



**Fapas<sup>®</sup> – Food Chemistry Proficiency Test Report 09110**

**Pesticide Residues in Rice**

**June-August 2017**

## PARTICIPANT LABORATORY NUMBER

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

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

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## SUMMARY

1. The test material for Fapas<sup>®</sup> – Food Chemistry proficiency test 09110 was dispatched in June 2017. Each participant received a rice test material to be analysed for pesticide residues. Participants had to identify and quantify, from a list of 279 pesticide residues, those present in the test material.
2. An assigned value ( $x_a$ ) was determined for acetamiprid, cis-chlordane, deltamethrin, linuron, methacrifos, phosphamidon, pirimicarb, propargite and vinclozolin, and in conjunction with the standard deviations for proficiency ( $\sigma_p$ ) was used to calculate a z-score for each result. However, those for methacrifos and phosphamidon are given *for information only*.
3. Results for this proficiency test are summarised as follows:

analyte	assigned value, $x_a$ $\mu\text{g/kg}$	number of scores, $ z  \leq 2$	total number of scores	% $ z  \leq 2$
Acetamiprid	73.3	65	73	89
cis-Chlordane	31.5	56	74	76
Deltamethrin	58.1	72	82	88
Linuron	40.0	62	65	95
Methacrifos	<i>10.0</i>	<i>17</i>	<i>33</i>	<i>52</i>
Phosphamidon	<i>14.5</i>	<i>36</i>	<i>56</i>	<i>64</i>
Pirimicarb	80.2	70	73	96
Propargite	54.5	64	70	91
Vinclozolin	50.2	70	76	92

*italics indicate for information only*

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## 1. INTRODUCTION

### 1.1. Proficiency Testing

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

Further details of the Fapas<sup>®</sup> – 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 commercially available organic white rice flour. This was mixed and split into two batches: one to use for the spiking material and one to use for the blank material.

Acetamiprid, cis-chlordane, deltamethrin, linuron, methacrifos, phosphamidon, pirimicarb, propargite and vinclozolin were spiked into the test material.

Samples were stored at ambient temperature until dispatch.

### 2.2. Screening and Homogeneity

A sample of rice flour was screened for the presence of incurred pesticide residues. No residues that were on the Fapas<sup>®</sup> target list were found at or above 16 µg/kg.

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. Despite sufficient homogeneity, the levels for methacrifos and phosphamidon had decreased significantly from the intended spike value.

### 2.3. Dispatch

The start date was 21 June 2017. Test materials were sent to 102 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 percentage (%) recovery and limit of quantification (LoQ). PLEASE NOTE:
  - Report results for all pesticide residues as the parent compound only, unless specified otherwise.

- For each residue analysed, please enter the LoQ, even if the residue was not detected.

Results were submitted by 95 participants (93%) before the closing date for this test, 09 August 2017.

Each participant was given a laboratory number, assigned in order of receipt of results. The reported analyte concentrations are given in Table 1 to Table 3.

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 acetamiprid, cis-chlordane, deltamethrin, linuron, methacrifos, phosphamidon, pirimicarb, propargite and vinclozolin at a level  $\geq 16 \mu\text{g}/\text{kg}$  are listed in Table 4 together with the percentage recovery and LoQ.

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 deviations for proficiency,  $\sigma_p$ , 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 whose recovery was outside the range 60-140% [8],
- iii) results where no limit of quantification was reported.

For acetamiprid, cis-chlordane, deltamethrin, linuron, phosphamidon, pirimicarb, propargite and vinclozolin, this procedure was straightforward and the robust mean was chosen as the assigned value.

For methacrifos, the median was chosen as the assigned value because of the low number of reported results.

For methacrifos and phosphamidon, the assigned values and z-scores are issued *for information only* due to the levels being very close to most participants' LoQs.

The assigned values for acetamiprid, cis-chlordane, deltamethrin, linuron, methacrifos, phosphamidon, pirimicarb, propargite and vinclozolin are shown in Table 6.

## 4.2. Standard Deviation for Proficiency, $\sigma_p$

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

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

The values for  $\sigma_p$  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  $\sigma_p$  = the standard deviation for proficiency.

Participants' z-scores for acetamiprid, cis-chlordane and deltamethrin are given in Table 1 and shown as histograms in Figures 1–3. Participants' z-scores for linuron, methacrifos and phosphamidon are given in Table 2 and shown as histograms in Figures 4–6. Participants' z-scores for pirimicarb, propargite and vinclozolin 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 [7].

## 6. REFERENCES

- 1 Adobe Approved Trust List, <https://helpx.adobe.com/acrobat/kb/approved-trust-list2.html#Whatisit> accessed 01/06/2017.
- 2 GlobalSign PDF Signing Tool, <https://www.globalsign.com/en/pdf-signing/> accessed 01/06/2017
- 3 ISO/IEC 17043:2010, Conformity assessment – General requirements for proficiency testing.
- 4 Fapas<sup>®</sup>, 2017, Protocol for Proficiency Testing Schemes, Version 6, April 2017, Part 1 – Common Principles.
- 5 Fapas<sup>®</sup>, 2017, Protocol for Proficiency Testing Schemes, Version 5, April 2017, Part 2 – Fapas<sup>®</sup> 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 Method Validation and Quality Control Procedures for Pesticide Residue Analysis in Food and Feed, Document No. SANTE/11945/2015.
- 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 Acetamiprid, cis-Chlordane and Deltamethrin**

laboratory number	analyte											
	Acetamiprid assigned value: 73.3 µg/kg				cis-Chlordane assigned value: 31.5 µg/kg				Deltamethrin assigned value: 58.1 µg/kg			
	result	rec. %	LoQ	z-score	result	rec. %	LoQ	z-score	result	rec. %	LoQ	z-score
001	0		10	<b>-4.5</b>	29	100	10	-0.4	50	100	10	-0.6
002	68.3			-0.3	34.1			0.4	80.6			1.8
003	66.4	105	10	-0.4	26.9	123	10	-0.7	74.1	94	10	1.3
004	93	100	5	1.2	0	100	5	<b>-4.5</b>	#			
005	#				#				53	96.6	10	-0.4
006	#				#				59	92	5	0.1
007	72	103	10	-0.1	38	98	10	0.9	78	98	10	1.6
008	79.8	102	10	0.4	#				67.7	118	10	0.8
009	79.7	119	10	0.4	37.4	109	10	0.8	73.8	105	10	1.2
010	71	114	10	-0.1	26.6	94	5	-0.7	45.8	107.7	10	-1.0
011	68	90.3	10	-0.3	125	101	10	<b>13.5</b>	70	106	10	0.9
012	90.3	82.8	10	1.1	0			<b>-4.5</b>	60.7	107.7	10	0.2
013	0			<b>-4.5</b>	30.569	91.12		-0.1	64.637	80.02		0.5
014	67	91	10	-0.4	164	100	10	<b>19.1</b>	67	100	10	0.7
015	70.91	102.0	10	-0.1	33.13	99.8	10	0.2	60.14	98.2	10	0.2
016	#				#				#			
017	67.3	110	40	-0.4	33.9	105	10	0.3	67.7	101	10	0.8
018	68	>90	10	-0.3	25	>90	10	-0.9	60	>90	10	0.1
019	79.0	86.0	10.0	0.4	#				92.0	105.0	10.0	<b>2.7</b>
020	#				#				#			
021	63	77	10	-0.6	155	90	10	<b>17.8</b>	55	83	10	-0.2
022	#				#				#			
023	#				25.0	83.40	10.0	-0.9	73.6	108.82	10.0	1.2
024	#				38.80	92	5	1.1	68.34	90	14	0.8
025	78.4	89.5	10.0	0.3	97.5	95.0	10.0	<b>9.5</b>	82.4	91.4	10.0	1.9

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

# not analysed

LoQ = limit of quantification

**Table 1 (continued): Results and z-Scores for Acetamiprid, cis- Chlordane and Deltamethrin**

laboratory number	analyte											
	Acetamiprid assigned value: 73.3 µg/kg				cis-Chlordane assigned value: 31.5 µg/kg				Deltamethrin assigned value: 58.1 µg/kg			
	result	rec. %	LoQ	z-score	result	rec. %	LoQ	z-score	result	rec. %	LoQ	z-score
026	82	90	10	0.5	36	90	10	0.6	61	90	10	0.2
027	#				25.1	126	10	-0.9	28.9	123	10	<b>-2.3</b>
028	#				#				#			
029	123	67	10	<b>3.1</b>	#				#			
030	70.23	86.3	10	-0.2	26.40	70.8	10	-0.7	51.41	65.7	10	-0.5
031	81.0	110	10	0.5	36.0	105.6	10	0.6	61.2	102	10	0.2
032	#				<LOQ	120	20		<LOQ	72	100	
033	#				#				53.8	102.7	10	-0.3
034	55	85	10	-1.1	34	119	10	0.4	80	85	10	1.7
035	82	70	10	0.5	24	80	10	-1.1	57	120	10	-0.1
036	64.89	98.61 %	20	-0.5	22.84	108.94 %	20	-1.3	46.90	93.8%	20	-0.9
037	57	82	10	-1.0	31	95	10	-0.1	55	85	10	-0.2
038	#				#				#			
039	78.5	105	5	0.3	36.4	91	5	0.7	40.9	93	5	-1.3
040	73.7	104.5	20	0.0	24.0	100.0	20	-1.1	57.3	100.1	20	-0.1
041	67.2	121	10.0	-0.4	69.1	101	10.0	<b>5.4</b>	47.0	128	10.0	-0.9
042	#				0			<b>-4.5</b>	100	106	10.0	<b>3.3</b>
043	#				22.20	90.3	10	-1.3	54.35	107.5	10	-0.3
044	55.0	89.1	10.0	-1.1	16.0	85.0	10.0	<b>-2.2</b>	48.0	83.2	10.0	-0.8
045	66.3	100	10	-0.4	126.5	100	10	<b>13.7</b>	73.1	100	10	1.2
046	30.75	102.55	10	<b>-2.6</b>	0			<b>-4.5</b>	0			<b>-4.5</b>
047	#				22.0	104%	10	-1.4	51.7	88%	10	-0.5
048	53.15	80	10	-1.2	26.71	109	10	-0.7	#			
049	#				28	94.58	10	-0.5	#			
050	63.71	70	10	-0.6	33.18	108	10	0.2	41.20	82	10	-1.3

