Fapas® – Food Chemistry Proficiency Test Report 19294

Pesticide Residues in Strawberry Purée

August-September 2020

PARTICIPANT LABORATORY NUMBER

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SUMMARY

1. The test materials for Fapas® – Food Chemistry proficiency test 19294 were dispatched in August 2020. Each participant received a strawberry 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. However, those for Phthalimide are given *for information only.*
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
Biphenyl	50.4	25	50	50
Bupirimate	64.1	59	70	84
Cyhalothrin-lambda	133	69	81	85
Fenbuconazole	143	83	87	95
Fenhexamid	69.6	78	86	91
Indoxacarb (sum of indoxacarb and its R enantiomer)	58.5	79	85	93
Isoprothiolane	87.3	68	72	94
Methamidophos	34.4	52	63	83
Phosalone	97.9	80	88	91
Phthalimide	*85.9*	*15*	28	*54*
Propargite	49.6	62	65	95
Pyridalyl	61.2	50	63	79
Pyridaphenthion	82.1	67	76	88
Triadimefon	66.4	84	92	91
Tricyclazole	64.1	71	83	86

italics indicate for information only

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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 strawberries. 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.

Biphenyl, Bupirimate, Cyhalothrin-lambda, Fenbuconazole, Fenhexamid, Indoxacarb, Isoprothiolane, Methamidophos, Phosalone, Phthalimide, Propargite, Pyridalyl, Pyridaphenthion, Triadimefon and Tricyclazole 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 10 August 2020. Test materials were sent to 105 participants.

3. RESULTS

The instructions for reporting results were as follows:

> 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 100 participants (95%) before the closing date for this test, 18 September 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 5.

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 Biphenyl, Bupirimate, Cyhalothrin-lambda, Fenbuconazole, Fenhexamid, Indoxacarb, Isoprothiolane, Methamidophos, Phosalone, Phthalimide, Propargite, Pyridalyl, Pyridaphenthion, Triadimefon and Tricyclazole at a level ≥15 µg/kg are listed in Table 6 together with the percentage recovery and LoQ.

Participants’ comments are given in Table 7.

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 procedure used to derive this consensus involved:

i) exclusion, if present, of any non numerical results i.e. qualitative or semi-quantitative results,

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

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

iv) exclusion of results where no recovery was reported,

v) exclusion of results where limit of quantification was higher than level reported,

vi) exclusion of results where no limit of quantification was reported.

For Biphenyl, Bupirimate, Cyhalothrin-lambda, Fenbuconazole, Fenhexamid, Indoxacarb, Isoprothiolane, Methamidophos, Phosalone, Propargite, Pyridalyl, Pyridaphenthion, Triadimefon and Tricyclazole, this procedure was straightforward and the robust mean was chosen as the assigned value.

For Phthalimide the median was chosen as the assigned value due to the low number of submitted results. The assigned value and z-scores are given for *information only* due to the high uncertainty on the assigned value.

The assigned values for all analytes are shown in Table 8.

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 8.

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 8,
and σ_p = the standard deviation for proficiency, see Table 8.

Participants' z-scores for all analytes are given in Table 1-Table 5 and shown as histograms in Figures 1–15. Those for Phthalimide are given for *information only*. 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 8.

The number and percentage of z-scores in the range $-2 \leq z \leq 2$ for all analytes are given in Table 9.

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 14/05/2020.

2 GlobalSign PDF Signing Tool, https://www.globalsign.com/en/digital-signatures/ accessed 14/05/2020.

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 Biphenyl, Bupirimate and Cyhalothrin-lambda

laboratory number	analyte											
	Biphenyl assigned value: 50.4 µg/kg				Bupirimate assigned value: 64.1 µg/kg				Cyhalothrin-lambda assigned value: 133 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
001	41.99	123	10	-0.8	56.21	107	10	-0.6	157.13	82	10	0.8
002	51.2	103.4	4	0.1	71.7	99.4	4	0.5	170.7	104.7	4	1.3
003	#				#				150	66	5	0.6
004	51	100	10	0.1	55	100	10	-0.6	117	100	10	-0.6
005	#				62.96	82	10	-0.1	111.86	81	10	-0.7
006	27	117	20	**-2.1**	61	102	20	-0.2	64	141	20	**-2.4**
007	#				#				#			
008	96	100	10	**4.1**	69	100	10	0.3	132	100	10	0.0
009	28	70-120	10 ppb	-2.0	60	70-120	10 ppb	-0.3	106	70-120	10 ppb	-0.9
010	#				#				129	90	5	-0.2
011	#				#				153	66	5	0.7
012	51	90	10	0.1	74	80	10	0.7	195	100	10	**2.1**
013	22.70	84	10	**-2.5**	40.10	96	10	-1.7	124.94	90	10	-0.3
014	#				#				118.24	93	10	-0.5
015	80	88	10	**2.7**	93	104	10	2.0	146	92	10	0.4

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 Biphenyl, Bupirimate and Cyhalothrin-lambda

laboratory number	analyte											
	Biphenyl assigned value: 50.4 µg/kg				Bupirimate assigned value: 64.1 µg/kg				Cyhalothrin-lambda assigned value: 133 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
016	#				#				152.8	80~120	10	0.7
017	#				#				152.8	90	10	0.7
018	57.2	86	10	0.6	68.3	98	10	0.3	158	92	10	0.9
019	0	50-150%	10 ppb	**-4.5**	56	50-150%	10 ppb	-0.6	176	50-150%	10 ppb	1.5
020	18	44	12	**-2.9**	65	100	9	0.1	125	115	12	-0.3
021	#				#				#			
022	#				#				#			
023	59.3	60-140	10	0.8	66.1	60-140	10	0.1	126.3	60-140	10	-0.2
024	#				43.62	83	5	-1.5	134.17	91	5	0.0
025	#				#				135	85	5	0.1
026	#				39	91	0.01ppm	-1.8	129	97	0.01ppm	-0.2
027	#				59	90.9	10	-0.4	112	85.6	10	-0.7
028	77		10	**2.4**	75		10	0.8	175		10	1.4
029	74.56		10	**2.2**	71.1		10	0.5	118.82		10	-0.5
030	#				#				171	85	10	1.3

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 Biphenyl, Bupirimate and Cyhalothrin-lambda

laboratory number	analyte											
	Biphenyl assigned value: 50.4 µg/kg				Bupirimate assigned value: 64.1 µg/kg				Cyhalothrin-lambda assigned value: 133 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
031	#				#				136	95	5	0.1
032	#				#				152	103	5	0.6
033	#				#				304	85	9	**5.9**
034	48.3	97	10	-0.2	68.5	101	10	0.3	97.0	96	10	-1.3
035	#				32.73	100	10	**-2.2**	#			
036	#				0	90	0.01 mg/kg (muestra)	**-4.5**	0	90	0.01 mg/kg (muestra)	**-4.6**
037	65	94	10	1.3	66	95	10	0.1	137	90	10	0.1
038	54.6	100	10	0.4	72.9	100	10	0.6	116	100	10	-0.6
039	#				63.5	88	10	0.0	124.1	93	10	-0.3
040	#				71	101.9	10	0.5	#			
041	#				71	74	10	0.5	44	96	10	**-3.1**
042	#				#				123	95	10	-0.4
043	#				#				0			**-4.6**
044	#				#				103.78	81	10	-1.0
045	42			-0.8	70			0.4	112			-0.7

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 Biphenyl, Bupirimate and Cyhalothrin-lambda

laboratory number	analyte											
	Biphenyl assigned value: 50.4 µg/kg				Bupirimate assigned value: 64.1 µg/kg				Cyhalothrin-lambda assigned value: 133 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
046	#				70.1	88.9	10.0	0.4	173.8	113.5	10.0	1.4
047	#				#				127.55	104.91		-0.2
048	42	70	10	-0.8	60	80	10	-0.3	73	60	10	**-2.1**
049	#				55.4	102	1	-0.6	#			
050	#				31.7	117.2	15.2	**-2.3**	#			
051	#				70	107	10	0.4	111	92	10	-0.8
052	#				#				123.2	100	5	-0.4
053	#				47	77	*	-1.2	117	77	*	-0.6
054	40	100	10	-0.9	69	100	10	0.3	135	100	10	0.1
055	61.6		10	1.0	93.7		10	**2.1**	138		10	0.2
056	37	87	5	-1.2	72	130	5	0.6	130	136	5	-0.1
057	0	70-92	0,01-0,02	**-4.5**	#				117.5	92	0.01	-0.5
058	15	70% to 120%	10	**-3.2**	#				135	70% to 120%	10	0.1
059	48.77	70-120	10 ppb	-0.1	72.76	70-120	10 ppb	0.6	#			
060	61	106	10	1.0	18	105	10	**-3.3**	146	114	10	0.4

z-scores outside |z| >2 are shown in **bold,** see Section 5 # = not tested % Rec = % recovery LoQ = limit of quantification
* participant comment = 10 microgram/kg

Table 1 (continued): Results and z-Scores for Biphenyl, Bupirimate and Cyhalothrin-lambda

laboratory number	analyte											
	Biphenyl assigned value: 50.4 µg/kg				Bupirimate assigned value: 64.1 µg/kg				Cyhalothrin-lambda assigned value: 133 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
061	41	100	10	-0.9	67	100	10	0.2	136	100	10	0.1
062	#				#				#			
063	79	100	10	**2.6**	58.1	100	10	-0.4	171.9	100	20	1.3
064	#				66	100	10	0.1	125	91	10	-0.3
065	#				#				#			
066	#				#				#			
067	#				95.20		10ppb	**2.2**	#			
068	55	85	0.01	0.4	83	93	0.01	1.3	164	112	0.01	1.1
069	#				#				#			
070	#				51	111	10	-0.9	#			
071	#				64.405	90.3	10	0.0	128.957	86.3	10	-0.2
072	#				85	80 - 120%	10	1.5	#			
073	#				#				#			
074	75	104	10	**2.2**	71	97	10	0.5	155	93	10	0.7
075	#				#				118.70	101.90	2.16	-0.5

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 Biphenyl, Bupirimate and Cyhalothrin-lambda

laboratory number	analyte											
	Biphenyl assigned value: 50.4 µg/kg				Bupirimate assigned value: 64.1 µg/kg				Cyhalothrin-lambda assigned value: 133 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
076	36.1	93	13	-1.3	38.5	88	15	-1.8	73.4	90	7	**-2.1**
077	74	107	5	**2.1**	78	99	5	1.0	154	111	5	0.7
078	0		0.01 mg/kg	**-4.5**	66.0	92	0.01 mg/kg	0.1	167.0	84	0.01 mg/kg	1.2
079	15	70 to 120 %	10	**-3.2**	#				135	70 to 120 %	10	0.1
080	#				64.156	90%		0.0	#			
081	#				#				15	70-80.3	250	**-4.1**
082	0	% 90	0.010	**-4.5**	68.562	% 90	0.010	0.3	168.623	% 90	0.010	1.2
083	#				51.2	93	10	-0.9	24	93	10	**-3.8**
084	#				73.27	86	10 ppb	0.6	155.0	89	10 ppb	0.7
085	0			**-4.5**	0			**-4.5**	102		10	-1.1
086	59		10	0.8	88		10	1.7	180		10	1.6
087	38.70	114	10	-1.1	69.90	92	10	0.4	127.00	110	10	-0.2
088	#				67.49		25	0.2	#			
089	76	33	10	**2.3**	90	95	10	1.8	152	92	10	0.6
090	0	95	0.01 mg/kg	**-4.5**	133	95	10	**4.9**	200	95	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 Biphenyl, Bupirimate and Cyhalothrin-lambda

laboratory number	analyte											
	Biphenyl assigned value: 50.4 µg/kg				Bupirimate assigned value: 64.1 µg/kg				Cyhalothrin-lambda assigned value: 133 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
091	69.6	104	10	1.7	55.9	96	10	-0.6	82.6	91	10	-1.8
092	87	119	10	**3.3**	64	93	10	0.0	115	105	10	-0.6
093	112	100	10	**5.5**	153	100	10	**6.3**	166	100	10	1.1
094	0		0.010	**-4.5**	66		10	0.1	180		10	1.6
095	54.30	70-120 %	0.01	0.3	76.18	70-120 %	0.01	0.9	77.04	70-120 %	0.01	-1.9
096	#				#				#			
097	26	101.5	10	**-2.2**	53	108.5	10	-0.8	130	95.5	10	-0.1
098	0.047	70-130	0.01	**-4.5**	0.050	70-130	0.01	**-4.5**	0.12	70-130	0.01	**-4.6**
099	40.73	70-120	6	-0.9	70.01	70-120	6	0.4	107.42	70-120	6	-0.9
100	23.42	100	0.01	**-2.4**	33.00	100	0.01	**-2.2**	94.54	100	0.01	-1.3

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 Fenbuconazole, Fenhexamid and Indoxacarb

laboratory number	analyte											
	Fenbuconazole assigned value: 143 µg/kg				Fenhexamid assigned value: 69.6 µg/kg				Indoxacarb assigned value: 58.5 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
001	146.2	103	10	0.1	68.79	123	10	-0.1	51.69	97	10	-0.5
002	166.3	95.3	4	0.8	78.6	91.8	4	0.6	65.2	107.4	4	0.5
003	161	89	5	0.6	76	92	5	0.4	61	81	5	0.2
004	102	100	10	-1.3	#				46	100	10	-1.0
005	119.18	73	10	-0.8	65.67	93	10	-0.3	59.15	97	10	0.1
006	91	96	20	-1.7	143	102	20	**4.8**	37	128	20	-1.7
007	197	93	6	1.8	80	82	5	0.7	60	87	8	0.1
008	92	100	10	-1.7	53	100	10	-1.1	55	100	10	-0.3
009	128	70-120	10 ppb	-0.5	71	70-120	10 ppb	0.1	53	70-120	10 ppb	-0.4
010	145	97	5	0.1	70	97	5	0.0	47	84	5	-0.9
011	160	89	5	0.6	76	92	5	0.4	60	81	5	0.1
012	255	120	10	**3.7**	69	80	10	0.0	66	100	10	0.6
013	141.81	103	10	0.0	62.10	99	20	-0.5	60.89	101	10	0.2
014	158.67	94	10	0.5	70.00	87	10	0.0	55.38	84	10	-0.2
015	188	89	10	1.5	76	82	10	0.4	75	84	10	1.3

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 Fenbuconazole, Fenhexamid and Indoxacarb

laboratory number	analyte											
	Fenbuconazole assigned value: 143 µg/kg				Fenhexamid assigned value: 69.6 µg/kg				Indoxacarb assigned value: 58.5 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
016	148.8	80~120	10	0.2	72.7	80~120	10	0.2	64.0	80~120	10	0.4
017	143.5	92	10	0.0	62.4	95	10	-0.5	70.3	93	10	0.9
018	153	102	10	0.3	68.1	100	10	-0.1	62.0	102	10	0.3
019	174	50-150%	10 ppb	1.0	64	50-150%	10 ppb	-0.4	57	50-150%	10 ppb	-0.1
020	145	94	9	0.1	67	92	9	-0.2	57	106	6	-0.1
021	#				#				#			
022	145.5	88.2	0.1	0.1	67.7	94.7	0.1	-0.1	66.5	95.0	0.1	0.6
023	178.3	60-140	10	1.2	83	60-140	10	0.9	67.2	60-140	10	0.7
024	126.43	82	5	-0.5	69.29	86	10	0.0	50.65	73	10	-0.6
025	143	93	5	0.0	69	90	5	0.0	59	91	5	0.0
026	#				#				#			
027	127	91.1	10	-0.5	62	85.2	10	-0.5	55	89.6	10	-0.3
028	170		10	0.9	150		10	**5.3**	80		10	1.7
029	175.1		10	1.1	89.1		10	1.3	#			
030	159	99	10	0.5	67	96	10	-0.2	86	108	10	**2.1**

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 Fenbuconazole, Fenhexamid and Indoxacarb

laboratory number	analyte											
	Fenbuconazole assigned value: 143 µg/kg				Fenhexamid assigned value: 69.6 µg/kg				Indoxacarb assigned value: 58.5 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
031	96	117	5	-1.5	0			**-4.5**	48	71	5	-0.8
032	163	97	5	0.7	71	98	5	0.1	66	101	5	0.6
033	197	93	6	1.8	80	82	5	0.7	60	87	8	0.1
034	136.5	101	10	-0.2	67.2	99	10	-0.2	56.0	106	10	-0.2
035	#				#				#			
036	#				#				0	90	0.01 mg/kg (muestra)	**-4.5**
037	137	93	10	-0.2	63	100	10	-0.4	54	95	10	-0.3
038	141	100	10	-0.1	69.6	100	10	0.0	52.7	100	10	-0.4
039	137	87	10	-0.2	67	110	10	-0.2	52	87	10	-0.5
040	145	106.5	10	0.1	79	101.9	10	0.6	52	109.5	10	-0.5
041	116	74	10	-0.9	62	91	10	-0.5	44	92	10	-1.1
042	135	95	10	-0.2	61	95	10	-0.6	41	95	10	-1.4
043	106	83	5	-1.2	0			**-4.5**	0			**-4.5**
044	144.38	101	10	0.1	83.16	88	10	0.9	#			
045	159			0.5	70			0.0	60			0.1

