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LABORATORY NUMBER: [Available to participants from FAPAS SecureWeb](#)

FAPAS® Proficiency Test 19117

Pesticide Residues in Grape Purée

February–April 2011

Report

Prepared and authorised on behalf of FAPAS by

A handwritten signature in black ink that reads 'Michael Knaggs'.

Michael Knaggs

SUMMARY

1. The test material for FAPAS® proficiency test 19117 was dispatched in February 2011. Each participant received a grape purée test material. From a list of 146 pesticide residues, participants had to identify and quantify those present in the test material.
2. The test material contained deltamethrin, imidacloprid, kresoxim-methyl, monocrotophos, quinalphos and quinoxifen. For each analyte, an assigned value (x_a) was determined. In conjunction with the standard deviation for proficiency (σ_p), a z-score for each result was calculated.
3. Results for this proficiency test are summarised as follows:

analyte	assigned value, x_a µg/kg	number of scores, $ z \leq 2$	total number of scores	% $ z \leq 2$
deltamethrin	184	59	75	79
imidacloprid	139	60	68	88
kresoxim-methyl	162	65	73	89
monocrotophos	330	53	69	77
quinalphos	39.0	54	67	81
quinoxifen	250	45	49	92

4. Surplus test materials are available for sale, see APPENDIX II.
5. Whereas this report has been produced in good faith and in accordance with best industry practice, neither The Food and Environment Research Agency nor the Secretary of State for Environment, Food and Rural Affairs accepts any liability whatsoever as to the application or use of the information contained therein.

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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[®] proficiency testing scheme is available in our protocols [1, 2].

2. TEST MATERIAL

2.1. Preparation

Sample preparation was carried out by a laboratory contracted to do so by FAPAS[®].

The test material was prepared from organic grapes that were obtained from a commercial supplier. They were frozen, cryogenically milled and thawed to produce a purée.

A sample of the purée was screened for the presence of incurred pesticide residues. No residues were found above 30 µg/kg.

The purée was split into two batches: one was used to prepare the blank test material, the other to prepare the spiked test material.

The following pesticides were spiked into the spiked test material: deltamethrin, imidacloprid, kresoxim-methyl, monocrotophos, quinalphos and quinoxyfen.

Samples were stored at -20°C until dispatch.

2.2. Homogeneity

To test for homogeneity, randomly selected test materials were analysed in duplicate by a laboratory contracted to do so by FAPAS[®].

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

2.3. Dispatch

The start date was 21 February 2011. Test materials were sent to 82 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 79 participants (96%) before the closing date for this test, 6 April 2011.

Each participant was given a laboratory number, assigned in order of receipt of results. The reported analyte concentrations are given in Table 1 for deltamethrin, imidacloprid and kresoxim-methyl and in Table 2 for monocrotophos, quinalphos and quinoxyfen.

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 -2.0, they were assessed as if their result was zero.

Participants' comments are given in Table 3.

Any participant identifying pesticide residues other than deltamethrin, imidacloprid, kresoxim-methyl, monocrotophos, quinalphos and quinoxyfen at levels ≥ 30 $\mu\text{g}/\text{kg}$ is listed in Table 4 together with the pesticide residues reported and the level determined.

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. In combination with the standard deviation for proficiency, σ_p , a z-score was calculated for each result. The procedure follows that recommended in the IUPAC International Harmonised Protocol for the Proficiency Testing of Analytical Chemistry Laboratories [3].

Further details on the procedure followed can be found in the relevant protocols [1, 2].

4.1. Calculation of the Assigned Value, x_a

The assigned value, x_a , was set as the consensus of the results submitted by participants.

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

- i) results reported as approximately 10, 100 or 1000 \times greater or smaller than the majority of submitted results (as these were considered to be reporting errors),
- ii) results where no percentage recovery was reported,
- iii) results whose recovery was outside the range 70–120% [4],
- iv) results where no limit of quantification was reported,
- v) results lower than the participant's reported limit of quantification.

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

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

4.2. Standard Deviation for Proficiency, σ_p

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

For all residues, σ_p was derived from the appropriate form of the Horwitz equation [5].

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

4.3. Individual z-Scores

Participants' z-scores were calculated as:

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

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

Participants' z-scores are given in Table 1 for deltamethrin, imidacloprid and kresoxim-methyl and in Table 2 for monocrotophos, quinalphos and quinoxyfen. They are shown as histograms in Figures 1–6. It is possible for the z-scores published in this report to differ slightly from the z-score that can be calculated using the formula given above. These differences arise from the necessary rounding of the actual assigned values and standard deviations for proficiency prior to their publication in Table 5.

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

5. ASSESSMENT 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 [3].

6. REFERENCES

- 1 FAPAS, 2010, Protocol for Proficiency Testing Schemes, Part 1 – Common Principles, Version 2, Issued December 2010.
- 2 FAPAS, 2009, Protocol for Proficiency Testing Schemes, Part 2 – FAPAS®, Revision 2009, Version 1, Issued November 2009.
- 3 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.
- 4 Pihlström, T (Co-ordinator), 2010, *Method Validation and Quality Control Procedures for Pesticide Residues Analysis in Food and Feed*, Document No. SANCO/10684/2009.
- 5 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 Deltamethrin, Imidacloprid and Kresoxim-methyl

laboratory number	analyte											
	deltamethrin assigned value 184 µg/kg				imidacloprid assigned value 139 µg/kg				kresoxim-methyl assigned value 162 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	179	86	20	-0.1	185	90	10	1.6	166	64	10	0.1
002	157.5	92	10	-0.7	113.6	89	10	-0.8	174.6	88	10	0.4
003	#				#				#			
004	174	73.6	50	-0.3	213	92.8	10	2.5	170	102.2	10	0.2
005	220	120	50	1.0	115	92	20	-0.8	100	94	25	-1.8
006	126	107	5	-1.5	#				143	70	5	-0.6
007	163	92	10	-0.5	139	107	10	0.0	144	100	10	-0.5
008	143	85	40	-1.1	#				160	70	40	-0.1
009	144.7		40	-1.0	#				164.4		20	0.1
010	9.94	91.6	100	-4.6	0			-4.6	301.9	112.3	10.00	4.1
011	226	70-110	10	1.1	128	70-110	10	-0.4	181	70-110	10	0.5
012	405	92	10	5.8	#				#			
013	133	98		-1.3	141	83.8		0.1	0		10	-4.8
014	190		10	0.2	90		10	-1.6	140		10	-0.7
015	199			0.4	110			-1.0	205			1.2
016	137	89	5	-1.2	154	90	5	0.5	166	115	5	0.1
017	181.5	87	10	-0.1	99.5	85	10	-1.3	140.5	106	10	-0.6
018	177		50	-0.2	177		20	1.3	134		20	-0.8
019	193	101	5	0.2	135	92	5	-0.1	199	95	5	1.1
020	208	104	10	0.6	120	105	10	-0.6	174	92	10	0.3
021	241	118	10	1.5	150	90	10	0.4	#			
022	101	82	10	-2.2	103	119	10	-1.2	151	76	10	-0.3
023	117	87	10	-1.8	122	80	10	-0.6	116	74	10	-1.4
024	180	98	10	-0.1	137	95	10	-0.1	175	101	10	0.4
025	32	100	10	-4.0	151	100	25	0.4	179	100	10	0.5
026	96.6			-2.3	106.3			-1.1	74.6			-2.6
027	179.3	125	10	-0.1	0		10	-4.6	118.7	72	10	-1.3

LoQ = limit of quantification
z-scores outside |z| >2 are shown in **bold**, see Section 5

= pesticide not analysed for

Table 1 (continued): Results and z-Scores for Deltamethrin, Imidacloprid and Kresoxim-methyl

laboratory number	analyte											
	deltamethrin assigned value 184 µg/kg				imidacloprid assigned value 139 µg/kg				kresoxim-methyl assigned value 162 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
028	212	105	10	0.7	94	93	5	-1.5	125	85	5	-1.1
029	#				151	90	10	0.4	356	90	10	5.7
030	205	90	10	0.6	150	92	10	0.4	170	83	10	0.2
031	138			-1.2	164			0.9	141			-0.6
032	167	92.0	10	-0.4	125	89.9	10	-0.5	138	102.4	10	-0.7
033	89	89	1	-2.5	128	85	1	-0.4	175	93	1	0.4
034	221.04		50.00	1.0	#				#			
035	164	86.2	50	-0.5	108	83.1	10	-1.0	110	84.2	10	-1.5
036	170	84	10	-0.4	#				188	88	10	0.7
037	#				198.1	95	1	2.0	#			
038	239	90.0	10	1.5	131	103.0	10	-0.3	133	99.0	10	-0.9
039	394	98	10	5.5	330	99	10	6.4	301	100	10	4.1
040	195			0.3	131			-0.3	143			-0.6
041	186.87	85	10	0.1	122.17	90	10	-0.6	174.33	95	10	0.3
042	173	102	10	-0.3	160	104	10	0.7	150	107	10	-0.4
043	95	70		-2.3	111	85	10	-0.9	158	80	10	-0.1
044	186.03	93.72	10	0.1	150	96	10	0.4	180	96	10	0.5
045	301	69 M	50	3.1	132	82.7	30	-0.2	151	90.3	30	-0.3
046	#				#				#			
047	147			-1.0	0			-4.6	145			-0.5
048	164	M	10	-0.5	117	M	10	-0.7	148	M	10	-0.4
049	195.00	98	10.0	0.3	101.00	97	10.0	-1.3	140.00	98	10.0	-0.7
050	175	100	30	-0.2	110	100	10	-1.0	170	97	10	0.2
051	143	85	25	-1.1	142	102	10	0.1	125	96	10	-1.1
052	162.00	104	10	-0.6	128.50	84	10	-0.3	108.71	107	10	-1.6
053	205.6	86	10	0.6	155.8	93.7	10	0.6	221.6	102.9	10	1.7

LoQ = limit of quantification
z-scores outside |z| >2 are shown in **bold**, see Section 5

= pesticide not analysed for
M = matrix-based calibration

Table 1 (continued): Results and z-Scores for Deltamethrin, Imidacloprid and Kresoxim-methyl

laboratory number	analyte											
	deltamethrin assigned value 184 µg/kg				imidacloprid assigned value 139 µg/kg				kresoxim-methyl assigned value 162 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
054	211	98	20	0.7	83.31	97	5	-1.9	42	92	5	-3.5
055	289	101	10	2.8	#				162	83	10	0.0
056	180	91	10	-0.1	120	91	20	-0.6	170	89	10	0.2
057	100		10	-2.2	131		10	-0.3	109		10	-1.6
058	159	96	10	-0.7	114	98	10	-0.8	143	92	10	-0.6
059	168.8	91	10	-0.4	176.2	95	10	1.3	149.5	90	10	-0.4
060	170		10	-0.4	135		10	-0.1	136		10	-0.8
061	195	114	0.01	0.3	110	78	0.01	-1.0	170	113	0.01	0.2
062	154	105	10	-0.8	166	93	10	0.9	201	95	10	1.1
063	218	114	10	0.9	250	113	10	3.7	157	71	10	-0.2
064	190	100-120	10	0.2	144	90-100	10	0.2	201	100	10	1.1
065	159		10	-0.7	160		10	0.7	123		10	-1.2
066	0		100	-4.8	#				156.4	41	100	-0.2
067	178	92	10	-0.2	153	96	10	0.5	140	103	10	-0.7
068	321	94	20	3.6	136	94	10	-0.1	152	98	10	-0.3
069	128	77.5	50	-1.5	116	79.5	10	-0.8	0			-4.8
070	150	95	10	-0.9	120	96	10	-0.6	150	99	10	-0.4
071	189.0	88	10	0.1	110.0	98	10	-1.0	120.0	106	10	-1.2
072	155	99	10	-0.8	151	96	10	0.4	146	96	10	-0.5
073	245	90	25	1.6	#				205	90	25	1.2
074	171	97	10	-0.3	142	99	10	0.1	206	99.9	10	1.3
075	160	99.5	5	-0.6	150	90	5	0.4	138	71	20	-0.7
076	0	88		-4.8	190	87	10	1.7	190	91	10	0.8
077	0	88	20	-4.8	200	88	10	2.1	183	93	10	0.6
078	300	105	10	3.1	130	128	10	-0.3	305	110	10	4.2
079	240	92	30	1.5	220	91	30	2.7	193	88	30	0.9

LoQ = limit of quantification
z-scores outside |z| >2 are shown in **bold**, see Section 5