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

# not analysed

LoQ = limit of quantification

**Table 1 (continued): Results and z-Scores for Acetamiprid, cis- Chlordane and Deltamethrin**

laboratory number	analyte											
	Acetamiprid assigned value: 73.3 µg/kg				cis-Chlordane assigned value: 31.5 µg/kg				Deltamethrin assigned value: 58.1 µg/kg			
	result	rec. %	LoQ	z-score	result	rec. %	LoQ	z-score	result	rec. %	LoQ	z-score
051	#				27.0	104	10	-0.7	57.0	85	3	-0.1
052	92.5		10	1.2	#				95.5		10	<b>2.9</b>
053	86	97	10	0.8	#				67	103	10	0.7
054	75.8	86.3	20	0.2	25.9	69.9	20	-0.8	52.2	90.2	20	-0.5
055	0			<b>-4.5</b>	22	85	10	-1.4	43	83	10	-1.2
056	#				31	99.2	10	-0.1	60	87	10	0.1
057	56.04	89.3	10	-1.1	20.33	68.5	10	-1.6	52.67	99.0	10	-0.4
058	88	95	10	0.9	#				45	92	10	-1.0
059	#				#				#			
060	76	100	10	0.2	41	100	10	1.4	62	100	10	0.3
061	84	93	10	0.7	84	90	10	<b>7.6</b>	42	114	10	-1.3
062	75	107	10	0.1	43	89	10	1.7	65	96	10	0.5
063	81.3	101.0	20	0.5	26.8	92.7	20	-0.7	52.8	90.2	20	-0.4
064	77	97	10	0.2	106	78	10	<b>10.7</b>	56	75	10	-0.2
065	67.32	-	10.0	-0.4	28.60	100.7	10.0	-0.4	36.26	85.4	10.0	-1.7
066	#				#				#			
067	86.32	99	10	0.8	23.02	112	10	-1.2	48.41	99.2	10	-0.8
068	75		10	0.1	27		10	-0.7	51		10	-0.6
069	80.6		10	0.5	0		10	<b>-4.5</b>	41.3		10	-1.3
070	54.8	78.6	10	-1.1	0			<b>-4.5</b>	#			
071	74	100	10	0.0	24	100	10	-1.1	85	100	10	<b>2.1</b>
072	76	90	10	0.2	34	100	10	0.4	47	80	10	-0.9
073	73.2	102	10	0.0	27.6	75	10	-0.6	48.0	78	10	-0.8
074	60	80%	5	-0.8	24	97%	5	-1.1	44	80%	5	-1.1
075	75	100	10	0.1	#				48	100	10	-0.8

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

# not analysed

LoQ = limit of quantification

**Table 1 (continued): Results and z-Scores for Acetamiprid, cis- Chlordane and Deltamethrin**

laboratory number	analyte											
	Acetamiprid assigned value: 73.3 µg/kg				cis-Chlordane assigned value: 31.5 µg/kg				Deltamethrin assigned value: 58.1 µg/kg			
	result	rec. %	LoQ	z-score	result	rec. %	LoQ	z-score	result	rec. %	LoQ	z-score
076	67	100	10	-0.4	29	85	10	-0.4	61	70	10	0.2
077	85.97	90	10	0.8	27.84	85	10	-0.5	69.39	89	10	0.9
078	85.05	100	10	0.7	24.96	76	10	-0.9	51.26	83	10	-0.5
079	8.8	68.7	10	<b>-4.0</b>	23.3	100.5	10	-1.2	29.2	49.0	10	<b>-2.3</b>
080	77	100	10	0.2	30	100	10	-0.2	63	100	10	0.4
081	59	97	10	-0.9	35	101	10	0.5	64	99	10	0.5
082	0			<b>-4.5</b>	0			<b>-4.5</b>	9	89	10	<b>-3.8</b>
083	84	98	10	0.7	38	105	10	0.9	64	102	10	0.5
084	73	92	5	0.0	27	87	5	-0.7	48	80	5	-0.8
085	78	96	10	0.3	34	107	10	0.4	60	126	10	0.1
086	117		10	<b>2.7</b>	11		10	<b>-3.0</b>	100		10	<b>3.3</b>
087	73	100	10	0.0	54	107	10	<b>3.2</b>	66	117	10	0.6
088	#				#				59	95	10	0.1
089	71	100	10	-0.1	31	100	10	-0.1	64	100	10	0.5
090	73.7	78	10	0.0	26.5	92	10	-0.7	44.3	78	10	-1.1
091	74	72	10	0.0	#				45	91	60	-1.0
092	74.5	99.3	10	0.1	26	105.1	10	-0.8	66.2	93.3	10	0.6
093	73.2	99-102	10	0.0	36.5	77-85	10	0.7	55.3	70-120	20	-0.2
094	75		10	0.1	30		10	-0.2	51		10	-0.6
095	#				#				0			<b>-4.5</b>

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

# not analysed

LoQ = limit of quantification

**Table 2: Results and z-Scores for Linuron, Methacrifos and Phosphamidon**

laboratory number	analyte											
	Linuron assigned value: 40.0 µg/kg				Methacrifos assigned value: 10.0 µg/kg				Phosphamidon assigned value: 14.5 µg/kg			
	result	rec. %	LoQ	z-score	result	rec. %	LoQ	z-score	result	rec. %	LoQ	z-score
001	15	100	10	<b>-2.8</b>	<LOQ		10		<LOQ		10	
002	41.4			0.2	#				14.3			-0.1
003	42.3	101	10	0.3	0			-4.5	13.8	97	10	-0.2
004	50	100	5	1.1	16	100	5	2.7	#			
005	#				7	82.1	5	-1.4	#			
006	#				#				#			
007	39	95	10	-0.1	11	107	10	0.5	11	95	10	-1.1
008	36.4	88	10	-0.4	#				13.1	79	10	-0.4
009	53.7	126	10	1.6	<LOQ		10		18.1	120	10	1.1
010	#				7.9	99.3	5	-1.0	#			
011	#				#				#			
012	43.8	113.5	10	0.4	10.0	106.1	10	0.0	16.1	100.7	10	0.5
013	#				#				27.722	103.53		4.1
014	#				10	100	10	0.0	0			-4.5
015	44.65	108.0	10	0.5	<LOQ		10		17.44	97.5	10	0.9
016	#				#				#			
017	#				<LOQ		10		<LOQ		10	
018	36	>90	10	-0.5	<LOQ		10		12	>90	10	-0.8
019	#				#				10.0	95.0	10.0	-1.4
020	#				#				#			
021	35	85	10	-0.6	#				14	118	10	-0.2
022	36.4	89	10	-0.4	#				0			-4.5
023	#				#				<LOQ		10.0	
024	#				#				#			
025	#				#				18.2	91.5	10.0	1.1

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

*italics indicate for information only*

# not analysed

LoQ = limit of quantification

**Table 2 (continued): Results and z-Scores for Linuron, Methacrifos and Phosphamidon**

laboratory number	analyte											
	Linuron assigned value: 40.0 µg/kg				Methacrifos assigned value: 10.0 µg/kg				Phosphamidon assigned value: 14.5 µg/kg			
	result	rec. %	LoQ	z-score	result	rec. %	LoQ	z-score	result	rec. %	LoQ	z-score
026	45	90	10	0.6	5	90	5	-2.3	<LOQ		10	
027	#				0			-4.5	0			-4.5
028	#				#				#			
029	#				#				#			
030	38.10	93.2	10	-0.2	0			-4.5	0			-4.5
031	38.2	104	10	-0.2	<LOQ		10		13.7	106	10	-0.3
032	#				#				#			
033	37.1	94.7	10	-0.3	#				<LOQ		10	
034	39	91	10	-0.1	#				#			
035	43	90	10	0.3	<LOQ		10		<LOQ		10	
036	47.80	93.17 %	20	0.9	<LOQ		20		<LOQ		20	
037	#				<LOQ		10		<LOQ		10	
038	#				#				#			
039	42.5	103	5	0.3	9.7	93	5	-0.1	17.8	103	5	1.0
040	37.7	100.1	20	-0.3	#				#			
041	37.4	126	10.0	-0.3	0			-4.5	0			-4.5
042	#				#				0			-4.5
043	#				#				0			-4.5
044	30.0	83.6	10.0	-1.1	<LOQ		10		<LOQ		10	
045	40.4	100	10	0.0	10.2	100	10	0.1	15.1	100	10	0.2
046	27.95	87.85	10	-1.4	#				0			-4.5
047	#				<LOQ		10		#			
048	34.21	88	10	-0.7	#				11.33	104	10	-1.0
049	#				#				#			
050	28.54	100	10	-1.3	0			-4.5	12.62	84	10	-0.6

