Fapas® – Food Chemistry Proficiency Test Report 19253 – Version 2

Pesticide Residues in Strawberry Purée

August-September 2018

PARTICIPANT LABORATORY NUMBER

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SUMMARY

1. This report has been reissued following an investigation into the behaviour of Etoxazole. The assigned value and z-scores for this analyte are now given *for information only*. All other data remains unchanged.
2. The test materials for Fapas® – Food Chemistry proficiency test 19253 were dispatched in August 2018. Each participant received a strawberry purée test material to be analysed for a selection of pesticide residues.
3. 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 Etoxazole and Spirotetramat (sum spirotetramat and spirotetramat-enol expressed as spirotetramat) are given *for information only.*
4. 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
Aldicarb-sulfoxide	106	35	49	71
Cyhalothrin-lambda	182	42	59	71
Dimethoate	56.7	61	70	87
Endosulfan II (beta)	147	48	58	83
Etoxazole	*57.9*	*34*	58	*59*
Fenpyroximate	116	47	61	77
Monocrotophos	134	49	62	79
Phosphamidon	101	42	53	79
Pirimicarb	46.8	53	62	85
Spirotetramat	*178*	*11*	40	*28*
Triticonazole	97.9	31	45	69

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.

Aldicarb-sulfoxide, Cyhalothrin-lambda, Dimethoate, Endosulfan II (beta), Etoxazole, Fenpyroximate, Monocrotophos, Phosphamidon, Pirimicarb, Spirotetramat and Triticonazole were spiked into the test material.

Samples were stored at -20°C until dispatch.

2.2. Screening and 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 August 2018. Test materials were sent to 80 participants.

3. RESULTS

The instructions for reporting results were as follows:

- Determine the level of pesticide residues present in the test material, in µg/kg, as received, uncorrected for recovery, 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.

- If any residues are reported not in the form specified, please note this in the comments box.
- For residues tested for but not detected, please enter an LoQ via the 'review/resubmit your results' link on your list of tests. This has to be done after you have submitted results for all pesticide residues detected.
- This is an identification and quantification test, therefore if you analyse for a pesticide that is in the test material, and do not identify it, and your limit of quantification is below the level needed for a satisfactory z-score or you do not submit an LoQ, you will be assessed as if your result was zero.

Results were submitted by 75 participants (94%) before the closing date for this test, 27 September 2018.

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 Aldicarb-sulfoxide, Cyhalothrin-lambda, Dimethoate, Endosulfan II (beta), Etoxazole, Fenpyroximate, Monocrotophos, Phosphamidon, Pirimicarb, Spirotetramat and Triticonazole at a level ≥15 µg/kg are listed in Table 5 together with the percentage recovery and LoQ.

Participants' comments are given in Table 6.

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

4. STATISTICAL EVALUATION OF RESULTS

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

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

4.1. Calculation of the Assigned Value, x_a

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

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

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

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

iii) results where no percentage recovery was reported,

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

v) results where no limit of quantification was reported.

For Aldicarb-sulfoxide, Dimethoate, Endosulfan II (beta), Etoxazole, Fenpyroximate, Monocrotophos, Phosphamidon, Pirimicarb and Triticonazole, this procedure was straightforward and the robust mean was chosen as the assigned value. However, an investigation into the behaviour of Etoxazole has revealed that it may be unstable in certain conditions. Consequently, the assigned value now is given *for information only*.

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

For Cyhalothrin-lambda, the mode was chosen as the assigned value because the distribution of results was skewed. See Figure 2.

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. Those for Etoxazole and Spirotetramat 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 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 01/06/2017.

2 GlobalSign PDF Signing Tool, https://www.globalsign.com/en/pdf-signing/ accessed 01/06/2017.

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 Aldicarb-Sulfoxide, Cyhalothrin-Lambda and Dimethoate

laboratory number	analyte											
	Aldicarb-sulfoxide assigned value: 106 µg/kg				Cyhalothrin-lambda assigned value: 182 µg/kg				Dimethoate assigned value: 56.7 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
001	#				#				59.9	97.4	2.5	0.3
002	0			**-4.5**	250		5	1.8	46		10	-0.9
003	#				#				47	82	100	-0.8
004	#				188.8	99	10	0.2	61.7	96	10	0.4
005	#				121	90	10	-1.6	35.2	100	10	-1.7
006	#				#				#			
007	0			**-4.5**	0			**-4.8**	0			**-4.5**
008	#				#				50.2	94.8	10	-0.5
009	#				#				70	93	100	1.1
010	#				45.5	82.0	10.0	**-3.6**	60.88	89.9	10.0	0.3
011	#				197	91.6	10	0.4	53.3	91.6	10	-0.3
012	111.8	105.7	10	0.2	193.3	117.7	10	0.3	68.0	114.8	10	0.9
013	#				#				54	96	30	-0.2
014	97.06	95.54	0.01	-0.4	162.45	91.87	0.01	-0.5	45.91	85.33	0.01	-0.9
015	118.3	100	5	0.5	219.3	100	10	1.0	48.6	100	10	-0.6

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

Table 1 (continued): Results and z-Scores for Aldicarb-Sulfoxide, Cyhalothrin-Lambda and Dimethoate

laboratory number	analyte											
	Aldicarb-sulfoxide assigned value: 106 µg/kg				Cyhalothrin-lambda assigned value: 182 µg/kg				Dimethoate assigned value: 56.7 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
016	0			**-4.5**	0			**-4.8**	66.5678	98.12	11	0.8
017	#				175.2	99	0.5	-0.2	63.3	99	1.0	0.5
018	0			**-4.5**	194.5	91	3.519	0.3	0			**-4.5**
019	#				#				58.5	95.0	20	0.1
020	#				174	98	10	-0.2	63	96	10	0.5
021	#				#				#			
022	0			**-4.5**	0			**-4.8**	0			**-4.5**
023	0			**-4.5**	0			**-4.8**	0			**-4.5**
024	#				173.0	99	5	-0.2	65.3	100	5	0.7
025	113.9	91	10	0.3	153.2	85	10	-0.8	58.1	96	10	0.1
026	0			**-4.5**	248		10	1.8	77		10	1.6
027	#				#				62	98	20	0.4
028	101		10	-0.2	160		10	-0.6	56		10	-0.1
029	97			-0.4	#				62			0.4
030	99.8	100	10	-0.3	188	100	10	0.2	55	100	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 Aldicarb-Sulfoxide, Cyhalothrin-Lambda and Dimethoate

laboratory number	analyte											
	Aldicarb-sulfoxide assigned value: 106 µg/kg				Cyhalothrin-lambda assigned value: 182 µg/kg				Dimethoate assigned value: 56.7 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
031	124.0	100	10	0.8	178.6	110	10	-0.1	67.0	98	10	0.8
032	#				#				#			
033	125.3	100	10	0.8	215.3	100	10	0.9	66.3	100	10	0.8
034	89	90	10	-0.7	163	100	10	-0.5	44	100	10	-1.0
035	116	94	10	0.4	155	100	30	-0.7	56	103	10	-0.1
036	#				347	112	10	**4.4**	60.26	110	2	0.3
037	91	94	10	-0.6	168	95	10	-0.4	47	89	10	-0.8
038	0			**-4.5**	Detected	NA	Qualitative		0			**-4.5**
039	132	84	30	1.1	163	63	30	-0.5	54	88	30	-0.2
040	#				#				43.15	72.00	32.51	-1.1
041	0			**-4.5**	0			**-4.8**	53	96.2	10	-0.3
042	63	86	10	-1.8	185	119	10	0.1	50	97	10	-0.5
043	#				188	98.7	2	0.2	64	97.6	3	0.6
044	0		20	**-4.5**	242	109	10	1.6	65.2	93	10	0.7
045	#				36	114	10	**-3.9**	#			

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 Aldicarb-Sulfoxide, Cyhalothrin-Lambda and Dimethoate

laboratory number	analyte											
	Aldicarb-sulfoxide assigned value: 106 µg/kg				Cyhalothrin-lambda assigned value: 182 µg/kg				Dimethoate assigned value: 56.7 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
046	119.37	98.5	10	0.6	198.93	77.1	10	0.5	63.00	92.8	10	0.5
047	#				#				64.70	111	5	0.6
048	108.0	95	10	0.1	184.8	88	5	0.1	51.8	95	1	-0.4
049	#				142.20		30	-1.1	0			**-4.5**
050	125	100	10	0.8	190	100	10	0.2	53	100	10	-0.3
051	96	140		-0.4	212	120		0.8	53	72		-0.3
052	115.2	73.75	10	0.4	139.8	75.75	10	-1.1	79.8	74.25	10	1.9
053	125	98	10	0.8	103	95	10	**-2.1**	75	96	10	1.5
054	360	100.130	5	**10.9**	#				#			
055	123.2			0.7	221.3			1.0	57.6			0.1
056	104	91	10	-0.1	199	133	10	0.5	54	97	10	-0.2
057	72.77	80	10	-1.4	183.24	80	10	0.0	36.07	80	10	-1.7
058	142		10	1.5	207		10	0.7	69		10	1.0
059	#				132		10	-1.3	63		10	0.5
060	108	84	10	0.1	101	115	10	**-2.2**	68	98	10	0.9

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 Aldicarb-Sulfoxide, Cyhalothrin-Lambda and Dimethoate

laboratory number	analyte											
	Aldicarb-sulfoxide assigned value: 106 µg/kg				Cyhalothrin-lambda assigned value: 182 µg/kg				Dimethoate assigned value: 56.7 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
061	98	87	10	-0.3	185	84	10	0.1	49	86	10	-0.6
062	91		20	-0.6	266		10	**2.2**	60		10	0.3
063	65	100	10	-1.8	125	100	10	-1.5	35	100	10	-1.7
064	135	108	10	1.2	183	94	10	0.0	620	105	10	**45.2**
065	#				87.8	69	20	**-2.5**	46.8	82	10	-0.8
066	0			**-4.5**	0			**-4.8**	0			**-4.5**
067	99	77	10	-0.3	177	105	10	-0.1	56	83	10	-0.1
068	0			**-4.5**	202	101	10	0.5	58	94	10	0.1
069	80	78.9	10	-1.1	100	74.5	10	**-2.2**	50	81.95	10	-0.5
070	89	84	10	-0.7	355	73.5	10	**4.6**	58	88	10	0.1
071	#				#				63.5320	89.70	10	0.5
072	123	100	10	0.7	91	100	10	**-2.4**	62	100	10	0.4
073	0			**-4.5**	0			**-4.8**	0			**-4.5**
074	72.83	99.15	10	-1.4	220.14	96.55	10	1.0	46.42	99.60	10	-0.8
075	118			0.5	132			-1.3	51			-0.5