= pesticide not analysed for

Table 2: Results and z-Scores for Monocrotophos, Quinalphos and Quinoxifen

laboratory number	analyte											
	monocrotophos assigned value 330 µg/kg				quinalphos assigned value 39.0 µg/kg				quinoxifen assigned value 250 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	163	63	50	-2.7	0		20	-4.5	#			
002	285.5		10	-0.7	51.2	83	10	1.4	246.7	98	10	-0.1
003	#				0		10	-4.5	225	90	10	-0.5
004	315	87.2	10	-0.2	36	91.9	10	-0.4	212	93.4	10	-0.8
005	265	98	25	-1.0	26	71	20	-1.5	160	96	30	-1.8
006	0			-5.3	#				#			
007	327	110	10	0.0	33	103	10	-0.7	216	82	10	-0.7
008	#				39	100	30	0.0	227	80	20	-0.5
009	#				#				#			
010	#				#				319.80	80.80	10.00	1.4
011	388	70-110	10	0.9	44	70-110	10	0.6	303	70-110	10	1.1
012	263	109	20	-1.1	52	96	20	1.5	#			
013	255	108		-1.2	#				158	74		-1.9
014	320		10	-0.2	30		10	-1.1	170		10	-1.6
015	225			-1.7	0			-4.5	0			-5.1
016	333		5	0.1	32	116	5	-0.8	255	114	5	0.1
017	236	106	10	-1.5	#				206	77	10	-0.9
018	#				32		10	-0.8	247		10	-0.1
019	310	86	5	-0.3	38	95	5	-0.1	289	101	5	0.8
020	284	127	10	-0.7	34	130	10	-0.6	230	114	10	-0.4
021	416	96	10	1.4	53	92	10	1.6	#			
022	303	100	10	-0.4	28	97	10	-1.3	#			
023	350	101	10	0.3	41	97	10	0.2	#			
024	324	95	10	-0.1	56	101	10	2.0	260	97	10	0.2
025	450	100	25	1.9	39	100	10	0.0	254	100	10	0.1
026	#				#				#			
027	167.2	83	10	-2.6	30.4	73	10	-1.0	#			

LoQ = limit of quantification
z-scores outside $|z| > 2$ are shown in **bold**, see Section 5

= pesticide not analysed for

Table 2 (continued): Results and z-Scores for Monocrotophos, Quinalphos and Quinoxifen

laboratory number	analyte											
	monocrotophos assigned value 330 µg/kg				quinalphos assigned value 39.0 µg/kg				quinoxifen assigned value 250 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
028	321	92	5	-0.1	28	81	5	-1.3	#			
029	336	90	10	0.1	29	90	10	-1.2	254	90	10	0.1
030	340	89	10	0.2	50	88	10	1.3	#			
031	198			-2.1	30			-1.1	280			0.6
032	252	91.3	10	-1.2	36	85.0	10	-0.4	186	92	10	-1.3
033	#				#				265	91	1	0.3
034	358.79	102.33	200.00	0.5	#				#			
035	341	96.6	10	0.2	20	82	10	-2.2	#			
036	0		10	-5.3	46	100	10	0.8	250	92	10	0.0
037	390.5	98	5	1.0	#				#			
038	321	100.0	10	-0.1	84	90.0	10	5.2	#			
039	767	95	10	7.0	66	98	10	3.1	450	97	10	4.1
040	322			-0.1	29			-1.2	201			-1.0
041	355.67	90	10	0.4	41.93	95	10	0.3	#			
042	325	95	10	-0.1	36	101	10	-0.4	#			
043	193	80	10	-2.2	50	80	10	1.3	#			
044	350	99	10	0.3	50	99	10	1.3	#			
045	312	78.7	30	-0.3	39	91.4	30	0.0	139	67.3 M	30	-2.2
046	#				#				#			
047	157			-2.8	90			5.9	236			-0.3
048	261	M	10	-1.1	32.4	M	10	-0.8	#			
049	301.00	97	10.0	-0.5	35.00	96	10.0	-0.5	#			
050	320	100	10	-0.2	45	105	10	0.7	290	101	10	0.8
051	375	94	10	0.7	28	110	10	-1.3	217	95	10	-0.7
052	260.00	84	10	-1.1	33.50	88	10	-0.6	#			
053	380.4	102	10	0.8	59.4	100.6	10	2.4	#			

LoQ = limit of quantification
z-scores outside |z| >2 are shown in **bold**, see Section 5

= pesticide not analysed for
M = matrix-based calibration

Table 2 (continued): Results and z-Scores for Monocrotophos, Quinalphos and Quinoxifen

laboratory number	analyte											
	monocrotophos assigned value 330 µg/kg				quinalphos assigned value 39.0 µg/kg				quinoxifen assigned value 250 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
054	445	97	5	1.9	6.88	96	5	-3.7	#			
055	0		10	-5.3	33.1	87	10	-0.7	#			
056	326	105	17	-0.1	45	99.5	15	0.7	266	96	15	0.3
057	190		10	-2.2	43.2		10	0.5	199		10	-1.0
058	293	98	10	-0.6	26	92	10	-1.5	235	86	10	-0.3
059	329.8	103	10	0.0	31.5	88	10	-0.9	246	91	10	-0.1
060	#				#				#			
061	221	90	0.01	-1.7	38	106	0.01	-0.1	289	112	0.01	0.8
062	400	93	10	1.1	30	92	10	-1.1	317	82	10	1.4
063	450	109	10	1.9	38	102	10	-0.1	258	91	10	0.2
064	323	80-90	10	-0.1	42	100-120	10	0.3	152	100	10	-2.0
065	0		10	-5.3	24		10	-1.8	220		10	-0.6
066	0		100	-5.3	99.6	11	100	7.0	192	6	100	-1.2
067	347	94	10	0.3	33	92	10	-0.7	210	86	10	-0.8
068	263	74	10	-1.1	0		10	-4.5	292	96	10	0.9
069	318	85.4	10	-0.2	36	83.9	10	-0.4	262	92.3	10	0.3
070	410	90	10	1.3	29	99	10	-1.2	170	85	10	-1.6
071	366.0	94	10	0.6	29.0	92	10	-1.2	193.0	100	10	-1.1
072	336	96	10	0.1	33	99	10	-0.7	251	99	10	0.0
073	0		25	-5.3	65	80	25	3.0	#			
074	352	96	10	0.4	39	105	10	0.0	217	93	10	-0.7
075	#				#				#			
076	145	90	10	-3.0	40	89	20	0.1	275	91	10	0.5
077	200	91	10	-2.1	35	89	10	-0.5	290	91	10	0.8
078	735	118	10	6.5	60	103	10	2.4	430	95	10	3.7
079	310	92	30	-0.3	45	90	30	0.7	#			

LoQ = limit of quantification
z-scores outside |z| >2 are shown in **bold**, see Section 5

= pesticide not analysed for

Table 3: Participants' Comments

participant number	comments
011	We also found Fenpropimorph at about 4µg/kg which is below our LOQ
050	we found fenpropimorph below 10 µg/kg, our limit of quatification
066	% recovery based on solvent standards with PTV , NOT MATRIX MATCHED!, RESULT BASED ON SPIKED BLANK
070	Traces: Fenpropimorph, Spinosad, Spiroxamine
072	Fenpropimorph : < LOQ

comments are as submitted by participants

Table 4: Additional Pesticide Residues Reported

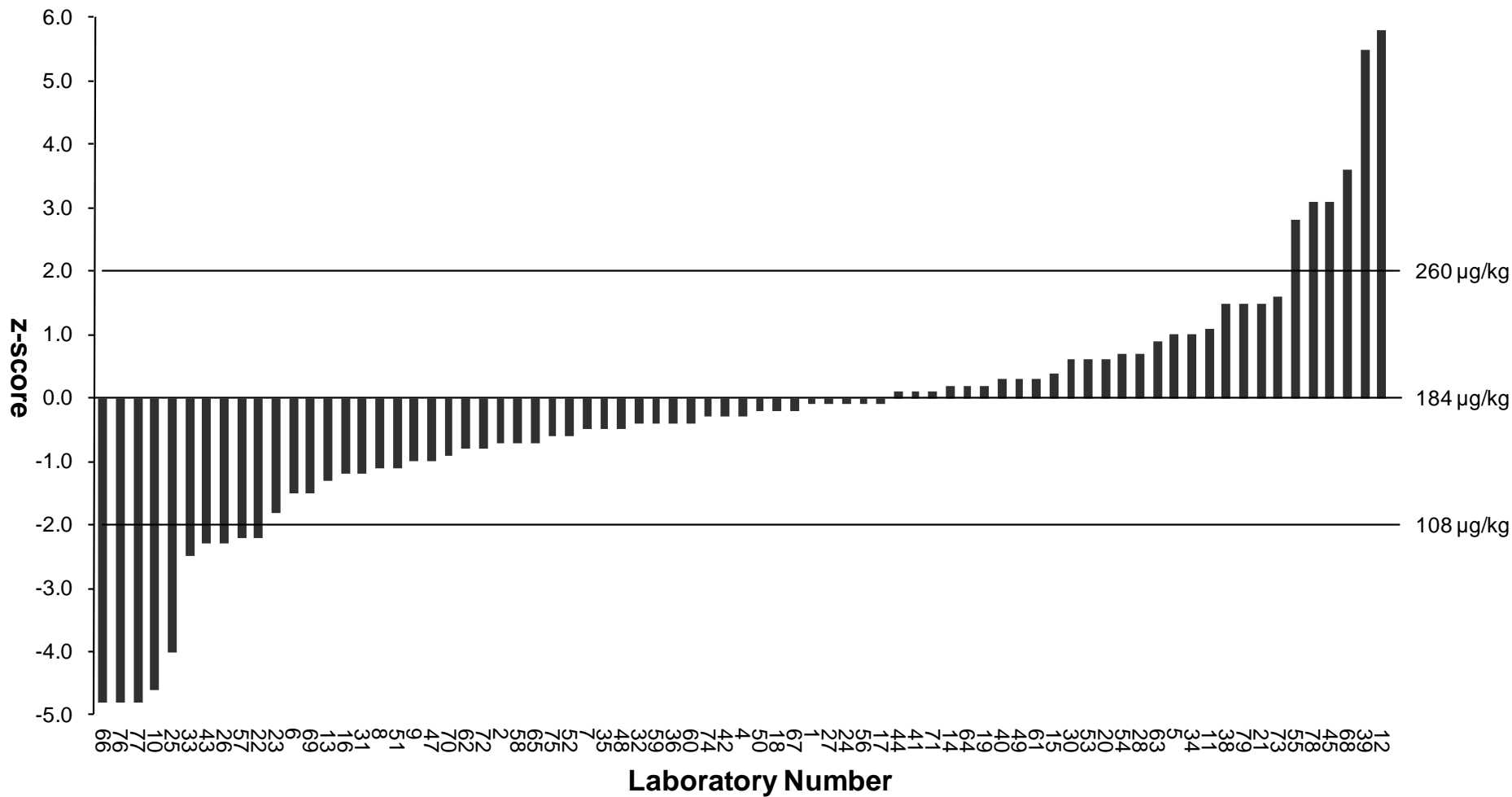
laboratory number	pesticide residue >30 µg/kg	result µg/kg	recovery %	LoQ µg/kg
020	buprofezin	159	96	10
034	pp'-DDT	43.84		10.00
037	pp'-DDT	41.1	—	—

Table 5: Assigned Values and Standard Deviations for Proficiency

analyte	assigned value, x_a , µg/kg			standard deviation for proficiency, µg/kg	
	data points, n	robust mean, \hat{X}	uncertainty, u	derived from	σ_p
deltamethrin	54	184	5.96	Horwitz Equation [5]	37.9
imidacloprid	53	139	4.24	Horwitz Equation [5]	29.9
kresoxim-methyl	57	162	4.53	Horwitz Equation [5]	34.2
monocrotophos	51	330	9.33	Horwitz Equation [5]	62.3
quinalphos	53	39.0	1.48	Horwitz Equation [5]	8.59
quinoxifen	38	250	7.67	Horwitz Equation [5]	49.2

Table 6: 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$
deltamethrin	59	75	79
imidacloprid	60	68	88
kresoxim-methyl	65	73	89
monocrotophos	53	69	77
quinalphos	54	67	81
quinoxifen	45	49	92



