FAPAS® Report 19140

Pesticide Residues in Lettuce

October-December 2012

PARTICIPANT LABORATORY NUMBER

Participants can log in to FAPAS SecureWeb at any time to obtain their laboratory number for this proficiency test.

Laboratory numbers are displayed in SecureWeb next to the download link for this report.

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SUMMARY

1. The test material for FAPAS® proficiency test 19140 was dispatched in October 2012. Each participant received a lettuce test material. From a list of 200 pesticide residues, participants had to identify and quantify those present.
2. The test material contained beta-endosulfan, parathion (-ethyl), pencyuron, phosalone, propyzamide, tecnazene and thiamethoxam. An assigned value (x_a) was determined for each analyte and in conjunction with the standard deviation for proficiency (σ_p) was used to calculate a z-score for each result.
3. Results for this proficiency test are summarised as follows:

analyte	assigned value, x_a µg/kg	number of scores, |z| ≤2	total number of scores	% |z| ≤2
beta-endosulfan	93.6	72	89	81
parathion (-ethyl)	25.6	59	73	81
pencyuron	73.2	55	67	82
phosalone	70.4	73	85	86
propyzamide	89.9	68	77	88
tecnazene	38.0	48	54	89
thiamethoxam	48.8	70	78	90

4. Surplus test materials are available for sale, see APPENDIX II.

CONTENTS

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® proficiency testing scheme are available in our protocols [3, 4].

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 [2].

The test material was prepared from organic lettuces, which were frozen and cryogenically milled. 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 incurred residues were detected at, or above, 25 µg/kg.

Beta-endosulfan, parathion (-ethyl), pencyuron, phosalone, propyzamide, tecnazene and thiamethoxam 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 [2].

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

2.3. Dispatch

The start date was 22 October 2012. Test materials were sent to 128 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 on the results form.

Results were submitted by 116 participants (91%) before the closing date for this test, 06 December 2012.

Each participant was given a laboratory number, assigned in order of receipt of results. The reported analyte concentrations are given in Table 1 for beta-endosulfan, parathion (-ethyl), pencyuron and phosalone and in Table 2 for propyzamide, tecnazene and thiamethoxam.

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 LoQ was *above* the level needed for a z-score of -3.0, then the result was recorded as <LoQ.

Any participant identifying pesticide residues other than beta-endosulfan, parathion (-ethyl), pencyuron, phosalone, propyzamide, tecnazene and thiamethoxam at levels above 25 µg/kg are listed in Table 3.

Participants' comments are given in Table 4.

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 for each result. The procedure follows that recommended in the IUPAC International Harmonised Protocol for the Proficiency Testing of Analytical Chemistry Laboratories [5].

Further details on the procedure followed can be found in the relevant protocols [3, 4].

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) 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),

ii) results where no percentage recovery was reported,

iii) results whose recovery was outside the range 70-120% [6],

iv) results where no limit of quantification was reported,

v) results less than the reported LoQ,

vi) quantitative and semi-quantitative results, e.g. <LoQ.

For all analytes, this procedure was straightforward and the robust mean was chosen as the assigned value.

The assigned values for all analytes are shown in Table 5.

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 [7].

The values for σ_p used to calculate z-scores from the reported results of this test are given in Table 5.

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 are given in Table 1 for beta-endosulfan, parathion (-ethyl), pencyuron and phosalone and in Table 2 for propyzamide, tecnazene and thiamethoxam. They are shown as histograms in Figures 1–7. 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 5.

The number and percentage of z-scores in the range $-2 \leq z \leq 2$ for all analytes are given in Table 6.

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 [5].

6. REFERENCES

1 Adobe Certified Document Services, http://www.adobe.com/security/partners_cds.html, accessed 08/05/2012.

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

3 FAPAS, 2012, Protocol for Proficiency Testing Schemes, Part 1 – Common Principles, Version 3, Issued January 2012.

4 FAPAS, 2012, Protocol for Proficiency Testing Schemes, Part 2 – FAPAS®, Version 2, Issued January 2012.

5 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.

6 Method Validation and Quality Control Procedures for Pesticide Residue Analysis in Food and Feed, Document No. SANCO/12495/2011.

7 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 beta-Endosulfan, Parathion (-ethyl), Pencyuron and Phosalone

laboratory number	analyte															
	beta-endosulfan assigned value 93.6 µg/kg				parathion (-ethyl) assigned value 25.6 µg/kg				pencyuron assigned value 73.2 µg/kg				phosalone assigned value 70.4 µg/kg			
	result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score
001	60	98.4	10	-1.6	30	89.8	10	0.8	#				0			**-4.5**
002	#				#				#				65.1	96	30	-0.3
003	#				#				#				#			
004	73	100	10	-1.0	22	100	10	-0.6	47	100	10	-1.6	51	100	10	-1.3
005	#				#				#				#			
006	102	116	10	0.4	29	111	10	0.6	72	86	10	-0.1	80	87	10	0.6
007	#				#				#				68.0	98	30	-0.2
008	#				#				#				#			
009	136	100	10	**2.1**	#				<LOQ		50		106	91	10	**2.3**
010	#				#				#				64	103.9	30	-0.4
011	64.5			-1.4	34.6			1.6	68.2			-0.3	61.8			-0.6
012	#				#				#				78.5	103	30	0.5
013	#				#				#				65.7	92	30	-0.3
014	0			**-4.5**	20.15	94.8	10	-1.0	76.91	92.4	10	0.2	70.47	84.5	10	0.0
015	132	97.3		1.9	#				#				#			

= not analysed

z-scores outside |z| >2 are shown in **bold,** see Section 5

Table 1 (continued): Results and z-Scores for beta-Endosulfan, Parathion (-ethyl), Pencyuron and Phosalone

laboratory number	analyte															
	beta-endosulfan assigned value 93.6 µg/kg				parathion (-ethyl) assigned value 25.6 µg/kg				pencyuron assigned value 73.2 µg/kg				phosalone assigned value 70.4 µg/kg			
	result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score
016	#				20±10	75	20	-1.0	#				#			
017	80.0	81	30	-0.7	30.0	85	30	0.8	90.0	82	60	1.0	60.0	78	30	-0.7
018	#				#				#				60.7	82	30	-0.6
019	#				#				#				#			
020	62	88	10	-1.5	<LOQ		<10		87	96	10	0.9	80	97	10	0.6
021	0	100	10	**-4.5**	30	100	10	0.8	90	100	10	1.0	50	100	10	-1.3
022	52	82.5	20	-2.0	<LOQ	65	20		#				49	82.5	20	-1.4
023	63	90.3	10	-1.5	18	100.8	10	-1.3	57	93	10	-1.0	49	77.1	10	-1.4
024	109.00	89	32	0.7	31.25	105	6	1.0	85.41	100	5	0.8	76.5	94	6	0.4
025	58	100	10	-1.7	23	79	10	-0.5	66	90	10	-0.4	48	90	10	-1.4
026	92	96	10	-0.1	26	80	10	0.1	82	87	10	0.5	49	95	10	-1.4
027	#				#				76.3	95.4	10	0.2	#			
028	127.96			1.7	#				#				#			
029	46.7		20	**-2.3**	<LOQ		30		#				<LOQ		30	
030	115	90	10	1.0	30	92	10	0.8	75	94		0.1	73	102	10	0.2

= not analysed

z-scores outside |z| >2 are shown in **bold,** see Section 5

Table 1 (continued): Results and z-Scores for beta-Endosulfan, Parathion (-ethyl), Pencyuron and Phosalone

laboratory number	analyte															
	beta-endosulfan assigned value 93.6 µg/kg				parathion (-ethyl) assigned value 25.6 µg/kg				pencyuron assigned value 73.2 µg/kg				phosalone assigned value 70.4 µg/kg			
	result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score
031	76	92.5	10	-0.9	13	96.6	10	**-2.2**	62	91.9	10	-0.7	63	88.9	10	-0.5
032	#				<LOQ	97.3	25		#				#			
033	#				#				71.3	93.2	10.0	-0.1	#			
034	120	99.50	5.00	1.3	27.7	93.00	5.00	0.4	79.9	96.00	5.00	0.4	80.1	95.00	5.00	0.6
035	#				#				66.99	92.5	8	-0.4	#			
036	#				#				#				#			
037	89.3		10	-0.2	26.4			0.1	#				72.5			0.1
038	#				#				#				78	104	50	0.5
039	102	100	10	0.4	24.1	91	10	-0.3	67.8	88	10	-0.3	69.9	88	10	0.0
040	77	90	10	-0.8	25	90	10	-0.1	96	90	10	1.4	#			
041	61	87	25	-1.6	<LOQ	85	30		69	85	50	-0.3	418	87	60	**22.4**
042	100			0.3	#				#				#			
043	100	96	10	0.3	<LOQ	91	10		#				80	104	10	0.6
044	144.654		10	**2.5**	50.874		10	**4.5**	61.070		10	-0.8	66.781		10	-0.2
045	#				<LOQ	87.5	25		#				<LOQ	83.15	50	