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 II, Etoxazole and Fenpyroximate

laboratory number	analyte											
	Endosulfan II (beta) assigned value: 147 µg/kg				Etoxazole assigned value: *57.9* µg/kg				Fenpyroximate assigned value: 116 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
001	#				#				125.4	97.3	2.5	0.4
002	150		5	0.1	47		10	*-0.9*	83		10	-1.3
003	#				11	88	30	*-3.7*	#			
004	162.3	105	10	0.5	62.0	94	10	*0.3*	#			
005	#				0			*-4.5*	57.1	92	10	**-2.3**
006	#				#				#			
007	0			**-4.7**	0			*-4.5*	0			**-4.5**
008	130	102.2	10	-0.5	#				#			
009	#				19	110	30	*-3.1*	#			
010	#				36.18	85.8	10.0	*-1.7*	117.3	91.3	10.0	0.1
011	154	99.0	10	0.2	29.9	109.8	10	*-2.2*	113	106	10	-0.1
012	127.1	109.4	10	-0.6	11.3	112.7	10	*-3.7*	121.9	103.9	10	0.2
013	#				14	106	10	*-3.4*	#			
014	125.13	103.29	0.01	-0.7	59.13	86.34	0.01	*0.1*	103.60	87.87	0.01	-0.5
015	181.5	100	10	1.1	#				#			

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 2 (continued): Results and z-Scores for Endosulfan II, Etoxazole and Fenpyroximate

laboratory number	analyte											
	Endosulfan II (beta) assigned value: 147 µg/kg				Etoxazole assigned value: *57.9* µg/kg				Fenpyroximate assigned value: 116 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
016	0			**-4.7**	43.6631	98.65	6	*-1.1*	106.5753	100.15	7	-0.4
017	151	100	0.5	0.1	47.4	99	0.5	*-0.8*	112	100	0.5	-0.1
018	0			**-4.7**	0			*-4.5*	0			**-4.5**
019	#				#				#			
020	153	96	10	0.2	49	96	10	*-0.7*	112	99	10	-0.1
021	#				#				#			
022	0			**-4.7**	0			*-4.5*	0			**-4.5**
023	145.7	95	5	0.0	0			*-4.5*	0			**-4.5**
024	151.1	96	5	0.1	48.9	98	5	*-0.7*	109.8	96	5	-0.2
025	103.6	82	10	-1.4	73.9	95	10	*1.3*	108.8	98	10	-0.3
026	172		10	0.8	90		10	*2.5*	127		10	0.4
027	160	110	10	0.4	74	73	10	*1.3*	130	108	10	0.6
028	191		10	1.4	87		10	*2.3*	149		10	1.3
029	128			-0.6	50			*-0.6*	131			0.6
030	175	99	10	0.9	#				114	100	10	-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 2 (continued): Results and z-Scores for Endosulfan II, Etoxazole and Fenpyroximate

laboratory number	analyte											
	Endosulfan II (beta) assigned value: 147 µg/kg				Etoxazole assigned value: *57.9* µg/kg				Fenpyroximate assigned value: 116 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
031	176.3	75	10	0.9	63.9	72	10	*0.5*	142.8	83	10	1.1
032	#				#				#			
033	157.0	100	10	0.3	88.4	100	10	*2.4*	145.6	100	10	1.2
034	146	80	10	0.0	42	70	10	*-1.3*	98	90	10	-0.7
035	168	95	10	0.7	84	101	10	*2.0*	114	100	10	-0.1
036	#				247	153	*2*	*14.8*	119.83	108	*2*	0.2
037	163	96	10	0.5	69	109	10	*0.9*	104	106	10	-0.5
038	Detected	NA	Qualitative		0			*-4.5*	0			**-4.5**
039	168	64	30	0.7	<LOQ		30		141	98	30	1.0
040	#				65.89	104.00	0.70	*0.6*	128.51	104.00	7.91	0.5
041	0			**-4.7**	0			*-4.5*	38.5	25.6	10	**-3.0**
042	130	87	10	-0.5	46	107	10	*-0.9*	119	110	10	0.1
043	159	100.9	4	0.4	44	100.4	2	*-1.1*	112	96.7	0.6	-0.1
044	270	106	10	**3.9**	#				103	92	10	-0.5
045	92	99	10	-1.8	75	109	10	*1.3*	45	37	10	**-2.8**

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 2 (continued): Results and z-Scores for Endosulfan II, Etoxazole and Fenpyroximate

laboratory number	analyte											
	Endosulfan II (beta) assigned value: 147 µg/kg				Etoxazole assigned value: *57.9* µg/kg				Fenpyroximate assigned value: 116 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
046	159.12	79.5	10	0.4	45.40	103.6	10	*-1.0*	127.40	92.4	10	0.5
047	#				#				#			
048	149.1	102	5	0.1	99.6	98	5	*3.3*	123.5	100	5	0.3
049	0			**-4.7**	#				84.71		30	-1.2
050	195	100	10	1.5	54	100	10	*-0.3*	122	100	10	0.2
051	175	102		0.9	79	101		*1.7*	119	104		0.1
052	114.2	77.75	10	-1.0	36.0	111.50	10	*-1.7*	80.6	115.75	10	-1.4
053	79	94	10	**-2.2**	144	87	10	*6.8*	162	89	10	1.8
054	#				#				#			
055	139.5			-0.2	78.8			*1.6*	145.5			1.2
056	168	73	10	0.7	81	115	10	*1.8*	115	98	10	0.0
057	#				65.30	80	10	*0.6*	119.21	80	10	0.1
058	184		10	1.2	65		10	*0.6*	134		10	0.7
059	134		10	-0.4	68		10	*0.8*	0			**-4.5**
060	162	107	10	0.5	96	96	10	*3.0*	146	107	10	1.2

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 2 (continued): Results and z-Scores for Endosulfan II, Etoxazole and Fenpyroximate

laboratory number	analyte											
	Endosulfan II (beta) assigned value: 147 µg/kg				Etoxazole assigned value: *57.9* µg/kg				Fenpyroximate assigned value: 116 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
061	162	95	10	0.5	70	87	10	*0.9*	105	93	10	-0.4
062	155		10	0.3	58		10	*0.0*	121		10	0.2
063	100	100	10	-1.5	20	100	10	*-3.0*	55	100	10	**-2.4**
064	125	93	10	-0.7	540	87	10	*37.8*	460	106	10	**13.5**
065	115.3	73	10	-1.0	#				85.2	122	10	-1.2
066	178.1	88	10	1.0	0			*-4.5*	0			**-4.5**
067	149	95	10	0.1	#				#			
068	163	98	10	0.5	64	78	10	*0.5*	123	93	10	0.3
069	30	84.8	20	**-3.7**	10	76.1	10	*-3.8*	70	68.15	10	-1.8
070	124	102	10	-0.7	#				95	82	10	-0.8
071	#				#				#			
072	144	100	10	-0.1	62	100	10	*0.3*	125	100	10	0.4
073	0			**-4.7**	0			*-4.5*	0			**-4.5**
074	128.11	99.80	10	-0.6	42.21	99.10	10	*-1.2*	122.49	100.65	10	0.3
075	100.7			-1.5	34			*-1.9*	179			**2.5**

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 3: Results and z-Scores for Monocrotophos, Phosphamidon and Pirimicarb

laboratory number	analyte											
	Monocrotophos assigned value: 134 µg/kg				Phosphamidon assigned value: 101 µg/kg				Pirimicarb assigned value: 46.8 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
001	#				#				47.8	95.4	2.5	0.1
002	0			**-4.6**	0			**-4.5**	75		5	**2.7**
003	#				#				#			
004	172.2	96	10	1.3	#				49.7	101	10	0.3
005	#				#				#			
006	#				#				#			
007	0			**-4.6**	0			**-4.5**	0			**-4.5**
008	#				102.8	96.4	10	0.1	44.8	94.4	10	-0.2
009	#				#				#			
010	154.85	96.5	10.0	0.7	#				28.65	92.4	10.0	-1.8
011	133	111.6	10	0.0	#				47.0	87.7	10	0.0
012	133.1	98.7	10	0.0	137.5	123.1	10	1.6	50.1	107.4	10	0.3
013	#				#				#			
014	116.36	89.36	0.01	-0.6	69.11	94.67	0.01	-1.4	17.47	84.30	0.01	**-2.8**
015	149.9	100	10	0.5	171.7	100	10	**3.2**	51.0	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 Monocrotophos, Phosphamidon and Pirimicarb

laboratory number	analyte											
	Monocrotophos assigned value: 134 µg/kg				Phosphamidon assigned value: 101 µg/kg				Pirimicarb assigned value: 46.8 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
016	0			**-4.6**	102.2699	92.02	11	0.0	47.9391	94.49	8	0.1
017	133	97	0.5	0.0	104.2	96	0.5	0.1	44.7	99	0.5	-0.2
018	0			**-4.6**	0			**-4.5**	0			**-4.5**
019	#				#				#			
020	133	96	10	0.0	110	96	10	0.4	45	99	10	-0.2
021	129.9	98.5	5	-0.1	#				#			
022	0			**-4.6**	96.7	95	10	-0.2	49.5	95	10	0.3
023	0			**-4.6**	0			**-4.5**	0			**-4.5**
024	136.0	95	5	0.1	111.1	94	5	0.4	46.0	97		-0.1
025	137.4	92	10	0.1	96.3	75	10	-0.2	44.2	93	10	-0.2
026	137		10	0.1	121		10	0.9	50		10	0.3
027	123	125	10	-0.4	109	78	10	0.3	47	88	10	0.0
028	131		10	-0.1	101		10	0.0	42		10	-0.5
029	132			-0.1	#				40			-0.7
030	125	100	10	-0.3	112.7	98	10	0.5	44	99	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 Monocrotophos, Phosphamidon and Pirimicarb

laboratory number	analyte											
	Monocrotophos assigned value: 134 µg/kg				Phosphamidon assigned value: 101 µg/kg				Pirimicarb assigned value: 46.8 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
031	145.8	99	10	0.4	110.4	65	10	0.4	52.0	98	10	0.5
032	125.1	103.1	0.2	-0.3	#				#			
033	142.8	100	10	0.3	106.1	100	10	0.2	49.9	100	10	0.3
034	118	80	10	-0.6	102	100	10	0.0	39	90	10	-0.8
035	149	97	10	0.5	97	103	10	-0.2	51	103	10	0.4
036	#				#				#			
037	112	97	10	-0.8	#				41	96	10	-0.6
038	0			**-4.6**	0			**-4.5**	Detected	NA	Qualitative	
039	0		30	**-4.6**	91	88	30	-0.5	51	92	30	0.4
040	134.61	98.00	24.75	0.0	101.96	99.33	0.15	0.0	49.94	103.33	0.58	0.3
041	0			**-4.6**	99.9	114.2	10	-0.1	51.8	109.6	10	0.5
042	119	70	10	-0.5	92	110	10	-0.4	46	98	10	-0.1
043	136	104.6	4	0.1	114	98.5	3	0.6	45	95.4	3	-0.2
044	130	114	10	-0.1	#				33.9	117	10	-1.3
045	#				0			**-4.5**	49	52	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 Monocrotophos, Phosphamidon and Pirimicarb

laboratory number	analyte											
	Monocrotophos assigned value: 134 µg/kg				Phosphamidon assigned value: 101 µg/kg				Pirimicarb assigned value: 46.8 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
046	142.05	90.2	10	0.3	102.36	93.9	10	0.1	51.12	96.7	10	0.4
047	120.70	92	5	-0.5	95.55	110	5	-0.3	#			
048	161.9	100	5	1.0	66.7	112	5	-1.6	46.1	90	5	-0.1
049	154.15		30	0.7	79.02		30	-1.0	37.90		30	-0.9
050	150	100	10	0.6	99	100	10	-0.1	51	100	10	0.4
051	149	109		0.5	80	85		-1.0	49	85		0.2
052	129.4	74.75	10	-0.2	67.6	93.75	10	-1.5	#			
053	116	88	10	-0.6	167	93	10	**3.0**	102	98	10	**5.4**
054	#				#				56	100.130	5	0.9
055	132.3			-0.1	90.2			-0.5	51.5			0.5
056	142	95	10	0.3	97	99	10	-0.2	47	97	10	0.0
057	62.33	80	10	**-2.5**	103.34	80	10	0.1	37.80	80	10	-0.9
058	170		10	1.2	104		10	0.1	58		10	1.1
059	#				#				52		10	0.5
060	145	90	10	0.4	103	120	10	0.1	60	119	10	1.3

