FAPAS® Report 19205

Pesticide Residues in Pea (with pods) Purée

February-April 2016

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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UKAS PROFICIENCY TESTING
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SUMMARY

1. The test material for FAPAS® proficiency test 19205 was dispatched in February 2016. Each participant received a pea (with pods) purée test material.
2. From a list of 213 pesticide residues, participants had to identify and quantify those present. The test material contained azoxystrobin, cyhalothrin-lambda, dimethoate, fenvalerate, methiocarb, permethrin, pirimicarb, tebuconazole and dithiocarbamates.
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.
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
azoxystrobin	117	72	77	94
cyhalothrin-lambda	36.8	58	74	78
dimethoate	173	67	76	88
fenvalerate	78.5	68	77	88
methiocarb	152	64	71	90
permethrin	115	66	75	88
pirimicarb	159	65	71	92
tebuconazole	77.6	74	77	96
dithiocarbamates	297	38	63	60

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 fresh peas, 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.

Azoxystrobin, cyhalothrin-lambda, dimethoate, fenvalerate, methiocarb, permethrin, pirimicarb, tebuconazole and dithiocarbamates were spiked into the test material.

No other residues were detected at, or above, 30 µg/kg.

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 February 2016. Test materials were sent to 91 participants.

3. RESULTS

The instructions for reporting results were as follows:

1) Determine the concentration 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.

- 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 87 participants (96%) before the closing date for this test, 7 April 2016.

Each participant was given a laboratory number, assigned in order of receipt of results. The reported analyte concentrations are given in Table 1 for azoxystrobin, cyhalothrin-lambda and dimethoate, in Table 2 for fenvalerate, methiocarb and permethrin and in Table 3 for pirimicarb, tebuconazole and dithiocarbamates.

If a participant analysed for a pesticide residue that was in the test material, but did not identify it, and their LoQ (limit of quantification) was below the level needed for a z-score of -3.0 or an LoQ was not reported, 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.

Additional pesticides reported by participants, other than azoxystrobin, cyhalothrin-lambda, dimethoate, fenvalerate, methiocarb, permethrin, pirimicarb, tebuconazole and dithiocarbamates are shown in Table 4.

Participants' comments are given in Table 5.

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) 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% [6],

v) results where no limit of quantification was reported.

For azoxystrobin, cyhalothrin-lambda, dimethoate, fenvalerate, methiocarb, permethrin, pirimicarb, tebuconazole and dithiocarbamates, this procedure was straightforward and the robust mean was chosen as the assigned value.

The assigned values for all analytes are shown in Table 6.

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 6.

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 azoxystrobin, cyhalothrin-lambda, dimethoate are given in Table 1 and shown as histograms in Figures 1–3. Participants' z-scores for fenvalerate, methiocarb and permethrin are given in Table 2 and shown as histograms in Figures 4–6. Participants' z-scores for pirimicarb, tebuconazole and dithiocarbamates are given in Table 3 and shown as histograms in Figures 7–9. 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 6.

The number and percentage of z-scores in the range $-2 \leq z \leq 2$ for all analytes are given in Table 7.

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/misc/pki/cds_cp.html, accessed 10/05/2015.

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

3 FAPAS, 2014, Protocol for Proficiency Testing Schemes, Part 1 – Common Principles, Version 4, Issued May 2014.

4 FAPAS, 2014, Protocol for Proficiency Testing Schemes, Part 2 – FAPAS®, Version 3, Issued May 2014.

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 Analytical Quality Control and Validation Procedures for Pesticide Residues Analysis in Food and Feed, Document No. SANTE/11945/2015.

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 Azoxystrobin, Cyhalothrin-Lambda and Dimethoate

laboratory number	analyte											
	azoxystrobin assigned value 117 µg/kg				cyhalothrin-lambda assigned value 36.8 µg/kg				dimethoate assigned value 173 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	95.6	98	10	-0.8	37.1	103	10	0.0	183.2	107	10	0.3
002	85	90	10	-1.3	30	88	10	-0.8	172	117	20	0.0
003	87		10	-1.2	30		10	-0.8	145		10	-0.8
004	87		10	-1.2	30		10	-0.8	145		10	-0.8
005	132	93	10	0.6	40	99	10	0.4	194	108	10	0.6
006	131	100	10	0.5	39.7	100	20	0.4	157	100	10	-0.4
007	92	86	10	-1.0	39	93	10	0.3	162	91	10	-0.3
008	97	89	10	-0.8	40	102	50	0.4	156	89	10	-0.5
009	87.8	94.74	10	-1.1	70.2	105.12	10	**4.1**	165	93.02	10	-0.2
010	117	102	10	0.0	38	90	10	0.2	136	115	10	-1.0
011	91	92	10	-1.0	32	112	10	-0.6	183	90	10	0.3
012	110	86	10	-0.3	55	96	10	**2.3**	163	100	10	-0.3
013	102	105	10	-0.6	0	89	10	**-4.5**	167	85	10	-0.2
014	119	103	10	0.1	38	89	10	0.2	192	92	10	0.5
015	118.3	106.5	10	0.0	50.4	115.4	10	1.7	172.8	96.2	10	0.0

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

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

laboratory number	analyte											
	azoxystrobin assigned value 117 µg/kg				cyhalothrin-lambda assigned value 36.8 µg/kg				dimethoate assigned value 173 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
016	66.0			-2.0	27.0			-1.2	146.0			-0.7
017	120	95	10	0.1	33	83	10	-0.5	205	92	10	0.9
018	#				#				#			
019	110	85	10	-0.3	34	80	10	-0.3	172	90	10	0.0
020	170		10	2.0	60		10	**2.9**	200		10	0.8
021	86.00	108.04	10.00	-1.2	37.00	99.00	10.00	0.0	160.00	89.00	10.00	-0.4
022	101	90	0.01	-0.6	34	124	0.01	-0.3	158	84	0.01	-0.4
023	138	100	10	0.8	0			**-4.5**	171	100	5	0.0
024	82.1	87.5	10	-1.4	36.9	119.0	10	0.0	174	89.0	10	0.0
025	122	84	5	0.2	41	102	5	0.5	196	97	5	0.6
026	#				#				#			
027	116.50		10	0.0	33.60		10	-0.4	130.47		10	-1.2
028	124	109	10	0.3	41	103	20	0.5	151	88	10	-0.6
029	154	100	10	1.4	36	100	10	-0.1	214	100	10	1.1
030	92.06	87	8.5	-1.0	12.71	132	5	**-3.0**	93	67.9	10	**-2.2**

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

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

laboratory number	analyte											
	azoxystrobin assigned value 117 µg/kg				cyhalothrin-lambda assigned value 36.8 µg/kg				dimethoate assigned value 173 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
031	#				34	85.29	20	-0.3	#			
032	0.122	102	0.005	**-4.5**	0.035	99	0.005	**-4.5**	0.170	97	0.005	**-4.8**
033	98.6965	88.0	10	-0.7	#				149.10065	87.8	10	-0.7
034	121	108	10	0.1	53	105	10	2.0	193	105	10	0.6
035	128.49		10	0.4	21.24		10	-1.9	0			**-4.8**
036	109	100	10	-0.3	36	90	10	-0.1	142	108	10	-0.9
037	#				#				#			
038	116.9	104	10	0.0	39.7	106	10	0.4	197.5	93	10	0.7
039	129	78	10	0.5	39	130	10	0.3	#			
040	#				#				#			
041	141	105	10	0.9	#				#			
042	115	96	10	-0.1	32	68	10	-0.6	0			**-4.8**
043	119	114	10	0.1	#				174	110	10	0.0
044	100	100	10	-0.7	#				181	100	10	0.2
045	79.4			-1.5	43.338			0.8	129.22			-1.2

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

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

laboratory number	analyte											
	azoxystrobin assigned value 117 µg/kg				cyhalothrin-lambda assigned value 36.8 µg/kg				dimethoate assigned value 173 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
046	144.7	110.6	10	1.1	36.5	102.9	10	0.0	180.4	117.2	10	0.2
047	110.5	100	25	-0.3	0			**-4.5**	114.63	100	25	-1.6
048	97	100	10	-0.8	32.5	100	10	-0.5	175	97	10	0.1
049	169	99.5	10	2.0	39	98.2	10	0.3	252	98	10	**2.2**
050	#				#				#			
051	113.1	107	10	-0.2	38.27	72	10	0.2	144.08	89	10	-0.8
052	119.6	98	10	0.1	34.9	83	10	-0.2	137.9	93	10	-1.0
053	107	85	10	-0.4	30.8	88	10	-0.7	152	86	10	-0.6
054	#				#				#			
055	136	100	10	0.7	31	101	10	-0.7	188	97	10	0.4
056	117	105	10	0.0	39	105	10	0.3	193	90	10	0.6
057	124	100	10	0.3	21	158	10	-1.9	195	87	10	0.6
058	117.1	97	10	0.0	0	112	10	**-4.5**	146.5	91	10	-0.7
059	250	94	10	**5.1**	#				240	73	10	1.9
060	114		10	-0.1	35		10	-0.2	125		10	-1.3

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

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

laboratory number	analyte											
	azoxystrobin assigned value 117 µg/kg				cyhalothrin-lambda assigned value 36.8 µg/kg				dimethoate assigned value 173 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
061	121	97	10	0.1	37.1	95	10	0.0	143	89	10	-0.8
062	114	91	10	-0.1	#				160	86	10	-0.4
063	185	95	10	**2.6**	67	86	10	**3.7**	160	92	10	-0.4
064	106	89	10	-0.4	35	94	10	-0.2	140	98	10	-0.9
065	121	97	10	0.1	35	95	10	-0.2	180	102	10	0.2
066	#				38		10	0.2	123		100	-1.4
067	133	100	10	0.6	33	100	10	-0.5	206	100	10	0.9
068	206.77			**3.5**	0			**-4.5**	343.11			**4.7**
069	139.3	85.2	10	0.9	43.4		10	0.8	205.6	91.5	10	0.9
070	124	101	10	0.3	44	101	20	0.9	139	98	10	-0.9
071	#				32.4	101.6	10	-0.5	#			
072	#				#				#			
073	135	100	10	0.7	44	100	10	0.9	188	100	10	0.4
074	140		0.01	0.9	65		0.01	**3.5**	400		0.01	**6.3**
075	0.101			**-4.5**	0.039			**-4.5**	0.157			**-4.8**

