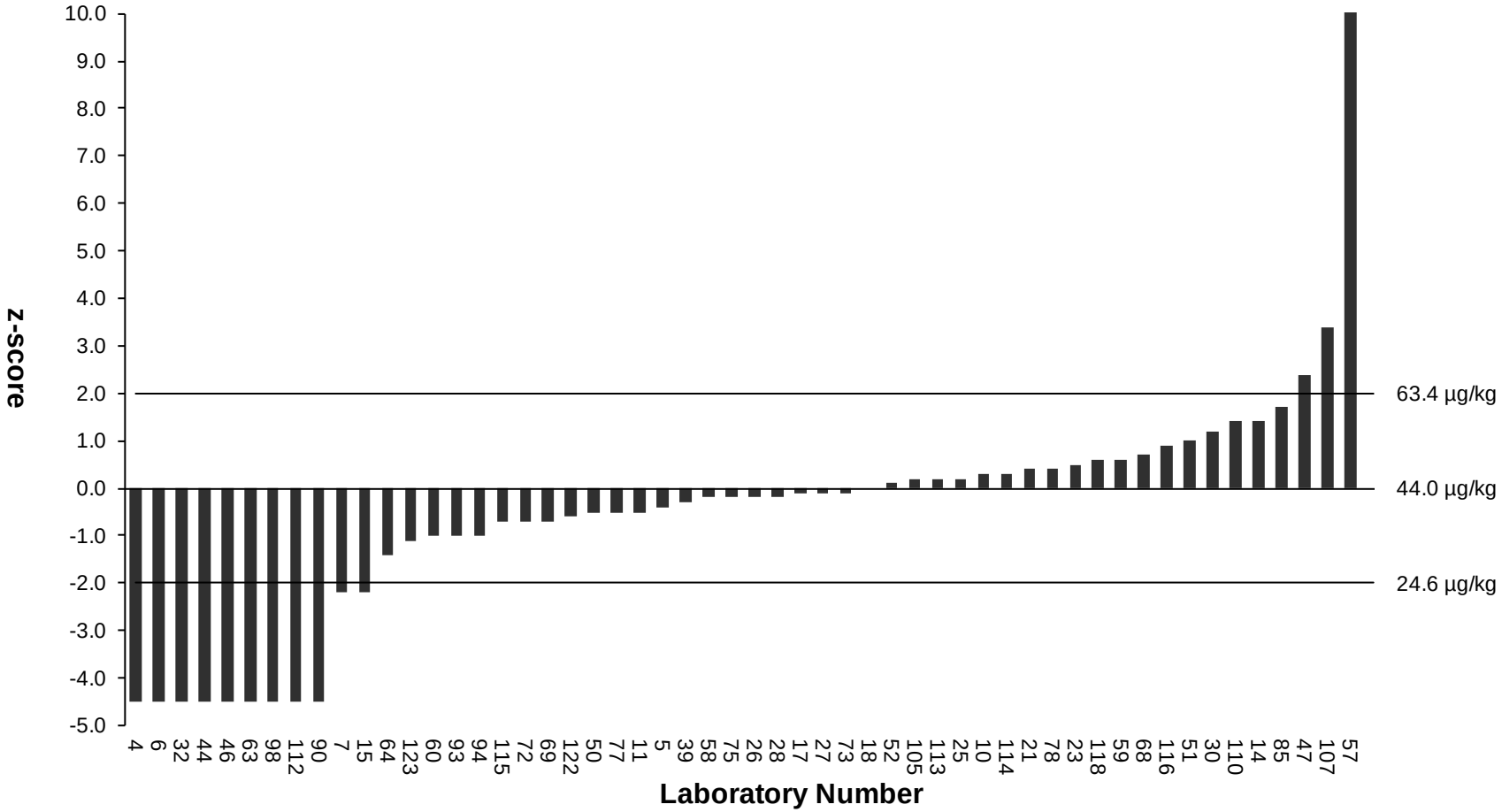

Figure 10: z-Scores for Pyridalyl

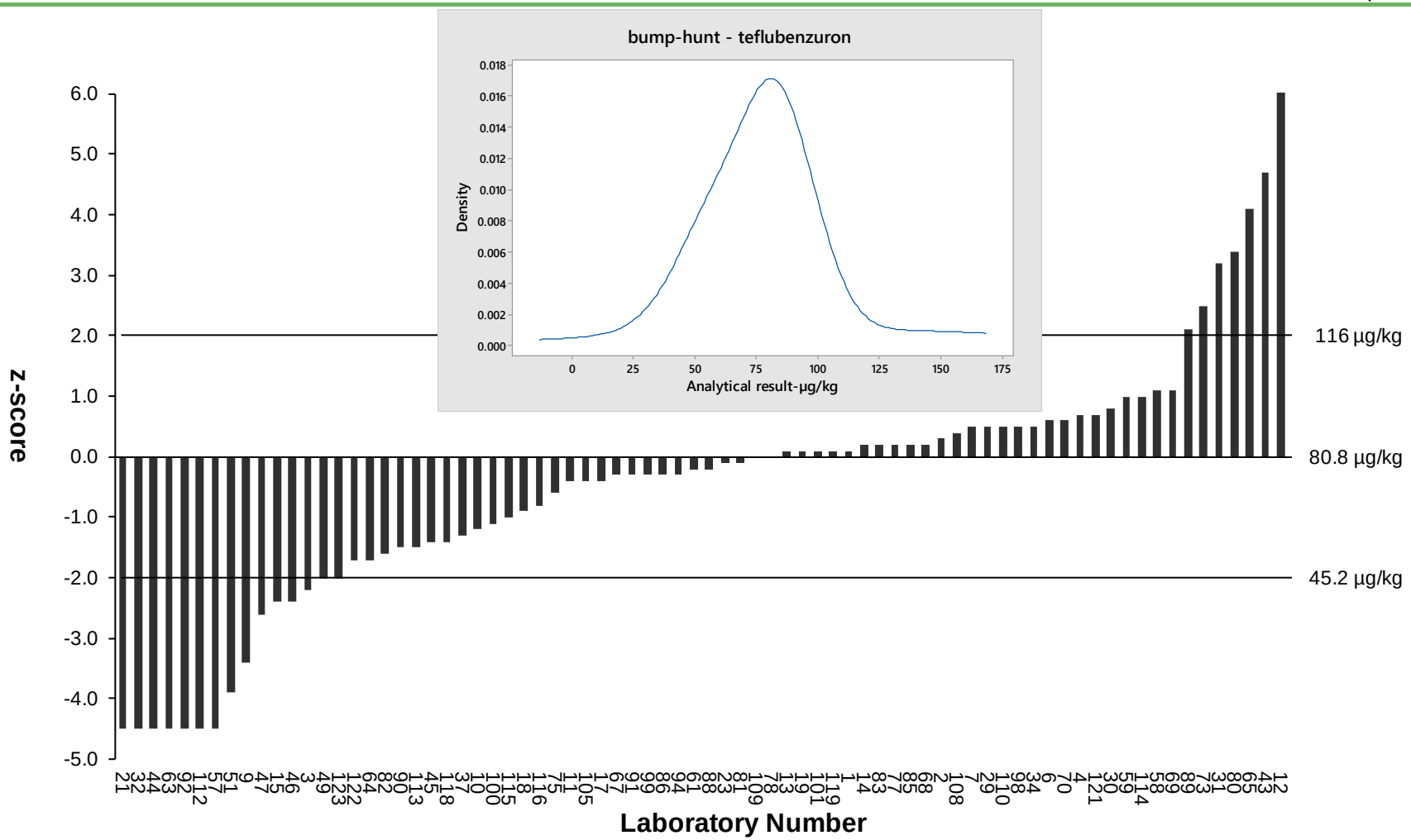

Figure 11: z-Scores for Teflubenzuron

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	001 003 005 013 016 031 042 053 067 074 080 092 096 097 104
yes	002 006 007 008 009 011 012 014 015 017 018 019 020 021 022 023 025 026 027 028 029 032 033 034 035 036 037 038 041 044 045 046 047 048 049 050 051 054 055 056 057 058 059 060 061 064 065 066 068 069 071 072 077 078 079 081 084 085 086 087 091 094 095 100 103 106 108 109 110 111 112 113 117 118 119 120 121 122

Method Based On	laboratory number
International Standard	002 003 007 016 018 019 022 026 027 028 031 036 037 042 046 053 056 057 060 065 067 069 071 074 077 079 085 092 095 096 097 100 103 109 112 113 118 119
National Standard	005 013 021 025 029 032 035 038 041 045 049 050 051 054 055 058 061 078 081 086 091 094 108 110
Paper Published In An International Journal	017 023 047 048 059 117 121
Manufacturer/Kit Instructions/Technical Note	014 072
In house method	001 006 008 011 012 015 020 033 034 044 064 066 068 080 084 087 104 106 111 120 122

Quoted percentage recovery measured in same analytical batch as test material	laboratory number
no	029 041 047 085 086 096 108 121
yes	001 002 003 005 006 007 008 009 011 012 013 014 015 016 017 018 019 020 021 022 023 025 026 027 028 031 032 033 034 035 036 037 038 042 044 045 046 048 049 050 054 055 056 057 058 059 060 061 064 065 066 067 068 069 071 072 074 077 078 079 080 081 084 087 091 092 094 095 097 100 103 104 106 109 110 111 112 113 117 118 119 120 122

