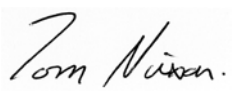
FAPAS® Proficiency Test Report 19128

Pesticide Residues in Green Bean

November 2011–January 2012

Prepared and authorised on behalf of FAPAS by



Tom Nixon, Round Co-ordinator

0009 PT

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® proficiency test 19128 was dispatched in November 2011. Each participant received a green bean test material. From a list of 171 pesticide residues, participants had to identify and quantify those present.

2. The test material contained chlorpyrifos-methyl, indoxacarb (measured as sum of R & S isomers), omethoate, pirimiphos-methyl, propiconazole and spinosad (measured as spinosyn A & spinosyn D). An assigned value (x_a) was determined for each residue and in conjunction with the standard deviation for proficiency (σ_p) was used to calculate a z-score for each result.

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

analyte	assigned value, x_a µg/kg	number of scores, |z| ≤2	total number of scores	% |z| ≤2
chlorpyrifos-methyl	39.1	52	59	88
indoxacarb	101.1	44	47	94
omethoate	97.9	36	46	78
pirimiphos-methyl	143.6	49	53	92
propiconazole	51.9	39	49	80
spinosad	46.9	32	40	80

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 are available in our protocols [2, 3].

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 to ISO 17043.

The test material was prepared from organic green beans 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. The remainder was stored at -20 °C. No residues were detected at or above 30 µg/kg. Spinosad was detected below 30 µg/kg.

All pesticide residues were spiked into the test material including additional spinosad.

Samples were stored at -20 °C temperature until dispatch.

2.2. Homogeneity

To test for homogeneity, randomly selected test materials were analysed in duplicate. Testing was sub-contracted to a laboratory meeting the quality requirements of the scheme's accreditation to ISO 17043.

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 November 2011. Test materials were sent to 66 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. This included indoxacarb, which was to be

reported as the sum of R and S isomers and spinosad, which was to be reported as the sum of spinosyn A and spinosyn D.

Results were submitted by 65 participants (98%) before the closing date for this test, 4 January 2012.

Each participant was given a laboratory number, assigned in order of receipt of results. The reported analyte concentrations are given in Table 1 for chlorpyrifos-methyl, indoxacarb and omethoate and in Table 2 for pirimiphos-methyl, propiconazole and spinosad.

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.

If a participant analysed for a pesticide residue that was in the test material, but did not identify it and their LoQ was *above* the level needed for a z-score of -2.0, then the result was recorded as <LoQ.

Any participant identifying pesticide residues other than chlorpyrifos-methyl, indoxacarb, omethoate, pirimiphos-methyl, propiconazole and spinosad at levels above ≥30 µg/kg are listed in Table 3.

Participants' comments are given in Table 4.

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

4. STATISTICAL EVALUATION OF RESULTS

The results submitted by participants were statistically analysed in order to provide an assigned value for each analyte. The assigned values were then used in combination with the standard deviation for proficiency, σ_p, to calculate a z-score for each result. The procedure follows that recommended in the IUPAC International Harmonised Protocol for the Proficiency Testing of Analytical Chemistry Laboratories [4].

Further details on the procedure followed can be found in the relevant protocols [2, 3].

4.1. Calculation of the Assigned Value, x_a

The assigned value, x_a, for chlorpyrifos-methyl, indoxacarb, omethoate, pirimiphos-methyl, propiconazole and spinosad was derived from 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 × 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% [5],

iv) results where no limit of quantification was reported,

v) results below the reported limit of quantification.

For chlorpyrifos-methyl, indoxacarb, omethoate, pirimiphos-methyl, propiconazole and spinosad, 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 chlorpyrifos-methyl, indoxacarb, omethoate, pirimiphos-methyl, propiconazole and spinosad, σ_p was derived from the appropriate form of the Horwitz equation [6].

The values for σ_p used to calculate z-scores from the reported results of this test are given in Table 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 for chlorpyrifos-methyl, indoxacarb and omethoate are given in Table 1 and for pirimiphos-methyl, propiconazole and spinosad are given in Table 2. 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 [4].

6. REFERENCES

1 Adobe Certified Document Services http://www.adobe.com/security/partners_cds.html, accessed 24/05/2011.

2 FAPAS, 2010, Protocol for Proficiency Testing Schemes, Part 1 – Common Principles, Version 2, Issued December 2010.

3 FAPAS, 2009, Protocol for Proficiency Testing Schemes, Part 2 – FAPAS®, Revision 2009, Version 1, Issued November 2009.