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

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

laboratory number	analyte											
	azoxystrobin assigned value 117 µg/kg				cyhalothrin-lambda assigned value 36.8 µg/kg				dimethoate assigned value 173 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
076	116	115	10	0.0	36	96	10	-0.1	162	96	10	-0.3
077	120	100	10	0.1	34	101	10	-0.3	175	97	10	0.1
078	120	103	10	0.1	77	95	10	**5.0**	415	89	10	**6.7**
079	140	94	10	0.9	44	100	10	0.9	150	92	10	-0.6
080	120	99	10	0.1	38.5	108	10	0.2	163	107	10	-0.3
081	105	94	10	-0.5	28.3	112	10	-1.0	169	90	10	-0.1
082	111	100	10	-0.2	35	111	10	-0.2	197	111	10	0.7
083	106	112	10	-0.4	24	92	10	-1.6	134	89	10	-1.1
084	127.85	96	10	0.4	23.23	84	10	-1.7	187.19	104	10	0.4
085	115	95.0	10	-0.1	29	72.9	10	-1.0	201	102	10	0.8
086	132	112	0.01	0.6	0			**-4.5**	223	83	0.01	1.4
087	130.5	101	10	0.5	0		10	**-4.5**	180	100	10	0.2

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

Table 2: Results and z-Scores for Fenvalerate, Methiocarb and Permethrin

laboratory number	analyte											
	fenvalerate assigned value 78.5 µg/kg				methiocarb assigned value 152 µg/kg				permethrin assigned value 115 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	74.8	91	10	-0.2	145.8	101	10	-0.2	106.1	92	10	-0.4
002	65	97	10	-0.8	119	108	10	-1.0	93	105	10	-0.9
003	64		10	-0.8	96		10	-1.7	90		10	-1.0
004	64		10	-0.8	96		10	-1.7	90		10	-1.0
005	84	101	10	0.3	176	101	10	0.8	108	103	10	-0.3
006	107	100	20	1.7	134	100	10	-0.5	178	100	10	**2.5**
007	77	89	10	-0.1	146	90	10	-0.2	107	86	10	-0.3
008	65	109	30	-0.8	147	97	10	-0.1	#			
009	96.5	91.93	10	1.0	126.6	95.62	10	-0.8	119.5	95.56	10	0.2
010	79	105	10	0.0	184	113	10	1.0	119	83	10	0.2
011	75	113	10	-0.2	148	116	10	-0.1	98	109	10	-0.7
012	70	115	10	-0.5	115	98	10	-1.1	118	108	10	0.1
013	75	102	10	-0.2	90	90	10	-1.9	90	89	10	-1.0
014	68	80	10	-0.6	160	101	10	0.3	108	95	10	-0.3
015	93.8	108.1	10	0.9	142.5	98.1	10	-0.3	113.5	107.7	10	-0.1

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

Table 2 (continued): Results and z-Scores for Fenvalerate, Methiocarb and Permethrin

laboratory number	analyte											
	fenvalerate assigned value 78.5 µg/kg				methiocarb assigned value 152 µg/kg				permethrin assigned value 115 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
016	28.0			**-2.9**	44.5			**-3.3**	136.0			0.8
017	75	100	10	-0.2	168	80	10	0.5	120	102	10	0.2
018	#				#				#			
019	79	85	10	0.0	151	85	10	0.0	188	90	10	**2.9**
020	106		10	1.6	340		10	**5.8**	0			**-4.5**
021	118.56		10.00	**2.3**	108.00	75.00	10.00	-1.4	117.00	98.00	10.00	0.1
022	62	98	0.02	-1.0	121	87	0.01	-1.0	112	91	0.02	-0.1
023	67.2	100	10	-0.7	0			**-4.7**	121	100	10	0.2
024	77.3	111.8	10	-0.1	138	86.0	10	-0.4	112	113.4	10	-0.1
025	101	90	5	1.3	153	99	5	0.0	113	90	5	-0.1
026	#				#				#			
027	162.68		10	**4.9**	125.70		10	-0.8	#			
028	103	123	10	1.4	140	106	10	-0.4	0			**-4.5**
029	76	100	10	-0.1	200	100	10	1.5	121	100	10	0.2
030	87.16	93	9.9	0.5	191.5	115.2	10	1.2	0			**-4.5**

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

Table 2 (continued): Results and z-Scores for Fenvalerate, Methiocarb and Permethrin

laboratory number	analyte											
	fenvalerate assigned value 78.5 µg/kg				methiocarb assigned value 152 µg/kg				permethrin assigned value 115 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
031	128	96.78	20	**2.9**	#				98	79.87	20	-0.7
032	0.077	96	0.005	**-4.5**	0.134	92	0.005	**-4.7**	0.128	101	0.005	**-4.5**
033	#				#				#			
034	77	119	10	-0.1	170	104	10	0.6	127	94	10	0.5
035	81.90		10	0.2	#				114.48		10	0.0
036	75	96	10	-0.2	138	88	10	-0.4	111	99	10	-0.2
037	#				#				#			
038	71.6	113	10	-0.4	193.3	88	10	1.3	101.0	98	10	-0.6
039	69	104	10	-0.5	#				123	107	10	0.3
040	#				#				#			
041	#				153	101	10	0.0	#			
042	47	67	10	-1.8	#				89	72	10	-1.0
043	86	110	10	0.4	162	107	10	0.3	110	110	10	-0.2
044	75.2	100	10	-0.2	132	100	10	-0.6	#			
045	84.47			0.3	106.52			-1.4	97.097			-0.7

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

Table 2 (continued): Results and z-Scores for Fenvalerate, Methiocarb and Permethrin

laboratory number	analyte											
	fenvalerate assigned value 78.5 µg/kg				methiocarb assigned value 152 µg/kg				permethrin assigned value 115 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
046	88.1	109.7	10	0.6	198.1	97.0	10	1.4	91.4	73.9	10	-0.9
047	62.94	100	25	-0.9	92.10	100	25	-1.8	93.87	100	25	-0.8
048	81.6	100	10	0.2	128	99	10	-0.7	100	113	10	-0.6
049	102	100.5	10	1.4	198	99.4	10	1.4	105	99	10	-0.4
050	#				#				#			
051	90.36	91	10	0.7	163.16	96	10	0.4	109.13	116	10	-0.2
052	66.5	84	10	-0.7	157.5	90	10	0.2	100.4	79	10	-0.6
053	49.8	109	10	-1.7	71.9	90	10	**-2.5**	107	93	10	-0.3
054	#				#				#			
055	77	96	10	-0.1	190	101	10	1.2	127	98	10	0.5
056	80	102	10	0.1	157	85	10	0.2	128	107	10	0.5
057	#				168	92	10	0.5	123	128	10	0.3
058	77.0	111	10	-0.1	144.0	96	10	-0.2	134.7	88	50	0.8
059	#				#				250	76	10	**5.3**
060	59		10	-1.1	150		20	-0.1	96		10	-0.8

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

Table 2 (continued): Results and z-Scores for Fenvalerate, Methiocarb and Permethrin

laboratory number	analyte											
	fenvalerate assigned value 78.5 µg/kg				methiocarb assigned value 152 µg/kg				permethrin assigned value 115 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
061	74.2	93	10	-0.2	149	91	10	-0.1	115	93	10	0.0
062	54	86	10	-1.4	136	87	10	-0.5	98	84	10	-0.7
063	122	102	10	**2.5**	134	105	10	-0.5	141	87	10	1.0
064	98	75	10	1.1	160	61	10	0.3	147	86	10	1.3
065	75	105	10	-0.2	125	101	10	-0.8	110	93	10	-0.2
066	62		10	-1.0	#				89		10	-1.0
067	72	100	10	-0.4	210	100	10	1.8	125	100	10	0.4
068	0			**-4.5**	404.26			**7.8**	0			**-4.5**
069	75.7		10	-0.2	169.9	92.6	10	0.6	112	82.3	10	-0.1
070	75	94	20	-0.2	94	97	10	-1.8	101	102	20	-0.6
071	66.3	106.1	10	-0.7	#				98.9	100.5	10	-0.6
072	56	80	10	-1.3	#				#			
073	97	100	10	1.1	166	100	10	0.4	137	100	10	0.9
074	78		0.01	0.0	140		0.01	-0.4	134		0.01	0.7
075	0.048			**-4.5**	0.101			**-4.7**	0.093			**-4.5**

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

Table 2 (continued): Results and z-Scores for Fenvalerate, Methiocarb and Permethrin

laboratory number	analyte											
	fenvalerate assigned value 78.5 µg/kg				methiocarb assigned value 152 µg/kg				permethrin assigned value 115 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
076	77	88	10	-0.1	165	95	10	0.4	103	98	10	-0.5
077	76	96	10	-0.1	176	101	10	0.8	119	98	10	0.2
078	82	87	10	0.2	156	109	10	0.1	141	89	10	1.0
079	83	103	10	0.3	160	94	10	0.3	120	114	10	0.2
080	75.9	99	10	-0.1	134	93	10	-0.5	121	104	10	0.2
081	57.4	112	10	-1.2	139	84	10	-0.4	85.3	117	10	-1.2
082	93	100	10	0.8	149	111	10	-0.1	103	100	10	-0.5
083	57	101	10	-1.2	#				86	99	10	-1.1
084	81.16	95	10	0.2	184.93	98	10	1.0	143.57	135	10	1.1
085	69	87.5	10	-0.5	126	109	10	-0.8	159	97.9	10	1.7
086	111	72	0.01	1.9	166	115	0.01	0.4	163	116	0.01	1.9
087	126	83	10	**2.8**	166	98	10	0.4	127	101	10	0.5

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

Table 3: Results and z-Scores for Pirimicarb, Tebuconazole and Dithiocarbamates

laboratory number	analyte											
	pirimicarb assigned value 159 µg/kg				tebuconazole assigned value 77.6 µg/kg				dithiocarbamates assigned value 297 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	167.8	100	10	0.3	80.1	100	10	0.1	#			
002	152	122	10	-0.2	68	102	10	-0.6	280	81	40	-0.3
003	138		10	-0.6	45		10	-1.9	223		10	-1.3
004	138		10	-0.6	45		10	-1.9	223		10	-1.3
005	199	98	10	1.2	76	101	10	-0.1	160	74	10	**-2.4**
006	150	100	10	-0.3	74.0	100	10	-0.2	#			
007	174	96	10	0.4	62	78	10	-0.9	455	90	25	**2.8**
008	127	96	30	-1.0	64	82	30	-0.8	#			
009	149	94.77	10	-0.3	91.4	94.77	10	0.8	214.9	98.56	20	-1.4
010	166	118	10	0.2	82	102	10	0.3	334	98	50	0.7
011	146	98	10	-0.4	82	118	10	0.3	270	104	50	-0.5
012	#				70	88	10	-0.4	#			
013	140	80	10	-0.6	63	102	10	-0.9	#			
014	149	92	10	-0.3	80	113	10	0.1	368	368	50	1.3
015	151.9	89.5	10	-0.2	71.4	91.9	10	-0.4	116.74		100	**-3.2**

