FAPAS® Report 19167

Pesticide Residues in Soya Beans

March-May 2014

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
0009

SUMMARY

1. The test material for FAPAS® proficiency test 19167 was dispatched in March 2014. Each participant received a soya bean test material.
2. From a list of 209 pesticide residues, participants had to identify and quantify those present. The test material contained Buprofezin, Diazinon, Difenoconazole, Endosulfan sulfate, Metalaxyl, 4,4'-Methoxychlor, Parathion-methyl, Tebufenozide, and Glyphosate.
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
Buprofezin	38.9	23	26	88
Diazinon	120	26	29	90
Difenoconazole	107	22	27	81
Endosulfan sulfate	81.6	15	23	65
Metalaxyl	76.8	24	27	89
4,4'-Methoxychlor	43.5	14	20	70
Parathion-methyl	37.8	21	24	88
Tebufenozide	61.0	19	22	86
Glyphosate	500	5	10	50

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

CONTENTS

1. INTRODUCTION

1.1. Proficiency Testing

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

Further details of the FAPAS® proficiency testing scheme are available in our protocols [3, 4].

2. TEST MATERIAL

2.1. Preparation

Preparation of the samples for this proficiency test was sub-contracted to a laboratory meeting the quality requirements of the scheme's accreditation [2].

The test material was prepared from fresh soya beans. The spiked and blank test materials were both prepared from the same bulk material.

Sub-samples were taken to screen for the possible presence of incurred residues. No incurred residues were detected at or above, 25 µg/kg.

Buprofezin, Diazinon, Difenoconazole, Endosulfan sulfate, Metalaxyl, 4,4'-Methoxychlor, Parathion-methyl, Tebufenozide, and Glyphosate were spiked into the test material.

Samples were stored at -20°C until dispatch.

2.2. Homogeneity

To test for homogeneity, randomly selected test materials were analysed in duplicate. Testing was sub-contracted to a laboratory meeting the quality requirements of the scheme's accreditation [2].

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

2.3. Dispatch

The start date was 31 March 2014. Test materials were sent to 39 participants.

3. RESULTS

The instructions for reporting results were as follows:

- Determine the level of pesticide residues present in the test material, in µg/kg, as received, uncorrected for recovery, together with the percentage recovery and limit of quantification (LoQ).
- All pesticide residues are to be reported as the parent compound only, unless specified otherwise on the results form.

Results were submitted by 32 participants (82%) before the closing date for this test, 15 May 2014.

Each participant was given a laboratory number, assigned in order of receipt of results. The reported analyte concentrations are given in Table 1 for Buprofezin, Diazinon, Difenoconazole, Table 2 for Endosulfan sulfate, Metalaxyl, 4,4'-Methoxychlor and Table 3 for Parathion-methyl, Tebufenozide and Glyphosate.

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

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

Any participant identifying pesticide residues other than Buprofezin, Diazinon, Difenoconazole, Endosulfan sulfate, Metalaxyl, 4,4'-Methoxychlor Parathion-methyl, Tebufenozide and Glyphosate at levels above 25 µg/kg is listed 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) qualitative results, e.g. <LoQ,,

ii) results where no recovery percentage was reported,

iii) 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)

For all analytes, this procedure was straightforward and the median was chosen as the assigned value because of the low number of valid results.

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 [6].

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 all analytes are given in Table 1, Table 2 and Table 3 and shown as histograms in Figures 1–9. 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 14/05/2014.

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 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 Buprofezin, Diazinon and Difenoconazole

laboratory number	analyte											
	Buprofezin assigned value 38.9 µg/kg				Diazinon assigned value 120 µg/kg				Difenoconazole assigned value 107 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	38.9	84.2	10	0.0	142.1	106.6	10	0.8	113.0	99.7	10	0.3
002	#				#				#			
003	35	105	10.0	-0.5	106		10.0	-0.5	98.4		10.0	-0.4
004	47		10	0.9	136		10	0.6	55		10	**-2.2**
005	54	S	10	1.8	126	S	10	0.2	116	S	10	0.4
006	34.2063	93.865	6.9	-0.5	130.8149	79.833	6.1	0.4	#			
007	33		10	-0.7	84		10	-1.4	74		10	-1.4
008	45.8		10	0.8	128		10	0.3	121		10	0.6
009	#				82.3	90	5	-1.4	0			**-4.5**
010	#				63.62	100.1	10	**-2.1**	#			
011	#				114.45	83.62	10	-0.2	92.05	76.30	10	-0.6
012	46	91	10	0.8	153	105	10	1.2	110	92	10	0.1
013	36.5	84.5	10	-0.3	120	98.4	10	0.0	107	99.2	10	0.0
014	28	95	10	-1.3	94	103	10	-1.0	93	91	10	-0.6
015	38		10	-0.1	156		10	1.4	135		10	1.2

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

Table 1 (continued): Results and z-Scores for Buprofezin, Diazinon and Difenoconazole

laboratory number	analyte											
	Buprofezin assigned value 38.9 µg/kg				Diazinon assigned value 120 µg/kg				Difenoconazole assigned value 107 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	#				0.12		0.06	**-4.5**	#			
017	41.09	94.99	10	0.3	123.18	84.49	10	0.1	115.99	96.29	10	0.4
018	47.5	88	10	1.0	112.3	81	10	-0.3	114.5	100	10	0.3
019	#				#				#			
020	43	90	10	0.5	129	85	10	0.3	102	82	10	-0.2
021	45.3	93	10	0.7	129.5	98	10	0.4	98	100	10	-0.4
022	32.0		10	-0.8	93.14		10	-1.0	87.44		10	-0.8
023	40.3	88	10	0.2	#				95.2	92	10	-0.5
024	45	98	10	0.7	110	99	10	-0.4	110	99	10	0.1
025	36	85	10	-0.3	135	91	10	0.6	107	91	10	0.0
026	20.83	107.2	10	**-2.1**	73.68	105.81	10	-1.8	57.46	109.77	10	**-2.1**
027	0			**-4.5**	150	87	10	1.1	110	93	10	0.1
028	55	102	10	1.9	154	88	10	1.3	180	108	10	**3.1**
029	46.9		10	0.9	148.2		10	1.1	109.5		10	0.1
030	0.015	89.5	0.010	**-4.5**	0.053	95.0	0.010	**-4.5**	0.051	85.0	0.010	**-4.5**

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

Table 1 (continued): Results and z-Scores for Buprofezin, Diazinon and Difenoconazole

laboratory number	analyte											
	Buprofezin assigned value 38.9 µg/kg				Diazinon assigned value 120 µg/kg				Difenoconazole assigned value 107 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	37	106	10	-0.2	113	92	10	-0.3	105	86	10	-0.1
032	36.8	78	10	-0.2	119.6	82	10	0.0	135.2	82	10	1.2

Table 2: Results and z-Scores for Endosulfan sulfate, Metalaxyl and 4,4'-Methoxychlor

laboratory number	analyte											
	Endosulfan sulfate assigned value 81.6 µg/kg				Metalaxyl assigned value 76.8 µg/kg				4,4'-Methoxychlor assigned value 43.5 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	128.5	101.0	10	**2.6**	75.7	103.4	10	-0.1	29.6	85.1	10	-1.5
002	#				#				#			
003	#				76.2		10.0	0.0	50.0		10.0	0.7
004	92		10	0.6	113		10	**2.1**	46		10	0.3
005	110	S	10	1.6	64	S	10	-0.8	64	S	10	**2.1**
006	#				85.5527	95.500	5.3	0.5	#			
007	61		10	-1.1	95		10	1.1	0			**-4.5**
008	80.7		10	0.0	77.0		10	0.0	57.7		10	1.5
009	0			**-4.5**	79.7	95	5	0.2	#			
010	75.20	95.7	5	-0.4	#				21.19	92.8	5	**-2.3**
011	#				58.49	84.17	10	-1.1	42.08	81.62	10	-0.2
012	0			**-4.5**	75	97	10	-0.1	45	81	10	0.2
013	77.9	78.3	10	-0.2	65.4	93.4	10	-0.7	42.0	84.0	10	-0.2
014	#				#				#			
015	320		10	**13.3**	85		10	0.5	#			