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 Fenbuconazole, Fenhexamid and Indoxacarb

laboratory number	analyte											
	Fenbuconazole assigned value: 143 µg/kg				Fenhexamid assigned value: 69.6 µg/kg				Indoxacarb assigned value: 58.5 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
046	172.3	81.1	10.0	1.0	73.6	99.6	10.0	0.3	71.0	95.9	10.0	1.0
047	#				#				#			
048	135	80	5	-0.2	61	120	5	-0.6	50	90	5	-0.7
049	121.1	95	1	-0.7	56.5	92	1	-0.9	66.4	120	1	0.6
050	109.1	119.6	28.9	-1.1	49.7	119.6	8.9	-1.3	<LOQ	70-120%	Below 30ppb	
051	151	103	10	0.3	55	72	10	-1.0	72	102	10	1.1
052	135.9	90	5	-0.2	64.7	103	5	-0.3	52.2	101	5	-0.5
053	#				#				#			
054	147	100	10	0.1	66	100	10	-0.2	63	100	10	0.4
055	144.9		10	0.1	66		10	-0.2	66.4		10	0.6
056	143	102	5	0.0	54	103	5	-1.0	56	99	5	-0.2
057	#				#				49.8	75	0.02	-0.7
058	158	70% to 120%	10	0.5	78	70% to 120%	10	0.5	60	70% to 120%	10	0.1
059	168.86	70-120	10 ppb	0.9	72.59	70-120	10 ppb	0.2	#			
060	172	104	10	1.0	72	101	10	0.2	68	109	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 Fenbuconazole, Fenhexamid and Indoxacarb

laboratory number	analyte											
	Fenbuconazole assigned value: 143 µg/kg				Fenhexamid assigned value: 69.6 µg/kg				Indoxacarb assigned value: 58.5 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
061	100	100	10	-1.4	56	100	10	-0.9	40	100	10	-1.4
062	136			-0.2	59			-0.7	57			-0.1
063	144.2	100	10	0.1	66.1	100	10	-0.2	57.4	100	10	-0.1
064	148	102	10	0.2	59	81	10	-0.7	57	94	10	-0.1
065	152	89.9	0.41	0.3	#				#			
066	#				69.83			0.0	47.36			-0.9
067	124.22		10ppb	-0.6	#				75.71		10ppb	1.3
068	152	101	0.01	0.3	78	93	0.01	0.5	71	114	0.01	1.0
069	150.5826	92.670	8.254	0.3	70.403	86.870	18.245	0.1	#			
070	169	105	10	0.9	70	104	10	0.0	68	108	10	0.7
071	143.577	87.3	10	0.0	65.789	90.1	10	-0.2	60.916	91.2	10	0.2
072	#				#				57	80 - 120%	10	-0.1
073	#				#				#			
074	172	96	10	1.0	86	93	10	1.1	67	90	10	0.7
075	131.00	105.88	0.95	-0.4	65.23	99.06	0.52	-0.3	54.50	99.53	2.66	-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 Fenbuconazole, Fenhexamid and Indoxacarb

laboratory number	analyte											
	Fenbuconazole assigned value: 143 µg/kg				Fenhexamid assigned value: 69.6 µg/kg				Indoxacarb assigned value: 58.5 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
076	104	92	10	-1.3	82.2	95	10	0.8	40.7	89	12 ug/kg	-1.4
077	168	99	5	0.8	74	102	5	0.3	65	99	5	0.5
078	103.7	81	0.01 mg/kg	-1.3	75.6	100	0.01 mg/kg	0.4	#			
079	158	70 to 120 %	10	0.5	78	70 to 120 %	10	0.5	60	70 to 120 %	10	0.1
080	#				67.561	90%		-0.1	45.501	90%		-1.0
081	#				#				#			
082	96.751	% 90	0.010	-1.5	67.441	% 90	0.010	-0.1	53.828	% 90	0.010	-0.4
083	0	93		**-4.7**	0	93		**-4.5**	49.7	93	10	-0.7
084	148.28	99	10 ppb	0.2	0	105	10 ppb	**-4.5**	64.52	92	10 ppb	0.5
085	153		50	0.3	59		20	-0.7	65		20	0.5
086	152		10	0.3	77		10	0.5	72		10	1.1
087	136.20	84	10	-0.2	69.00	78	10	0.0	57.20	103	10	-0.1
088	140.30		25	-0.1	62.89		25	-0.4	59.43		25	0.1
089	155	100	10	0.4	65	100	10	-0.3	59	93	10	0.0
090	0	95	0.01 mg/kg	**-4.7**	158	95	10	**5.8**	90	95	10	**2.5**

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 Fenbuconazole, Fenhexamid and Indoxacarb

laboratory number	analyte											
	Fenbuconazole assigned value: 143 µg/kg				Fenhexamid assigned value: 69.6 µg/kg				Indoxacarb assigned value: 58.5 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
091	115	87	10	-0.9	87.3	95	10	1.2	45.4	92	10	-1.0
092	130	91	10	-0.4	61	79	10	-0.6	61	101	10	0.2
093	175	100	10	1.1	74	100	10	0.3	105	100	10	**3.6**
094	164		10	0.7	67		10	-0.2	59		10	0.0
095	122.74	70-120 %	0.01	-0.6	65.70	70-120 %	0.01	-0.3	61.27	70-120 %	0.01	0.2
096	#				#				#			
097	120	89.5	10	-0.7	60	103.5	10	-0.6	50	90	10	-0.7
098	0.078	70-130	0.01	**-4.7**	0.052	70-130	0.01	**-4.5**	0.048	70-130	0.01	**-4.5**
099	186.17	70-120	6	1.4	80.65	70-120	6	0.7	76.83	70-120	6	1.4
100	110.60	100	0.01	-1.0	64.08	100	0.01	-0.4	47.60	100	0.01	-0.8

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 Isoprothiolane, Methamidophos and Phosalone

laboratory number	analyte											
	Isoprothiolane assigned value: 87.3 µg/kg				Methamidophos assigned value: 34.4 µg/kg				Phosalone assigned value: 97.9 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
001	102.3	67	10	0.8	34.46	81	20	0.0	87.5	140	10	-0.5
002	95.8	106.5	4	0.4	35.8	91.6	8	0.2	109.1	98.7	4	0.5
003	88	80	5	0.0	#				117	53	5	0.9
004	#				#				83	100	10	-0.7
005	84.27	83	10	-0.2	32.90	85	10	-0.2	87.13	78	10	-0.5
006	123	100	20	1.9	92	96	20	**7.6**	90	126	20	-0.4
007	100	84	6	0.7	#				97	83	9	0.0
008	90	100	10	0.1	62	100	10	**3.6**	84	100	10	-0.6
009	81	70-120	10 ppb	-0.3	30	70-120	10 ppb	-0.6	73	70-120	10 ppb	-1.2
010	84	103	5	-0.2	#				78	84	5	-0.9
011	87	80	5	0.0	#				120	53	5	1.0
012	#				30	70	10	-0.6	130	110	10	1.5
013	77.35	101	10	-0.5	37.73	59	10	0.4	94.05	103	10	-0.2
014	87.73	86	10	0.0	#				105.49	99	10	0.4
015	118	99	10	1.6	37	98	10	0.3	136	95	10	1.8

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 Isoprothiolane, Methamidophos and Phosalone

laboratory number	analyte											
	Isoprothiolane assigned value: 87.3 µg/kg				Methamidophos assigned value: 34.4 µg/kg				Phosalone assigned value: 97.9 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
016	76.0	80~120	10	-0.6	#				115.7	80~120	10	0.8
017	85.8	94	10	-0.1	#				119.6	91	10	1.0
018	90.8	100	10	0.2	32.9	86	10	-0.2	110	90	10	0.6
019	107	50-150%	10 ppb	1.0	22	50-150%	10 ppb	-1.6	131	50-150%	10 ppb	1.5
020	80	96	12	-0.4	28	76	3	-0.8	85	100	9	-0.6
021	#				#				#			
022	82.5	83.0	0.1	-0.3	#				117.5	94.9	0.1	0.9
023	87.5	60-140	10	0.0	32.5	60-140	10	-0.3	93.6	60-140	10	-0.2
024	#				#				#			
025	85	97	5	-0.1	#				91	81	5	-0.3
026	#				#				95	96	0.01ppm	-0.1
027	78	92.8	10	-0.5	35	78.5	10	0.1	82	84.5	10	-0.7
028	101		10	0.7	35		10	0.1	130		10	1.5
029	#				#				131.6		10	1.6
030	89	91	10	0.1	#				143	117	10	**2.1**

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 Isoprothiolane, Methamidophos and Phosalone

laboratory number	analyte											
	Isoprothiolane assigned value: 87.3 µg/kg				Methamidophos assigned value: 34.4 µg/kg				Phosalone assigned value: 97.9 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
031	52	112	5	-1.8	#				106	106	5	0.4
032	97	95	5	0.5	0			**-4.5**	124	104	5	1.2
033	100	84	6	0.7	#				97	83	9	0.0
034	#				33.8	104	10	-0.1	96.7	107	10	-0.1
035	79.00	100	10	-0.4	#				#			
036	#				#				0	90	0.01 mg/kg (muestra)	**-4.5**
037	78	99	10	-0.5	27	83	10	-1.0	89	101	10	-0.4
038	81.7	100	10	-0.3	35.9	100	10	0.2	91.8	100	10	-0.3
039	#				31.5	81	10	-0.4	94	82	10	-0.2
040	#				39	106.9	10	0.6	#			
041	64	84	10	-1.2	44	67	10	1.3	66	74	10	-1.5
042	77	95	10	-0.5	0	95	10	**-4.5**	72	95	10	-1.2
043	0			**-4.5**	#				463	95	5	**16.9**
044	80.12	111	10	-0.4	#				72.49	88	10	-1.2
045	94			0.3	35			0.1	106			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 Isoprothiolane, Methamidophos and Phosalone

laboratory number	analyte											
	Isoprothiolane assigned value: 87.3 µg/kg				Methamidophos assigned value: 34.4 µg/kg				Phosalone assigned value: 97.9 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
046	92.4	89.2	10.0	0.3	35.3	89.7	10.0	0.1	111.5	92.1	10.0	0.6
047	#				0			**-4.5**	86.03	76.11		-0.6
048	#				#				89	80	5	-0.4
049	#				34.0	93	1	-0.1	#			
050	53.3	114.8	3.0	-1.8	<LOQ	70-120%	Below 30ppb		#			
051	101	107	10	0.7	30	79	10	-0.6	115	101	10	0.8
052	77.1	98	5	-0.5	#				84.7	101	5	-0.6
053	#				#				67	77	*	-1.4
054	96	100	10	0.5	33	100	10	-0.2	101	100	10	0.1
055	88.8		10	0.1	26		10	-1.1	138.9		10	1.9
056	80	132	5	-0.4	45	101	5	1.4	94	112	5	-0.2
057	#				#				81.9	78	0.01	-0.7
058	81	••	10	-0.3	35	••	10	0.1	91	••	10	-0.3
059	#				43.53	70-120	10 ppb	1.2	135.68	70-120	10 ppb	1.8
060	104	109	10	0.9	35	91	10	0.1	131	106	10	1.5

z-scores outside |z| >2 are shown in **bold,** see Section 5 # = not tested % Rec = % recovery LoQ = limit of quantification
* participant comment = 10 microgram/kg •• participant comment = 70% to 120%

Table 3 (continued): Results and z-Scores for Isoprothiolane, Methamidophos and Phosalone

laboratory number	analyte											
	Isoprothiolane assigned value: 87.3 µg/kg				Methamidophos assigned value: 34.4 µg/kg				Phosalone assigned value: 97.9 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
061	#				#				75	100	10	-1.1
062	#				28			-0.8	#			
063	96.7	100	10	0.5	34.7	100	10	0.0	104.8	100	10	0.3
064	86	96	10	-0.1	25	84	10	-1.2	113	98	10	0.7
065	79	88.7	0.46	-0.4	#				#			
066	#				#				84.53			-0.6
067	#				#				115.86		10ppb	0.8
068	100	86	0.01	0.7	34	103	0.01	-0.1	117	111	0.01	0.9
069	91.248	92.260	7.749	0.2	#				#			
070	99	112	10	0.6	#				112	109	10	0.7
071	94.786	91.7	10	0.4	34.698	82.2	10	0.0	103.207	87.2	10	0.2
072	#				37	80 - 120%	10	0.3	216	80-120	10	**5.5**
073	#				#				#			
074	105	101	10	0.9	36	82	10	0.2	105	100	10	0.3
075	72.70	104.29	0.37	-0.8	#				81.90	105.33	3.67	-0.7

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 Isoprothiolane, Methamidophos and Phosalone

laboratory number	analyte											
	Isoprothiolane assigned value: 87.3 µg/kg				Methamidophos assigned value: 34.4 µg/kg				Phosalone assigned value: 97.9 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
076	#				29.1	88	10	-0.7	49.2	102	10	**-2.3**
077	97	98	5	0.5	41	81	5	0.9	111	102	5	0.6
078	#				0		0.01 mg/kg	**-4.5**	100.1	72	0.01 mg/kg	0.1
079	81	70 to 120 %	10	-0.3	35	70 to 120 %	10	0.1	91	70 to 120 %	10	-0.3
080	84.587	90%		-0.1	36.426	90%		0.3	82.362	90%		-0.7
081	#				#				#			
082	#				0	% 90	0.010	**-4.5**	75.104	% 90	0.010	-1.1
083	0	93		**-4.5**	0	93		**-4.5**	62.2	93	10	-1.7
084	89.86	89	10 ppb	0.1	34.92	81	10 ppb	0.1	98.44	99	10 ppb	0.0
085	106		100	1.0	32		50	-0.3	107		100	0.4
086	115		10	1.4	27		10	-1.0	114		10	0.7
087	64.40	73	10	-1.2	34.80	96	10	0.0	106.10	110	10	0.4
088	89.06		25	0.1	32.96		25	-0.2	109.01		25	0.5
089	90	99	10	0.1	39	46	10	0.6	108	98	10	0.5
090	250	95	10	**8.5**	57	95	10	**3.0**	150	95	10	**2.4**

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 Isoprothiolane, Methamidophos and Phosalone

laboratory number	analyte											
	Isoprothiolane assigned value: 87.3 µg/kg				Methamidophos assigned value: 34.4 µg/kg				Phosalone assigned value: 97.9 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
091	67.6	93	10	-1.0	39.6	107	10	0.7	61.8	85	10	-1.7
092	75	83	10	-0.6	35	77	10	0.1	97	97	10	0.0
093	99	100	10	0.6	20	100	10	-1.9	155	100	10	**2.6**
094	89		10	0.1	#				116		10	0.8
095	95.59	70-120 %	0.01	0.4	28.50	70-120 %	0.01	-0.8	81.98	70-120 %	0.01	-0.7
096	#				#				#			
097	85	85.5	10	-0.1	39	-	10	0.6	87	91	10	-0.5
098	0.055	70-130	0.01	**-4.5**	0.022	70-130	0.01	**-4.5**	0.085	70-130	0.01	**-4.5**
099	#				28.43	70-120	6	-0.8	107.83	70-120	6	0.5
100	83.56	100	0.01	-0.2	14.20	100	0.01	**-2.7**	58.06	100	0.01	-1.9

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 Phthalimide, Propargite and Pyridalyl

laboratory number	analyte											
	Phthalimide assigned value: *85.9* µg/kg				Propargite assigned value: 49.6 µg/kg				Pyridalyl assigned value: 61.2 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
001	78.89	57	10	*-0.4*	52.44	98	10	0.3	75.82	60	10	1.1
002	#				50.2	101.9	4	0.1	#			
003	#				#				74	75	5	0.9
004	#				47	100	10	-0.2	#			
005	#				46.40	87	10	-0.3	#			
006	#				37	115	20	-1.2	0		20	**-4.5**
007	#				#				57	78	9	-0.3
008	181	100	10	*5.0*	53	100	10	0.3	161	17.7	10	**7.4**
009	0	70-120	10 ppb	*-4.5*	41	70-120	10 ppb	-0.8	47	70-120	10 ppb	-1.1
010	#				#				55	86	5	-0.5
011	#				#				60	75	5	-0.1
012	#				#				#			
013	#				46.70	96	10	-0.3	57.78	74	10	-0.3
014	#				#				66.76	109	10	0.4
015	100	90	10	*0.7*	63	80	10	1.2	88	87	10	2.0