If measured in this batch, stage spike added	laboratory number
prior to extraction	001 002 003 005 006 007 008 009 011 012 013 014 015 016 017 018 019 020 021 022 023 025 026 027 028 029 031 032 033 034 035 036 037 038 041 042 045 046 047 048 050 051 053 055 056 057 058 059 061 064 065 066 067 069 071 072 074 077 078 080 081 084 087 091 092 094 095 096 097 100 103 104 106 108 109 110 111 112 113 117 118 119 122
prior to clean up	044
prior to instrument measurement	054 060 068 079

Concentration of Spike (µg/kg)	laboratory number
≥1 - <5	014 095
≥5 - <10	011 021 026 031 056 079 087 097 112 117 119
≥10 - <25	001 002 006 012 016 017 018 019 020 022 027 029 038 045 046 048 051 053 064 067 069 074 077 092 100 109 121
≥25 - <50	005 007 008 009 025 042 047 054 061 066 071 080 084 091 096 111 118 120
≥50 - <100	003 013 015 023 032 034 036 037 044 050 057 058 059 060 078 081 094 103 106 108 110 113 122
≥100	028 033 041 055 065 068 085 104

Composition of Blank Commodity used for Spiking	laboratory number
blank provided	001 002 003 005 007 008 009 011 014 016 019 020 021 023 025 026 027 028 029 032 034 035 036 037 038 041 042 044 045 048 050 051 053 054 055 056 057 058 059 060 061 064 066 067 068 074 077 078 080 081 084 087 091 092 094 095 096 097 100 103 104 106 108 109 111 113 118 119 120 122
Bio Cauliflower	085
blank material	006 013 017 018 022 046 065 071 072 079 110 112
Carrot	012
Cucumber	031
Leafy Vegetable	033
mustard greens	117

Composition of Blank Commodity used for Spiking (continued)	laboratory number
Previous used recovery data	047
Validation	121

Calibration	laboratory number
standard addition	011 013 025 026 028 034 037 051 054 068 084 091 095 108 110 113 120 122
matrix-matched	001 002 003 005 006 007 012 013 014 016 017 018 019 021 023 027 028 029 031 032 036 038 042 044 045 049 050 053 057 059 060 065 066 069 072 074 077 078 079 081 085 087 097 103 104 106 109 111 112 113 117 118 119 121
solvent	013 033 035 055 109
multi-level	001 002 007 008 009 012 013 015 017 020 022 033 035 037 041 046 047 048 051 056 057 058 059 061 064 066 067 068 071 074 077 078 080 091 092 094 096 100 108 109 110 113 117 118 121
single-level	047 086 109

Internal Standard Added	laboratory number
no	001 002 003 005 006 007 008 011 015 016 018 020 022 025 026 028 033 034 035 038 041 047 048 049 054 055 058 061 065 066 071 072 081 086 091 092 094 095 097 100 103 106 108 111 113 117 121
yes	009 012 013 014 017 019 021 023 027 029 031 032 036 037 042 044 045 046 050 053 056 057 059 060 064 067 068 069 074 077 078 079 080 084 085 087 096 104 109 110 112 118 119 120 122

Internal Standard Used	laboratory number
Antor	059
Atrazine-d5 and fenthion-d6	078
chlorpyrifos diethyl-d-10	084 109
DDT p,p' C13	014
Deuterated	069
Dichlorvos-d6	019
Malathion-d10	019
Dimethoate-d6	019

Internal Standard Used (continued)	laboratory number
,2,4-D-13C6	019
Parathion d10	014
Dimethachlor	053
Naphthalene D8	080
None	005 007 026 028 072 091
Paclubutrazol-D4, Triadimenol-D4	013
PCB 31/Sulfotep	085
PCB187, PCB20	118
TCA	112
TDCPP	037 119
TPHP	046
TPP	009 019 021 023 027 042 045 056 060 064 067 068 077 079 087 096 110 120 122
SULPROFOS	122
LCMSMSm PCB-52 for GCMSMS	056
Carbofuran D3	120
Isoproturon-d6	110 118
Mirex	110
Carbaryl D7	087
Trimethy phosphate	057
TRIS	109
Tris-(1,3-dichloro-2-propyl)-phosphate, Carbendazim d4, Imidacloprid d4, Nicarbazin.	067