= not analysed

z-scores outside |z| >2 are shown in **bold,** see Section 5

Table 1 (continued): Results and z-Scores for beta-Endosulfan, Parathion (-ethyl), Pencyuron and Phosalone

laboratory number	analyte															
	beta-endosulfan assigned value 93.6 µg/kg				parathion (-ethyl) assigned value 25.6 µg/kg				pencyuron assigned value 73.2 µg/kg				phosalone assigned value 70.4 µg/kg			
	result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score
046	69.42	111	10	-1.2	38	116	10	**2.2**	103.71	124	10	1.9	56	124	10	-0.9
047	95	98	10	0.1	0			**-4.5**	68	103	10	-0.3	68	99	10	-0.2
048	115.0	98	10	1.0	20.0	97	10	-1.0	62.3	110	10	-0.7	80.0	99	10	0.6
049	#				#				#				#			
050	88	94	10	-0.3	23	96	10	-0.5	46	77	10	-1.7	58	99	10	-0.8
051	69.12	94.50	0.10	-1.2	#				#				#			
052	117		20	1.1	#				74		10	0.0	84		20	0.9
053	#				25.6	89.8	10	0.0	#				#			
054	73	122	40	-1.0	29	167	10	0.6	#				66	138	40	-0.3
055	96.1	100	10	0.1	16.0	100	10	-1.7	#				63.0	100	10	-0.5
056	72.5	97		-1.0	20.65	91		-0.9	67.25	112		-0.4	63.85	107		-0.4
057	116	107	30	1.1	<LOQ		30		38	90	30	**-2.2**	#			
058	130.0	112	10	1.8	#				#				98.4	96	10	1.8
059	140	98	10	**2.2**	30	95	10	0.8	120	100	10	**2.9**	#			
060	95	83	10	0.1	29	87	10	0.6	66	96	10	-0.4	72	102	10	0.1

= not analysed

z-scores outside |z| >2 are shown in **bold,** see Section 5

Table 1 (continued): Results and z-Scores for beta-Endosulfan, Parathion (-ethyl), Pencyuron and Phosalone

laboratory number	analyte															
	beta-endosulfan assigned value 93.6 µg/kg				parathion (-ethyl) assigned value 25.6 µg/kg				pencyuron assigned value 73.2 µg/kg				phosalone assigned value 70.4 µg/kg			
	result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score
061	79	101	10	-0.7	21	123	10	-0.8	109	96	10	**2.2**	75	77	10	0.3
062	0	90	5	**-4.5**	19.0	90	10	-1.2	#				#			
063	71.35	100.04	5	-1.1	33.45	98.24	5	1.4	81.21	84.88	5	0.5	42.06	96.43	5	-1.8
064	65		10	-1.4	13		10	**-2.2**	64		10	-0.6	31		10	**-2.5**
065	95	90	10	0.1	46	95	10	**3.6**	73	100	10	0.0	158	90	10	**5.6**
066	141	97	20	**2.3**	25	97	20	-0.1	#				120	97	30	**3.2**
067	93.3	93	10.0	0.0	29.4	107	10.0	0.7	28.0	83	10.0	**-2.8**	68.9	105	10.0	-0.1
068	60			-1.6	20			-1.0	0		10	**-4.5**	0		10	**-4.5**
069	94.8	98.2	10	0.1	22.7	98.1	10	-0.5	#				66.1	97.9	10	-0.3
070	#				0			**-4.5**	#				#			
071	71	46.78	10	-1.1	19	63.67	10	-1.2	91	91.52	10	1.1	53	80.41	10	-1.1
072	0		0.02	**-4.5**	65		0.02	**7.0**	0			**-4.5**	120		0.02	**3.2**
073	#				#				#				#			
074	87.1			-0.3	12.8			**-2.3**	#				67.5			-0.2
075	0			**-4.5**	24	60	10	-0.3	#				46	62	10	-1.6

= not analysed

z-scores outside |z| >2 are shown in **bold,** see Section 5

Table 1 (continued): Results and z-Scores for beta-Endosulfan, Parathion (-ethyl), Pencyuron and Phosalone

laboratory number	analyte															
	beta-endosulfan assigned value 93.6 µg/kg				parathion (-ethyl) assigned value 25.6 µg/kg				pencyuron assigned value 73.2 µg/kg				phosalone assigned value 70.4 µg/kg			
	result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score
076	#				#				#				69.9	100	0.03	0.0
077	#				28	97		0.4	#				68	98		-0.2
078	121	99	60	1.3	#				83	89	30	0.6	91	98	10	1.3
079	89	96	10	-0.2	24	93	10	-0.3	81	100	10	0.5	63	91	10	-0.5
080	130	70	10	1.8	30	80	10	0.8	80	70	10	0.4	90	80	10	1.3
081	72	97	10	-1.1	11	112	10	**-2.6**	106	116	10	2.0	39	96	10	-2.0
082	83		10	-0.5	#				#				#			
083	84.9			-0.4	27.8			0.4	64.7			-0.5	49.3			-1.4
084	66.4179	87.22	50	-1.3	#				#				#			
085	84.0	84	10	-0.5	23.2	107	10	-0.4	40.0	106	10	**-2.1**	64.5	96	10	-0.4
086	110	90	10	0.8	29	90	10	0.6	78	97	10	0.3	75	89	10	0.3
087	#				#				#				#			
088	99	87	10	0.3	29	90	10	0.6	70	93	10	-0.2	75	93	10	0.3
089	101.39		10	0.4	29.81		10	0.8	79.64			0.4	82.69		10	0.8
090	101.9	87		0.4	30.9	73		0.9	#				61.2	75		-0.6

= not analysed

z-scores outside |z| >2 are shown in **bold,** see Section 5

Table 1 (continued): Results and z-Scores for beta-Endosulfan, Parathion (-ethyl), Pencyuron and Phosalone

laboratory number	analyte															
	beta-endosulfan assigned value 93.6 µg/kg				parathion (-ethyl) assigned value 25.6 µg/kg				pencyuron assigned value 73.2 µg/kg				phosalone assigned value 70.4 µg/kg			
	result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score
091	100	94	10	0.3	<LOQ	100	50		70	98	50	-0.2	66	76	10	-0.3
092	60.0	100.5	10	-1.6	26.3	99.4	10	0.1	63.5	99.4	10	-0.6	62.7	99.5	10	-0.5
093	77.7	92	10	-0.8	24.2	127	10	-0.2	65.2	81	10	-0.5	54.8	89	10	-1.0
094	88	100	10	-0.3	23	100	10	-0.5	#				#			
095	55.3	98.1	10	-1.9	25.4	106	20	0.0	55.0	95	10	-1.1	65.5	103.1	30	-0.3
096	77.0	68.7	10	-0.8	18.7	78.8	10	-1.2	54.2	70.2	10	-1.2	55.5	72.3	30	-1.0
097	130	76	10	1.8	25	89	10	-0.1	65	87	10	-0.5	68	76	10	-0.2
098	127	89	10	1.6	29	107	10	0.6	87	96	10	0.9	91	109	10	1.3
099	94.7	123	10	0.1	22.3	116	10	-0.6	66	130	10	-0.4	74	65	10	0.2
100	100		5	0.3	29		10	0.6	67		10	-0.4	70		10	0.0
101	85	95	6	-0.4	33	105	10	1.3	#				87	25	90	1.1
102	122	86	10	1.4	43	95	10	**3.1**	0			**-4.5**	93	100	10	1.5
103	#				<LOQ		<100		83.2	89		0.6	27.5	91		**-2.8**
104	116	134	10	1.1	31	83	10	1.0	#				#			
105	0		10	**-4.5**	<LOQ		50		69	89	10	-0.3	86	93	10	1.0

= not analysed

z-scores outside |z| >2 are shown in **bold,** see Section 5

Table 1 (continued): Results and z-Scores for beta-Endosulfan, Parathion (-ethyl), Pencyuron and Phosalone

laboratory number	analyte															
	beta-endosulfan assigned value 93.6 µg/kg				parathion (-ethyl) assigned value 25.6 µg/kg				pencyuron assigned value 73.2 µg/kg				phosalone assigned value 70.4 µg/kg			
	result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score
106	96.79	91.5	9.40	0.2	15.35	79.9	8.70	-1.8	#				#			
107	#				#				#				#			
108	150	105	10	**2.7**	18	95	10	-1.3	97	95	10	1.5	87	95	10	1.1
109	95.6	100	10	0.1	21.6	117	10	-0.7	113	115	10	**2.5**	64.6	110	10	-0.4
110	0			**-4.5**	0			**-4.5**	74			0.0	89			1.2
111	0	57	10	**-4.5**	<LOQ	87	10		0	0	10	**-4.5**	0	0	10	**-4.5**
112	0.085	76	0.01	**-4.5**	0.044	98	0.01	**-4.5**	0.072		0.01	**-4.5**	0.091	97	0.01	**-4.5**
113	88.9		20	-0.2	23.6		20	-0.4	#				58.8		10	-0.8
114	63			-1.5	23			-0.5	63			-0.6	62			-0.5
115	0		25	**-4.5**	<LOQ		25		53.20	98.08	25	-1.2	84.70	86.01	25	0.9
116	0			**-4.5**	0			**-4.5**	0			**-4.5**	0			**-4.5**

= not analysed

z-scores outside |z| >2 are shown in **bold,** see Section 5

Table 2: Results and z-Scores for Propyzamide, Tecnazene and Thiamethoxam

laboratory number	analyte											
	propyzamide assigned value 89.9 µg/kg				tecnazene assigned value 38.0 µg/kg				thiamethoxam assigned value 48.8 µg/kg			
	result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score
001	#				#				33	84.4	10	-1.5
002	#				#				45.7	97	20	-0.3
003	#				#				#			
004	80	100	10	-0.5	36	100	10	-0.2	75	100	10	**2.4**
005	#				#				47.8	108.05	50	-0.1
006	106	96	10	0.8	38	94	10	0.0	52	101	10	0.3
007	#				#				50.5	116	20	0.2
008	#				#				#			
009	117	105	10	1.4	#				#			
010	#				#				46	95.5	20	-0.3
011	74.4			-0.8	38.6			0.1	43.2			-0.5
012	#				#				59.5	111	20	1.0
013	#				#				45.4	98	20	-0.3
014	#				#				50.71	79.6	10	0.2
015	108	99.5		0.9	42.6	114		0.5	60.9	85.7		1.1
016	#				#				#			
017	80.0	83	30	-0.5	30.0	75	30	-1.0	42.0	82	18	-0.6
018	#				#				52.7	102	20	0.4
019	#				#				#			
020	99	96	10	0.5	41	97	10	0.4	52	97	10	0.3
021	80	100	10	-0.5	40	100	10	0.2	52	100	10	0.3
022	60	85	10	-1.5	#				#			
023	69	92.7	10	-1.1	24	98.5	10	-1.7	37	89	10	-1.1
024	105.13	101	5	0.8	40.25	87	6	0.3	49.28	100	5	0.0
025	76	104	10	-0.7	35	61	10	-0.4	44	89	10	-0.4
026	71	73	10	-1.0	40	77	10	0.2	45	93	10	-0.4
027	#				#				50.9	93.6	10	0.2
028	#				#				#			