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

Table 2 (continued): Results and z-Scores for Endosulfan sulfate, Metalaxyl and 4,4'-Methoxychlor

laboratory number	analyte											
	Endosulfan sulfate assigned value 81.6 µg/kg				Metalaxyl assigned value 76.8 µg/kg				4,4'-Methoxychlor assigned value 43.5 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	#				#				#			
017	83.17	102.36	10	0.1	77.62	87.97	10	0.0	45.31	79.94	10	0.2
018	100.9	102	10	1.1	78.7	100	10	0.1	53.9	92	10	1.1
019	#				#				#			
020	93	78	10	0.6	75	89	10	-0.1	57	76	10	1.4
021	79.5	99	10	-0.1	74	100	10	-0.2	#			
022	56.84		10	-1.4	52.04		10	-1.5	<LOQ		50	
023	#				69.5	94	10	-0.4	#			
024	84	93	10	0.1	87	99	10	0.6	35	92	10	-0.9
025	123	95	10	**2.3**	80	98	10	0.2	41	83	10	-0.3
026	44.22	101.28	10	**-2.1**	25.13	105.23	10	**-3.1**	#			
027	130	91	10	**2.7**	83	92	10	0.4	79	96	10	**3.7**
028	80	80	10	-0.1	89	102	10	0.7	45	80	10	0.2
029	101.0		10	1.1	82.0		10	0.3	0			**-4.5**
030	#				0.039	91.5	0.010	**-4.5**	#			

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

Table 2 (continued): Results and z-Scores for Endosulfan sulfate, Metalaxyl and 4,4'-Methoxychlor

laboratory number	analyte											
	Endosulfan sulfate assigned value 81.6 µg/kg				Metalaxyl assigned value 76.8 µg/kg				4,4'-Methoxychlor assigned value 43.5 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	37	97	10	**-2.5**	76	85	10	0.0	88	96	10	**4.6**
032	73.0	111	10	-0.5	77.6	99	10	0.0	39.8	107	10	-0.4

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

Table 3: Results and z-Scores for Parathion-methyl, Tebufenozide, and Glyphosate

laboratory number	analyte											
	Parathion-methyl assigned value 37.8 µg/kg				Tebufenozide assigned value 61.0 µg/kg				Glyphosate assigned value 500 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	48.9	95.3	10	1.3	#				#			
002	#				#				#			
003	40.2		10.0	0.3	56	79.5	10.0	-0.4	#			
004	51		10	1.6	44		10	-1.3	0			**-5.6**
005	45	S	10	0.9	58	S	10	-0.2	#			
006	#				#				#			
007	24		10	-1.7	77		10	1.2	#			
008	44.6		10	0.8	58.5		10	-0.2	466		100	-0.4
009	0			**-4.5**	#				#			
010	#				#				#			
011	38.30	89.60	10	0.1	#				#			
012	0			**-4.5**	0			**-4.5**	0			**-5.6**
013	38.1	92.9	10	0.0	#				#			
014	#				59	96	10	-0.1	#			
015	0			**-4.5**	64		10	0.2	#			

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

Table 3 (continued): Results and z-Scores for Parathion-methyl, Tebufenozide, and Glyphosate

laboratory number	analyte											
	Parathion-methyl assigned value 37.8 µg/kg				Tebufenozide assigned value 61.0 µg/kg				Glyphosate assigned value 500 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	#				#				#			
017	32.76	72.11	10	-0.6	63.74	118.0	10	0.2	503.23	93.18	10	0.0
018	37.6	77	10	0.0	61.9	100	10	0.1	496.4	100	100	0.0
019	#				#				#			
020	49	92	10	1.3	78	88	10	1.3	510	100	10	0.1
021	30.5	92	10	-0.9	45	100	10	-1.2	#			
022	44.33		10	0.8	47.29		10	-1.0	0		10	**-5.6**
023	#				60.0	86	10	-0.1	#			
024	28	96	10	-1.2	60	97	10	-0.1	452	92	10	-0.5
025	45	105	10	0.9	57	92	10	-0.3	#			
026	28.14	103.07	10	-1.2	#				#			
027	38	91	10	0.0	74	96	10	1.0	0			**-5.6**
028	22	80	10	-1.9	77	80	10	1.2	#			
029	46.2		10	1.0	51.3		10	-0.7	#			
030	#				0.027	93.0	0.010	**-4.5**	#			

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

Table 3 (continued): Results and z-Scores for Parathion-methyl, Tebufenozide, and Glyphosate

laboratory number	analyte											
	Parathion-methyl assigned value 37.8 µg/kg				Tebufenozide assigned value 61.0 µg/kg				Glyphosate assigned value 500 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	25	95	10	-1.5	78	104	10	1.3	0			**-5.6**
032	42.8	75	10	0.6	0			**-4.5**	#			

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

Table 4: Additional Pesticide Residues Reported

laboratory number	pesticide residue >25 µg/kg	result µg/kg	recovery %	LoQ µg/kg
032	tebuconazole	59.8	70	10

Table 5: Participants' Comments

participant number	comments
011	Difenoconazole has isomers Difenoconazole-1 and Difenoconazole-2.Difenoconazole was Calculated grand total of Difenoconazole-1 and Difenoconazole-2.
012	Glyphosate on list of potential pesticides, but not on result submission list!
029	Result reported for Metalaxyl is sum of Metalaxyl and Metalaxyl-M. Both substances in corresponding concentration of 41 µg/kg.

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	
Buprofezin	17	38.9	1.47	Horwitz [6]	8.56
Diazinon	19	120	3.68	Horwitz [6]	26.4
Difenoconazole	17	107	2.70	Horwitz [6]	23.5
Endosulfan sulfate	14	81.6	3.96	Horwitz [6]	17.9
Metalaxyl	18	76.8	1.07	Horwitz [6]	16.9
4,4'-Methoxychlor	14	43.5	2.43	Horwitz [6]	9.58
Parathion-methyl	14	37.8	2.87	Horwitz [6]	8.32
Tebufenozide	12	61.0	1.90	Horwitz [6]	13.4
Glyphosate	4	500	5.04	Horwitz [6]	88.8

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

analyte	number of scores where |z| ≤2	total number of scores	% |z| ≤2
Buprofezin	23	26	88
Diazinon	26	29	90
Difenoconazole	22	27	81
Endosulfan sulfate	15	23	65
Metalaxyl	24	27	89
4,4'-Methoxychlor	14	20	70
Parathion-methyl	21	24	88
Tebufenozide	19	22	86
Glyphosate	5	10	50