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

Table 4 (continued): Results and z-Scores for Phthalimide, Propargite and Pyridalyl

laboratory number	analyte											
	Phthalimide assigned value: *85.9* µg/kg				Propargite assigned value: 49.6 µg/kg				Pyridalyl assigned value: 61.2 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
016	#				#				61.1	80~120	10	0.0
017	#				#				71.3	88	10	0.7
018	#				54.6	90	10	0.5	79.5	88	10	1.4
019	#				57	50-150%	10 ppb	0.7	53	50-150%	10 ppb	-0.6
020	#				43	82	12	-0.6	41	107	15	-1.5
021	#				#				#			
022	#				#				69.0	94.7	0.1	0.6
023	174.4	60-140	10	*4.7*	57.6	60-140	10	0.7	48.7	60-140	10	-0.9
024	#				40.67	81	5	-0.8	#			
025	#				#				63	83	5	0.1
026	#				#				#			
027	#				48	88.7	10	-0.1	#			
028	0		0.01	*-4.5*	60		10	1.0	0		0.01	**-4.5**
029	#				54.65		10	0.5	0		10	**-4.5**
030	#				#				104	102	10	**3.2**

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

Table 4 (continued): Results and z-Scores for Phthalimide, Propargite and Pyridalyl

laboratory number	analyte											
	Phthalimide assigned value: *85.9* µg/kg				Propargite assigned value: 49.6 µg/kg				Pyridalyl assigned value: 61.2 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
031	#				#				48	110	5	-1.0
032	#				#				65	100	5	0.3
033	#				#				57	78	9	-0.3
034	60.0	96	10	*-1.4*	46.0	100	10	-0.3	#			
035	#				#				#			
036	#				#				#			
037	94	100	10	*0.4*	53	96	10	0.3	62	95	10	0.1
038	85.1	100	10	*0.0*	59.4	100	10	0.9	70.0	100	10	0.6
039	#				47.8	75	10	-0.2	#			
040	#				37	79.3	10	-1.2	#			
041	#				31	81	10	-1.7	44	79	10	-1.3
042	#				#				47	95	10	-1.1
043	#				#				0			**-4.5**
044	#				#				58.01	80	10	-0.2
045	76			*-0.5*	49			-0.1	62			0.1

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

Table 4 (continued): Results and z-Scores for Phthalimide, Propargite and Pyridalyl

laboratory number	analyte											
	Phthalimide assigned value: *85.9* µg/kg				Propargite assigned value: 49.6 µg/kg				Pyridalyl assigned value: 61.2 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
046	#				56.3	100.7	10.0	0.6	#			
047	#				#				#			
048	#				56	90	5	0.6	#			
049	#				#				#			
050	#				#				#			
051	#				51	94	10	0.1	#			
052	#				#				57.2	102	5	-0.3
053	#				#				#			
054	72	100	10	*-0.7*	47	100	10	-0.2	60	100	10	-0.1
055	56.4		10	*-1.6*	68.2		10	1.7	60.2		10	-0.1
056	0		5	*-4.5*	47	115	5	-0.2	65	70	5	0.3
057	#				#				#			
058	#				40	70% to 120%	10	-0.9	#			
059	#				67.87	70-120	10 ppb	1.7	75.63	70-120	10 ppb	1.1
060	#				56	105	10	0.6	71	106	10	0.7

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

Table 4 (continued): Results and z-Scores for Phthalimide, Propargite and Pyridalyl

laboratory number	analyte											
	Phthalimide assigned value: *85.9* µg/kg				Propargite assigned value: 49.6 µg/kg				Pyridalyl assigned value: 61.2 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
061	#				36	100	10	-1.2	49	100	10	-0.9
062	#				52			0.2	#			
063	86.7	100	10	*0.0*	67.3	100	10	1.6	82.9	100	10	1.6
064	#				50	98	10	0.0	#			
065	#				#				#			
066	#				#				39.01			-1.7
067	#				25.56		10ppb	**-2.2**	#			
068	#				59	106	0.01	0.9	0	60-140	0.01	**-4.5**
069	#				#				#			
070	#				50	106	10	0.0	60	108	10	-0.1
071	#				53.899	95.7	10	0.4	51.9	87.2	10	-0.7
072	#				49	80 - 120%	10	-0.1	#			
073	#				#				#			
074	98	99	10	*0.6*	59	94	10	0.9	85	95	10	1.8
075	#				#				55.60	102.47	1.67	-0.4

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

Table 4 (continued): Results and z-Scores for Phthalimide, Propargite and Pyridalyl

laboratory number	analyte											
	Phthalimide assigned value: *85.9* µg/kg				Propargite assigned value: 49.6 µg/kg				Pyridalyl assigned value: 61.2 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
076	0	85-116	5-8 ug/kg	*-4.5*	36.1	96	15 ug/kg	-1.2	41.9	112	10	-1.4
077	82	98	5	*-0.2*	57	103	5	0.7	80	101	5	1.4
078	#				42.9	90	0.01 mg/kg	-0.6	#			
079	#				40	70 to 120 %	10	-0.9	#			
080	#				#				#			
081	#				#				#			
082	#				38.741	% 90	0.010	-1.0	34.836	% 90	0.010	-2.0
083	0	93		*-4.5*	44.8	93	10	-0.4	0	93		**-4.5**
084	#				54.45	81	10 ppb	0.4	#			
085	0			*-4.5*	53		30	0.3	66		300	0.4
086	60		10	*-1.4*	43		10	-0.6	47		10	-1.1
087	#				41.60	111	10	-0.7	57.90	105	10	-0.2
088	#				59.50		25	0.9	61.05		25	0.0
089	64	117	10	*-1.2*	53	92	20	0.3	73	89	10	0.9
090	#				100	95	10	**4.6**	0	95	0.01 mg/kg	**-4.5**

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

Table 4 (continued): Results and z-Scores for Phthalimide, Propargite and Pyridalyl

laboratory number	analyte											
	Phthalimide assigned value: *85.9* µg/kg				Propargite assigned value: 49.6 µg/kg				Pyridalyl assigned value: 61.2 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
091	#				34.8	86	10	-1.4	31.7	86	10	**-2.2**
092	108	112	10	*1.2*	55	83	10	0.5	53	76	10	-0.6
093	0		10	*-4.5*	63	100	10	1.2	#			
094	0		0.010	*-4.5*	47		10	-0.2	72		10	0.8
095	40.52	70-120 %	0.01	*-2.4*	47.68	70-120 %	0.01	-0.2	0	70-120 %	0,01	**-4.5**
096	#				#				#			
097	60	130	10	*-1.4*	46	86	10	-0.3	55	103.5	10	-0.5
098	0	70-130		*-4.5*	0	70-130		**-4.5**	0	70-130		**-4.5**
099	#				47.21	70-120	6	-0.2	#			
100	0			*-4.5*	69.56	100	0.01	1.8	0			**-4.5**

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

Table 5: Results and z-Scores for Pyridaphenthion, Triadimefon and Tricyclazole

laboratory number	analyte											
	Pyridaphenthion assigned value: 82.1 µg/kg				Triadimefon assigned value: 66.4 µg/kg				Tricyclazole assigned value: 64.1 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
001	116.06	59	10	1.9	63.78	92	10	-0.2	50.95	95	0	-0.9
002	92	101.9	2	0.6	63.7	88.8	8	-0.2	67.9	85.5	0	0.3
003	88	87	5	0.3	66	76	5	0.0	68	86	1	0.3
004	#				56	100	10	-0.7	#			
005	68.49	76	10	-0.8	59.73	79	10	-0.5	60.07	92	1	-0.3
006	107	102	20	1.4	89	101	20	1.6	129	78	1	**4.6**
007	95	93	7	0.7	78	88	6	0.8	62	83	1	-0.2
008	66	100	10	-0.9	75	100	10	0.6	103	100	1	**2.8**
009	65	70-120	10 ppb	-0.9	61	70-120	10 ppb	-0.4	64	70-120	1	0.0
010	78	98	5	-0.2	67	101	5	0.0	66	100	1	0.1
011	86	87	5	0.2	65	76	5	-0.1	67	86	1	0.2
012	120	110	10	**2.1**	75	100	10	0.6	63	90	0	-0.1
013	81.12	102	10	-0.1	60.75	87	10	-0.4	61.99	91	0	-0.2
014	58.80	90	10	-1.3	72.00	95	10	0.4	43.60	80	0	-1.5
015	88	102	10	0.3	94	91	10	1.9	104	100	1	**2.8**

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

Table 5 (continued): Results and z-Scores for Pyridaphenthion, Triadimefon and Tricyclazole

laboratory number	analyte											
	Pyridaphenthion assigned value: 82.1 µg/kg				Triadimefon assigned value: 66.4 µg/kg				Tricyclazole assigned value: 64.1 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
016	63.1	80~120	10	-1.0	65.7	80~120	10	0.0	55.7	80~120	1	-0.6
017	78.9	98	10	-0.2	65.9	97	10	0.0	63.1	85	1	-0.1
018	89.4	102	10	0.4	68.9	97	10	0.2	70.7	97	1	0.5
019	103	50-150%	10 ppb	1.2	66	50-150%	10 ppb	0.0	63	50-150%	0	-0.1
020	72	96	3	-0.6	61	105	6	-0.4	66	95	1	0.1
021	#				#				#			
022	78.2	89.3	0.1	-0.2	69.1	92.7	0.1	0.2	59.5	83.3	1	-0.3
023	72.2	60-140	10	-0.5	63.8	60-140	10	-0.2	67.6	60-140	1	0.2
024	#				59.97	88	5	-0.4	#			
025	83	90	5	0.1	65	93	5	-0.1	63	84	1	-0.1
026	#				62	95	0.01ppm	-0.3	#			
027	72	89.1	10	-0.6	57	91.1	10	-0.6	63	87.4	1	-0.1
028	95		10	0.7	77		10	0.7	65		1	0.1
029	70.44		10	-0.6	82.22		10	1.1	0		0	**-4.5**
030	88	98	10	0.3	73	91	10	0.5	60	84	1	-0.3

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

Table 5 (continued): Results and z-Scores for Pyridaphenthion, Triadimefon and Tricyclazole

laboratory number	analyte											
	Pyridaphenthion assigned value: 82.1 µg/kg				Triadimefon assigned value: 66.4 µg/kg				Tricyclazole assigned value: 64.1 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
031	60	118	5	-1.2	51	112	5	-1.1	15	95	1	**-3.5**
032	88	99	5	0.3	73	99	5	0.5	69	97	1	0.3
033	95	93	7	0.7	78	88	6	0.8	62	83	1	-0.2
034	78.0	101	10	-0.2	61.0	98	10	-0.4	58.7	94	1	-0.4
035	#				70.58	100	10	0.3	54.05	100	1	-0.7
036	0	90	0.01 mg/kg (muestra)	**-4.5**	#				#			
037	86	96	10	0.2	63	94	10	-0.2	69	101	1	0.3
038	78.1	100	10	-0.2	67.6	100	10	0.1	67.5	100	1	0.2
039	#				59	84	10	-0.5	58	95	1	-0.4
040	#				78	107.7	10	0.8	#			
041	51	120	10	-1.7	64	113	10	-0.2	64	77	1	0.0
042	73	95	10	-0.5	60	95	10	-0.4	63	95	1	-0.1
043	0			**-4.5**	0			**-4.5**	0		0	**-4.5**
044	79.24	108	10	-0.2	62.99	101	10	-0.2	74.97	92	1	0.8
045	88			0.3	80			0.9	72		1	0.6

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

Table 5 (continued): Results and z-Scores for Pyridaphenthion, Triadimefon and Tricyclazole

laboratory number	analyte											
	Pyridaphenthion assigned value: 82.1 µg/kg				Triadimefon assigned value: 66.4 µg/kg				Tricyclazole assigned value: 64.1 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
046	91.1	82.9	10.0	0.5	67.8	87.5	10.0	0.1	72.9	97.7	1	0.6
047	#				#				#			
048	#				56	95	5	-0.7	#			
049	#				57.6	95	1	-0.6	64.9	95	1	0.1
050	#				46.4	119.8	20.6	-1.4	49.9	118.9	0	-1.0
051	#				77	110	10	0.7	63	95	1	-0.1
052	71.7	91	5	-0.6	62.9	92	5	-0.2	61.9	103	1	-0.2
053	#				#				#			
054	83	100	10	0.1	69	100	10	0.2	0	100	1	**-4.5**
055	117.9		10	2.0	98.7		10	**2.2**	92.2		1	2.0
056	88	112	5	0.3	62	117	5	-0.3	64	111	1	0.0
057	#				0	70-92	0,01-0,02	**-4.5**	#			
058	#				65	70% to 120%	10	-0.1	72	70% to 120%	1	0.6
059	96.31	70-120	10 ppb	0.8	73.08	70-120	10 ppb	0.5	68.68	70-120	1	0.3
060	107	109	10	1.4	77	107	10	0.7	81	106	1	1.2

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

Table 5 (continued): Results and z-Scores for Pyridaphenthion, Triadimefon and Tricyclazole

laboratory number	analyte											
	Pyridaphenthion assigned value: 82.1 µg/kg				Triadimefon assigned value: 66.4 µg/kg				Tricyclazole assigned value: 64.1 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
061	76	100	10	-0.3	63	100	10	-0.2	41	100	1	-1.6
062	73			-0.5	60			-0.4	#			
063	91	100	10	0.5	75.9	100	10	0.7	55	100	0	-0.6
064	86	103	10	0.2	66	95	10	0.0	#			
065	#				63	90.1	1.97	-0.2	#			
066	#				#				67.51		1	0.2
067	#				20.17		10ppb	**-3.2**	#			
068	100	110	0.01	1.0	77	98	0.01	0.7	72	93	1	0.6
069	83.339	90.080	20.246	0.1	69.665	92.330	19.227	0.2	65.524	96.330	1	0.1
070	94	111	10	0.7	75	106	10	0.6	69	103	0	0.3
071	79.532	91.3	10	-0.1	68.918	89.6	10	0.2	59.942	91.6	1	-0.3
072	218	80-120	10	**7.5**	59	80 - 120%	10	-0.5	44	80-120	0	-1.4
073	#				#				#			
074	96	98	10	0.8	75	97	10	0.6	73	92	1	0.6
075	73.81	94.84	0.44	-0.5	66.86	99.84	0.57	0.0	59.80	97.25	1	-0.3

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

Table 5 (continued): Results and z-Scores for Pyridaphenthion, Triadimefon and Tricyclazole

laboratory number	analyte											
	Pyridaphenthion assigned value: 82.1 µg/kg				Triadimefon assigned value: 66.4 µg/kg				Tricyclazole assigned value: 64.1 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
076	75.5	94	8 ug/kg	-0.4	36.4	114	10	**-2.1**	40.3	89	0	-1.7
077	92	99	5	0.6	77	94	5	0.7	73	103	1	0.6
078	69.4	81	0.01 mg/kg	-0.7	63.2	86	0.01 mg/kg	-0.2	55.9	86	0	-0.6
079	#				65	70 to 120 %	10	-0.1	72	70 to 120 %	1	0.6
080	73.166	90%		-0.5	66.672	90%		0.0	74.587	90%	1	0.7
081	#				#				#			
082	44.123	% 90	0.010	**-2.1**	30.982	% 90	0.010	**-2.4**	0	% 90	1	**-4.5**
083	69.5	93	10	-0.7	48.1	93	10	-1.3	0	93	1	**-4.5**
084	#				66.70	94	10 ppb	0.0	72.41	105	1	0.6
085	0			**-4.5**	70		10	0.2	72		1	0.6
086	82		10	0.0	44		10	-1.5	45		1	-1.4
087	73.20	68	10	-0.5	59.50	80	10	-0.5	49.40	60	1	-1.0
088	#				70.81		25	0.3	62.29		0	-0.1
089	95	84	10	0.7	75	97	10	0.6	71	92	1	0.5
090	140	95	10	**3.2**	120	95	10	**3.7**	80	95	1	1.1

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

Table 5 (continued): Results and z-Scores for Pyridaphenthion, Triadimefon and Tricyclazole

laboratory number	analyte											
	Pyridaphenthion assigned value: 82.1 µg/kg				Triadimefon assigned value: 66.4 µg/kg				Tricyclazole assigned value: 64.1 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
091	59.6	81	10	-1.2	53.4	83	10	-0.9	58.9	89	1	-0.4
092	81	75	10	-0.1	62	113	10	-0.3	67	103	1	0.2
093	98	100	10	0.9	83	100	10	1.1	64	100	1	0.0
094	70		10	-0.7	68		10	0.1	60		1	-0.3
095	0	70-120 %	0,01	**-4.5**	68.20	70-120 %	0.01	0.1	0	70-120 %	1	**-4.5**
096	#				#				#			
097	83	106	10	0.1	55	112.5	10	-0.8	59	-	1	-0.4
098	0.065	70-130	0.01	**-4.5**	0.053	70-130	0.01	**-4.5**	0.043	70-130	1	**-4.5**
099	89.20	70-120	6	0.4	77.69	70-120	6	0.8	63.69	70-120	1	0.0
100	67.22	100	0.01	-0.8	65.00	100	0.01	-0.1	0		0	**-4.5**

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

Table 6: Additional Pesticide Residues Reported

laboratory number	pesticide residue ≥15 µg/kg	result µg/kg	recovery %	LoQ µg/kg
010	Demeton-S-methyl-sulfone	91	99	5
054	Triticonazole	69	100	10
073	DDD-pp (TDE)	1731	95	10
073	DDE-pp	463	95	10
073	DDT-pp	2594	95	10
073	Endosulfan I	341	95	10
073	Endosulfan II (beta)	341	95	10
073	Endosulfan-sulfate	2551	95	10
073	HCH-B (beta hexachlorocyclohexane)	208	95	10
073	Heptachlor	325	95	10
076	Acrinathrin	72	89	10 ug/kg
081	Bifenthrin	25	70-82	40
081	Chlorpyrifos (ethyl)	60	70-80	80
081	Cypermethrin	58	70-80.0	100
081	Dieldrin	15	82.3	40
082	Ethirimol	32.871	% 90	0.010
100	Acrinathrin	56.77	100	0.01

Table 7: Participants' Comments

laboratory number	comments
008	The result for Pyridalyl has been recovery corrected.
013	Our submitted result for methamidophos was corrected for the 59% spike recovery due to known loss at ITSP.
026	It took 15days for transpotation.
035	It took 14 days for transportation.
040	GC MS was not operational during study, LC only
073	This is my first proficiency test and I hope my results will be suitable.
081	The results reported are below LOQ; The reported method used are modified and validated.
084	T19294 - Strawberry Pure
085	LoQ refer to procedure and for easy input, Non-accredited analytes are also ticked with "Not detected".
088	The LOQ values is given in µg/kg (as in the case of the results). In the analytes, which we "not detected", we have LOQ values as 50 µg/kg. Thank you!
093	Results corrected by ISTD and recovery samples.