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 Monocrotophos, Phosphamidon and Pirimicarb

laboratory number	analyte											
	Monocrotophos assigned value: 134 µg/kg				Phosphamidon assigned value: 101 µg/kg				Pirimicarb assigned value: 46.8 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
061	137	102	10	0.1	115	109	10	0.6	51	121	10	0.4
062	#				#				44		10	-0.3
063	95	100	10	-1.3	60	100	10	-1.9	40	100	10	-0.7
064	210	89	10	**2.6**	130	94	10	1.3	640	105	10	**57.7**
065	110.0	88	10	-0.8	75.9	82	10	-1.1	43.6	82	10	-0.3
066	0			**-4.6**	0			**-4.5**	0			**-4.5**
067	123	80	10	-0.4	#				43	100	10	-0.4
068	146	79	10	0.4	96	96	10	-0.2	55	98	10	0.8
069	120	97.05	10	-0.5	70	91.35	10	-1.4	30	87.8	10	-1.6
070	117	85	10	-0.6	0			**-4.5**	41	92	10	-0.6
071	147.9820	104.42		0.5	#				#			
072	157	100	10	0.8	#				48	100	10	0.1
073	0			**-4.6**	230			**5.8**	0			**-4.5**
074	173.93	101.7	10	1.4	107.46	97.5	10	0.3	48.24	100.25	10	0.1
075	138			0.1	91			-0.5	45			-0.2

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 Spirotetramat and Triticonazole

laboratory number	analyte							
	Spirotetramat assigned value: *178* µg/kg				Triticonazole assigned value: 97.9 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
001	0	96.1	2.5	*-4.8*	#			
002	417		10	*6.5*	79		10	-0.9
003	#				#			
004	0			*-4.8*	95.5	106	10	-0.1
005	#				62.4	96	10	-1.6
006	#				#			
007	0			*-4.8*	0			**-4.5**
008	#				#			
009	#				#			
010	#				90.02	86.3	10.0	-0.4
011	226	82.6	10	*1.3*	#			
012	#				109.1	76.2	10	0.5
013	#				#			
014	#				80.90	74.18	0.01	-0.8
015	#				140.1	100	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 Spirotetramat and Triticonazole

laboratory number	analyte							
	Spirotetramat assigned value: *178* µg/kg				Triticonazole assigned value: 97.9 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
016	0			*-4.8*	94.2384	100.18	7	-0.2
017	#				#			
018	0			*-4.8*	0			**-4.5**
019	#				#			
020	#				#			
021	#				#			
022	0			*-4.8*	0			**-4.5**
023	0			*-4.8*	0			**-4.5**
024	#				#			
025	174.0	99	10	*-0.1*	101.4	107	10	0.2
026	0			*-4.8*	0			**-4.5**
027	#				88	98	10	-0.5
028	0		10	*-4.8*	112		10	0.7
029	#				#			
030	#				#			

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 Spirotetramat and Triticonazole

laboratory number	analyte							
	Spirotetramat assigned value: *178* µg/kg				Triticonazole assigned value: 97.9 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
031	205.5	115	10	*0.7*	96.3	80	10	-0.1
032	#				#			
033	0	100	10	*-4.8*	#			
034	182	90	10	*0.1*	#			
035	126	80	10	*-1.4*	108	99	10	0.5
036	#				0			**-4.5**
037	0	104	10	*-4.8*	105	103	10	0.3
038	0			*-4.8*	0			**-4.5**
039	0		30	*-4.8*	116	104	30	0.8
040	#				#			
041	0			*-4.8*	0			**-4.5**
042	256	120	10	*2.1*	98	112	10	0.0
043	#				#			
044	#				#			
045	#				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 4 (continued): Results and z-Scores for Spirotetramat and Triticonazole

laboratory number	analyte							
	Spirotetramat assigned value: *178* µg/kg				Triticonazole assigned value: 97.9 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
046	0		10	*-4.8*	104.27	94.2	10	0.3
047	#				#			
048	129.5	72	5	*-1.3*	135.2	105	10	1.7
049	104.51		30	*-2.0*	40.58		30	**-2.7**
050	183	100	10	*0.1*	101	100	10	0.1
051	#				108	104		0.5
052	0		10	*-4.8*	#			
053	0			*-4.8*	41	93	10	**-2.6**
054	#				#			
055	#				#			
056	#				97	97	10	0.0
057	#				92.01	80	10	-0.3
058	538		10	*9.8*	126		10	1.3
059	#				#			
060	162	71	10	*-0.4*	100	88	10	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 Spirotetramat and Triticonazole

laboratory number	analyte							
	Spirotetramat assigned value: *178* µg/kg				Triticonazole assigned value: 97.9 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
061	#				90	102	10	-0.4
062	73		10	*-2.8*	#			
063	50	100	10	*-3.5*	55	100	10	-2.0
064	756	92	10	*15.7*	175	107	10	**3.6**
065	#				#			
066	0			*-4.8*	0			**-4.5**
067	#				#			
068	156	95	10	*-0.6*	124	86	10	1.2
069	360	76.4	10	*4.9*	74	82.65	10	-1.1
070	92	86	10	*-2.3*	84	94	10	-0.6
071	#				#			
072	172	100	10	*-0.2*	93	100	10	-0.2
073	0			*-4.8*	0			**-4.5**
074	313.93	98.2	10	*3.7*	110.87	98.05	10	0.6
075	0			*-4.8*	#			

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: Additional Pesticide Residues Reported

laboratory number	pesticide residue ≥15 µg/kg	result µg/kg	recovery %	LoQ µg/kg
002	Endrin	44		5
011	Endrin	31.4	91.5	10
014	Endrin	121.99	90.22	0.01
026	Aldicarb-sulfone (aldoxycarb)	140		10
036	Metconazole	108.26	98	2
037	Endrin	26	98	10
045	Chloridazon	72	30	10
045	Metconazole	15	57	10
053	Acrinathrin	103	110	10
053	fenpropidin (sum of fenpropidin and its salts, expressed as fenpropidin)	171	86	10
054	Iprovalicarb	79	100-130	5 µg/kg
054	Phosmet	29	100-130	5 µg/kg
059	Diuron	75		10

Table 6: Participants' Comments

laboratory number	comments
011	% RECOVERY FROM VALIDATION METHOD
028	Sample ID = 18-006645-001
036	All reported results are before recovery correction.
038	This is a qualitative screen (NDA-RESD-208) for pesticides and toxins.
050	Endosulfan I was detected in the sample below LoQ
060	Fluazifop: free acid only
063	100% recovery because we made standard addition
064	We found also traces of Pentachloranilin and Myclobutanil
068	The sample has arrived to the lab not frozen.
070	SE RECIBE LA MUESTRA Y BLANCO VISIBLEMENTE FERMENTADA Y CON DERRAME, LO CUAL SE EVIDENCIA CON EL ENVASE HINCHADO

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	
Aldicarb-sulfoxide	29	106	4	Horwitz [9]	23.3
Cyhalothrin-lambda	42	182	3	Horwitz [9]	37.6
Dimethoate	49	56.7	1.3	Horwitz [9]	12.5
Endosulfan II (beta)	41	147	4	Horwitz [9]	31.4
Etoxazole	36	*57.9*	*4.4*	Horwitz [9]	*12.7*
Fenpyroximate	41	116	3	Horwitz [9]	25.5
Monocrotophos	42	134	3	Horwitz [9]	29.0
Phosphamidon	37	101	2	Horwitz [9]	22.3
Pirimicarb	43	46.8	0.9	Horwitz [9]	10.3
Spirotetramat	16	*178*	*18*	Horwitz [9]	*36.9*
Triticonazole	29	97.9	3.5	Horwitz [9]	21.5

italics indicate for information only

Table 8: Number and Percentage of z-Scores where |z| ≤2

analyte	number of scores where |z| ≤2	total number of scores	% |z|≤2
Aldicarb-sulfoxide	35	49	71
Cyhalothrin-lambda	42	59	71
Dimethoate	61	70	87
Endosulfan II (beta)	48	58	83
Etoxazole	*34*	58	*59*
Fenpyroximate	47	61	77
Monocrotophos	49	62	79
Phosphamidon	42	53	79
Pirimicarb	53	62	85
Spirotetramat	*11*	40	*28*
Triticonazole	31	45	69