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

Table 3 (continued): Results and z-Scores for Pirimicarb, Tebuconazole and Dithiocarbamates

laboratory number	analyte											
	pirimicarb assigned value 159 µg/kg				tebuconazole assigned value 77.6 µg/kg				dithiocarbamates assigned value 297 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
016	141.0			-0.5	45.0			-1.9	73.0			**-3.9**
017	160	91	10	0.0	78	96	10	0.0	310	72	50	0.2
018	#				#				156.52	98	150	**-2.5**
019	170	85	10	0.3	82	95	10	0.3	220	80	10	-1.3
020	200		10	1.2	110		10	1.9	290			-0.1
021	159.00	104.00	10.00	0.0	55.00	92.00	10.00	-1.3	738.00		10.00	**7.7**
022	146	87	0.01	-0.4	69	77	0.01	-0.5	0			**-5.2**
023	157	100	10	-0.1	73.5	100	10	-0.2	287	67	50	-0.2
024	149	88.5	10	-0.3	66.5	115.1	10	-0.7	365	119.0	100	1.2
025	186	88	5	0.8	97	90	5	1.1	123	100	5	**-3.0**
026	#				<LOQ	105	40		#			
027	125.10		10	-1.0	79.40		10	0.1	#			
028	150	106	10	-0.3	82	94	10	0.3	216	104	10	-1.4
029	197	100	10	1.1	100	100	10	1.3	280	100	10	-0.3
030	#				48	77.83	10	-1.7	#			

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

Table 3 (continued): Results and z-Scores for Pirimicarb, Tebuconazole and Dithiocarbamates

laboratory number	analyte											
	pirimicarb assigned value 159 µg/kg				tebuconazole assigned value 77.6 µg/kg				dithiocarbamates assigned value 297 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
031	#				71	75.83	20	-0.4	#			
032	0.160	96	0.005	**-4.7**	0.084	96	0.005	**-4.5**	#			
033	#				63.4308	89.9	10	-0.8	#			
034	173	109	10	0.4	82	101	10	0.3	209	75	10	-1.5
035	146.75		10	-0.4	58.12		10	-1.1	#			
036	133	94	10	-0.8	84	85	10	0.4	#			
037	#				#				372		10	1.3
038	193.9	88	10	1.0	85.0	102	10	0.4	240	118	20	-1.0
039	#				86	99	10	0.5	#			
040	#				#				432	89.6	40	**2.4**
041	139	90	10	-0.6	#				#			
042	#				68	80	10	-0.6	275	84	50	-0.4
043	162	109	10	0.1	84.8	112	10	0.4	374	100	10	1.4
044	152	100	10	-0.2	71	100	10	-0.4	444	114	100	**2.6**
045	114.38			-1.3	78.775			0.1	0			**-5.2**

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

Table 3 (continued): Results and z-Scores for Pirimicarb, Tebuconazole and Dithiocarbamates

laboratory number	analyte											
	pirimicarb assigned value 159 µg/kg				tebuconazole assigned value 77.6 µg/kg				dithiocarbamates assigned value 297 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
046	179.2	97.5	10	0.6	92.4	94.6	10	0.9	355.7	72.5	50	1.0
047	130	100	25	-0.9	74.27	100	25	-0.2	510.95	100	25	**3.8**
048	160	93	10	0.0	65	95	10	-0.7	362	112	100	1.1
049	#				100	99.5	10	1.3	64.1	99.2	40	**-4.1**
050	#				#				280	91.2	100	-0.3
051	139.48	95	10	-0.6	81.75	114	10	0.2	343	89	20	0.8
052	127.5	91	10	-0.9	67.8	97	10	-0.6	310.2	97	50	0.2
053	98.3	84	10	-1.8	47.2	90	10	-1.8	750	90	50	**8.0**
054	#				#				0	0	100	**-5.2**
055	170	96	10	0.3	77	101	10	0.0	265	90	10	-0.6
056	170	106	10	0.3	81	104	10	0.2	489	117	40	**3.4**
057	167	99	10	0.2	97	115	10	1.1	#			
058	181.3	91	10	0.7	85.4	106	50	0.5	267.7	79	100	-0.5
059	#				150	108	10	**4.2**	#			
060	151		10	-0.2	66		10	-0.7	#			

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

Table 3 (continued): Results and z-Scores for Pirimicarb, Tebuconazole and Dithiocarbamates

laboratory number	analyte											
	pirimicarb assigned value 159 µg/kg				tebuconazole assigned value 77.6 µg/kg				dithiocarbamates assigned value 297 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
061	163	93	10	0.1	84.7	98	10	0.4	#			
062	137	81	10	-0.7	76	90	10	-0.1	0			**-5.2**
063	188	94	10	0.9	88	97	10	0.6	198	81	10	-1.7
064	138	85	10	-0.6	82	89	10	0.3	175	97	100	**-2.1**
065	169	104	10	0.3	84	97	10	0.4	211	102	10	-1.5
066	#				#				#			
067	187	100	10	0.8	102	100	10	1.4	270	96	50	-0.5
068	0			**-4.7**	48.95			-1.7	1390.5			**19.2**
069	194.3	77.2	10	1.1	78	89.2	10	0.0	275.3		10	-0.4
070	164	95	10	0.2	70	93	10	-0.4	296	84	50	0.0
071	#				#				#			
072	55	79	10	**-3.1**	#				#			
073	185	100	10	0.8	87	100	10	0.6	261	100	10	-0.6
074	234		0.01	**2.2**	99		0.01	1.3	690		0.05	**6.9**
075	0.159			**-4.7**	0.083			**-4.5**	0.125			**-5.2**

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

Table 3 (continued): Results and z-Scores for Pirimicarb, Tebuconazole and Dithiocarbamates

laboratory number	analyte											
	pirimicarb assigned value 159 µg/kg				tebuconazole assigned value 77.6 µg/kg				dithiocarbamates assigned value 297 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
076	162	94	10	0.1	71	103	10	-0.4	200	119	50	-1.7
077	155	96	10	-0.1	80	101	10	0.1	225		10	-1.3
078	259	107	10	**3.0**	90	90	10	0.7	840	89	10	**9.5**
079	190	90	10	0.9	85	113	10	0.4	370	97	10	1.3
080	154	89	10	-0.1	80.5	113	10	0.2	304	88	10	0.1
081	139	84	10	-0.6	63.0	106	10	-0.9	340	115	50	0.8
082	156	100	10	-0.1	72	100	10	-0.3	#			
083	130	90	10	-0.9	56	95	10	-1.3	260	59	50	-0.6
084	159.55	108	10	0.0	94.99	93	10	1.0	29.91	105	10	**-4.7**
085	144	102	10	-0.4	49	109	10	-1.7	754	91.7	100	**8.0**
086	182	106	0.01	0.7	78	98	0.01	0.0	0			**-5.2**
087	118	101	10	-1.2	73.0	99	10	-0.3	#			

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

Table 4: Additional Pesticide Residues Reported

laboratory number	pesticide residue >30 µg/kg	result µg/kg	recovery %	LoQ µg/kg
011	Acrinathrin	34	110	10
020	Methiocarb Sulfoxide	33		10
035	Epoxiconazole	62.2		10
049	Deltamethrin	698	98.5	10
063	Methiocarb Sulfoxide	32	95	10
068	Parathion-methyl	88.22		
072	Fenazaquin	82	79	10
074	Methiocarb Sulfoxide	50		0,01
078	Methiocarb Sulfoxide	42	100	10

Table 5: Participants' Comments

participant number	comments
022	Unfortunately the test material has been thawed more than once prior to analysis
024	ps1: Recovery: pp'-DDD 113.0%, pp'-DDE 104.5%, op'-DDT 87.4%, pp'-DDT 76.7 %; ps2: LoQ for each compound is 10 ug/kg; ps3: Spinosyn A 126.0%, Spinosyn D 123.0%
030	Lambda-Cyhalothrin deteced below LOD
044	use standard additonal method
046	Cyhalothrin(sum of isomers)
051	Test D for Dithiocarbamates-date start analyse 24.02.2016
079	Results are not corrected for recovery.
082	Dithiocarbamates only analyzed by UV-Vis, sample sent was not enough for analysis.

comments are as submitted by participants

Table 6: 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	
azoxystrobin	65	117	2.21	Horwitz [7]	25.8
cyhalothrin-lambda	52	36.8	0.803	Horwitz [7]	8.09
dimethoate	62	173	3.37	Horwitz [7]	36.0
fenvalerate	62	78.5	1.93	Horwitz [7]	17.3
methiocarb	59	152	3.60	Horwitz [7]	32.2
permethrin	61	115	2.35	Horwitz [7]	25.3
pirimicarb	59	159	3.02	Horwitz [7]	33.5
tebuconazole	65	77.6	1.51	Horwitz [7]	17.1
dithiocarbamates	44	297	17.6	Horwitz [7]	57.0

Table 7: Number and Percentage of z-Scores where |z| ≤2

analyte	number of scores where |z| ≤2	total number of scores	% |z| ≤2
azoxystrobin	72	77	94
cyhalothrin-lambda	58	74	78
dimethoate	67	76	88
fenvalerate	68	77	88
methiocarb	64	71	90
permethrin	66	75	88
pirimicarb	65	71	92
tebuconazole	74	77	96
dithiocarbamates	38	63	60