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

Table 8: 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	
Biphenyl	35	50.4	3.8	Horwitz [9]	11.1
Bupirimate	57	64.1	1.8	Horwitz [9]	14.1
Cyhalothrin-lambda	67	133	3	Horwitz [9]	28.9
Fenbuconazole	73	143	3	Horwitz [9]	30.6
Fenhexamid	69	69.6	1.1	Horwitz [9]	15.3
Indoxacarb	70	58.5	1.1	Horwitz [9]	12.9
Isoprothiolane	60	87.3	1.5	Horwitz [9]	19.2
Methamidophos	44	34.4	0.8	Horwitz [9]	7.58
Phosalone	71	97.9	2.6	Horwitz [9]	21.5
Phthalimide	14	*85.9*	*7.1*	Horwitz [9]	*18.9*
Propargite	53	49.6	1.3	Horwitz [9]	10.9
Pyridalyl	44	61.2	2.2	Horwitz [9]	13.5
Pyridaphenthion	61	82.1	1.8	Horwitz [9]	18.1
Triadimefon	77	66.4	1.0	Horwitz [9]	14.6
Tricyclazole	64	64.1	1.1	Horwitz [9]	14.1

italics indicate for information only

Table 9: Number and Percentage of z-Scores where |z| ≤2

analyte	number of scores where |z| ≤2	total number of scores	% |z|≤2
Biphenyl	25	50	50
Bupirimate	59	70	84
Cyhalothrin-lambda	69	81	85
Fenbuconazole	83	87	95
Fenhexamid	78	86	91
Indoxacarb	79	85	93
Isoprothiolane	68	72	94
Methamidophos	52	63	83
Phosalone	80	88	91
Phthalimide	*15*	*28*	*54*
Propargite	62	65	95
Pyridalyl	50	63	79
Pyridaphenthion	67	76	88
Triadimefon	84	92	91
Tricyclazole	71	83	86