GC Method: Sample Weight (g)	laboratory number
≥5 - <10	005 009 018 021 031 036 053 055 060 067 074 078 081 084 087 094 097 103 106 109 120
≥10 - <20	001 002 003 006 007 008 011 012 013 014 015 017 020 022 023 025 026 027 028 029 032 033 034 037 041 042 044 045 046 047 049 050 051 056 057 058 059 061 064 065 066 068 069 071 072 077 079 080 082 085 086 091 096 104 110 111 112 113 117 118 121 122
≥20 - <50	035 038 048 108

GC Method: Extraction Solvent Components	laboratory number
acetone	006 017 035 045 048 061 067
acetonitrile	001 002 003 005 007 008 009 011 012 013 014 018 020 021 022 023 025 026 027 028 029 031 032 033 034 035 036 037 038 041 042 044 046 047 049 050 051 053 055 056 057 058 060 064 065 066 068 069 071 072 074 077 078 079 080 081 084 085 086 087 091 094 096 097 104 106 108 109 110 112 113 117 118 120 121 122
dichloromethane	017 048
ethyl acetate	006 015 033 034 059 067 103 111
water	011 012 074 109
acetic acid	057 121
cyclohexane	006
hexane	045 048
n-hexane	033
petroleum ether	017

GC Method: Extraction pH Adjusted	laboratory number
no	001 002 006 007 011 014 015 017 018 021 022 023 025 026 027 028 029 032 033 034 035 036 037 038 041 042 044 045 048 050 055 056 057 058 060 061 065 066 067 068 069 072 074 078 079 080 081 082 084 085 086 087 091 094 095 096 097 100 104 106 108 109 110 111 112 117 120 121 122
yes	003 005 008 009 012 013 020 031 046 047 049 053 059 064 071 077 103 113 118

GC Method: Extraction Techniques Used	laboratory number
macerate at room temperature	017 035 055 094
QuEChERS	001 002 003 005 006 007 008 009 011 012 013 014 015 018 020 021 022 023 025 026 027 028 029 031 032 033 034 036 037 042 044 045 046 047 050 051 053 056 057 059 060 065 066 067 068 069 071 072 074 077 078 079 080 081 082 084 085 087 095 096 097 103 104 106 108 109 110 111 112 113 117 118 120 121 122
liquid-liquid partition	035 038 041 048 049 061 086 091
extraction at elevated pressure	091
column chromatography - flash	037

GC Method: Sample Clean-up Technique	laboratory number
liquid-liquid partition	011 055 061
none	015 017 035 046 059 085 109 111
solid phase extraction (SPE) (column/cartridge)	026 033 037 038 041 048 049 058 086 091 094 104 117
solid phase extraction (SPE) (dispersive)	001 002 003 005 006 007 008 009 011 012 013 014 018 020 021 022 023 025 027 028 029 031 032 034 036 042 044 045 050 051 053 056 057 060 064 065 066 067 068 069 071 072 074 077 078 079 080 081 084 087 095 096 103 106 108 110 112 113 118 120 121 122
QuECgERs	047

GC Method: SPE Sorbent Type	laboratory number
C18	001 003 018 023 029 058 077 085 103
Envicarb/GCB	044 049 091 117
PSA	001 002 006 007 008 009 011 012 013 014 021 022 023 025 026 027 028 031 032 033 034 036 037 045 047 050 051 053 056 057 060 065 066 067 069 071 072 074 079 084 087 096 104 108 109 110 112 113 118 120 121
NH2	020 038 081 086 094
Mixed Mode	042 064 078 080
COLUMN POLIXILENOS	122
florisil	048
magnesium sulfate	057 072
graphitized carbon black	072

GC Method: GC Column Packing	laboratory number
50% methyl 50% phenyl polysiloxane	018 032 055 066 112 117
65% methyl 35% phenyl polysiloxane	022 027 028 029 079
95% methyl 5% phenyl polysiloxane	002 003 005 006 007 008 009 011 012 013 014 015 017 020 021 023 025 026 031 034 035 036 037 038 042 044 045 046 047 049 050 051 056 057 058 059 060 061 064 065 067 068 069 071 072 074 077 078 081 084 085 087 091 094 096 103 104 106 108 109 110 111 113 118 121 122
100% Dimethylpolysiloxane, 6%-(cyanopropyl-phenyl) methylpolysiloxane, 50% Diphenyl 50% Dimethylpolysiloxane	033

GC Method: GC Column Packing (continued)	laboratory number
DB-XLB	080

GC Method: GC Detector Type	laboratory number
ECD	013 033 048 055 058 061 086 096 112
FPD	033 035 061 086
MS	002 013 037 047 061 080 091 094 104 108 113 120
MS-MS	001 003 005 006 007 008 009 011 012 014 015 017 018 020 021 022 023 025 026 027 028 029 031 032 034 036 038 042 044 045 046 049 050 051 053 056 057 059 060 064 065 066 067 068 069 071 072 074 077 078 079 081 082 084 085 087 097 103 106 109 111 117 118 121 122
MS-TOF	110
NPD	033