= not analysed z-scores outside |z| >2 are shown in **bold,** see Section 5

Table 2 (continued): Results and z-Scores for Propyzamide, Tecnazene and Thiamethoxam

laboratory number	analyte											
	propyzamide assigned value 89.9 µg/kg				tecnazene assigned value 38.0 µg/kg				thiamethoxam assigned value 48.8 µg/kg			
	result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score
029	53.9		20	-1.8	#				#			
030	90	98	10	0.0	40	68		0.2	51	104	10	0.2
031	68	92.3	10	-1.1	25	78.9	10	-1.6	37	87.9	10	-1.1
032	#				#				#			
033	#				#				53.7	97.4	10.0	0.5
034	103	95.00	5.00	0.7	45.6	96.00	5.00	0.9	50.1	95.00	5.00	0.1
035	#				#				44.85	90.6	8	-0.4
036	#				#				#			
037	#				#				#			
038	88	96	10	-0.1	#				#			
039	94.8	89	10	0.2	38.3	84	10	0.0	47.2	88	10	-0.1
040	89	90	10	0.0	#				41	90	10	-0.7
041	107	86	60	0.9	#				38	80	10	-1.0
042	#				#				#			
043	100	96	10	0.5	40	81	10	0.2	#			
044	#				#				29.450		10	-1.8
045	#				#				#			
046	75	84	10	-0.8	39.75	114	10	0.2	57.2	99	10	0.8
047	84	108	10	-0.3	35	84	10	-0.4	43	101	10	-0.5
048	97.2	99	10	0.4	34.5	86	10	-0.4	52.6	104	10	0.4
049	31	70	10	**-3.0**	#				#			
050	83	97	10	-0.4	35	86	10	-0.4	37	95	10	-1.1
051	#				#				#			
052	99		10	0.5	#				45		20	-0.4
053	93.3	96.3	10	0.2	#				#			
054	132	124	40	**2.1**	32	117	10	-0.7	#			
055	92.3	100	10	0.1	38.5	100	10	0.1	51.0	100	5	0.2
056	87.3	107		-0.1	#				49.6	106		0.1

= not analysed z-scores outside |z| >2 are shown in **bold,** see Section 5

Table 2 (continued): Results and z-Scores for Propyzamide, Tecnazene and Thiamethoxam

laboratory number	analyte											
	propyzamide assigned value 89.9 µg/kg				tecnazene assigned value 38.0 µg/kg				thiamethoxam assigned value 48.8 µg/kg			
	result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score
057	77	89	22	-0.7	#				60	84	15	1.0
058	119.3	104	10	1.5	#				45.8	88.4	10	-0.3
059	140	99	10	**2.5**	#				#			
060	94	96	10	0.2	39	84	10	0.1	48	96	10	-0.1
061	69	124	20	-1.1	33	123	10	-0.6	77	98	10	**2.6**
062	#				#				#			
063	47.98	97.13	5	**-2.1**	24.86	97.72	5	-1.6	46.01	83.13	5	-0.3
064	52		10	-1.9	18		10	**-2.4**	37		10	-1.1
065	110	100	10	1.0	50	95	10	1.4	50	95	10	0.1
066	0		30	**-4.5**	#				95	102	30	**4.3**
067	#				#				20.9	88	10.0	**-2.6**
068	60			-1.5	19			**-2.3**	33			-1.5
069	92.5	97.4	10	0.1	44.5	91.7	10	0.8	#			
070	#				#				#			
071	98	96.96	10	0.4	13	60.53	10	**-3.0**	62	92.16	10	1.2
072	148		0.02	**2.9**	66		0.02	**3.3**	#			
073	#				#				#			
074	#				#				#			
075	#				#				#			
076	#				#				#			
077	#				#				#			
078	111	101	50	1.1	#				55	98	20	0.6
079	88	98	10	-0.1	38	81	10	0.0	49	105	10	0.0
080	110	80	10	1.0	40	80	10	0.2	50	90	10	0.1
081	58	102	10	-1.6	24	88	10	-1.7	55	110	10	0.6
082	#				#				#			
083	84.9			-0.3	38.0			0.0	51.8			0.3
084	#				#				#			

= not analysed z-scores outside |z| >2 are shown in **bold,** see Section 5

Table 2 (continued): Results and z-Scores for Propyzamide, Tecnazene and Thiamethoxam

laboratory number	analyte											
	propyzamide assigned value 89.9 µg/kg				tecnazene assigned value 38.0 µg/kg				thiamethoxam assigned value 48.8 µg/kg			
	result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score
085	90.8	106	10	0.0	31.6	93	10	-0.8	38.8	89	10	-0.9
086	93	96	10	0.2	47	96	10	1.1	50	95	10	0.1
087	#				#				#			
088	98	94	10	0.4	41	84	10	0.4	50	93	10	0.1
089	90.51		10	0.0	41.98		10	0.5	42.21		10	-0.6
090	56.1	78		-1.7	43	63		0.6	#			
091	78	95	10	-0.6	<LOQ	100	50		38	94	10	-1.0
092	79.3	100.4	10	-0.5	#				51.2	99.4	10	0.2
093	82.4	93	10	-0.4	35.2	97	10	-0.3	46.3	97	10	-0.2
094	85	100	10	-0.2	#				51			0.2
095	75.6	93.3	10	-0.7	#				48.8	95.8	10	0.0
096	#				#				53.7	106.2	10	0.5
097	75	86	25	-0.8	29	90	10	-1.1	46	76	10	-0.3
098	99	104	10	0.5	46	110	10	1.0	54	99	10	0.5
099	95.8	125	10	0.3	35.9	109	10	-0.3	44.2	122	10	-0.4
100	82		10	-0.4	#				48		10	-0.1
101	106	10	90	0.8	#				#			
102	116	103	10	1.3	47	95	10	1.1	53	98	10	0.4
103	76.3	88		-0.7	#				#			
104	#				36	76	10	-0.2	#			
105	0		10	**-4.5**	47	91	10	1.1	48	95	10	-0.1
106	#				#				#			
107	#				#				53	101	40	0.4
108	108	92	10	0.9	42	96	10	0.5	48	102	10	-0.1
109	112	87	10	1.1	44.4	102	10	0.8	0	89	10	**-4.5**
110	55			-1.8	44			0.7	51			0.2
111	84	97	10	-0.3	0	74	10	**-4.5**	0	0	10	**-4.5**
112	0.076	82	0.01	**-4.5**	#				0.031	98	0.01	**-4.5**

= not analysed z-scores outside |z| >2 are shown in **bold,** see Section 5

Table 2 (continued): Results and z-Scores for Propyzamide, Tecnazene and Thiamethoxam

laboratory number	analyte											
	propyzamide assigned value 89.9 µg/kg				tecnazene assigned value 38.0 µg/kg				thiamethoxam assigned value 48.8 µg/kg			
	result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score
113	82.3		10	-0.4	35.7		10	-0.3	#			
114	76			-0.7	28			-1.2	40			-0.8
115	76.10	111.59	25	-0.7	<LOQ		25		45.06	94.70	25	-0.3
116	0			**-4.5**	0			**-4.5**	0			**-4.5**

= not analysed z-scores outside |z| >2 are shown in **bold,** see Section 5

Table 3: Additional Pesticide Residues Reported

laboratory number	pesticide residue >25 µg/kg	result µg/kg	recovery %	LoQ µg/kg
021	endosulfan sulfate	80	100	10
072	alpha endosulfan	82		
072	permethrin	46		0.02
110	parathion-methyl	30		
111	chlorfenapyr	44	67	10
111	deltamethrin	34	73	10
111	fenpropathrin	62	61	10
111	indoxacarb	59	134	10
111	methamidophos	74.2	55	10
111	triazophos	52	76	10

Table 4: Participants' Comments

participant number	comments
036	Triflumizol: < 10 µg/kg, Rec:106% , LoQ: 10µg/kg // Cyromazina:<10 µg/kg, Rec:120%, LoQ: 10 µg/kg. It has been detected Imidacloprid: 2µg/kg
044	Reported results are against aqueous calibration curve except for beta endosulfan, thiamethoxam, pencycuron & phosalone which are against MMC.
065	Imidacloprid was detected under the limit of quantification (5 µg/kg)
070	Organochlorine and Synthetic Pyrethroid were not tested due to instrument (GC-ECD) breakdown.
102	Trace of Endosulfan-alfa: 2 µg/kg and Imidacloprid: 5 µg/kg
105	A trace of Imidacloprid was detected among limit of quantification 10 µg/kg.
108	Imidacloprid detected < LOQ
115	matrix matched calibration
116	A problem occured at the mass spectrometer during gas chromatography analysis. We were unable to complete the determination for the pesticides.

comments are as submitted by participants

Table 5: 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, µg/kg	
beta-endosulfan	54	93.6	3.72	Horwitz [7]	20.60
parathion (-ethyl)	47	25.6	0.874	Horwitz [7]	5.63
pencyuron	47	73.2	2.61	Horwitz [7]	16.11
phosalone	58	70.4	2.12	Horwitz [7]	15.50
propyzamide	53	89.9	2.39	Horwitz [7]	19.79
tecnazene	37	38.0	1.12	Horwitz [7]	8.37
thiamethoxam	59	48.8	0.880	Horwitz [7]	10.73

