Fapas® – Food Chemistry Proficiency Test Report 19278

Pesticide Residues in Raspberry Purée

October-November 2019

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SUMMARY

1. The test materials for Fapas® – Food Chemistry proficiency test 19278 were dispatched in October 2019. Each participant received a raspberry purée test material to be analysed for a selection of pesticide residues.

2. An assigned value (x_a) was determined for each analyte and in conjunction with the standard deviation for proficiency (σ_p) was used to calculate a z-score for each result.

3. Results for this proficiency test are summarised as follows:

analyte	assigned value, x_a µg/kg	number of scores, |z| ≤2	total number of scores	% |z|≤2
Bifenthrin	58.4	72	78	92
Chlorpyrifos (ethyl)	56.5	74	79	94
Cyproconazole	38.9	67	74	91
Diazinon	53.0	68	76	89
Endosulfan I (alpha)	37.8	61	72	85
Epoxiconazole	50.7	68	70	97
Fenhexamid	70.7	69	74	93
Heptachlor	49.7	63	71	89
Iprodione	118	68	77	88
Piperonyl butoxide	89.6	66	70	94
Propoxur	32.0	66	71	93

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1. INTRODUCTION

1.1. Proficiency Testing

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

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

2. TEST MATERIAL

2.1. Preparation

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

The test material was prepared from fresh raspberries. The bulk sample was split into two batches: one for spiking and one for the blank test material.

Sub-samples were taken to screen for the possible presence of incurred residues and the remainder was stored at -20°C. No residues were detected at, or above, 15 µg/kg.

Bifenthrin, Chlorpyrifos (ethyl), Cyproconazole, Diazinon, Endosulfan I, Epoxiconazole, Fenhexamid, Heptachlor, Iprodione, Piperonyl butoxide and Propoxur were spiked into the test material.

Samples were stored at -20°C until dispatch.

2.2. Homogeneity

To test for homogeneity, randomly selected test materials were analysed in duplicate. Testing was sub-contracted to a laboratory meeting the quality requirements of the scheme's accreditation [3].

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

2.3. Dispatch

The start date was 7 October 2019. Test materials were sent to 86 participants.

3. RESULTS

The instructions for reporting results were as follows:

- Determine the level of residues present in the test material, **in µg/kg, in the material in the form it is received (no further correction for wet weight or dry weight or reconstitution),** together with percentage (%) recovery and limit of quantification (LoQ).
- All residues are to be reported **as specified**. If this is not possible, use the comments box to note any residues that are *not* reported in the form specified.

  - For each residue, select either “Not Detected”, “Not Tested” or “Provide Result”.
  - Enter a default value for “% recovery” and “limit of quantification (LOQ) µg/kg”.
- AFTER you have entered your results for each residue you MUST review and if necessary, edit the values for “% recovery” and “limit of quantification (LOQ) µg/kg” that differ from the default value you gave.

Results were submitted by 81 participants (94%) before the closing date for this test, 15 November 2019.

Each participant was given a laboratory number, assigned in order of receipt of results. The reported analyte concentrations are given in Table 1-Table 4.

If a participant analysed for a pesticide residue that was in the test material, but did not identify it, and their limit of quantification was below the level needed for a z-score of -3.0, they were assessed as if their result was zero.

If a participant analysed for a pesticide residue that was in the test material, but did not identify it, and their limit of quantification was above the level needed for a z-score of -3.0, the result was recorded as <LoQ.

Any participant identifying pesticide residues other than Bifenthrin, Chlorpyrifos (ethyl), Cyproconazole, Diazinon, Endosulfan I, Epoxiconazole, Fenhexamid, Heptachlor, Iprodione, Piperonyl butoxide and Propoxur at a level ≥15 µg/kg are listed in Table 5 together with the percentage recovery and LoQ.

Participants’ comments are given in Table 6.

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

4. STATISTICAL EVALUATION OF RESULTS

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

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

4.1. Calculation of the Assigned Value, x_a

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

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

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

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

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

iv) results where no recovery was reported,

v) results which were below the participant’s limit of quantification,

vi) results where no limit of quantification was reported.

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 7.

4.2. Standard Deviation for Proficiency, σ_p

The standard deviation for proficiency, σ_p, was set at a value that reflects best practice for the analyses in question.

For all analytes, σ_p was derived from the appropriate form of the Horwitz equation [9].

The values for σ_p used to calculate z-scores from the reported results of this test are given in Table 7.

4.3. Individual z-Scores

Participants' z-scores were calculated as:

$$z = \frac{(x - x_a)}{\sigma_p}$$

where x = the participant's reported result,
x_a = the assigned value, see Table 7,
and σ_p = the standard deviation for proficiency, see Table 7.

Participants' z-scores for all analytes are given in Table 1-Table 4 and shown as histograms in Figures 1–11. It is possible for the z-scores published in this report to differ slightly from the z-score that can be calculated using the formula given above. These differences arise from the necessary rounding of the actual assigned values and standard deviations for proficiency prior to their publication in Table 7.

The number and percentage of z-scores in the range $-2 \leq z \leq 2$ for all analytes are given in Table 8.

5. INTERPRETATION OF SCORES

In normal circumstances, over time, about 95% of z-scores will lie in the range $-2 \leq z \leq 2$. Occasional scores in the range $2 < |z| < 3$ are to be expected, at a rate of 1 in 20. Whether or not such scores are of importance can only be decided by considering them in the context of the other scores obtained by that laboratory.

Scores where $|z| > 3$ are to be expected at a rate of about 1 in 300. Given this rarity, such z-scores very strongly indicate that the result is not fit-for-purpose and almost certainly requires investigation.

The consideration of a set or sequence of z-scores over time provides more useful information than a single z-score. Examples of suitable methods of comparison are provided in the IUPAC International Harmonised Protocol for the Proficiency Testing of Analytical Chemistry Laboratories [7].

6. REFERENCES

1 Adobe Approved Trust List, https://helpx.adobe.com/acrobat/kb/approved-trust-list2.html#Whatisit accessed 13/11/2018.

2 GlobalSign PDF Signing Tool, https://www.globalsign.com/en/digital-signatures/ accessed 13/11/2018.

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

4 Fapas®, 2017, Protocol for Proficiency Testing Schemes, Version 6, April 2017, Part 1 – Common Principles.

5 Fapas®, 2017, Protocol for Proficiency Testing Schemes, Version 5, April 2017, Part 2 – Fapas® Food Chemistry scheme (FAPAS).

6 AMC Tech Brief No. 74, z-Scores and other scores in chemical proficiency testing – their meanings, and some common misconceptions, *Anal. Methods*, 2016, **8**, 5553.

7 Thompson, M., Ellison, S.L.R. and Wood, R., 2006, The International Harmonised Protocol for the Proficiency Testing of Analytical Chemistry Laboratories, *Pure Appl. Chem.*, **78**, No. 1, 145–196.

8 Guidance document on analytical quality control and method validation procedures for pesticide residues and analysis in food and feed, Document No. SANTE/11813/2017.

9 Thompson, M., 2000, Recent trends in inter-laboratory precision at ppb and sub-ppb concentrations in relation to fitness for purpose criteria in proficiency testing, *Analyst*, **125**, 385-386.

Table 1: Results and z-Scores for Bifenthrin, Chlorpyrifos (ethyl) and Cyproconazole

laboratory number	analyte											
	Bifenthrin assigned value: 58.4 µg/kg				Chlorpyrifos (ethyl) assigned value: 56.5 µg/kg				Cyproconazole assigned value: 38.9 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
001	53.2	100.4	2	-0.4	59.1	95.1	2	0.2	39.1	94.9	2	0.0
002	54		10	-0.3	59.6	91	10	0.3	#			
003	48	86	9	-0.8	52	87	6	-0.4	38	92	9	-0.1
004	47	68	10	-0.9	39	71	10	-1.4	34.7	107	10	-0.5
005	37.4		10	-1.6	72.9		10	1.3	40		10	0.1
006	61	87	10	0.2	59	94	10	0.2	0			**-4.5**
007	66	70-120	10	0.6	62	70-120	10	0.4	55	70-120	10	1.9
008	61.2	89	10	0.2	64.0	86	10	0.6	37.6	93	10	-0.1
009	56.7	98.52	10	-0.1	55.0	117	10	-0.1	41.1	84.5	10	0.3
010	59		10	0.0	56		10	0.0	48		10	1.1
011	57.51	98	10	-0.1	56.50	98	10	0.0	36.91	102	10	-0.2
012	50	100	10	-0.7	73	100	10	1.3	32	100	10	-0.8
013	63	106	10	0.4	60	99	5	0.3	45	101	10	0.7
014	65	50-150	10	0.5	61	50-150	10	0.4	#			
015	60			0.1	61			0.4	46			0.8

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

Table 1 (continued): Results and z-Scores for Bifenthrin, Chlorpyrifos (ethyl) and Cyproconazole

laboratory number	analyte											
	Bifenthrin assigned value: 58.4 µg/kg				Chlorpyrifos (ethyl) assigned value: 56.5 µg/kg				Cyproconazole assigned value: 38.9 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
016	59.61	90	10	0.1	59.61	90	10	0.3	0	90	10	**-4.5**
017	#				53	83	5	-0.3	30	96	5	-1.0
018	44	108	10	-1.1	55	102	10	-0.1	31	119	10	-0.9
019	43	113	10	-1.2	47	104	10	-0.8	44	112	10	0.6
020	59.6	95	10	0.1	57.5	93	10	0.1	34.9	94	10	-0.5
021	53.26	100.32	10	-0.4	55.68	85.04	10	-0.1	44.38	107.97	10	0.6
022	63	90-110	10	0.4	55	90-110	10	-0.1	20	90-110	10	**-2.2**
023	62.6	88	10	0.3	37.1	95	10	-1.6	27.4	85	10	-1.3
024	#				#				#			
025	61.5	115	10	0.2	55.5	100	10	-0.1	38.5	102	10	0.0
026	50	90	10	-0.7	54	90	10	-0.2	34	90	10	-0.6
027	64	90	10	0.4	60	106	10	0.3	47	78	10	1.0
028	111.311			**4.1**	61.904			0.4	136.823			**11.5**
029	56.79	85.38	6.045	-0.1	53.94	94.14	6.045	-0.2	39.25	88.98	6.045	0.0
030	60	80-120	0.01mg/kg	0.1	60	80-120	0.01mg/kg	0.3	#			

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

Table 1 (continued): Results and z-Scores for Bifenthrin, Chlorpyrifos (ethyl) and Cyproconazole

laboratory number	analyte											
	Bifenthrin assigned value: 58.4 µg/kg				Chlorpyrifos (ethyl) assigned value: 56.5 µg/kg				Cyproconazole assigned value: 38.9 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
031	66	99	10	0.6	61	97	10	0.4	48	97	10	1.1
032	59.28	70-120	10	0.1	64.7	70-120	10	0.7	32.64	70-120	10	-0.7
033	75.1	100	10	1.3	62.5	100	10	0.5	0	100	10	**-4.5**
034	53.0	85	10	-0.4	45.8	85	10	-0.9	29.6	85	10	-1.1
035	61.5	100	5	0.2	48.2	104	5	-0.7	39.1	116	5	0.0
036	73		10	1.1	54		10	-0.2	38	93	10	-0.1
037	39	64	10	-1.5	40	86	10	-1.3	23	60	10	-1.9
038	50.42	92.66	10	-0.6	#				#			
039	325	89	10	**20.7**	123	88	10	**5.4**	0			**-4.5**
040	52	100	10	-0.5	56.1	100	10	0.0	44.5	100	10	0.7
041	76	114	10	1.4	63	91	10	0.5	42	117	10	0.4
042	65	96	10	0.5	60	95	10	0.3	46	96	10	0.8
043	59.57	105	10	0.1	57.22	101	10	0.1	38.82	97	10	0.0
044	42.14	70-135	10	-1.3	52.829	70-135	10	-0.3	56.05	70-135	10	2.0
045	60	114.1	10	0.1	20	92.3	10	**-2.9**	23	96.4	10	-1.9

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

Table 1 (continued): Results and z-Scores for Bifenthrin, Chlorpyrifos (ethyl) and Cyproconazole

laboratory number	analyte											
	Bifenthrin assigned value: 58.4 µg/kg				Chlorpyrifos (ethyl) assigned value: 56.5 µg/kg				Cyproconazole assigned value: 38.9 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
046	78	85	10	1.5	64	85	10	0.6	42	85	10	0.4
047	92			**2.6**	83			**2.1**	#			
048	68.39			0.8	51.06			-0.4	47.72			1.0
049	61		10	0.2	27		10	**-2.4**	43		10	0.5
050	62	125	10	0.3	58	112	10	0.1	33	90	10	-0.7
051	58.1	101.5	10	0.0	56.5	101.5	10	0.0	34.1	93.2	10	-0.6
052	57	94	10	-0.1	53	87	10	-0.3	39	88	10	0.0
053	56	94	10	-0.2	55	97	10	-0.1	41	102	10	0.2
054	0		10	**-4.5**	0		10	**-4.5**	14.68		10	**-2.8**
055	66	72	10	0.6	56	81	10	0.0	41	88	5	0.2
056	#				37.6	60	5	-1.5	39.8	95	5	0.1
057	66	101	10	0.6	61	101	10	0.4	43	98	10	0.5
058	63	88	10	0.4	53.1	88	10	-0.3	30.7	88	10	-1.0
059	53	S	10	-0.4	59	S	10	0.2	46	108	10	0.8
060	43.4	95	10	-1.2	47.1	89	10	-0.8	35.5	108	10	-0.4