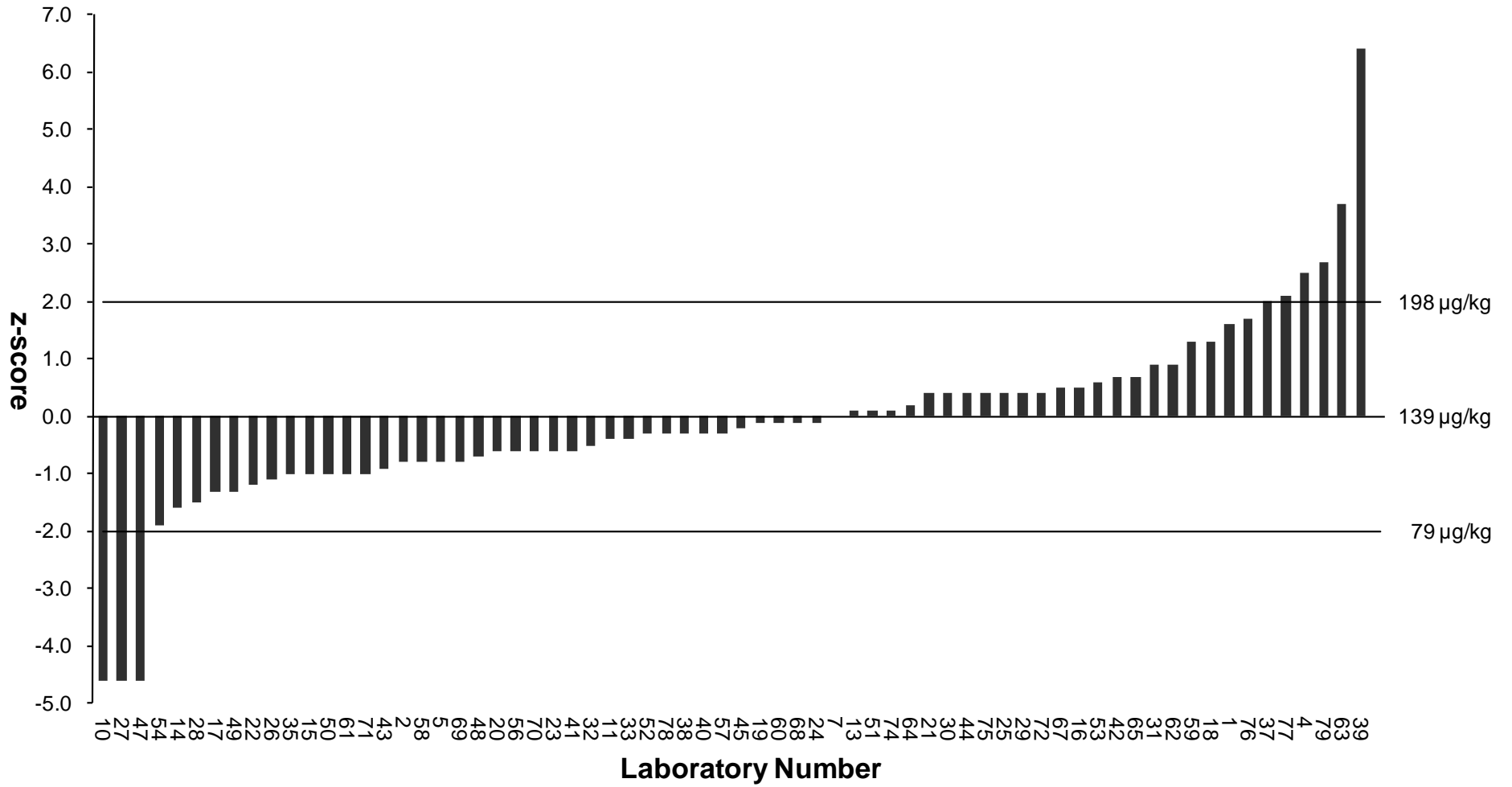
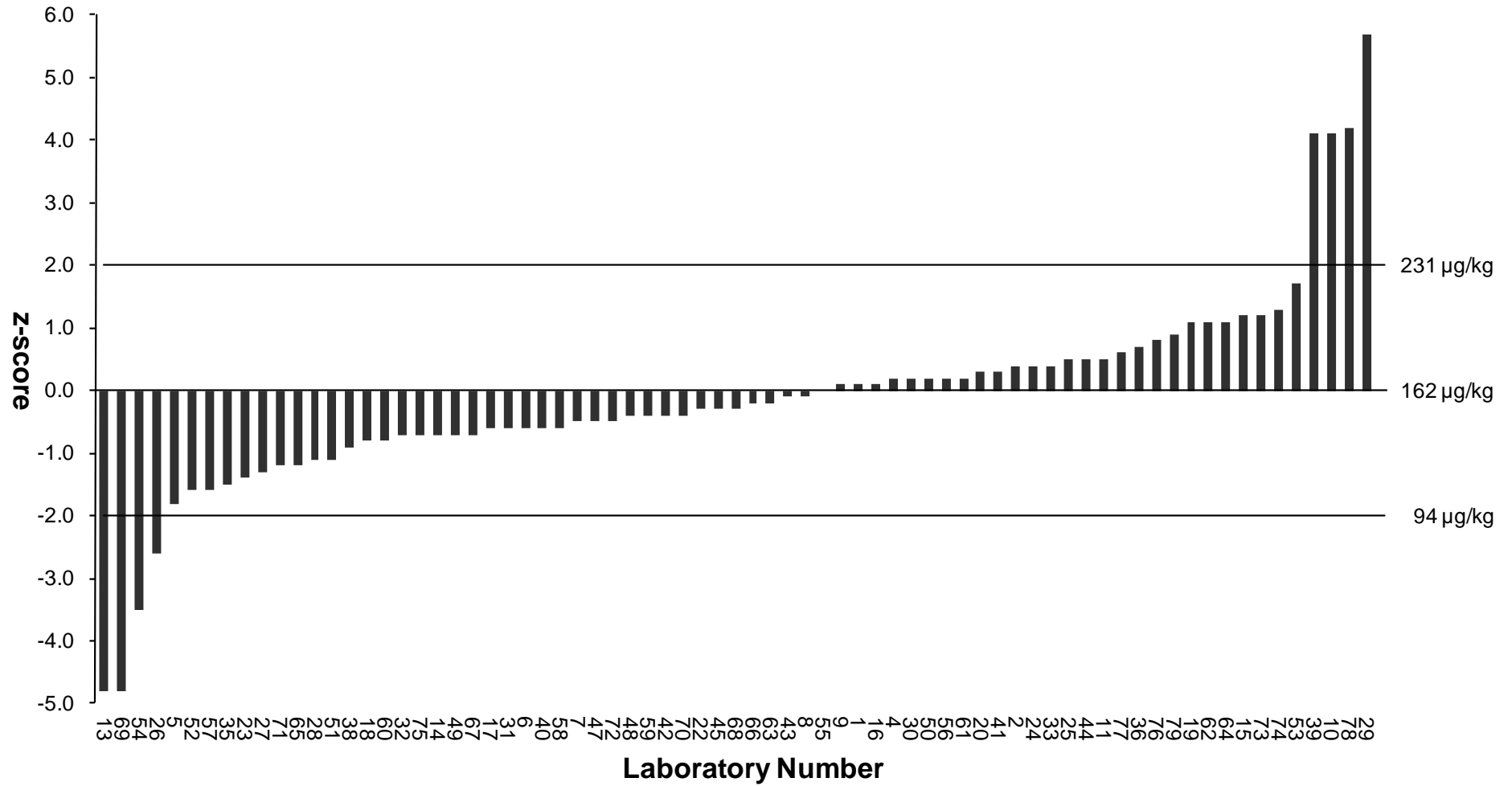