italics indicate for information only

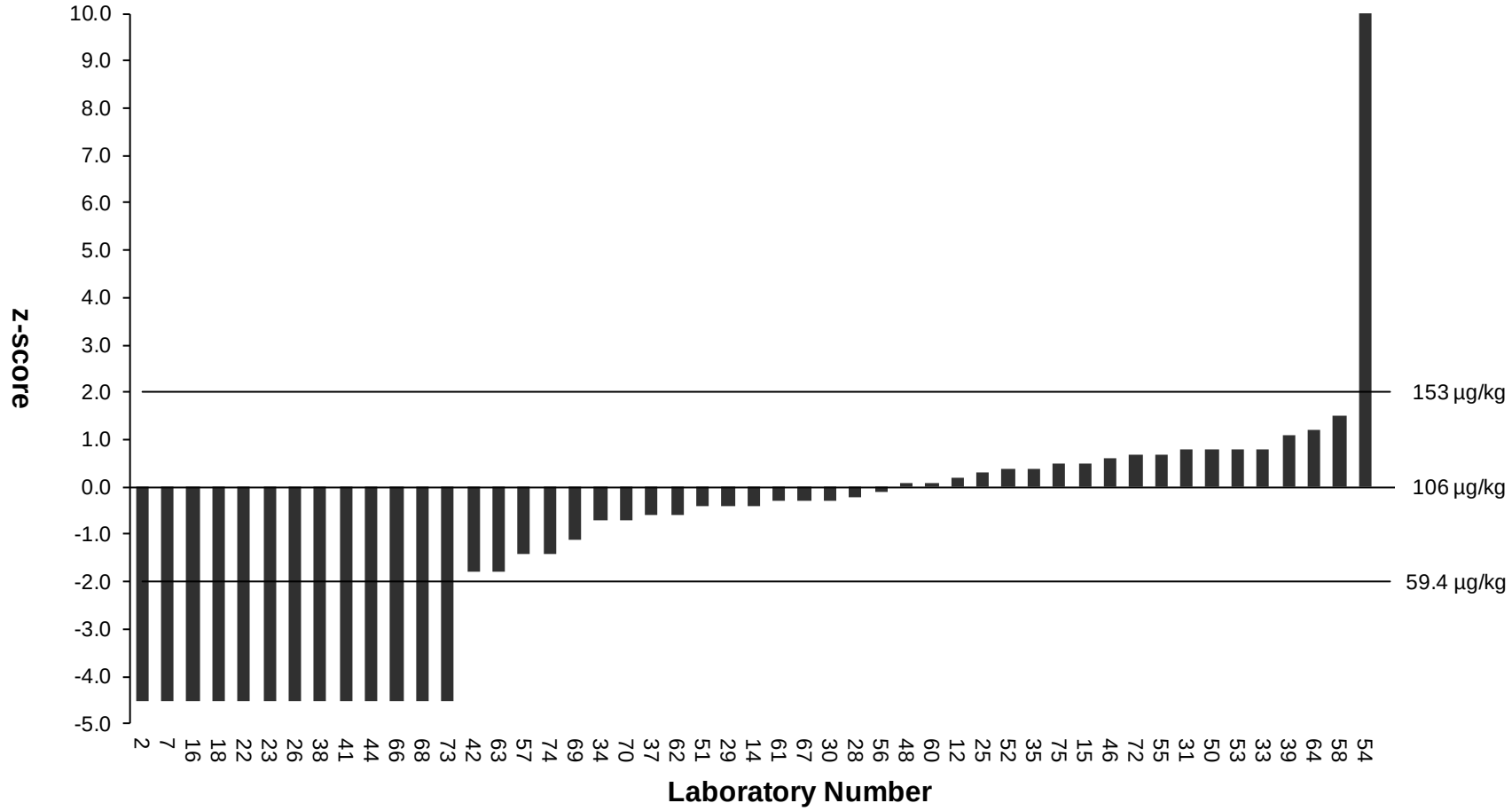

Figure 1: z-Scores for Aldicarb-sulfoxide

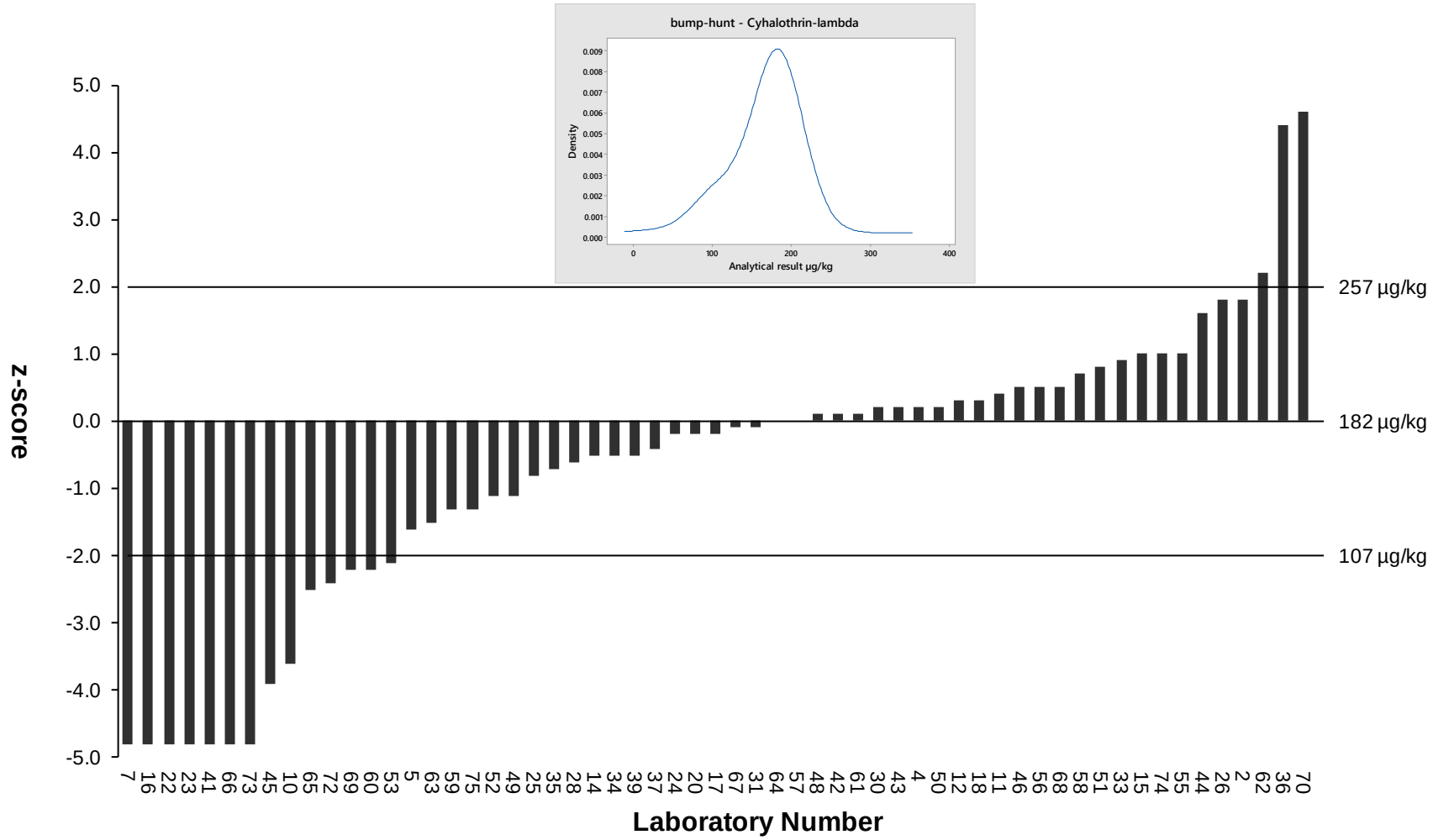

Figure 2: z-Scores for Cyhalothrin-lambda

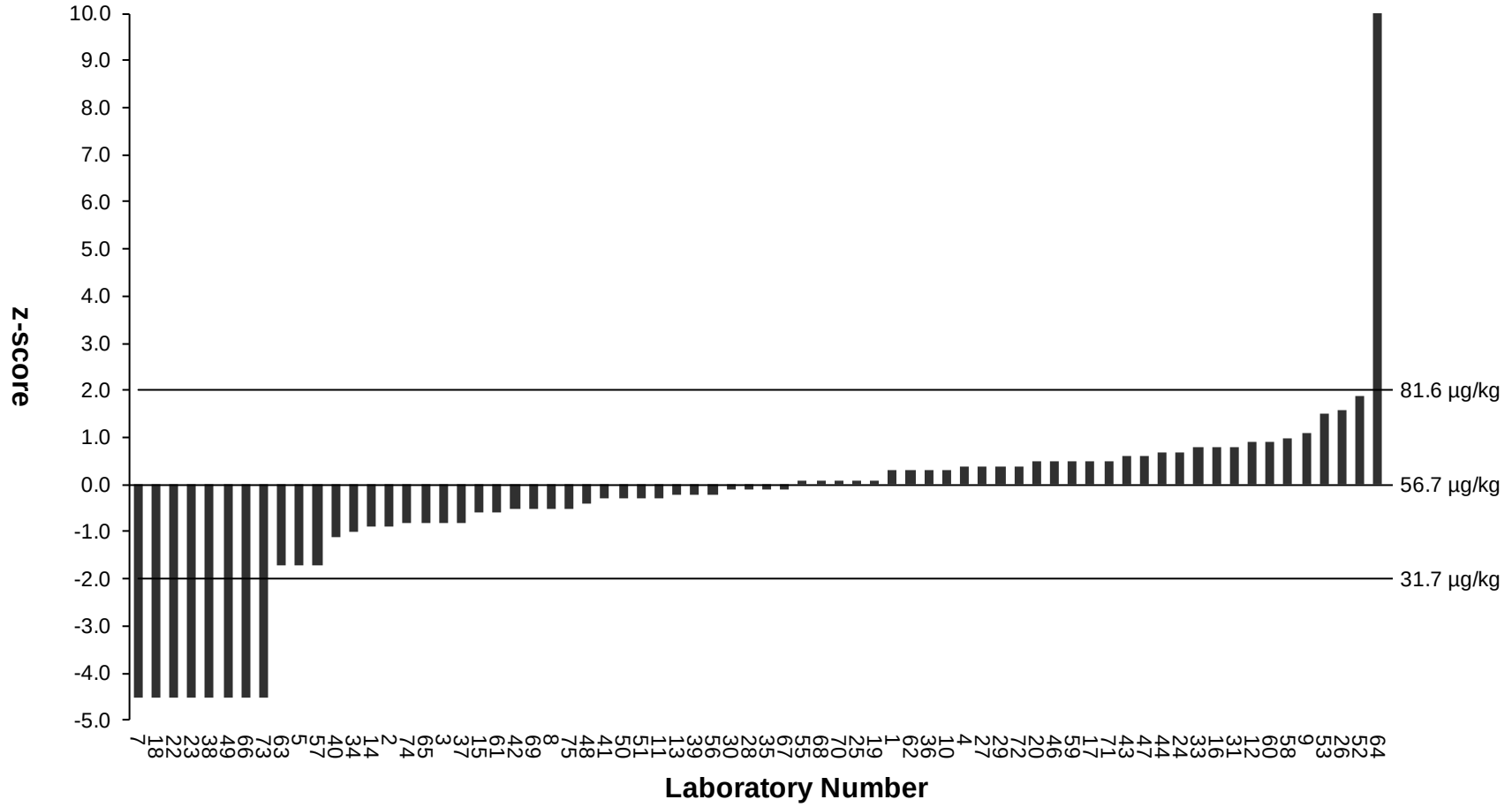

Figure 3: z-Scores for Dimethoate

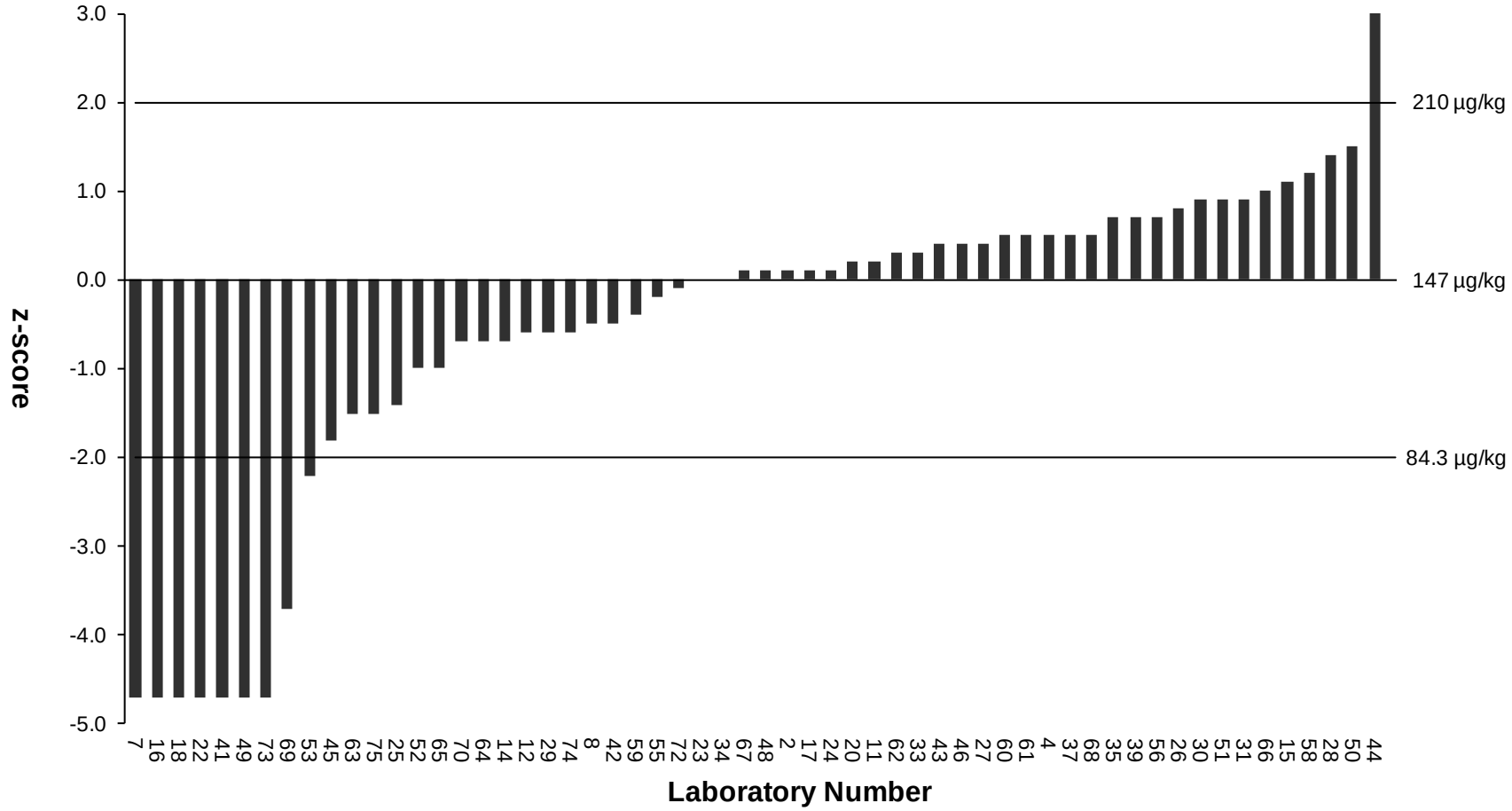

Figure 4: z-Scores for Endosulfan II (beta)