italics indicate for information only

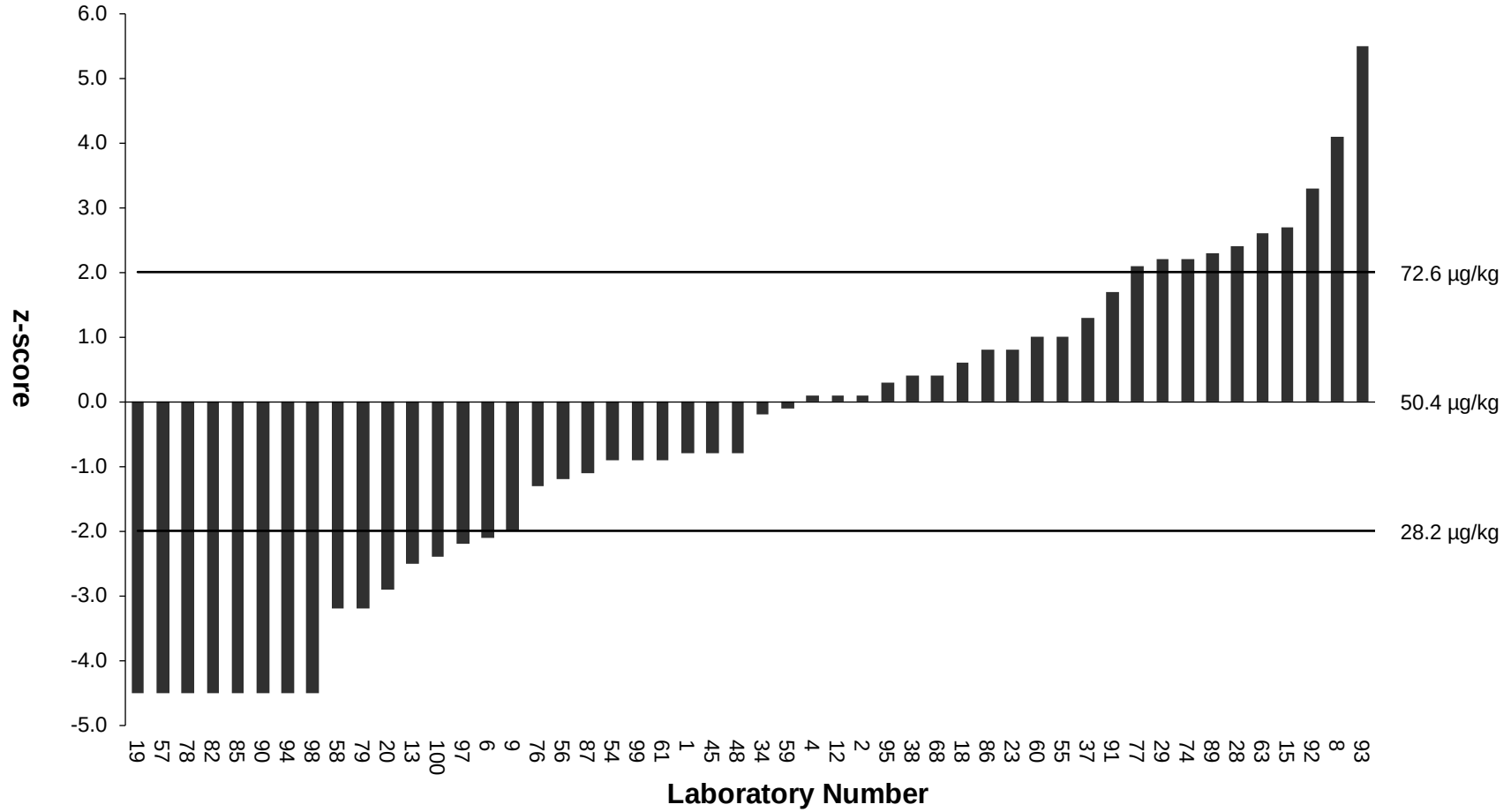

Figure 1: z-Scores for Biphenyl

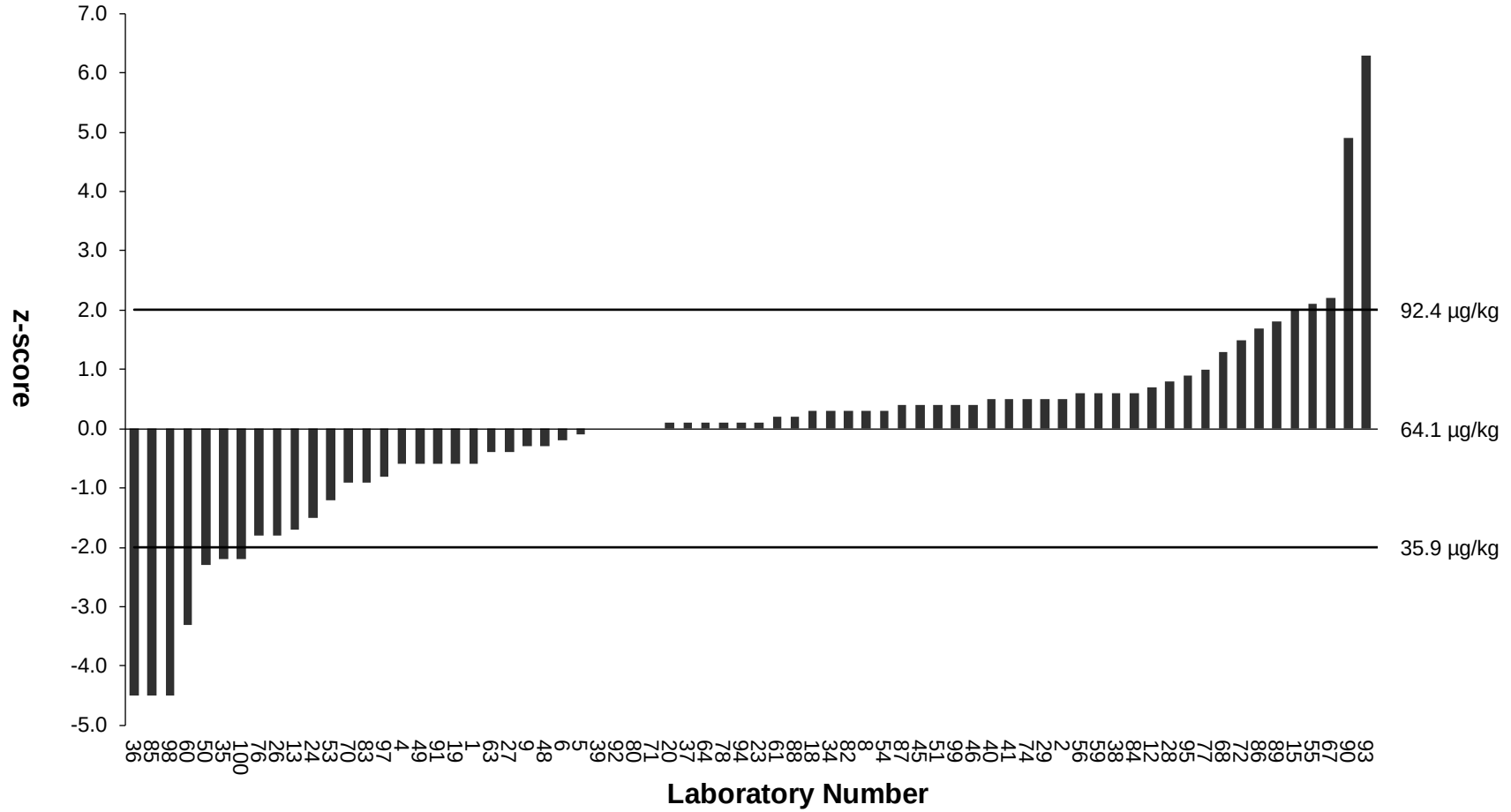

Figure 2: z-Scores for Bupirimate

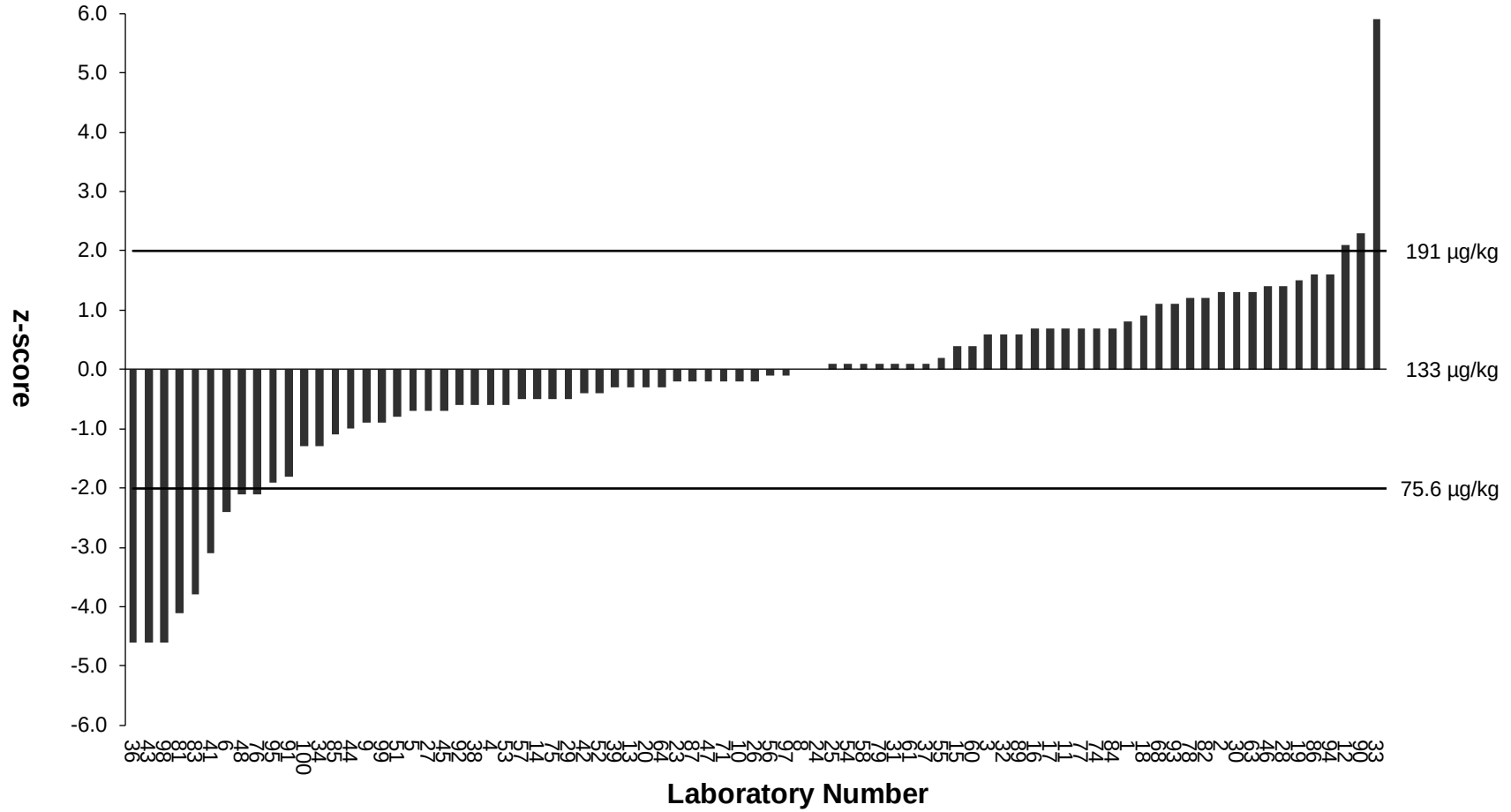

Figure 3: z-Scores for Cyhalothrin-lambda

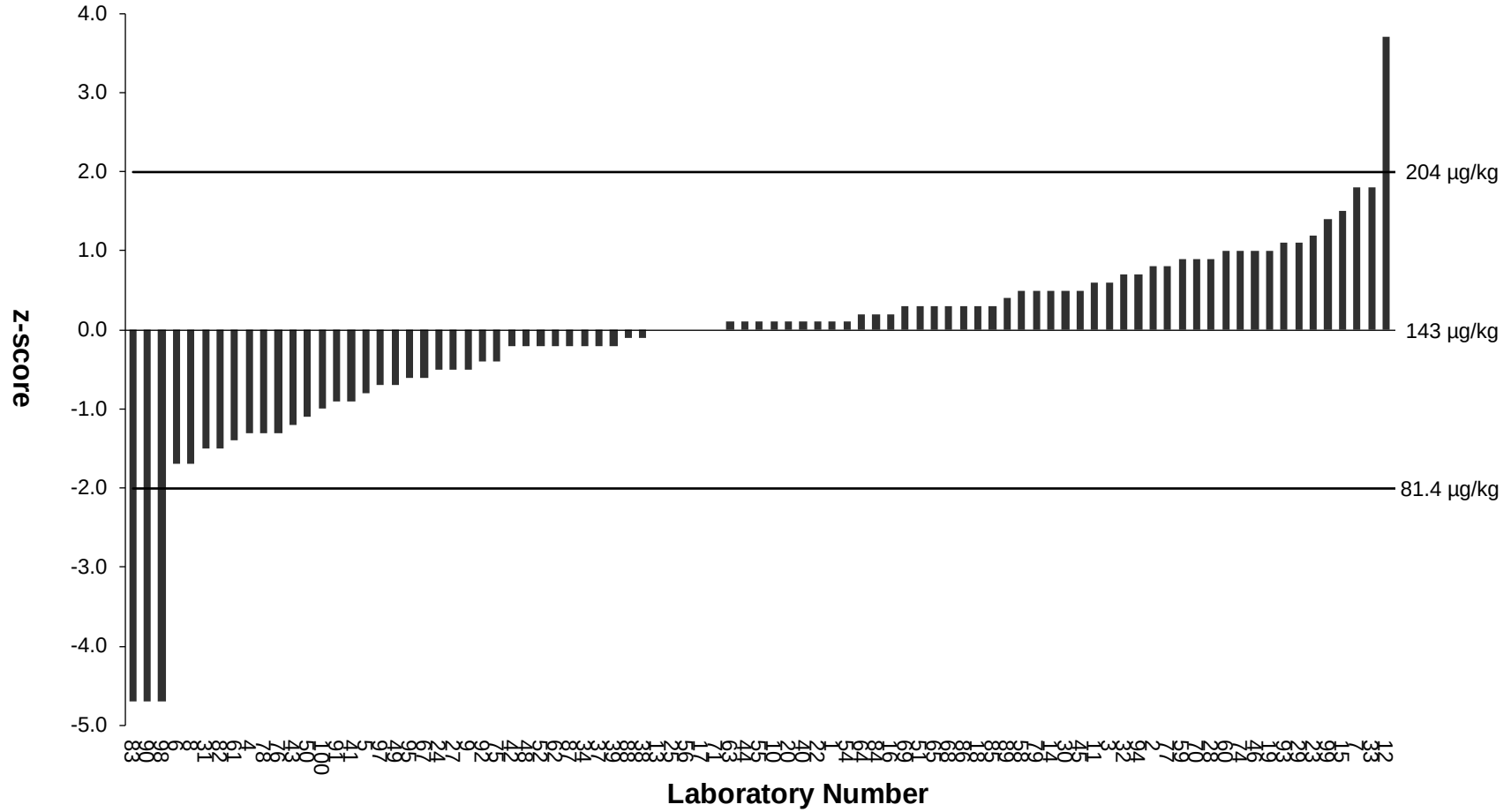

Figure 4: z-Scores for Fenbuconazole

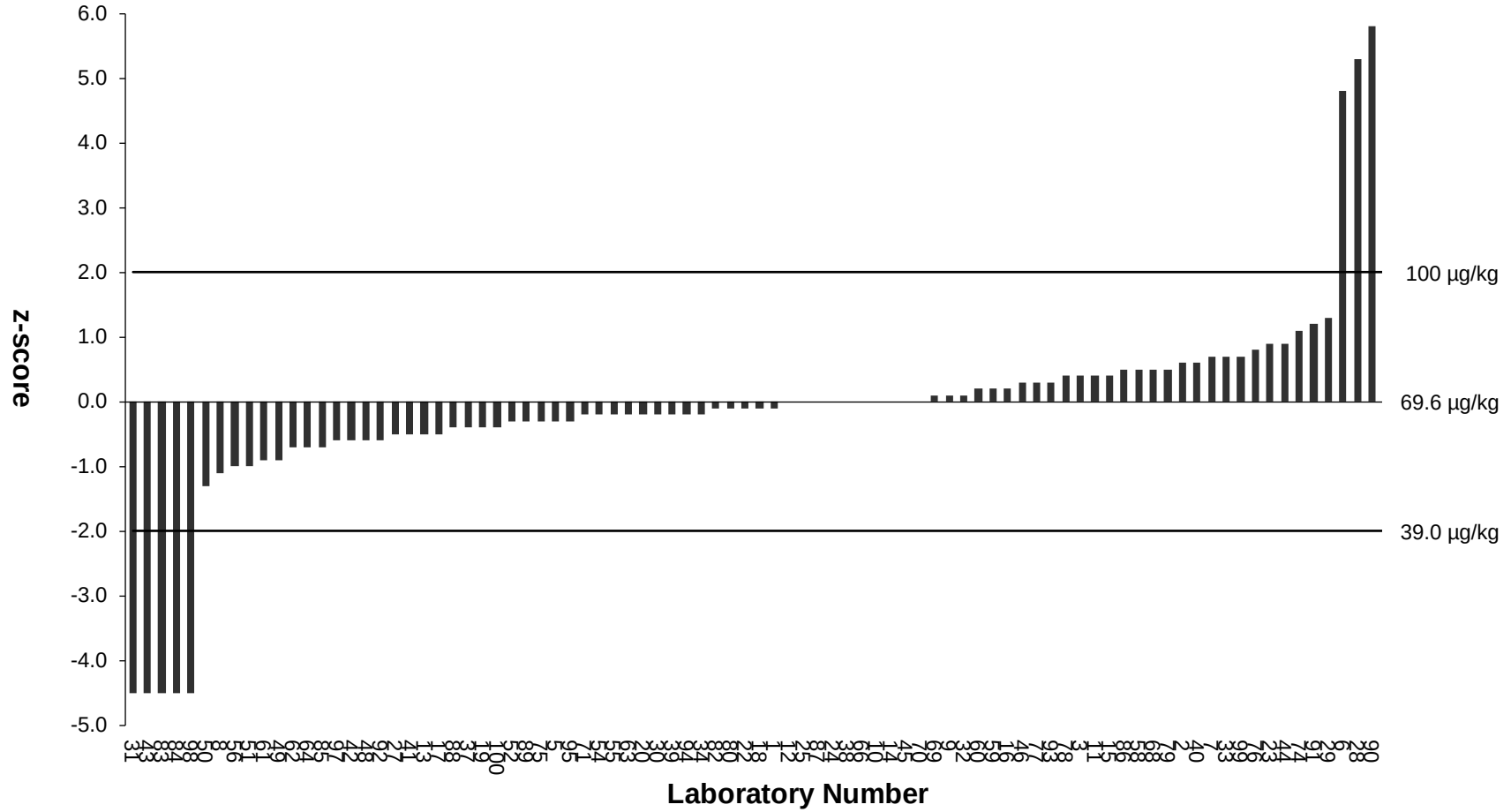

Figure 5: z-Scores for Fenhexamid

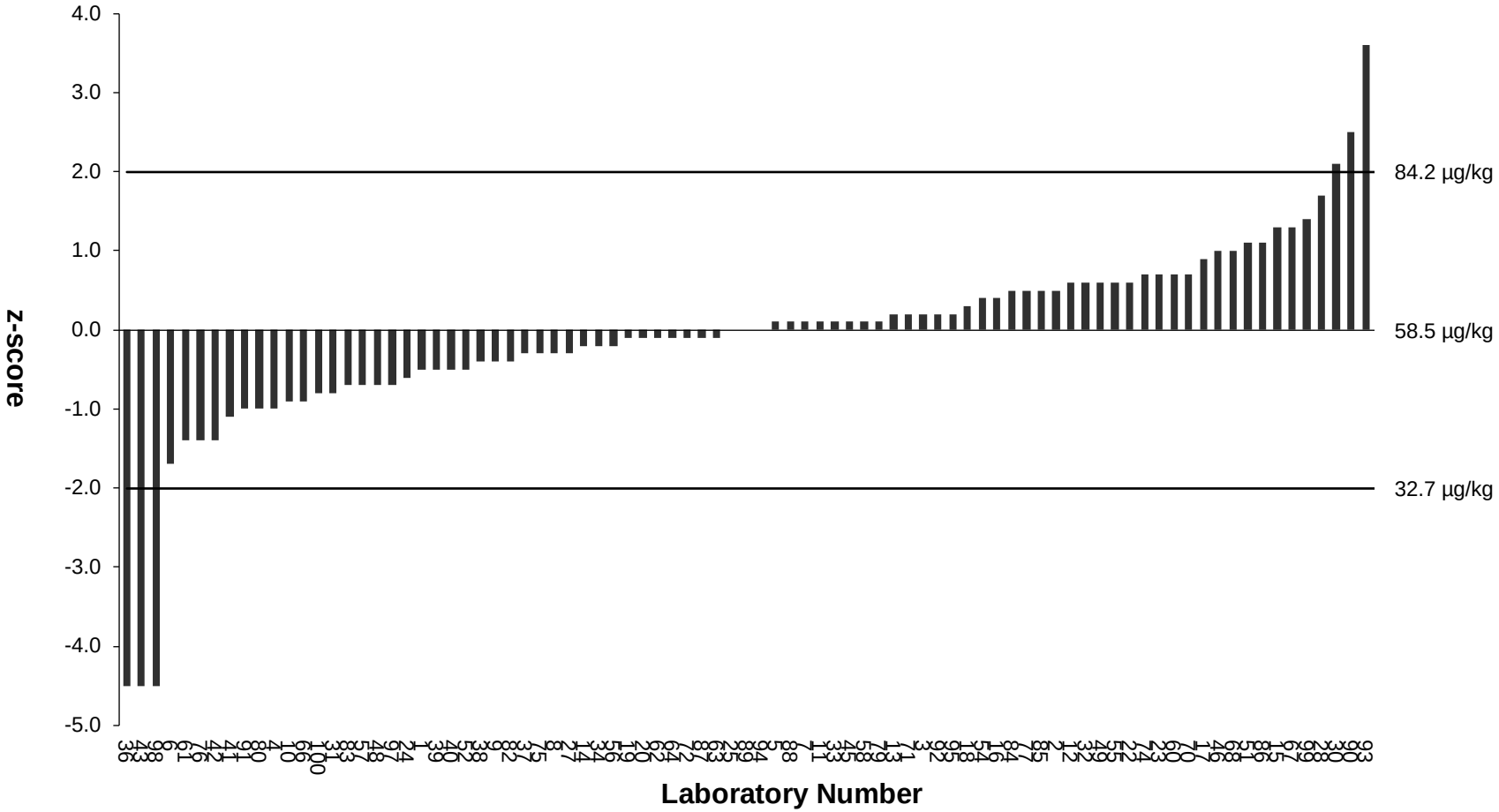

Figure 6: z-Scores for Indoxacarb

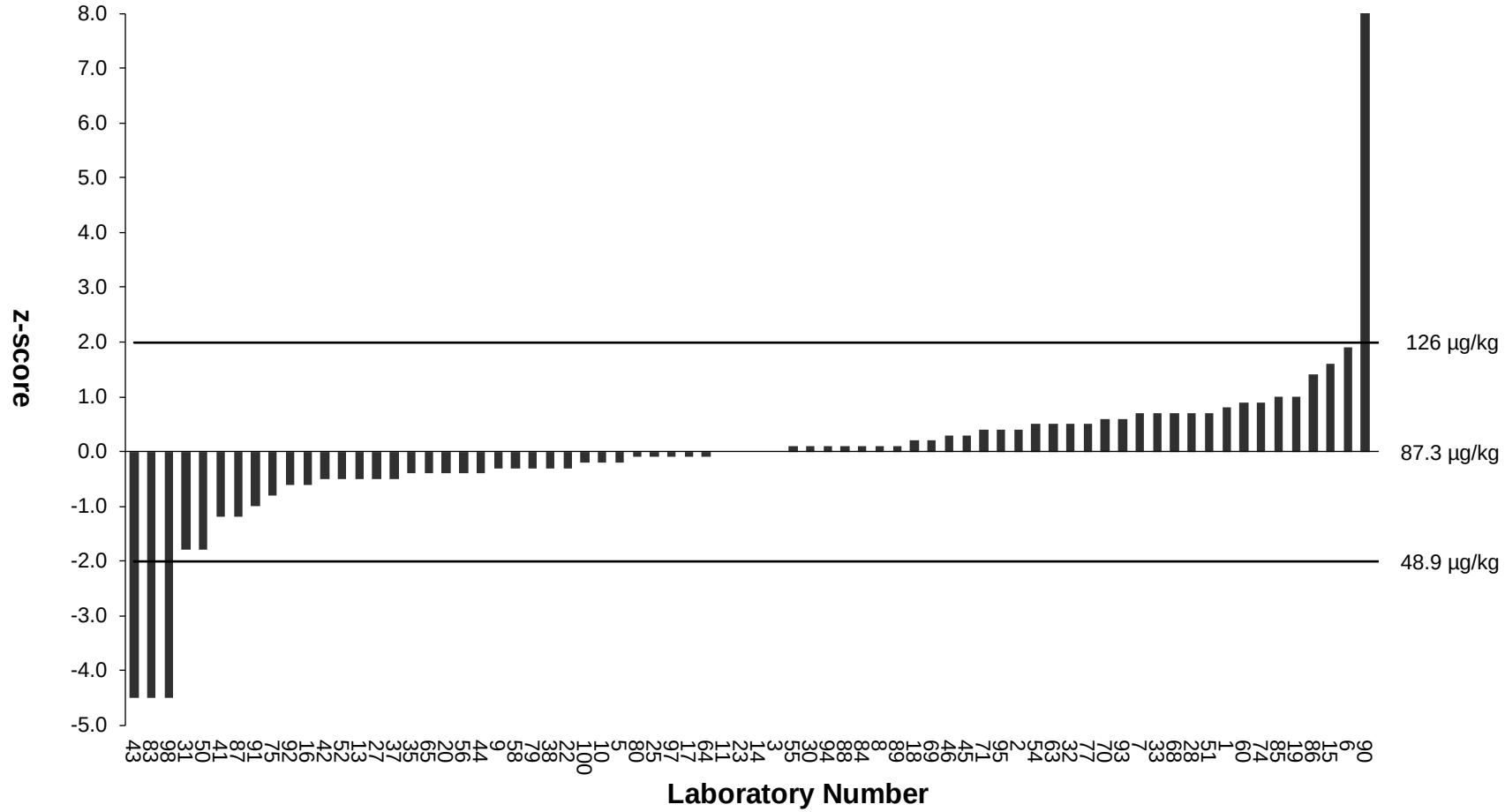

Figure 7: z-Scores for Isoprothiolane

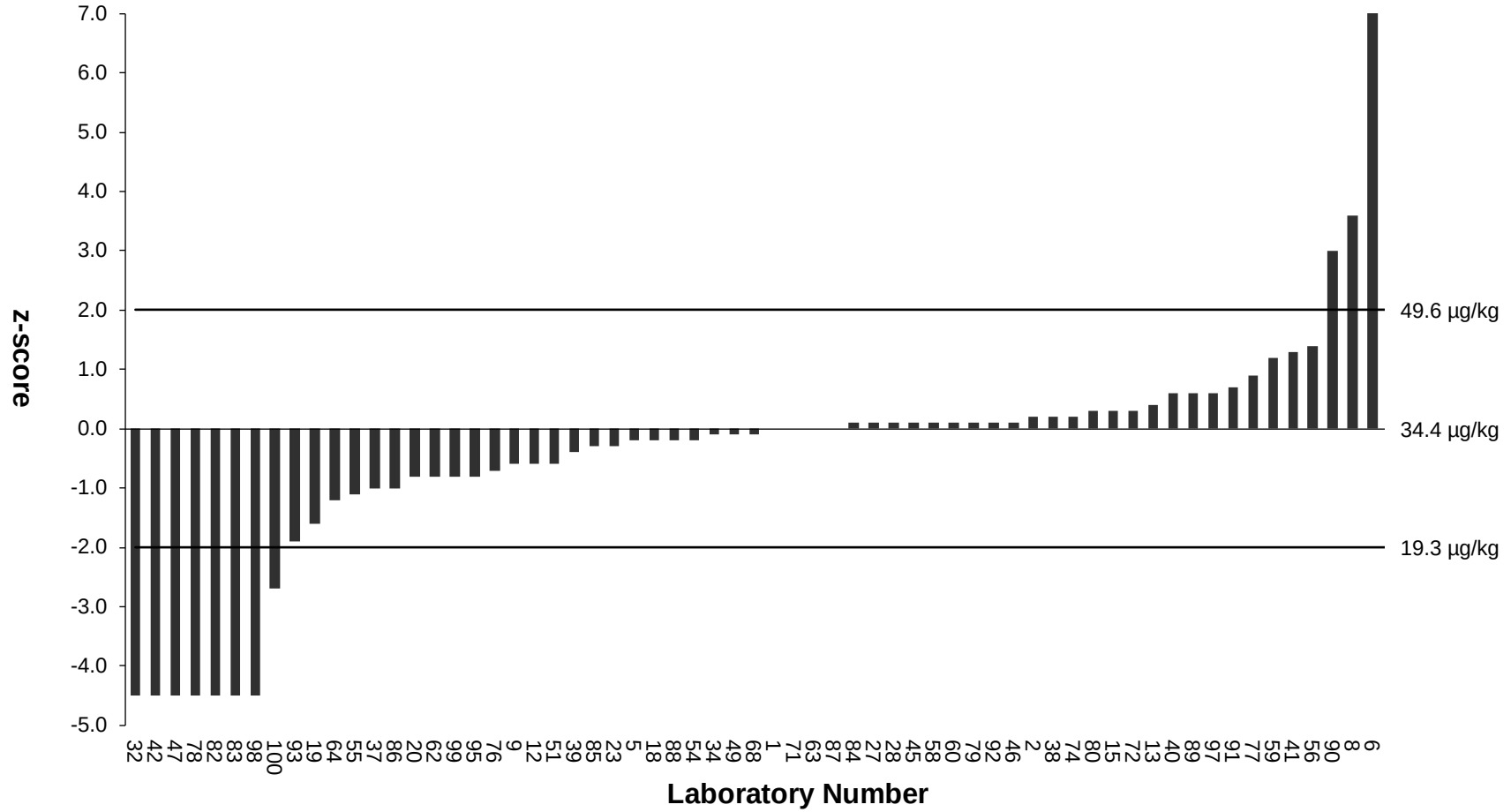

Figure 8: z-Scores for Methamidophos

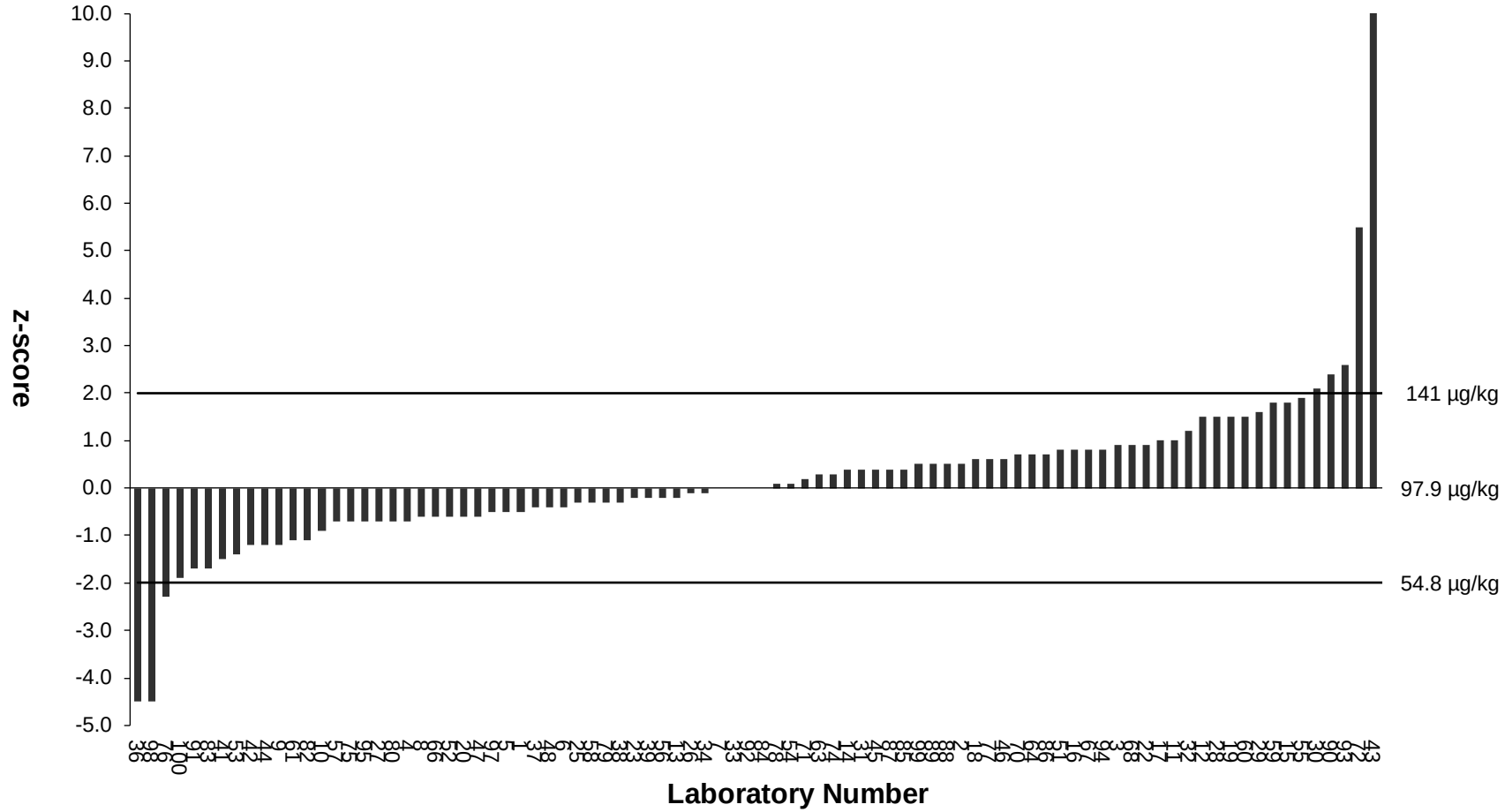

Figure 9: z-Scores for Phosalone

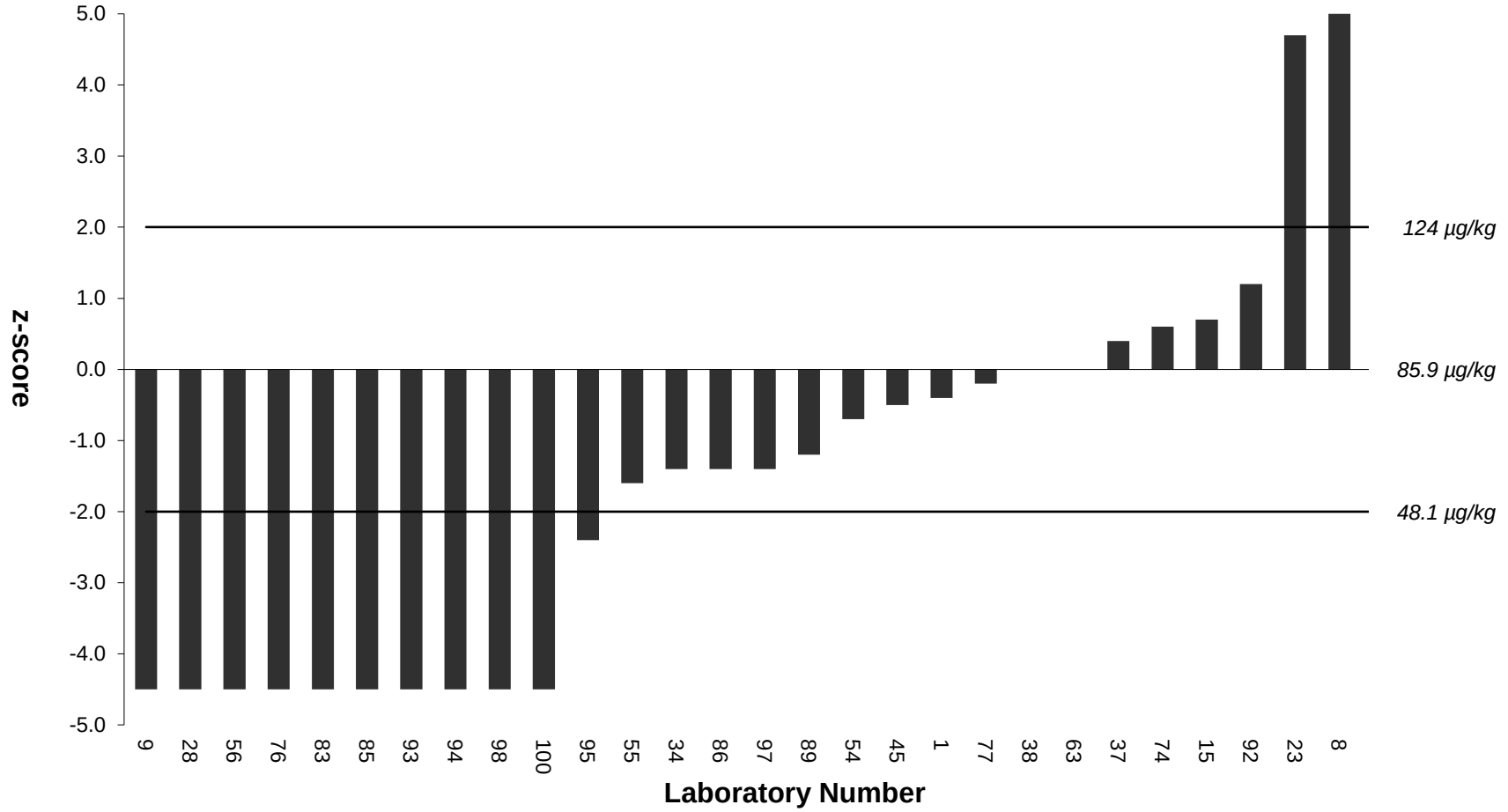

Figure 10: z-Scores for Phthalimide

this histogram is given for information only

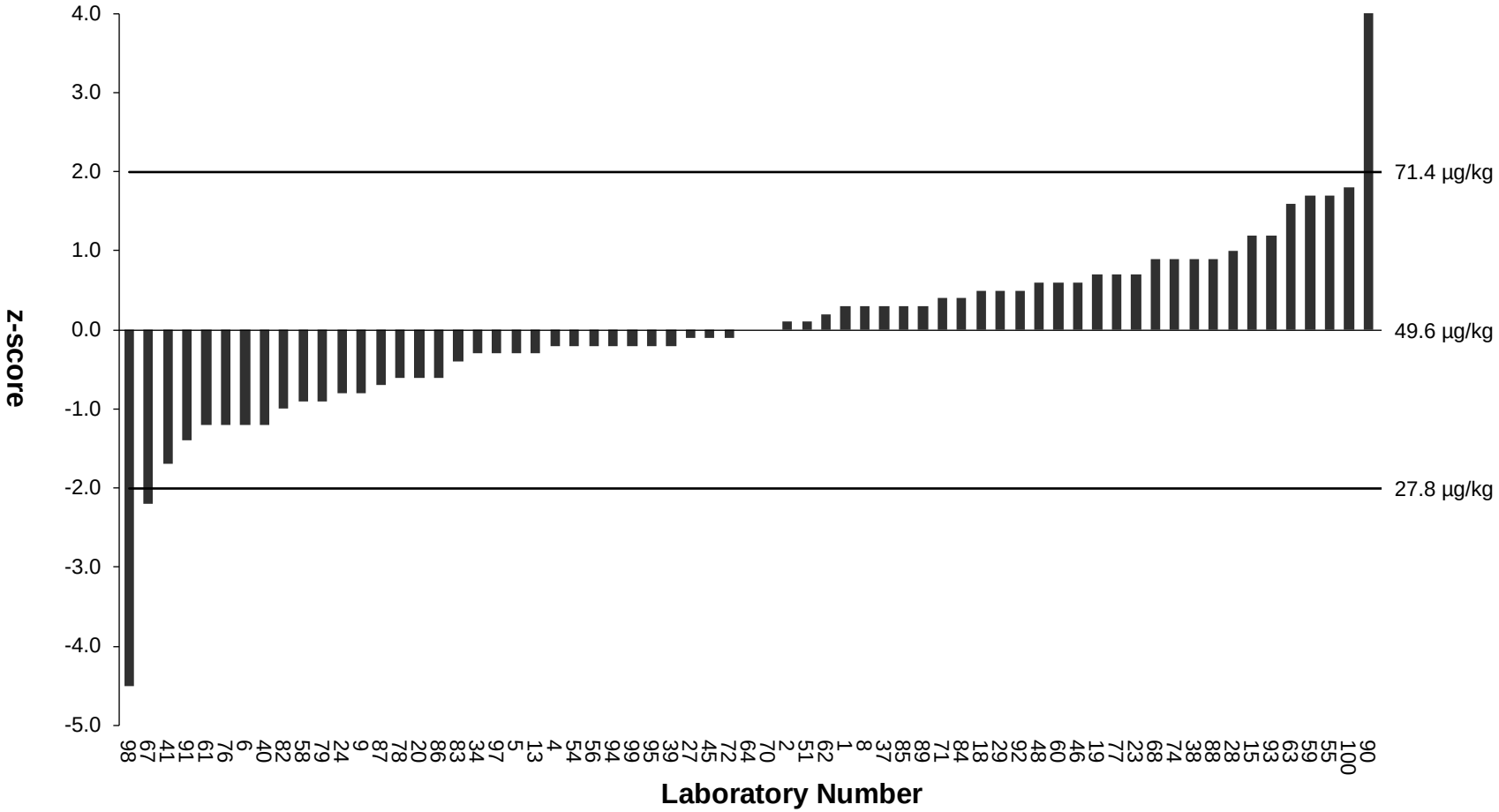

Figure 11: z-Scores for Propargite

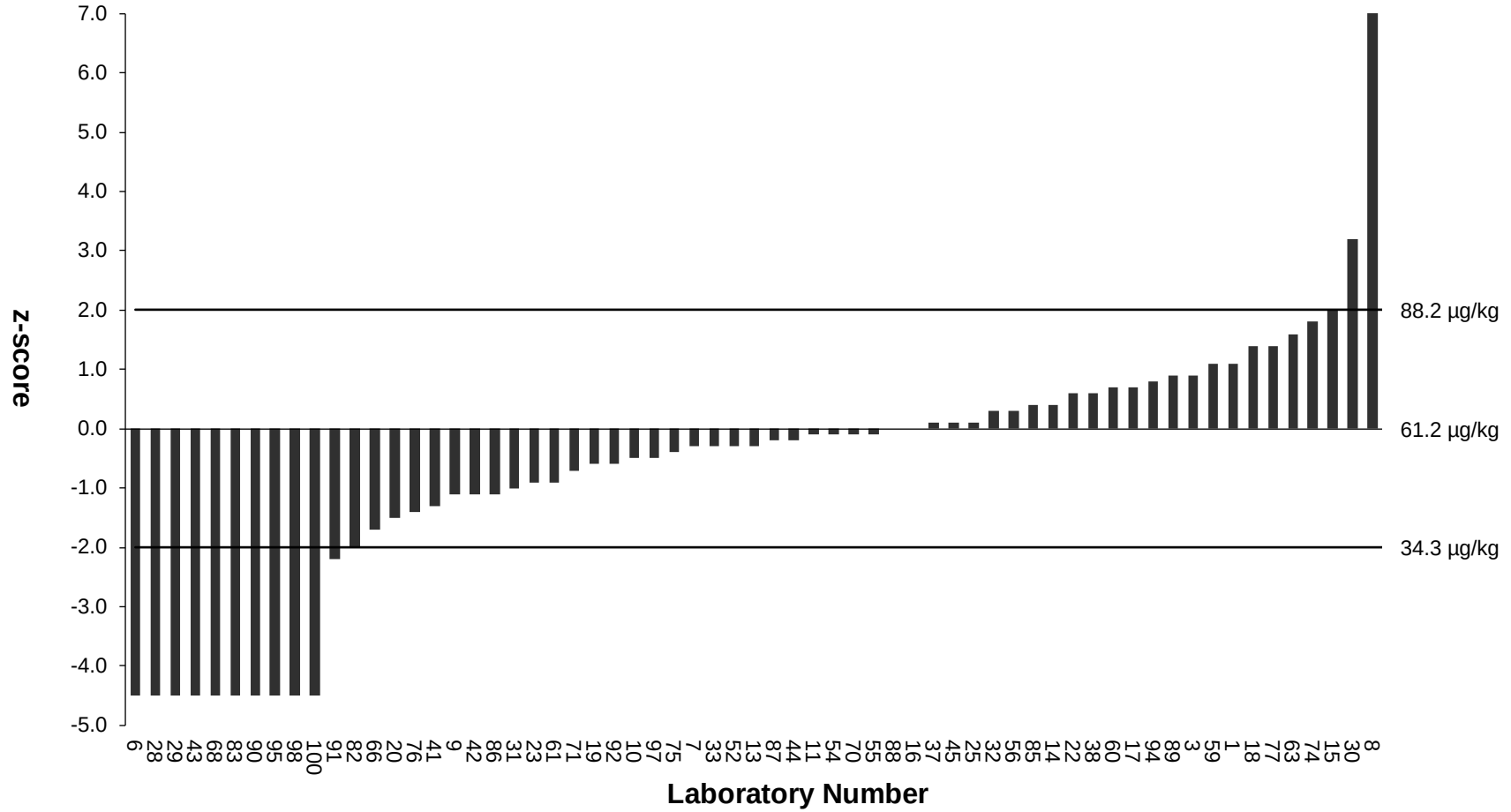

Figure 12: z-Scores for Pyridalyl

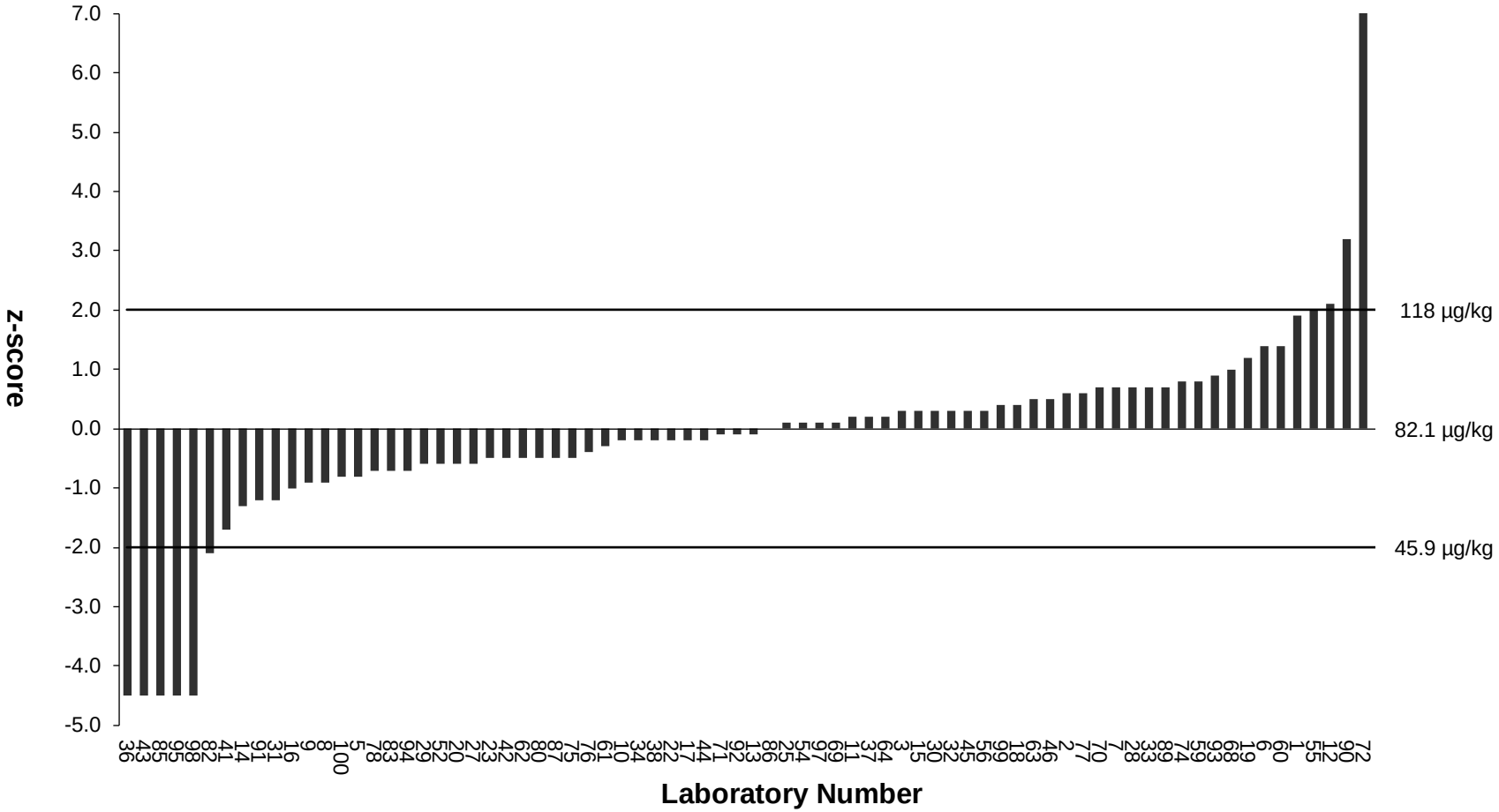

Figure 13: z-Scores for Pyridaphenthion

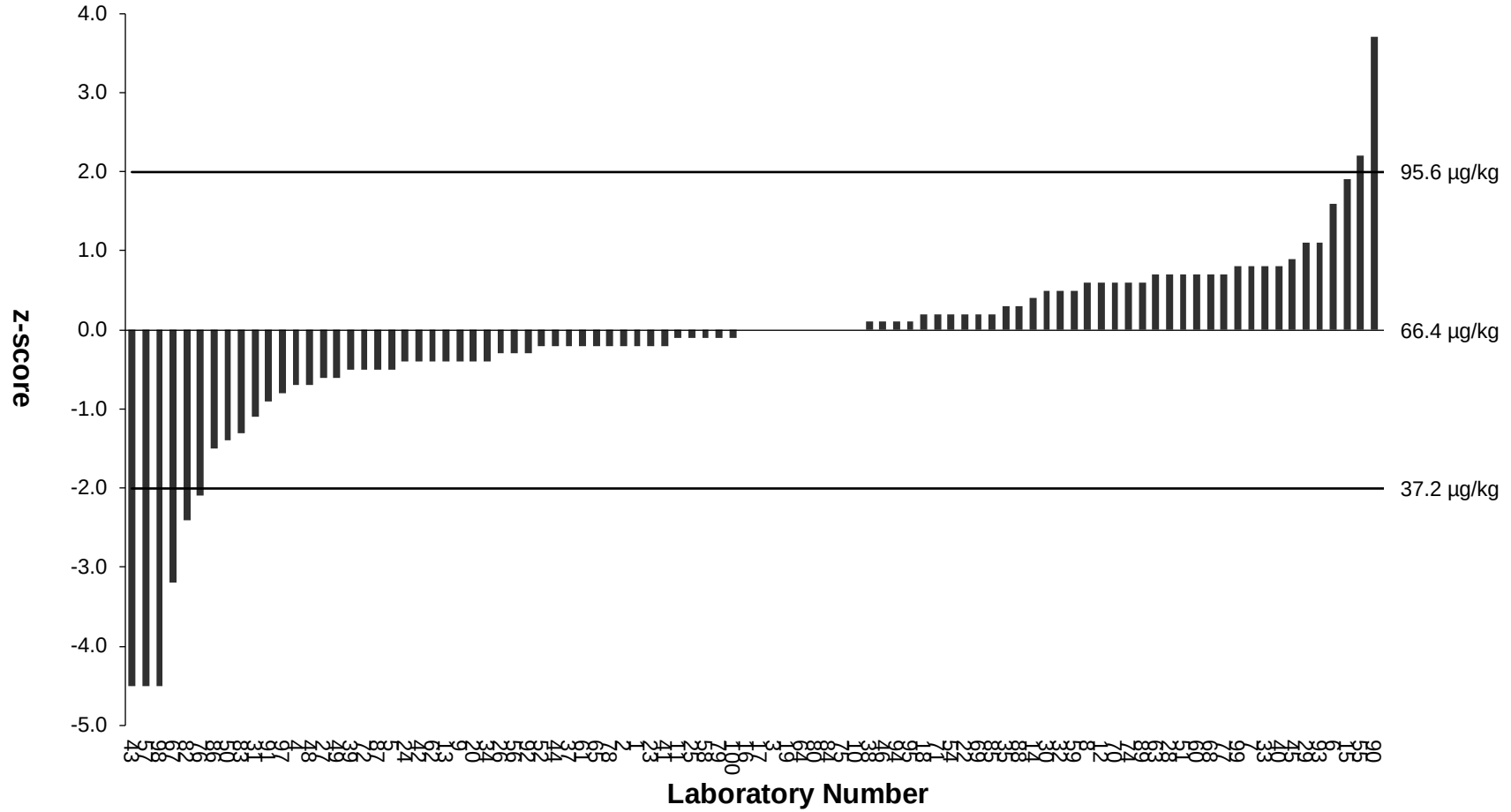

Figure 14: z-Scores for Triadimefon

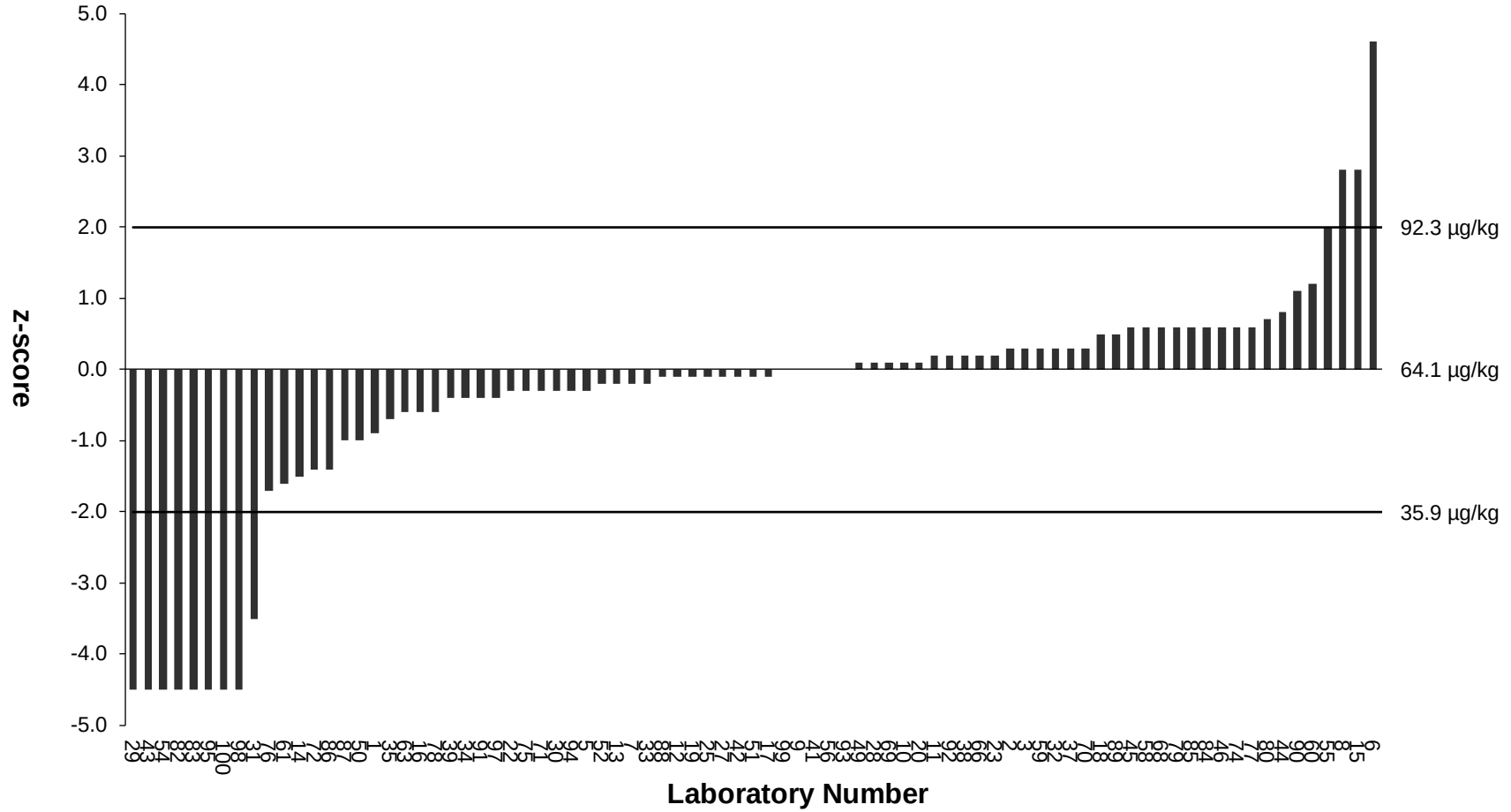

Figure 15: z-Scores for Tricyclazole

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	004 014 019 029 050 064 070 076 088 100
yes	002 003 005 006 007 008 009 010 011 013 015 016 017 018 020 022 023 025 026 027 028 030 031 032 033 035 036 037 038 039 040 042 043 044 046 048 049 053 055 056 057 058 060 065 066 068 073 075 079 081 084 086 087 089 090 092 096 097 099

Method Based On	laboratory number