*italics indicate for information only*

# not analysed

LoQ = limit of quantification

**Table 2 (continued): Results and z-Scores for Linuron, Methacrifos and Phosphamidon**

laboratory number	analyte											
	Linuron assigned value: 40.0 µg/kg				Methacrifos assigned value: 10.0 µg/kg				Phosphamidon assigned value: 14.5 µg/kg			
	result	rec. %	LoQ	z-score	result	rec. %	LoQ	z-score	result	rec. %	LoQ	z-score
051	27.1	102	9	-1.5	#				#			
052	46.6		10	0.7	#				#			
053	#				#				0			<i>-4.5</i>
054	38.3	102.0	20	-0.2	10.8	61.9	10	<i>0.4</i>	10.5	86.8	10	<i>-1.3</i>
055	32	56	10	-0.9	<10		10		0			<i>-4.5</i>
056	41	100	10	0.1	<LOQ (around 9)	100	10		11	101	10	<i>-1.1</i>
057	32.47	104.8	10	-0.9	0			<i>-4.5</i>	0			<i>-4.5</i>
058	50	96	10	1.1	#				19	94	10	<i>1.4</i>
059	#				#				30	98	10	<i>4.8</i>
060	43	100	10	0.3	0			<i>-4.5</i>	12	100	10	<i>-0.8</i>
061	43	86	10	0.3	#				17	97	10	<i>0.8</i>
062	42	108	10	0.2	13	113	10	<i>1.4</i>	15	105	10	<i>0.1</i>
063	41.7	98	20	0.2	8.2	88.5	20	<i>-0.8</i>	9.6	98.8	20	<i>-1.5</i>
064	21	82	10	<b>-2.2</b>	#				13	87	10	<i>-0.5</i>
065	33.60	-	10.0	-0.7	#				10.72	-	10.0	<i>-1.2</i>
066	#				#				#			
067	#				10.25	102.3	10	<i>0.1</i>	14.80	85	10	<i>0.1</i>
068	42		10	0.2	0			<i>-4.5</i>	11		10	<i>-1.1</i>
069	35.0		10	-0.6	<LOQ		10		<LOQ		10	
070	33.5	77.3	10	-0.7	#				#			
071	39	100	10	-0.1	0			<i>-4.5</i>	23	100	10	<i>2.7</i>
072	35	89	10	-0.6	7	98	5	<i>-1.4</i>	18	85	10	<i>1.1</i>
073	49.8	102	10	1.1	<LOQ		10		11.3	89	10	<i>-1.0</i>
074	31	65%	5	-1.0	8	100%	5	<i>-0.9</i>	10	90%	5	<i>-1.4</i>
075	37.6	100	10	-0.3	#				0			<i>-4.5</i>

z-scores outside  $|z| > 2$  are shown in **bold**, see Section 5*italics indicate for information only*

# not analysed

LoQ = limit of quantification

**Table 2 (continued): Results and z-Scores for Linuron, Methacrifos and Phosphamidon**

laboratory number	analyte											
	Linuron assigned value: 40.0 µg/kg				Methacrifos assigned value: 10.0 µg/kg				Phosphamidon assigned value: 14.5 µg/kg			
	result	rec. %	LoQ	z-score	result	rec. %	LoQ	z-score	result	rec. %	LoQ	z-score
076	43	104	10	0.3	0			-4.5	11	102	10	-1.1
077	0			<b>-4.5</b>	#				0			-4.5
078	43.65	115	10	0.4	0			-4.5	90.00	100	10	23.6
079	#				#				#			
080	43	100	10	0.3	9	100	5	-0.5	15	100	10	0.1
081	43	101	10	0.3	14	97	10	1.8	14	98	10	-0.2
082	49	78	10	1.0	#				0			-4.5
083	52	88	10	1.4	0			-4.5	0			-4.5
084	44	102	5	0.5	<10	90	10		14	90	5	-0.2
085	42	96	10	0.2	0			-4.5	16	101	10	0.5
086	44		10	0.5	10		10	0.0	27		10	3.9
087	43	91	10	0.3	10.1	117	10	0.0	10.6	104	10	-1.2
088	#				#				#			
089	45	100	10	0.6	#				#			
090	40.7	80	10	0.1	#				<LOQ		10	
091	39	91	10	-0.1	#				#			
092	42.2	96	10	0.2	#				<LOQ		10	
093	36.5	70-120	10	-0.4	<LOQ	70-120	10		#			
094	41		10	0.1	0			-4.5	#			
095	#				#				#			

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

*italics indicate for information only*

# not analysed

LoQ = limit of quantification



**Table 3: Results and z-Scores for Pirimicarb, Propargite and Vinclozolin**

laboratory number	analyte											
	Pirimicarb assigned value: 80.2 µg/kg				Propargite assigned value: 54.5 µg/kg				Vinclozolin assigned value: 50.2 µg/kg			
	result	rec. %	LoQ	z-score	result	rec. %	LoQ	z-score	result	rec. %	LoQ	z-score
001	43	100	10	<b>-2.1</b>	49	100	10	-0.5	39	100	10	-1.0
002	77.7			-0.1	60.2			0.5	50.5			0.0
003	74.8	104	10	-0.3	60.8	110	10	0.5	47.3	108	10	-0.3
004	81	100	5	0.0	#				#			
005	#				#				#			
006	54	88	5	-1.5	#				0	85	5	<b>-4.5</b>
007	88	104	10	0.4	57	97	10	0.2	52	104	10	0.2
008	91.5	89	10	0.6	50.8	94	10	-0.3	#			
009	92.3	94	10	0.7	71.4	107	10	1.4	74.5	112	10	<b>2.2</b>
010	#				52.1	94.2	10	-0.2	52.9	98.5	10	0.2
011	52	109	10	-1.6	78	113	10	2.0	51	102	10	0.1
012	85.8	100.6	10	0.3	51.4	79.2	10	-0.3	56.0	101.4	10	0.5
013	#				63.066	106.68		0.7	#			
014	81	93	10	0.0	52	79	10	-0.2	52	100	10	0.2
015	82.49	99.4	10	0.1	64.07	101.8	10	0.8	58.67	101.3	10	0.8
016	#				#				#			
017	72.3	104	10	-0.4	43.5	102	10	-0.9	46.2	109	10	-0.4
018	77	>90	10	-0.2	58	>90	10	0.3	54	>90	10	0.3
019	#				#				#			
020	52	110	5	-1.6	#				34	110	5	-1.5
021	84	94	10	0.2	41	80	10	-1.1	56	95	10	0.5
022	61.3	98	10	-1.1	59.8	100	10	0.4	41.1	97	10	-0.8
023	#				#				#			
024	#				#				#			
025	#				0			<b>-4.5</b>	80.5	92.4	10.0	<b>2.7</b>

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

# not analysed

LoQ = limit of quantification

**Table 3 (continued): Results and z-Scores for Pirimicarb, Propargite and Vinclozolin**

laboratory number	analyte											
	Pirimicarb assigned value: 80.2 µg/kg				Propargite assigned value: 54.5 µg/kg				Vinclozolin assigned value: 50.2 µg/kg			
	result	rec. %	LoQ	z-score	result	rec. %	LoQ	z-score	result	rec. %	LoQ	z-score
026	82	90	10	0.1	55	90	10	0.0	61	90	10	1.0
027	#				#				37.3	119	10	-1.2
028	#				#				#			
029	#				#				#			
030	75.71	78.6	10	-0.3	#				48.21	71.8	10	-0.2
031	109.0	124	10	1.6	60.9	125	10	0.5	53.0	94.5	10	0.3
032	74	119	20	-0.4	52	106	20	-0.2	51	93	20	0.1
033	85.8	94.0	10	0.3	#				0		10	<b>-4.5</b>
034	90	106	10	0.6	74	112	10	1.6	53	116	10	0.3
035	80	90	10	0.0	53	90	10	-0.1	46	90	10	-0.4
036	66.87	99.19 %	20	-0.8	43.31	92.11 %	20	-0.9	33.51	98.39 %	20	-1.5
037	75	73	10	-0.3	87	73	10	<b>2.7</b>	48	119	10	-0.2
038	#				#				#			
039	95.1	105	5	0.8	63.6	104	5	0.8	64.2	88	5	1.3
040	75.1	101.6	20	-0.3	45.7	102.3	20	-0.7	43.2	103.4	20	-0.6
041	76.9	120	10.0	-0.2	66.6	98	10.0	1.0	59.3	110	10.0	0.8
042	#				#				50.0	102	10.0	0.0
043	48.72	102.3	10	-1.8	25.75	96.9	10	<b>-2.4</b>	43.63	103.2	10	-0.6
044	55.0	76.5	10.0	-1.4	34.7	83.1	10.0	-1.7	44.0	82.2	10.0	-0.6
045	73.5	100	10	-0.4	55.0	100	10	0.0	51.2	100	10	0.1
046	#				#				42.83	102.6	10	-0.7
047	#				#				34.9	109%	10	-1.4
048	58.82	70	10	-1.2	40.73	91	10	-1.1	48.54	100	10	-0.1
049	#				#				#			
050	76.98	86	10	-0.2	47.50	96	10	-0.6	39.70	85	10	-1.0

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

# not analysed

LoQ = limit of quantification

**Table 3 (continued): Results and z-Scores for Pirimicarb, Propargite and Vinclozolin**

laboratory number	analyte											
	Pirimicarb assigned value: 80.2 µg/kg				Propargite assigned value: 54.5 µg/kg				Vinclozolin assigned value: 50.2 µg/kg			
	result	rec. %	LoQ	z-score	result	rec. %	LoQ	z-score	result	rec. %	LoQ	z-score
051	83.4	117	10	0.2	#				52.9	78	3	0.2
052	92.6		10	0.7	60.4		10	0.5	49.4		10	-0.1
053	93	107	10	0.7	#				#			
054	86.3	80.8	20	0.3	60.6	90.5	20	0.5	45.0	75.4	20	-0.5
055	67	111	10	-0.7	28	67	10	<b>-2.2</b>	42	102	10	-0.7
056	81	101	10	0.0	46	97.9	10	-0.7	46	100.1	10	-0.4
057	75.35	100.7	10	-0.3	43.17	109.4	10	-0.9	32.51	68.2	10	-1.6
058	#				70	95	10	1.3	#			
059	101	100	10	1.2	#				58	106	10	0.7
060	92	100	10	0.7	60	100	10	0.5	64	100	10	1.3
061	81	88	10	0.0	72	82	10	1.5	55	90	10	0.4
062	86	105	10	0.3	68	96	10	1.1	67	67	10	1.5
063	80.6	97.8	20	0.0	54.5	90.8	20	0.0	48.1	99.7	20	-0.2
064	#				74	89	10	1.6	60	107	10	0.9
065	81.18	-	10.0	0.1	51.55	-	10.0	-0.2	50.83	90.2	10.0	0.1
066	#				#				#			
067	85.70	75.2	10	0.3	#				#			
068	86		10	0.3	53		10	-0.1	54		10	0.3
069	65.7		10	-0.8	34.8		10	-1.6	39.4		10	-1.0
070	73.0	82.1	10	-0.4	71.8	103	10	1.4	65.9	106	10	1.4
071	86	100	10	0.3	56	100	10	0.1	50	100	10	0.0
072	80	93	10	0.0	62	105	10	0.6	51	105	10	0.1
073	88.5	104	10	0.5	68.4	100	10	1.2	48.5	93	10	-0.2
074	69	88%	5	-0.6	45	93%	5	-0.8	40	88%	5	-0.9
075	78	100	20	-0.1	50	100	10	-0.4	#			