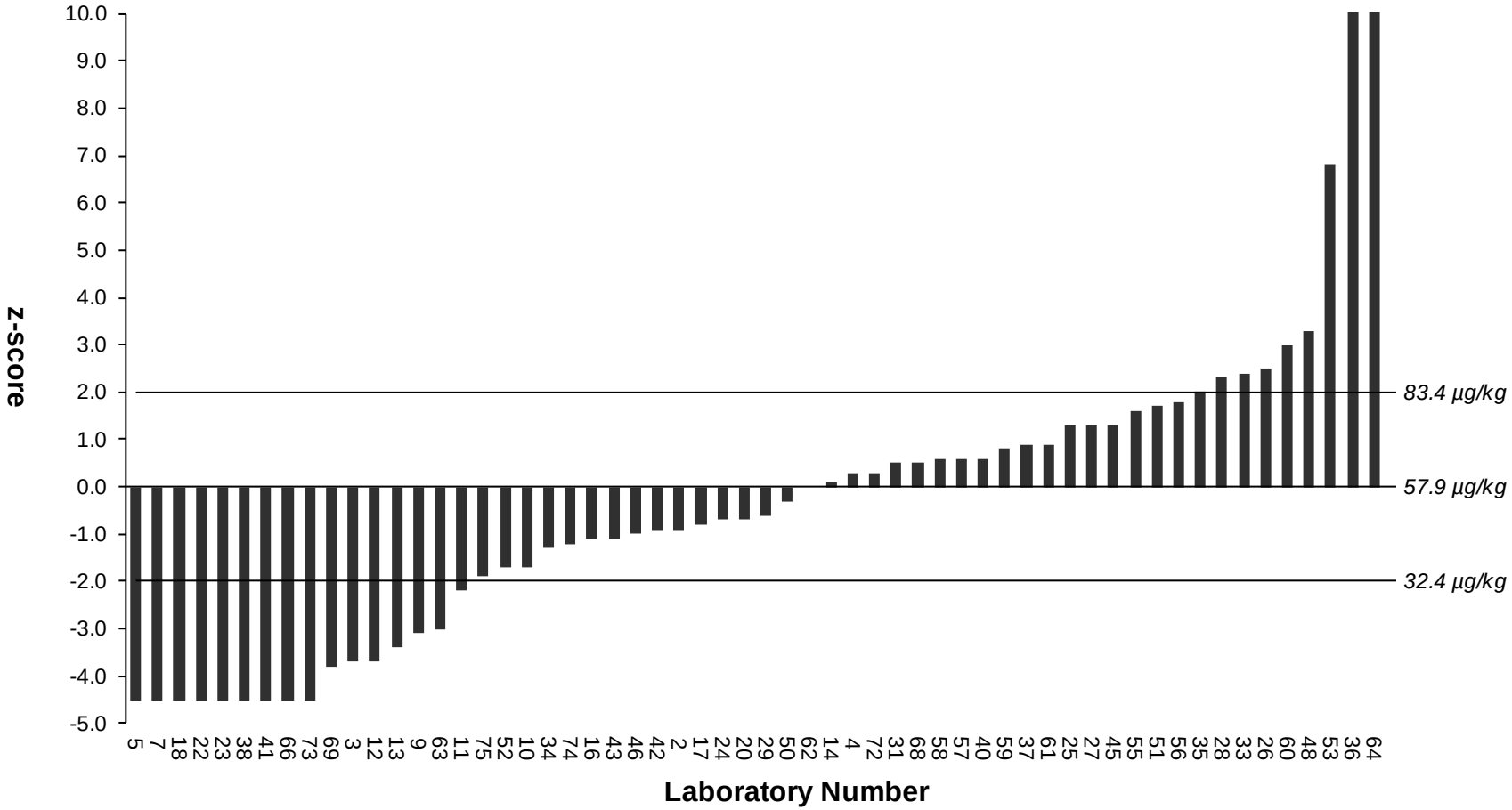

Figure 5: z-Scores for Etoxazole

this histogram is given for information only

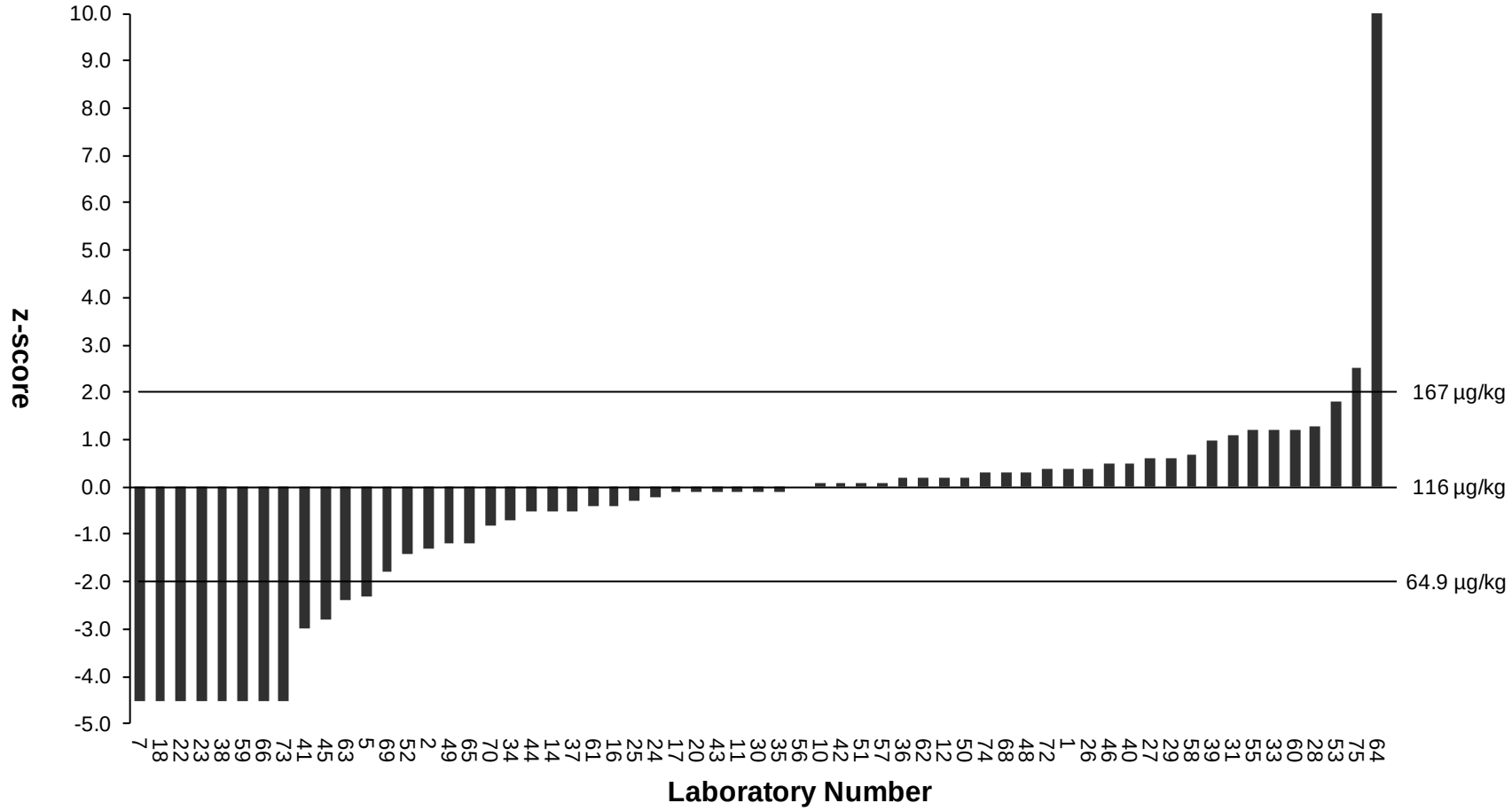

Figure 6: z-Scores for Fenpyroximate

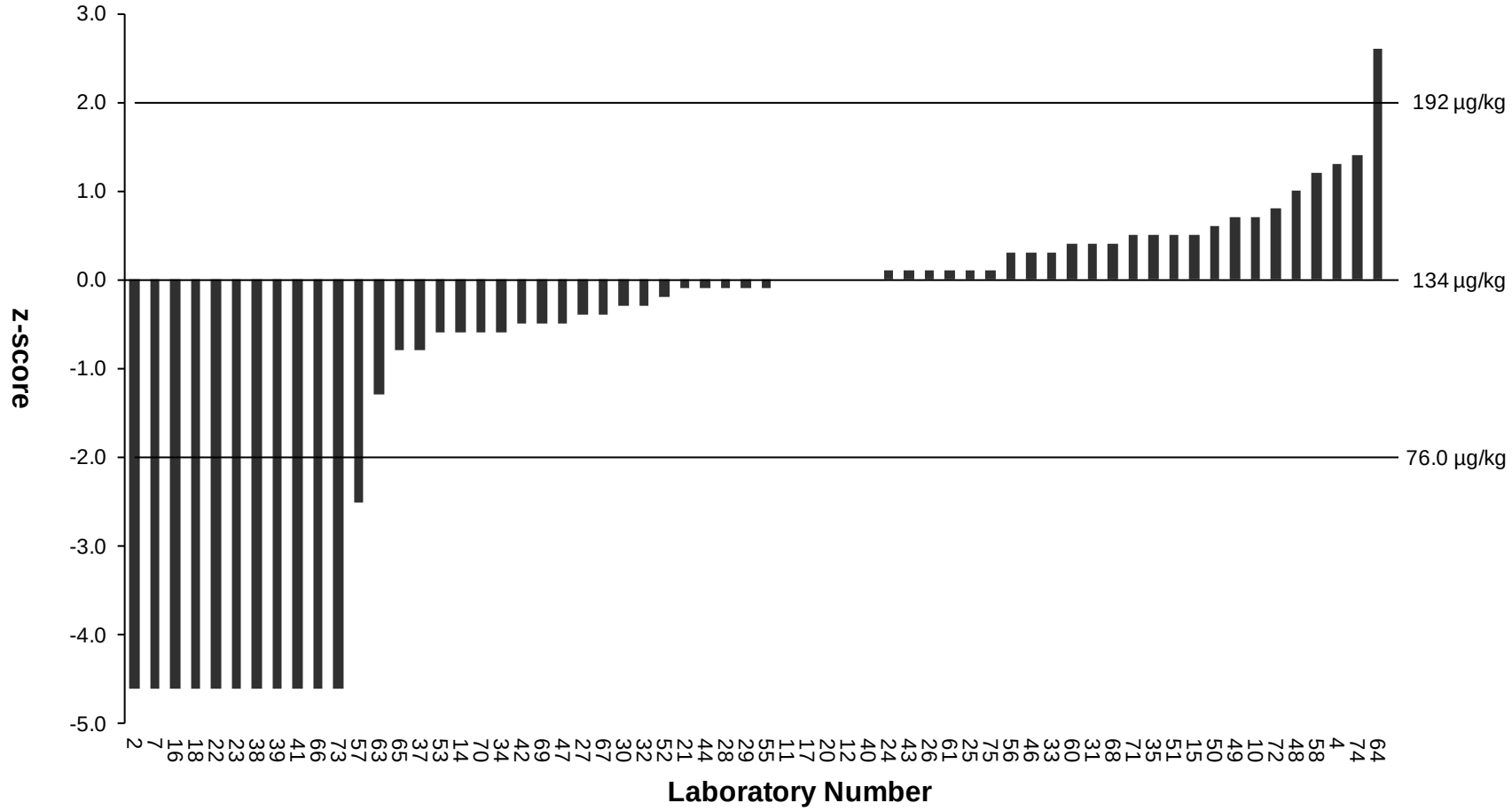

Figure 7: z-Scores for Monocrotophos

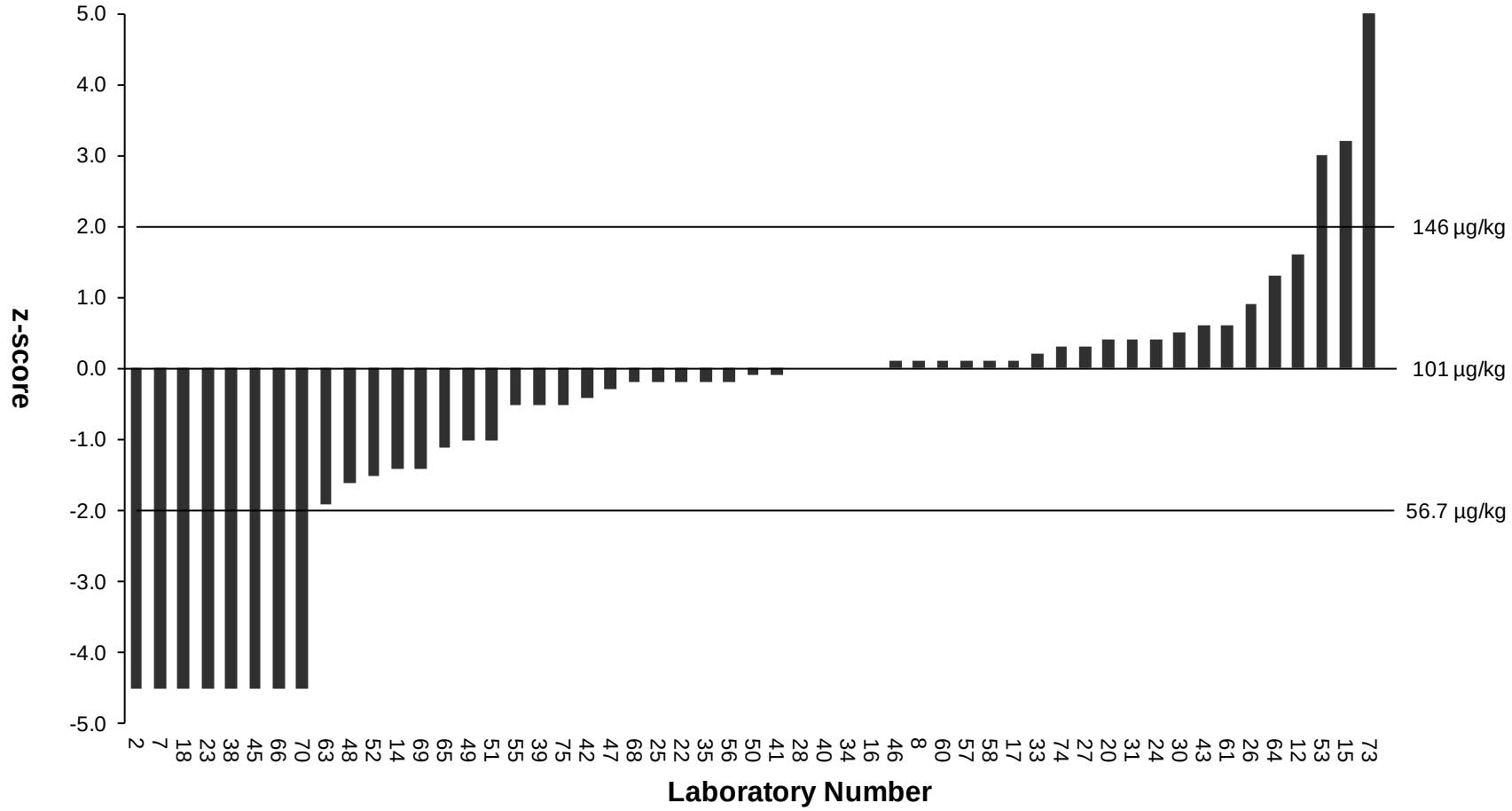

Figure 8: z-Scores for Phosphamidon

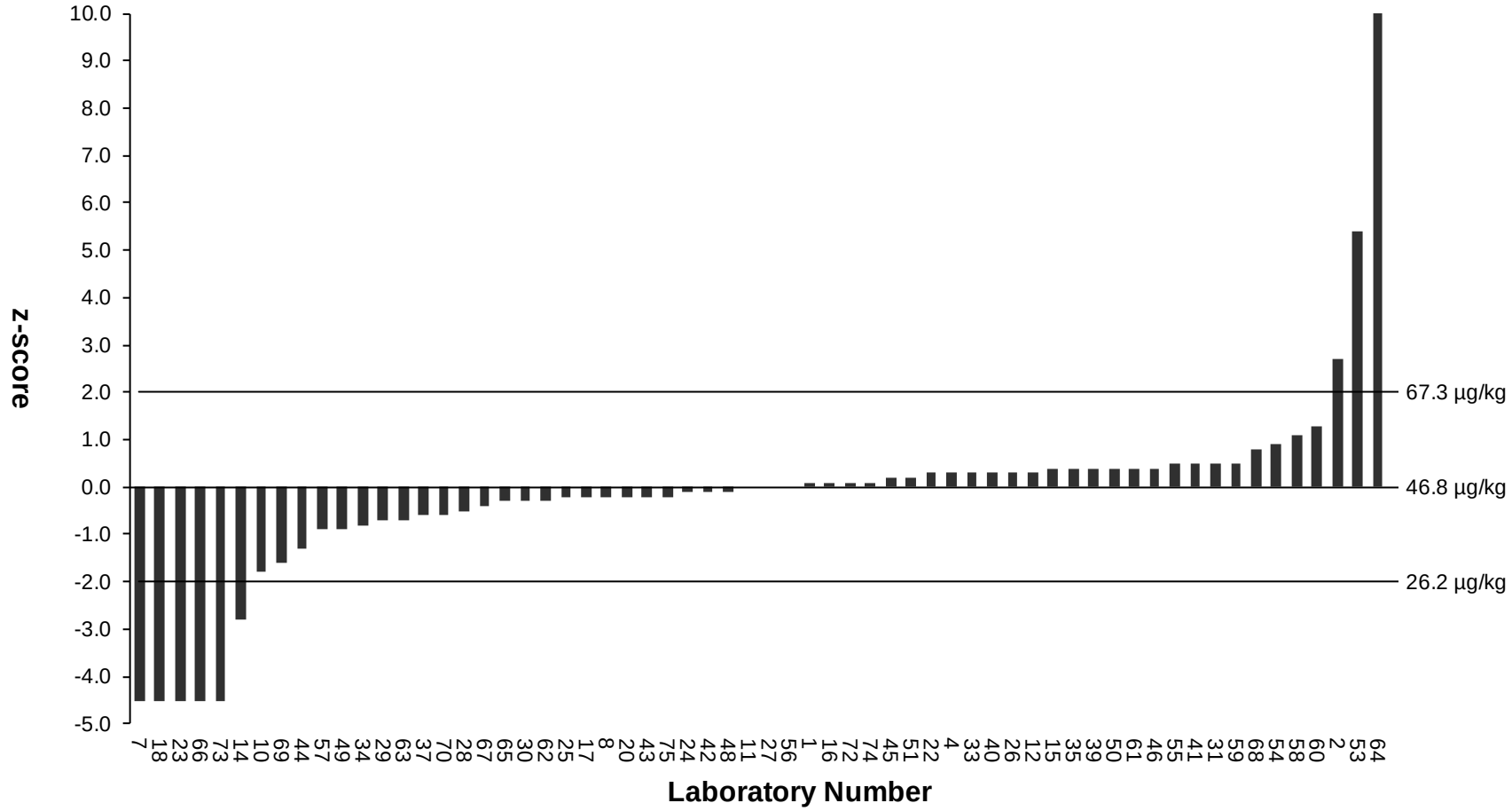

Figure 9: z-Scores for Pirimicarb

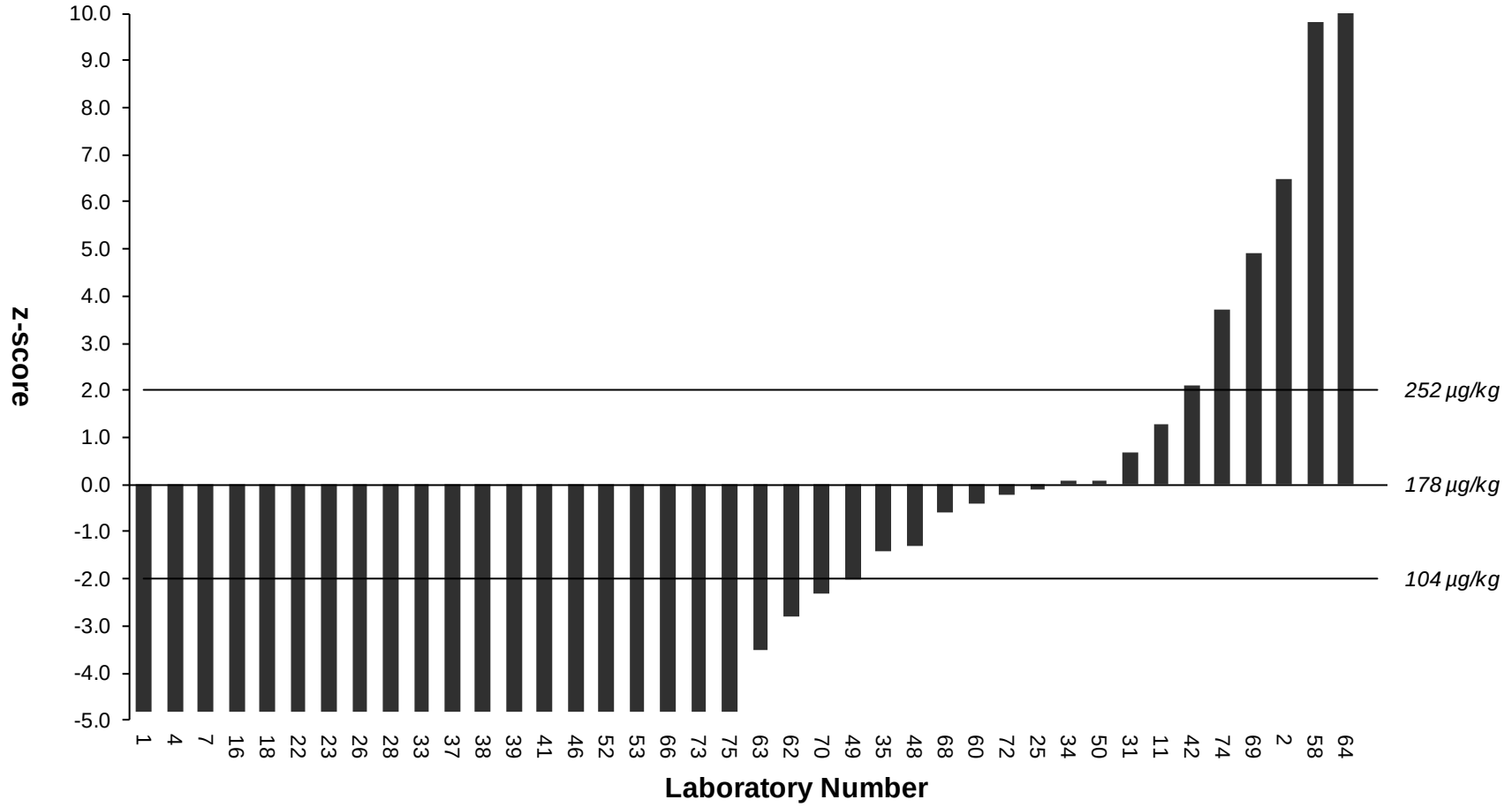

Figure 10: z-Scores for Spirotetramat

this histogram is given for information only

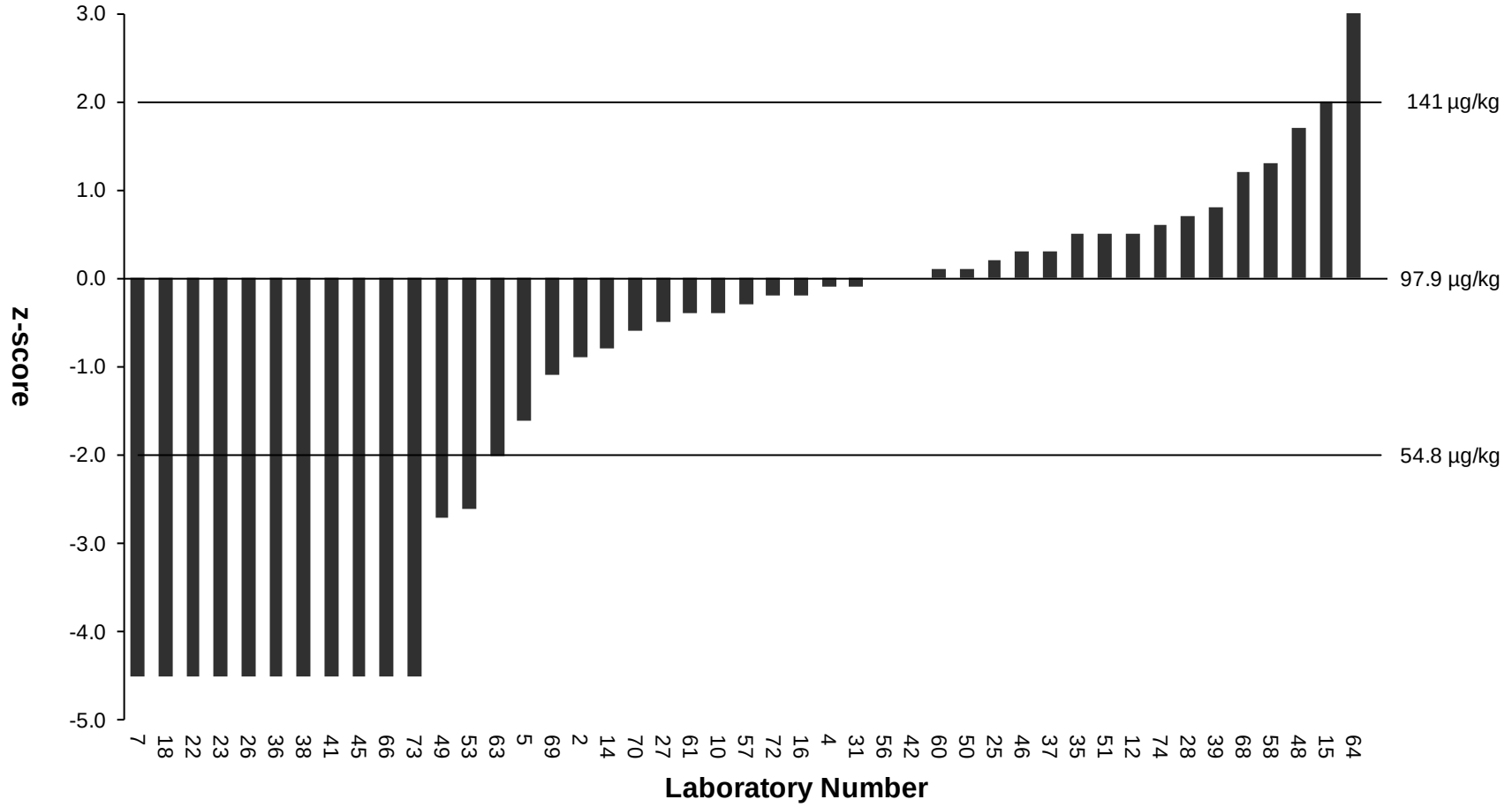

Figure 11: z-Scores for Triticonazole

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	005 009 012 013 044 057 065 071
yes	001 003 004 006 007 008 010 011 014 015 016 017 019 020 021 022 023 025 027 028 029 030 031 032 033 035 037 038 039 040 041 042 043 046 048 050 051 052 053 054 055 056 060 062 064 067 068 069 070 074

Method Based On	laboratory number
International Standard	002 005 011 014 017 020 027 029 031 035 039 040 041 042 045 048 054 056 063 069 070 071 074
National Standard	003 007 008 012 013 016 019 021 022 023 030 032 033 043 046 052 055 060 064