International Standard	002 007 008 009 010 011 013 015 022 025 028 029 030 031 032 033 037 042 043 053 058 060 068 070 075 076 079 081 084 086 088 090 092 096 099 100
National Standard	003 005 014 016 017 018 026 027 046 048 065 066
Paper Published In An International Journal	039 040 064
Manufacturer/Kit Instructions/Technical Note	019 044 049 073
In house method	004 006 020 023 035 036 050 055 056 057 087 089 097

Quoted percentage recovery measured in same analytical batch as test material	laboratory number
no	019 026 053 066 086 088 096 100
yes	002 003 004 005 006 007 008 009 010 011 013 014 015 016 017 018 020 022 023 025 027 028 029 030 031 032 033 035 036 037 039 040 042 043 044 046 048 049 050 056 057 058 060 064 065 068 070 073 075 076 079 081 084 087 089 090 092 097 099

If measured in this batch, stage spike added	laboratory number
prior to extraction	002 003 004 005 006 007 008 009 010 011 013 014 015 016 017 018 019 020 022 023 025 026 027 028 029 030 031 032 033 035 036 037 039 040 042 043 044 046 050 056 057 058 060 064 065 068 070 075 076 079 081 084 087 089 090 092 097 099

If measured in this batch, stage spike added (continued)	laboratory number
prior to clean up	049
prior to instrument measurement	066 100

Concentration of Spike (µg/kg)	laboratory number
≥5 - <10	003 009 039 056 099
≥10 - <25	002 015 019 020 023 026 028 036 049 058 064 068 070 076 079 084
≥25 - <50	004 006 013 029 046 050 087 092 100
≥50 - <100	005 007 010 011 014 016 018 022 025 027 030 031 032 033 035 037 042 043 044 057 060 065 066 075 090 097
≥100	017 040 081 089

Source of Standards	laboratory number
Accu Standard	002 006 007 008 009 010 011 016 017 022 025 029 030 031 032 033 039 042 043 046 053 066 075 090
Dr Ehrenstorfer	004 008 014 015 018 026 028 036 037 039 046 048 049 056 057 058 060 065 068 079 084 089 092 097