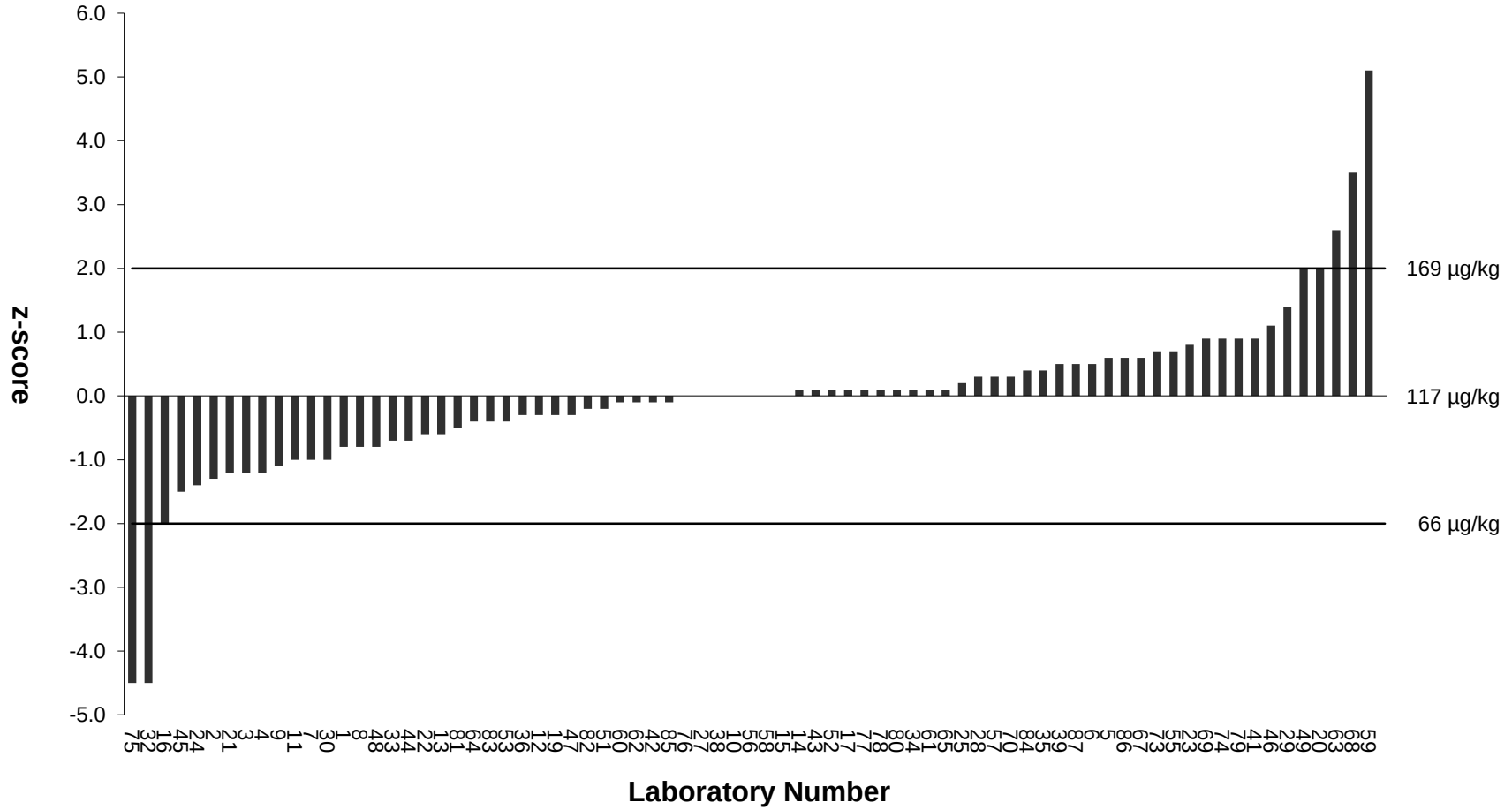

Figure 1: z-Scores for Azoxystrobin

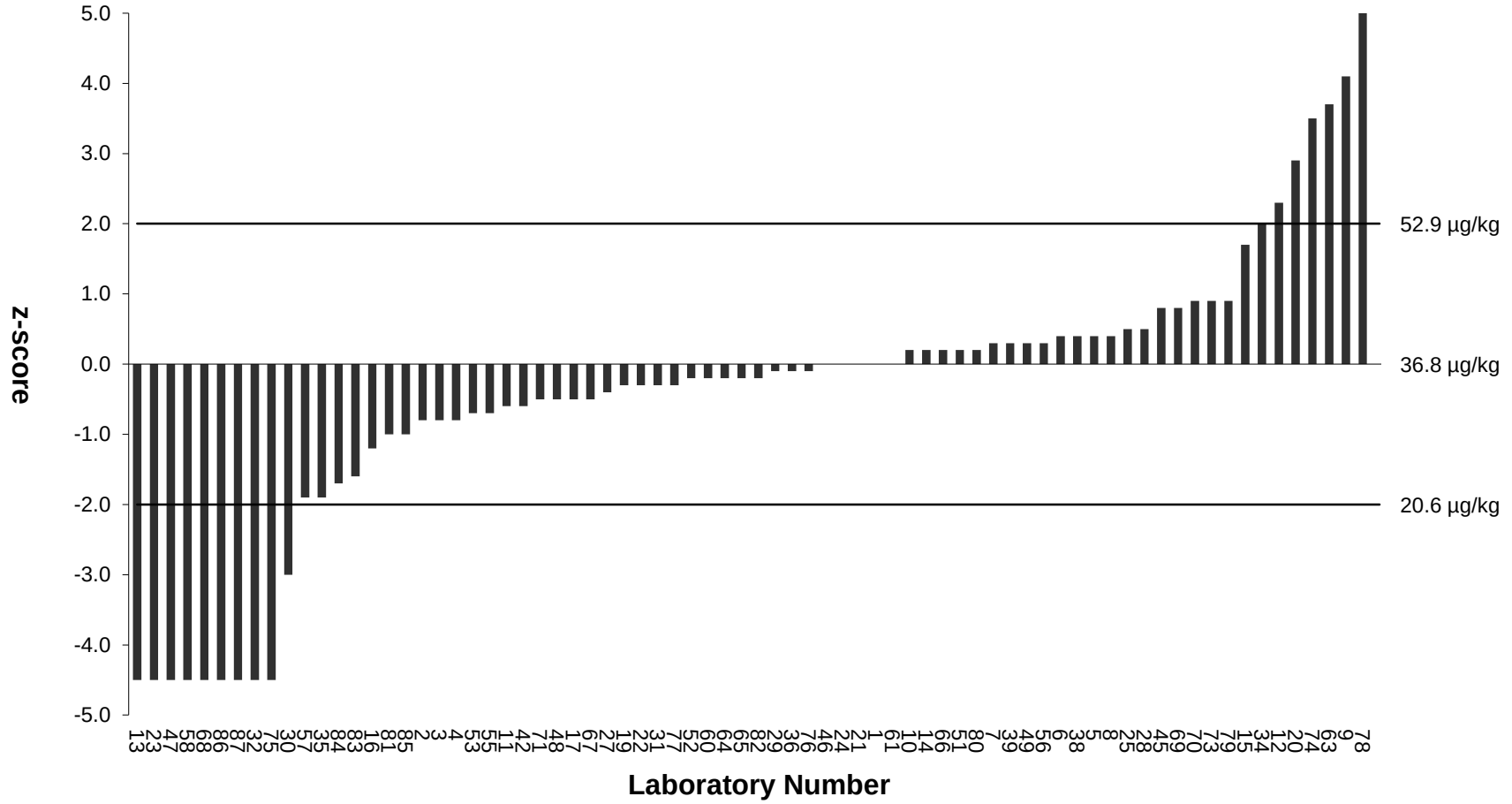

Figure 2: z-Scores for Cyhalothrin-Lambda

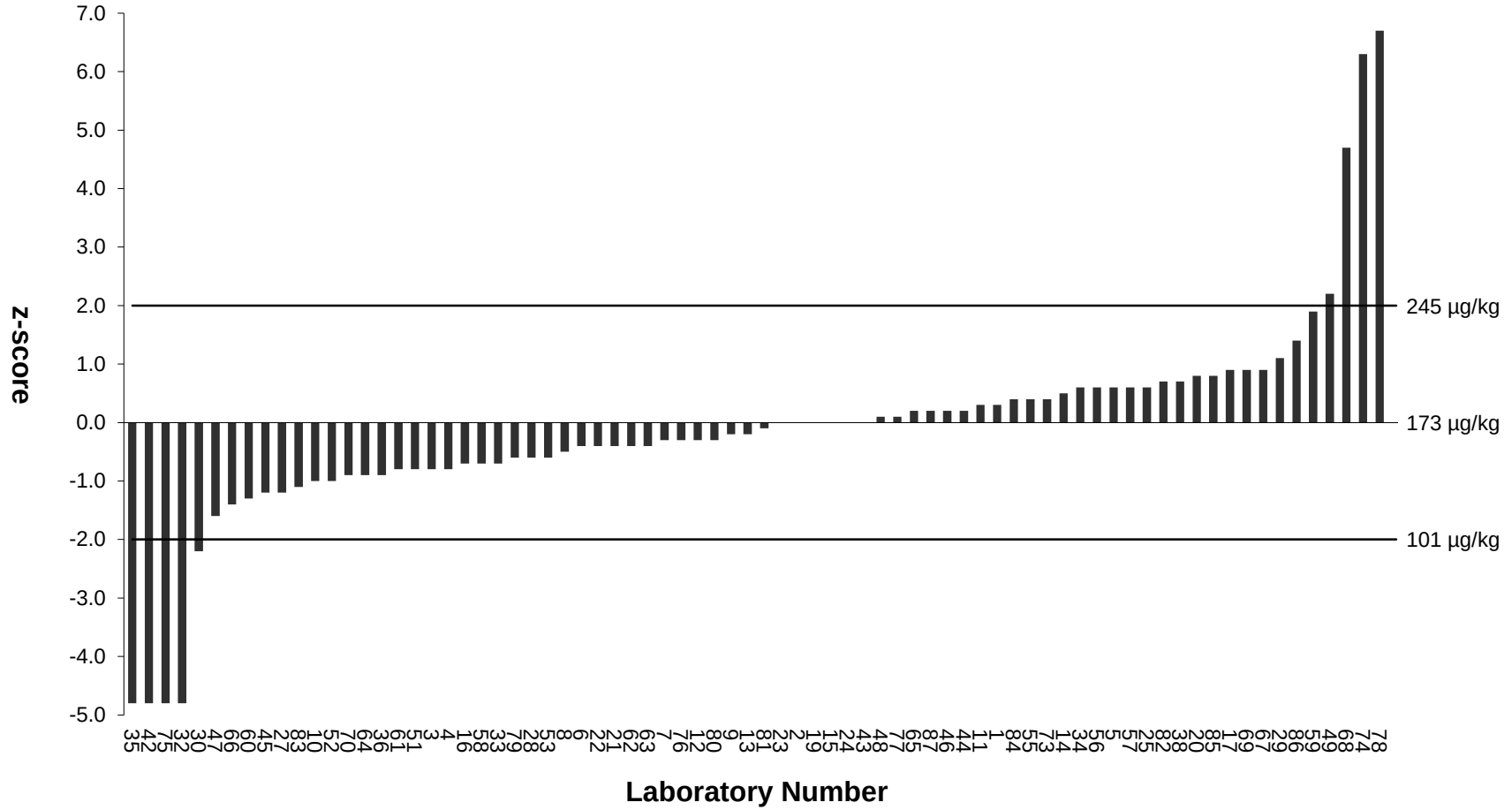

Figure 3: z-Scores for Dimethoate

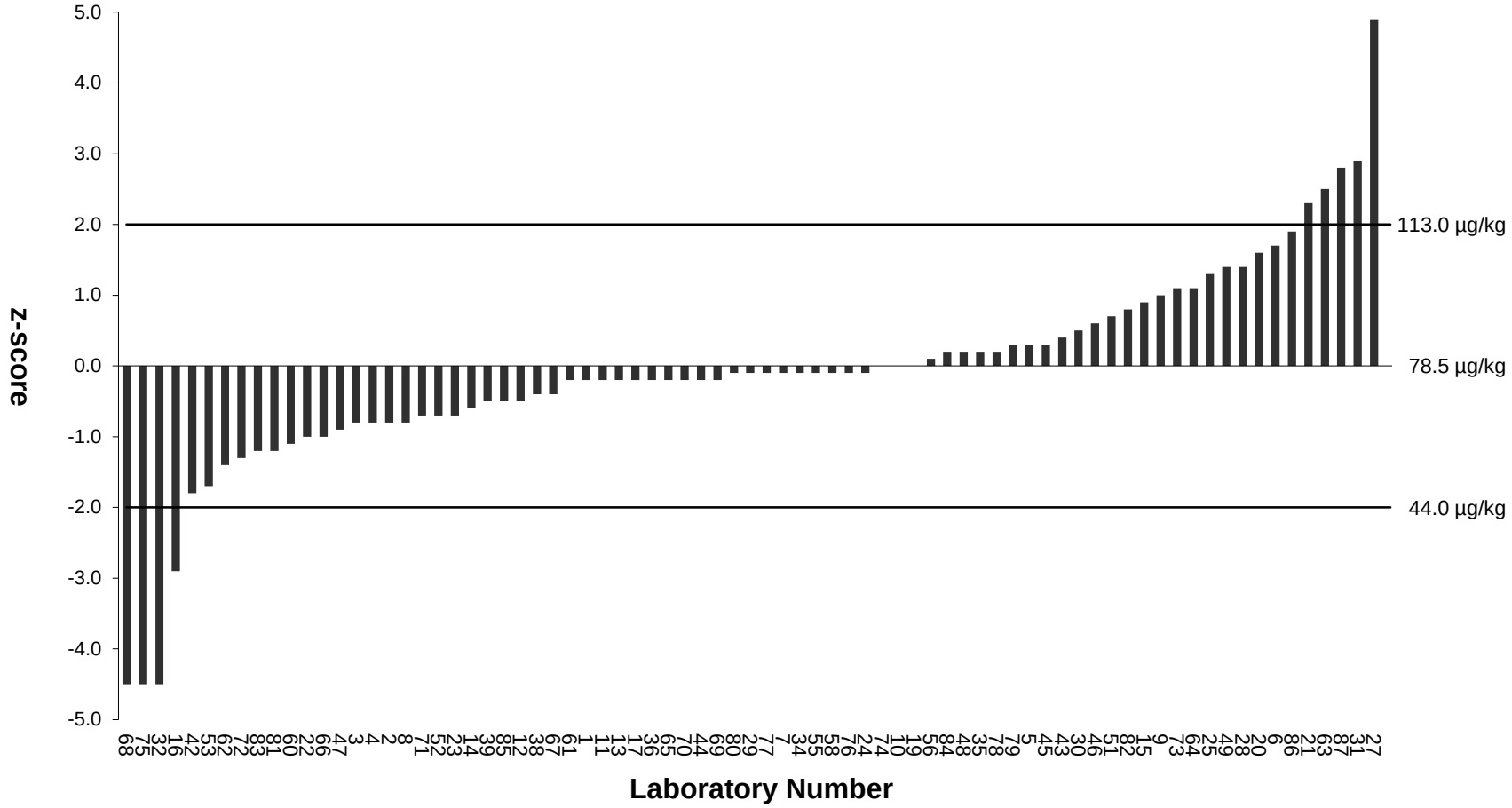

Figure 4: z-Scores for Fenvalerate

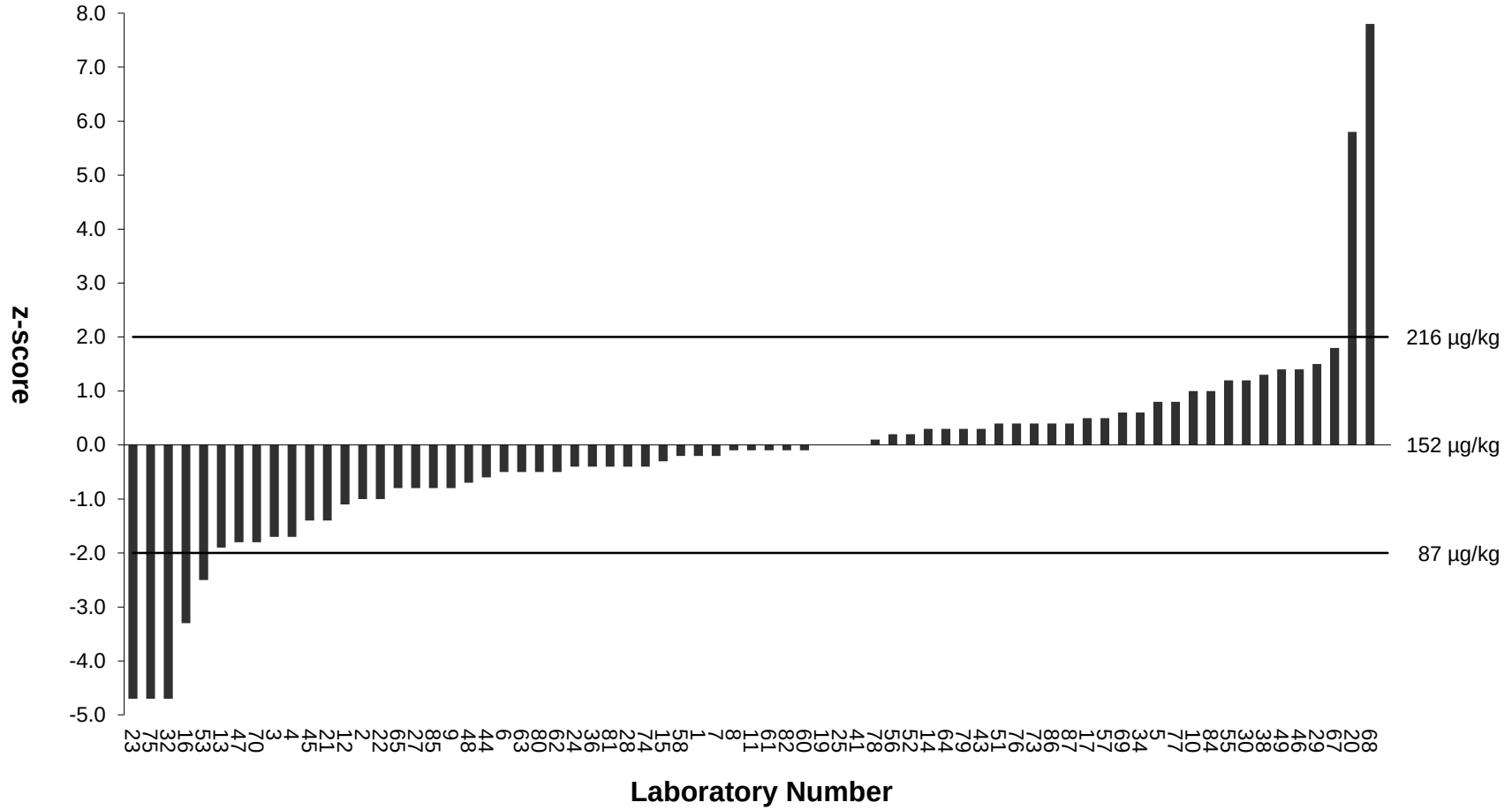

Figure 5: z-Scores for Methiocarb

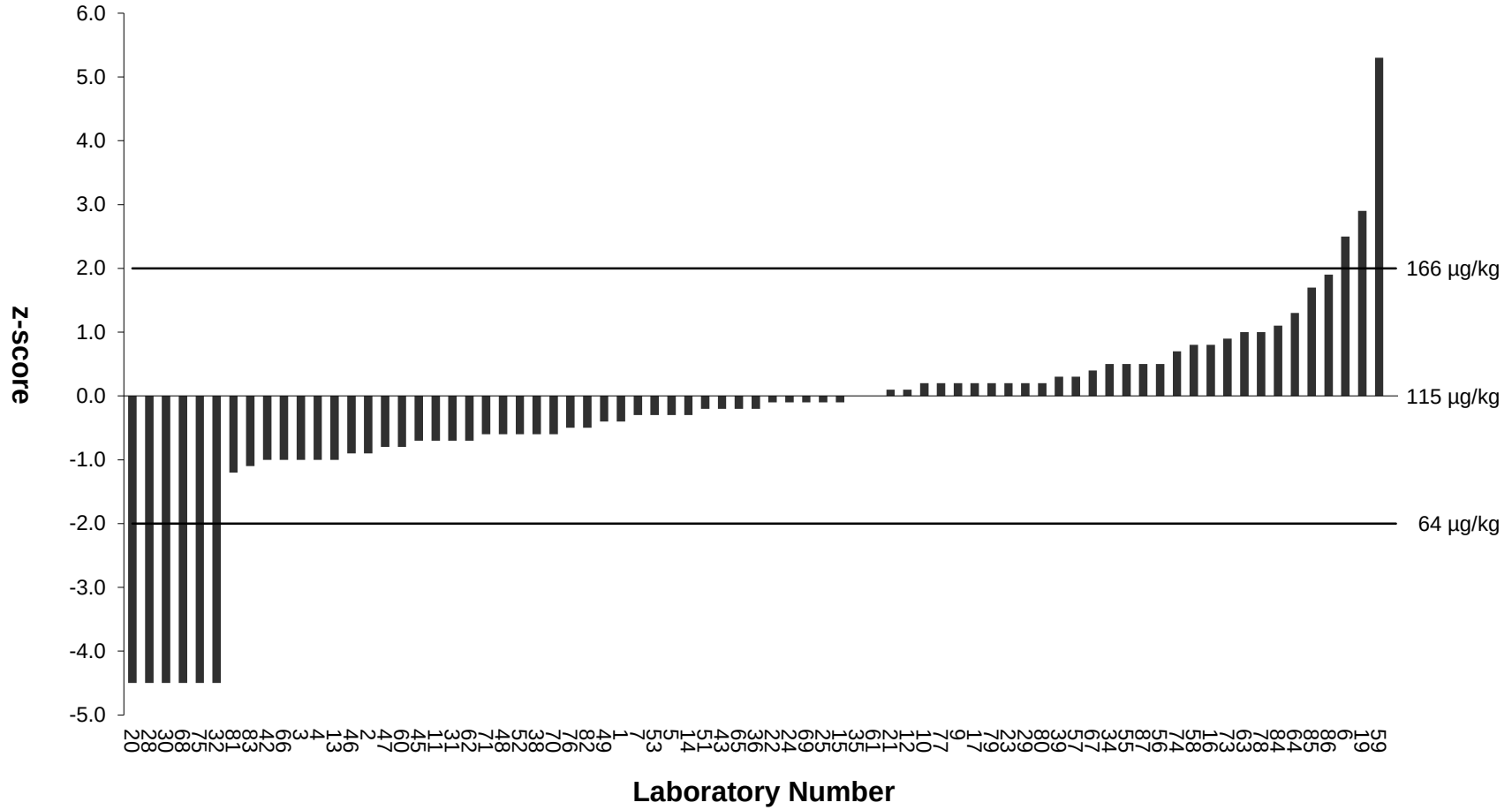

Figure 6: z-Scores for Permethrin

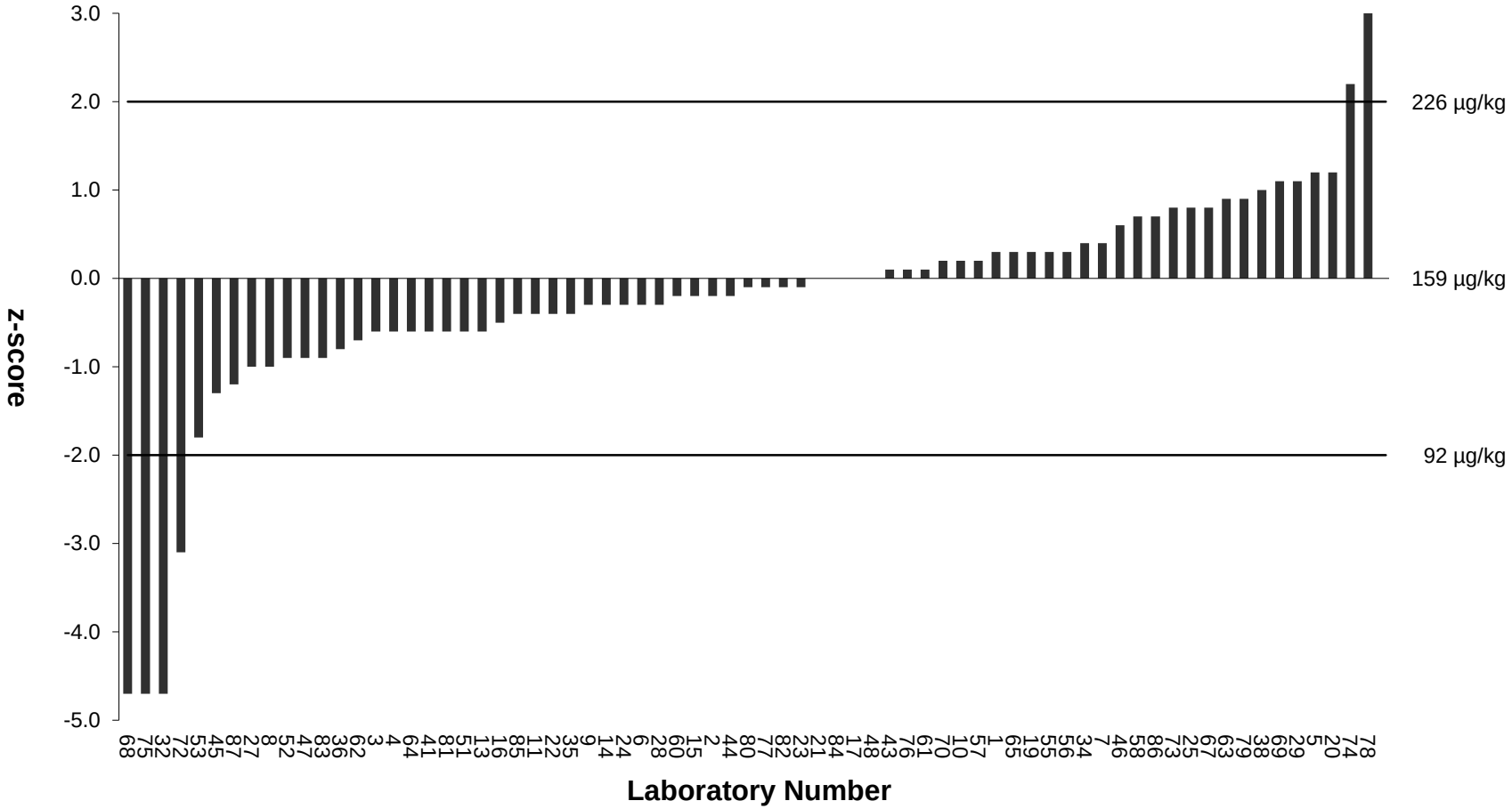

Figure 7: z-Scores for Pirimicarb

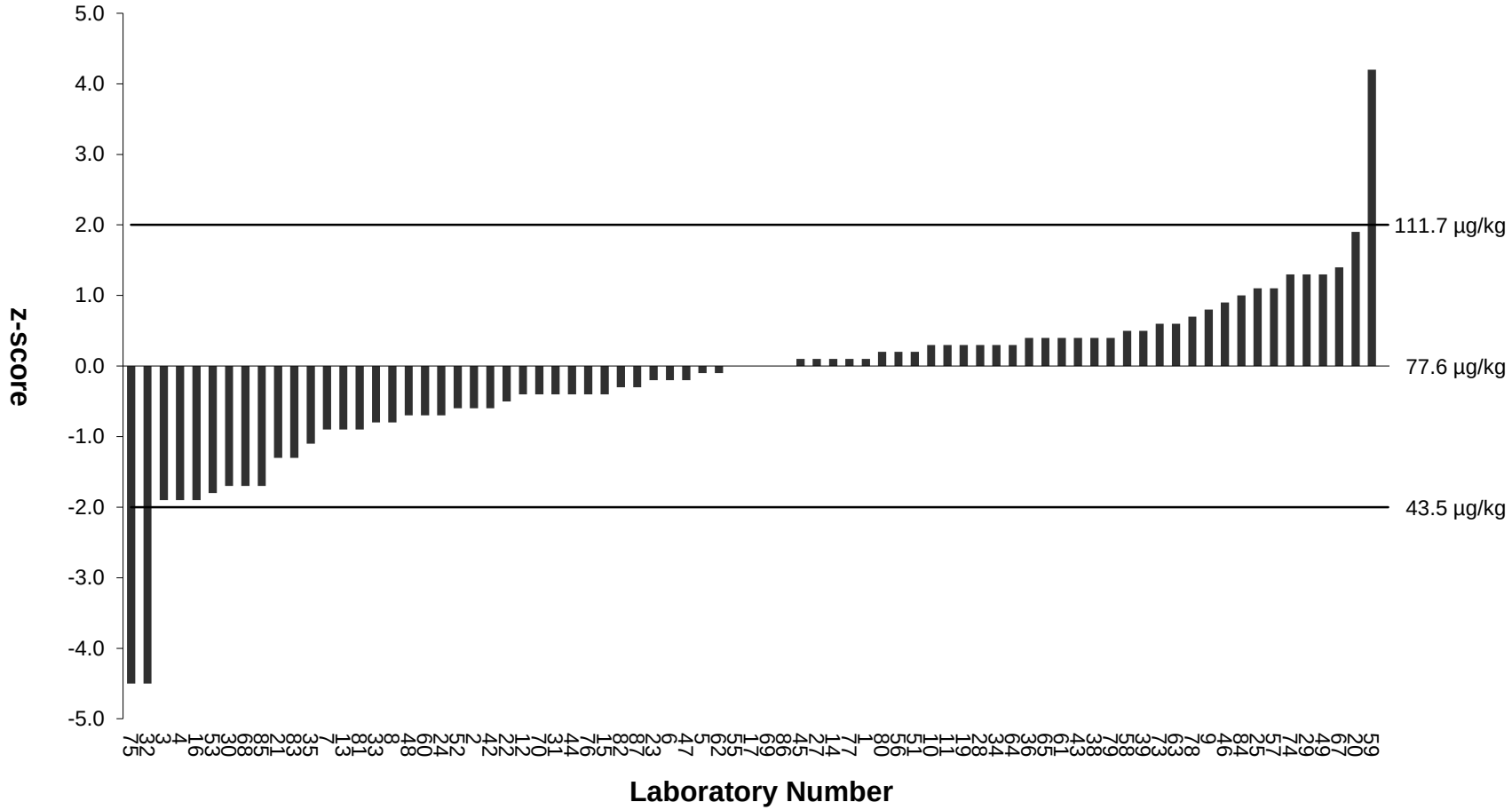

Figure 8: z-Scores for Tebuconazole

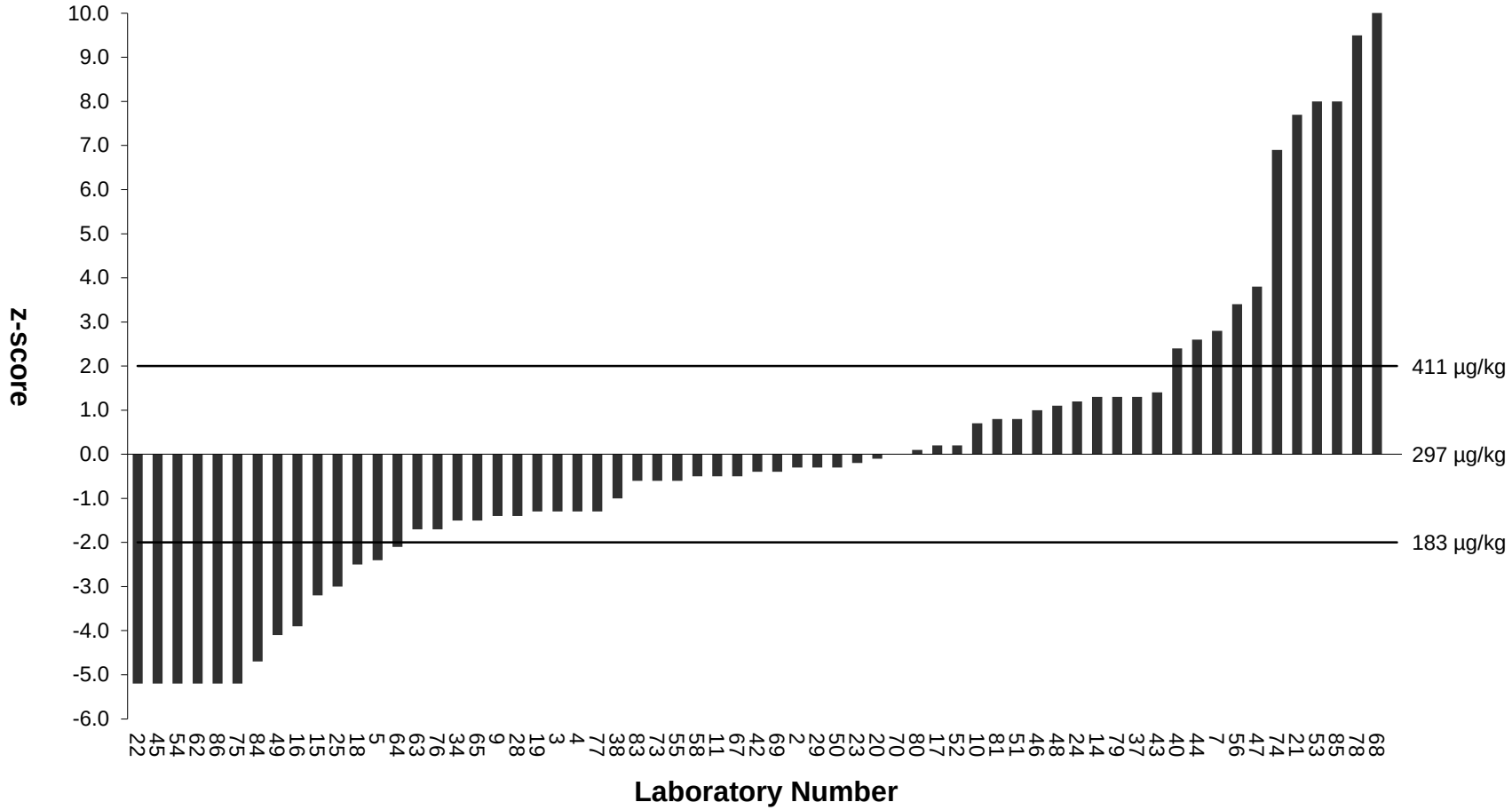

Figure 9: z-Scores for Dithiocarbamates

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	002 005 007 008 009 010 012 014 015 017 021 022 023 024 025 027 028 032 034 036 038 040 044 047 048 049 050 051 052 053 055 056 058 059 060 061 063 065 067 070 073 076 077 078 079 080 081 082 085 086
no	013 018 026 029 030 031 035 039 041 042 043 046 057 062 083 087

Method Based On	laboratory number
International Standard	005 009 014 021 028 031 034 035 036 040 041 048 049 055 061 063 065 067 077 078 082 087
National Standard	007 015 024 025 027 029 038 050 051 056 058 059 073 085 086
Paper Published In An International Journal	008 017 039 081 083
Manufacturer/Kit Instructions/Technical Note	026 042 043 060
In house method	002 010 012 013 018 022 023 030 032 044 046 047 052 053 057 062 070 076 080

Quoted percentage recovery measured in same analytical batch as test material	laboratory number
yes	002 005 007 008 010 013 014 015 017 018 021 022 023 024 025 026 029 030 032 034 036 039 040 041 042 043 044 046 049 051 052 053 057 058 060 061 062 065 067 070 073 076 078 080 081 082 083 085 087