4 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.

5 Method Validation and Quality Control Procedures for Pesticide Residues Analysis in Food and Feed, Document No. SANCO/12495/2011

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

Table 1: Results and z-Scores for Chlorpyrifos-methyl, Indoxacarb and Omethoate

laboratory number	analyte											
	chlorpyriphos-methyl assigned value 39.1 µg/kg				indoxacarb assigned value 101.1 µg/kg				omethoate assigned value 97.9 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	29.8		10	-1.1	66.5		20	-1.6	86.7		10	-0.5
002	32	87	10	-0.8	94	99	10	-0.3	63	73	10	-1.6
003	41.1	99	20	0.2	127.0	81	10	1.2	#			
004	50	90		1.3	124	94		1.0	192	86		**4.4**
005	#				#				#			
006	45	104	10	0.7	#				#			
007	39.9	78	20	0.1	96.4	86	10	-0.2	#			
008	49.95	85	10	1.3	#				0			**-4.5**
009	35.7	96	10	-0.4	99.0	101	10	-0.1	83.3	96	10	-0.7
010	30	71	10	-1.1	85	99	10	-0.7	119	93	10	1.0
011	42.1	81	20.0	0.3	98.9	90	10.0	-0.1	#			
012	45	114	5	0.7	106	78	5	0.2	118	89	5	0.9
013	34			-0.6	71			-1.4	0		20	**-4.5**
014	40	97	10	0.1	105	103	10	0.2	105	80	10	0.3
015	31	96	30	-0.9	73	112	30	-1.3	115	94	60	0.8
016	43	92	5	0.4	104	99	5	0.1	100	91	5	0.1
017	42.7	102	20	0.4	100.2	79	10	0.0	#			
018	36.3	89	20	-0.3	96.5	76	10	-0.2	#			
019	41.8	94	20	0.3	102.6	79		0.1	#			
020	30.6	78	10	-1.0	77.9	84	10	-1.0	71.7	111	10	-1.2
021	38	92	10	-0.1	112	106	10	0.5	89	100	10	-0.4
022	#				#				91.82		0.04	-0.3
023	45.66	92.78	10	0.8	#				#			
024	24	80.0	10	-1.8	110	96.2	10	0.4	97	79.5	10	0.0
025	10.6	98	10	**-3.3**	90.4	93	10	-0.5	21.7	100	10	**-3.5**
026	32	75	10	-0.8	99	111	10	-0.1	80	80	10	-0.8
027	30		10	-1.1	97		10	-0.2	103		10	0.2
028	33.9	90	10	-0.6	#				#			

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

Table 1 (continued): Results and z-Scores for Chlorpyrifos-methyl, Indoxacarb and Omethoate

laboratory number	analyte											
	chlorpyriphos-methyl assigned value 39.1 µg/kg				indoxacarb assigned value 101.1 µg/kg				omethoate assigned value 97.9 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
029	22		10	-2.0	63		10	-1.7	67		10	-1.4
030	31.0		10	-0.9	#				122		10	1.1
031	79.5	40	5	**4.7**	#				#			
032	50	94	10	1.3	#				#			
033	43.28		10	0.5	113.96		10	0.6	100.08		10	0.1
034	#				101	80	50	0.0	106	75	10	0.4
035	77	90	10	**4.4**	83	90	10	-0.8	86	90	10	-0.6
036	44.69		10	0.6	93.57		10	-0.3	75.59		10	-1.0
037	26.2	79	10	-1.5	66.5	84	10	-1.6	104	90	10	0.3
038	47	75	10	0.9	110	92	10	0.4	120	70	10	1.0
039	45	93	10	0.7	111	108	10	0.4	102	110	10	0.2
040	45	85	5	0.7	105	102	5	0.2	105	95	3	0.3
041	39	105	10	0.0	119	111	10	0.8	114	93	10	0.7
042	26		10	-1.5	99		10	-0.1	86		10	-0.6
043	33	91	10	-0.7	0	90	10	**-4.5**	0	0	10	**-4.5**
044	49	104	20	1.1	106	107	50	0.2	51	82	30	**-2.2**
045	42.2	82.80	10	0.4	#				#			
046	#				#				#			
047	30	102.1	10	-1.1	49	91.1	10	**-2.3**	150	75.3	10	**2.4**
048	50			1.3	118			0.8	86			-0.6
049	37	97	10	-0.2	109	117	10	0.4	103	89	10	0.2
050	0		10.00	**-4.5**	#				0		40.00	**-4.5**
051	37.0	42	32.7	-0.2	#				#			
052	#				#				#			
053	30.66		50	-1.0	#				105.46		50	0.4
054	47	91	10	0.9	#				#			
055	60	90	10	**2.4**	116	90	10	0.7	112	96	10	0.7
056	43	100	10	0.4	140	100	10	1.8	#			