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

Table 1 (continued): Results and z-Scores for Bifenthrin, Chlorpyrifos (ethyl) and Cyproconazole

laboratory number	analyte											
	Bifenthrin assigned value: 58.4 µg/kg				Chlorpyrifos (ethyl) assigned value: 56.5 µg/kg				Cyproconazole assigned value: 38.9 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
061	48.5		10	-0.8	64.9		10	0.7	33		10	-0.7
062	56	91	5	-0.2	60	97	5	0.3	45	93	5	0.7
063	64.32	112.04	5.00	0.5	56.80	109.00	5.00	0.0	43.65	106.36	5.00	0.6
064	36	100	10	-1.7	51	100	10	-0.4	25	100	10	-1.6
065	54	100	5	-0.3	56	100	5	0.0	40	100	5	0.1
066	65	105	10	0.5	56	97	10	0.0	40	110	10	0.1
067	60	6.163	0.01	0.1	52	10.768	0.01	-0.4	37	-4.338	0.01	-0.2
068	63	92	10	0.4	64	100	10	0.6	51	95	10	1.4
069	65	105	10	0.5	64	106	10	0.6	44	106	10	0.6
070	77	79	0.010mg/kg	1.4	55	94	0.010mg/kg	-0.1	37	97	0.010mg/kg	-0.2
071	60	100	10	0.1	57	100	10	0.0	40	100	10	0.1
072	29	80	30	**-2.3**	40	80	30	-1.3	34	80	30	-0.6
073	69	95	10	0.8	54	91	10	-0.2	39	99	10	0.0
074	72	99	10	1.1	63	98	10	0.5	52	96	10	1.5
075	66.5	101	10	0.6	66.6	105	10	0.8	44.4	98	10	0.6

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

Table 1 (continued): Results and z-Scores for Bifenthrin, Chlorpyrifos (ethyl) and Cyproconazole

laboratory number	analyte											
	Bifenthrin assigned value: 58.4 µg/kg				Chlorpyrifos (ethyl) assigned value: 56.5 µg/kg				Cyproconazole assigned value: 38.9 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
076	59	96	5	0.0	59	108	5	0.2	40	106	5	0.1
077	37	70	10	-1.7	58	80	10	0.1	30	60	10	-1.0
078	40.52	103	8.12	-1.4	38.33	106	32.32	-1.5	#			
079	53	97	10	-0.4	56	106	10	0.0	38	94	10	-0.1
080	28.99	70-130	0.01mg/kg	**-2.3**	57.97	70-130	0.01mg/kg	0.1	35.98	70-130	0.01mg/kg	-0.3
081	79	114	0.003	1.6	57	102	0.003	0.0	51	114	0.003	1.4

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

Table 2: Results and z-Scores for Diazinon, Endosulfan I and Epoxiconazole

laboratory number	analyte											
	Diazinon assigned value: 53.0 µg/kg				Endosulfan I assigned value: 37.8 µg/kg				Epoxiconazole assigned value: 50.7 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
001	54.4	81.7	2	0.1	40.4	87.4	2	0.3	46.7	87.3	2	-0.4
002	57.4	92	10	0.4	#				#			
003	44	100	6	-0.8	37	97	9	-0.1	61	94	6	0.9
004	27	82	10	**-2.2**	23	75	10	-1.8	34.3	76	10	-1.5
005	58.3		10	0.5	26.4		10	-1.4	44		10	-0.6
006	61	98	10	0.7	49	89	10	1.3	#			
007	78	70-120	10	**2.1**	55	70-120	10	**2.1**	47	70-120	10	-0.3
008	56.0	87	10	0.3	46.6	90	10	1.1	45.0	90	10	-0.5
009	39.1	75	10	-1.2	43.7	111	20	0.7	44.6	91	10	-0.5
010	51		10	-0.2	31		10	-0.8	56		10	0.5
011	37.76	97	10	-1.3	36.69	96	10	-0.1	50.99	113	10	0.0
012	88	100	10	**3.0**	45	100	10	0.9	#			
013	61	103	10	0.7	42	89	10	0.5	58	116	10	0.7
014	59	50-150	10	0.5	23	50-150	10	-1.8	59	50-150	10	0.7
015	60			0.6	45			0.9	57			0.6

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

Table 2 (continued): Results and z-Scores for Diazinon, Endosulfan I and Epoxiconazole

laboratory number	analyte											
	Diazinon assigned value: 53.0 µg/kg				Endosulfan I assigned value: 37.8 µg/kg				Epoxiconazole assigned value: 50.7 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
016	59.61	90	10	0.6	41.69	90	10	0.5	#			
017	#				#				37	93	5	-1.2
018	41	101	10	-1.0	40	113	10	0.3	40	92	10	-1.0
019	47	94	10	-0.5	24	92	10	-1.7	49	93	10	-0.1
020	43.8	90	10	-0.8	27.7	84	10	-1.2	67.9	104	10	1.5
021	52.79	99.33	10	0.0	34.52	77.99	10	-0.4	59.28	108.76	10	0.8
022	51	90-110	10	-0.2	43	90-110	10	0.6	54	90-110	10	0.3
023	34.7	77	10	-1.6	51.4	83	10	1.6	44.1	71	10	-0.6
024	#				#				#			
025	55.5	95	10	0.2	42.5	110	10	0.6	48.5	98	10	-0.2
026	50	90	10	-0.3	29	90	10	-1.1	48	90	10	-0.2
027	54	105	10	0.1	47	127	10	1.1	51	90	10	0.0
028	52.435			0.0	40.87			0.4	#			
029	51.43	92.94	6.045	-0.1	#				45.62	86.64	6.045	-0.5
030	36	80-120	0.01mg/kg	-1.5	0	80-120	0.01mg/kg	**-4.5**	#			

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

Table 2 (continued): Results and z-Scores for Diazinon, Endosulfan I and Epoxiconazole

laboratory number	analyte											
	Diazinon assigned value: 53.0 µg/kg				Endosulfan I assigned value: 37.8 µg/kg				Epoxiconazole assigned value: 50.7 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
031	62	105	10	0.8	0	100	10	**-4.5**	50	96	10	-0.1
032	60.1	70-120	10	0.6	#				65	70-120	10	1.3
033	62	100	10	0.8	26.2	100	10	-1.4	61	100	10	0.9
034	40.98	85	10	-1.0	25.9	85	10	-1.4	47.0	85	10	-0.3
035	55	110	5	0.2	35.5	110	5	-0.3	47.8	102	5	-0.3
036	56	93	10	0.3	<LOQ		50		51	90	10	0.0
037	30	108	10	-2.0	21	72	10	-2.0	58	118	10	0.7
038	#				30.79	88.53	10	-0.8	#			
039	56	78	10	0.3	0			**-4.5**	0			**-4.5**
040	80.8	100	10	**2.4**	42.5	100	10	0.6	59.2	100	10	0.8
041	61	115	10	0.7	40	112	10	0.3	38	109	10	-1.1
042	67	90	10	1.2	40	81	10	0.3	58	92	10	0.7
043	54.71	98	10	0.1	0		5	**-4.5**	51.25	127	10	0.1
044	28.68	70-135	10	**-2.1**	0	70-135	10	**-4.5**	57.9	70-135	10	0.7
045	53	90.2	10	0.0	53	98.6	10	1.8	50	92.8	10	-0.1

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

Table 2 (continued): Results and z-Scores for Diazinon, Endosulfan I and Epoxiconazole

laboratory number	analyte											
	Diazinon assigned value: 53.0 µg/kg				Endosulfan I assigned value: 37.8 µg/kg				Epoxiconazole assigned value: 50.7 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
046	68	85	10	1.3	0	85	10	**-4.5**	58	85	10	0.7
047	79			**2.2**	62			**2.9**	#			
048	56.64			0.3	36.09			-0.2	62.48			1.1
049	66		10	1.1	46		10	1.0	50		10	-0.1
050	50	98	10	-0.3	37	110	10	-0.1	46	95	10	-0.4
051	49.9	101.1	10	-0.3	37.0	108.3	10	-0.1	46.0	104.8	10	-0.4
052	53	97	10	0.0	42	98	10	0.5	47	95	10	-0.3
053	44	97	10	-0.8	46	101	10	1.0	50	98	10	-0.1
054	0		10	**-4.5**	51.86		10	1.7	27.24		10	**-2.1**
055	57	77	10	0.3	46	71	10	1.0	62	90	5	1.0
056	#				#				#			
057	56	98	10	0.3	33	100	10	-0.6	53	98	10	0.2
058	56.1	88	10	0.3	29.7	88	10	-1.0	41.2	88	10	-0.8
059	53	116	10	0.0	36	S	10	-0.2	54	120	10	0.3
060	42.6	105	10	-0.9	30.0	89	10	-0.9	33.8	111	10	-1.5

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

Table 2 (continued): Results and z-Scores for Diazinon, Endosulfan I and Epoxiconazole

laboratory number	analyte											
	Diazinon assigned value: 53.0 µg/kg				Endosulfan I assigned value: 37.8 µg/kg				Epoxiconazole assigned value: 50.7 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
061	34.6		10	-1.6	51.4		10	1.6	42.9		10	-0.7
062	50	87	5	-0.3	45	81	5	0.9	49	93	5	-0.1
063	57.10	110.81	5.00	0.4	38.47	108.47	10.00	0.1	60.63	107.38	5.00	0.9
064	45	100	10	-0.7	26	100	10	-1.4	42	100	10	-0.8
065	48	100	5	-0.4	30	100	5	-0.9	49	100	5	-0.1
066	55	90	10	0.2	39	100	10	0.1	48	87	10	-0.2
067	63	11.538	0.01	0.9	50	5.353	0.01	1.5	47	-6.473	0.01	-0.3
068	51	105	10	-0.2	45	97	10	0.9	66	110	10	1.4
069	62	96	10	0.8	45	93	10	0.9	55	100	10	0.4
070	51	100	0.010mg/kg	-0.2	0		0.010mg/kg	**-4.5**	47	91	0.010mg/kg	-0.3
071	50	100	10	-0.3	36	100	10	-0.2	64	100	10	1.2
072	49	80	30	-0.3	#				40	80	30	-1.0
073	46	91	10	-0.6	45	95	10	0.9	51	102	10	0.0
074	60	95	10	0.6	45	99	10	0.9	55	99	10	0.4
075	62.3	100	10	0.8	0			**-4.5**	52.1	96	10	0.1

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

Table 2 (continued): Results and z-Scores for Diazinon, Endosulfan I and Epoxiconazole

laboratory number	analyte											
	Diazinon assigned value: 53.0 µg/kg				Endosulfan I assigned value: 37.8 µg/kg				Epoxiconazole assigned value: 50.7 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
076	54	92	5	0.1	48	110	5	1.2	52	106	5	0.1
077	55	90	10	0.2	28	70	10	-1.2	48	100	10	-0.2
078	98.67	73	12.24	**3.9**	< LOQ	120	49.19		#			
079	54	87	10	0.1	36	124	10	-0.2	45	94	10	-0.5
080	#				24.99	70-130	0.01mg/kg	-1.5	45.98	70-130	0.01mg/kg	-0.4
081	58	109	0.003	0.4	12	106	0.003	**-3.1**	53	97	0.003	0.2

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

Table 3: Results and z-Scores for Fenhexamid, Heptachlor and Iprodione

laboratory number	analyte											
	Fenhexamid assigned value: 70.7 µg/kg				Heptachlor assigned value: 49.7 µg/kg				Iprodione assigned value: 118 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
001	38.9	56	2	-2.0	54.9	87.5	2	0.5	129.8	91.8	2	0.5
002	#				#				129		10	0.4
003	60	82	9	-0.7	48	98	9	-0.2	110	90	21	-0.3
004	60	106	10	-0.7	41	89	10	-0.8	75	79	10	-1.7
005	77		10	0.4	52.7		10	0.3	104.9		10	-0.5
006	67	98	10	-0.2	51	88	10	0.1	143	88	10	1.0
007	65	70-120	10	-0.4	74	70-120	10	**2.2**	85	70-120	10	-1.3
008	58.0	91	10	-0.8	60.2	80	10	1.0	107	94	10	-0.4
009	74.2	48	10	0.2	57.9	107	10	0.7	84.4	111	10	-1.3
010	67		10	-0.2	40		10	-0.9	178		10	**2.3**
011	60.37	101	20	-0.7	41.81	80	10	-0.7	92.51	102	10	-1.0
012	#				87	100	10	**3.4**	64	100	10	**-2.1**
013	77	102	10	0.4	56	93	5	0.6	123	96	10	0.2
014	64	50-150	10	-0.4	0	50-150	10	**-4.5**	116	50-150	10	-0.1
015	77			0.4	62			1.1	105			-0.5

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

Table 3 (continued): Results and z-Scores for Fenhexamid, Heptachlor and Iprodione

laboratory number	analyte											
	Fenhexamid assigned value: 70.7 µg/kg				Heptachlor assigned value: 49.7 µg/kg				Iprodione assigned value: 118 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
016	#				#				125.13	90	10	0.3
017	57	100	5	-0.9	#				106	123	5	-0.5
018	64	109	10	-0.4	45	98	10	-0.4	103	103	10	-0.6
019	59	98	10	-0.8	42	95	10	-0.7	124	108	10	0.2
020	91.6	70	10	1.3	33.4	88	10	-1.5	91.4	78	10	-1.0
021	86.40	107.97	10	1.0	38.55	97.34	10	-1.0	112.04	102.08	10	-0.2
022	71	90-110	10	0.0	50	90-110	10	0.0	119	90-110	10	0.0
023	44.5	75	10	-1.7	<LOQ		50		86.8	70	10	-1.2
024	#				53.4	100	20	0.3	#			
025	58.7	92	10	-0.8	55	90	10	0.5	145	105	10	1.0
026	67	90	10	-0.2	41	90	10	-0.8	93	90	10	-1.0
027	81	94	10	0.7	61	119	10	1.0	134	92	10	0.6
028	76.495			0.4	#				#			
029	#				55.11	97.72	6.045	0.5	143.92	99.42	6.045	1.0
030	#				48	80-120	0.01mg/kg	-0.2	100	80-120	0.01mg/kg	-0.7