LC Method: Sample Weight (g)	laboratory number
≥1 - <5	095
≥5 - <10	005 009 018 021 031 036 053 060 074 078 081 084 086 087 094 097 103 106 109 120
≥10 - <20	001 002 003 006 007 008 011 012 013 014 015 016 017 019 020 022 023 025 026 027 028 029 032 034 037 044 045 046 047 049 050 051 054 056 057 058 059 061 064 065 066 067 068 069 071 072 077 079 080 082 085 091 092 096 100 110 111 113 117 118 119 121 122
≥20 - <50	048 108

LC Method: Extraction Solvent Components	laboratory number
acetone	048
acetonitrile	001 002 003 005 006 007 008 009 011 012 013 014 016 017 018 019 020 021 022 023 025 026 027 028 029 031 032 034 036 037 044 046 047 049 050 051 053 054 056 057 058 059 060 061 064 065 066 067 068 069 071 072 074 077 078 079 080 081 084 085 086 087 091 092 094 095 096 097 100 106 108 109 110 113 117 118 119 120 121 122
dichloromethane	048

LC Method: Extraction Solvent Components (continued)	laboratory number
ethyl acetate	015 048 103 111
methanol	045 048
1% acetic acid	057 121
Water	109

LC Method: Extraction pH Adjusted	laboratory number
no	001 002 006 007 011 014 015 016 017 018 019 022 023 025 026 027 028 029 032 034 035 036 037 044 045 047 048 056 058 060 061 065 066 067 068 069 072 078 079 080 081 082 084 085 086 087 091 094 096 097 106 108 109 110 111 117 119 120 121 122
yes	003 005 008 009 012 013 020 021 031 046 049 050 053 054 057 059 064 071 074 077 092 095 100 103 113 118

LC Method: Extraction Techniques Used	laboratory number
macerate at room temperature	094
QuEChERS	001 002 003 005 006 007 008 009 011 012 013 014 015 016 017 018 019 020 021 022 023 025 026 027 028 029 031 032 034 036 037 044 045 046 047 050 051 053 054 056 057 059 060 065 066 067 068 069 071 072 074 077 078 079 080 081 082 084 085 087 092 095 096 097 100 103 106 108 109 110 111 113 117 118 119 120 121 122
liquid-liquid partition	048 049 061 086 091
extraction at elevated pressure	091

LC Method: Sample Clean-up Technique	laboratory number
GPC/HPGPC	044
liquid-liquid partition	011 061
none	005 015 018 019 025 028 032 050 051 054 059 060 068 071 085 109 110 111
solid phase extraction (SPE) (column/cartridge)	036 048 049 058 086 091 094 117

LC Method: Sample Clean-up Technique (continued)	laboratory number
solid phase extraction (SPE) (dispersive)	001 002 003 006 007 008 009 011 012 013 014 016 017 020 022 023 026 027 029 031 034 037 045 053 056 057 064 065 066 067 072 074 077 078 079 080 081 084 087 095 096 100 103 106 108 113 119 120 121 122
QuEChERs	047

LC Method: SPE Sorbent Type	laboratory number
C18	001 025 029 032 067 077 085 103 122
Envicarb/GCB	049 091 117
PSA	001 002 003 006 007 008 009 011 012 013 014 016 017 022 026 031 034 036 037 044 045 047 050 053 056 065 066 072 074 079 084 087 096 100 108 109 111 113 118 119 120 121 023
NH2	020 048 058 081 086 094
Mixed Mode	027 064 078 080
no	060
C18	023
graphitized carbon black, magnesium sulfate	072
MgSO4	037

LC Method: HPLC Column Packing	laboratory number
C18	001 002 003 005 006 007 008 009 011 012 013 014 015 016 017 020 021 022 025 026 027 028 029 031 032 034 036 037 044 045 046 047 048 049 050 051 053 054 056 057 059 060 064 065 066 067 068 069 071 072 074 077 078 079 080 081 084 086 087 091 092 094 095 096 100 103 106 108 109 110 111 113 117 118 119 120 121 122
C8	019 085
CORTEX T3	023

LC method: Mobile Phase Components	laboratory number
ethanoic acid (acetic acid)	011 110
acetonitrile	007 011 016 022 023 026 029 034 049 067 078 086 091 108 118