Figure 2: z-Scores for Imidacloprid



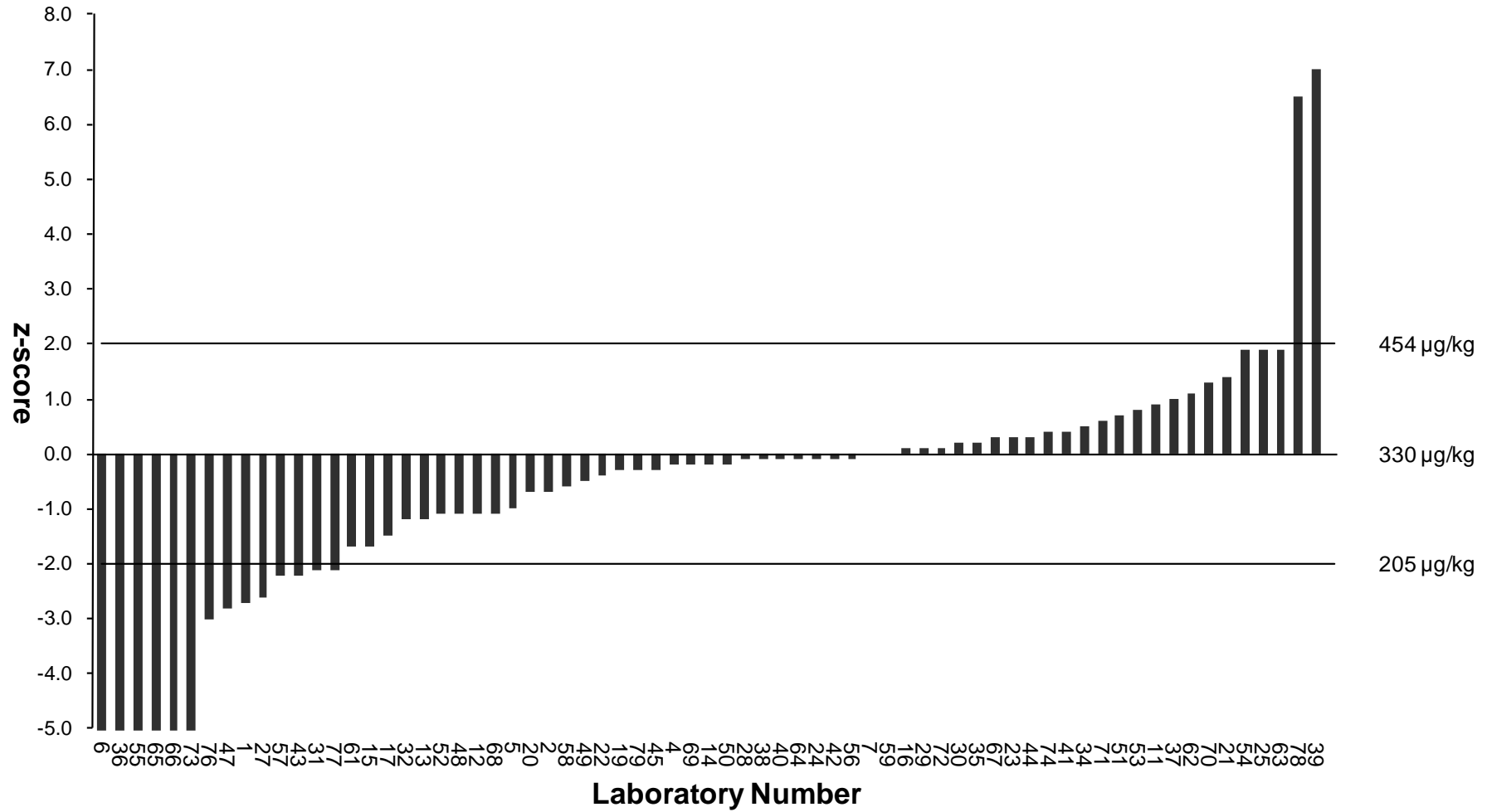


Figure 4: z-Scores for Monocrotophos

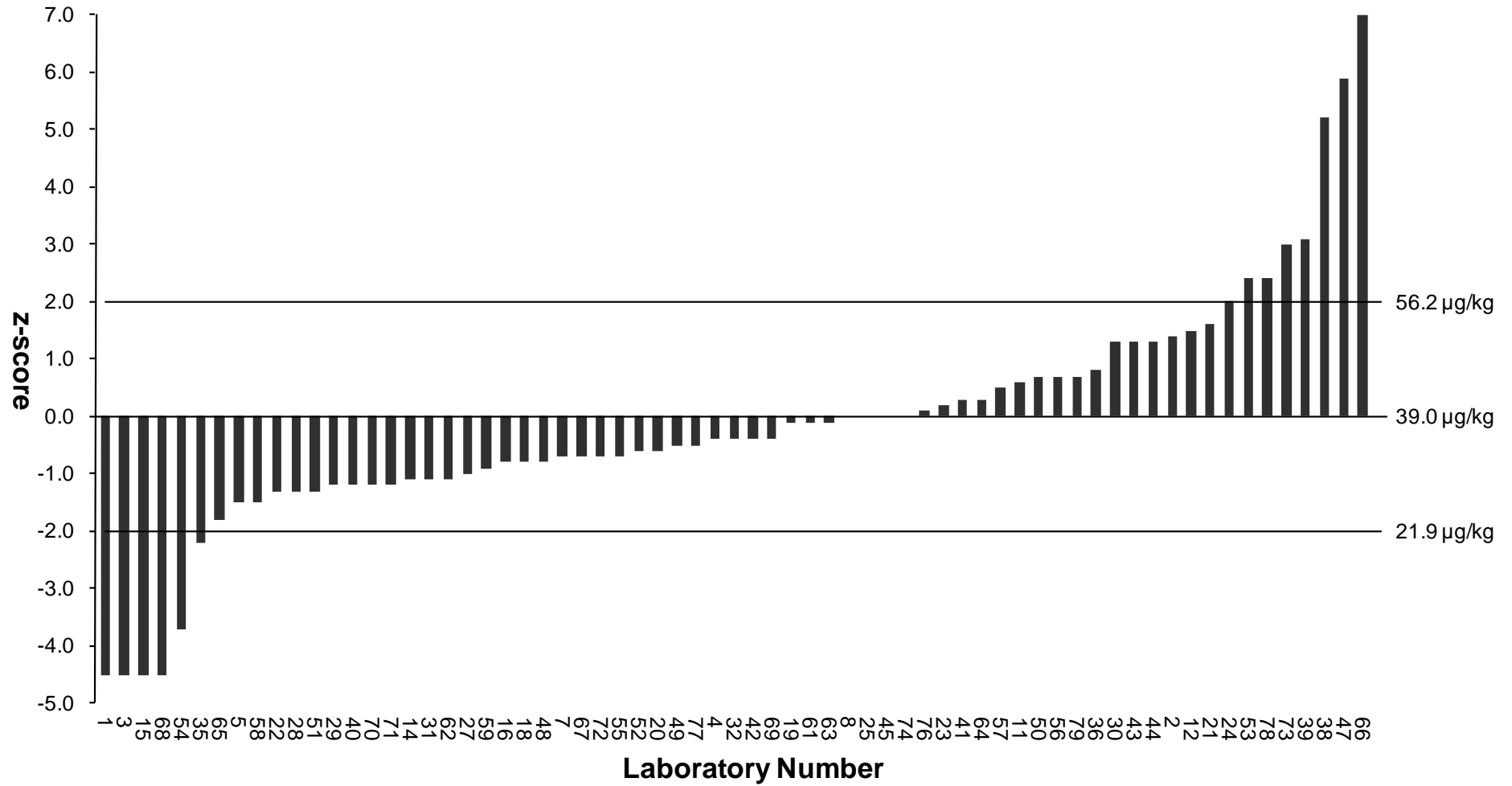


Figure 5: z-Scores for Quinalphos

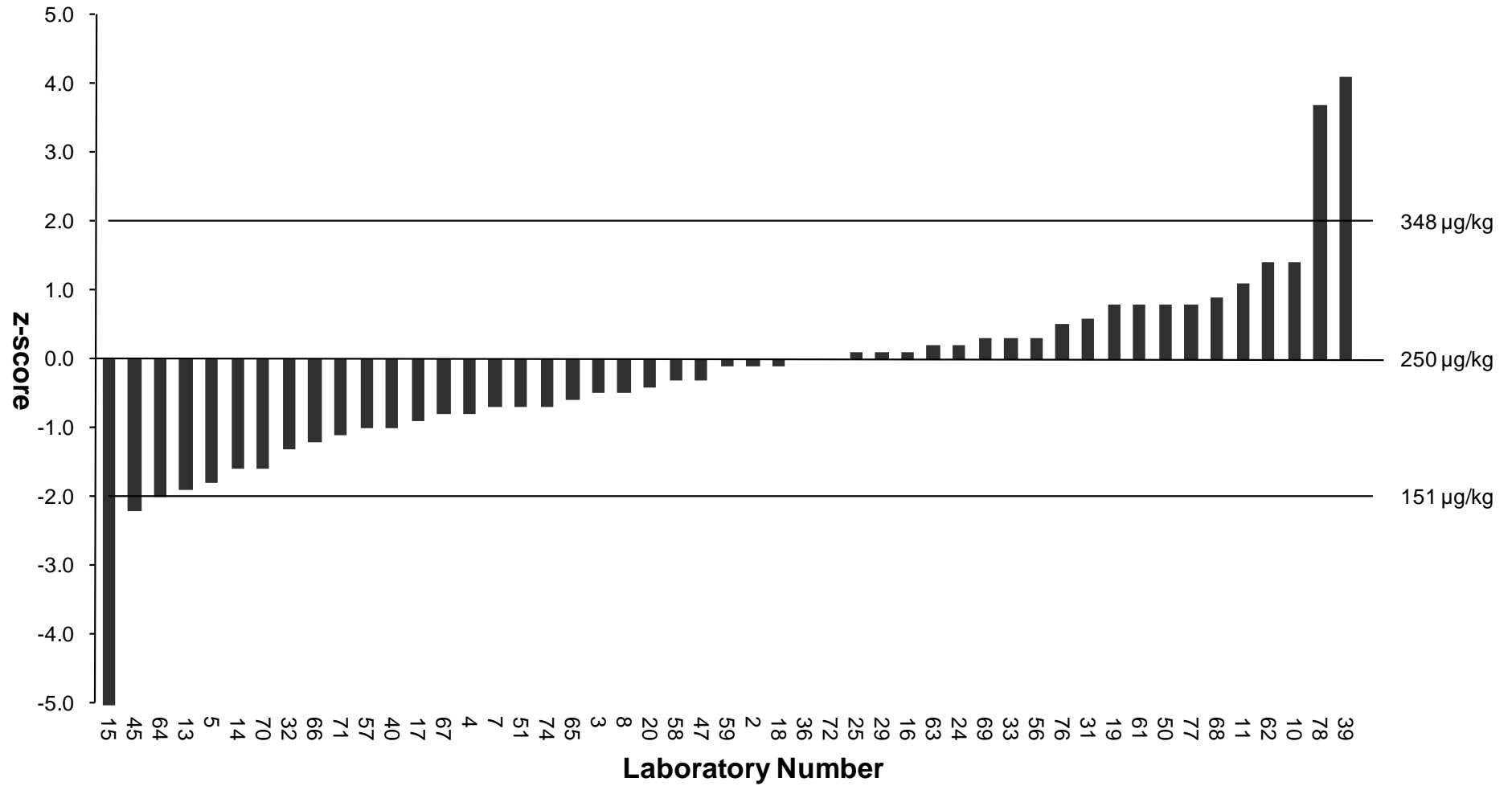


Figure 6: z-Scores for Quinoxifen

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.

Deltamethrin

Accredited Method Used	laboratory number
yes	002 004 005 006 008 009 010 011 012 013 014 015 016 018 022 023 024 025 026 027 028 030 031 032 033 034 035 038 039 041 042 043 044 048 049 050 051 052 053 054 056 057 059 061 062 063 064 065 067 068 069 070 071 072 074 078 079
no	001 017 021 036 045 075

Sample Weight (g)	laboratory number
≥1 - <2	006 052
≥2 - <5	011 061
≥5 - <10	023 024 043 045 053 059 070 078
≥10 - <25	001 004 005 008 009 010 012 013 014 015 017 018 021 022 025 026 027 028 030 031 032 033 035 036 038 039 041 042 044 047 048 049 050 051 054 056 057 063 064 065 067 068 069 071 072 074 075 079
≥25 - <50	002 062
≥50	016 034

Extraction Solvent Components	laboratory number
acetic acid	015 036 067 072 075
acetone	001 004 006 008 009 012 013 016 050 064 074
acetonitrile	005 010 011 015 017 021 024 026 032 033 034 036 039 045 056 061 065 067 068 070 071 072 075
cyclohexane	016 025 064
dichloromethane	001 004 008 050 074
ethyl acetate	002 014 016 018 022 023 025 027 028 030 031 035 038 041 042 043 044 048 049 051 052 053 054 057 059 062 063 064 069 079
hexane	006
petroleum ether/spirit	001 004 008 050 074
sodium sulfate	051
water	013

Extraction Technique Used	laboratory number
ASE	025 057
cold solvent extraction at atmospheric pressure	001 002 004 005 006 008 010 011 012 013 014 017 018 021 022 023 024 027 028 030 031 032 033 035 039 042 044 045 048 049 050 051 053 054 059 062 064 065 067 068 069 070 071 074
solid liquid extraction	043
solvent extraction	034
solvent extraction and partitioning with sodium sulphate and sodium acetate	036
solvent extraction at increased pressure/PLE	015

Extraction pH Adjusted	laboratory number
yes	011 016 017 032 061 062 067 069 070 072 075 078
no	001 002 004 005 006 008 009 010 012 013 014 015 018 021 022 023 024 025 027 028 030 031 033 034 035 036 038 039 041 042 043 044 045 048 049 050 051 052 053 054 056 057 059 063 064 065 068 071 074 079

Sample Clean-up Technique	laboratory number
alumina column	014
C18	005
extraction	015 053 069 071
Extrelut	044
filter	010 074
GPC/HPGPC	004 013 016 025 057
liquid/liquid extraction	006 041 052
liquid-liquid partitioning	038
NH ₂ /aminopropyl column	011
primary secondary amine	022 043
PSA	028
PSA cleanup	035
QuEChERS	056 075
solid phase extraction (SPE) (column/cartridge)	012 034 059
solid phase extraction (SPE) (dispersive)	010 011 017 021 024 027 030 032 033 036 038 039 041 042 045 048 049 052 054 061 065 067 068 070 072 075 079
none	001 002 008 018 023 050 051 062 063

SPE Sorbent Type	laboratory number
alumina	014
C18	005 011 012 078 079
Envicarb/GCB	059
Florisil	034
PSA	010 021 024 027 028 030 033 036 038 039 041 042 043 044 045 048 049 052 053 054 065 067 070 072 075
PSA + MgSO ₄	017
PSA/C18	032
silica	016

Certified Standards Used	laboratory number
yes	001 002 004 005 006 008 009 010 011 012 014 015 016 017 018 021 022 023 024 025 027 028 030 031 032 033 034 035 036 038 039 041 042 043 044 045 047 048 049 050 051 052 053 054 056 057 059 061 062 063 064 065 067 068 069 070 072 074 075 078 079
no	013

MS Confirmation	laboratory number
yes	001 002 004 005 006 008 009 010 011 012 013 014 015 016 017 018 022 023 024 025 027 028 030 031 032 033 034 035 036 038 039 041 042 043 044 045 047 048 049 050 051 052 053 054 056 057 059 061 062 063 064 065 067 068 069 070 072 074 075 078 079
no	021

Calibrations	laboratory number
matrix-matched	001 002 004 005 006 008 010 011 014 015 016 017 025 026 028 030 031 032 033 035 036 038 039 041 042 043 044 048 049 050 051 052 057 059 061 062 063 067 069 071 072 075 078
multi-level	001 004 008 009 014 016 017 023 025 027 031 034 035 036 038 045 051 054 059 061 063 064 065 068 074
single-level	006 013 016 021 032 033 056 070
solvent	024 027 053 068 074
standard addition	012 018 022

Source of Standards	laboratory number
Accu Standard	044 054 065 079
Chemservice, Restek and Supelco	075
Dr Ehrenstorfer	001 002 004 005 006 008 009 010 011 013 015 016 018 022 023 024 025 028 031 032 033 035 036 038 039 041 044 047 048 050 051 052 056 057 059 061 062 063 064 067 068 069 070 072 074
Fluka	011 012 042 053 063 074
Merck	071
Restek	034
Sigma/Aldrich	006 010 014 027 030 039 043 044 045 049 063 074
Supelco	044 063 074
Ultra Scientific	021
various	078

Is Quoted Percentage Recovery Measured in Same Analytical Batch as Test Material?	laboratory number
yes	001 004 008 009 011 012 013 016 017 018 021 022 023 025 027 028 030 032 033 036 038 039 041 042 044 045 047 048 049 050 051 052 053 056 057 059 062 063 064 067 069 071 072 074 075 079
no	002 006 010 014 015 024 031 034 035 043 054 061 065 070 078

If Measured in the same Batch, When was the Spike Added?	laboratory number
prior to extraction	001 004 006 008 009 011 012 013 014 016 017 018 021 022 023 024 025 027 028 030 032 033 036 038 039 041 042 044 045 048 049 050 052 053 056 057 059 061 062 063 064 065 067 069 071 072 074 075 078 079
prior to clean up	051

Ratio of Isomers	laboratory number
1:4	038

Level of Spike (µg/kg)	laboratory number
<25	001 017 018 036 044 048 049 052 059 063 065 075
≥25 - <50	004 013 018 027 038 048 057 059 062 079
≥50 - <100	005 016 017 018 022 023 024 025 028 030 032 035 048 053 063 072 074
≥100 - <150	011 025 033 039 042 045 048 051 056 063 064
≥150 - <200	008 025 048 050 067 069
≥200 - <250	018 025 048 064
≥250 - <300	021 025
≥300 - <400	025 041 064
≥400 - <500	025
≥500	012 018 025 061

Composition of Blank Commodity used for Spiking	laboratory number
grape blank provided	004 006 008 011 013 016 017 018 021 022 024 027 030 032 033 035 038 041 042 044 045 048 049 050 051 052 053 057 059 061 063 065 067 072 075 078 079
grape test material provided	005 012 025 028 039 056 074
grape	001
tomato	014
in house blank	023 036 062 068

GC Column Type	laboratory number
capillary	001 002 004 006 008 009 011 012 013 014 015 016 017 018 021 022 024 025 027 028 030 031 032 033 034 035 036 038 041 042 043 044 048 049 050 051 052 053 054 056 057 059 061 062 063 064 065 068 069 070 071 072 075 078 079
narrowbore	023 045 074
widebore	005

GC Column Packing	laboratory number
14% cyanopropylphenyl 86%methyl polysiloxane	006
65% methyl 35% phenyl polysiloxane	012 062

GC Column Packing (continued)	laboratory number
95% methyl 5% phenyl polysiloxane	001 002 004 005 006 008 011 014 017 018 021 022 023 024 025 027 028 030 032 033 035 036 038 041 042 043 044 048 049 050 051 052 054 056 057 059 061 064 065 068 069 070 072 074 075 078 079
5% phenyl methyl siloxane	034
5% phenyl-polysiloxane	009
5%phenyl-95%dimethyl polysiloxane	063
100% methyl polysiloxane	016
CP-sil 8 CB	031
Zebron ZB-XLB	053