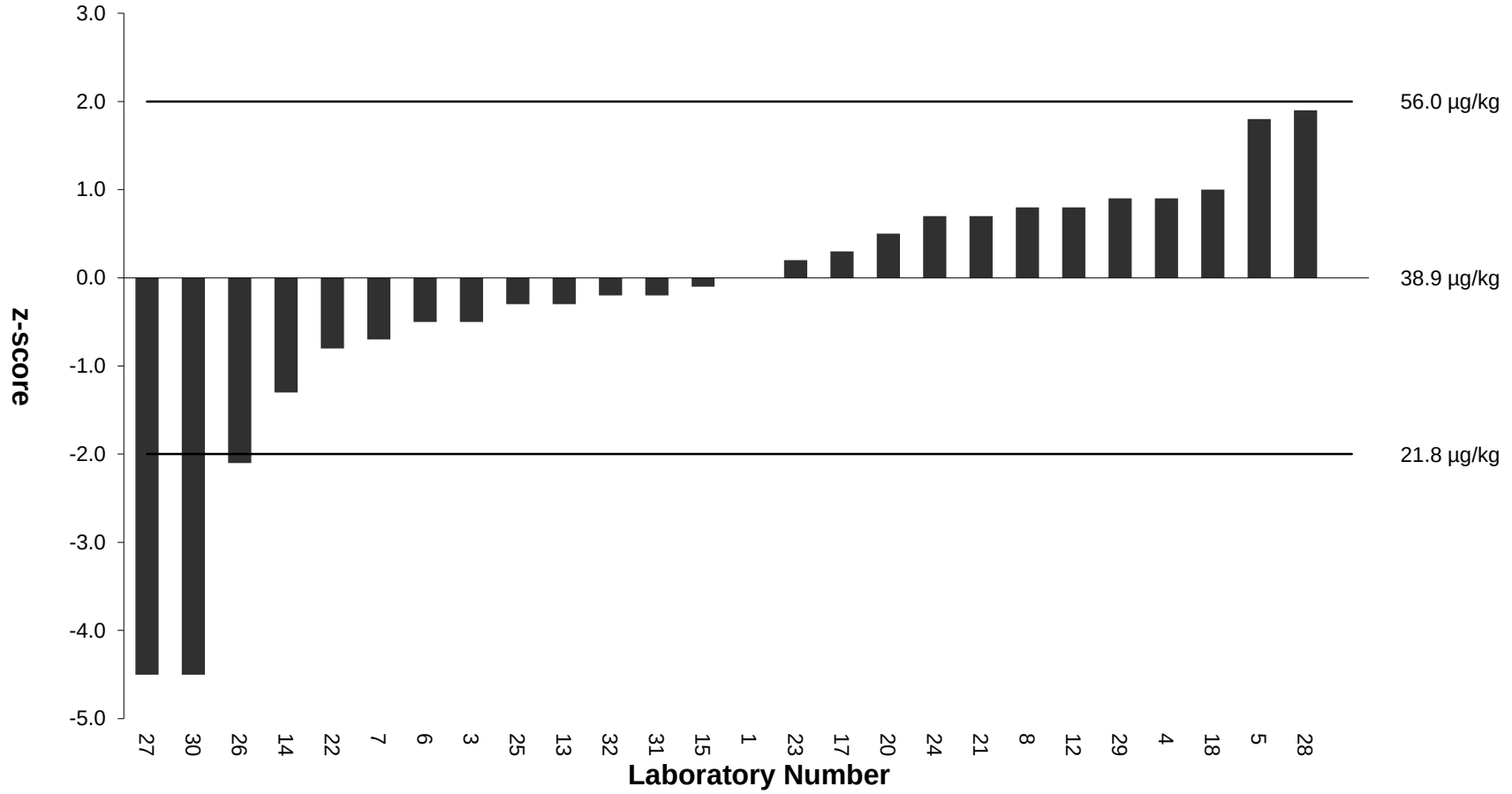

Figure 1: z-Scores for Buprofezin

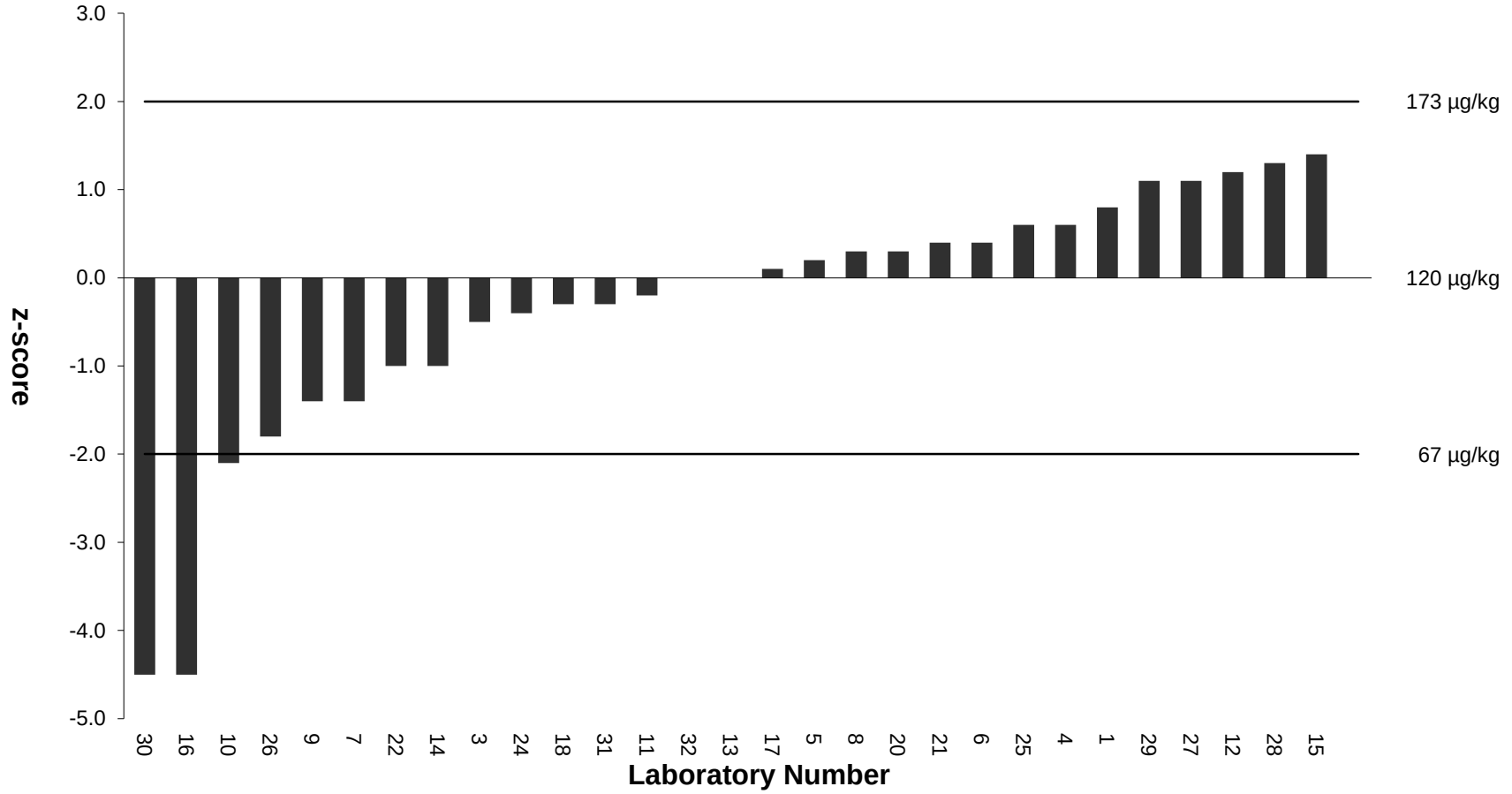

Figure 2: z-Scores for Diazinon

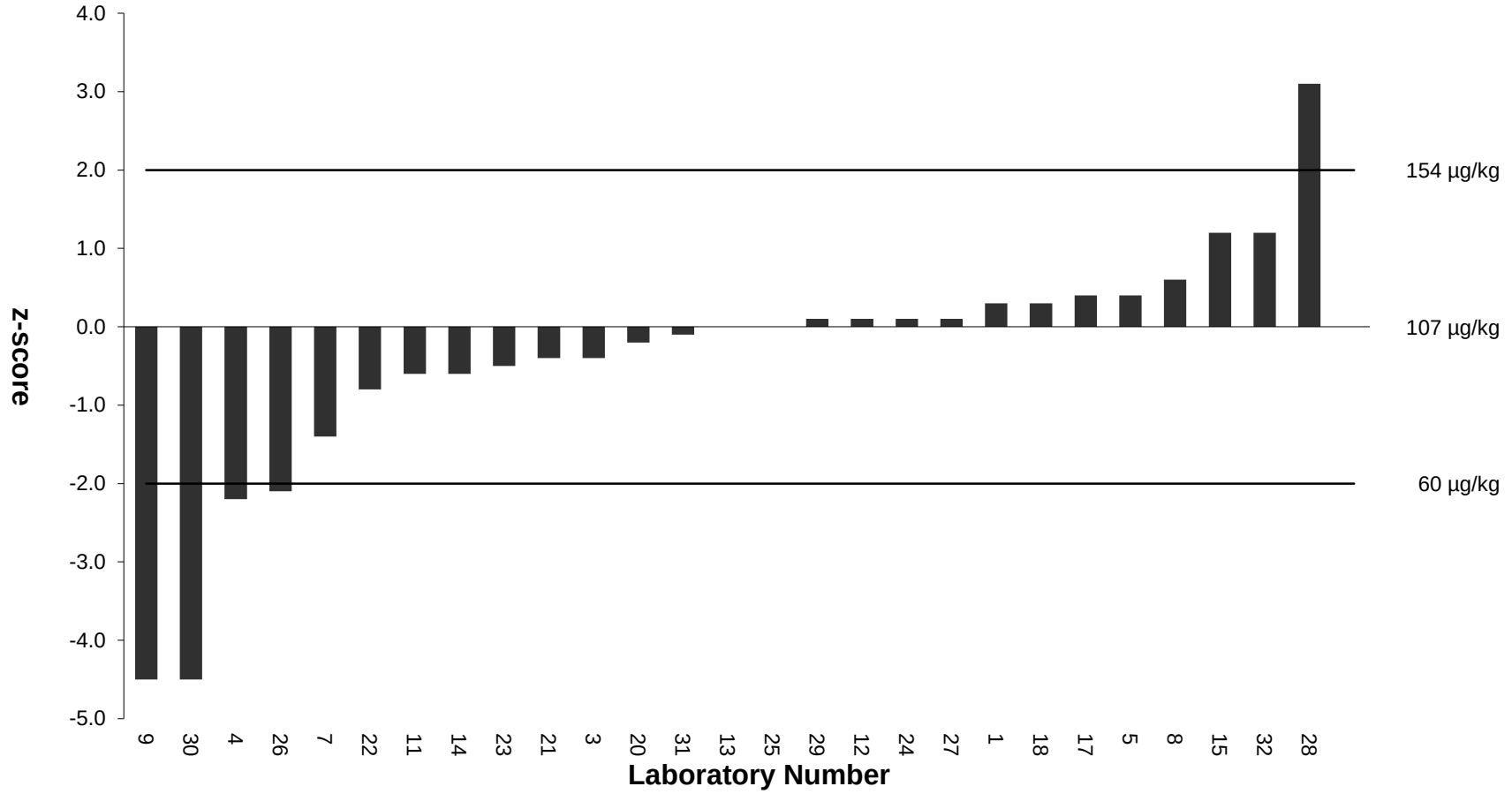

Figure 3: z-Scores for Difenoconazole

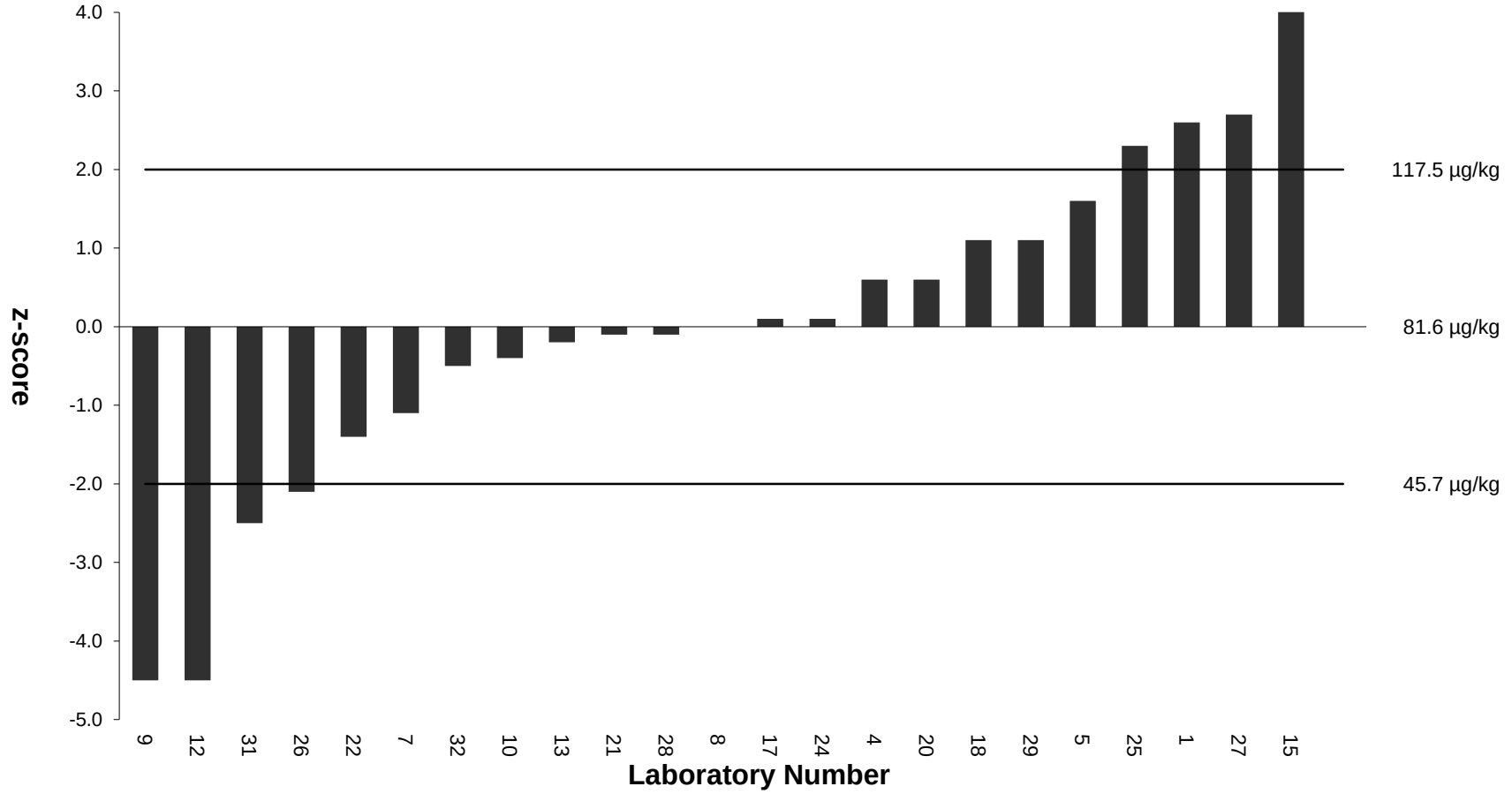

Figure 4: z-Scores for Endosulfan sulfate

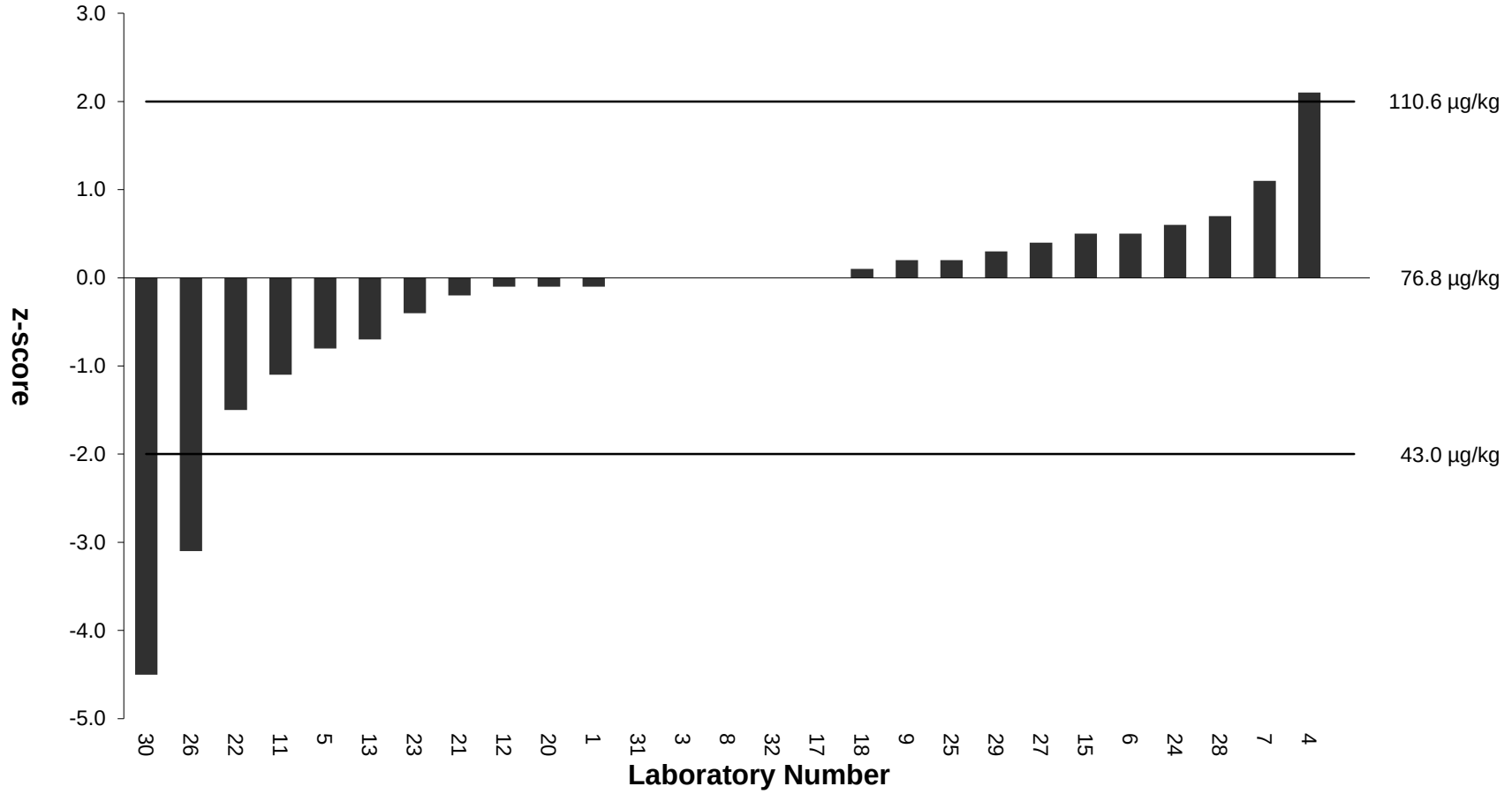

Figure 5: z-Scores for Metalaxyl

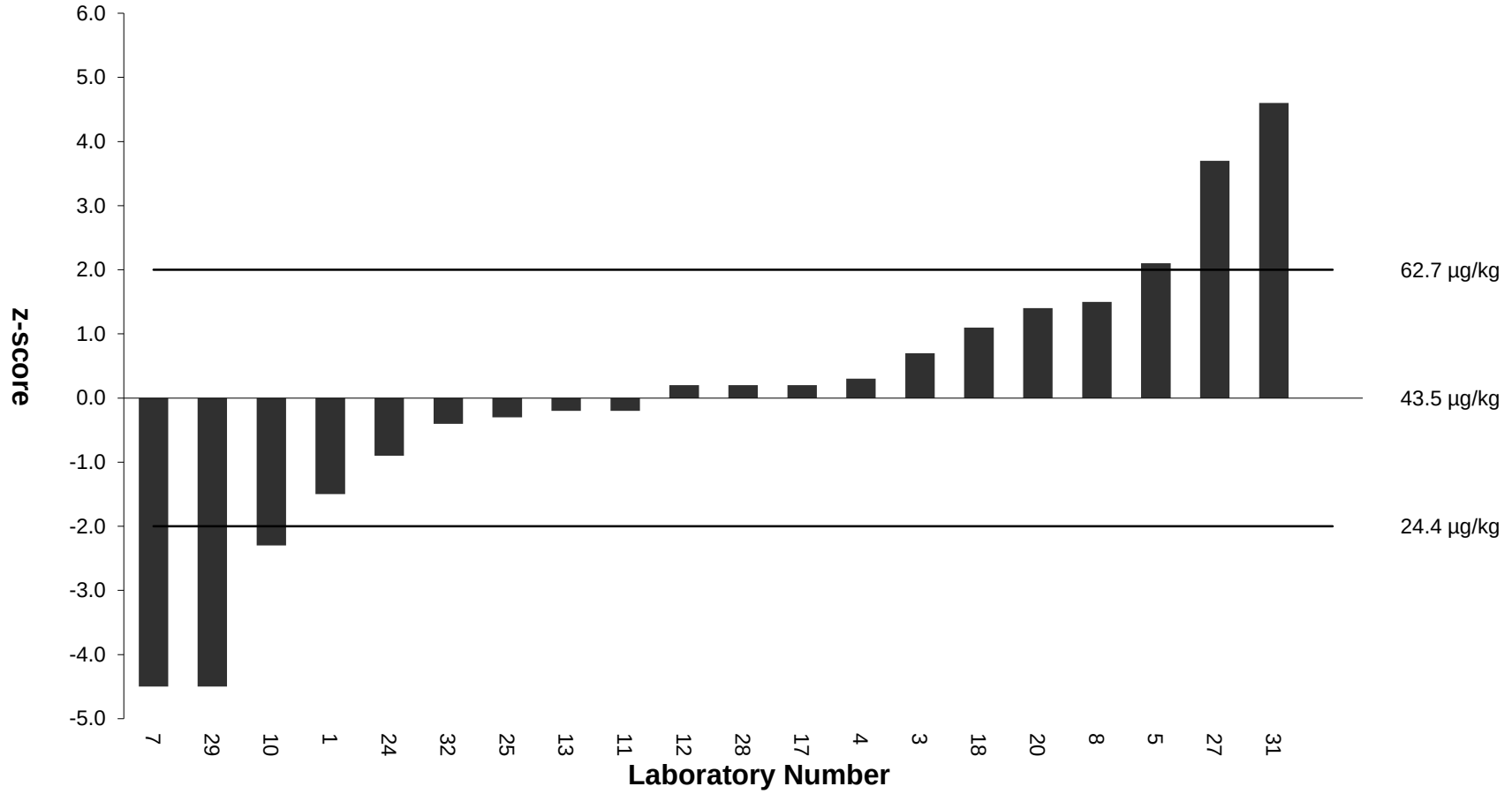

Figure 6: z-Scores for 4,4'-Methoxychlor

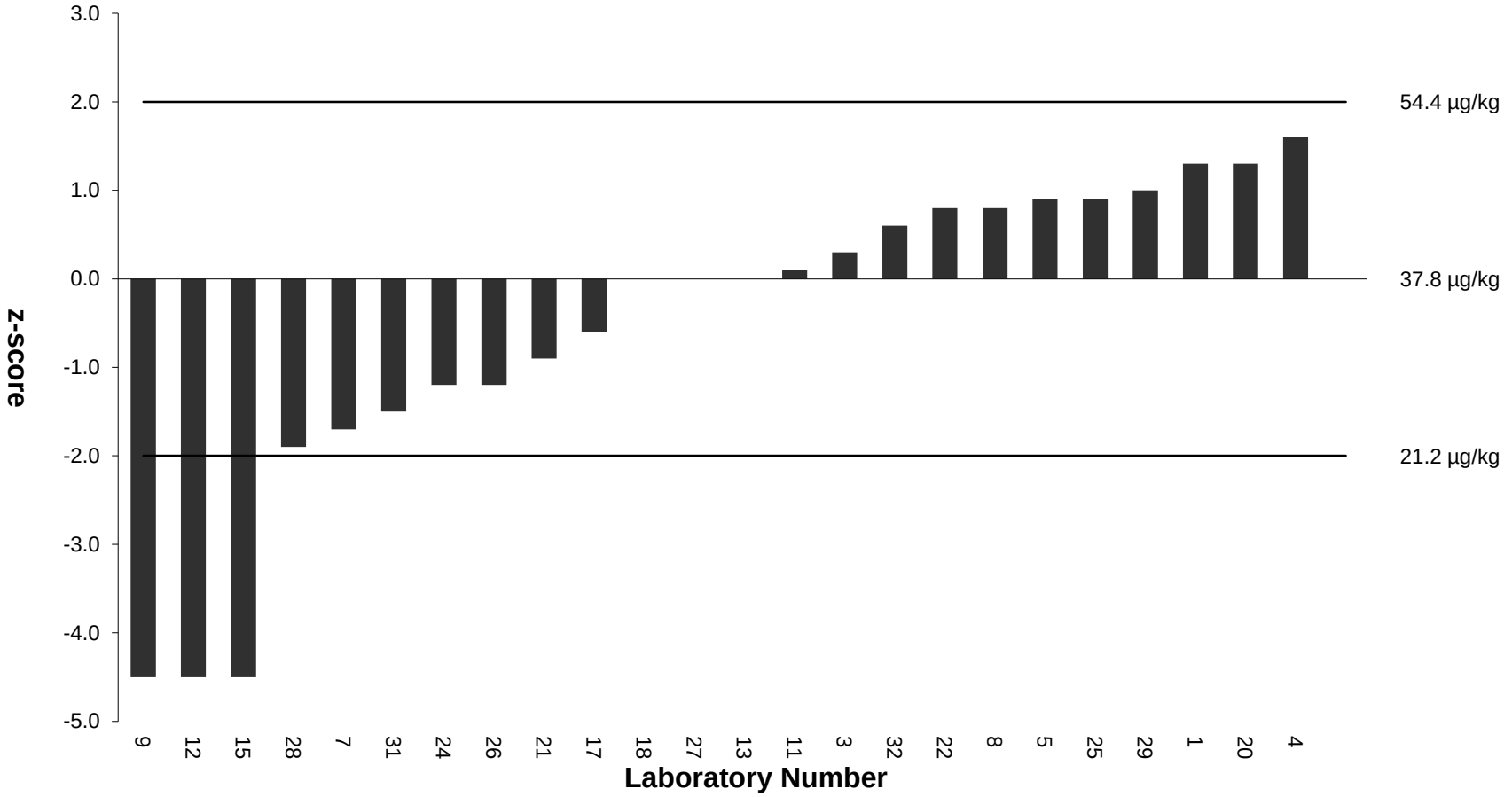

Figure 7: z-Scores for Parathion-methyl

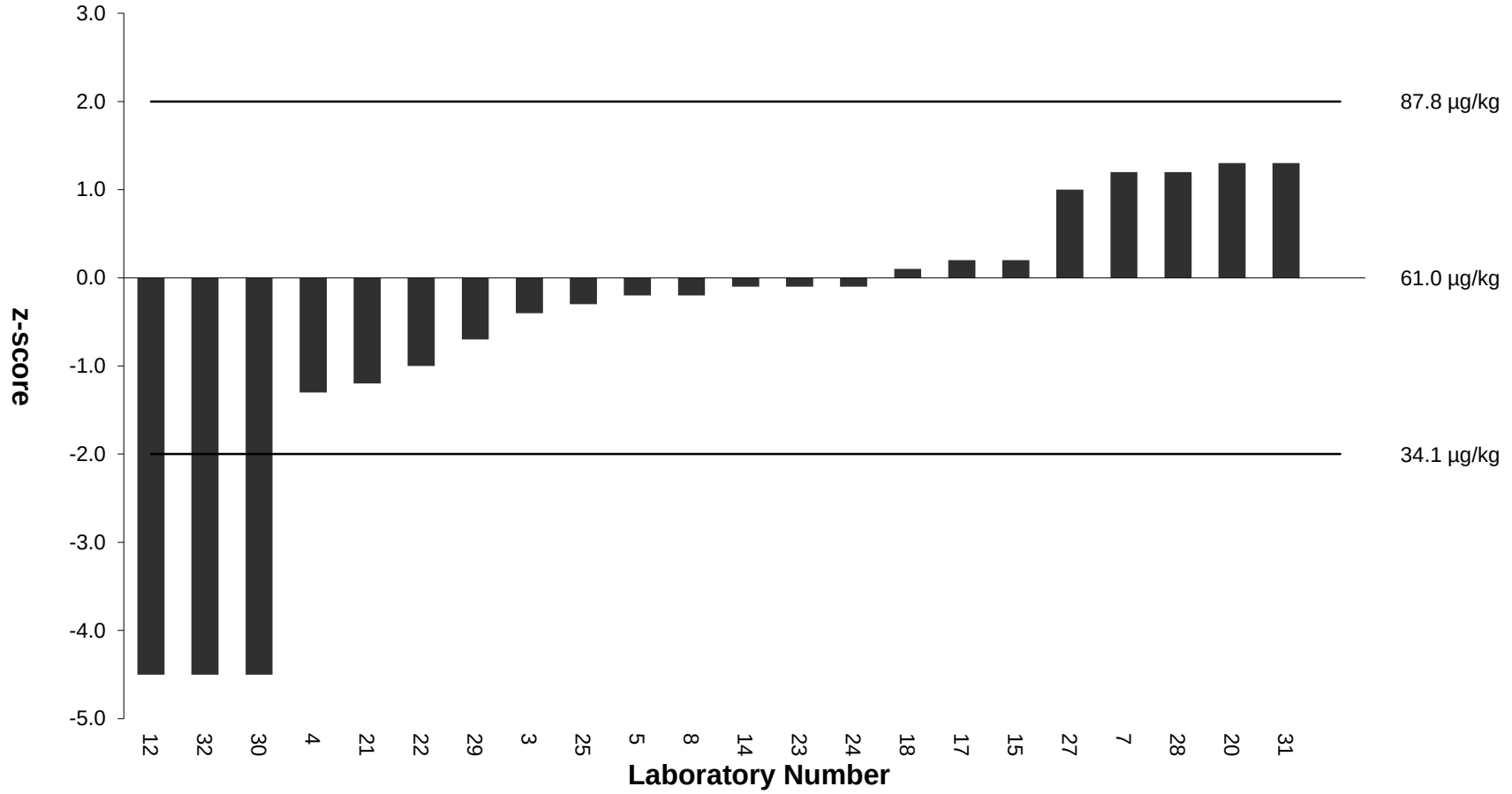