no	009 012 027 028 031 035 038 047 048 050 055 056 059 063 077 086

If measured in this batch, stage spike added	laboratory number
prior to extraction	002 005 007 008 009 010 012 013 014 015 017 018 021 022 023 024 025 026 029 030 032 034 036 039 040 041 042 043 044 046 047 048 049 050 051 052 053 057 058 059 060 061 062 063 065 070 073 076 078 080 081 082 083 085 087

If measured in this batch, stage spike added (continued)	laboratory number
prior to instrument measurement	067

Concentration of Spike	laboratory number
≥1 - <5	041
≥5 - <10	009 023 032 046 058
≥10 - <25	007 008 014 024 031 052 053 055 060 081 083 087
≥25 - <50	017 025 026 030 036 047 059 063 085
≥50 - <100	005 010 012 013 015 021 022 029 034 051 057 061 073 078 082
≥100	002 018 039 040 042 043 044 048 049 050 062 065 067 070 076 077 080

Composition of Blank Commodity used for Spiking	laboratory number
blank provided	002 005 007 008 009 010 012 013 015 018 021 022 023 024 025 026 027 029 030 031 032 036 039 041 042 043 044 046 047 048 049 050 051 057 058 060 061 062 065 067 076 077 078 080 081 082 083 085
apple	040
carrot	053
organic pea	034 059 073

Calibration	laboratory number
standard addition	038 041 044 048 056 057 063 073
matrix-matched	005 007 008 014 018 024 029 031 035 036 046 049 052 055 060 061 062 065 067 073 077 080 081 085
solvent	005 021 028 036 050 087
multi-level	002 005 009 012 013 015 017 022 023 025 026 027 029 031 032 034 036 038 039 040 042 043 047 052 053 058 059 061 062 070 076 078 081 082 083
single-level	005 007 010 030 036 051 073 086

Internal Standard Added	laboratory number
yes	002 005 007 009 010 014 021 022 023 024 025 029 031 032 034 035 036 038 039 044 046 047 048 049 051 053 055 056 058 060 063 065 067 073 076 077 078 080 082 085 086