Fisher	046
Fluka	002 008 046
Merck	008 097
NIST	081
Sigma/Aldrich	002 008 046 055 060 073 097
Supelco	008 046
Wako	019 026 046 070
Agilent Standard mix kit	088
CPAchem	096
Specht Hamburg	092
Hayashi Pure Chemical	035
HPC	015 060 086
Standards GmbH	015
KEMIDAS	003
Pesticide Mix - Scharlab	064
Restek	008 013 040 099

Composition of Blank Commodity used for Spiking	laboratory number
blank provided	002 003 004 005 006 007 009 010 011 013 014 015 016 017 018 019 020 023 025 026 027 029 030 031 032 033 035 036 037 039 040 042 043 044 046 048 056 058 064 065 066 068 070 075 076 079 081 084 087 089 090 092 099
Blank Fruit Sample	050
Generic mixed matrix.	008
laboratory blank	057 060
Strawberries	028 049 055

Calibration	laboratory number
standard addition	013 035 046 048 053 058 066 068 073 081 084
matrix-matched	003 005 007 008 010 011 014 015 016 017 018 020 022 023 025 026 027 028 029 030 031 032 033 037 040 042 043 044 046 055 056 057 060 064 065 068 075 076 088 090 092 099
solvent	002 013 019 050 070
multi-level	002 008 009 013 019 020 035 036 039 040 044 046 049 050 060 065 068 079 086 087 089 096 097 100
single-level	004 018 092

Internal Standard Added	laboratory number
no	003 007 010 011 014 016 023 025 026 027 028 030 031 032 033 036 043 050 058 064 065 070 073 079 081 084 090
yes	002 004 005 006 009 013 015 017 018 019 020 022 029 035 037 039 040 042 044 046 048 049 053 055 056 057 060 066 068 075 076 086 087 088 089 092 096 097 099 100

Internal Standard Used	laboratory number
ACCUSTANDARD	066
Anthrachinon, Chlorpyriphos-ethyl // Carbendazim, Isoproturon, Propamocarb, Imidacloprid	086
Atrazine deutered and Nicarbazine	039
Bromophos-methyl	013