Figure 8: z-Scores for Tebufenozide

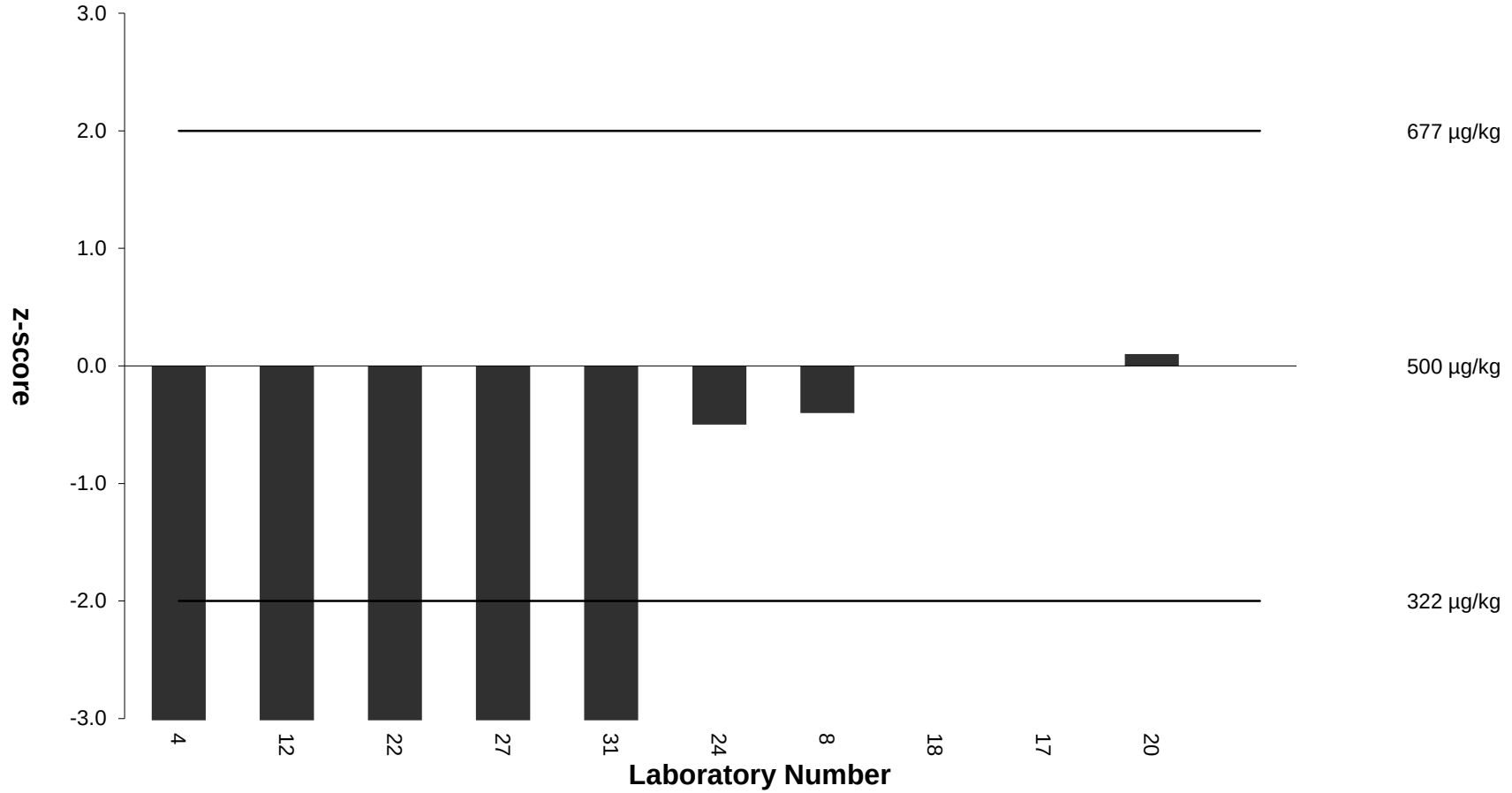

Figure 9: z-Scores for Glyphosate

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.

Is The Method Used Accredited	laboratory number
yes	001 002 003 004 005 006 007 008 009 011 012 013 014 016 018 020 021 022 024 025 026 029 030 031 032
no	010 015 017 019 023

What is your method based on	laboratory number
International Standard	001 002 004 005 007 009 010 012 018 020 021 024 026 031 032
National Standard	003 006 008 013 022
Paper Published In An International Journal	015 016
Manufacturer/Kit Instructions/Technical Note	011