Paper Published In An International Journal	010 037 044 062 065
In house method	001 004 006 009 015 025 038 050 053 057 067 068

quoted percentage recovery measured in same analytical batch as test material	laboratory number
no	007 038 048 054 057 060 062 064
yes	001 003 004 005 008 009 010 011 012 013 014 015 016 017 019 020 021 023 025 027 029 030 031 032 033 035 037 039 040 041 042 043 044 045 046 050 051 052 053 056 063 065 067 068 069 070 071 074

If measured in this batch, stage spike added	laboratory number
prior to extraction	001 002 003 004 005 006 008 009 010 011 012 013 014 015 016 020 022 023 025 027 029 030 031 032 033 035 037 038 039 040 042 044 046 048 050 052 054 055 056 063 064 065 067 068 069 070 071 074
prior to clean up	021 041 043
prior to instrument measurement	017 019 053

Concentration of Spike (µg/kg)	laboratory number
≥1 - <5	021
≥5 - <10	015 017 044 048
≥10 - <25	002 004 006 010 016 022 025 030 042 050 052 053 062 067 068 069 071 074
≥25 - <50	051 054 057
≥50 - <100	012 014 019 020 023 029 033 035 038 039 043 046 056 060 063 064 070
≥100	001 003 005 008 009 011 013 027 031 032 037 040 041 065