no	008 012 013 015 017 018 026 027 028 030 040 041 042 043 050 052 057 059 061 062 070 081 083 087

Internal Standard Used	laboratory number
aldrin	021
Carbendazim D4, Nicarbazin	034
Coffein (GC) Tricyclazole (LC)	002
combination of 6	029
DiazinonD	046
HCB-13C, PCB209, Quinalphos	032
Isoproturon D6, Anthracene D10	065
isotope labeled standards	010
Nicarbzain, Tritan	036
only to check extraction efficiency	005
p,p-DDE-d8	085
PCB 31	034 055 063 077
Sulfotep	055 063 077
PCB Mixture	031
PCB-153	032 076
PCB187, PCB20, Anilofos, Isoproturon	051
PCB77	025
Pirimicarb-D6	007 056
Azoxystrobin-D4	056
TEPP and Pyrimiphos methyl D6	049
tetraphenylethylene	014
TPP	009 022 023 024 034 035 038 039 047 063 073 076 080 082
deuterated pesticides	078 080
Sulprofos	082
Isoproturon-d6	038

Internal Standard Used (continued)	laboratory number
Carbaryl D7	023
HBB/Nicarbazin	073

GC Method: Sample Weight (g)	laboratory number
≥1 - <5	018 029 047 050 062 082 087
≥5 - <10	009 010 014 015 017 023 025 026 027 028 036 041 048 049 061 065 067 070 073 077 085 086
≥10 - <20	002 005 007 008 012 022 024 031 032 034 035 038 039 042 043 044 046 051 052 053 055 056 057 058 060 063 076 080 081 083
≥20 - <50	021 030 040 059

GC Method: Extraction Solvent Components	laboratory number
acetone	021 030 032 076
acetonitrile	002 005 008 009 010 012 014 015 021 022 023 024 026 027 028 029 031 034 036 038 039 041 042 043 046 047 049 051 053 055 056 057 058 059 060 061 062 063 065 067 070 073 077 080 081 082 085 086 087
dichloromethane	032 076
ethyl acetate	007 017 021 025 044 048 052
methanol	078
acetic acid	027 035
Hexane	030 032
iso-octane	040
petroleumether	076
Stannous chloride in 10% HCl	018

GC Method: Extraction pH Adjusted	laboratory number
yes	007 010 022 023 025 027 034 036 041 046 051 052 053 073 080 085 086