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

# not analysed

LoQ = limit of quantification

**Table 3 (continued): Results and z-Scores for Pirimicarb, Propargite and Vinclozolin**

laboratory number	analyte											
	Pirimicarb assigned value: 80.2 µg/kg				Propargite assigned value: 54.5 µg/kg				Vinclozolin assigned value: 50.2 µg/kg			
	result	rec. %	LoQ	z-score	result	rec. %	LoQ	z-score	result	rec. %	LoQ	z-score
076	97	101	10	1.0	55	110	10	0.0	59	88	10	0.8
077	#				43.4	89	10	-0.9	0			<b>-4.5</b>
078	84.45	95	10	0.2	55.75	100	10	0.1	48.30	80	10	-0.2
079	25.6	41.0	10	<b>-3.1</b>	37.3	108.4	10	-1.4	40.1	124.6	10	-0.9
080	88	100	10	0.4	60	100	10	0.5	52	100	10	0.2
081	85	96	10	0.3	55	100	10	0.0	58	100	10	0.7
082	0			<b>-4.5</b>	#				69	83	10	1.7
083	94	87	10	0.8	52	96	10	-0.2	59	108	10	0.8
084	86	104	5	0.3	43	85	5	-1.0	48	96	5	-0.2
085	87	96	10	0.4	63	123	10	0.7	57	108	10	0.6
086	88		10	0.4	0			<b>-4.5</b>	40		10	-0.9
087	88	93	10	0.4	68	90	10	1.1	0			<b>-4.5</b>
088	#				#				#			
089	76	100	10	-0.2	46	100	10	-0.7	45	100	10	-0.5
090	72.5	69	10	-0.4	49.3	73	10	-0.4	30.1	107	10	-1.8
091	69	83	10	-0.6	31	84	10	-2.0	51	84	10	0.1
092	87.2	101.8	10	0.4	36.2	97.5	10	-1.5	47.2	101.3	10	-0.3
093	91.5	70-120	10	0.6	51.6	70-120	10	-0.2	48.7	70-120	10	-0.1
094	88		10	0.4	50		10	-0.4	44		10	-0.6
095	#				0			<b>-4.5</b>	#			

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

# not analysed

LoQ = limit of quantification

**Table 4: Additional Pesticide Residues Reported**

laboratory number	pesticide residue $\geq 16 \mu\text{g}/\text{kg}$	result $\mu\text{g}/\text{kg}$	recovery %	LoQ $\mu\text{g}/\text{kg}$
012	trans-Chlordane	123	94.5	10
025	Bifenthrin	24.2	92.4	10.0
025	Cyfluthrin (sum)	23.9	91.4	10.0
025	lambda-Cyhalothrin	22.4	92.0	10.0
025	Cypermethrin (sum isomers)	26.4	89.0	10.0
025	pp'-DDD	23.5	89.6	10.0
025	pp'-DDE	22.8	92.5	10.0
025	op'-DDT	21.4	93.4	10.0
025	pp'-DDT	22.6	96.4	10.0
025	Endosulfan sulfate	19.4	95.1	10.0
025	Ethion	22.6	92.4	10.0
025	Fenvalerate (sum of all isomers)	22.4	95.4	10.0
025	Hexachlorobenzene (HCB)	23.5	91.0	10.0
025	alpha-HCH	21.9	96.0	10.0
025	beta-HCH	19.4	89.5	10.0
025	gamma-HCH/Lindane	17.2	91.4	10.0
025	Phorate	17.5	90.4	10.0
059	trans-Chlordane	310	96	10
069	trans-Chlordane	86.4		10
070	trans-Chlordane	35.7	85.8	10
082	trans-Chlordane	48	90	10
088	alpha-Endosulfan	18	93	10
095	Bifenthrin	70		30

LoQ = limit of quantification

**Table 5: Participants' Comments**

laboratory number	comments
001	Traces of acetamiprid and methacriphos were detected (<10 µg/kg)
008	The results of pesticides analyzed on GC could not be supplied because our GC/MS/MS instrument is out of order because of an electronic card error.
010	For Metalaxyl was determined only Metalaxyl, without Metalaxyl-M.
012	Carbendazim,Carbofuran,chlorpyrifos ,Difenoconazole and Tebuconazole are in traces and below our Lab LOQ ie 10 ppb
021	appropriate standard of cis-Chlordane was not available for proper quantification of cis-Chlordane
025	Matrix Match Calibration applied and Not detected results was assessed as Below Limit of Quantification
030	This is for accreditation purpose. Analytes Methacrifos (4.31 ppb) and Phosphamidon (6.55 ppb) detected but below the LOQ values.
031	Methacrifos was detected (about 8 µg/kg ), but lower than LOQ ( 10 µg/kg ), so the result was not reported.
038	Method Not accredited
044	LOQ of all of the not detected Analytes is 10.0 µg/kg
056	Methacrifos detected but just below LOQ of 10 µg/kg
058	Analysis was done on matrix based linearity
060	Carbofuran=0.31 ug/kg was detected in the blank sample
066	QuEChERS (lab modified) method for sample extraction. Instrument was HPLC.
071	We also found traces of methacrifos but these are below our reporting limit.
073	Methacrifos <10 µg/kg (LOQ)
085	Trace of Methacrifos
091	quantification with standard-addition approach, result for deltamethrin in routine samples would be reported as <RL""
093	Phosphamidon is detected, but it's amount is decreased significantly in the difference analysis days. So it's result is not submitted. Carbendazim is detected at trace concentration (1 µg/kg), but we report 'not detected' with LOQ = 10 µg/kg.

comments are as submitted by participants

**Table 6: Assigned Values and Standard Deviations for Proficiency**

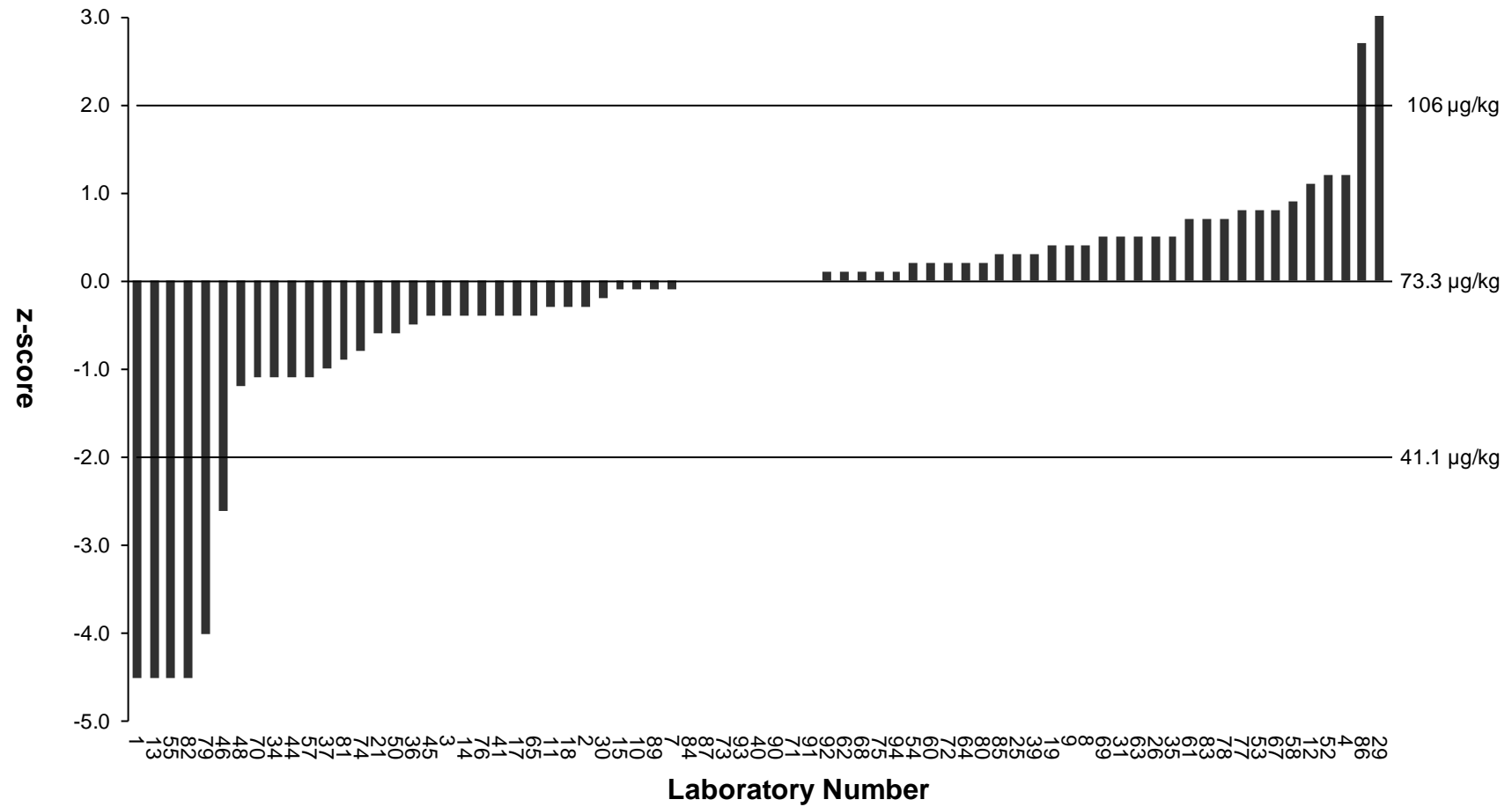
analyte	data points, <i>n</i>	assigned value, $x_a$ $\mu\text{g/kg}$	uncertainty, <i>u</i>	standard deviation for proficiency, $\sigma_p$ , $\mu\text{g/kg}$	
Acetamiprid	60	73.3	1.3	Horwitz [9]	16.1
cis-Chlordane	61	31.5	1.1	Horwitz [9]	6.93
Deltamethrin	69	58.1	1.5	Horwitz [9]	12.8
Linuron	55	40.0	0.9	Horwitz [9]	8.80
Methacrifos	17	<i>10.0</i>	<i>0.4</i>	Horwitz [9]	<i>2.20</i>
Phosphamidon	34	<i>14.5</i>	<i>0.6</i>	Horwitz [9]	<i>3.20</i>
Pirimicarb	63	80.2	1.4	Horwitz [9]	17.6
Propargite	59	54.5	1.7	Horwitz [9]	12.0
Vinclozolin	65	50.2	1.1	Horwitz [9]	11.0