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

Table 1 (continued): Results and z-Scores for Chlorpyrifos-methyl, Indoxacarb and Omethoate

laboratory number	analyte											
	chlorpyriphos-methyl assigned value 39.1 µg/kg				indoxacarb assigned value 101.1 µg/kg				omethoate assigned value 97.9 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
057	44	91	10	0.6	123	115	10	1.0	132	100	10	1.6
058	20	91	5	**-2.2**	#				108	100	10	0.5
059	28.6	76	10	-1.2	65.7	86	10	-1.6	86.1	96	10	-0.5
060	30	101	10	-1.1	140	100	10	1.8	44	85	10	**-2.5**
061	22	87	10	-2.0	39	93	10	**-2.8**	95	120	10	-0.1
062	40	126	10	0.1	90	121	10	-0.5	12	90	10	**-4.0**
063	62	90-110	10	**2.7**	124	90-110	10	1.0	118	90-110	10	0.9
064	51	84	10	1.4	108	75	10	0.3	126	70	20	1.3
065	#				#				#			

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

Table 2: Results and z-Scores for Pirimiphos-methyl, Propiconazole and Spinosad

laboratory number	analyte											
	pirimiphos-methyl assigned value 143.6 µg/kg				propiconazole assigned value 51.9 µg/kg				spinosad assigned value 46.9 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	97.6		10	-1.5	39.1		10	-1.1	25.1		10	**-2.1**
002	121	91	10	-0.7	44	90	10	-0.7	41	77	10	-0.6
003	#				#				65.4	82	20	1.8
004	258	97		**3.7**	61	91		0.8	53	97		0.6
005	172.25	88	10	0.9	86.25	105	10	**3.0**	#			
006	180	103	10	1.2	106	109	10	**4.7**	#			
007	#				#				45.7	78	20	-0.1
008	101.49	85	10	-1.4	#				#			
009	135	95	10	-0.3	49.0	96	10	-0.3	50.8	83	10	0.4
010	122	72	10	-0.7	39	118	10	-1.1	33	69	10	-1.3
011	#				#				#			
012	256	168	5	**3.7**	85	170	50	**2.9**	55	91	5	0.8
013	121			-0.7	16			**-3.1**	#			
014	122	102	10	-0.7	57	106	10	0.4	35	95	10	-1.2
015	127	94	30	-0.5	64	108	21	1.1	70	73	15	**2.2**
016	140	96	5	-0.1	101	109	5	**4.3**	#			
017	#				#				44.3	82	20	-0.3
018	#				#				48.6	90	20	0.2
019	#				#				#			
020	110	78	10	-1.1	46.4	79	10	-0.5	#			
021	168	94	10	0.8	53	104	10	0.1	52	84	10	0.5
022	142.53	111	0.02	0.0	#				#			
023	150.04	90.90	10	0.2	#				#			
024	130	80.0	10	-0.4	51	95.2	10	-0.1	41	80.7	10	-0.6
025	#				17.7	107	10	**-3.0**	#			
026	124	104	10	-0.6	44	109	10	-0.7	28	96	10	-1.8
027	135		10	-0.3	37		10	-1.3	34		10	-1.3
028	183	105	10	1.3	56.7	94	10	0.4	#			