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

Table 3 (continued): Results and z-Scores for Fenhexamid, Heptachlor and Iprodione

laboratory number	analyte											
	Fenhexamid assigned value: 70.7 µg/kg				Heptachlor assigned value: 49.7 µg/kg				Iprodione assigned value: 118 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
031	72	107	10	0.1	58	104	10	0.8	125	96	10	0.3
032	93.9	70-120	10	1.5	#				152	70-120	10	1.3
033	73	100	10	0.1	48.1	100	10	-0.1	138.1	100	10	0.8
034	79.4	85	10	0.6	43.5	85	10	-0.6	87.7	85	10	-1.2
035	67.5	74	5	-0.2	42.3	108	1	-0.7	119.2	106	5	0.0
036	82	121	10	0.7	46		10	-0.3	249		10	**5.1**
037	86	114	10	1.0	28	66	10	-2.0	98	100	10	-0.8
038	#				30.88	83.53	10	-1.7	110.76	84.88	10	-0.3
039	0			**-4.5**	525	81	10	**43.5**	0			**-4.5**
040	70.6	100	10	0.0	58.7	100	10	0.8	119.5	100	10	0.1
041	77	91	10	0.4	63	80	10	1.2	139	110	10	0.8
042	68	97	10	-0.2	42	84	10	-0.7	151	100	10	1.3
043	66.01	108	10	-0.3	56.32	117	5	0.6	129.13	110	10	0.4
044	87.7	70-135	10	1.1	42.32	70-135	10	-0.7	0	70-135	10	**-4.5**
045	71	82.3	10	0.0	#				120	142.3	10	0.1

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

Table 3 (continued): Results and z-Scores for Fenhexamid, Heptachlor and Iprodione

laboratory number	analyte											
	Fenhexamid assigned value: 70.7 µg/kg				Heptachlor assigned value: 49.7 µg/kg				Iprodione assigned value: 118 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
046	69	85	10	-0.1	0	85	10	**-4.5**	122	85	10	0.2
047	83			0.8	#				204			**3.3**
048	68.29			-0.2	50.75			0.1	114.57			-0.1
049	72		10	0.1	58		10	0.8	150		10	1.2
050	65	95	10	-0.4	36	92	10	-1.3	169	120	10	2.0
051	56.0	91.1	10	-0.9	57.7	100.0	10	0.7	110.2	98.3	10	-0.3
052	58	92	10	-0.8	47	86	10	-0.2	100	92	10	-0.7
053	53	80	10	-1.1	54	94	10	0.4	120	75	10	0.1
054	19.61		10	**-3.3**	71.62		10	2.0	0		10	**-4.5**
055	95	58	10	1.6	60	92	10	0.9	137	51	10	0.7
056	65.8	117	10	-0.3	#				154.1	126	5	1.4
057	103	95	10	**2.1**	62	100	10	1.1	139	100	10	0.8
058	122.1	88	10	**3.3**	146.3	88	10	**8.8**	0	88		**-4.5**
059	72	103	10	0.1	63	S	10	1.2	130	S	10	0.5
060	65.5	97	10	-0.3	35.3	92	10	-1.3	105	94	10	-0.5

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

Table 3 (continued): Results and z-Scores for Fenhexamid, Heptachlor and Iprodione

laboratory number	analyte											
	Fenhexamid assigned value: 70.7 µg/kg				Heptachlor assigned value: 49.7 µg/kg				Iprodione assigned value: 118 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
061	52		10	-1.2	91.7		10	**3.8**	80		10	-1.5
062	59	87	5	-0.8	41	85	5	-0.8	127	86	5	0.3
063	76.40	91.45	5.00	0.4	41.72	113.54	5.00	-0.7	136.47	95.32	5.00	0.7
064	77	100	10	0.4	35	100	10	-1.3	116	100	10	-0.1
065	59	100	5	-0.8	32	100	5	-1.6	91	100	5	-1.0
066	71	80	10	0.0	60	89	10	0.9	104	79	10	-0.5
067	62	6.473	0.01	-0.6	61	8.147	0.01	1.0	107	5.162	0.01	-0.4
068	59	100	10	-0.8	41	163	10	-0.8	150	90	10	1.2
069	72	112	10	0.1	57	105	10	0.7	137	102	10	0.7
070	70	98	0.010mg/kg	0.0	53	80	0.010mg/kg	0.3	75	85	0.010mg/kg	-1.7
071	85	100	10	0.9	54	100	10	0.4	120	100	10	0.1
072	25	80	30	**-2.9**	36	80	30	-1.3	#			
073	69	99	10	-0.1	63	102	10	1.2	125	98	10	0.3
074	70	98	10	0.0	66	96	10	1.5	132	98	10	0.5
075	69.8	97	10	-0.1	43.3	90	10	-0.6	119	87	10	0.0

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

Table 3 (continued): Results and z-Scores for Fenhexamid, Heptachlor and Iprodione

laboratory number	analyte											
	Fenhexamid assigned value: 70.7 µg/kg				Heptachlor assigned value: 49.7 µg/kg				Iprodione assigned value: 118 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
076	71	110	5	0.0	66	101	5	1.5	115	99	5	-0.1
077	88	90	10	1.1	40	80	10	-0.9	107	70	10	-0.4
078	63.25	94	37.58	-0.5	0			**-4.5**	< LOQ	120	149	
079	98	103	10	1.8	#				122	90	10	0.2
080	65.97	70-130	0.01mg/kg	-0.3	35.98	70-130	0.01mg/kg	-1.3	112.94	70-130	0.01mg/kg	-0.2
081	86	102	0.005	1.0	40	117	0.003	-0.9	280	82	0.005	**6.2**

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

Table 4: Results and z-Scores for Piperonyl butoxide and Propoxur

laboratory number	analyte							
	Piperonyl butoxide assigned value: 89.6 µg/kg				Propoxur assigned value: 32.0 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
001	90.8	91.3	2	0.1	27.3	104	2	-0.7
002	105		10	0.8	#			
003	80	98	6	-0.5	30	88	2	-0.3
004	56.6	100	10	-1.7	29.6	111	10	-0.3
005	59		10	-1.6	31		10	-0.1
006	89	98	10	0.0	#			
007	114	70-120	10	1.2	29	70-120	10	-0.4
008	95.0	87	10	0.3	31.7	94	10	0.0
009	95.1	75	10	0.3	31.4	74	10	-0.1
010	113		10	1.2	38		10	0.8
011	84.95	102	10	-0.2	32.43	103	10	0.1
012	71	100	10	-0.9	#			
013	91	109	10	0.1	33	90	10	0.1
014	103	50-150	10	0.7	28	50-150	10	-0.6
015	91			0.1	34			0.3

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

Table 4 (continued): Results and z-Scores for Piperonyl butoxide and Propoxur

laboratory number	analyte							
	Piperonyl butoxide assigned value: 89.6 µg/kg				Propoxur assigned value: 32.0 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
016	#				28.20	90	10	-0.5
017	#				#			
018	85	107	10	-0.2	31	106	10	-0.1
019	86	88	10	-0.2	33	94	10	0.1
020	70.4	70	10	-1.0	44.1	92	10	1.7
021	82.16	96.67	10	-0.4	31.87	107.53	10	0.0
022	95	90-110	10	0.3	43	90-110	10	1.6
023	71.0	66	10	-0.9	34.3	71	10	0.3
024	#				#			
025	#				32.2	99	10	0.0
026	74	90	10	-0.8	38	90	10	0.8
027	98	91	10	0.4	31	86	10	-0.1
028	#				#			
029	89.57	85.77	6.045	0.0	31.08	97.98	6.045	-0.1
030	#				32	80-120	0.01mg/kg	0.0

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

Table 4 (continued): Results and z-Scores for Piperonyl butoxide and Propoxur

laboratory number	analyte							
	Piperonyl butoxide assigned value: 89.6 µg/kg				Propoxur assigned value: 32.0 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
031	100	104	10	0.5	34	101	10	0.3
032	121.41	70-120	10	1.6	80	70-120	10	**6.8**
033	97	100	10	0.4	#			
034	78.1	85	10	-0.6	33.31	85	10	0.2
035	111.3	106	5	1.1	25.9	88	10	-0.9
036	118		10	1.4	28	95	10	-0.6
037	50	70	10	-2.0	30	94	10	-0.3
038	#				#			
039	100	76	10	0.5	0			**-4.5**
040	100.9	100	10	0.6	24.9	100	10	-1.0
041	78	113	10	-0.6	24	99	10	-1.1
042	99	75	10	0.5	37	98	10	0.7
043	93.49	107	10	0.2	30.63	97	10	-0.2
044	45.78	70-135	10	**-2.2**	29.2	70-135	10	-0.4
045	93	120.8	10	0.2	32	66.9	10	0.0

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

Table 4 (continued): Results and z-Scores for Piperonyl butoxide and Propoxur

laboratory number	analyte							
	Piperonyl butoxide assigned value: 89.6 µg/kg				Propoxur assigned value: 32.0 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
046	0	85	10	**-4.5**	38	85	10	0.8
047	#				31			-0.1
048	120.62			1.6	32.12			0.0
049	94		10	0.2	35		10	0.4
050	111	125	10	1.1	24	95	10	-1.1
051	83.2	97.2	10	-0.3	27.2	89.5	10	-0.7
052	91	92	10	0.1	32	93	10	0.0
053	89	97	10	0.0	30	97	10	-0.3
054	0		10	**-4.5**	0		10	**-4.5**
055	91	78	10	0.1	33	90	10	0.1
056	77.1	80	5	-0.6	#			
057	96	97	10	0.3	43	99	10	1.6
058	112.3	88	10	1.2	68.7	88	10	**5.2**
059	65	117	10	-1.2	43	111	10	1.6
060	69.2	105	10	-1.0	30.7	99	10	-0.2

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

Table 4 (continued): Results and z-Scores for Piperonyl butoxide and Propoxur

laboratory number	analyte							
	Piperonyl butoxide assigned value: 89.6 µg/kg				Propoxur assigned value: 32.0 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
061	61.7		10	-1.4	26.4		10	-0.8
062	93	93	5	0.2	28	87	5	-0.6
063	116.61	114.49	5.00	1.4	#			
064	71	100	10	-0.9	30	100	10	-0.3
065	83	100	5	-0.3	29	100	5	-0.4
066	125	110	10	1.8	39	95	10	1.0
067	#				26	-12.982	0.01	-0.9
068	100	115	10	0.5	27	75	10	-0.7
069	102	88	10	0.6	35	96	10	0.4
070	98	95	0.010mg/kg	0.4	25	93	0.010mg/kg	-1.0
071	101	100	10	0.6	39	100	10	1.0
072	48	80	30	**-2.1**	37	80	30	0.7
073	87	100	10	-0.1	33	108	10	0.1
074	93	97	10	0.2	35	99	10	0.4
075	109	109	10	1.0	33.2	90	10	0.2

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

Table 4 (continued): Results and z-Scores for Piperonyl butoxide and Propoxur

laboratory number	analyte							
	Piperonyl butoxide assigned value: 89.6 µg/kg				Propoxur assigned value: 32.0 µg/kg			
	result	% Rec	LoQ	z-score	result	% Rec	LoQ	z-score
076	97	93	5	0.4	34	140	5	0.3
077	67	60	10	-1.1	23	60	10	-1.3
078	#				0			**-4.5**
079	#				29	92	10	-0.4
080	68.97	70-130	0.01mg/kg	-1.0	37.98	70-130	0.01mg/kg	0.8
081	123	98	0.003	1.7	19	110	0.003	-1.8

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

Table 5: Additional Pesticide Residues Reported

laboratory number	pesticide residue ≥15 µg/kg	result µg/kg	recovery %	LoQ µg/kg
014	Endosulfan II (beta)	22	50-150	10
027	Fenpropathrin	31	80	10
032	Fenazaquin	40	70 - 120	10 µg/kg
039	Fenpropathrin	77	85	10
054	Procymidone	245.9		10ppb
058	Azoxystrobin	40	88	10
058	Chlorobenzilate	17.3	88	10
058	Fenpropathrin	97.8	88	10
072	Endrin	32	80	30
081	Chlorobenzilate	46	99	0.003