*italics indicate for information only*

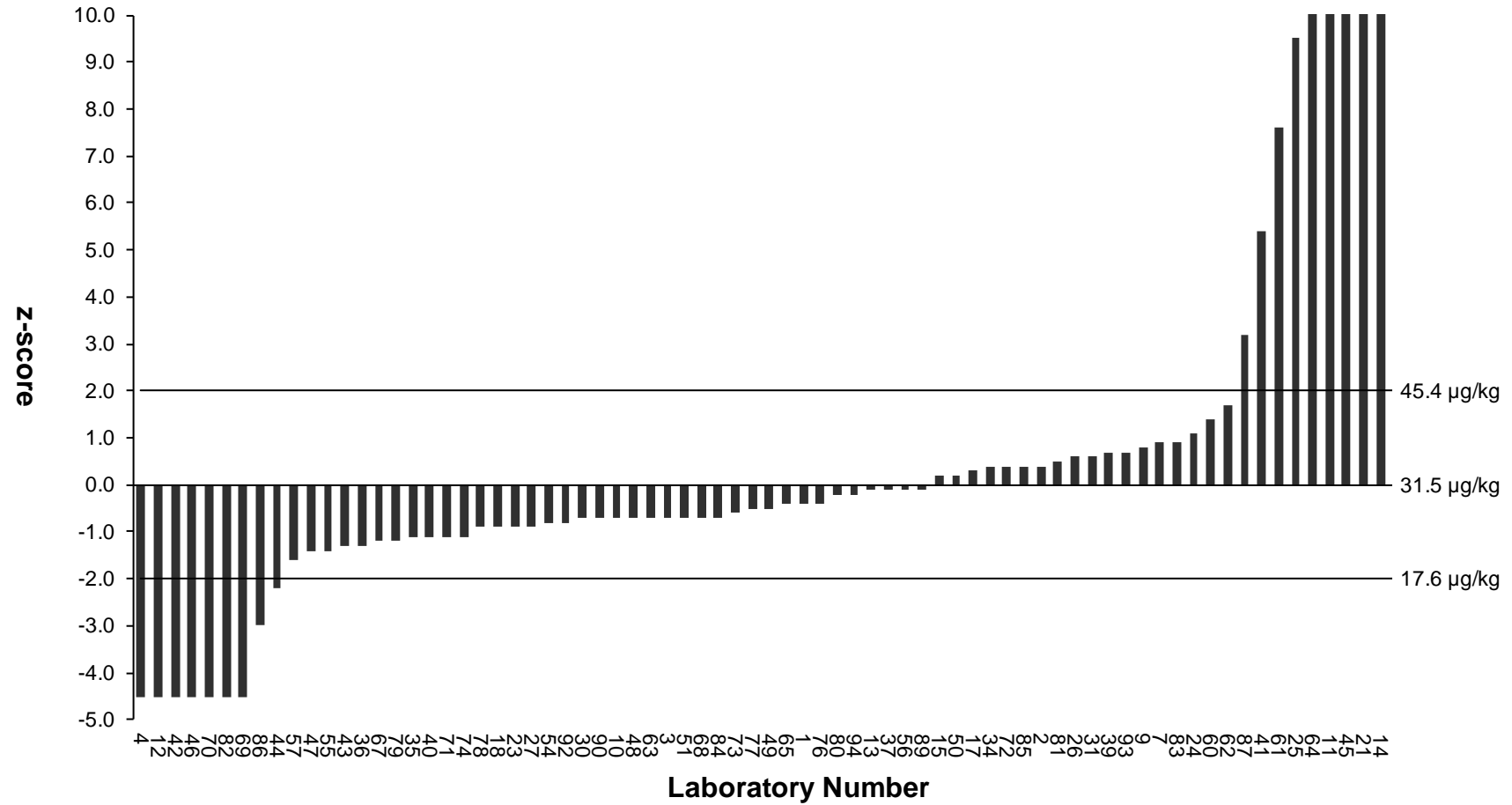
**Table 7: Number and Percentage of z-Scores where  $|z| \leq 2$** 

analyte	number of scores where $ z  \leq 2$	total number of scores	% $ z  \leq 2$
Acetamiprid	65	73	89
cis-Chlordane	56	74	76
Deltamethrin	72	82	88
Linuron	62	65	95
Methacrifos	<i>17</i>	33	<i>52</i>
Phosphamidon	<i>36</i>	56	<i>64</i>
Pirimicarb	70	73	96
Propargite	64	70	91
Vinclozolin	70	76	92