GC Injection Volume (µL)	laboratory number
<1	012 032
≥1 - <2	005 006 011 015 016 018 021 023 024 025 027 030 033 034 045 052 053 054 057 059 061 064 065 069 070 075 078 079
≥2 - <5	004 008 009 014 017 028 035 038 041 042 044 048 050 062
≥5 - <10	002 049 056 063 068 072 074
≥10	001 013 022 031 036 043 051

GC Injection Mode	laboratory number
large volume	013
PTV	001 002 011 014 021 022 027 028 031 035 036 041 043 049 050 051 056 064 068 072 074
pulsed splitless	034
split	012 053 069
splitless	004 006 008 009 015 016 017 018 023 024 025 030 032 033 042 044 045 048 052 054 057 059 061 062 063 065 070 075 078 079

GC Detector	laboratory number
ECD	005 008 009 012 021 034 053 074 075 079
HR-MS	069
ITD	001 031 050 068 074

GC Detector (continued)	laboratory number
MS	008 013 014 015 016 018 022 023 024 032 033 042 043 045 049 051 057 059 062 064 070 071 072 078
MS-MS	001 002 004 011 017 025 027 028 030 035 036 038 041 044 048 052 054 056 061 063 065 074

HPLC Column Packing	laboratory number
C18	010 013 015 024 030 032 033 039 041 043 044 056 062 067 069 074
none	075

HPLC Guard Column Used	laboratory number
yes	010 024 030 033 039 041 043 044 056 062 069 071 074
no	013 015 016 022 025 027 028 032 035 051 065 067 075

Mobile Phase Programme	laboratory number
isocratic	028 035
gradient	010 013 015 016 022 024 026 027 030 032 033 039 041 043 044 056 062 065 067 069 071 074 075 079

Mobile Phase Components	laboratory number
acetate	056
acetonitrile	013 024 030 032 033 062
ammonium formate	041 044 067
formic acid	010 030
methanol	010 015 026 033 039 041 043 044 067 069 071 074
water	010 013 015 030 032 033 039 067 069 071
none	075

HPLC Column Temperature (°C)	laboratory number
ambient	013 033 039 041 043 044 056 062
>ambient - <50	010 015 024 030 032 069 071 074
≥50	067

HPLC Injection Volume (µL)	laboratory number
<5	013 024 032 062
≥5 - <10	043 056 074
≥10 - <25	010 015 030 033 039 041 044 067 069

Mobile Phase Flow Rate (mL/min)	laboratory number
<0.25	056 069
≥0.25 - <0.75	010 013 015 024 030 032 033 039 043 044 067 074
≥0.75 - <1.25	041 062

HPLC Detector Type	laboratory number
MS-MS	010 013 015 016 024 030 032 033 039 041 043 044 056 062 067 069 071 074

Imidacloprid

Accredited Method Used	laboratory number
yes	001 002 004 005 011 013 015 016 018 022 023 024 025 028 030 031 032 033 035 037 038 039 041 042 043 044 048 049 050 051 052 053 054 056 057 059 062 063 065 067 069 070 071 072 074 077 078 079
no	017 021 045 068 075

Sample Weight (g)	laboratory number
≥2 - <5	011
≥5 - <10	018 023 024 028 030 037 043 045 053 059 070 078

Sample Weight (g) (continued)	laboratory number
≥10 - <25	001 005 013 015 017 021 022 025 031 032 033 035 038 039 041 042 044 048 049 050 051 052 054 056 057 062 063 065 067 068 069 071 072 074 075 079
≥25 - <50	002
≥50	016

Extraction Solvent Components	laboratory number
acetic acid	004 015 067 072 075
acetone	013 016 050 074
acetonitrile	001 004 005 011 015 017 018 021 024 032 033 037 039 045 056 062 063 065 067 068 070 071 072 075 079
cyclohexane	016
dichloromethane	050 074
ethyl acetate	002 016 022 025 028 030 035 038 041 042 043 044 048 049 051 052 053 054 057 059 069
methanol	022 023
petroleum ether/spirit	050 074
sodium sulfate	051
water	013 022 023

Extraction Technique Used	laboratory number
ASE	057
cold solvent extraction at atmospheric pressure	001 002 004 005 011 013 017 018 021 022 024 028 030 032 033 035 037 039 042 044 045 048 049 050 051 053 054 059 062 065 067 068 069 070 071 074
solid liquid extraction	043
solvent extraction at increased pressure/PLE	015

Extraction pH Adjusted	laboratory number
yes	001 004 011 016 017 025 032 037 059 067 069 070 072 075 078
no	002 005 013 015 018 021 022 023 024 028 030 031 033 035 038 039 041 042 043 044 045 048 049 050 051 052 053 054 056 057 062 063 065 068 071 074 077 079

Sample Clean-up Technique	laboratory number
C18	005
DisQuE Extraction Tube	025
extraction	015 069 071
Extrelut	044
filter	074
GPC/HPGPC	013 016
liquid/liquid extraction	037 038 041 052 053 063
NH ₂ /aminopropyl column	011
primary secondary amine	022
PSA	028
PSA cleanup	035
QuEChERS	056 079
solid phase extraction (SPE) (column/cartridge)	059
solid phase extraction (SPE) (dispersive)	001 004 011 017 018 021 024 032 033 038 039 041 045 048 049 052 054 065 067 068 070 072 075 077 079
none	002 023 042 050 051 062 063

SPE Sorbent Type	laboratory number
C18	005 011 078 079
Envicarb/GCB	059
PSA	001 004 018 021 024 028 030 033 038 039 041 043 044 045 048 049 053 054 065 067 072 075
PSA & GCB	052
PSA + MgSO ₄	017
PSA/C18	032
silica	016

Certified Standards Used	laboratory number
yes	001 002 004 005 011 015 016 017 018 021 022 023 024 025 028 030 031 032 033 035 037 038 039 041 042 043 044 045 048 049 050 051 052 053 054 056 057 059 062 063 065 067 068 069 070 072 074 075 077 078 079
no	013

MS Confirmation	laboratory number
yes	001 002 004 005 011 013 015 016 017 018 022 023 024 025 028 030 031 032 033 035 037 038 039 041 042 043 044 045 048 049 050 051 052 053 054 056 057 059 062 063 065 067 068 069 070 072 074 075 077 078 079
no	021

Calibrations	laboratory number
matrix-matched	001 002 004 005 011 015 016 017 018 025 028 030 031 032 033 035 037 038 039 041 042 043 044 045 048 049 050 051 052 057 059 062 063 067 069 071 072 075 078 079
multi-level	001 004 016 017 023 025 031 035 038 051 054 059 063 065 068 074
single-level	013 016 021 032 033 056 070
solvent	024 053 068 074
standard addition	022

Source of Standards	laboratory number
Accu Standard	044 054 065 079
Chemservices	075
Dr Ehrenstorfer	001 002 004 005 011 013 015 016 017 018 021 022 023 024 028 031 032 033 037 038 039 041 044 048 050 051 052 056 057 059 062 063 067 068 069 070 072 074
Fluka	011 025 030 042 063 074
Merck	071
Sigma/Aldrich	030 035 039 043 044 045 049 053 063 074
Supelco	044 063 074 075
various	078

Is Quoted Percentage Recovery Measured in Same Analytical Batch as Test Material?	laboratory number
yes	001 004 011 013 016 017 018 021 022 023 025 028 030 032 033 037 038 039 041 042 044 045 048 049 050 051 052 053 056 057 059 062 063 067 069 071 072 074 075 079
no	002 015 024 031 035 043 054 065 070 078

If Measured in the same Batch, When was the Spike Added?	laboratory number
prior to extraction	001 004 011 013 016 017 018 021 022 023 024 025 028 030 032 033 037 038 039 041 042 044 045 048 049 050 052 053 056 057 059 062 063 065 067 069 071 072 074 075 078 079
prior to clean up	051

Level of Spike (µg/kg)	laboratory number
<25	001 017 018 021 035 037 044 048 049 052 059 063 065 075
≥25 - <50	013 018 038 048 057 059 062 079
≥50 - <100	004 005 016 018 022 023 024 028 030 032 048 053 063 072 074
≥100 - <150	011 033 039 042 045 048 050 051 056
≥150 - <200	025 048 067 069
≥200 - <250	018 025 048
≥250 - <300	025
≥300 - <400	025 041
≥400 - <500	025
≥500	018 025

Composition of Blank Commodity used for Spiking	laboratory number
grape blank provided	004 011 013 016 017 018 021 022 024 030 032 033 035 038 041 042 044 045 048 049 050 051 052 053 057 059 063 065 067 072 075 077 078 079
grape test material provided	005 025 028 039 056 074
grape	001
in house blank	023 062 068

GC Column Type	laboratory number
capillary	011 013 015 016 024 032 037 041 043 044 056 069 078
narrowbore	074

GC Column Packing	laboratory number
100% methyl polysiloxane	016
95% methyl 5% phenyl polysiloxane	005 011 024 032 041 043 044 056 069 074 078
none	075

GC Injection Volume (µL)	laboratory number
<1	032
≥1 - <2	011 015 016 024 069 078
≥2 - <5	041 044
≥5 - <10	056 074
≥10	013 043

GC Injection Mode	laboratory number
large volume	013
PTV	011 041 043 056 068 074
split	069
splitless	015 016 024 032 044 078

GC Detector	laboratory number
ECD	074
HR-MS	069
ITD	074
MS	013 015 016 024 032 043 078
MS-MS	011 041 044 056 074

HPLC Column Packing	laboratory number
C18	001 002 004 005 013 015 017 018 021 022 023 024 028 030 031 032 033 035 037 038 039 041 042 043 044 045 048 049 050 051 052 053 054 056 057 059 062 063 065 067 068 069 070 072 074 075 077
C18 X-Terra type	011
endcapped	021 042 068
UPLC HSS T3 (Waters)	025

HPLC Guard Column Used	laboratory number
yes	001 002 017 018 021 023 024 025 030 031 033 035 038 039 041 042 043 044 045 048 049 051 056 057 059 062 068 069 070 071 072 074 075 079
no	004 005 011 013 015 016 022 028 032 037 050 052 053 054 063 065 067 077

Mobile Phase Programme	laboratory number
isocratic	042 054
gradient	001 002 004 005 011 013 015 016 017 018 021 022 023 024 025 028 030 031 032 033 035 037 038 039 041 043 044 045 048 049 050 051 052 053 056 057 059 062 063 065 067 068 069 070 071 072 074 075 077 079

Mobile Phase Components	laboratory number
acetate	011 021 056
acetic acid	011 021
acetonitrile	002 013 021 024 030 031 032 033 037 054 059 062
ammonium formate	001 002 005 017 031 035 041 044 051
ammonium formate buffer	052
formic acid	001 002 025 030 031 048 057 075
methanol	001 002 004 011 015 017 018 022 023 025 028 031 033 035 038 039 041 042 043 044 045 048 049 050 051 052 053 054 057 063 065 067 068 069 070 071 072 074 075
water	001 004 011 013 015 017 018 021 022 023 025 030 032 033 035 038 039 042 045 048 049 050 051 053 054 059 063 067 068 069 070 071 072 075

HPLC Column Temperature (°C)	laboratory number
ambient	013 023 028 033 037 038 039 041 043 044 049 056 065
>ambient - <50	001 002 004 005 011 015 017 018 021 022 024 025 030 031 032 035 042 045 048 051 052 053 054 057 059 062 063 068 069 070 071 072 074 075 077 079
≥50	050 067

HPLC Injection Volume (µL)	laboratory number
<5	013 024 032 050 072 079
≥5 - <10	011 018 043 053 056 057 068 074 077
≥10 - <25	001 002 004 005 015 017 021 022 023 025 028 030 031 033 035 038 039 041 042 044 048 049 051 052 054 059 062 063 065 067 069 070 075
≥50 - <100	037 045

Mobile Phase Flow Rate (mL/min)	laboratory number
<0.25	001 002 017 018 023 031 054 056 059 068 069
≥0.25 - <0.75	005 011 013 015 021 024 025 028 030 032 033 035 037 039 042 043 044 045 048 050 051 052 053 063 065 067 070 072 074 075 079
≥0.75 - <1.25	004 022 038 041 049 062 077

HPLC Pre Column Derivatisation	laboratory number
none	001 002 004 016 022 023 024 025 028 030 033 035 037 038 039 042 043 044 048 050 051 052 053 054 056 062 067 069 074 075 079

HPLC Post Column Derivatisation	laboratory number
none	001 002 004 016 022 023 024 025 028 030 033 035 037 038 039 042 043 044 048 050 051 052 053 054 056 062 067 069 074 075 079

HPLC Detector Type	laboratory number
MS-MS	001 002 004 005 013 015 016 017 018 021 022 023 024 025 028 030 031 032 033 035 037 038 039 041 042 043 044 045 048 050 051 052 053 054 056 057 059 062 063 067 068 069 070 071 072 074 075 077 079
UV	049