LC method: Mobile Phase Components (continued)	laboratory number
ammonium acetate	001 022 034 045 065 078 081 091 092 106 108 110 118
ammonium formate	001 002 003 006 008 011 014 015 016 017 019 022 023 025 053 054 056 057 059 060 064 065 066 067 068 071 074 077 079 084 095 100 103 109 111 119 121
formic acid (methanoic acid)	002 003 006 007 008 012 017 019 021 022 023 045 049 054 057 058 059 064 065 067 069 074 077 078 079 080 092 094 100 109 111 120 121
methanol	002 003 005 006 008 009 011 012 013 017 018 019 025 027 028 031 032 036 044 045 046 048 050 053 054 059 061 064 065 067 068 072 074 077 079 080 084 085 087 094 096 100 109 110 111 113 117 121 122
water	001 002 003 006 007 008 012 013 017 019 020 022 023 031 037 045 046 047 048 051 053 054 057 059 064 065 066 067 068 072 074 078 079 085 092 094 109 110 111 113 117 118 121 122
0.1% formic acid + 5mm In ACN, DW	060

LC Method: Detector Type	laboratory number
fluorescence	048
HR-MS	019
MS-MS	001 002 003 005 006 007 008 009 011 012 013 014 015 016 017 018 020 021 022 023 025 026 027 028 029 031 032 034 036 037 044 045 046 047 049 050 051 053 054 056 058 059 060 061 064 065 066 067 068 069 071 072 074 077 078 079 080 081 082 084 085 086 087 091 094 095 096 097 100 103 106 108 109 110 111 113 117 118 119 120 121 122
LC TOF	057

Chloridazon

Source of Standards	laboratory number
Accu Standard	077
Dr Ehrenstorfer	003 018 044 049 078 081 088 113 118
Sigma/Aldrich	002 012 023 031 046
Chemlab	065

Source of Standards (continued)	laboratory number
ChemService	047
HPC Standards	110
Lab Instruments	064
LabStandard	006
national standard	091
Restek	014 119
SPEX	011 067

Method Principle	laboratory number
GC	023 047
LC	002 003 006 011 012 014 018 031 044 046 049 064 065 066 067 068 078 081 088 091 110 113 118 119

Identification by Mass Spectrometry	laboratory number
yes	002 003 006 011 012 014 018 023 031 046 047 049 064 065 066 067 068 078 081 088 091 110 113 118 119

Cymoxanil

Source of Standards	laboratory number
Accu Standard	045 058
Dr Ehrenstorfer	001 003 005 009 018 029 034 044 051 057 078 080 081 087 088 113 118
Sigma/Aldrich	002 012 031 046 059 072 097 101
Supelco	079 100
Chemlab	065
chemservice	117
CPA CHEM	017
EPA Standards Repository	007
GB/T 20769-2008	086
HPC Standards	110
LabStandard	019
national standard	091
Restek	014 070 089
Scharlab	121

Source of Standards (continued)	laboratory number
SPEX	011
spex Certiprep	084
US EPA National Pesticide Repository	008

Method Principle	laboratory number
LC	001 002 003 005 006 007 008 009 011 012 014 017 018 019 020 023 029 031 034 037 044 045 046 051 057 058 059 061 064 065 066 068 070 072 078 079 080 081 084 086 087 088 089 091 097 100 101 110 113 117 118 119 120 121

Identification by Mass Spectrometry	laboratory number
yes	001 002 003 005 006 007 008 009 011 012 014 017 018 019 020 023 029 031 034 037 044 045 046 051 057 058 059 061 064 065 066 068 070 072 078 079 080 081 084 086 087 088 089 091 097 100 101 110 113 117 118 119 120 121

Dimethomorph

Source of Standards	laboratory number
Accu Standard	045 058
Dr Ehrenstorfer	001 003 009 013 018 029 034 049 051 057 071 078 080 081 087 088 097 111 113
Sigma/Aldrich	002 012 031 046 059 096 101
Supelco	079 100
Chemlab	065
chemservice	117
CPA CHEM	017
EPA Standards Repository	007
GB/T 20769-2008	086
hpc	106
HPC standards	110
national standard	091
Restek	089 119
SPEX	011 067 109

Source of Standards (continued)	laboratory number
spex Certiprep	084
US EPA National Pesticide Repository	008

Method Principle	laboratory number
GC	044 049 087
LC	001 002 003 006 007 008 009 011 012 013 014 017 018 019 020 023 029 031 034 037 045 046 047 051 057 058 059 061 064 065 066 067 068 070 071 072 078 079 080 081 084 086 088 089 091 096 097 100 101 106 109 110 111 113 117 118 119 121