Internal Standard Used (continued)	laboratory number
Carbendazim D3	049
Chlorpyrifos-D10, Isoproturon D6, Octachlorstyrene	015
GC-MS (TPM), LC-MS-MS (Atrd5)	020
Nicarbazin, Tritan	092
NONE	023 058 081
PCB 28	053
PCB 52 (GC); Albendazole (LC)	076
PCB209	057
Pentabromotoluol	004
Pirimicarb-d6	018
PPT as surrogate standard.	088
TPP	002 005 006 008 017 019 029 040 042 044 046 060 068 087 089 097 099
TDCPP	099
Imazalil D5	068 087
Lindane D6, Metholachlor D6, Delthamethrin D6, parathion D10	087
TDCPP, PCB-153	089
Pirimicarb-D6	060
Anthracene D10	015 068
Tritan, Paration, Diphenyl, Captan, Phtalimid (Deuteriert)	037
TTP	009

GC Method: Sample Weight (g)	laboratory number
≥1 - <5	004 006 015 016 042 057
≥5 - <10	002 007 009 010 011 013 025 030 031 032 033 039 043 048 053 056 058 075 079 081 084 086 087 092 096 100
≥10 - <20	003 005 008 014 017 018 019 020 022 023 027 028 029 035 036 037 040 044 046 055 060 064 066 068 070 073 076 089 090 097 099
≥20 - <50	026

GC Method: Extraction Solvent Components	laboratory number
acetone	035 036 057 081 089
acetonitrile	002 003 005 007 008 009 010 011 013 014 015 016 017 019 020 022 025 026 027 028 029 030 031 032 033 035 037 040 042 043 044 046 048 053 056 060 064 066 068 070 073 075 076 081 086 087 090 092 096 097 099 100
dichloromethane	036 089
ethyl acetate	004 006 009 018 023 039 048 058 079 084
water	002 015 086
acetic acid	046 064
eter petroleo	036
PE	089

GC Method: Extraction pH Adjusted	laboratory number
no	003 004 005 007 008 009 010 011 014 015 017 019 020 022 023 025 026 028 029 031 032 033 036 037 040 043 044 048 053 055 056 057 058 064 066 068 070 073 075 079 081 084 086 089 090 092 096 097
yes	002 013 016 018 027 030 035 039 042 046 060 076 087 099 100

GC Method: Extraction Techniques Used	laboratory number
macerate at room temperature	018 035
QuEChERS	002 003 005 006 007 008 009 010 011 013 014 015 016 017 019 020 022 023 025 027 028 029 030 031 032 033 037 039 040 042 043 044 046 048 053 056 058 060 064 066 068 070 073 075 076 081 084 086 087 090 092 096 097 099 100
liquid-liquid partition	004 026 035 057 079 081 089
miniluke	036

GC Method: Sample Clean-up Technique	laboratory number
GPC/HPGPC	057
liquid-liquid partition	017 018 035 056 066
none	023 036 087 089 100

GC Method: Sample Clean-up Technique (continued)	laboratory number
solid phase extraction (SPE) (column/cartridge)	013 019 026 035
solid phase extraction (SPE) (dispersive)	002 003 004 006 007 008 009 010 011 014 015 016 020 022 025 027 028 029 030 031 032 033 037 039 040 042 043 044 046 048 053 058 060 064 068 070 073 075 076 079 081 084 086 090 092 096 097 099

GC Method: SPE Sorbent Type	laboratory number
C18	009 014 017 023 028 029 073 090
Envicarb/GCB	100
PSA	002 007 008 010 011 015 016 020 022 025 027 030 031 032 033 035 037 039 040 042 043 044 053 056 058 060 064 066 068 070 075 076 079 081 084 086 096 097 099
Mixed Mode	013 019 046 092
Extrelut GCB	048
GC/NH2	026
No SPE	057

GC Method: GC Column Packing	laboratory number
50% methyl 50% phenyl polysiloxane	090 100
65% methyl 35% phenyl polysiloxane	009
95% methyl 5% phenyl polysiloxane	002 003 004 007 008 010 011 013 014 015 016 017 018 019 022 023 025 027 028 030 031 032 033 035 036 037 039 040 042 043 046 048 053 056 057 058 060 064 066 068 070 075 076 079 081 084 087 089 092 096 097 099
cyanopropylphenyl	020
HP -5MS 5% Phenyl Methyl Silox 30m x 250µm x 0.25µm column	029
methylpolysiloxane	020 026
TR-5MS 30 m x 0,25 mm	073

GC Method: GC Detector Type	laboratory number
ECD	057 081
MS	004 019 020 039 048 053 057 092 096

GC Method: GC Detector Type (continued)	laboratory number
MS-MS	002 003 005 006 007 008 009 010 011 013 014 015 016 017 018 022 023 025 026 027 028 029 030 031 032 033 035 036 037 040 042 043 044 046 055 056 058 060 064 066 068 070 073 075 076 079 084 087 089 090 097 099 100
TOF	086

LC Method: Sample Weight (g)	laboratory number
≥1 - <5	006 015 016 042 086
≥5 - <10	002 007 009 010 011 013 025 030 031 032 033 039 043 048 056 058 065 079 084 087 092
≥10 - <20	003 005 008 014 017 018 019 020 022 023 027 028 029 036 037 040 044 046 049 050 055 057 060 064 066 068 070 075 076 089 090 097 099

LC Method: Extraction Solvent Components	laboratory number
acetone	036 057 089
acetonitrile	002 003 005 007 008 009 010 011 013 014 015 016 017 019 020 022 023 025 027 028 029 030 031 032 033 037 039 040 042 043 044 046 048 049 050 056 060 064 065 066 068 070 075 076 084 086 087 088 090 092 097 099
dichloromethane	036 089
ethyl acetate	006 018 058 079 084
methanol	048 057
acetic acid	046 064
water	086
eter petroleo	036
PE	089

LC Method: Extraction pH Adjusted	laboratory number
no	005 007 008 009 010 011 014 015 017 019 020 022 023 025 028 029 031 032 033 036 037 040 043 044 048 050 055 056 058 064 066 068 070 075 079 084 086 089 090 092 097

LC Method: Extraction pH Adjusted (continued)	laboratory number
yes	002 003 013 016 018 027 030 039 042 046 049 057 060 065 076 087 088 099

LC Method: Extraction Techniques Used	laboratory number
macerate at room temperature	018
QuEChERS	002 003 005 006 007 008 009 010 011 013 014 015 016 017 019 020 022 023 025 027 028 029 030 031 032 033 037 039 040 042 043 044 046 048 049 050 056 058 060 064 065 066 068 070 075 076 079 084 086 087 088 090 092 097 099
liquid-liquid partition	057 089
miniluke	036

LC Method: Sample Clean-up Technique	laboratory number
GPC/HPGPC	057
liquid-liquid partition	018 042 048 056 066 086
none	002 003 014 016 017 019 020 022 023 036 044 049 050 075 087 089 097
solid phase extraction (SPE) (column/cartridge)	013
solid phase extraction (SPE) (dispersive)	006 007 008 009 010 011 015 025 027 028 029 030 031 032 033 037 039 040 043 046 058 060 064 068 070 076 079 084 088 090 092 099
QuEchERS	065

LC Method: SPE Sorbent Type	laboratory number
C18	009 017 023 028 029 042 090
PSA	007 008 010 011 015 022 025 027 030 031 032 033 037 039 040 043 056 058 060 064 066 068 070 076 079 084 088 099
Mixed Mode	013 046 092
no SPE	057
none	002 020

LC Method: HPLC Column Packing	laboratory number
C18	002 003 005 007 008 009 010 011 013 014 016 017 018 019 020 022 023 025 027 028 029 030 031 032 033 036 037 039 040 042 043 044 046 048 049 050 056 057 058 060 064 065 066 068 070 075 076 079 084 086 087 088 089 090 092 097 099
C8	015

LC method: Mobile Phase Components	laboratory number
ethanoic acid (acetic acid)	039 89
acetonitrile	002 015 040 056 065 086 087 090
ammonium acetate	005 018 019 046 049 089
ammonium formate	002 003 006 008 009 013 020 023 028 037 039 042 048 050 056 058 060 064 070 075 076 079 088 092 099
formic acid (methanoic acid)	002 003 006 007 008 010 011 016 020 023 025 028 030 031 032 033 040 042 043 044 046 050 056 060 064 066 070 076 086 088 099
methanol	002 003 006 008 013 014 018 019 020 022 023 027 028 029 036 039 042 044 046 048 049 050 056 057 060 064 068 070 084 086 088 089 092 097
phosphate	057
water	002 003 006 008 013 015 017 019 023 028 029 036 039 040 042 044 046 048 049 050 060 064 065 070 084 086 087 089 092 097

LC Method: Detector Type	laboratory number
Diode Array Detector	057
HR-MS	019 040
MS	067
MS-MS	002 003 005 006 007 008 009 010 011 013 014 015 016 017 018 020 022 023 025 027 028 029 030 031 032 033 036 037 039 042 043 044 046 048 049 055 056 058 060 064 065 066 068 070 075 076 079 084 086 087 088 089 090 092 097 099
HRAM	050

Biphenyl

Method Principle	laboratory number
GC	002 004 008 013 015 018 023 028 029 034 037 048 056 058 060 068 076 079 086 087 089 092 097 099 100
LC	009

Identification by Mass Spectrometry	laboratory number
yes	002 004 008 009 013 015 018 023 028 029 034 037 048 056 058 060 068 076 079 086 087 089 092 097 099 100

Bupirimate

Method Principle	laboratory number
GC	004 005 009 023 026 027 035 046 048 053 056 060 064 070 072 076 086 089 100
LC	002 008 013 015 018 019 028 029 034 037 039 040 049 050 068 082 083 084 087 088 090 092 097 099

Identification by Mass Spectrometry	laboratory number
no	026
yes	002 004 008 009 013 015 018 019 023 027 028 029 034 035 037 039 040 046 048 049 050 053 056 060 064 068 070 072 076 082 083 084 086 087 088 089 090 092 097 099 100

Cyhalothrin-lambda

Method Principle	laboratory number
GC	002 003 004 005 007 008 009 011 013 014 015 017 018 023 025 026 027 028 029 030 031 032 033 034 035 037 039 042 044 046 048 053 056 057 058 060 064 068 075 076 079 081 083 084 086 087 089 090 092 097 100
LC	019 082 099

Identification by Mass Spectrometry	laboratory number
no	026 081
yes	002 003 004 007 008 009 011 013 014 015 017 018 019 023 025 027 028 029 030 031 032 033 034 035 037 039 042 044 046 048 053 056 057 058 060 064 068 075 076 079 082 083 084 086 087 089 090 092 097 099 100

Fenbuconazole

Method Principle	laboratory number
GC	004 005 027 034 035 046 056 064 086 097 100
LC	002 003 007 008 009 011 013 014 015 017 018 019 023 025 028 029 030 031 032 033 037 039 040 042 043 044 048 049 050 058 060 068 069 070 075 076 079 082 084 087 088 089 092 099

Identification by Mass Spectrometry	laboratory number
yes	002 003 004 007 008 009 011 013 014 015 017 018 019 023 025 027 028 029 030 031 032 033 034 035 037 039 040 042 043 044 046 048 049 050 056 058 060 064 068 069 070 075 076 079 082 084 086 087 088 089 092 097 099 100

Fenhexamid

Method Principle	laboratory number
GC	009 013 028 034 100
LC	002 003 005 007 008 011 014 015 017 018 019 023 025 027 029 030 032 033 037 039 040 042 044 046 048 049 050 056 058 060 064 066 068 069 070 075 076 079 082 086 087 088 089 090 092 097 099

Identification by Mass Spectrometry	laboratory number
yes	002 003 007 008 009 011 013 014 015 017 018 019 023 025 027 028 029 030 032 033 034 037 039 040 042 044 046 048 049 050 056 058 060 064 066 068 069 070 075 076 079 082 086 087 088 089 090 092 097 099 100

Indoxacarb

Method Principle	laboratory number
GC	004 007 011 014 017 025 030 031 032 033 042 057 064 066 075 076 090 097 100
LC	002 003 005 008 009 013 015 018 019 023 027 028 034 037 039 040 046 048 049 056 058 060 068 070 072 079 082 083 084 086 087 088 089 092 099

Identification by Mass Spectrometry	laboratory number
yes	002 003 004 007 009 011 013 014 015 017 018 019 023 025 027 028 030 031 032 033 034 037 039 040 042 046 048 049 056 057 058 060 064 066 068 070 072 075 076 079 082 083 084 086 087 088 089 090 092 097 099 100

Isoprothiolane

Method Principle	laboratory number
GC	002 005 008 009 015 023 027 028 035 037 046 056 058 064 072 079 100
LC	003 007 011 013 014 017 018 019 025 030 031 032 033 042 044 050 060 068 069 070 075 084 086 087 088 089 090 092 097

Identification by Mass Spectrometry	laboratory number
yes	002 003 007 008 009 011 013 014 015 017 018 019 023 025 027 028 030 031 032 033 035 037 042 044 046 050 056 058 060 064 068 069 070 072 075 079 084 086 087 088 089 090 092 097 100

Methamidophos

Method Principle	laboratory number
GC	009 034 100
LC	002 005 008 013 015 018 019 023 027 028 037 039 040 046 049 056 058 060 064 068 072 076 079 084 086 087 088 089 090 092 097 099

Identification by Mass Spectrometry	laboratory number
yes	002 008 009 013 015 018 019 023 027 028 034 037 039 040 046 049 056 058 060 064 068 072 076 079 084 086 087 088 089 090 092 097 099 100

Phosalone

Method Principle	laboratory number
GC	002 003 004 005 007 008 011 014 015 017 018 023 025 026 027 030 031 032 033 034 035 042 043 044 046 053 056 057 058 064 066 070 075 076 079 086 087 097 100
LC	009 013 019 028 029 037 039 048 060 068 082 083 084 088 089 090 092 099

Identification by Mass Spectrometry	laboratory number
no	009 026
yes	002 003 004 007 008 011 013 014 015 017 018 019 023 025 027 028 029 030 031 032 033 034 035 037 039 042 043 044 046 048 053 056 057 058 060 064 066 068 070 075 076 079 082 083 084 086 087 088 089 090 092 097 099 100

Phthalimide

Method Principle	laboratory number
GC	008 015 023 034 037 086 089 092 097

Identification by Mass Spectrometry	laboratory number
yes	008 015 023 034 037 086 089 092 097

Propargite

Method Principle	laboratory number
GC	004 009 018 028 029 035 064 068 076 087 089 090 100
LC	002 005 008 013 015 019 023 027 034 037 039 040 046 048 056 058 060 070 072 079 082 083 084 086 088 092 097 099

Identification by Mass Spectrometry	laboratory number
yes	002 004 008 009 013 015 018 019 023 027 028 029 034 035 037 039 040 046 048 056 058 060 064 068 070 072 076 079 082 083 084 086 087 088 089 090 092 097 099 100

Pyridalyl

Method Principle	laboratory number
GC	003 007 011 014 017 018 025 030 031 032 033 037 042 044 066 070 075 092 097
LC	008 009 013 015 019 023 056 060 076 082 086 087 088 089

Identification by Mass Spectrometry	laboratory number
yes	003 007 008 009 011 013 014 015 017 018 019 023 025 030 031 032 033 037 042 044 056 060 070 075 076 082 086 087 088 089 092 097

Pyridaphenthion

Method Principle	laboratory number
GC	005 008 023 027 029 034 035 046 056 064 076 086 089 092 100

Method Principle (continued)	laboratory number
LC	002 003 007 009 011 013 014 015 017 018 019 025 028 030 031 032 033 037 042 044 060 068 069 070 075 082 083 087 090 097 099

Identification by Mass Spectrometry	laboratory number
yes	002 003 007 008 009 011 013 014 017 018 019 023 025 027 028 029 030 031 032 033 034 035 037 042 044 046 056 060 064 068 069 070 075 076 082 083 086 087 089 090 092 097 099 100

Triadimefon

Method Principle	laboratory number
GC	004 005 008 009 013 023 026 027 029 034 035 046 056 064 070 076 086 090 097 100
LC	002 003 007 011 014 015 017 018 019 025 028 030 031 032 033 037 039 040 042 044 048 049 050 058 060 068 069 072 075 079 082 083 084 087 088 089 092 099

Identification by Mass Spectrometry	laboratory number
no	026
yes	002 003 004 007 008 009 011 013 014 015 017 018 019 023 025 027 028 029 030 031 032 033 034 035 037 039 040 042 044 046 048 049 050 056 058 060 064 068 069 070 072 075 076 079 082 083 084 086 087 088 089 090 092 097 099 100

Tricyclazole

Method Principle	laboratory number
GC	009 035 086
LC	002 003 005 007 008 011 013 014 015 017 018 019 023 025 027 028 030 031 032 033 034 037 039 042 044 046 049 050 056 058 060 066 068 069 070 075 076 079 084 087 088 089 090 092 097 099

Identification by Mass Spectrometry	laboratory number
no	046
yes	002 003 007 008 009 011 013 014 015 017 018 019 023 025 027 028 030 031 032 033 034 035 037 039 042 044 049 050 056 058 060 066 068 069 070 075 076 079 084 086 087 088 089 090 092 097 099

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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