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

Table 2 (continued): Results and z-Scores for Pirimiphos-methyl, Propiconazole and Spinosad

laboratory number	analyte											
	pirimiphos-methyl assigned value 143.6 µg/kg				propiconazole assigned value 51.9 µg/kg				spinosad assigned value 46.9 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
029	120		10	-0.8	46		10	-0.5	50		10	0.3
030	112.0		10	-1.0	43.0		10	-0.8	0		10	**-4.5**
031	#				#				#			
032	170	91	10	0.9	65	98	10	1.1	#			
033	173.30		10	1.0	71.72		10	1.7	56.90		10	1.0
034	#				#				34	86	10	-1.3
035	160	90	10	0.5	55	90	10	0.3	41	90	10	-0.6
036	169.44		10	0.8	54.22		10	0.2	33.6		1	-1.3
037	97.3	69	10	-1.5	34.0	75	10	-1.6	20.0	68	10	**-2.6**
038	138	77	10	-0.2	51	85	10	-0.1	51	92	10	0.4
039	167	89	10	0.8	58	92	10	0.5	56	85	10	0.9
040	210	85	5	**2.2**	63	85	5	1.0	43	95	5	-0.4
041	155	113	10	0.4	56	118	10	0.4	55	124	10	0.8
042	105		10	-1.3	0		10	**-4.5**	19		10	**-2.7**
043	145.5	85	10	0.1	64.3	105	10	1.1	#			
044	228	104	20	**2.7**	58	104	20	0.5	#			
045	202.8	120.02	10	1.9	62.0	126.68	10	0.9	#			
046	#				73.8	118.1	10	1.9	#			
047	95	87.3	10	-1.6	35	90.9	10	-1.5	63	87.6	10	1.6
048	190			1.5	68			1.4	50			0.3
049	111	101	10	-1.1	49	94	10	-0.3	33	100	10	-1.3
050	107.00		10.00	-1.2	#				#			
051	120.4	41	28.0	-0.8	0			**-4.5**	#			
052	#				#				#			
053	123.36	74.8	50	-0.7	#				#			
054	149	86	10	0.2	44	91	10	-0.7	35	80	10	-1.2
055	149	104	10	0.2	65	98	10	1.1	106	102	10	**5.7**
056	171.3	100	10	0.9	#				47.2	100	10	0.0

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

Table 2 (continued): Results and z-Scores for Pirimiphos-methyl, Propiconazole and Spinosad

laboratory number	analyte											
	pirimiphos-methyl assigned value 143.6 µg/kg				propiconazole assigned value 51.9 µg/kg				spinosad assigned value 46.9 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
057	155	84	10	0.4	53	117	10	0.1	56	108	10	0.9
058	125	99	40	-0.6	52	139	10	0.0	#			
059	106.9	80	10	-1.2	35.3	79	10	-1.5	NQ		10	
060	120	100	10	-0.8	38	97	10	-1.2	0			**-4.5**
061	94	94	10	-1.6	17	86	10	**-3.1**	23	118	10	**-2.3**
062	170	121	10	0.9	50	120	10	-0.2	50	106	10	0.3
063	184	90-110	10	1.3	53	90-110	10	0.1	47	90-110	10	0.0
064	182	71	10	1.2	23	112	20	**-2.5**	44	75	10	-0.3
065	#				#				#			

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

NQ = not quantified

Table 3: Additional Pesticide Residues Reported

laboratory number	pesticide residue >30 µg/kg	result µg/kg	recovery %	LoQ µg/kg
043	cyfluthrin	99.9	89	10
043	methoxyfenozide	135	50	10
043	propamocarb	117	0	10
043	quinalphos	209	0	10

Table 4: Participants' Comments

participant number	comments
003	The result of Spinosad is a sum of isomers(Spinosyn A:51.0 ?g/kg, Spinosyn D:14.4 ?g/kg)
016	The result of Propiconazole is the sum of Cis- and Trans- Propiconazole.
038	spinosad = spinosyn A x 0.95 + spinosyn D x 0.05
059	Spinosad was detected but not quantified due to chromatographic interference
060	Traces of Endosulfansulfat at the blank

comments are as submitted by participants

Table 5: Assigned Values and Standard Deviations for Proficiency

analyte	data points, n	assigned value, x_a, µg/kg	uncertainty, u	standard deviation for proficiency, σ_p, µg/kg	
chlorpyrifos-methyl	44	39.1	1.5	Horwitz [6]	8.6
indoxacarb	35	101.1	3.3	Horwitz [6]	22.2
omethoate	31	97.9	4.5	Horwitz [6]	21.5
pirimiphos-methyl	37	143.6	5.2	Horwitz [6]	30.8
propiconazole	35	51.9	2.5	Horwitz [6]	11.4
spinosad	27	46.9	2.4	Horwitz [6]	10.3