Identification by Mass Spectrometry	laboratory number
yes	001 002 003 006 007 008 009 011 012 013 014 017 018 019 020 023 029 031 034 037 044 045 046 047 049 051 057 058 059 061 064 065 066 067 068 070 071 072 078 079 080 081 084 086 087 088 089 091 096 097 100 101 106 109 110 111 113 117 118 119 121

Endosulfan-sulfate

Source of Standards	laboratory number
Accu Standard	045 055 058
Dr Ehrenstorfer	001 003 009 013 018 029 034 044 048 051 057 067 078 081 084 087 088 111 113 118 120 124
Fluka	106
Sigma/Aldrich	002 012 023 031 046 059 096 110 112
Supelco	079
Agilent (formerly Ultra Scientific)	074
chemservice	117
CPA CHEM	017
EPA Standards Repository	007
GB/T 5009.19-2008	086
national standard	091
Restek	014
SPEX	011 109

Source of Standards (continued)	laboratory number
US EPA National Pesticide Repository	008

Method Principle	laboratory number
GC	001 002 003 006 007 008 009 011 012 014 017 018 020 023 029 031 034 037 044 045 046 047 048 051 055 057 058 059 061 062 064 066 067 068 072 074 078 079 080 081 084 086 087 088 091 096 104 106 109 110 111 112 117 118 120 121 124
LC	013 113

Identification by Mass Spectrometry	laboratory number
no	048 096 124
yes	001 002 003 006 007 008 009 011 012 013 014 017 018 020 023 029 031 034 037 044 045 046 047 051 055 057 058 059 061 062 064 066 067 068 072 074 078 079 080 081 084 086 087 088 091 104 106 109 110 111 112 113 117 118 120 121

Etrimfos

Source of Standards	laboratory number
Accu Standard	045 054 058
Dr Ehrenstorfer	001 003 005 013 018 029 044 049 051 078 087 118
Fluka	106
Sigma/Aldrich	012 023 031 041 046 059 084
GB/T 5009.20-2003	086
HPC standards	110
Internal standards repository	014
national standard	091
O2Si	034
SPEX	011 067 109

Method Principle	laboratory number
GC	001 006 011 012 013 018 029 031 034 041 044 045 046 058 059 061 064 068 084 086 087 091 110
LC	003 005 014 023 049 051 054 067 078 106 109 118

Identification by Mass Spectrometry	laboratory number
yes	001 003 005 006 011 012 013 014 018 023 029 031 034 041 044 045 046 049 051 054 058 059 061 064 067 068 078 084 086 087 091 106 109 110 118

Fluopyram

Source of Standards	laboratory number
Accu Standard	045 060
Dr Ehrenstorfer	001 003 013 018 021 025 027 028 029 032 034 036 046 051 052 071 078 087 113
Sigma/Aldrich	012 023 031 059 096 101
Supelco	079 100
Agilent (formerly Ultra Scientific)	074
Chemlab	065
chemservice	117
CPA CHEM	017
EPA Standards Repository	007
hpc	106
HPC standards	110
Restek	014
SPEX	011 109
US EPA National Pesticide Repository	008

Method Principle	laboratory number
GC	009 013 017 021 025 027 028 031 032 036 037 051 052 059 060 064 110 121
LC	001 003 006 007 008 011 012 014 018 019 020 023 029 034 045 046 047 065 068 071 072 074 078 079 087 096 100 101 106 109 113 117 118

Identification by Mass Spectrometry	laboratory number
yes	001 003 006 007 008 009 011 012 013 014 017 018 019 020 021 023 025 027 028 029 031 032 034 036 037 045 046 047 051 052 059 060 064 065 068 071 072 074 078 079 087 096 100 101 106 109 110 113 117 118 121

Methoxyfenozide

Source of Standards	laboratory number
Accu Standard	045 058
Dr Ehrenstorfer	001 003 013 018 025 029 034 044 046 049 051 052 057 078 081 087 088 113
Sigma/Aldrich	002 012 031 059 101 106
Supelco	079 100
Agilent (formerly Ultra Scientific)	074
Chemlab	065
chemservice	117
CPA CHEM	017
EPA Standards Repository	007
GB/T 20769-2008	086
HPC standards	110
national standard	091
Restek	014 056 089 119
SPEX	011 067 109
Spex Certiprep	084
US EPA National Pesticide Repository	008

Method Principle	laboratory number
LC	001 002 003 006 007 008 009 011 012 013 014 017 018 019 020 023 025 029 031 034 037 044 045 046 047 051 052 056 057 058 059 061 064 065 066 067 068 070 072 074 078 079 081 084 086 087 088 089 091 100 101 106 109 110 113 117 118 119 120 121