no	002 005 008 009 012 014 015 017 018 021 024 026 028 029 030 031 032 035 038 039 040 042 043 044 047 048 049 050 055 056 057 058 059 060 061 062 063 065 067 070 076 077 078 081 082 083 087

GC Method: Extraction Techniques Used	laboratory number
macerate at room temperature	007 017 031 044 052 059
QuEChERS	002 005 008 009 010 012 014 015 021 022 023 024 026 027 028 029 031 034 035 036 038 039 042 046 047 049 051 053 055 056 057 058 060 061 063 065 067 070 073 077 078 080 081 082 083 085 086 087
liquid-liquid partition	025 030 032 040 041 043 062 076
extraction at elevated pressure	048
DFG S-19	021
Headspace	018

GC Method: Sample Clean-up Technique	laboratory number
GPC/HPGPC	021
liquid-liquid partition	040 059
none	014 017 018 024 030 032 043 047 055 063 070 076 077 086 087
solid phase extraction (SPE) (column/cartridge)	025 026 029 042 044 046 048 052 059 062
solid phase extraction (SPE) (dispersive)	002 005 008 009 010 012 015 022 023 027 028 031 034 035 036 038 039 041 051 053 057 058 060 061 065 067 073 078 080 081 082 083 085
filtration	007

GC Method: SPE Sorbent Type	laboratory number
C18	042
Envicarb	052 061
PSA	002 005 008 010 012 015 022 023 027 034 036 038 039 051 053 055 057 058 060 065 067 070 073 078 080 081 082 083 085
Mixed Mode	026 029 031 041 044 046 048 059 062
None	018
GCB	002 052 061

GC Method: GC Column Packing	laboratory number
50% methyl 50% phenyl polysiloxane	012 014 041
65% methyl 35% phenyl polysiloxane	021 026

GC Method: GC Column Packing (continued)	laboratory number
95% methyl 5% phenyl polysiloxane	002 007 008 009 010 015 017 022 023 024 025 027 028 029 030 031 032 034 035 036 038 039 042 043 044 046 051 052 053 055 056 057 059 060 061 062 063 065 067 070 073 076 077 078 080 081 083 085
DB-1, DB-5, DB-1701	005
DB-624	018
SULFUR column	040