Table 6: Participants' Comments

laboratory number	comments
002	Bifenthrin, Iprodione, Piperonyl Butoxide are not part of our fortification process
005	esfenvalerate not tested
007	Iprodion and Propoxur were analyzed with a modified Quechers method. The pH value was not adjusted.
013	Azoxystrobin detected but < LoQ (10 µg/kg)
019	Azoxystrobin detected below LOQ in blank as well as in sample
028	we found two traces of pesticides (Difenoconazole & Dimethomorph)
030	All the values are corrected with the recoveries.
036	Azoxystrobin positive detected via LC-MS/MS (Quechers) and GC-MS (DFG S19). With approx. 7 µg/kg below LOQ. Same Result in Test Material 19278b (Blank Raspberry).
037	The analyte Azoxystrobin has been reported with 10 ppb, it should be noted that the blank samplet received from FAPAS also contained this compound (10ppb), both meet the confirmation criteria so it is likely that the blank already contained this compound.
039	Lcmsms-gcmsms
040	azoxystrobine 8µg/kg
041	Azoxystrobin: 6 µg/kg
042	Traces found of azoxystrobine (8µg/kg) and boscalid and pendimethalin (<5µg/kg)
053	Azoxystrobin detected in blank sample : 7 µg/kg
055	Azoxystrobin was detedted in traces.
057	We have being detected Azoxystrobin <LOQ.
059	Standard-Addition for Bifenthrin, Chlorpyrifos (ethyl), Endosulfan I (alpha), Heptachlor and Iprodione
068	Heptachlor corrected for recovery
070	when the sample arrived in lab was unfrozen.
071	The sample contains 0.008 mg/kg of azoxistrobin

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

Table 7: Assigned Values and Standard Deviations for Proficiency

analyte	data points, *n*	assigned value, x_a µg/kg	uncertainty, *u*	standard deviation for proficiency, σ_p	
Bifenthrin	64	58.4	1.3	Horwitz [9]	12.9
Chlorpyrifos (ethyl)	67	56.5	0.7	Horwitz [9]	12.4
Cyproconazole	61	38.9	0.9	Horwitz [9]	8.55
Diazinon	65	53.0	1.1	Horwitz [9]	11.7
Endosulfan I	53	37.8	1.3	Horwitz [9]	8.32
Epoxiconazole	60	50.7	1.0	Horwitz [9]	11.1
Fenhexamid	58	70.7	1.6	Horwitz [9]	15.6
Heptachlor	57	49.7	1.6	Horwitz [9]	10.9
Iprodione	60	118	3	Horwitz [9]	25.9
Piperonyl butoxide	59	89.6	2.2	Horwitz [9]	19.7
Propoxur	59	32.0	0.7	Horwitz [9]	7.05

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

analyte	number of scores where $\|z\| \leq 2$	total number of scores	% $\|z\| \leq 2$
Bifenthrin	72	78	92
Chlorpyrifos (ethyl)	74	79	94
Cyproconazole	67	74	91
Diazinon	68	76	89
Endosulfan I	61	72	85
Epoxiconazole	68	70	97
Fenhexamid	69	74	93
Heptachlor	63	71	89
Iprodione	68	77	88
Piperonyl butoxide	66	70	94
Propoxur	66	71	93

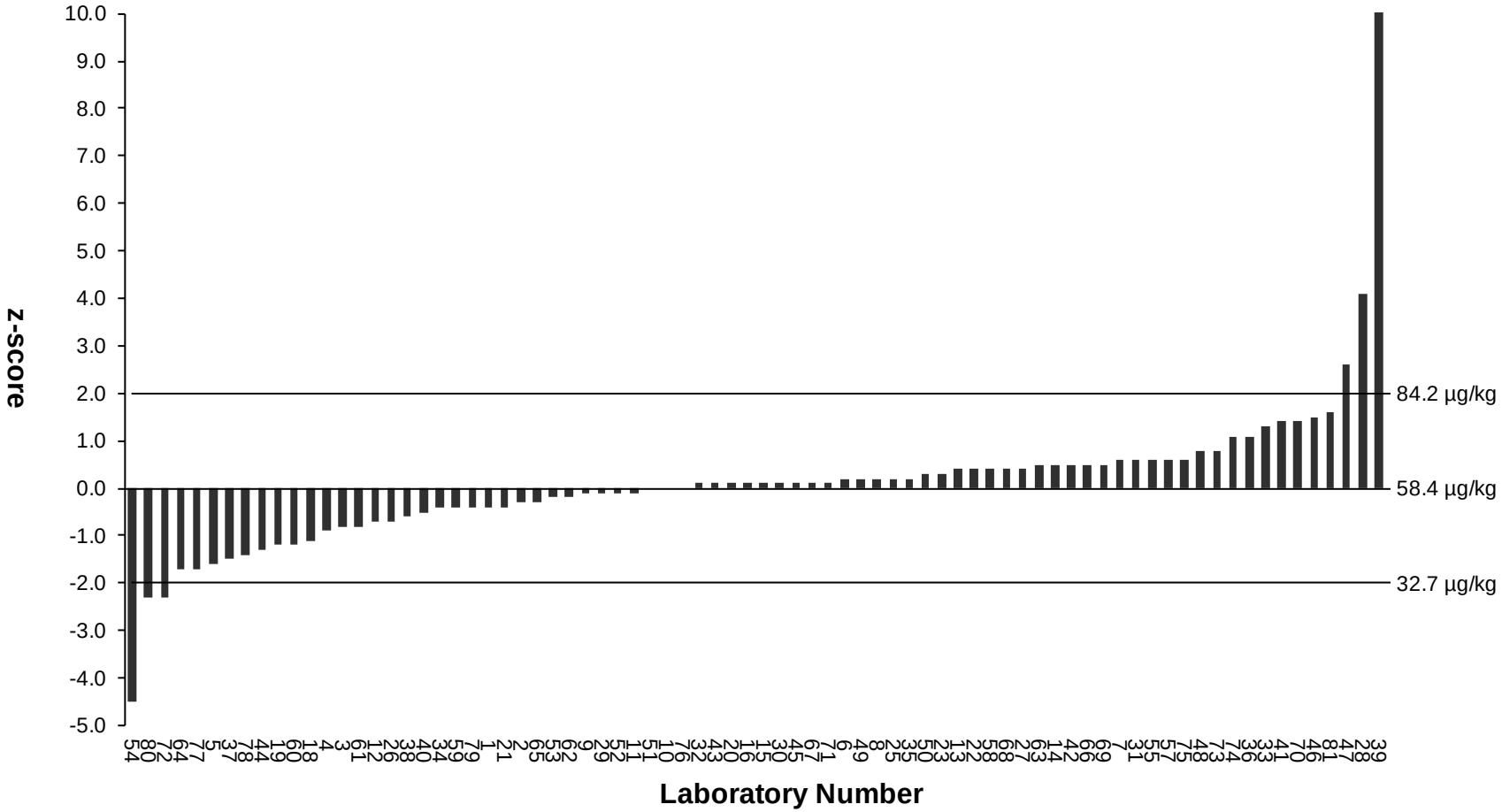

Figure 1: z-Scores for Bifenthrin

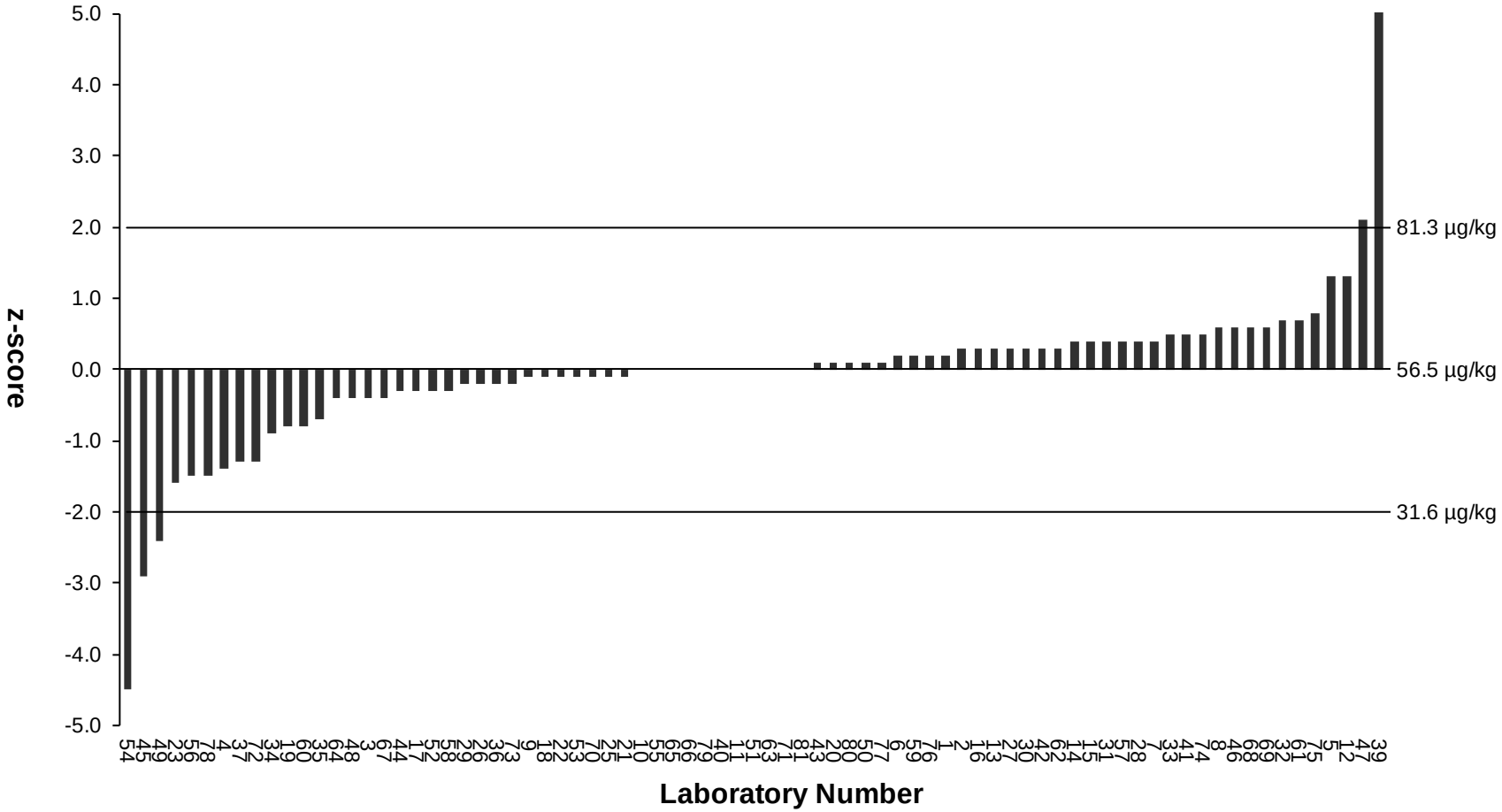

Figure 2: z-Scores for Chlorpyrifos (ethyl)