Table 6: Number and Percentage of z-Scores where |z| ≤2

analyte	number of scores where |z| ≤2	total number of scores	% |z| ≤2
beta-endosulfan	72	89	81
parathion (-ethyl)	59	73	81
pencyuron	55	67	82
phosalone	73	85	86
propyzamide	68	77	88
tecnazene	48	54	89
thiamethoxam	70	78	90

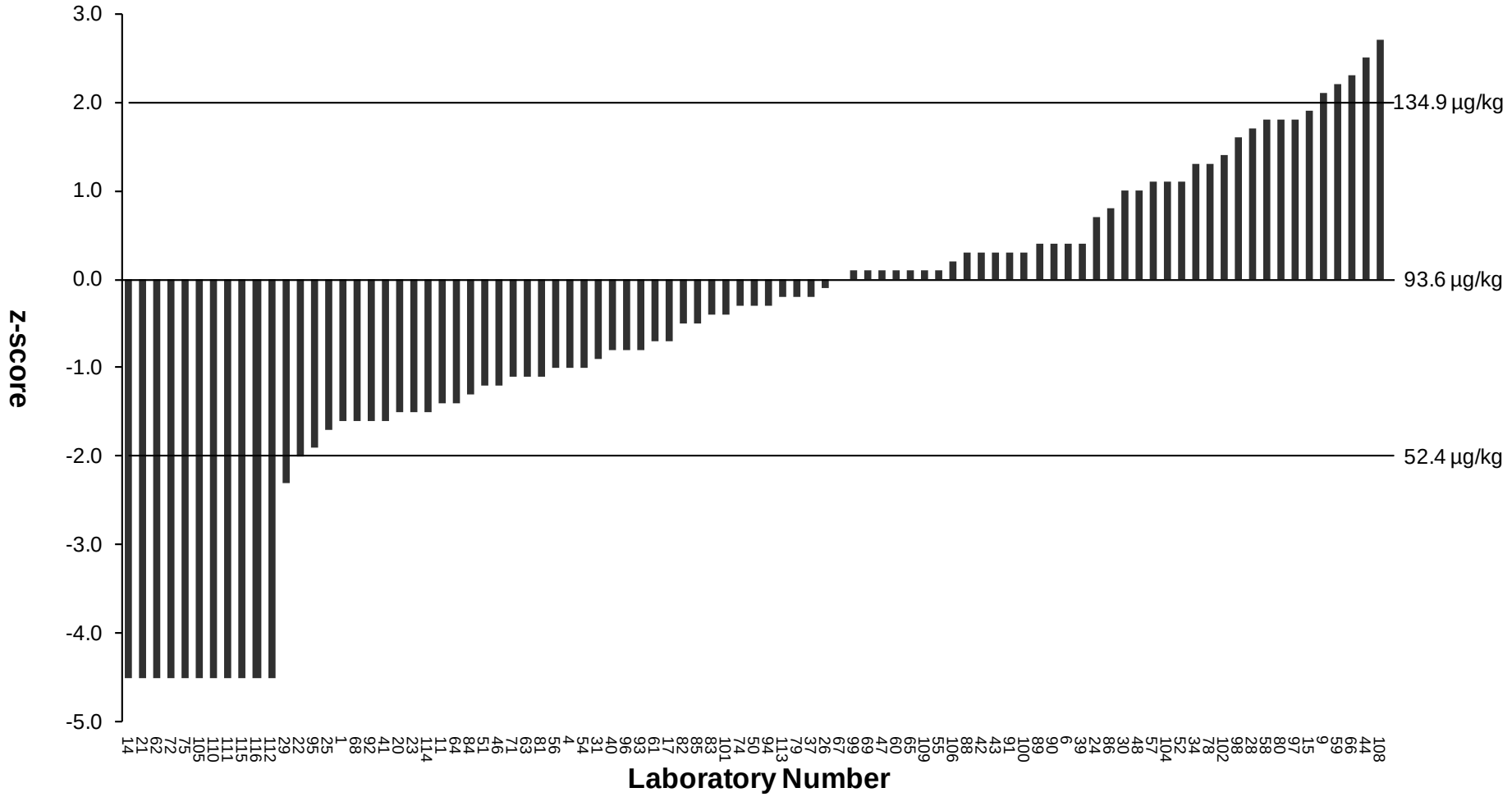

Figure 1: z-Scores for beta-Endosulfan

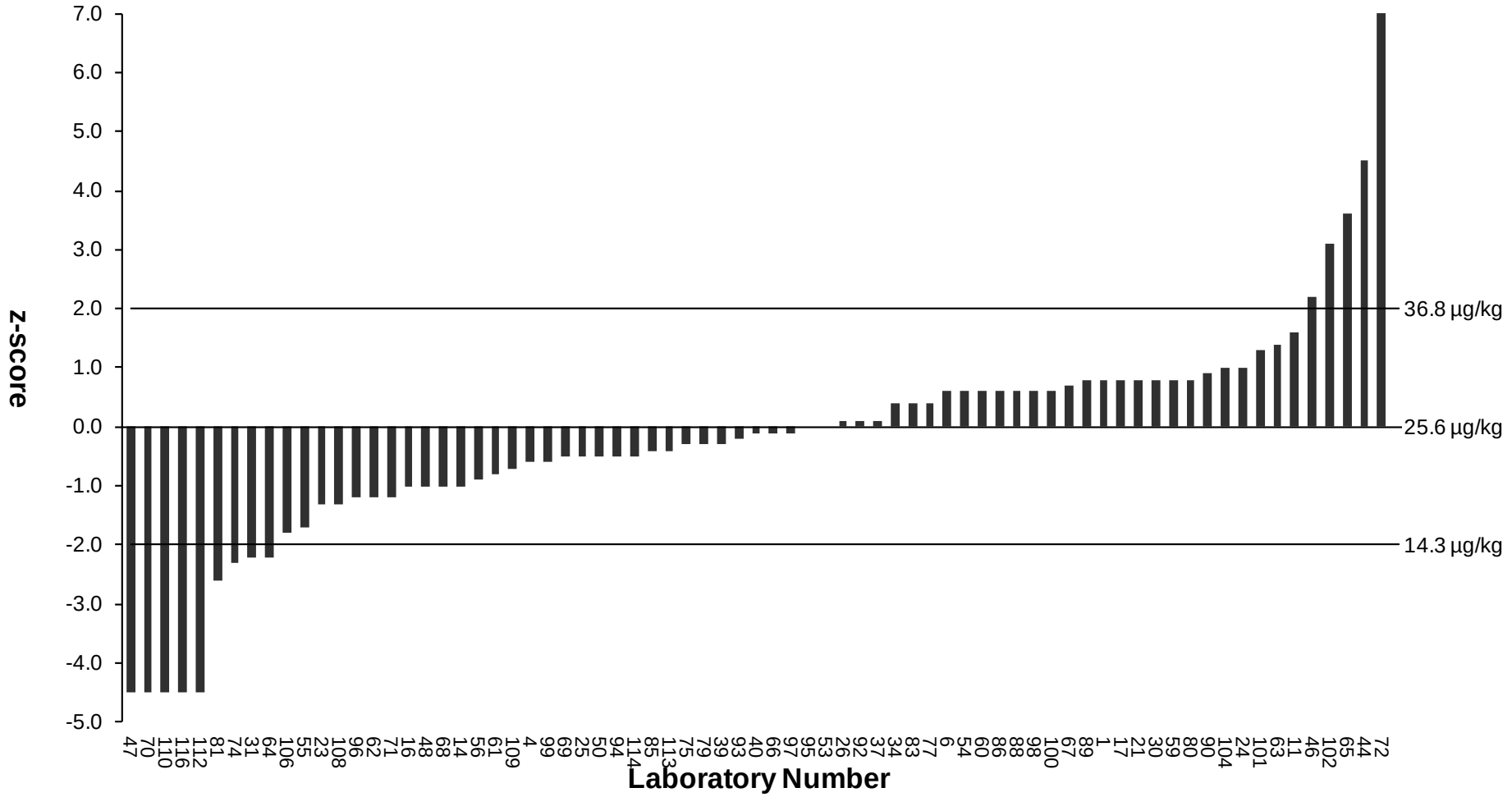

Figure 2: z-Scores for Parathion (-ethyl)