GC Method: GC Detector Type	laboratory number
ECD	005 021
FPD	005 050
MS	005 008 017 018 021 026 028 031 036 040 041 046 051 063 070 073 077 078
MS-MS	002 007 009 010 012 014 015 022 023 024 025 027 029 032 034 035 039 042 043 044 047 048 049 052 053 055 056 057 058 059 060 061 062 065 067 073 076 080 081 082 083 085 086 087
TOF-MS	038

LC Method: Sample Weight (g)	laboratory number
≥1 - <5	029 047 062 087
≥5 - <10	009 010 014 015 017 018 023 025 027 028 030 036 041 048 049 061 065 067 070 073 077 078 085 086
≥10 - <20	002 005 007 008 012 013 022 024 032 034 038 042 043 046 051 052 053 055 056 057 058 060 063 076 080 081 082 083
≥20 - <50	021 044 059

LC Method: Extraction Solvent Components	laboratory number
acetone	032 076
acetonitrile	002 005 008 009 010 012 013 015 017 018 021 022 023 024 025 027 028 030 034 036 038 041 042 043 046 047 048 049 051 052 053 055 056 057 058 059 060 061 062 063 065 067 070 073 077 080 081 082 085 086 087
dichloromethane	032 076

LC Method: Extraction Solvent Components (continued)	laboratory number
ethyl acetate	007 044
methanol	014 078
acetic acid	027
hexaan	032
petroleumether	076

LC Method: Extraction pH Adjusted	laboratory number
yes	007 010 012 013 022 025 027 029 030 034 036 041 046 051 053 061 073 080 085 086
no	002 005 008 009 014 015 017 018 021 023 024 028 032 038 039 042 043 044 047 048 049 052 055 056 057 058 059 060 062 063 065 067 070 076 077 078 081 082 083 087

LC Method: Extraction Techniques Used	laboratory number
macerate at room temperature	007 044 059
QuEChERS	002 005 008 009 010 012 013 014 015 017 018 021 022 023 024 025 027 028 029 030 034 036 038 042 046 047 049 051 052 053 055 056 057 058 060 061 063 065 067 070 073 077 078 080 081 082 083 085 086 087
liquid-liquid partition	032 041 043 062 076
extraction at elevated pressure	048

LC Method: Sample Clean-up Technique	laboratory number
liquid-liquid partition	029 059 078
none	009 014 017 021 024 032 038 043 044 047 048 049 051 052 055 063 065 067 070 076 077 086 087
solid phase extraction (SPE) (column/cartridge)	041 046 059 061 062 080
solid phase extraction (SPE) (dispersive)	002 005 008 010 012 013 015 018 022 023 027 028 034 036 053 057 058 060 073 081 082 083 085
Dilution and filtering	042
Filtration	007

LC Method: SPE Sorbent Type	laboratory number
C18	057
Envicarb	061
PSA	002 005 008 009 010 012 013 015 018 022 023 025 027 034 036 053 055 058 060 067 070 073 081 082 083 085
Mixed Mode	029 041 046 059 062 080
GCB	002 061

LC Method: HPLC Column Packing	laboratory number
C18	005 007 008 009 010 012 013 014 015 017 018 021 022 023 024 025 027 028 029 030 032 036 038 041 042 043 044 046 047 051 052 053 055 056 057 059 060 061 062 065 067 070 073 076 077 078 080 081 082 083 085 086
Mixed Mode	034

LC method: Mobile Phase Components	laboratory number
acetonitrile	014 015 021 023 029 034 041 080
ammonium acetate	007 021 024 030 042 046 047 049 052 062 073 078 081
ammonium formate	005 025 029 032 036 038 044 053 059
formic acid	018 043 047 053 059 062
methanol	002 005 007 008 010 024 032 034 036 038 047 048 052 053 055 059 061 062 073 076 077 081 087
water	002 009 010 012 013 018 021 022 024 027 028 029 034 036 038 047 051 052 053 056 057 058 060 061 062 063 065 067 070 073 076 081 082 083 085 086
acetic acid	002 005 036

LC Method: Detector Type	laboratory number
MS-MS	002 005 007 008 009 010 012 013 014 015 017 018 021 022 023 024 025 027 028 029 030 032 034 036 038 041 042 043 044 046 047 048 049 051 052 053 055 056 057 058 059 060 061 062 063 065 067 070 073 076 077 078 080 081 082 083 085 086 087

Azoxystrobin

Method Principle	laboratory number
GC	002 010 017 021 022 030 034 035 038 039 056 061 080
LC	005 007 008 009 012 013 014 015 023 024 025 027 028 029 032 036 041 042 043 044 046 047 049 051 052 053 055 057 058 059 060 062 063 065 067 070 073 076 077 079 081 082 083 085 086 087

Identification by Mass Spectrometry	laboratory number
yes	002 005 007 008 009 010 012 013 014 015 017 021 022 023 024 025 027 028 029 030 032 034 035 036 038 039 041 043 044 046 047 049 051 052 053 055 056 057 058 059 060 061 062 063 065 067 070 073 076 077 079 080 081 082 083 085 086 087
no	042

Cyhalothrin-Lambda

Method Principle	laboratory number
GC	002 005 007 008 009 010 012 014 015 017 021 022 024 027 028 029 030 031 032 034 035 036 038 039 042 046 049 051 052 053 055 056 057 060 061 063 065 067 070 073 076 077 079 080 081 082 083 085
LC	025

Identification by Mass Spectrometry	laboratory number
yes	002 005 007 008 009 010 014 015 017 021 022 024 025 027 028 029 030 031 032 034 035 036 038 039 046 049 051 052 053 055 056 060 061 063 065 067 070 073 076 077 079 080 081 082 083 085
no	012 042

Dimethoate

Method Principle	laboratory number
GC	009 010 017 021 030 043 059 060 076 083
LC	002 005 007 008 012 013 014 015 022 023 024 025 027 028 029 032 034 036 038 044 046 047 049 051 052 053 055 056 057 058 061 062 063 065 067 070 073 077 079 080 081 082 085 087

Identification by Mass Spectrometry	laboratory number
yes	002 005 007 008 009 010 012 013 014 015 017 021 022 023 024 025 027 028 029 030 032 034 036 038 043 044 046 047 049 051 052 053 055 056 057 058 059 060 061 062 063 065 067 070 073 076 077 079 080 081 082 083 085 087

Fenvalerate (sum of all isomers)

Method Principle	laboratory number
GC	002 005 008 009 010 014 015 017 021 022 023 024 025 027 028 029 030 031 032 034 035 036 038 039 042 043 044 046 049 051 052 053 055 056 058 060 061 062 063 065 067 070 073 076 077 079 080 081 082 083 085 086 087
LC	007 012 013 047

Identification by Mass Spectrometry	laboratory number
yes	002 005 007 008 009 010 012 013 014 015 017 021 022 023 024 025 027 028 029 030 031 032 034 035 036 038 039 043 044 046 047 049 051 052 053 055 056 058 060 061 062 063 065 067 070 073 076 077 079 080 081 082 083 085 086 087
no	042

Methiocarb

Method Principle	laboratory number
GC	009 060 082
LC	002 005 007 008 010 012 013 014 015 017 021 022 024 025 027 028 029 030 032 034 036 038 041 043 044 046 047 049 051 052 053 055 056 058 061 062 063 065 067 070 073 076 077 079 080 081 085 087

Identification by Mass Spectrometry	laboratory number
yes	002 005 007 008 009 010 012 013 014 015 017 021 022 024 025 027 028 029 032 034 036 038 041 043 044 046 047 049 051 052 053 055 056 058 060 061 062 063 065 067 070 073 076 077 079 080 081 082 085 087

Permethrin (sum isomers)

Method Principle	laboratory number
GC	002 005 007 009 010 012 014 015 017 021 022 023 024 025 029 031 032 034 035 036 038 039 042 043 049 051 052 053 055 056 057 058 059 060 061 062 063 065 067 070 076 077 079 080 081 082 083 086 087
LC	013 046 047 073 085

Identification by Mass Spectrometry	laboratory number
yes	002 005 007 009 010 012 013 014 015 017 021 022 023 024 025 029 031 032 034 035 036 038 039 043 046 047 049 051 052 053 055 056 058 059 060 061 062 063 065 067 070 073 076 077 079 080 081 082 083 085 086 087
no	042

Pirimicarb

Method Principle	laboratory number
GC	008 009 017 021 022 023 035 055 060 061 062 063 076 077 082 083
LC	002 005 007 010 013 014 015 024 025 027 028 029 032 034 036 038 041 043 044 046 047 051 052 053 056 058 065 067 070 073 079 080 081 085 087

Identification by Mass Spectrometry	laboratory number
yes	002 005 007 008 009 010 013 014 015 017 021 022 023 024 025 027 028 029 032 034 035 036 038 041 043 044 046 047 051 052 053 055 056 058 060 061 062 063 065 067 070 073 076 077 079 080 081 082 083 085 087

Tebuconazole

Method Principle	laboratory number
GC	002 008 009 010 017 021 023 024 025 030 031 034 035 038 039 042 053 055 058 060 061 062 063 067 077 079 080 081 082 083 086 087
LC	005 007 012 013 014 015 022 027 028 029 032 036 043 044 046 047 049 051 052 056 057 059 065 070 073 076 085

Identification by Mass Spectrometry	laboratory number
yes	002 005 007 008 009 010 012 013 014 015 017 021 022 023 024 025 027 028 029 030 031 032 034 035 036 038 039 043 044 046 047 049 051 052 053 055 056 057 058 059 060 061 062 063 065 067 070 073 076 077 079 080 081 082 083 085 086 087
no	042

Dithiocarbamates

Method Principle	laboratory number
GC	002 005 007 009 010 014 017 018 021 024 025 028 029 034 038 040 043 044 046 047 049 050 051 052 053 055 056 058 063 065 067 070 073 076 077 079 080 081 083 085
LC	042

Identification by Mass Spectrometry	laboratory number
yes	002 009 010 014 017 018 021 029 034 040 044 046 047 051 052 053 055 056 058 065 067 070 076 079 081
no	005 007 024 025 028 038 042 043 049 050 063 073 077 080 083 085

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® proficiency tests.

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 Simon Hunter (Round Coordinator). Participants with any comments or concerns about this proficiency test should contact:

FAPAS

Fera Science Ltd (Fera)
Sand Hutton
York
YO41 1LZ
UK

Tel: +44 (0)1904 462100
Fax: +44 (0)1904 500440

info@fapas.com

www.fapas.com