Kresoxim-methyl

Accredited Method Used	laboratory number
yes	001 002 004 005 006 008 009 010 011 015 016 018 022 023 024 025 027 028 030 031 032 033 035 038 039 041 042 043 044 048 049 050 051 052 053 054 056 057 059 061 062 063 064 065 067 068 070 071 072 074 078 079
no	017 036 045 047 066 075 077

Sample Weight (g)	laboratory number
≥1 - <2	006 052
≥2 - <5	011 061
≥5 - <10	023 024 028 030 043 045 053 059 070 078
≥10 - <25	001 004 005 008 009 010 015 017 018 022 025 027 031 032 033 035 036 038 039 041 042 044 047 048 049 050 051 054 056 057 062 063 064 065 066 067 068 071 072 074 075 077 079
≥25 - <50	002
≥50	016

Extraction Solvent Components	laboratory number
acetic acid	015 036 067 072 075
acetone	004 006 008 009 016 047 050 064 074 077
acetonitrile	001 005 010 011 015 017 024 032 033 036 039 045 056 061 062 065 066 067 068 070 071 072 075 079
cyclohexane	016 025 064
dichloromethane	004 008 050 074
ethyl acetate	002 016 018 022 023 025 027 028 030 031 035 038 041 042 043 044 048 049 051 052 053 054 057 059 063 064
hexane	006
methanol	022
petroleum ether/spirit	004 008 050 074
sodium sulfate	051
water	022

Extraction Technique Used	laboratory number
ASE	025 057
cold solvent extraction at atmospheric pressure	001 002 004 005 006 008 010 011 017 018 022 023 024 027 028 030 031 032 033 035 039 042 044 045 048 049 050 051 053 054 059 061 062 064 065 066 067 068 070 071 074 077
solid liquid extraction	043
solvent extraction and partitioning with sodium sulphate and sodium acetate	036
solvent extraction at increased pressure/PLE	015

Extraction pH Adjusted	laboratory number
yes	001 011 016 017 032 061 066 067 070 072 075 078
no	002 004 005 006 008 009 010 015 018 022 023 024 025 027 028 030 031 033 035 036 038 039 041 042 043 044 045 047 048 049 050 051 052 053 054 056 057 059 062 063 064 065 068 071 074 077 079

Sample Clean-up Technique	laboratory number
C18	005
extraction	015 071
Extrelut	044
filter	010 074
GPC/HPGPC	004 016 025
liquid/liquid extraction	006 038 041 052 053
NH ₂ /aminopropyl column	011
primary secondary amine	022
PSA	028
PSA cleanup	035
QuEChERS	056 079
solid phase extraction (SPE) (column/cartridge)	059
solid phase extraction (SPE) (dispersive)	001 010 011 017 024 027 032 033 036 038 039 041 045 048 049 052 054 061 065 067 068 070 072 075 077 079
none	002 008 018 023 042 050 051 062 063 066

SPE Sorbent Type	laboratory number
C18	005 011 078 079
Envicarb/GCB	059
PSA	001 010 024 027 028 030 033 036 038 039 041 043 044 045 048 049 052 053 054 061 065 067 070 072 075
PSA + MgSO ₄	017
PSA/C18	032
silica	016
none	066

Certified Standards Used	laboratory number
yes	001 002 004 005 006 008 009 010 011 015 016 017 018 022 023 024 025 027 028 030 031 032 033 035 036 038 039 041 042 043 044 045 047 048 049 050 051 052 053 054 056 057 059 061 062 063 064 065 066 067 068 070 072 074 075 077 078 079

MS Confirmation	laboratory number
yes	001 002 004 005 006 008 009 010 011 015 016 017 018 022 023 024 025 027 028 030 031 032 033 035 036 038 039 041 042 043 044 045 047 048 049 050 051 052 053 054 056 057 059 061 062 063 064 065 066 067 068 070 072 074 075 077 078 079

Calibrations	laboratory number
matrix-matched	001 002 004 005 006 008 010 011 015 016 017 025 028 030 031 032 033 035 036 038 039 041 042 043 044 045 048 049 050 051 052 057 059 061 062 063 067 071 072 075 078 079
multi-level	001 004 008 009 016 017 023 025 027 031 035 036 038 051 054 059 061 063 064 065 068 074
single-level	006 016 032 033 047 056 066 070 077
solvent	024 027 053 066 068 074
standard addition	018 022

Source of Standards	laboratory number
Accu Standard	038 044 054 065 079
Chemservices and Supelco	075
Dr Ehrenstorfer	001 002 004 005 006 008 009 010 011 015 016 017 018 022 023 024 025 028 032 033 036 039 041 044 048 050 051 052 056 057 059 061 062 063 064 066 067 068 070 072 074 077
Fluka	011 030 042 053 063 066 074
Merck	071
Sigma/Aldrich	006 010 027 030 031 035 039 043 044 045 049 063 074
Supelco	044 063 074
various	078

Is Quoted Percentage Recovery Measured in Same Analytical Batch as Test Material?	laboratory number
yes	001 004 008 009 011 016 017 018 022 023 025 027 028 030 032 033 036 038 039 041 042 044 045 048 049 050 051 052 053 056 057 059 061 062 063 064 066 067 071 072 074 075 077 079
no	002 006 010 015 024 031 035 043 047 054 065 070 078

If Measured in the same Batch, When was the Spike Added?	laboratory number
prior to extraction	001 004 006 008 009 011 016 017 018 022 023 025 027 028 030 032 033 036 038 039 041 042 044 045 048 049 050 052 053 056 057 059 061 062 063 064 065 066 067 071 072 074 075 077 078 079
prior to clean up	051
prior to instrument measurement	024

Level of Spike (µg/kg)	laboratory number
<25	001 017 018 035 036 044 048 049 052 059 063 065 075
≥25 - <50	004 018 025 027 038 048 057 059 062 079

Level of Spike (µg/kg) (continued)	laboratory number
≥50 - <100	005 016 018 022 023 024 025 028 030 032 048 053 061 063 072 074
≥100 - <150	011 025 033 039 042 045 048 051 056 063 064 066
≥150 - <200	008 025 048 050 067 077
≥200 - <250	018 025 048 064
≥250 - <300	025
≥300 - <400	025 041 064
≥400 - <500	025
≥500	018 025

Composition of Blank Commodity used for Spiking	laboratory number
grape blank provided	004 006 008 011 016 017 018 022 027 030 032 033 035 038 041 042 044 045 048 049 050 051 052 053 057 059 061 063 065 066 067 072 075 077 078 079
grape test material provided	005 024 025 028 039 056 074
grape	001
in house blank	023 036 062 068

GC Column Type	laboratory number
capillary	002 004 006 008 009 011 015 016 018 024 025 031 032 033 036 041 043 044 047 050 052 056 059 061 063 064 066 068 070 071 077 078
narrowbore	023 074
widebore	005

GC Column Packing	laboratory number
14% cyanopropylphenyl 86%methyl polysiloxane	006
95% methyl 5% phenyl polysiloxane	002 004 005 006 008 011 018 023 024 025 032 033 036 041 043 044 050 052 056 059 061 064 066 068 070 074 077 078
5% phenyl-polysiloxane	009
5%phenyl-95%dimethyl polysiloxane	063
100% methyl polysiloxane	016
CP-sil 8 CB	031
none	075

GC Injection Volume (µL)	laboratory number
<1	032
≥1 - <2	005 006 011 015 016 018 023 024 025 033 047 052 059 061 064 070 077 078
≥2 - <5	004 008 009 041 044 050
≥5 - <10	002 056 063 068 074
≥10	031 036 043 066

GC Injection Mode	laboratory number
PTV	002 011 031 036 041 043 050 056 064 066 068 074 077
splitless	004 006 008 009 015 016 018 023 024 025 032 033 044 047 052 059 061 063 070 078

GC Detector	laboratory number
ECD	005 009 047 074
FPD	047
ITD	031 050 068 074
MS	008 015 016 018 023 024 032 033 043 047 059 064 066 070 071 077 078
MS-MS	002 004 011 025 036 041 044 052 056 061 063 074
NPD	008 047

HPLC Column Packing	laboratory number
C18	001 010 015 017 022 024 028 030 032 033 035 038 039 041 042 043 044 045 048 049 051 053 054 056 057 061 062 065 067 072 074 075 077
C18 X-Terra type	011
C8	027
endcapped	042
none	066

HPLC Guard Column Used	laboratory number
yes	001 010 017 024 030 033 035 038 039 041 042 043 044 045 048 049 051 056 057 062 071 072 074 075 079
no	011 015 016 022 025 027 028 032 053 054 061 065 067 077

Mobile Phase Programme	laboratory number
isocratic	042 054
gradient	001 010 011 015 016 017 022 024 027 028 030 032 033 035 038 039 041 043 044 045 048 049 051 053 056 057 061 062 065 067 071 072 074 075 077 079

Mobile Phase Components	laboratory number
acetate	011 056
acetic acid	011
acetonitrile	024 030 032 033 054 062
ammonium formate	001 017 027 035 041 044 051 061
formic acid	001 010 030 048 057 061 075
methanol	001 010 011 015 017 022 027 028 033 035 038 039 041 042 043 044 045 048 049 051 053 054 057 061 065 067 071 072 074 075
water	001 010 011 015 017 022 027 030 032 033 035 038 039 042 045 048 049 051 053 054 061 067 071 072 075

HPLC Column Temperature (°C)	laboratory number
ambient	028 033 038 039 041 043 044 049 056 065
>ambient - <50	001 010 011 015 017 022 024 027 030 032 035 042 045 048 051 053 054 057 061 062 071 072 074 075 077 079
≥50	067

HPLC Injection Volume (µL)	laboratory number
<5	024 032 072 079
≥5 - <10	011 043 053 056 057 074 077
≥10 - <25	001 010 015 017 022 028 030 033 035 038 039 041 042 044 048 049 051 054 062 065 067 075
≥25 - <50	027 061
≥50 - <100	045

Mobile Phase Flow Rate (mL/min)	laboratory number
<0.25	001 017 054 056
≥0.25 - <0.75	010 011 015 024 027 028 030 032 033 035 039 042 043 044 045 048 051 053 061 065 067 072 074 075 079
≥0.75 - <1.25	022 038 041 049 062 077

HPLC Pre Column Derivatisation	laboratory number
none	001 010 016 022 024 027 028 030 033 035 038 039 042 043 044 048 051 053 054 056 062 067 074 075 079

HPLC Post Column Derivatisation	laboratory number
none	001 010 016 022 024 027 028 030 033 035 038 039 042 043 044 048 051 053 054 056 062 067 074 075 079

HPLC Detector Type	laboratory number
MS-MS	001 010 015 016 017 022 024 027 028 030 032 033 035 038 039 041 042 043 044 045 048 049 051 053 054 056 057 061 062 067 071 072 074 075 077 079

Monocrotophos

Accredited Method Used	laboratory number
yes	004 005 011 012 013 015 016 022 023 024 025 027 028 030 031 032 034 035 037 038 039 041 042 043 044 048 049 050 051 052 053 054 056 057 059 062 063 067 069 070 071 072 074 078 079
no	001 002 017 021 045 068 077

Sample Weight (g)	laboratory number
≥2 - <5	011
≥5 - <10	023 024 028 030 037 043 045 053 059 070 078

Sample Weight (g) (continued)	laboratory number
≥10 - <25	001 004 005 012 013 015 017 021 022 025 027 031 032 035 038 039 041 042 044 048 049 050 051 052 054 056 057 062 063 067 068 069 071 072 074 077 079
≥25 - <50	002
≥50	016 034

Extraction Solvent Components	laboratory number
acetic acid	015 067 072
acetone	001 004 012 013 016 050 074 077
acetonitrile	005 011 015 017 021 024 032 034 037 039 045 056 062 063 067 068 070 071 072 079
cyclohexane	016
dichloromethane	001 004 050 074
ethyl acetate	002 016 022 025 027 028 030 031 035 038 041 042 043 044 048 049 051 052 053 054 057 059 069
methanol	022 023
petroleum ether/spirit	001 004 050 074
sodium sulfate	051
water	013 022 023

Extraction Technique Used	laboratory number
ASE	057
cold solvent extraction at atmospheric pressure	001 002 004 005 011 012 013 017 021 022 024 027 028 030 031 032 035 037 039 042 044 045 048 049 050 051 053 054 059 062 067 068 069 070 071 074 077
solid liquid extraction	043
solvent extraction	034
solvent extraction at increased pressure/PLE	015

Extraction pH Adjusted	laboratory number
yes	011 016 017 025 032 037 067 069 070 072 078
no	001 002 004 005 012 013 015 021 022 023 024 027 028 030 031 034 035 038 039 041 042 043 044 045 048 049 050 051 052 053 054 056 057 059 062 063 068 071 074 077 079