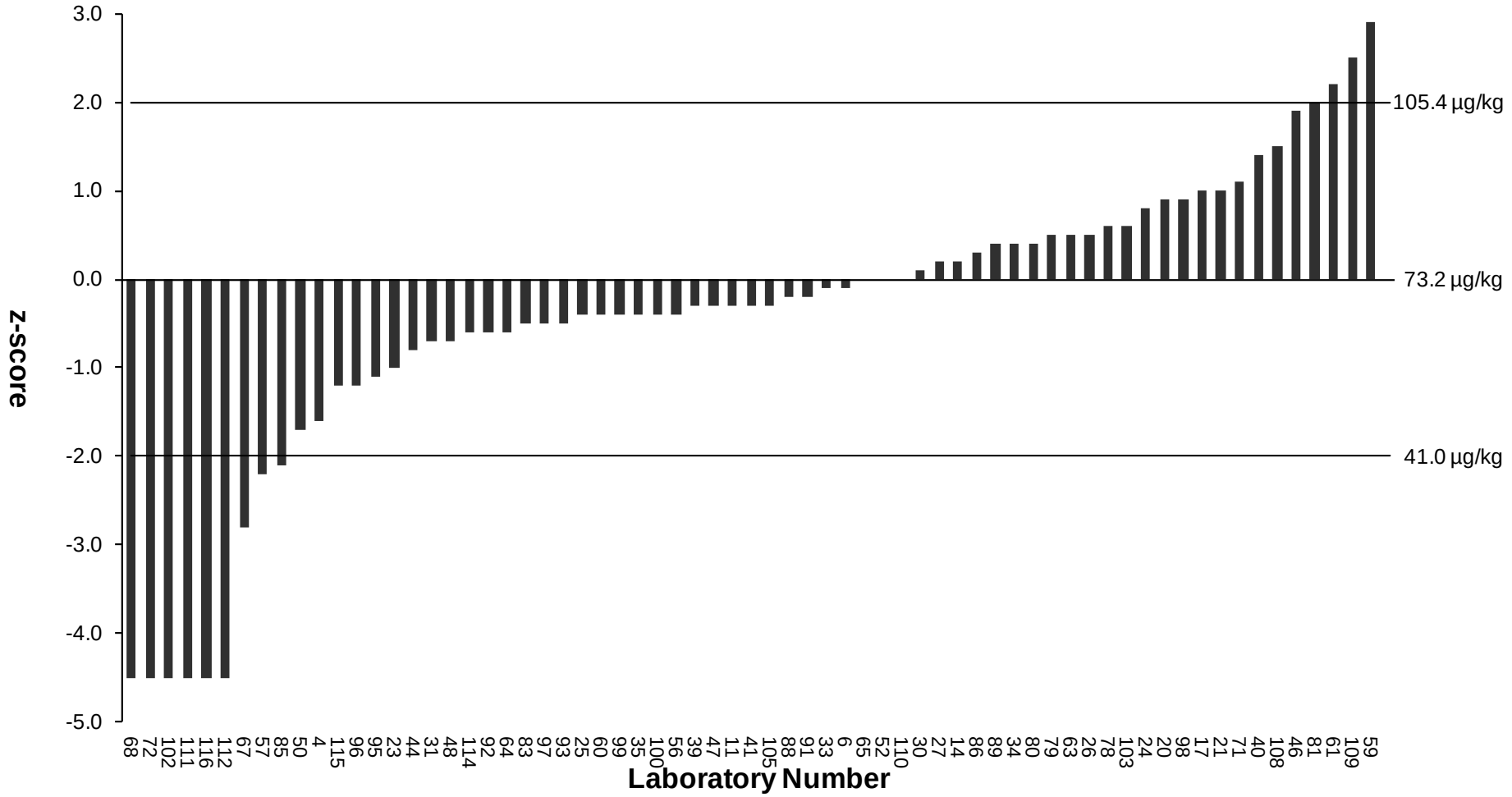

Figure 3: z-Scores for Pencyuron

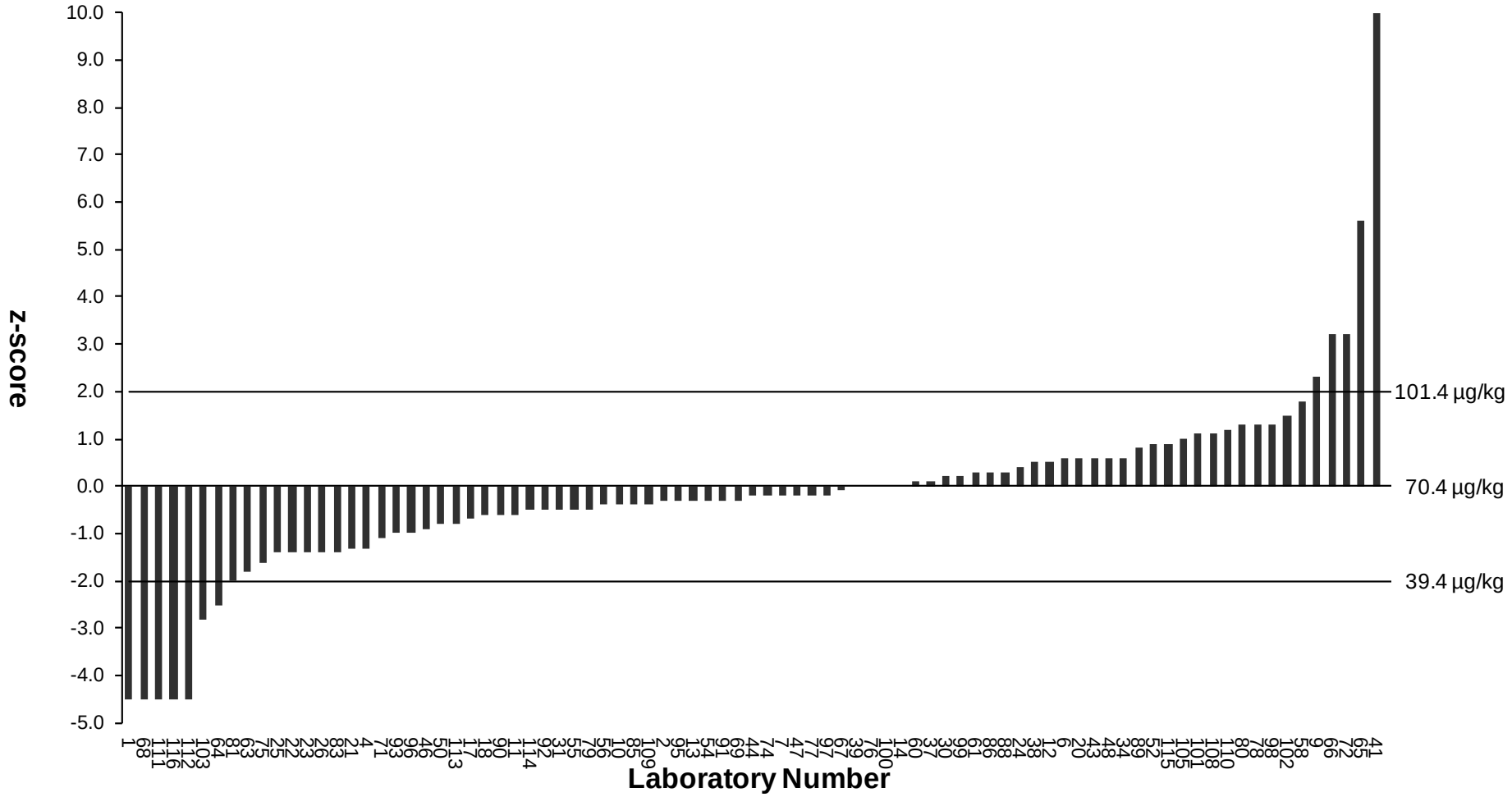

Figure 4: z-Scores for Phosalone

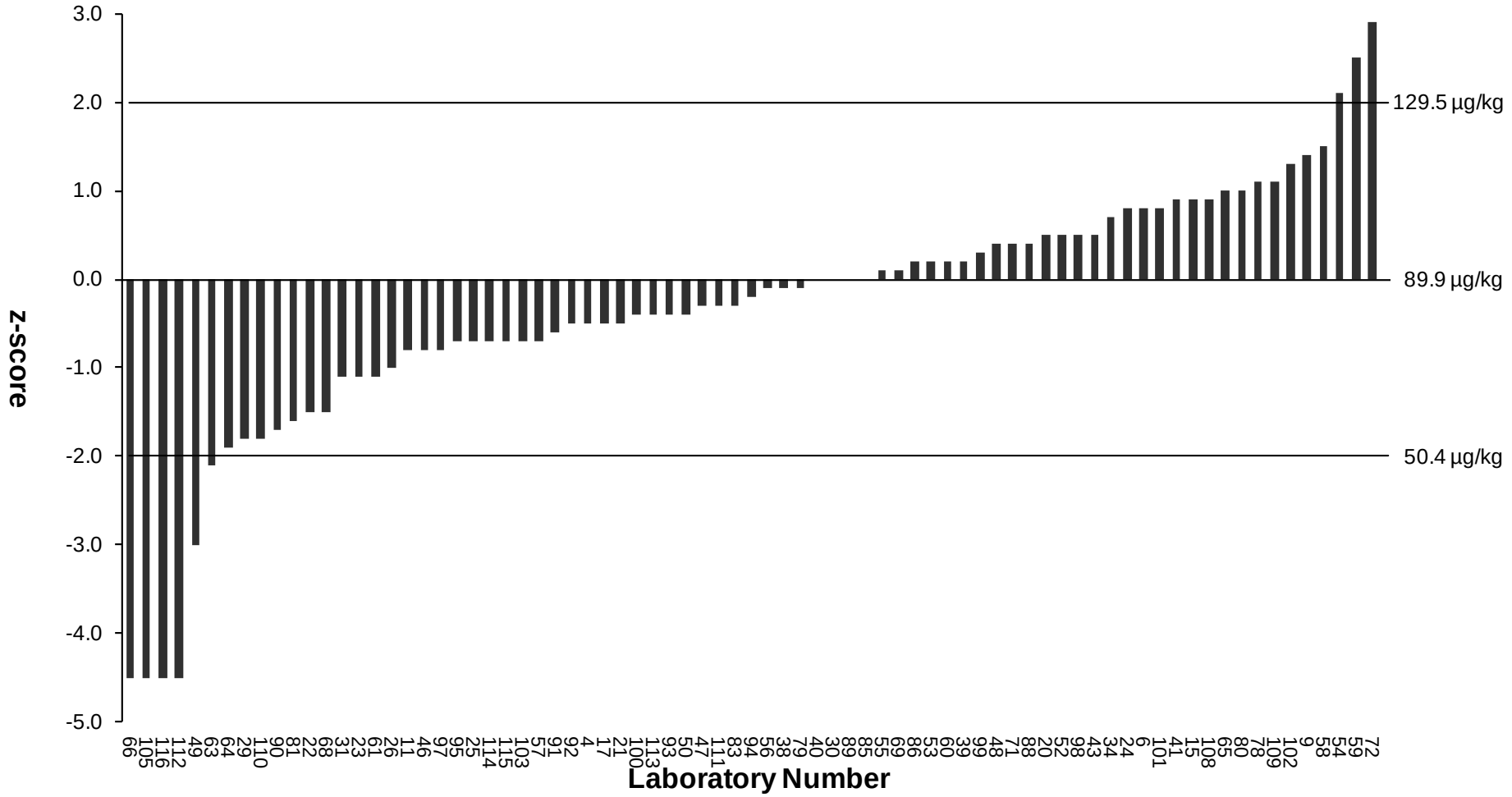

Figure 5: z-Scores for Propyzamide

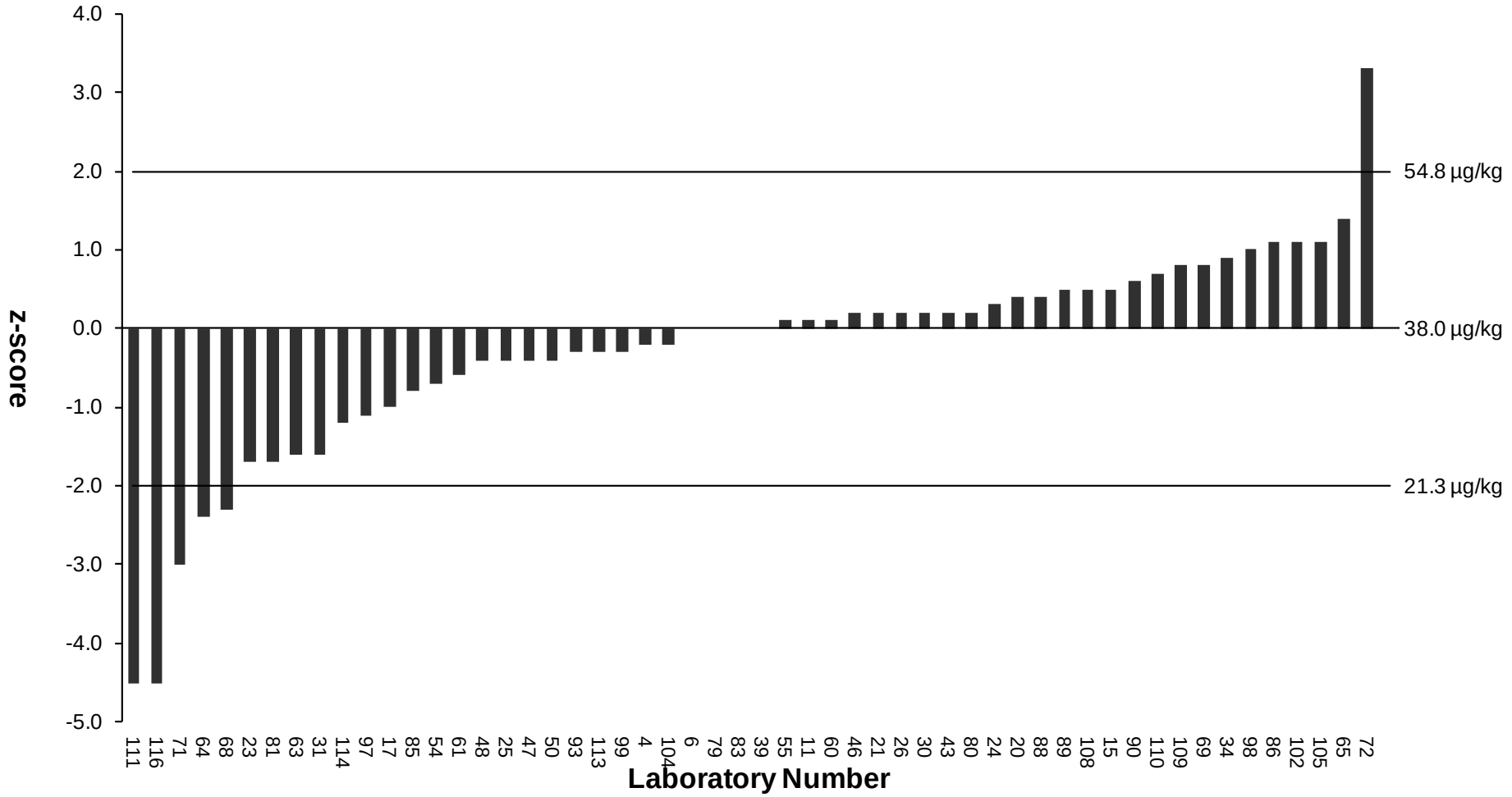

Figure 6: z-Scores for Tecnazene

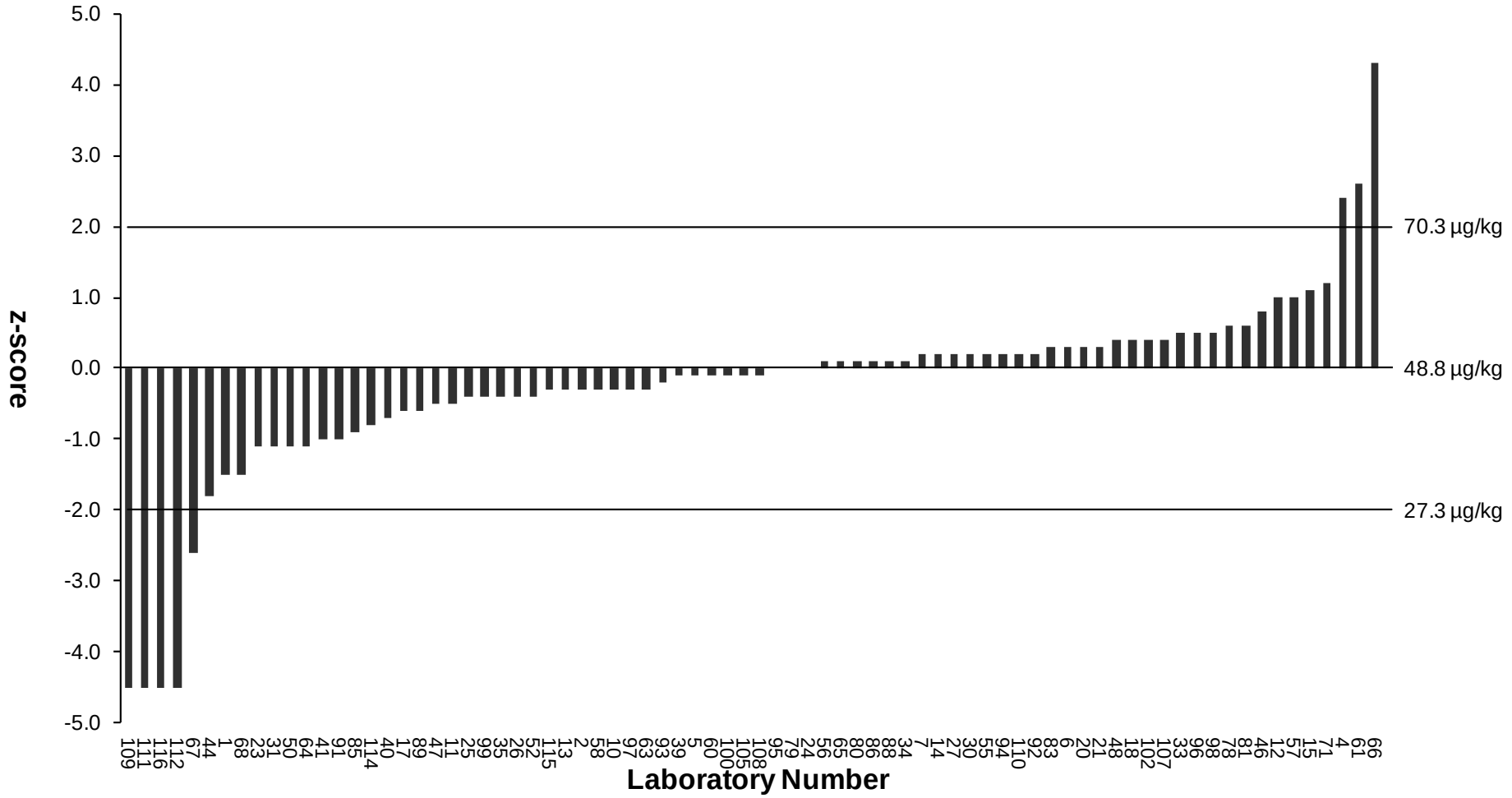

Figure 7: z-Scores for Thiamethoxam

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
yes	001 002 004 005 006 007 010 012 013 014 017 020 021 022 023 024 026 027 029 030 031 032 033 034 035 036 041 042 043 044 045 046 048 049 050 051 052 053 054 055 057 058 060 061 062 065 067 068 069 070 072 075 076 078 079 085 086 087 088 089 091 092 093 094 096 097 098 099 100 101 102 103 104 105 106 107 108 109 111 112 113 114
no	003 019 025 038 039 047 059 063 071 073 081 084 115

Quoted percentage recovery measured in same analytical batch as test material	laboratory number
yes	002 003 006 007 010 012 013 014 017 020 021 022 023 024 025 026 027 030 031 033 034 035 036 039 041 042 043 044 045 046 047 050 051 052 053 054 055 057 059 060 061 062 063 067 069 071 072 073 075 079 080 081 084 085 086 088 091 092 093 094 095 096 099 102 104 107 108 109 111 112 113
no	001 004 005 019 032 038 048 049 058 065 068 070 076 078 087 089 097 098 101 103 105 106 114 115