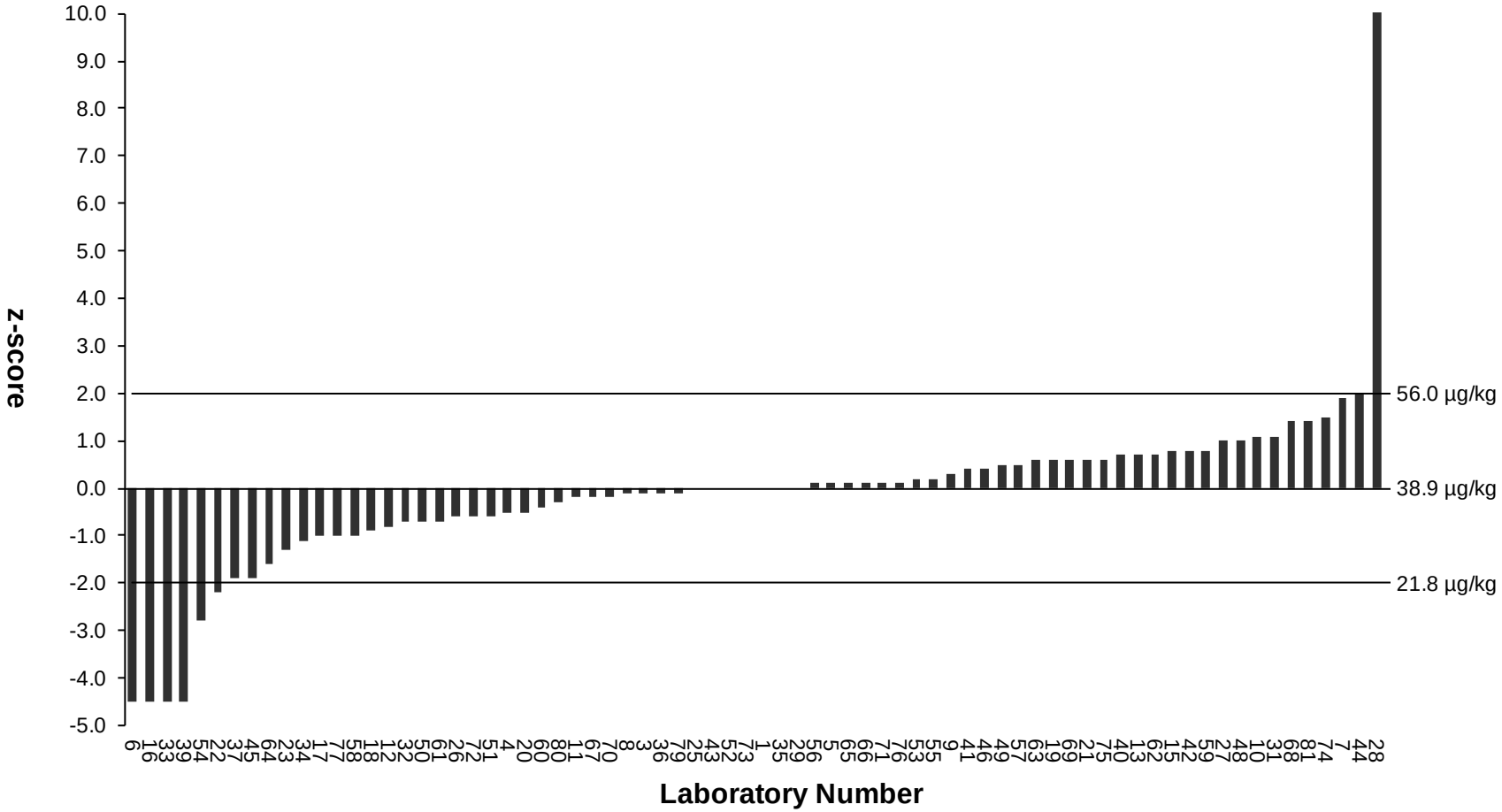

Figure 3: z-Scores for Cyproconazole

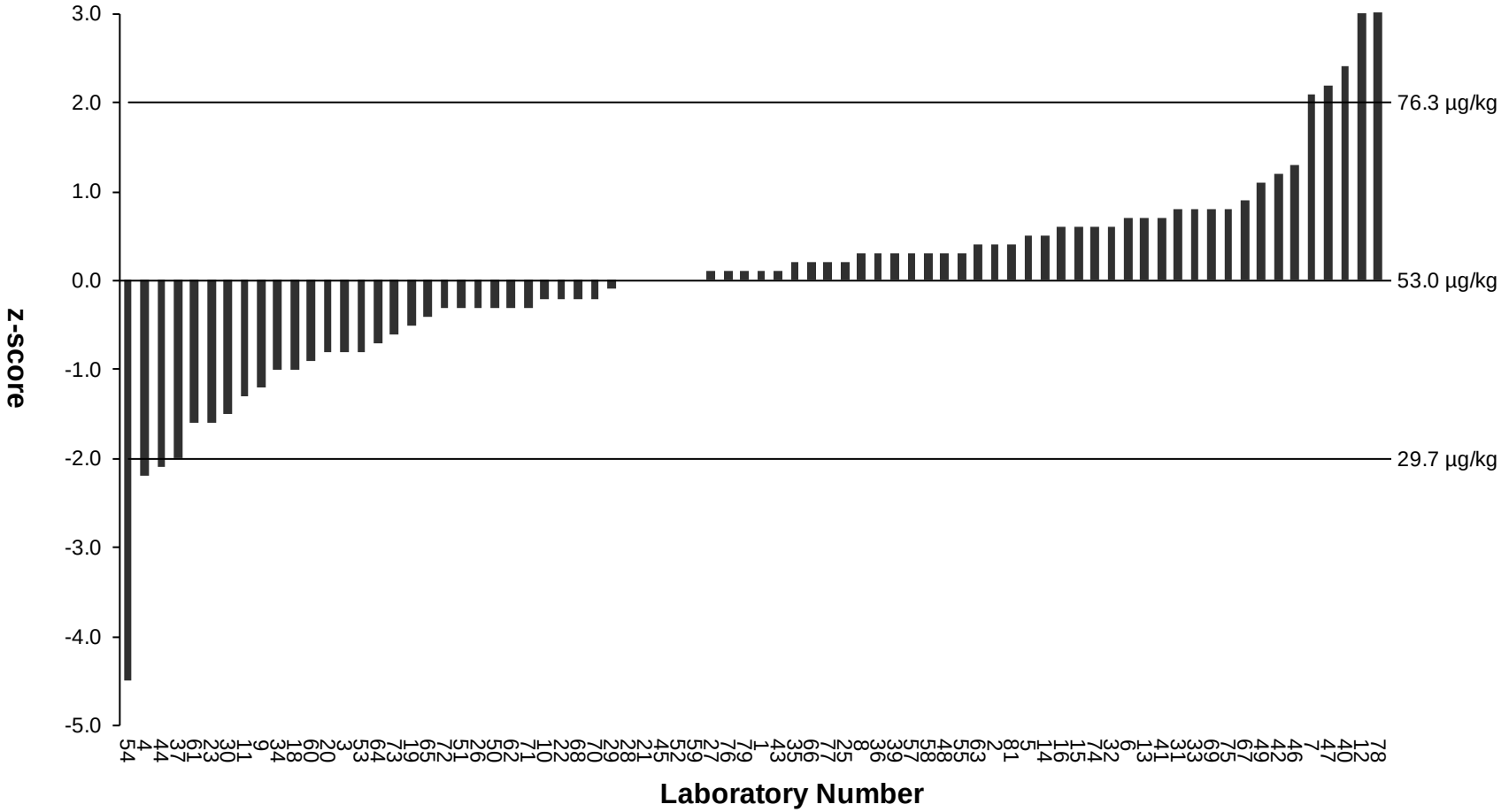

Figure 4: z-Scores for Diazinon

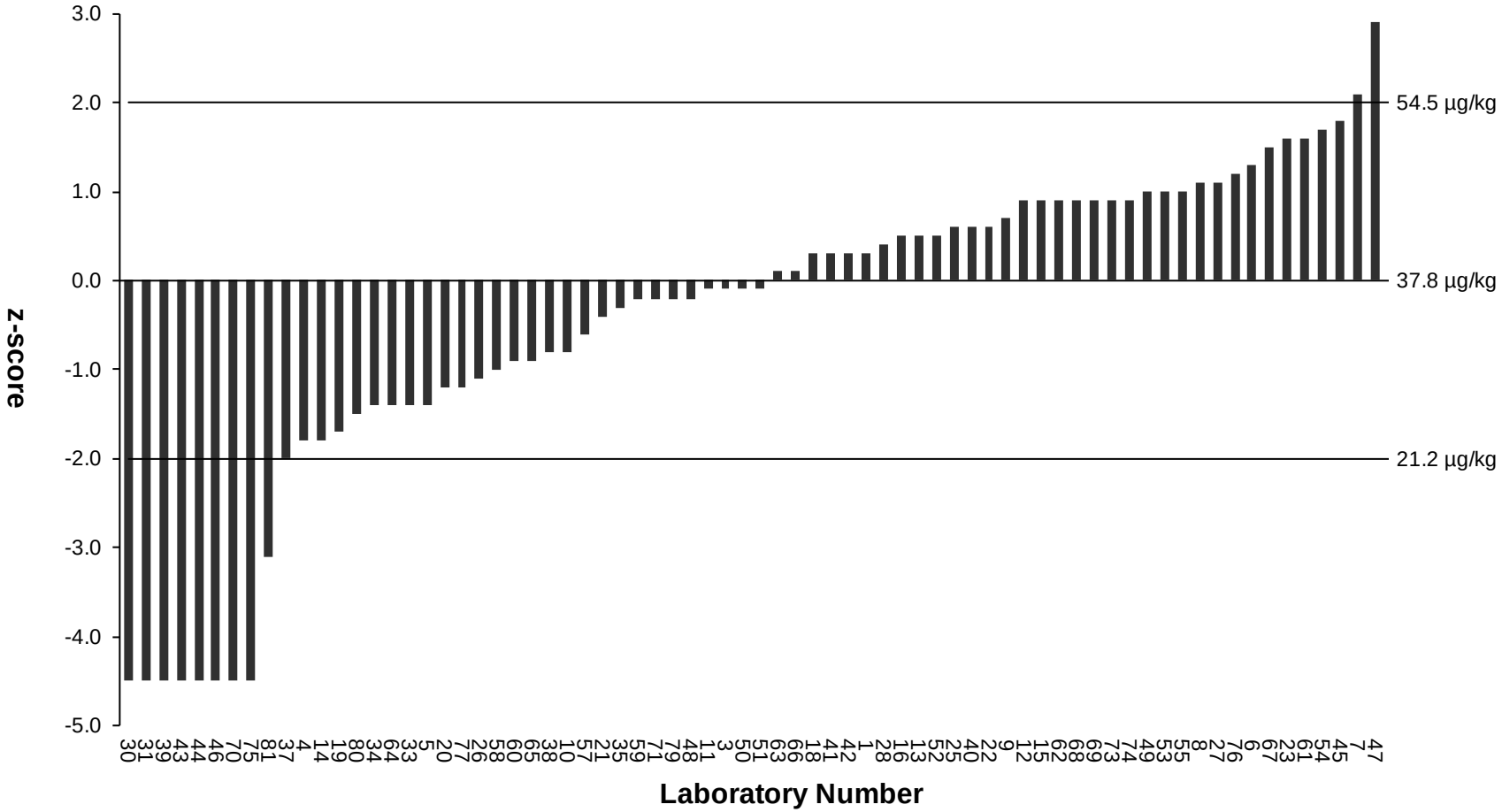

Figure 5: z-Scores for Endosulfan I

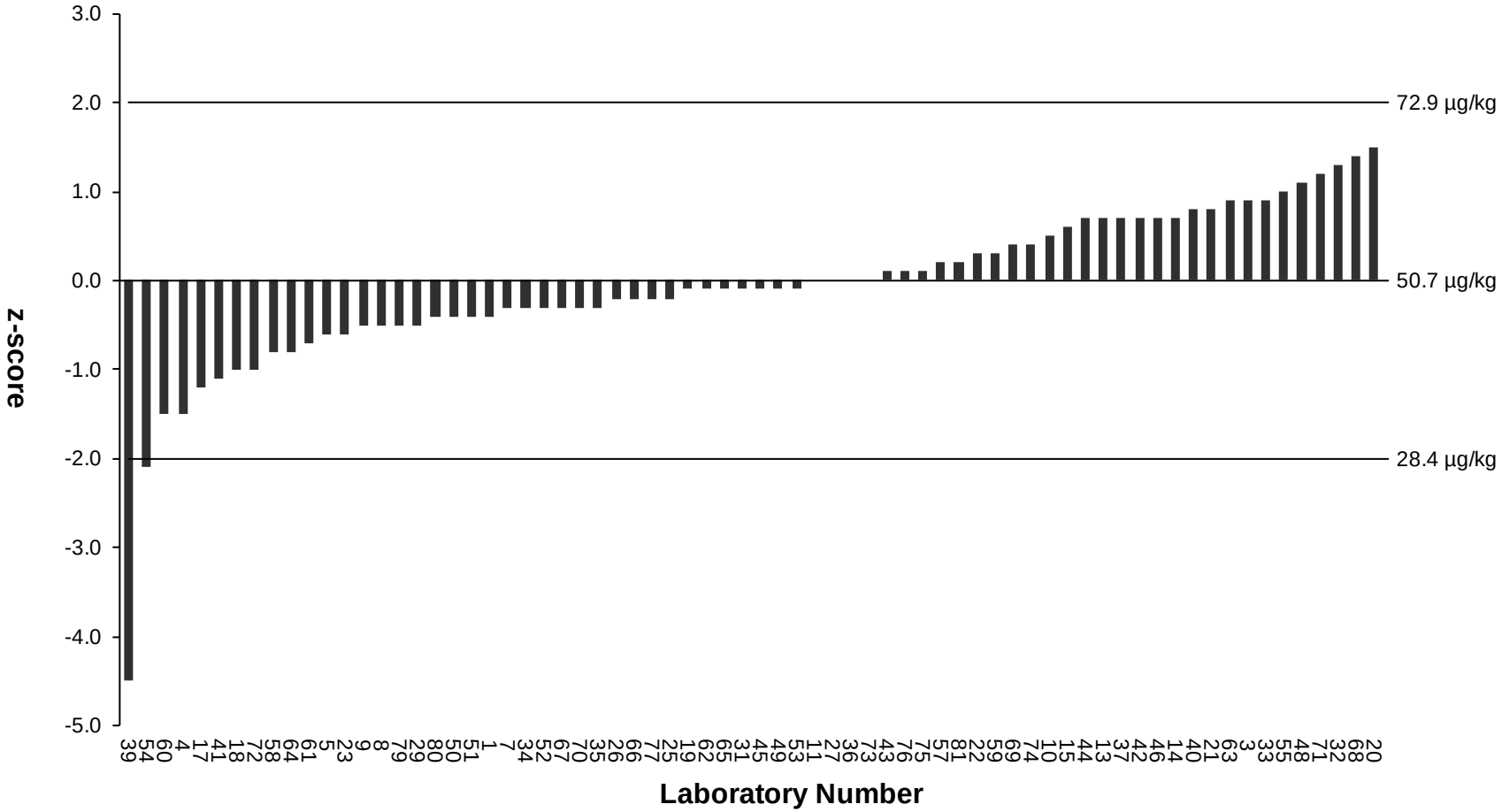

Figure 6: z-Scores for Epoxiconazole

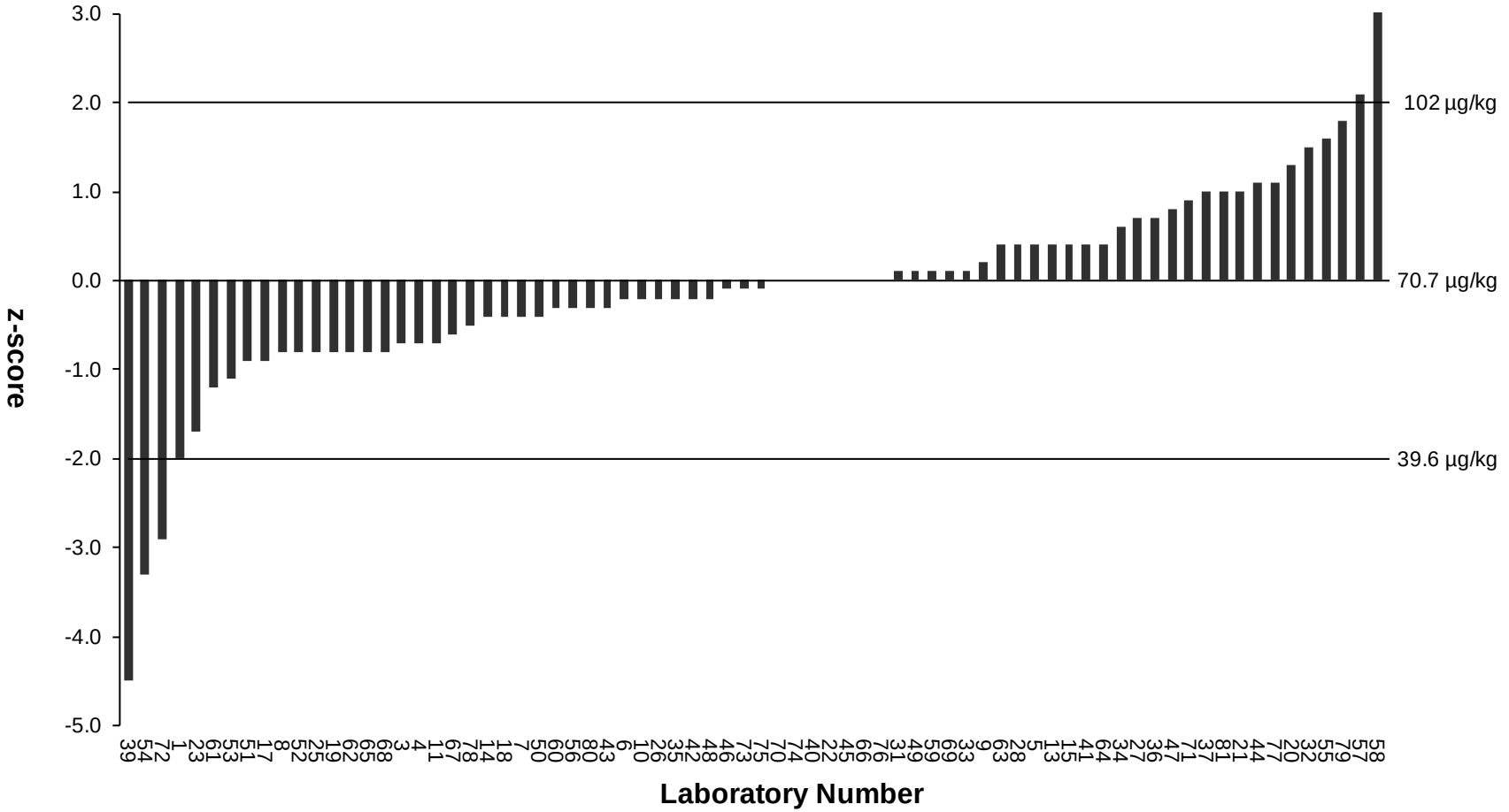

Figure 7: z-Scores for Fenhexamid

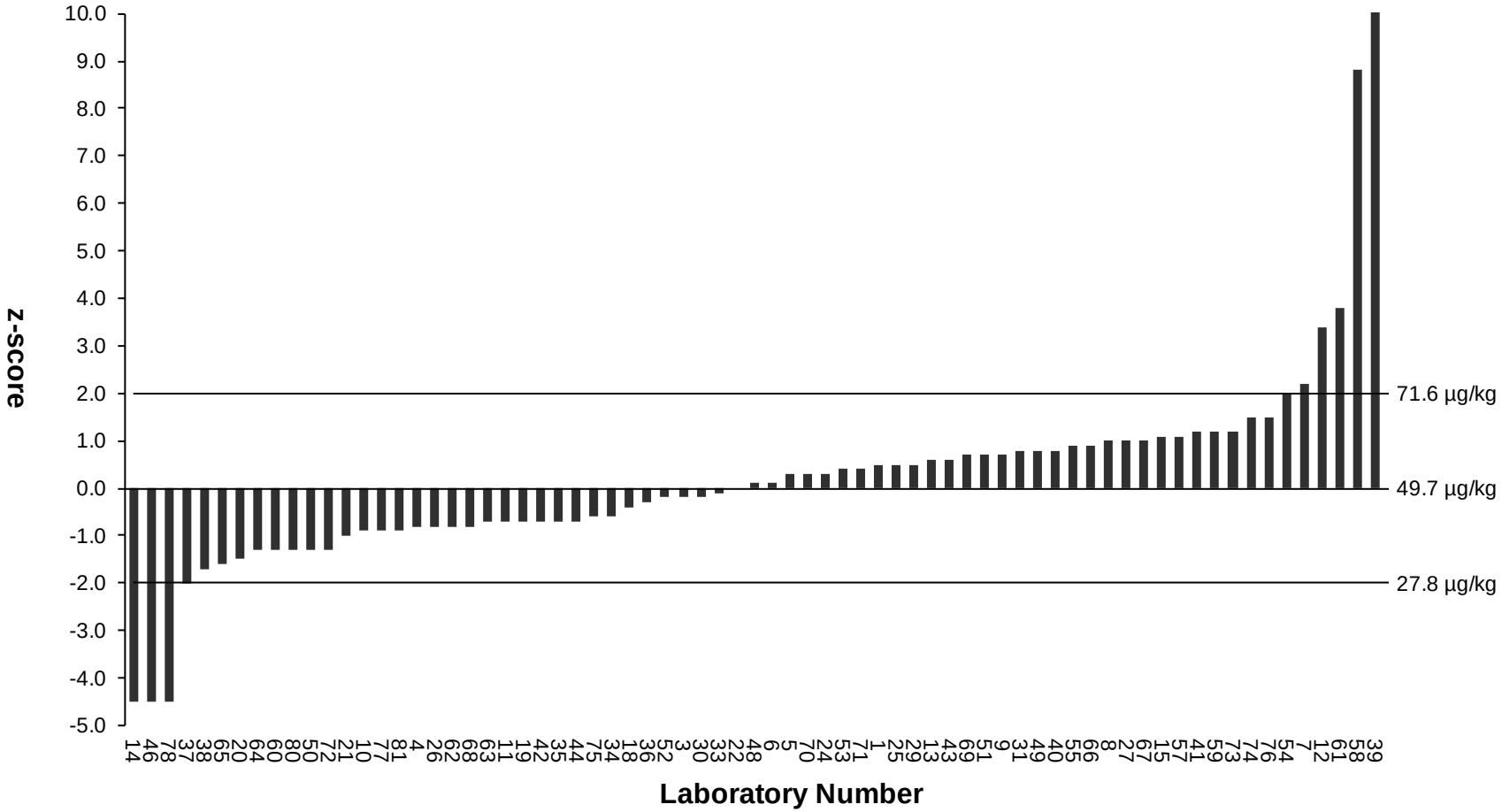

Figure 8: z-Scores for Heptachlor

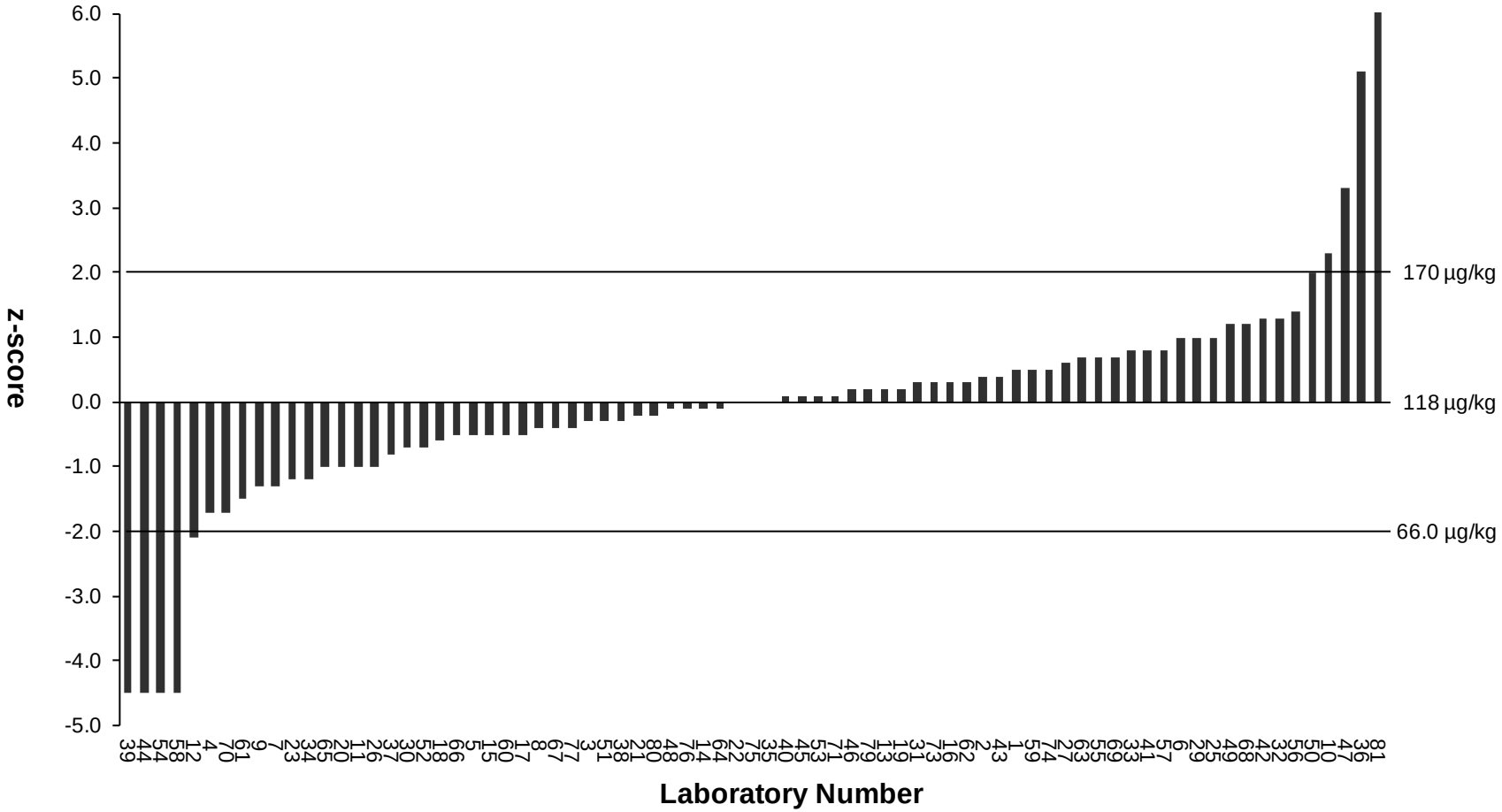

Figure 9: z-Scores for Iprodione

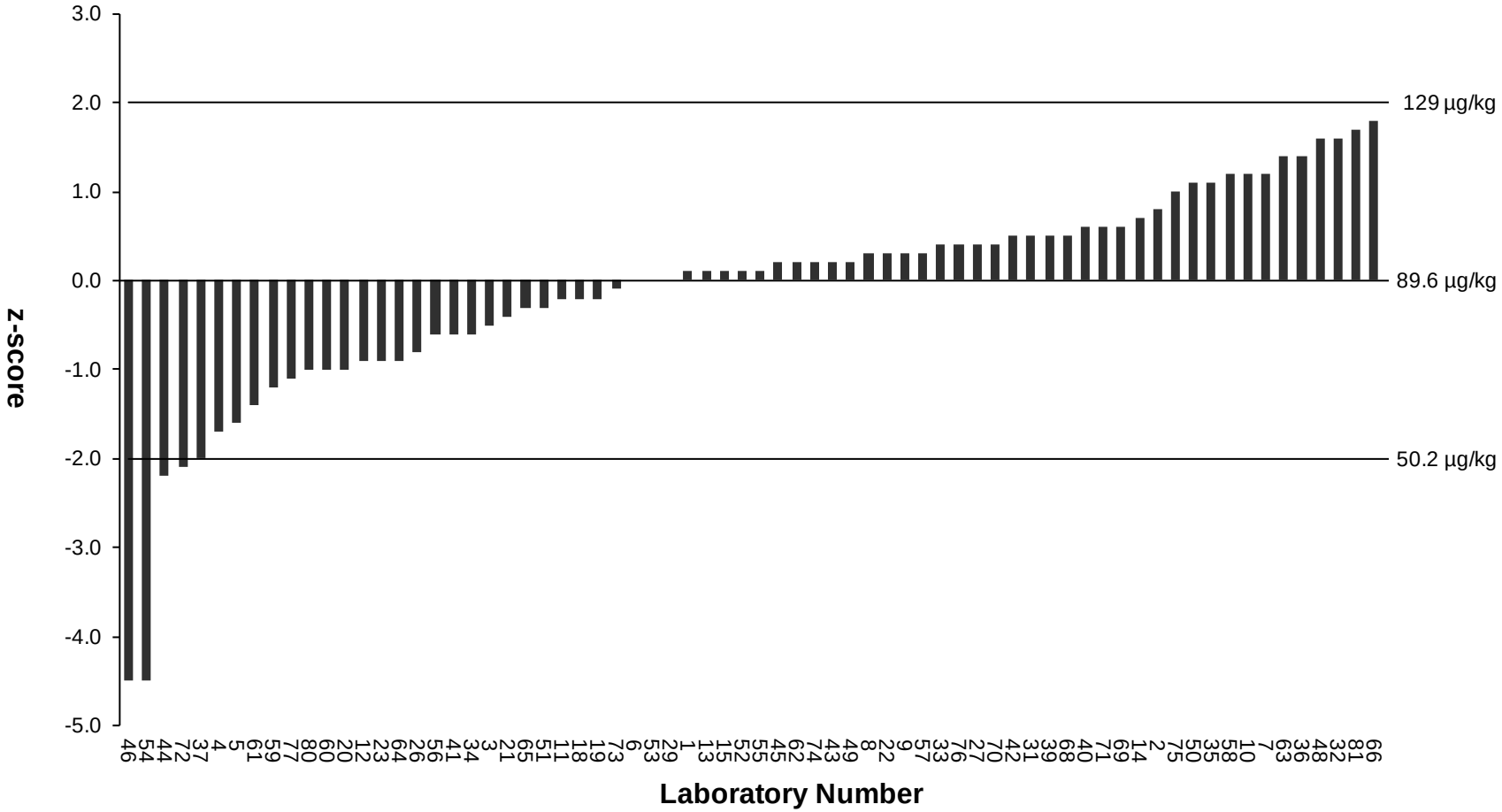

Figure 10: z-Scores for Piperonyl butoxide

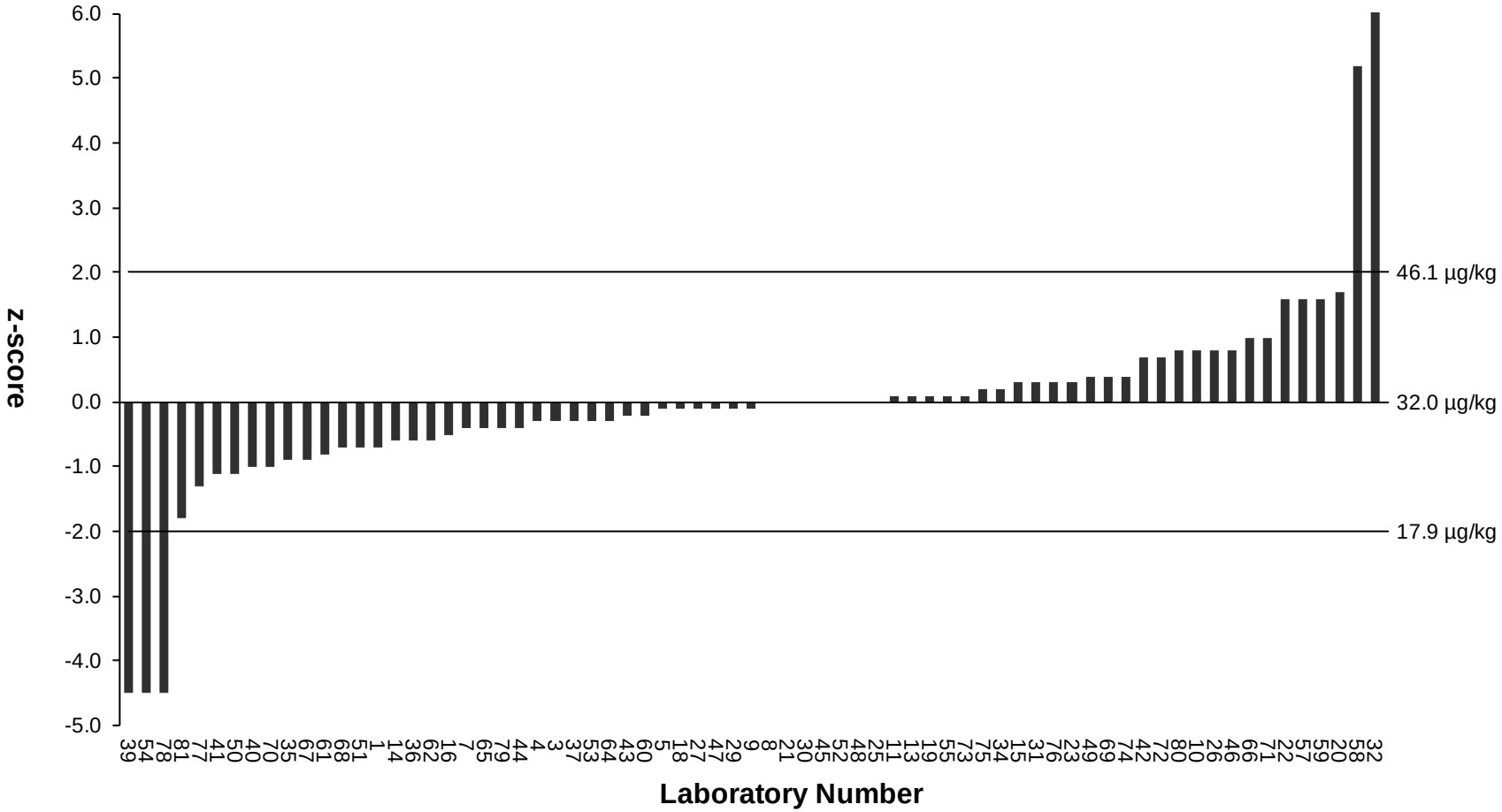

Figure 11: z-Scores for Propoxur

APPENDIX I: Analytical Methods Used by Participants

Methods are tabulated according to the information supplied by participants, but some responses may have been combined or edited for clarity.

Accredited Method Used	laboratory number
no	002 007 012 016 021 024 027 029 063 074 079
yes	001 003 004 005 006 008 011 013 018 019 020 022 025 026 028 031 032 033 034 035 036 038 041 042 043 044 045 046 049 050 052 053 055 057 059 060 061 064 065 066 068 070 073 075 076 078 080

Method Based On	laboratory number
International Standard	001 005 007 011 018 022 027 028 031 032 035 036 038 041 043 045 052 057 063 064 065 068 070 073 075
National Standard	008 013 029 049 055 059 066
Paper Published In An International Journal	002 004 046 060 076 079
Manufacturer/Kit Instructions/Technical Note	021
In house method	003 006 012 016 019 020 024 025 026 034 042 044 050 053 061 074 078 080

Quoted percentage recovery measured in same analytical batch as test material	laboratory number
no	002 005 007 026 028 034 057 066 073
yes	001 003 004 006 008 011 012 013 016 018 019 020 021 022 024 025 027 029 031 032 033 035 036 038 041 042 043 044 045 046 049 050 052 053 055 059 060 063 064 065 068 070 074 075 076 078 079 080

If measured in this batch, stage spike added	laboratory number
prior to extraction	001 002 003 004 005 006 008 011 012 013 016 018 019 020 021 022 024 025 026 027 029 031 032 033 034 035 036 038 041 043 044 045 046 049 050 052 053 055 059 060 063 065 066 068 070 073 074 075 076 078 079 080
prior to instrument measurement	042 064

Concentration of Spike (µg/kg)	laboratory number
≥5 - <10	035 053 063 076
≥10 - <25	001 003 004 005 006 016 020 022 024 025 029 032 034 038 041 045 050 059 060 079 080
≥25 - <50	011 012 021 027 044 065 068
≥50 - <100	002 008 013 018 026 031 036 043 046 052 055 066 070 075
≥100	019 042 049 057 073 078

Composition of Blank Commodity used for Spiking	laboratory number