Sample Clean-up Technique	laboratory number
C18	005
DisQuE Extraction Tube	025
extraction	015 069 071
Extrelut	044
filter	034 074
GPC/HPGPC	004 013 016
liquid/liquid extraction	037 038 041 052 053 063
NH ₂ /aminopropyl column	011
Primary Secondary Amine	022
PSA	028
PSA cleanup	035
QuEChERS	056 079
solid phase extraction (SPE) (column/cartridge)	012 059
solid phase extraction (SPE) (dispersive)	011 017 021 024 027 032 038 039 041 042 045 048 049 052 054 067 068 070 072 077 079
none	001 002 023 050 051 062 063

SPE Sorbent Type	laboratory number
C18	005 011 012 078 079
Envicarb/GCB	059
PSA	021 024 027 028 030 038 039 041 042 043 044 045 048 049 053 054 067 070 072
PSA & GCB	052
PSA + MgSO ₄	017
PSA/C18	032
silica	016

Certified Standards Used	laboratory number
yes	001 002 004 005 011 012 015 016 017 021 022 023 024 025 027 028 030 031 032 034 035 037 038 039 041 042 043 044 045 048 049 050 051 052 053 054 056 057 059 062 063 067 068 069 070 072 074 077 078 079
no	013

MS Confirmation	laboratory number
yes	001 002 004 005 011 012 013 015 016 017 022 023 024 025 027 028 030 031 032 034 035 037 038 039 041 042 043 044 045 048 049 050 051 052 053 054 056 057 059 062 063 067 068 069 070 072 074 077 078 079
no	021

Calibrations	laboratory number
matrix-matched	001 002 004 005 011 015 016 017 025 028 030 031 032 035 037 038 039 041 042 043 044 045 048 049 050 051 052 057 059 062 063 067 069 071 072 078 079
multi-level	001 004 016 017 023 025 027 031 034 035 038 051 054 059 063 068 074
single-level	013 016 021 032 056 070 077
solvent	024 027 053 068 074
standard addition	012 022

Source of Standards	laboratory number
Accu Standard	044 054 079
Dr Ehrenstorfer	001 002 004 005 011 012 013 015 016 017 021 022 023 024 028 032 037 038 039 041 044 048 050 051 052 056 057 059 062 063 067 068 069 070 072 074 077
Fluka	011 025 030 042 063 074
Merck	071
Restek	034
Sigma/Aldrich	027 030 031 035 039 043 044 045 049 053 063 074
Supelco	044 063 074
various	078

Is Quoted Percentage Recovery Measured in Same Analytical Batch as Test Material?	laboratory number
yes	001 004 011 012 013 016 017 021 022 023 025 027 028 030 032 037 038 039 041 042 044 045 048 049 050 051 052 053 056 057 059 062 063 067 069 071 072 074 077 079
no	002 015 024 031 034 035 043 054 070 078

If Measured in the same Batch, When was the Spike Added?	laboratory number
prior to extraction	001 004 011 012 013 016 017 021 022 023 025 027 028 030 032 037 038 039 041 042 044 045 048 049 050 052 053 056 057 059 062 063 067 069 071 072 074 077 078 079
prior to clean up	051
prior to instrument measurement	024

Level of Spike (µg/kg)	laboratory number
<25	001 017 021 035 037 044 048 049 052 059 063
≥25 - <50	004 013 027 038 048 057 059 062 079
≥50 - <100	005 016 022 023 024 028 030 032 048 053 063 072 074
≥100 - <150	011 039 042 045 048 056
≥150 - <200	025 048 077
≥200 - <250	025 048
≥250 - <300	025 050
≥300 - <400	025 041 051 067 069
≥400 - <500	025
≥500	012 025

Composition of Blank Commodity used for Spiking	laboratory number
grape blank provided	004 011 013 016 017 021 022 027 030 032 035 038 041 042 044 045 048 049 050 051 052 053 057 059 063 067 072 077 078 079
grape test material provided	005 012 024 025 028 039 056 074
grape	001
in house blank	023 062 068

GC Column Type	laboratory number
capillary	001 002 004 011 012 013 015 016 024 031 032 034 037 041 042 043 044 056 059 069 070 071 077 078
narrowbore	074

GC Column Packing	laboratory number
65% methyl 35% phenyl polysiloxane	012
95% methyl 5% phenyl polysiloxane	001 002 004 005 011 024 032 041 042 043 044 056 059 069 070 074 077 078
5% phenyl methyl siloxane	034
100% methyl polysiloxane	016
CP-sil 8 CB	031

GC Injection Volume (µL)	laboratory number
<1	012 032
≥1 - <2	011 015 016 024 034 059 069 070 077 078
≥2 - <5	004 041 042 044
≥5 - <10	002 056 074
≥10	001 013 031 043

GC Injection Mode	laboratory number
large volume	013
PTV	001 002 011 031 041 043 056 068 074 077
pulsed splitless	034
split	069
splitless	004 012 015 016 024 032 042 044 059 070 078

GC Detector	laboratory number
ECD	074
FPD	012 034 070
HR-MS	069
ITD	001 031 074
MS	013 015 016 024 032 042 043 059 070 071 077 078
MS-MS	001 002 004 011 041 044 056 074

HPLC Column Packing	laboratory number
C18	005 013 015 017 021 022 023 024 028 030 032 035 037 038 039 041 043 044 045 048 049 050 051 052 053 054 056 057 062 063 067 068 069 072 074 077
C18 X-Terra type	011
C8	027
endcapped	021 068
UPLC HSS T3 (Waters)	025

HPLC Guard Column Used	laboratory number
yes	017 021 023 024 025 030 035 038 039 041 043 044 045 048 049 051 056 057 062 068 069 071 072 074 079
no	005 011 013 015 016 022 027 028 032 037 050 052 053 054 063 067 077

Mobile Phase Programme	laboratory number
isocratic	054
gradient	005 011 013 015 016 017 021 022 023 024 025 027 028 030 032 035 037 038 039 041 043 044 045 048 049 050 051 052 053 056 057 062 063 067 068 069 071 072 074 077 079

Mobile Phase Components	laboratory number
acetate	011 021 056
acetic acid	011 021
acetonitrile	013 021 024 030 032 037 054 062
ammonium formate	005 017 023 035 041 044 048
ammonium formate buffer	052
formic acid	025 030 048 057
methanol	011 015 017 022 023 025 027 028 035 038 039 041 043 044 045 048 049 050 051 052 053 054 057 063 067 068 069 071 072 074
water	011 013 015 017 021 022 023 025 027 030 032 035 038 039 045 048 049 050 051 053 054 063 067 068 069 071 072

HPLC Column Temperature (°C)	laboratory number
ambient	013 023 028 037 038 039 041 043 044 049 056
>ambient - <50	005 011 015 017 021 022 024 025 027 030 032 035 045 048 051 052 053 054 057 062 063 068 069 071 072 074 077 079
≥50	050 067

HPLC Injection Volume (µL)	laboratory number
<5	013 024 032 050 072 079
≥5 - <10	011 043 053 056 057 068 074 077
≥10 - <25	005 015 017 021 022 023 025 028 030 035 038 039 041 044 048 049 051 052 054 062 063 067 069
≥25 - <50	027
≥50 - <100	037 045

Mobile Phase Flow Rate (mL/min)	laboratory number
<0.25	017 023 054 056 068 069
≥0.25 - <0.75	005 011 013 015 021 024 025 027 028 030 032 035 037 039 043 044 045 048 050 051 052 053 063 067 072 074 079
≥0.75 - <1.25	022 038 041 049 062 077

HPLC Detector Type	laboratory number
MS-MS	005 013 015 016 017 021 022 023 024 025 027 028 030 032 035 037 038 039 041 043 044 045 048 049 050 051 052 053 054 056 057 062 063 067 068 069 071 072 074 077 079

Quinalphos

Accredited Method Used	laboratory number
yes	002 004 005 008 011 012 016 018 022 023 024 025 027 028 030 031 032 035 038 039 041 042 043 044 048 049 050 051 052 053 054 056 057 059 061 062 063 064 065 067 069 070 071 072 074 078 079
no	021 036 045 066 077

Sample Weight (g)	laboratory number
≥2 - <5	011
≥5 - <10	023 024 028 030 043 045 053 059 070 078
≥10 - <25	004 005 008 012 018 021 022 025 027 031 032 035 036 038 039 041 042 044 048 049 050 051 052 054 056 057 061 063 064 065 066 067 069 071 072 074 077 079
≥25 - <50	002 062
≥50	016

Extraction Solvent Components	laboratory number
acetic acid	036 067 072
acetone	004 008 012 016 050 064 074 077
acetonitrile	005 011 021 024 032 036 039 045 056 061 065 066 067 070 071 072 079
cyclohexane	016 025 064
dichloromethane	004 008 050 074
ethyl acetate	002 016 018 022 023 025 027 028 030 031 035 038 041 042 043 044 048 049 051 052 053 054 057 059 062 063 064 069
methanol	022
petroleum ether/spirit	004 008 050 074
sodium sulfate	051
water	022

Extraction Technique Used	laboratory number
ASE	025 057
cold solvent extraction at atmospheric pressure	002 004 005 008 011 012 018 021 022 023 024 027 028 030 031 032 035 039 042 044 045 048 049 050 051 053 054 059 061 062 064 065 066 067 069 070 071 074 077
solid liquid extraction	043
solvent extraction and partitioning with sodium sulphate and sodium acetate	036

Extraction pH Adjusted	laboratory number
yes	011 016 032 061 062 066 067 069 070 072 078
no	002 004 005 008 012 018 021 022 023 024 025 027 028 030 031 035 036 038 039 041 042 043 044 045 048 049 050 051 052 053 054 056 057 059 063 064 065 071 074 077 079

Sample Clean-up Technique	laboratory number
C18	005
extraction	069 071
Extrelut	044
filter	074
GPC/HPGPC	004 016 025 057
liquid/liquid extraction	038 041 052 053
NH ₂ /aminopropyl column	011
Primary Secondary Amine	022
PSA	028
PSA cleanup	035
QuEChERS	056 079
solid phase extraction (SPE) (column/cartridge)	012 059
solid phase extraction (SPE) (dispersive)	011 021 024 027 032 036 038 039 041 042 045 048 049 052 054 065 067 070 072 077 079
SPD	061
none	002 008 018 023 050 051 062 063 066

SPE Sorbent Type	laboratory number
C18	005 011 012 078 079
Envicarb/GCB	059
PSA	021 024 027 028 030 036 038 039 041 042 043 044 045 048 049 053 054 065 067 070 072
silica	016
PSA & GCB	052
PSA/C18	032
none	066

Certified Standards Used	laboratory number
yes	002 004 005 008 011 012 016 018 021 022 023 024 025 027 028 030 031 032 035 036 038 039 041 042 043 044 045 048 049 050 051 052 053 054 056 057 059 061 062 063 064 065 066 067 069 070 072 074 077 078 079

MS Confirmation	laboratory number
yes	002 004 005 008 011 012 016 018 022 023 024 025 027 028 030 031 032 035 036 038 039 041 042 043 044 045 048 049 050 051 052 053 054 056 057 059 061 062 063 064 065 066 067 069 070 072 074 077 078 079
no	021

Calibrations	laboratory number
matrix-matched	002 004 005 008 011 016 025 028 030 031 032 035 036 038 039 041 042 043 044 045 048 049 050 051 052 057 059 061 062 063 067 069 071 072 078 079
multi-level	004 008 016 023 025 027 031 035 036 038 051 054 059 061 063 064 065 074
single-level	016 021 032 056 066 070 077
solvent	024 027 053 061 066 074
standard addition	012 018 022

Source of Standards	laboratory number
Accu Standard	044 054 065 079
Dr Ehrenstorfer	002 004 005 008 011 012 016 018 022 023 024 025 028 032 036 038 039 041 044 048 050 051 052 056 057 059 061 062 063 064 066 067 069 070 072 074 077
Fluka	011 030 042 053 063 066 074
Merck	071
Sigma/Aldrich	027 030 031 035 039 043 044 045 049 063 074
Supelco	044 063 074
Ultra scientific	021
various	078

Is Quoted Percentage Recovery Measured in Same Analytical Batch as Test Material?	laboratory number
yes	004 008 011 012 016 018 021 022 023 025 027 028 030 032 036 038 039 041 042 044 045 048 049 050 051 052 053 056 057 059 062 063 064 066 067 069 071 072 074 077 079
no	002 024 031 035 043 054 061 065 070 078

If Measured in the same Batch, When was the Spike Added?	laboratory number
prior to extraction	004 008 011 012 016 018 021 022 023 025 027 028 030 032 036 038 039 041 042 044 045 048 049 050 052 053 056 057 059 061 062 063 064 065 066 067 069 071 072 074 077 078 079
prior to clean up	051
prior to instrument measurement	024

Level of Spike (µg/kg)	laboratory number
<25	018 021 035 036 044 048 049 052 059 063 065
≥25 - <50	004 008 018 025 027 038 048 050 051 057 059 061 062 079
≥50 - <100	005 016 018 022 023 024 025 028 030 032 048 053 063 067 069 072 074
≥100 - <150	011 025 039 042 045 048 056 063 064 066
≥150 - <200	025 048 077
≥200 - <250	018 025 048 064
≥250 - <300	025
≥300 - <400	025 041 064
≥400 - <500	025
≥500	012 018 025