Composition of Blank Commodity used for Spiking	laboratory number
blank provided	001 002 006 008 010 012 013 014 015 016 017 019 020 021 022 023 027 029 030 031 032 033 035 037 038 040 041 043 044 046 048 051 052 053 054 057 062 064 070 071
Apple (tested for Pesticides free)	005
commercial blank	003 069
cucumber	004 050
In house blank strawberry	067
mix of blank matrices	056
NEGATIVE SAMPLE	011
Organic Matrix	063 068
Organic strawberry	025 060
Purchased material	074

Calibration	laboratory number
standard addition	007 009 013 022 031 040 054 055 060 062 063 064
matrix-matched	004 006 008 010 011 014 015 016 017 020 021 025 027 030 032 033 035 037 039 041 043 044 045 046 048 053 056 065 067 068 070 071
solvent	001 006 019 054 069
multi-level	001 002 003 004 005 012 013 022 023 025 029 031 037 044 050 052 057 065 068
single-level	042 064 074

Internal Standard Added	laboratory number
no	005 010 016 019 020 021 023 029 030 032 039 044 046 052 054 055 057 065 068 071
yes	001 003 004 006 007 008 009 011 012 013 014 015 017 025 027 031 033 035 037 038 040 042 043 045 048 050 051 053 056 060 062 063 064 067 069 070 074