If measured in this batch, at what stage was the spike added	laboratory number
prior to extraction	002 003 006 007 010 012 013 014 017 020 021 022 023 024 025 026 027 030 031 032 033 034 035 036 039 042 043 044 045 046 047 048 050 051 052 053 054 055 057 059 060 061 062 063 065 067 069 071 072 073 075 078 079 080 081 084 085 086 091 092 093 094 095 096 099 102 104 107 108 109 111 112 113 115
prior to clean up	041 089
prior to instrument measurement	076

Concentration of Spike (mg/kg)	laboratory number
≥0.01 - <0.05	003 006 021 022 023 024 034 036 039 042 046 048 050 052 055 060 061 063 065 079 081 085 086 093 094 097 102 103 112 115
≥0.05 - <0.1	014 020 024 025 026 027 030 031 035 036 044 045 047 054 057 059 060 062 065 069 071 080 086 091 092 096 097 104 108 109 112 113
≥0.1 - <0.25	002 007 012 013 017 032 036 041 053 060 065 072 075 076 078 084 095 099 102 107 111
≥0.25 - <0.50	005 036 041 045 058 060 094
≥0.5 - <1	010 041 051 067 070
0.01 - 0.07	105
1 & 5	073

Composition of Blank Commodity used for Spiking	laboratory number
lettuce blank provided	003 006 010 014 023 024 026 027 031 032 033 034 035 036 039 042 043 046 048 050 052 053 054 055 057 058 060 063 069 071 073 078 079 080 081 085 086 088 091 092 095 096 100 102 103 104 107 111 113 115
lettuce test material provided	021 025 030 044 047 051 059 067 072 075 084 099 106 108
lettuce	002 007 012 013 022 065 076 109 112
mustard	070
in house blank	020 045 061 062 093 094
tested samples	041

Calibration	laboratory number
standard addition	002 004 007 010 012 013 014 033 049 050 053 072 085 089 094 095
matrix-matched	003 005 006 017 021 023 024 027 030 031 032 034 035 036 038 041 044 047 048 050 052 055 057 058 060 063 065 069 071 079 081 088 091 092 093 095 096 097 099 100 102 103 104 105 106 107 108 109 111 112 113 115
solvent	001 010 012 013 025 041 043 045 051 062 075 086 101
multi-level	001 007 010 012 013 017 020 021 022 023 026 029 030 032 034 039 041 042 044 045 050 055 057 058 059 061 062 063 067 069 071 073 075 076 078 079 080 084 087 094 098 101 107 112
single-level	046 070

Internal Standard Added	laboratory number
yes	002 006 007 010 012 013 017 019 021 023 024 026 029 030 031 035 036 042 046 052 053 054 055 059 065 072 076 086 088 091 093 094 097 098 099 100 101 102 104 105 106 107 109 111 112 113 115
no	001 003 004 005 014 020 022 025 027 032 033 034 038 039 041 043 044 045 047 048 049 050 051 057 058 060 061 062 063 067 068 069 070 071 073 075 078 079 080 081 084 085 087 089 092 095 096 103 108 114

Internal Standard Used	laboratory number
1-bromo-2-nitrobenzene	042
aldrin GC	017
bromophos methyl	021
d10-parathion	104
deuterated pirimicarb	024
ethion	101
ethoprofos	006
fenchlorphos	094
GC: hexaclorobenzene D	109
Isoprocarb LC	017
LC: parathion D-10	109
methamidophos D6	035
nicarbazin	024
PCB 31	065 105
PCB 209	059
pyrene D10	046
Quinalphos	006
tetraphenylethylene	093
TPP (Triphenyl Phosphate)	002 007 012 019 021 023 024 026 031 052 053 055 059 086 097 099 100 102 107 112 113 115
Triphenyl phenol	072

GC Method: Sample Weight (g)	laboratory number
≥1 - <5	029 032 069 072 080 092 115
≥5 - <10	020 021 022 039 044 047 048 050 055 059 068 095 102 106 108 114
≥10 - <20	001 002 004 006 014 017 019 023 024 025 026 038 043 046 052 053 057 058 060 061 063 065 070 071 076 079 081 084 085 086 088 089 094 096 097 098 099 100 103 104 109 111 112
≥20 - <50	007 010 012 013 030 031 034 041 042 045 051 062 067 073 075 078 087 091 093 101 113
10	105

GC Method: Extraction Solvent Components	laboratory number
acetic acid	070 104
acetone	006 014 026 029 032 038 041 057 061 063 073 075 078 087 096 101 112
acetonitrile	002 007 010 012 013 017 019 021 024 030 031 034 039 042 043 044 046 047 048 050 051 052 053 055 058 059 060 065 067 068 069 070 071 076 079 080 081 085 086 088 089 092 095 097 098 099 100 102 103 104 105 106 108 111 113 114 115
CO2	072
cyclohexane	004 057 063
dichloromethane	006 026 038 075 112
diethyl ether	051
ethyl acetate	001 004 020 022 023 025 032 045 057 062 063 073 091 093
hexane	073 084
methanol	094
petroleum ether (petroleum spirit)	006 026 038 042

GC Method: Extraction pH Adjusted	laboratory number
yes	002 007 012 024 039 046 062 069 080 081 086 091 097 098 102 108
no	001 004 006 010 013 014 017 019 020 021 022 023 025 026 029 030 031 032 034 038 041 042 043 044 045 047 048 050 051 052 053 055 057 058 059 060 061 063 065 067 068 070 071 072 073 075 076 078 079 084 085 087 088 089 092 093 094 095 096 099 100 101 103 104 105 106 109 111 112 113 114 115

GC Method: Extraction Techniques Used	laboratory number
macerate at room temperature	002 020 022 026 031 034 045 047 057 061 062 069 078 080 089 092 093 095 112 113
macerate at elevated temperature	067
QuEChERS	012 017 019 021 024 025 030 039 044 046 052 055 058 059 060 063 065 068 079 086 088 097 098 099 100 102 103 104 105 106 108 109 111 114 115
liquid-liquid partition	010 012 013 023 032 042 043 045 048 051 053 069 071 075 076 078 081 084 087 091 094
extraction at elevated pressure	001 004 014 029
cold solvent extraction at atmospheric pressure	007 041
high-speed homogenizer	085
JICA method	073
Mini-Luke	006 112
Modified QuEChERS	070
SFE	072
shake 30 min at room temperature	050
Solid/Liquid partition	038

GC Method: Sample Clean-up Technique	laboratory number
GPC/HPGPC	001 004 057 078 080 093
liquid-liquid partition	007 031 032 034 041 047 051 080 088 091 100 103 109
solid phase extraction (SPE) (column/cartridge)	002 007 010 012 013 014 029 030 031 039 042 045 047 048 050 053 058 062 067 069 071 072 073 075 076 080 081 085 089 094 095 099 101 111 113 115
solid phase extraction (SPE) (dispersive)	017 019 021 024 025 043 044 052 059 063 078 079 086 102 106 108 114
PSA	097
QuEChERS (PSA)	104
silica gel column chromatography	087
none	006 020 023 026 038 046 060 061 065 070 084 092 096 105 112

GC Method: SPE Sorbent Type	laboratory number
Bond Elut AccuCAT	067
C18	014 051 071 075 076 094 099 101 106
C18,PSA,PLS-3	039
Carbon/NH2	069
ENVICarb (GCB/NH2)	007 010 012 013 062 085
Envicarb/GCB	031 034 043 047 048 059 080 111
GCB/NH2	078 081 089
Florisil	042 062
Mixed Mode	030 050 072 096 103 113
NH2	002 053 068 092 095 114
PSA	019 021 024 025 029 045 052 058 063 086 088 091 098 100 102 108 109 115
PSA,GCB,MgSO4	017 044
SAX/PSA	073
none	026

GC Method: GC Column Packing	laboratory number
100% methyl polysiloxane	034
95% methyl 5% phenyl polysiloxane	002 004 006 007 010 012 013 014 019 020 021 023 024 025 026 030 038 039 041 043 044 045 047 048 050 052 057 059 060 061 062 063 065 067 069 070 071 072 076 078 079 080 081 084 085 086 087 088 089 091 092 094 096 097 100 101 102 104 106 108 109 111 112 115
65% methyl 35% phenyl polysiloxane	001 042 053 105
50% methyl 50% phenyl polysiloxane	051 055 058 073 093 095 103
14% cyanopropylphenyl 86% methylpolysiloxane	017 032 075
trifluoropropylmethyl polysiloxane	068
DB-1701p	113
VF-5ms	022

GC Method: GC Detector Type	laboratory number
ECD	001 045 051 057 058 059 060 062 067 073 075 095 101
MS	002 006 007 010 012 013 017 020 021 029 031 034 039 041 043 045 047 048 050 057 060 065 068 069 072 078 084 088 089 092 093 094 095 097 099 100 101 103 104 105 106 111 113 114
MS-MS	004 006 014 019 022 023 025 026 030 031 038 041 044 046 052 053 055 061 063 071 079 080 081 085 086 091 096 098 102 108 109 112 115
FPD	001 032 045 070 075 078 087
MSD	024
NPD	057 076 078
PFPD	062

LC Method: Sample Weight (g)	laboratory number
≥1 - <5	048 080 081 092 114 115
≥5 - <10	020 021 036 039 041 044 047 049 050 055 059 061 095 102 108
≥10 - <20	001 002 003 004 005 006 014 017 023 024 025 026 027 030 033 034 035 043 046 052 057 058 060 063 065 071 076 079 085 086 088 089 093 096 097 098 099 100 103 105 107 109 112
≥20 - <50	007 010 012 013 031 067 078 091 094 113