In house method	014 017 019 023 025 029 030

Is quoted percentage recovery measured in same analytical batch as test material	laboratory number
yes	001 002 003 004 005 006 010 011 012 013 016 017 020 021 023 025 026 030 032
no	007 009 014 015 018 019 022 024 031

If measured in this batch, at what stage was the spike added	laboratory number
prior to extraction	001 002 003 004 005 006 010 011 012 013 016 017 018 020 021 022 023 025 026 030 032

Concentration of Spike (µg/kg)	laboratory number
≥1 - <5	002
≥5 - <10	022 032
≥10 - <25	012 019 030
≥25 - <50	005 023
≥50 - <100	001 003 004 006 010 011 013 017 018 020 021 031
≥100	016 024 025 026

Composition of Blank Commodity used for Spiking	laboratory number
blank provided	003 005 006 011 012 013 014 017 018 020 022 024 026 030
test material provided	001 004 019 023 025 029
Lab. blank material	010

Calibration	laboratory number
standard addition	005 017 018 020 029
matrix-matched	001 003 007 008 011 012 014 018 020 021 022 023 024 025 026 030 032
solvent	010 020
multi-level	001 003 004 006 009 010 015 016 019 020 029 030 031
single-level	007 013 020

Was an Internal Standard Added	laboratory number
yes	001 004 005 012 015 017 018 019 020 021 022 023 024 025 026 029 031 032
no	002 003 006 007 008 009 010 011 013 014 016 030

What Internal Standard was Used	laboratory number
2,4,5,6-tetrachloro-m-xylene	019
2-amino-benzymidazole and deuterated benzene hexachloro	032
coffein for GC analysis.	012
DiazinonD	017

What Internal Standard was Used (continued)	laboratory number
Malathion D6	004
only for ectraction efficiency	020
PCB 31/ Sulfotep	024
sulfotep / HBB / Tris / Carbedazim D3 / Propamocarb D7	031
tetraphenylethylene	021
TPP and deuterated pesticides	025
TPP, 1,2-C13N15	018
Triphenyl phosphate	001 005 015 022 023 026 029