Composition of Blank Commodity used for Spiking	laboratory number
grape blank provided	004 008 011 016 018 021 022 027 030 032 035 038 041 042 044 045 048 049 050 051 052 053 057 059 061 063 065 066 067 072 077 078 079
grape test material provided	005 012 024 025 028 039 056 074
in house blank	023 036 062

GC Column Type	laboratory number
capillary	002 004 008 011 012 016 018 024 025 031 032 036 041 042 043 044 056 057 059 061 062 063 064 065 066 067 069 070 071 072 077 078
narrowbore	023 074

GC Column Packing	laboratory number
65% methyl 35% phenyl polysiloxane	012 062
95% methyl 5% phenyl polysiloxane	002 004 005 008 011 018 023 024 025 032 036 041 042 043 044 056 057 059 064 065 066 067 069 070 072 074 077 078
5%phenyl-95%dimethyl polysiloxane	063
100% methyl polysiloxane	016
CP-sil 8 CB	031

GC Injection Volume (µL)	laboratory number
<1	012 032
≥1 - <2	011 016 018 023 024 025 057 059 064 065 069 070 077 078
≥2 - <5	004 008 041 042 044 061 062
≥5 - <10	002 056 063 072 074
≥10	031 036 043 066 067

GC Injection Mode	laboratory number
PTV	002 011 031 036 041 043 056 064 066 067 072 074 077
split	069
splitless	004 008 012 016 018 023 024 025 032 042 044 057 059 061 062 063 065 070 078

GC Detector	laboratory number
ECD	074
FPD	008 012 070
HR-MS	069
ITD	031 074
MS	008 016 018 023 024 032 042 043 057 059 062 064 066 067 070 071 072 077 078
MS-MS	002 004 011 025 036 041 044 056 063 065 074

HPLC Column Packing	laboratory number
C18	005 021 022 024 028 030 032 035 038 039 041 043 044 045 048 049 050 051 052 053 054 056 061 062 069 074 077
C18 X-Terra type	011
C8	027
endcapped	021
none	066

HPLC Guard Column Used	laboratory number
yes	021 024 030 035 038 039 041 043 044 045 048 049 051 056 062 069 071 074 079
no	005 011 016 022 025 027 028 032 050 052 053 054 061 065 067 077

Mobile Phase Programme	laboratory number
isocratic	054
gradient	005 011 016 021 022 024 027 028 030 032 035 038 039 041 043 044 045 048 049 050 051 052 053 056 061 062 065 067 069 071 074 077 079

Mobile Phase Components	laboratory number
acetate	011 021 056
acetic acid	011 021
acetonitrile	021 024 030 032 054 062
ammonium formate	005 027 035 041 044 051 052
ammonium formate buffer	052
formic acid	030 048 061
methanol	011 022 027 028 035 038 039 041 043 044 045 048 049 050 051 052 053 054 061 069 071 074
water	011 021 022 027 030 032 035 038 039 045 048 049 050 051 053 054 061 069 071

HPLC Column Temperature (°C)	laboratory number
ambient	028 038 039 041 043 044 049 056 062
>ambient - <50	005 011 021 022 024 027 030 032 035 045 048 051 052 053 054 061 069 071 074 077 079
≥50	050

HPLC Injection Volume (µL)	laboratory number
<5	024 032 050 062 079
≥5 - <10	011 043 053 056 061 074 077
≥10 - <25	005 021 022 028 030 035 038 039 041 044 048 049 051 052 054 069
≥25 - <50	027
≥50 - <100	045

Mobile Phase Flow Rate (mL/min)	laboratory number
<0.25	054 056 069
≥0.25 - <0.75	005 011 021 024 027 028 030 032 035 039 043 044 045 048 050 051 052 053 061 074 079
≥0.75 - <1.25	022 038 041 049 062 077

HPLC Detector Type	laboratory number
MS-MS	005 016 021 022 024 027 028 030 032 035 038 039 041 043 044 045 048 049 050 051 052 053 054 056 061 062 069 071 074 077 079

Quinoxifen

Accredited Method Used	laboratory number
yes	002 004 005 008 010 011 013 016 018 024 025 031 032 033 039 050 051 056 057 059 061 062 063 064 065 067 069 070 071 072 074 078
no	003 017 036 045 066 068 077

Sample Weight (g)	laboratory number
≥2 - <5	011
≥5 - <10	018 024 045 059 070 078
≥10 - <25	003 005 008 010 013 017 025 031 032 033 036 039 050 051 056 057 061 062 063 064 065 066 067 068 069 071 072 074 077
≥25 - <50	002
≥50	016

Extraction Solvent Components	laboratory number
acetic acid	003 004 036 067 072
acetone	008 013 016 050 064 074 077
acetonitrile	003 004 005 010 011 017 018 024 032 033 036 039 045 056 061 062 065 066 067 068 070 071 072
cyclohexane	016 025 064
dichloromethane	008 050 074
ethyl acetate	002 016 025 051 057 059 063 064 069
petroleum ether/spirit	008 050 074
sodium sulfate	051
water	013

Extraction Technique Used	laboratory number
ASE	025 057
cold solvent extraction at atmospheric pressure	002 003 004 005 008 010 011 013 017 018 024 032 033 039 045 050 051 059 061 062 064 065 066 067 068 069 070 071 074 077
solvent extraction and partitioning with sodium sulphate and sodium acetate	036

Extraction pH Adjusted	laboratory number
yes	004 011 016 017 032 061 066 067 069 070 072 078
no	002 003 005 008 010 013 018 024 025 031 033 036 039 045 050 051 056 057 059 062 063 064 065 068 071 074 077

Sample Clean-up Technique	laboratory number
C18	005
extraction	069 071
filter	010 074
GPC/HPGPC	013 016 025 057
NH ₂ /aminopropyl column	011
QuEChERS	056
solid phase extraction (SPE) (column/cartridge)	059
solid phase extraction (SPE) (dispersive)	003 004 010 011 017 018 024 032 033 036 039 045 061 065 067 068 070 072 077
none	002 008 050 051 062 063 066

SPE Sorbent Type	laboratory number
C18	005 011 078
Envicarb/GCB	059
PSA	003 004 010 018 024 033 036 039 045 061 065 067 070 072
PSA + MgSO ₄	017
PSA/C18	032
silica	016
none	066

Certified Standards Used	laboratory number
yes	002 003 004 005 008 010 011 016 017 018 024 025 031 032 033 036 039 045 050 051 056 057 059 061 062 063 064 065 066 067 068 069 070 072 074 077 078
no	013

MS Confirmation	laboratory number
yes	002 003 004 005 008 010 011 013 016 017 018 024 025 031 032 033 036 039 045 050 051 056 057 059 061 062 063 064 065 066 067 068 069 070 072 074 077 078

Calibrations	laboratory number
matrix-matched	002 003 004 005 008 010 011 016 017 018 025 031 032 033 036 039 045 050 051 057 059 061 062 063 067 069 071 072 078
multi-level	003 004 008 016 017 025 031 036 051 059 061 063 064 065 068 074
single-level	013 016 032 033 056 066 070 077
solvent	024 066 068 074

Source of Standards	laboratory number
Accu Standard	065
Dr Ehrenstorfer	002 003 004 005 008 010 011 013 016 017 018 024 025 032 033 036 039 050 056 057 059 062 063 064 066 067 068 069 070 072 074 077

Source of Standards (continued)	laboratory number
Fluka	011 063 066 074
Merck	071
Sigma/Aldrich	010 031 039 045 051 063 074
Supelco	063 074
various	078

Is Quoted Percentage Recovery Measured in Same Analytical Batch as Test Material?	laboratory number
yes	003 004 008 011 013 016 017 018 025 032 033 036 039 045 050 051 056 057 059 062 063 064 066 067 069 071 072 074 077
no	002 010 024 031 061 065 070 078

If Measured in the same Batch, When was the Spike Added?	laboratory number
prior to extraction	003 004 008 011 013 016 017 018 025 032 033 036 039 045 050 056 057 059 061 062 063 064 065 066 067 069 071 072 074 077 078
prior to clean up	051
prior to instrument measurement	024

Level of Spike (µg/kg)	laboratory number
<25	017 018 036 059 063 065
≥25 - <50	013 018 025 057 059 062
≥50 - <100	004 005 016 018 024 025 032 063 072 074
≥100 - <150	003 011 025 033 039 045 056 061 063 064 066
≥150 - <200	025 077
≥200 - <250	018 025 051 064 067
≥250 - <300	025 050
≥300 - <400	025 064 069
≥400 - <500	008 025
≥500	018 025

Composition of Blank Commodity used for Spiking	laboratory number
grape blank provided	004 008 011 013 016 017 018 032 033 045 050 051 057 059 061 063 065 066 067 072 077 078
grape test material provided	005 024 025 039 056 074
grape	003
in house blank	036 062 068

GC Column Type	laboratory number
capillary	002 008 011 013 016 024 025 032 033 036 050 051 056 057 059 063 064 066 067 068 069 070 071 072 077 078
narrowbore	061 074
widebore	005

GC Column Packing	laboratory number
100% methyl polysiloxane	016
95% methyl 5% phenyl polysiloxane	002 005 008 011 024 025 032 033 036 050 051 056 057 059 064 066 067 068 069 070 072 074 077 078
5%phenyl-95%dimethyl polysiloxane	063

GC Injection Volume (µL)	laboratory number
<1	032
≥1 - <2	005 011 016 024 025 033 057 059 061 064 069 070 077 078
≥2 - <5	008 050
≥5 - <10	002 056 063 068 072 074
≥10	013 036 051 066 067

GC Injection Mode	laboratory number
large volume	013
PTV	002 011 036 050 051 056 064 066 067 068 072 074 077
split	069
splitless	008 016 024 025 032 033 057 059 061 063 070 078

GC Detector	laboratory number
ECD	005 008 074
HR-MS	069
ITD	050 068 074
MS	008 013 016 024 032 033 051 057 059 064 066 067 070 071 072 077 078
MS-MS	002 011 025 036 056 063 074
NPD	061

HPLC Column Packing	laboratory number
C18	003 004 010 013 017 018 024 031 032 033 039 045 056 062 065 069 074 077
C18 X-Terra type	011
endcapped	003
none	066

HPLC Guard Column Used	laboratory number
yes	003 010 017 018 024 031 033 039 045 056 062 069 071 074
no	004 011 013 016 025 032 051 061 065 067 077

Mobile Phase Programme	laboratory number
gradient	003 004 010 011 013 016 017 018 024 031 032 033 039 045 056 061 062 065 067 069 071 074 077

Mobile Phase Components	laboratory number
acetate	011 056
acetic acid	011
acetonitrile	013 024 031 032 033 062
ammonium formate	017 031
formic acid	003 010 031 061
methanol	003 004 010 011 017 018 031 033 039 045 061 065 069 071 074
water	003 004 010 011 013 017 018 032 033 039 045 061 069 071

HPLC Column Temperature (°C)	laboratory number
ambient	013 033 039 056 065
>ambient - <50	003 004 010 011 017 018 024 031 032 045 061 062 069 071 074 077

HPLC Injection Volume (µL)	laboratory number
<5	013 024 032
≥5 - <10	011 018 056 074 077
≥10 - <25	003 004 010 017 031 033 039 061 062 065 069
≥50 - <100	045

Mobile Phase Flow Rate (mL/min)	laboratory number
<0.25	017 018 031 056 069
≥0.25 - <0.75	003 010 011 013 024 032 033 039 045 061 065 074
≥0.75 - <1.25	004 062 077

HPLC Pre Column Derivatisation	laboratory number
none	003 004 010 016 024 033 039 056 062 069 074

HPLC Post Column Derivatisation	laboratory number
none	003 004 010 016 024 033 039 056 062 069 074

HPLC Detector Type	laboratory number
MS-MS	003 004 010 013 016 017 018 024 031 032 033 039 045 056 061 062 069 071 074 077

APPENDIX II: FAPAS SecureWeb, Reports and Protocol

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, including surplus test materials from the batch used in this proficiency test.
- Freely download copies of reports, in Acrobat PDF format, of proficiency tests in which they have participated.

2. REPORTS

The Acrobat PDF version of this report is available to all participants as a free download from FAPAS SecureWeb.

A printed and bound version of this report can be purchased, please contact FAPAS for current pricing.

3. PROTOCOL

The Protocols [1, 2] set out how FAPAS® is organised. Copies can be downloaded from our website.

4. QUALITY SYSTEMS

FAPAS® is accredited by UKAS as complying with the requirements of ISO/IEC Guide 43 - 1: 1997, through assessment against ILAC Guide G13:2007.

The Food and Environment Research Agency is an ISO 9001 certified organisation.



5. CONTACT DETAILS

Participants with any comments or concerns about this proficiency test should contact:

FAPAS
The Food and Environment Research Agency
Sand Hutton, York
YO41 1LZ
UK

Tel: +44 (0)1904 462100
Fax: +44 (0)1904 462040
e-mail: info@fapas.com
testmaterials@fapas.com
web: www.fapas.com