LC Method: Extraction Solvent Components	laboratory number
acetone	006 014 026 027 033 078 096 112
acetonitrile	002 003 005 007 010 012 013 017 020 021 023 024 030 031 035 036 039 041 043 044 046 047 049 050 052 055 057 058 059 060 061 063 065 067 071 076 079 080 081 085 086 088 089 092 093 094 095 097 098 099 100 102 103 105 108 109 113 114 115
dichloromethane	006 026 112
ethyl acetate	001 004 025 091
methanol	034 048
acetic acid	004 020
petroleum ether	006 026

LC Method: Extraction pH Adjusted	laboratory number
yes	002 007 012 023 024 031 035 039 046 080 081 086 091 094 097 098 102 107 108
no	001 003 004 005 006 010 013 014 017 020 021 025 026 027 030 033 034 036 041 042 043 044 047 048 049 050 052 057 058 059 060 061 063 065 067 071 076 078 079 085 088 089 092 093 095 096 099 100 103 105 109 112 113 114 115

LC Method: Extraction Techniques Used	laboratory number
macerate at room temperature	002 026 027 031 033 034 047 078 080 081 089 091 092 095 112 113
macerate at elevated temperature	067
QuEChERS	004 005 012 017 020 021 023 024 025 030 035 036 039 041 044 046 049 052 055 057 058 059 060 061 063 065 079 086 088 093 097 098 099 100 102 103 105 107 108 109 114 115
liquid-liquid partition	003 010 012 013 043 048 071 076 078 094
extraction at elevated pressure	001 014
cold solvent extraction at atmospheric pressure	007
high-speed homogenizer	085
Mini-Luke	006 112
shake 30 min at room temperature	050

LC Method: Sample Clean-up Technique	laboratory number
GPC/HPGPC	001 078 080
liquid-liquid partition	007 027 031 033 034 047 080 088 091 100 109
solid phase extraction (SPE) (column/cartridge)	002 003 007 010 012 013 014 030 031 039 047 050 058 067 071 076 080 081 085 089 095 099 103 113 115
solid phase extraction (SPE) (dispersive)	005 021 024 025 035 036 043 044 052 057 059 061 063 078 079 086 107 108 114
PSA	097
QuEChERS	041
none	004 006 017 020 023 026 046 048 049 060 065 092 093 096 105 112

LC Method: SPE Sorbent Type	laboratory number
C18	014 071
Envicarb/GCB	031 043 047 059 080
PSA	021 023 024 025 035 036 052 058 061 063 086 088 098 100 108 109 115
NH2	002 067 076 092 095 099
Mixed Mode	030 050 057 096 103 113
C18,PSA	039 081
ENVICarb (GCB/NH2)	007 010 012 013 085
GCB-NH2	003 078 089
PSA, GCB	005
PSA, Magnesium Sulphate, Carbon	044
none	017 026

LC Method: HPLC Column Packing	laboratory number
C18	001 002 003 004 005 006 007 010 012 013 014 017 020 023 026 027 030 031 033 034 035 036 039 041 043 044 046 047 048 049 050 052 055 057 058 059 061 063 065 067 071 076 078 079 080 081 085 086 089 091 092 093 095 096 097 099 102 103 105 107 108 109 112 113 114 115
C8	088 100
Mixed Mode	025
RP-Amide	060
Zorbax eclipped, C18	024

LC method: Mobile Phase Components	laboratory number
acetic acid	097
acetonitrile	001 003 021 026 049 050 059 067 071 076 078 085 086 088 094 099 102 109 115
ammonium acetate	002 005 006 007 010 012 013 017 021 027 030 033 039 047 050 079 081 086 096 107 113
ammonium formate	020 023 025 035 036 041 044 048 052 058 060 063 065 080 091 097 100 108 109 112
formic acid	004 005 021 023 030 031 036 041 048 050 052 055 060 063 065 071 078 080 086 091 094 095 105 107 108 112 115

LC method: Mobile Phase Components (continued)	laboratory number
methanol	002 004 005 006 007 010 012 013 014 017 020 023 024 027 030 031 033 034 035 036 039 041 043 044 046 047 048 052 055 057 058 060 061 063 065 079 080 089 091 092 093 095 096 097 103 107 108 112 114 115
water	001 004 005 006 007 012 013 014 020 021 023 024 026 031 033 034 036 041 046 047 048 049 050 052 055 057 058 059 060 063 065 067 076 078 079 080 086 091 094 096 097 102 107 108 115

LC Method: Detector Type	laboratory number
fluorescence	076
MS	002 007 012 013 043 078 092 108
MS-MS	001 003 004 005 006 010 014 017 020 021 023 024 025 026 027 030 031 033 034 035 036 039 041 044 046 047 048 049 050 052 055 057 058 060 061 063 065 071 079 080 081 085 086 088 089 091 093 094 095 096 097 098 099 100 102 103 105 107 109 112 113 114 115
UV/Vis	059
UV and FLD	067

beta-Endosulfan

Method Principle	laboratory number
GC	001 004 006 017 020 022 023 024 025 026 029 030 031 034 039 041 042 043 044 046 047 048 050 051 052 054 055 057 058 059 060 061 063 065 067 068 069 071 078 079 080 081 082 084 085 086 088 089 091 092 093 094 095 096 097 100 101 102 104 106 108 109 112 113

Identification by Mass Spectrometry	laboratory number
yes	004 006 017 020 022 023 024 025 026 029 030 031 034 039 041 042 043 044 046 047 048 050 052 054 055 057 058 060 061 063 065 067 068 069 071 078 079 080 081 084 085 086 088 089 091 092 093 094 095 096 097 100 101 102 104 106 108 109 112 113
no	001 051 059 082

Parathion (-ethyl)

Method Principle	laboratory number
GC	001 004 006 014 016 017 021 023 024 025 026 030 031 032 034 044 046 048 050 053 054 055 059 061 062 065 067 068 069 071 072 075 079 080 081 085 086 088 089 092 093 094 095 096 099 100 101 102 104 106 108 109 112 113
LC	039 060 063 097

Identification by Mass Spectrometry	laboratory number
yes	004 006 014 016 017 021 023 024 025 026 030 031 034 039 044 046 048 050 053 054 055 060 061 063 065 067 068 069 071 072 079 080 081 085 086 088 089 092 093 094 095 096 097 099 100 101 104 106 108 109 112 113
no	001 032 059 062 075 102

Pencyuron

Method Principle	laboratory number
GC	017 041 059 078 092
LC	004 006 014 020 021 023 024 025 026 030 031 033 034 035 039 044 046 047 048 050 052 057 060 061 063 065 067 071 079 080 081 085 086 088 089 091 093 095 096 097 099 100 103 105 109 112 115

Identification by Mass Spectrometry	laboratory number
yes	004 006 014 017 020 021 023 024 025 026 030 031 033 034 035 039 041 044 046 047 048 050 052 057 060 061 063 065 067 071 078 079 080 081 085 086 088 089 091 092 093 095 096 097 099 100 103 105 109 112 115
no	059

Phosalone

Method Principle	laboratory number
GC	002 004 007 010 012 013 017 020 021 022 023 024 025 026 030 034 038 041 043 046 048 050 052 054 055 063 065 067 069 071 072 075 076 078 079 080 081 085 086 089 091 092 093 095 101 102 105 109 112 113
LC	006 014 031 039 044 047 058 060 061 088 096 097 100 103 108 115

Identification by Mass Spectrometry	laboratory number
yes	002 004 006 007 010 012 013 014 017 020 021 022 023 024 025 026 030 031 034 038 039 041 043 044 046 047 048 050 052 054 055 058 060 061 063 065 067 069 071 072 078 079 080 081 085 086 088 089 091 092 093 095 096 097 100 101 103 105 108 109 112 113 115
no	075 076 102

Propyzamide

Method Principle	laboratory number
GC	004 017 020 021 022 023 026 029 030 031 034 038 041 043 046 047 048 050 052 053 054 055 059 061 063 065 068 069 072 078 079 080 081 085 089 091 092 094 095 101 111 112 113
LC	006 024 025 039 049 057 058 060 071 086 088 093 097 100 102 103 108 109 115

Identification by Mass Spectrometry	laboratory number
yes	004 006 017 020 021 022 023 024 025 026 029 030 031 034 038 039 041 043 046 047 048 049 050 052 053 054 055 057 058 060 061 063 065 068 069 071 072 078 079 080 081 085 086 088 089 091 092 093 094 095 097 100 101 102 103 108 109 111 112 113 115
no	059

Tecnazene

Method Principle	laboratory number
GC	004 006 020 021 023 024 025 026 030 031 034 039 043 046 047 048 050 054 055 060 061 063 065 068 069 071 072 079 080 081 085 086 088 089 093 097 102 104 105 108 109 113

Identification by Mass Spectrometry	laboratory number
yes	004 006 020 021 023 024 025 026 030 031 034 039 043 046 047 048 050 054 055 060 061 063 065 068 069 071 072 079 080 081 085 086 088 089 093 097 102 104 105 108 109 113

Thiamethoxam

Method Principle	laboratory number
GC	068 089 099
LC	001 002 004 005 006 007 010 012 013 014 017 020 021 023 024 025 026 030 031 033 034 035 039 041 044 046 047 048 050 052 055 057 058 060 061 063 065 067 071 078 079 080 081 085 086 088 091 092 093 094 095 096 097 100 102 105 107 108 112

Identification by Mass Spectrometry	laboratory number
yes	001 002 004 005 006 007 010 012 013 014 017 020 021 023 024 025 026 030 031 033 034 035 039 041 044 046 047 048 050 052 055 057 058 060 061 063 065 067 068 071 078 079 080 081 085 086 088 089 091 092 093 094 095 096 097 099 100 102 105 107 108 112 115

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 and quality control materials.
- Freely download copies of reports (PDF file), of proficiency tests in which they have participated.

2. PROTOCOL

The Protocols [3, 4] set out how FAPAS® is organised. Copies can be downloaded from our website.

3. CONTACT DETAILS

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

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