Internal Standard Used	laboratory number
Atrazine D5	069
BNB	067
Coffein for GC and Carburan-d3 for LC	027
D10-Fluorene	074
Deuterated	001 050 063
Didn't Use An Internal Standard	054
GC:ChlorpyrifosD10,Clorthal-dimethylD6,DiflufenicanD3	012
Mixture of p,p-DDT, p,p-DDD, p,p-DDE	007
no internal standard used	005 021 029
Phosphoric Acid Triphenyl Ester	003
Pirimicarb D6, Nicarbazim	035
PCB 31	064
Carbaryl D7	015
Nicarbazin	048 056 060 069
Trifluralin D14, pp-DDE D8	025
TPP	002 008 009 011 013 015 035 037 038 046 048 051 056 060 064
Phenanthrene-d10	038

GC Method: Sample Weight (g)	laboratory number
≥1 - <5	006 023 038 041 044
≥5 - <10	014 015 016 032 042 043 048 051 057
≥10 - <20	002 004 008 010 011 012 017 020 022 025 027 030 031 033 035 037 039 040 045 046 050 053 055 056 060 062 063 064 065 067 068 069 074
≥20 - <50	003 007 009 013 019 021 052
10	070
50 g	029
No GC detection	005

GC Method: Extraction Solvent Components	laboratory number
acetone	019 023 029
acetonitrile	002 003 006 008 009 010 011 012 013 014 015 016 017 020 021 022 027 030 031 032 033 035 037 038 039 040 041 042 043 044 046 048 051 052 055 056 057 060 062 063 064 067 068 070 074
dichloromethane	029
ethyl acetate	004 025 045 050 069
methanol	053
water	042
acetic acid	046
Hexane	007
No GC detection	005

GC Method: Extraction pH Adjusted	laboratory number
no	004 005 007 008 009 010 012 014 015 016 017 019 021 023 029 030 032 033 037 040 044 045 048 050 052 053 055 056 057 060 062 063 064 065 069 070 074
yes	003 006 011 013 020 025 027 031 035 038 039 041 042 043 046 067 068

GC Method: Extraction Techniques Used	laboratory number
macerate at room temperature	007 009 012 025
QuEChERS	002 004 006 008 010 011 014 015 016 017 020 021 022 023 027 030 031 032 033 035 037 039 040 041 042 043 044 045 046 048 050 051 053 055 056 057 060 062 063 064 065 067 068 069 070 074
liquid-liquid partition	003 012 013 019 029 038 052
No GC detection	005

GC Method: Sample Clean-up Technique	laboratory number
GPC/HPGPC	025
liquid-liquid partition	012 023 029
none	004 005 019 038 050 064

GC Method: Sample Clean-up Technique (continued)	laboratory number
solid phase extraction (SPE) (column/cartridge)	003 007 009 012 013 021 052 057
solid phase extraction (SPE) (dispersive)	006 008 011 014 015 016 017 020 022 027 030 031 032 035 037 039 041 042 043 044 046 048 053 056 060 063 065 067 068 069 070 074
Dispersive PSA	010
QuEChERS	040

GC Method: SPE Sorbent Type	laboratory number
C18	069
GCB	041 053
PSA	004 008 011 014 015 016 017 020 021 022 023 027 030 031 035 037 039 042 043 048 056 057 060 063 067 068
NH2	003 012 013
Mixed Mode	007 009 010 032 046 070
Carbon/NH2	052
Envicarb	003 004 013 053
none	005 019 029 050

GC Method: GC Column Packing	laboratory number
50% methyl 50% phenyl polysiloxane	019 044 074
65% methyl 35% phenyl polysiloxane	021
95% methyl 5% phenyl polysiloxane	003 004 008 009 010 013 014 015 016 017 020 022 023 025 027 030 032 035 037 039 040 041 042 043 046 048 050 052 053 060 062 063 064 065 067 068 069 070
5% diphenyl / 95% dimethyl polysiloxane	011 012 038 056
n/a	029
No GC detection	005
supelco SPB-5	007
Trifluoropropyl methyl polysiloxane	031

GC Method: GC Detector Type	laboratory number
ECD	007 040 060
FPD	019 029
MS	003 009 013 038 039 060 063 064 067 069
MS-MS	002 004 006 008 010 011 012 014 015 016 017 020 021 022 023 025 027 029 030 031 032 033 035 037 041 042 043 044 045 046 048 050 051 052 053 055 056 060 062 064 065 068 070 074
NPD	040
No GC detection	005

LC Method: Sample Weight (g)	laboratory number
≥1 - <5	041
≥5 - <10	015 016 023 032 042 043 044 048 051 054 057
≥10 - <20	001 002 004 005 008 010 011 012 014 017 020 022 025 027 030 031 033 035 037 039 040 045 046 050 053 055 056 060 062 063 064 065 067 068 069 071 074
≥20 - <50	003 009 013 021 052
10	070
50	029

LC Method: Extraction Solvent Components	laboratory number
acetone	029
acetonitrile	001 002 003 004 005 008 009 010 011 012 013 014 015 016 017 020 021 022 023 025 027 030 031 032 033 035 037 039 040 041 042 043 044 046 048 050 051 052 053 054 055 056 057 060 062 063 064 067 068 069 070 071 074
dichloromethane	029
ethyl acetate	045
acetic acid	046 071

LC Method: Extraction pH Adjusted	laboratory number
no	004 008 009 010 012 014 015 017 021 023 025 029 030 032 033 037 040 044 048 050 052 053 054 055 056 057 060 062 063 064 065 067 069 070 071 074
yes	001 003 005 011 013 016 020 027 031 035 039 042 043 046 068

LC Method: Extraction Techniques Used	laboratory number
macerate at room temperature	009 012
QuEChERS	001 002 004 005 008 010 011 014 015 016 017 020 021 022 023 025 027 030 031 032 033 035 037 039 040 041 042 043 044 045 046 048 050 051 053 054 055 056 057 060 062 063 064 065 067 068 069 070 071 074
liquid-liquid partition	003 012 013 029 052

LC Method: Sample Clean-up Technique	laboratory number
GPC/HPGPC	053
liquid-liquid partition	012 020 023
none	004 005 017 022 025 029 032 043 050 056 064 065
solid phase extraction (SPE) (column/cartridge)	003 009 012 013 016 041 052 057
solid phase extraction (SPE) (dispersive)	011 014 015 021 027 030 031 035 037 039 042 044 046 048 054 060 063 067 068 069 070 071 074
Dilution	008
Dispersive PSA	010
QuEChERS	040

LC Method: SPE Sorbent Type	laboratory number
C18	001 016 017 053 054 069
GCB	041
PSA	004 010 011 015 027 030 031 035 037 039 042 048 057 060 063 067 068 071
NH2	003 012 013 021 052
Mixed Mode	009 014 023 046 070

LC Method: SPE Sorbent Type (continued)	laboratory number
Carbon	052
EnviCarb	003 013 041
n/a	029
NONE	005 020
Magnesium sulphate	071

LC Method: HPLC Column Packing	laboratory number
C18	001 003 004 005 008 009 010 011 012 013 014 015 017 020 021 022 023 025 027 029 030 031 032 033 035 037 039 040 041 042 043 044 046 048 050 052 053 054 056 060 062 063 065 067 068 069 070 071 074
Mixed Mode	016
biphenyl	057
PFP	064

LC method: Mobile Phase Components	laboratory number
ethanoic acid (acetic acid)	027 060 069
acetonitrile	014 029 037 048 054 060 064 065 071
ammonium acetate	003 009 010 012 013 025 030 033 042 044 046 052 056 071
ammonium formate	001 005 008 011 014 016 017 020 021 023 029 031 032 035 039 043 048 054 060 062 067 068 069
formic acid (methanoic acid)	004 008 010 011 020 032 037 042 048 051 056 062 064 065 067 068
methanol	001 003 005 008 010 011 012 013 014 015 020 023 025 027 029 030 031 032 035 039 040 042 044 050 052 053 056 062 067 069 071
water	001 008 010 011 012 013 014 020 022 023 025 027 029 030 031 032 035 039 041 042 048 050 056 057 062 063 064 065 069 071
FASE A FORMATO DE AMONIO Y ACIDO FORMICO EN AGUA GRADO MASAS; FASE B METANOL GRADO MASAS	070

LC Method: Detector Type	laboratory number
HR-MS	037
MS-MS	001 002 003 004 005 008 009 010 011 012 013 014 015 016 017 020 021 022 023 025 027 029 030 031 032 033 035 039 040 041 042 043 044 045 046 048 050 051 052 053 054 055 056 057 058 060 062 063 064 065 067 069 070 071 074
qTOF	068

Aldicarb-sulfoxide

Method Principle	laboratory number
LC	012 030 033 048 062 064 069 074

Identification by Mass Spectrometry	laboratory number
yes	012 030 033 048 062 064 069 074

Cyhalothrin-lambda

Method Principle	laboratory number
GC	010 012 030 033 038 048 062 064 065 069 074
LC	005

Identification by Mass Spectrometry	laboratory number
yes	005 010 012 030 033 038 048 062 064 065 069 074

Dimethoate

Method Principle	laboratory number
GC	003 013 016 040 062 064
LC	005 010 012 030 033 041 048 065 069 074

Identification by Mass Spectrometry	laboratory number
yes	003 005 010 012 013 016 030 033 040 041 048 062 064 065 069 074

Endosulfan II (beta)

Method Principle	laboratory number
GC	012 023 030 033 038 048 062 064 065 069 074

Identification by Mass Spectrometry	laboratory number
yes	012 023 030 033 038 048 062 064 065 069 074

Etoxazole

Method Principle	laboratory number
GC	003 012 013 016 062 064
LC	010 033 040 048 069 074

Identification by Mass Spectrometry	laboratory number
yes	003 010 012 013 016 033 040 048 062 064 069 074

Fenpyroximate

Method Principle	laboratory number
LC	005 010 012 016 030 033 040 041 048 062 064 065 069 074

Identification by Mass Spectrometry	laboratory number
yes	005 010 012 016 030 033 040 041 048 062 064 065 069 074

Monocrotophos

Method Principle	laboratory number
LC	010 012 021 030 032 033 040 048 064 065 069 074

Identification by Mass Spectrometry	laboratory number
yes	010 012 021 030 032 033 040 048 064 065 069 074

Phosphamidon

Method Principle	laboratory number
GC	012 016 048
LC	030 033 040 041 064 065 069 074

Identification by Mass Spectrometry	laboratory number
yes	012 016 030 033 040 041 048 064 065 069 074

Pirimicarb

Method Principle	laboratory number
GC	012 016 038 048 062
LC	010 030 033 040 041 064 065 069 074

Identification by Mass Spectrometry	laboratory number
yes	010 012 016 030 033 038 040 041 048 062 064 069 074

Spirotetramat

Method Principle	laboratory number
LC	048 062 064 069 074

Identification by Mass Spectrometry	laboratory number
yes	048 062 064 069 074

Triticonazole

Method Principle	laboratory number
LC	005 010 012 016 048 064 069 074

Identification by Mass Spectrometry	laboratory number
yes	005 010 012 016 048 064 069 074

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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