blank provided	001 003 004 008 011 012 016 018 019 020 021 022 024 025 026 027 029 031 032 034 035 036 038 042 043 046 049 052 053 055 059 060 065 066 068 073 074 075 076 078 079 080
Bio Kiwi	057 061
potato blank sample.	008
commercial blank	045
control sample	070
DI water	002
In house blank raspberry	005 006
organic raspberry	013
Raspberry extract	063
Strawberry	044

Calibration	laboratory number
standard addition	029 059 063 066 080
matrix-matched	003 004 005 006 007 008 013 018 021 025 028 031 034 035 038 041 043 044 046 049 052 053 055 057 059 060 063 068 070 073 074 075 076 078 079
solvent	001 002 011 032 036 042 045 052 053 055 061 070
multi-level	001 002 003 004 011 013 016 019 020 022 024 025 026 027 031 035 036 038 042 043 049 050 053 055 060 063 064 065 068
single-level	008 012 029 053 066 068

Internal Standard Added	laboratory number
no	016 018 021 025 027 028 029 032 060 074 075 079
yes	001 002 003 004 005 006 007 008 011 012 013 019 020 022 024 026 031 034 035 036 038 041 042 043 044 045 046 049 050 052 053 055 057 059 061 063 064 065 066 068 070 073 076 078 080

Internal Standard Used	laboratory number
Atrazine D5	045 073
BNB	006
Bromophos-methyl	011
C13-metalaxyl; D10-malathion; D4-paclobutrazol; D4-myclobutanil; D5-bifenthrin; D5-Bupirimate	078
Difenoconazol d6, Propyzamid d3	013
Chlorpyrifos d10	013 053
TRIS	053
D3-Acetamiprid, D4-Azoxystrobin, D10-Chlorpyrifos, D5-Iprodion, D14-Trifluralin	007
D6-Isoproturon	043 049
Deuterated	041
GC-MS(TPM), LC-MS-MS (ATRd5)	003
HCH D6, PCB C13	005
heptachlor-13C10	024
Multiple	061
Tritan	068
Parathion D10	052
PCB 20, PCB187	043
Sulfotep	057
phenanthrene d-10	002
PCB 31	057 066
Pirimicarb-D6	008 031
TDCP	059
Tributhylphosphate, Anthracene, BDMC	055
Trifenil metano	073
Nicarbazim	036 045 055 059 068 073

Internal Standard Used (continued)	laboratory number
TTP	001 004 019 020 022 026 031 035 036 042 044 046 049 050 059 063 065 066 070 076
TDCPP, PCB-153	019

GC Method: Sample Weight (g)	laboratory number
≥1 - <5	005 012 024 074 075
≥5 - <10	001 006 007 011 019 025 026 028 029 044 053 059 068 076 078 080
≥10 - <20	002 003 004 008 013 016 018 020 021 022 027 031 032 034 035 038 041 042 043 045 046 049 050 052 057 060 061 063 064 065 066 070 073 079
≥20 - <50	036 055

GC Method: Extraction Solvent Components	laboratory number
acetone	007 016 019 022 029 036 055
acetonitrile	001 002 003 005 006 011 013 018 020 021 025 026 027 028 031 032 035 038 041 042 043 044 046 049 050 052 053 057 059 060 063 065 066 068 070 073 074 075 076 078 079
dichloromethane	016 019 080
ethyl acetate	004 008 012 034 036 045 055 064
water	001 036 046 053
1% acetic acid	079
Cyclohexane	036 055
iso octane	024
Petroleum ether	019

GC Method: Extraction pH Adjusted	laboratory number