*italics indicate for information only*







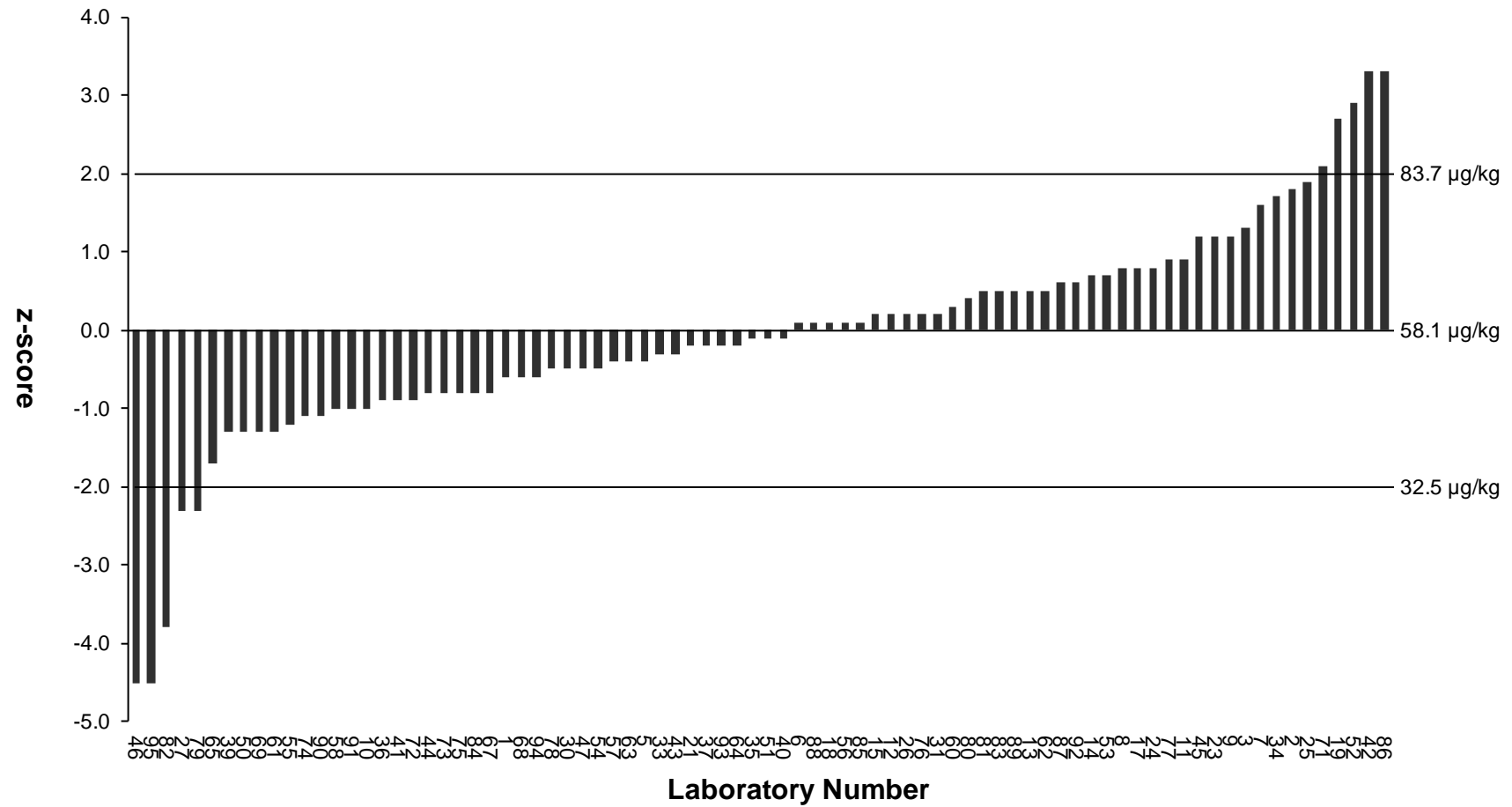


Figure 3: z-Scores for Deltamethrin

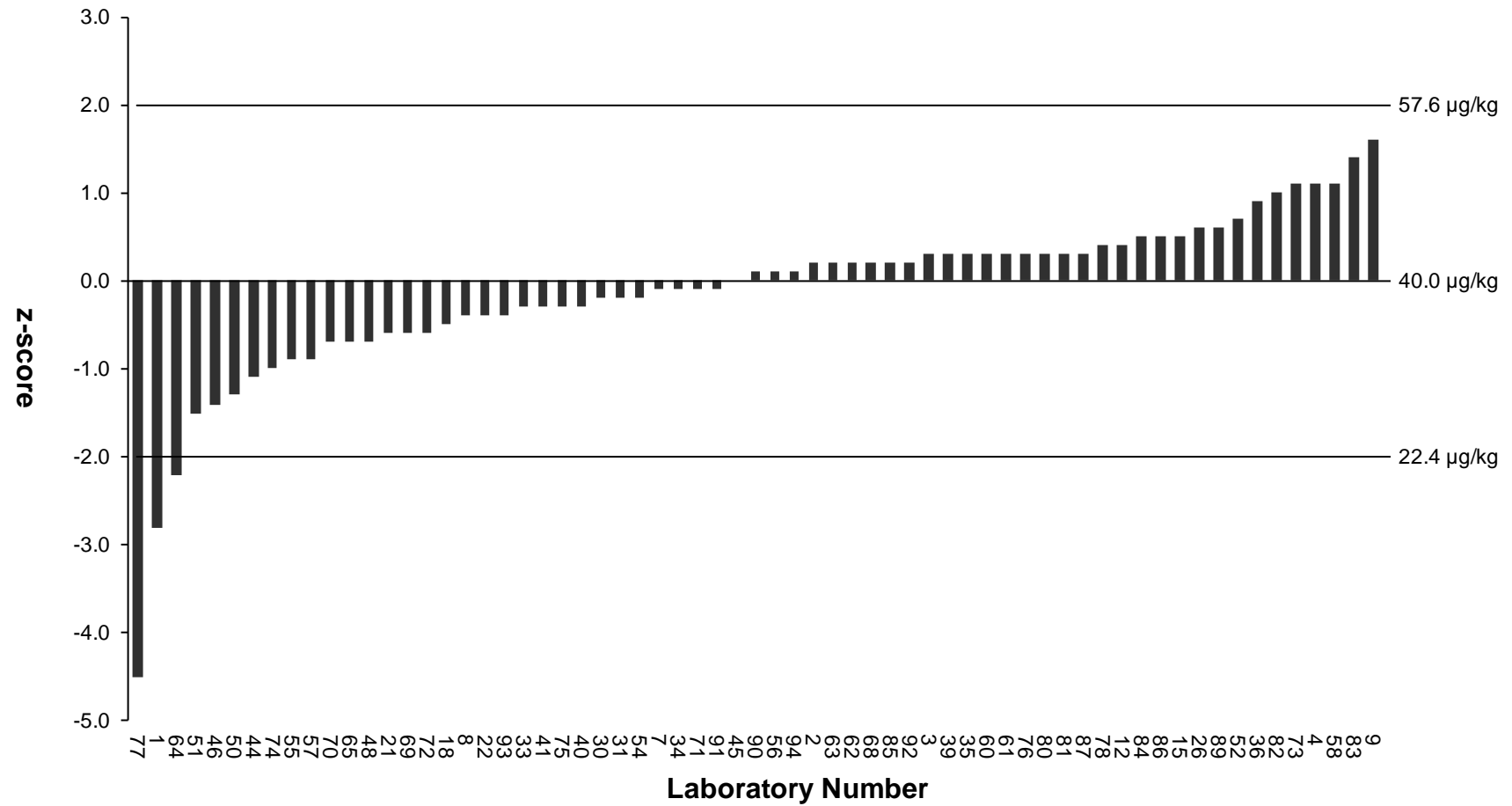
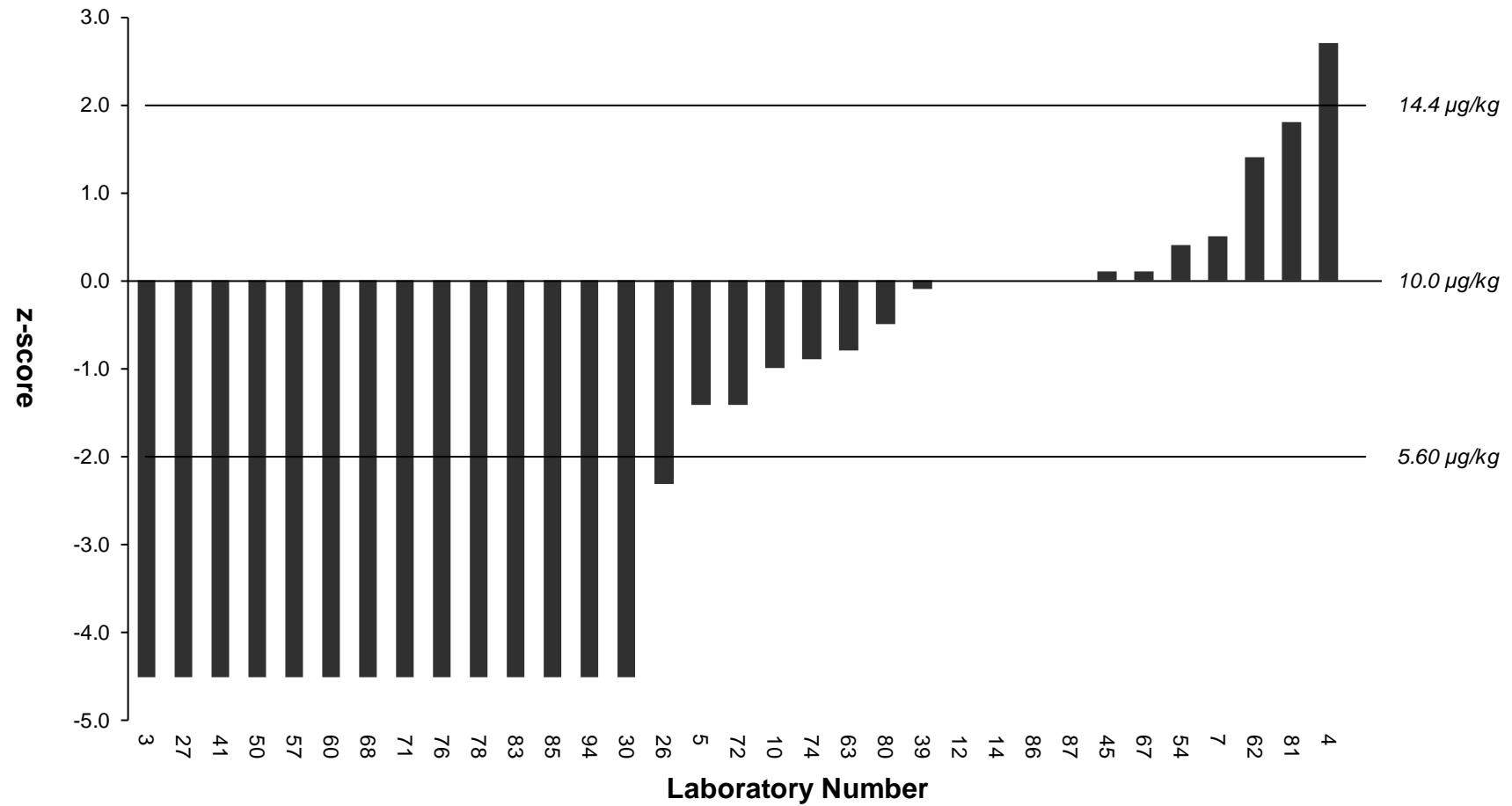
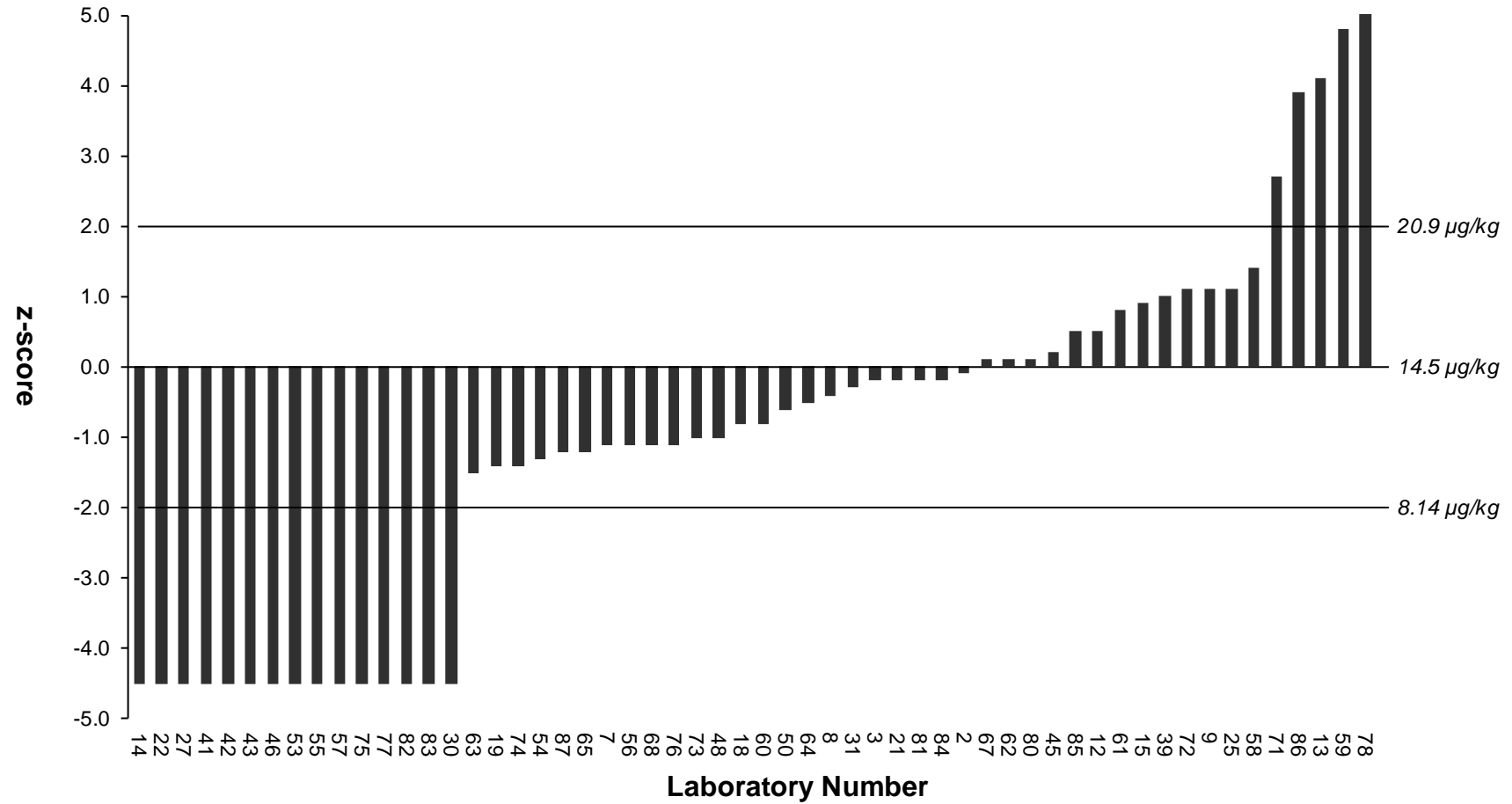