Identification by Mass Spectrometry	laboratory number
yes	001 002 003 006 007 008 009 011 012 013 014 017 018 019 020 023 025 029 031 034 037 044 045 046 047 049 051 052 056 057 058 059 061 064 065 066 067 068 070 072 074 078 079 081 084 086 087 088 089 091 100 101 106 109 110 113 117 118 119 120 121

Oxadixyl

Source of Standards	laboratory number
Accu Standard	045 060
Dr Ehrenstorfer	001 003 009 018 021 027 028 029 032 034 036 038 042 044 050 051 052 057 078 081 087 088 113 118
Sigma/Aldrich	002 012 023 031 046 059 097 106
Supelco	079 100
Wako	049
chemservice	117
CPA CHEM	017
GB 23200.8-2016	086
HPC standards	110
national standard	091
Restek	014 056 070 089 119
SPEX	011 067
Spex Certiprep	084

Method Principle	laboratory number
GC	001 009 012 014 017 023 029 031 038 042 044 045 046 047 059 064 079 086 087 088 117 121
LC	002 003 006 011 018 020 021 027 028 032 034 036 049 050 051 052 056 057 060 061 065 067 068 070 078 081 084 089 091 097 100 106 110 113 118 119 120

Identification by Mass Spectrometry	laboratory number
yes	001 003 006 009 011 012 014 017 018 020 021 023 027 028 029 031 032 034 036 038 042 044 045 046 047 049 050 051 052 056 057 059 060 061 064 065 067 068 070 078 079 081 084 086 087 088 089 091 097 100 106 110 113 117 118 119 120 121

Pirimicarb

Source of Standards	laboratory number
Accu Standard	045 060
Dr Ehrenstorfer	001 003 009 013 018 021 029 032 034 036 044 050 051 052 057 071 078 081 087 088 102 113 118
Sigma/Aldrich	002 012 031 046 059 096 101
Supelco	079 100
Wako	049
Chemlab	065
chemservice	117
CPA CHEM	017
GB/T 20769-2008	086
hpc	106
HPC standards	110
national standard	091
Restek	014 056 070 089 119
SPEX	011 067 109
Spex Certiprep	084

Method Principle	laboratory number
GC	009 013 017 031 044 046 049 064 066 079 080 087 102 117 121
LC	001 002 003 006 011 012 014 018 019 020 021 023 029 032 034 036 037 045 047 050 051 052 056 057 059 060 061 065 067 068 070 071 078 081 084 086 088 089 091 096 100 101 106 109 110 113 118 119

Identification by Mass Spectrometry	laboratory number
yes	001 002 003 006 009 011 012 013 014 017 018 019 020 021 023 029 031 032 034 036 037 044 045 046 047 049 050 051 052 056 057 059 060 061 064 065 066 067 068 070 071 078 079 080 081 084 086 087 088 089 091 096 100 101 102 106 109 110 113 117 118 119 121

Pyridalyl

Source of Standards	laboratory number
Accu Standard	058 060
Dr Ehrenstorfer	005 018 021 025 027 028 050 051 052 057 078 113 118
Sigma/Aldrich	059
CPA CHEM	017
EPA Standards Repository	007
HPC standards	110
Restek	014
SPEX	011

Method Principle	laboratory number
GC	005 007 014 017 021 025 027 028 047 050 051 052 057 058 060 068
LC	011 018 023 059 064 072 078 110 113 118

Identification by Mass Spectrometry	laboratory number
yes	005 007 011 014 017 018 021 023 025 027 028 047 050 051 052 057 058 059 060 064 068 072 078 110 113 118

Teflubenzuron

Source of Standards	laboratory number
Accu Standard	045 058
Dr Ehrenstorfer	001 003 013 018 029 034 051 078 080 081 088 113 118
Sigma/Aldrich	002 012 023 031 046 059 101

Source of Standards (continued)	laboratory number
Supelco	100
Wako	049
Chemlab	065
CPA CHEM	017
EPA Standards Repository	007
GB/T 20769-2008	086
HPC standards	110
national standard	091
Restek	014 070 089 119
SPEX	011 067 109

Method Principle	laboratory number
GC	014 059
LC	001 002 003 006 007 011 012 013 017 018 019 023 029 031 034 037 045 046 047 049 051 058 061 064 065 067 068 070 078 080 081 086 088 089 091 100 101 109 110 113 118 119 121

Identification by Mass Spectrometry	laboratory number
no	046
yes	001 002 003 006 007 009 011 012 013 014 017 018 019 023 029 031 034 037 045 047 049 051 058 059 061 064 065 067 068 070 078 080 081 086 088 089 091 100 101 109 110 113 118 119 121

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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