no	002 003 005 012 016 019 021 022 024 025 026 029 032 038 041 042 045 049 050 052 053 057 059 060 064 066 068 073 075 076 079 080
yes	001 004 006 007 008 011 013 018 020 027 028 031 034 035 036 043 044 046 055 063 065 070 074 078

GC Method: Extraction Techniques Used	laboratory number
macerate at room temperature	004 008 016 042
QuEChERS	001 002 003 005 006 011 013 018 020 021 022 025 026 027 028 029 031 032 035 038 041 042 043 044 045 046 049 050 052 053 057 059 060 063 064 065 066 068 070 073 074 075 076 078 079 080
liquid-liquid partition	007 012 019 024 034 055
DFG S19	036

GC Method: Sample Clean-up Technique	laboratory number
GPC/HPGPC	007
liquid-liquid partition	055
none	004 016 019 022 024 029 036 057 064 066 070 074 075
solid phase extraction (SPE) (column/cartridge)	011 012
solid phase extraction (SPE) (dispersive)	001 002 003 005 006 013 018 020 021 025 026 027 028 031 032 035 038 041 042 043 044 045 046 049 050 052 053 059 060 063 065 068 073 078 079 080

GC Method: SPE Sorbent Type	laboratory number
C18	005 045 057 080
PSA	001 002 003 006 013 018 021 025 027 028 031 032 035 041 043 044 046 049 050 052 053 059 060 063 065 068 070 073 076 079
Mixed Mode	011 020 026 038 078
MgSO4	025
none	055

GC Method: GC Column Packing	laboratory number
50% methyl 50% phenyl polysiloxane	022 050 065
65% methyl 35% phenyl polysiloxane	060
95% methyl 5% phenyl polysiloxane	001 002 004 005 006 007 008 011 012 016 018 019 020 024 025 027 028 029 031 032 034 035 036 038 041 042 043 044 045 046 049 052 053 055 057 059 063 066 068 070 073 074 075 078 079
5% Difenil 95% Dimetilpolixiloxano	026

GC Method: GC Column Packing (continued)	laboratory number
50% Agua -50% metanol	080
95% Dimethylpolysiloxane 5% Polysilarylene	021
Ã¢Â€Â¢(14%-Cyanopropyl-phenyl)-methylpolysiloxane	003

GC Method: GC Detector Type	laboratory number
ECD	019
MS	003 006 012 028 032 036 044 045 046 049 066 068
MS-MS	001 002 004 005 007 008 011 013 016 018 019 020 021 022 024 025 026 027 029 031 034 035 038 041 042 043 050 052 053 055 057 059 060 063 064 065 066 070 073 074 075 076 078 079 080

LC Method: Sample Weight (g)	laboratory number
≥1 - <5	005 007 055 074 075
≥5 - <10	001 011 019 025 026 028 029 036 053 059 068 076 078 080
≥10 - <20	003 004 006 008 013 016 018 020 021 022 027 031 032 034 035 038 041 042 043 044 045 046 049 050 052 057 060 061 063 064 065 066 070 073

LC Method: Extraction Solvent Components	laboratory number
acetone	016 019 029
acetonitrile	001 003 004 005 006 007 011 013 018 020 021 022 025 026 027 028 031 032 034 035 036 038 041 042 043 044 045 046 049 050 052 053 055 057 059 060 063 064 065 066 068 070 073 074 075 076 078
dichloromethane	016 019 080
ethyl acetate	008
petroleum ether	019
water	053