Figure 4: z-Scores for Linuron



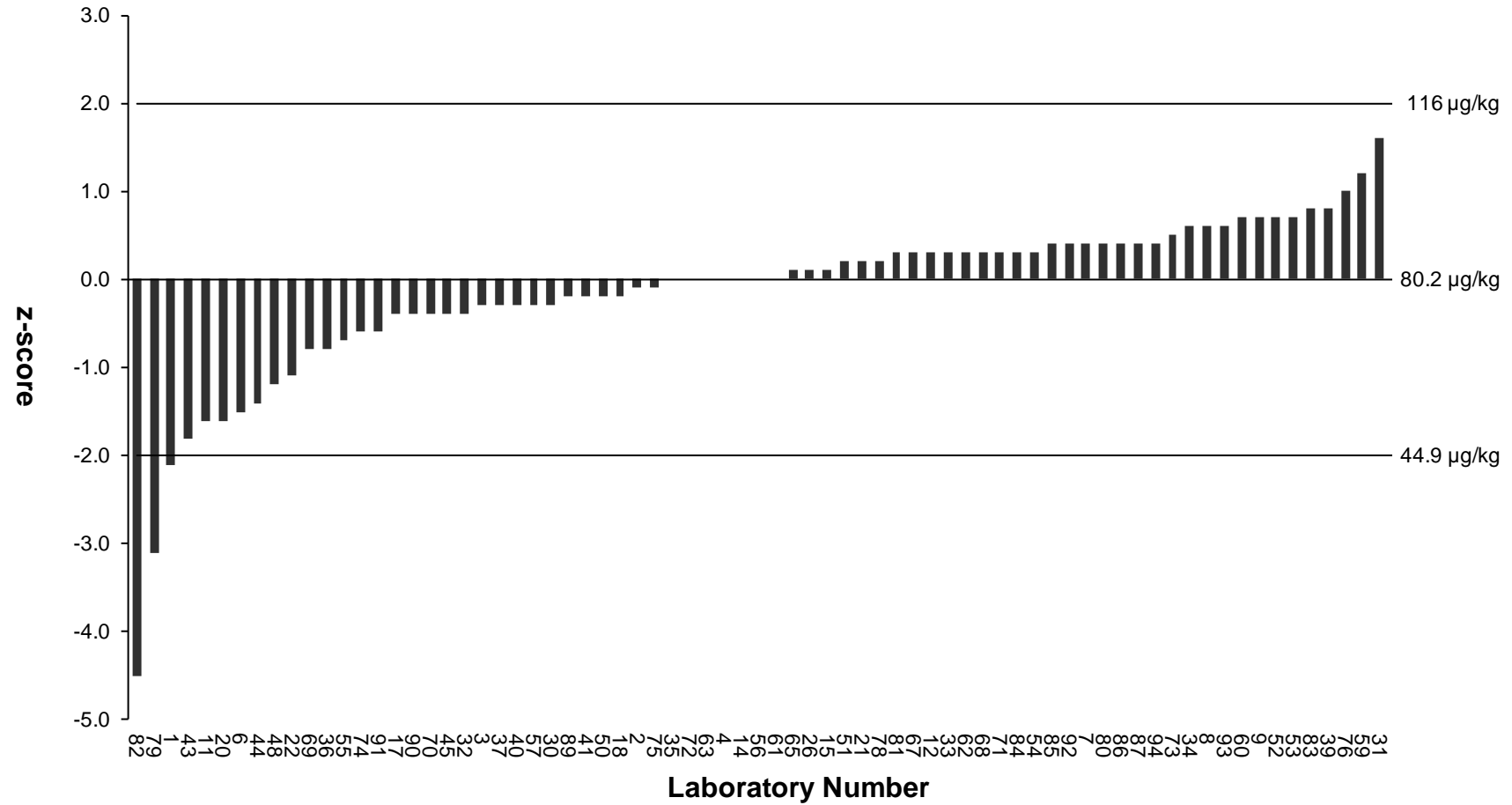
**Figure 5: z-Scores for Methacrifos**

*this histogram is given for information only*



**Figure 6: z-Scores for Phosphamidon**

*this histogram is given for information only*



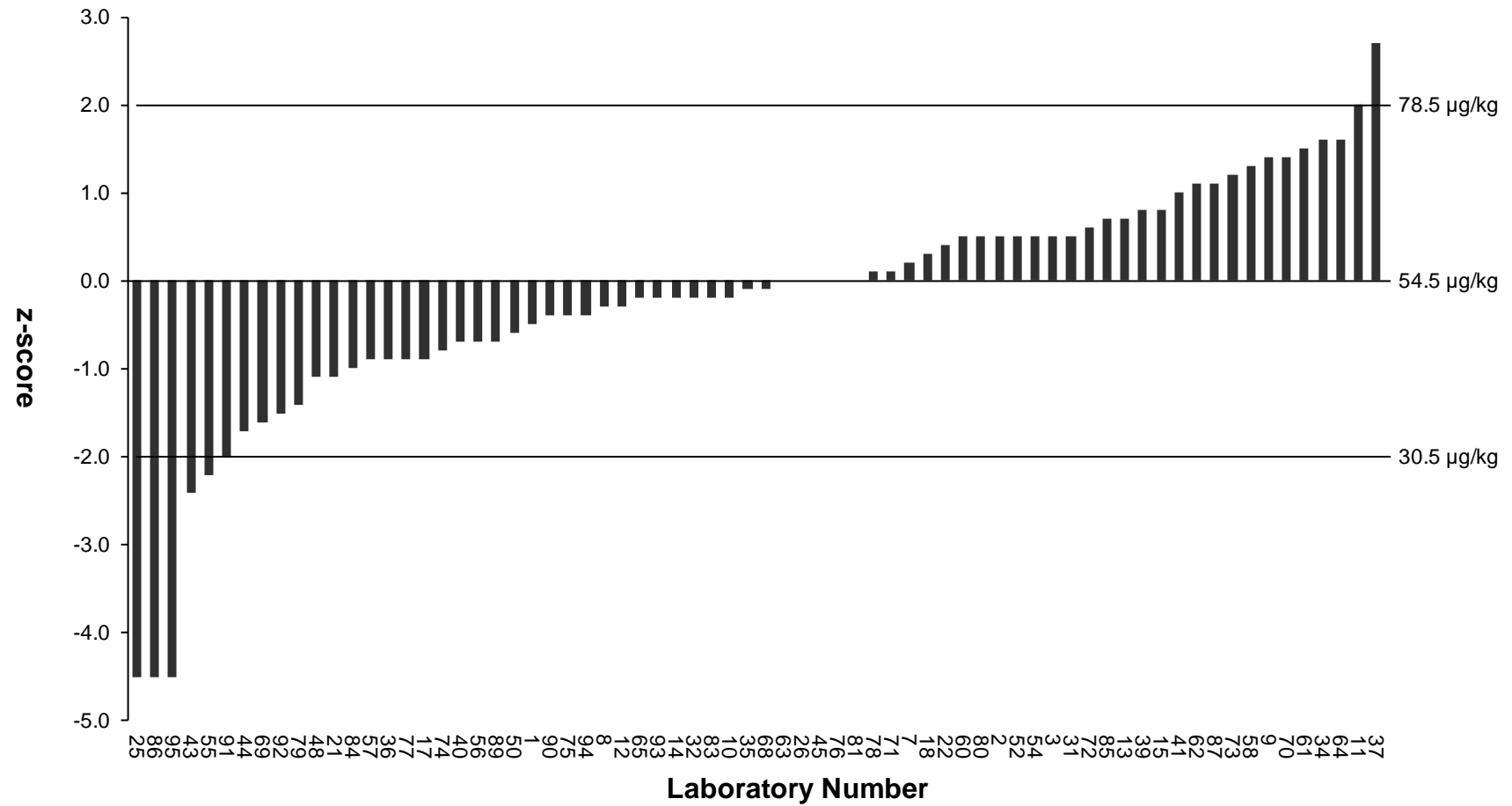


Figure 8: z-Scores for Propargite

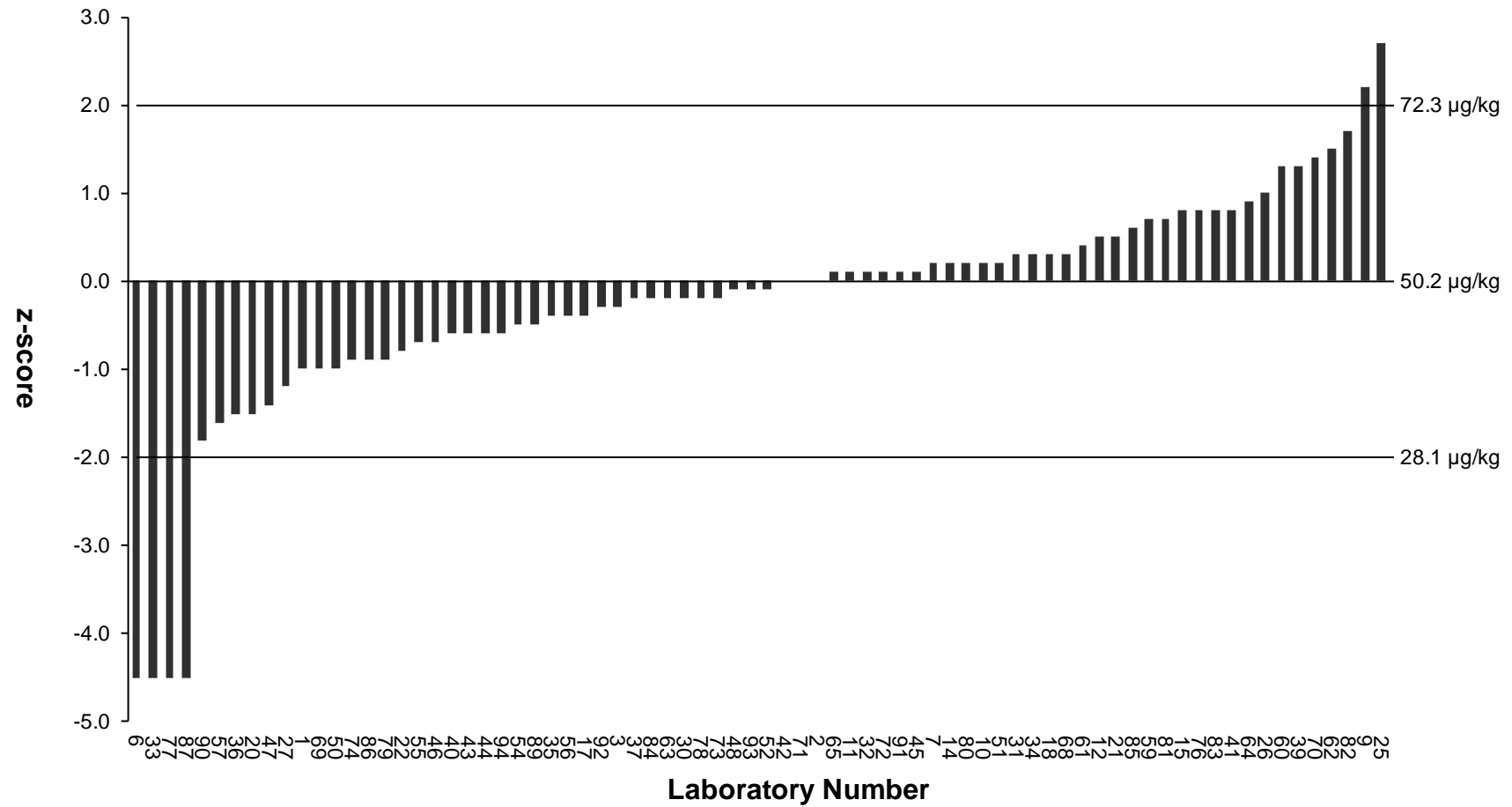


Figure 9: z-Scores for Vinclozolin



## 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.

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### Acetamiprid

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Method Principle	laboratory number
GC	017 030
LC	002 007 008 009 010 011 012 015 018 019 021 025 029 031 034 035 036 037 039 041 044 045 046 048 050 054 057 058 060 061 062 063 064 065 067 069 070 071 072 073 074 075 076 077 078 079 080 083 087 089 090 091 092 093

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Identification by Mass Spectrometry?	laboratory number
yes	002 007 008 009 011 012 015 017 018 019 021 025 029 030 031 034 035 036 037 039 041 044 045 046 048 050 054 057 058 060 061 062 063 064 065 067 069 070 071 072 073 074 075 076 077 078 079 080 083 087 089 090 091 092 093
no	010

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### cis-Chlordane

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Method Principle	laboratory number
GC	001 002 007 009 010 011 015 017 018 021 023 024 025 030 031 034 035 036 037 039 041 043 044 045 047 048 049 050 051 054 055 056 057 060 061 062 063 064 065 067 071 072 073 074 076 077 078 079 080 083 087 089 090 092 093

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Identification by Mass Spectrometry?	laboratory number
yes	001 002 007 009 011 015 017 018 021 023 025 030 031 034 035 036 037 039 041 043 044 045 048 049 050 051 054 055 056 057 060 061 062 063 064 065 067 071 072 073 074 076 077 078 079 080 087 089 090 092 093
no	010 024 047 083

## Deltamethrin

Method Principle	laboratory number
GC	002 005 007 009 010 012 015 017 018 019 023 024 025 030 034 035 036 037 039 041 043 044 045 047 050 051 054 055 057 058 060 061 063 064 065 067 071 073 075 076 077 078 079 080 083 087 088 089 092 093
LC	006 008 011 021 031 033 056 062 069 072 074 090 091

Identification by Mass Spectrometry?	laboratory number
yes	002 005 007 008 009 011 012 015 017 018 019 021 023 025 030 031 033 034 035 036 037 039 041 043 044 045 050 054 055 056 057 058 060 061 062 063 064 065 067 069 071 072 073 074 076 077 078 079 080 083 087 089 090 091 092 093
no	006 010 024 047 051 088

## Linuron

Method Principle	laboratory number
GC	030 055 069
LC	002 007 008 009 012 015 018 021 022 031 033 034 035 036 039 041 044 045 046 048 050 051 054 056 057 058 060 061 062 063 064 065 070 071 072 073 074 075 076 078 080 083 087 089 090 091 092 093

Identification by Mass Spectrometry?	laboratory number
yes	002 007 008 009 012 015 018 021 022 030 031 033 034 035 036 039 041 044 045 046 048 050 051 054 055 056 057 058 060 061 062 063 064 065 069 070 071 072 073 074 075 076 078 080 083 087 089 090 091 092 093

## Methacrifos

Method Principle	laboratory number
GC	005 010 012 039 045 054 055 062 063 067 072 074 080 087
LC	007 056