GC Method: Sample Weight (g)	laboratory number
≥1 - <5	002 004 005
≥5 - <10	003 008 010 011 015 016 017 018 029
≥10 - <20	001 006 009 012 013 014 019 020 022 024 025 026 031 032
≥20 - <50	007 021

GC Method: Extraction Solvent Components	laboratory number
acetone	007 017
acetonitrile	001 002 003 004 006 008 009 010 011 012 013 015 016 020 022 024 025 026 029 031 032
dichloromethane	014
ethyl acetate	005 018 019 021

GC Method: Extraction pH Adjusted	laboratory number
yes	004 005 008 010 012 014 018 021
no	001 002 003 006 007 009 011 013 015 016 017 019 020 022 024 025 026 029 031 032

GC Method: Extraction Techniques Used	laboratory number
macerate at room temperature	019 020 021
macerate at elevated temperature	018
QuEChERS	001 002 003 004 006 008 009 010 011 012 014 015 017 020 022 024 025 026 029 031 032
liquid-liquid partition	005 007 013 016

GC Method: Sample Clean-up Technique	laboratory number
GPC/HPGPC	007 019 021
liquid-liquid partition	009
none	006 024
solid phase extraction (SPE) (column/cartridge)	002 005 011 013 016 017 018 029
solid phase extraction (SPE) (dispersive)	001 003 004 008 010 012 014 015 020 022 025 026 032

GC Method: SPE Sorbent Type	laboratory number
C18	008 011 029
Envicarb/GCB	018
PSA	001 002 010 012 014 015 020 022 024 025 026 031 032
Mixed Mode	005 017
ENVCarb+NH2	013
florisil	016
PSA/GCB	003

GC Method: GC Column Packing	laboratory number
50% methyl 50% phenyl polysiloxane	021
65% methyl 35% phenyl polysiloxane	009 018
95% methyl 5% phenyl polysiloxane	001 002 003 004 006 007 008 010 011 012 013 014 016 020 022 024 025 026 029 032
(14% Cyanopropyl-phenyl)-methylpolysiloxane	019

GC Method: GC Detector Type	laboratory number
ECD	002 007 010 016 019 020
MS	005 009 011 013 014 015 017 018 020 021 024 026 029 031
MS-MS	001 003 004 007 008 012 022 025 032
FPD	007 020
NPD	006
PFPD	010

LC Method: Sample Weight (g)	laboratory number
≥1 - <5	003 005 007 023 030
≥5 - <10	008 015 017 018 029
≥10 - <20	001 006 012 014 020 021 022 024 025 026 031 032

LC Method: Extraction Solvent Components	laboratory number
acetonitrile	001 003 006 007 008 014 015 017 020 021 022 023 024 025 026 029 030 031 032
ethyl acetate	005 018 030
methanol	007 012

LC Method: Extraction pH Adjusted	laboratory number
yes	005 008 014 018 025 030
no	001 003 006 007 012 015 017 020 021 022 023 024 026 029 031 032

LC Method: Extraction Techniques Used	laboratory number
macerate at room temperature	020
macerate at elevated temperature	005
QuEChERS	001 003 006 007 008 014 015 017 018 020 021 022 023 024 025 026 029 030 031 032
liquid-liquid partition	012

LC Method: Sample Clean-up Technique	laboratory number
none	003 005 006 012 018 021 024
solid phase extraction (SPE) (column/cartridge)	017 025 029
solid phase extraction (SPE) (dispersive)	001 008 014 015 020 022 023 026 030 032

LC Method: SPE Sorbent Type	laboratory number
C18	008 029
PSA	001 014 015 020 022 023 024 026 031 032
Mixed Mode	017 025 030

LC Method: HPLC Column Packing	laboratory number
C18	001 003 005 006 007 008 012 014 015 017 018 020 021 022 023 024 025 026 029 030 031 032

LC method: Mobile Phase Components	laboratory number
acetic acid	020
acetonitrile	005 015 025 029
ammonium acetate	017 018 030
ammonium formate	001 005 008 015 020 026
formic acid	023 030
methanol	001 003 006 007 014 018 020 021 022 024 030 031
water	001 003 005 06 007 012 015 018 020 029 032

LC Method: Detector Type	laboratory number
MS-MS	001 003 005 006 007 008 012 014 015 017 018 020 021 022 023 024 025 026 029 030 031 032

Buprofezin

Method Principle	laboratory number
GC	005 007 008 013 017 018 021 024 025 031 032
LC	001 003 006 012 014 015 020 022 023 026 029 030

Identification by Mass Spectrometry	laboratory number
yes	001 003 005 006 008 012 013 014 015 017 018 020 021 022 023 024 025 026 029 030 031 032

Diazinon

Method Principle	laboratory number
GC	003 005 007 008 009 010 011 012 013 016 017 018 021 022 024 025 026 031 032
LC	001 006 014 015 020 029 030

Identification by Mass Spectrometry	laboratory number
yes	001 003 005 006 008 009 011 012 013 014 015 017 018 020 021 022 024 025 026 029 030 031 032
no	007 010 016

Difenoconazole

Method Principle	laboratory number
GC	003 007 011 012 013 017 024 025 031
LC	001 005 008 014 015 018 020 021 022 023 026 029 030 032

Identification by Mass Spectrometry	laboratory number
yes	001 003 005 007 008 011 012 013 014 015 017 018 020 021 022 023 024 025 026 029 030 031 032

Endosulfan sulfate

Method Principle	laboratory number
GC	001 004 005 007 008 010 013 015 017 018 020 021 022 024 025 026 029 031 032

Identification by Mass Spectrometry	laboratory number
yes	001 004 005 008 013 015 017 018 020 021 022 024 025 026 029 031 032
no	007 010

Metalaxyl

Method Principle	laboratory number
GC	003 005 009 011 013 017 018 022 024 025 031 032
LC	001 006 007 008 012 015 021 023 026 029 030

Identification by Mass Spectrometry	laboratory number
yes	001 003 005 006 007 008 009 011 012 013 015 017 018 021 022 023 024 025 026 029 030 031 032

4,4'-Methoxychlor

Method Principle	laboratory number
GC	001 003 005 008 010 011 012 013 017 018 024 025 031 032
LC	020

Identification by Mass Spectrometry	laboratory number
yes	001 003 005 008 011 012 013 017 018 020 024 025 031 032
no	010

Parathion-methyl

Method Principle	laboratory number
GC	001 003 005 007 008 011 013 017 018 020 021 022 024 025 029 031 032
LC	026

Identification by Mass Spectrometry	laboratory number
yes	001 003 005 008 011 013 017 018 020 021 022 024 025 026 029 031 032
no	007

Tebufenozide

Method Principle	laboratory number
LC	003 004 005 007 008 014 015 017 018 020 021 022 023 024 025 029 030 031

Identification by Mass Spectrometry	laboratory number
yes	003 004 005 007 008 014 015 017 018 020 021 022 024 025 029 030 031
no	023

Glyphosate

Method Principle	laboratory number
LC	008 017 018 020 024

Identification by Mass Spectrometry	laboratory number
yes	008 017 018 020 024

APPENDIX II: FAPAS SecureWeb, Protocol and Contact Details

1. FAPAS SECUREWEB

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

- Obtain their laboratory numbers for the proficiency tests in which they have participated.
- View the results they submitted in past and current proficiency tests.
- Submit their results and methods for current tests.
- Review future tests they have ordered.
- Order proficiency tests and quality control materials.
- Freely download copies of reports (PDF file), of proficiency tests in which they have participated.
- 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 Ed Rawlings (Round Coordinator). Participants with any comments or concerns about this proficiency test should contact:

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