LC Method: Extraction pH Adjusted	laboratory number
no	003 005 016 019 021 022 025 026 029 032 038 041 042 045 050 052 053 057 059 060 064 066 068 073 075 076 080
yes	001 004 006 007 008 011 013 018 020 027 028 031 034 035 036 043 044 046 049 055 063 065 070 074 078

LC Method: Extraction Techniques Used	laboratory number
macerate at room temperature	008 016 042
QuEChERS	001 003 004 005 006 007 011 013 018 020 021 022 025 026 027 028 029 031 032 034 035 036 038 041 042 043 044 045 046 049 050 052 053 055 057 059 060 063 064 065 066 068 070 073 074 075 076 078 080
liquid-liquid partition	019

LC Method: Sample Clean-up Technique	laboratory number
liquid-liquid partition	034
none	003 007 016 019 022 029 042 043 049 055 057 065 066 070 074 075
solid phase extraction (SPE) (column/cartridge)	011
solid phase extraction (SPE) (dispersive)	001 004 005 006 013 018 020 021 025 026 027 028 031 032 035 036 038 044 045 046 050 052 053 059 060 063 064 068 073 078 080

LC Method: SPE Sorbent Type	laboratory number
C18	005 006 045 057 080
PSA	001 004 013 018 021 025 027 028 031 032 035 036 044 046 050 052 053 059 060 063 064 068 070 073 076
Mixed Mode	011 020 026 038 078
MgSO4	025
none	055

LC Method: HPLC Column Packing	laboratory number
C18	001 003 004 006 007 008 011 016 018 019 020 022 025 026 027 028 029 031 032 034 035 036 038 041 042 043 044 045 046 049 050 052 053 055 059 060 063 065 068 073 074 075 076 078 080
C8	005 057
Biphenyl	021
Fortis	070
PFP	066

LC method: Mobile Phase Components	laboratory number
ethanoic acid (acetic acid)	043 045 053 068
acetonitrile	004 013 029 035 043 063 066
ammonium acetate	008 019 027 028 060
ammonium formate	001 003 004 006 007 011 018 020 021 025 026 031 035 036 038 042 044 045 046 049 052 053 055 063 064 068 070 073 075
formic acid (methanoic acid)	001 003 005 013 020 021 022 026 027 028 029 035 038 041 053 063 064 066 076
methanol	001 003 007 008 011 016 018 019 020 021 025 026 027 028 031 032 034 036 038 042 044 045 046 049 050 052 053 055 057 059 060 064 065 068 070 074 075 076 078 080
THF	075
water	001 004 007 011 013 016 018 019 021 026 028 031 035 036 038 042 043 045 046 049 050 052 053 055 057 059 060 063 064 066 068 074 075 076 078

LC Method: Detector Type	laboratory number
MS-MS	001 003 004 005 006 007 008 011 013 016 018 019 020 021 022 025 026 027 028 029 031 032 034 035 036 038 041 042 043 044 045 046 049 050 051 052 053 055 057 059 060 063 064 065 066 068 073 074 075 076 078 080
LC-QTof	070

Bifenthrin

Source of Standards	laboratory number
Accu Standard	022
Dr Ehrenstorfer	006 008 016 019 032 034 035 041 043 050 055 057 058 059 060 065 066 076
Sigma/Aldrich	011 030 038 070 075
Wako	027
Chem service	003
CPAChem	025
HPC	049
Lab instruments	026 081
Restek	002 021
Scharlab (Pesticides mix)	079
SPEX	053

Method Principle	laboratory number
GC	002 003 006 007 008 011 013 016 019 021 022 025 026 027 030 032 035 036 038 041 042 043 049 050 055 057 058 059 060 065 066 068 070 076 078 079
LC	031 053 069 074 075 081

Identification by Mass Spectrometry	laboratory number
no	070
yes	002 003 006 007 008 011 013 016 019 021 022 025 026 027 030 031 032 034 035 036 038 041 042 043 049 050 053 055 057 058 059 060 065 066 068 069 074 075 076 078 079 081

Chlorpyrifos (ethyl)

Source of Standards	laboratory number
Accu Standard	022
Dr Ehrenstorfer	006 008 011 016 019 032 034 035 041 043 050 055 057 058 059 060 066 076
Sigma/Aldrich	030 065 070 075

Source of Standards (continued)	laboratory number
Wako	027
Chem service	003
CPAChem	025
HPC	049
Lab instruments	026 081
Restek	002 021
CertiPrep	002
SPEX	002 053

Method Principle	laboratory number
GC	002 003 006 007 008 013 016 019 021 022 026 027 030 031 034 035 036 041 043 049 050 055 057 058 059 060 065 066 069 076 078 079
LC	011 025 032 042 053 068 070 074 075 081

Identification by Mass Spectrometry	laboratory number
no	070
yes	002 003 006 007 008 011 013 016 019 021 022 025 026 027 030 031 032 034 035 036 041 042 043 049 050 053 055 057 058 059 060 065 066 068 069 074 075 076 078 079 081

Cyproconazole

Source of Standards	laboratory number
Accu Standard	022
Dr Ehrenstorfer	008 011 019 032 034 035 041 043 050 055 057 058 059 060 066 076
Sigma/Aldrich	065 069 070 075
Wako	027
Micotox	049
Chem service	003
CPAChem	025
HPC Standards	049
Lab instruments	026 081

Source of Standards (continued)	laboratory number
Restek	021
SPEX	053

Method Principle	laboratory number
GC	003 007 011 019 022 026 034 035 041 049 057 058 060 065 066 076 079
LC	008 013 021 025 027 031 032 036 042 043 050 053 055 059 068 069 070 074 075 081

Identification by Mass Spectrometry	laboratory number
no	070
yes	003 007 008 011 013 019 021 022 025 026 027 031 032 035 036 041 042 043 049 050 053 055 057 058 059 060 065 066 068 069 074 075 076 079 081

Diazinon

Source of Standards	laboratory number
Dr Ehrenstorfer	006 008 016 019 032 034 035 041 043 050 055 057 059 060 066 076
Sigma/Aldrich	011 030 065 069 075
Wako	027
Chem service	003
CPAChem	025
HPC	049
Lab instruments	026 081
Restek	002 021
CertiPrep	002
SPEX	002 053

Method Principle	laboratory number
GC	002 003 006 007 013 016 019 021 022 026 027 034 035 041 049 050 055 057 058 060 065 066 076 078 079
LC	008 011 025 030 031 032 036 042 043 053 059 068 069 074 075 081

Identification by Mass Spectrometry	laboratory number
yes	002 003 006 007 008 011 013 016 019 021 022 025 026 027 030 031 032 034 035 036 041 042 043 049 050 053 055 057 058 059 060 065 066 068 069 074 075 076 078 079 081

Endosulfan I (alpha)

Source of Standards	laboratory number
Accu Standard	022
Dr Ehrenstorfer	006 008 016 019 034 035 041 050 055 057 058 059 060 066 076
Sigma/Aldrich	011 038 065
Wako	027
Chem service	003
CPAChem	025
HPC Standards	049
Lab instruments	026 081
Restek	021
SPEX	053

Method Principle	laboratory number
GC	003 006 007 008 011 013 016 019 021 022 025 026 027 034 035 038 041 042 049 050 053 055 057 058 059 060 065 066 068 069 074 076 078 079
LC	081

Identification by Mass Spectrometry	laboratory number
yes	003 006 007 008 011 013 016 019 021 022 025 026 027 034 035 038 041 042 049 050 053 055 057 058 059 060 065 066 068 069 074 076 078 079 081

Epoxiconazole

Source of Standards	laboratory number
Accu Standard	022
BDH	060
Dr Ehrenstorfer	008 011 019 031 032 034 035 041 043 050 055 057 059 065 066 076
Fisher	058
Sigma/Aldrich	070 075
Wako	027
Chem service	003
CPAChem	025
HPC	049
Lab instruments	026 081
Restek	021
SPEX	053

Method Principle	laboratory number
GC	027 035 041 049 057 066 079
LC	003 007 008 011 013 019 021 022 025 026 031 032 036 042 043 050 053 055 058 059 060 065 068 069 070 074 075 076 081

Identification by Mass Spectrometry	laboratory number
no	070
yes	003 007 008 011 013 019 021 022 025 026 027 031 032 034 035 036 041 042 043 049 050 053 055 057 058 059 060 065 066 068 069 074 075 076 079 081

Fenhexamid

Source of Standards	laboratory number
Accu Standard	022
Dr Ehrenstorfer	006 008 011 019 032 034 035 041 043 050 055 057 058 059 060 066 070
Sigma/Aldrich	065 075
Wako	027

Source of Standards (continued)	laboratory number
Chem service	003
CPAChem	025
HPC	049
Lab instruments	026 081
Restek	021
SPEX	053

Method Principle	laboratory number
GC	006 011 031 055 057 058 065 069 079
LC	003 007 008 013 019 021 022 025 026 027 032 034 035 036 041 042 043 049 050 053 059 060 066 068 070 074 075 078 081

Identification by Mass Spectrometry	laboratory number
no	070
yes	003 006 007 008 011 013 019 021 022 025 026 027 031 032 034 035 036 041 042 043 049 050 053 055 057 058 059 060 065 066 068 069 074 075 078 079 081

Heptachlor

Source of Standards	laboratory number
Accu Standard	022
Dr Ehrenstorfer	006 008 019 031 034 035 041 043 050 055 057 058 059 060 066 070 076
Fisher	076
Sigma/Aldrich	011 030 038 065 069 075
Wako	027
Chem service	003
CPAChem	025
HPC	049
Lab instruments	026 081
Restek	021 024
SPEX	053

Method Principle	laboratory number
GC	003 006 007 008 011 013 019 021 022 024 025 026 027 030 031 035 036 038 041 042 043 049 050 053 055 057 058 059 060 065 066 068 069 070 074 075
LC	034 076 081

Identification by Mass Spectrometry	laboratory number
no	019 070
yes	003 006 007 008 011 013 021 022 024 025 026 027 030 031 034 035 036 038 041 042 043 049 050 053 055 057 058 059 060 065 066 068 069 074 075 076 081

Iprodione

Source of Standards	laboratory number
Accu Standard	022
Dr Ehrenstorfer	006 007 008 016 019 032 034 035 041 043 050 055 057 059 060 066 070 076
Sigma/Aldrich	011 030 038 065 069 075
Wako	027
Chem service	003
CPAChem	025
HPC	049
Lab instruments	026 081
Restek	002 021
SPEX	053

Method Principle	laboratory number
GC	002 003 006 008 011 013 016 019 022 025 026 030 031 034 035 036 038 041 042 043 049 050 053 055 057 059 060 065 070 075 076 078 079
LC	007 021 027 032 066 068 069 074 081

Identification by Mass Spectrometry	laboratory number
no	070
yes	002 003 006 007 008 011 013 016 019 021 022 025 026 027 030 031 032 034 035 036 038 041 042 043 049 050 053 055 057 059 060 065 066 068 069 074 075 076 078 079 081

Piperonyl butoxide

Source of Standards	laboratory number
Accu Standard	022
Dr Ehrenstorfer	006 008 019 032 034 035 041 043 050 055 057 058 059 060 066 070 076
Sigma/Aldrich	011 065 075
Wako	027
Chem service	003
HPC	049
Lab instruments	026 081
Restek	002 021
SPEX	053

Method Principle	laboratory number
GC	002 003 006 007 011 013 021 022 026 027 031 032 034 035 036 041 043 049 050 055 057 060 066 069 076
LC	008 019 042 053 058 059 065 068 070 074 075 081

Identification by Mass Spectrometry	laboratory number
no	070
yes	002 003 006 007 008 011 013 019 021 022 026 027 031 032 034 035 036 041 042 043 049 050 053 055 057 058 059 060 065 066 068 069 074 075 076 081

Propoxur

Source of Standards	laboratory number
Accu Standard	022
BDH	058
Dr Ehrenstorfer	008 016 019 032 034 035 041 043 050 055 057 058 059 060 066 070 076
Fluka	031
Sigma/Aldrich	011 030 065 075
Wako	027
Chem service	003
CPAChem	025
HPC	049
Lab instruments	026 081
Restek	021
SPEX	053

Method Principle	laboratory number
GC	013 019 049 057 058 060 066 069 079
LC	003 007 008 011 016 021 022 025 026 027 030 031 032 034 035 036 041 042 043 050 053 055 059 065 068 070 074 075 076 081

Identification by Mass Spectrometry	laboratory number
no	070
yes	003 007 008 011 013 016 019 021 022 025 026 027 030 031 032 034 035 036 041 042 043 049 050 053 055 057 058 059 060 065 066 068 069 074 075 076 079 081

APPENDIX II: Fapas® SecureWeb, Protocol and Contact Details

1. Fapas® SECUREWEB

Access to the secure area of our website is only available to participants in our proficiency tests. Please contact us if you require a UserID and Password. Fapas® SecureWeb allows participants to:

- Obtain their laboratory numbers for the proficiency tests in which they have participated.
- View the results they submitted in past and current proficiency tests.
- Submit their results and methods for current tests.
- Review future tests they have ordered.
- Order proficiency tests, reference materials and quality control materials.
- Freely download copies of reports (PDF file), of proficiency tests in which they have participated.
- View charts of their z-scores obtained in previous Fapas® – Food Chemistry proficiency tests.

2. PROTOCOL

The Protocols [4, 5] set out how Fapas® – Food Chemistry is organised. Copies can be downloaded from our website.

3. CONTACT DETAILS

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

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