Identification by Mass Spectrometry?	laboratory number
yes	005 007 012 039 045 054 055 056 062 063 067 072 074 080 087
no	010

## Phosphamidon

Method Principle	laboratory number
GC	002 012 018 019 021 045 071 072 073 080
LC	007 008 009 015 025 031 039 048 050 054 056 058 059 060 061 062 063 064 065 067 074 076 078 087

Identification by Mass Spectrometry?	laboratory number
yes	002 007 008 009 012 015 018 019 021 025 031 039 045 048 050 054 056 058 059 060 061 062 063 064 065 067 071 072 073 074 076 078 080 087

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## Pirimicarb

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Method Principle	laboratory number
GC	012 017 021 022 030 034 035 043 045 051 055 069 075 089 091 092
LC	002 006 007 008 009 011 015 018 031 033 036 037 039 041 044 048 050 054 056 057 059 060 061 062 063 065 067 070 071 072 073 074 076 078 079 080 083 087 090 093

Identification by Mass Spectrometry?	laboratory number
yes	002 007 008 009 011 012 015 017 018 021 022 030 031 033 034 035 036 037 039 041 043 044 045 048 050 051 054 055 056 057 059 060 061 062 063 065 067 069 070 071 072 073 074 076 078 079 080 083 087 089 090 091 092 093
no	006

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## Propargite

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Method Principle	laboratory number
GC	002 009 010 017 022 034 037 041 043 045 050 055 056 060 062 064 069 070 073 076 079 080 087 091 092
LC	007 008 011 012 015 018 021 031 035 036 039 044 048 054 057 058 061 063 065 071 072 074 075 077 078 083 089 090 093

Identification by Mass Spectrometry?	laboratory number
yes	002 007 008 009 011 012 015 017 018 021 022 031 034 035 036 037 039 041 043 044 045 048 050 054 055 056 057 058 060 061 062 063 064 065 069 070 071 072 073 074 075 076 077 078 079 080 083 087 089 090 091 092 093
no	010

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**Vinclozolin**

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<b>Method Principle</b>	<b>laboratory number</b>
GC	002 007 009 010 011 012 015 017 018 021 022 025 030 031 034 035 036 037 039 041 043 044 045 046 047 048 050 051 054 055 056 057 059 060 061 062 063 064 065 069 071 072 073 074 076 078 079 080 083 089 090 091 092
LC	070 093

---

<b>Identification by Mass Spectrometry?</b>	<b>laboratory number</b>
yes	002 007 009 011 012 015 017 018 021 022 025 030 031 034 035 036 037 039 041 043 044 045 046 048 050 054 055 056 057 059 060 061 062 063 064 065 069 070 071 072 073 074 076 078 079 080 083 089 090 091 092 093
no	010 047 051

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## APPENDIX II: Fapas<sup>®</sup> SecureWeb, Protocol and Contact Details

### 1. Fapas<sup>®</sup> 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<sup>®</sup> 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<sup>®</sup> – Food Chemistry proficiency tests.

### 2. PROTOCOL

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

### 3. CONTACT DETAILS

This report was prepared and authorised on behalf of Fapas<sup>®</sup> by Izabele Cincyte (Round Coordinator). Participants with any comments or concerns about this proficiency test should contact:

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