Table 6: Number and Percentage of z-Scores where |z| ≤2

analyte	number of scores where |z| ≤2	total number of scores	% |z| ≤2
chlorpyrifos-methyl	52	59	88
indoxacarb	44	47	94
omethoate	36	46	78
pirimiphos-methyl	49	53	92
propiconazole	39	49	80
spinosad	32	40	80

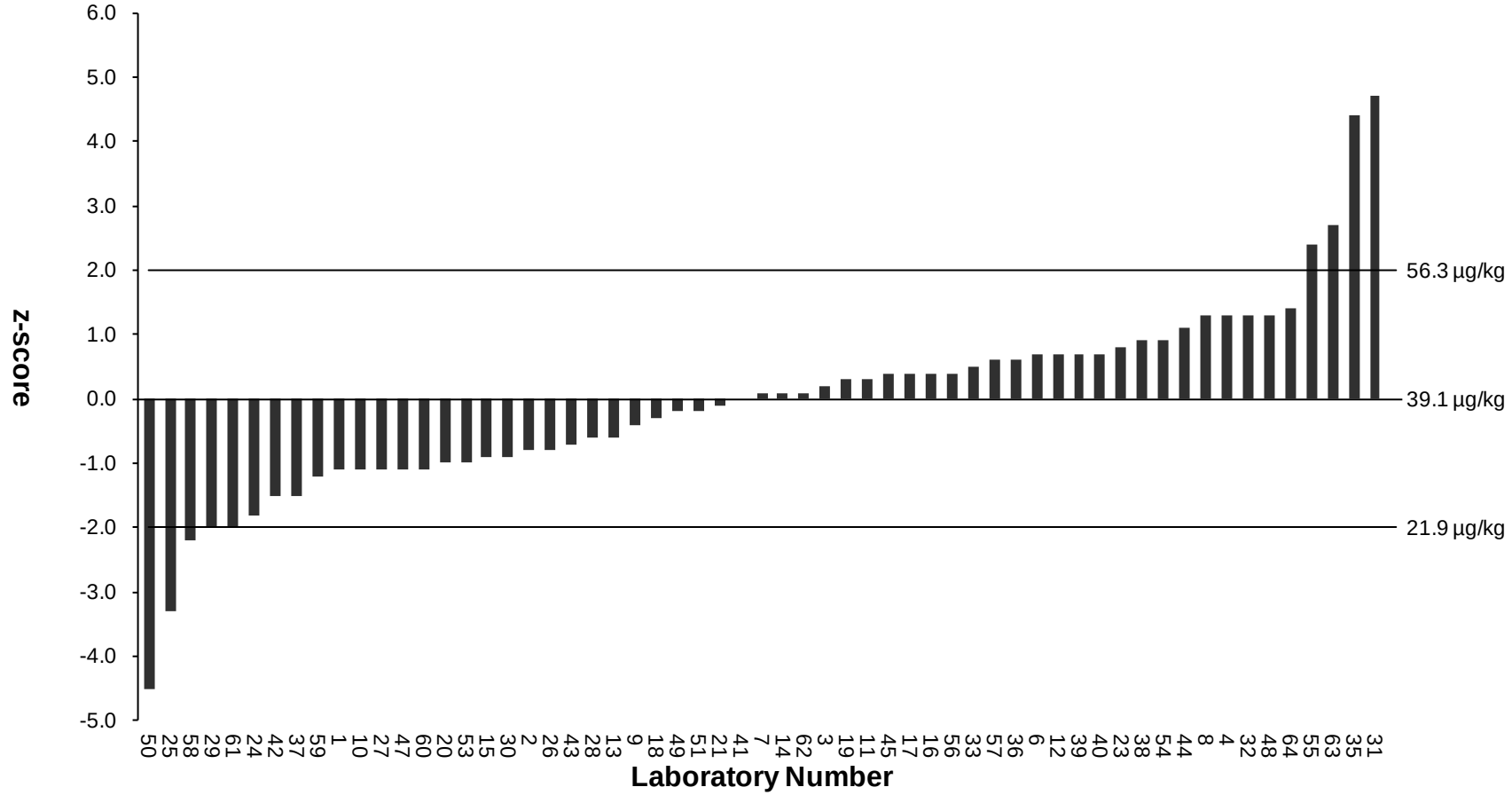

Figure 1: z-Scores for Chlorpyrifos-methyl

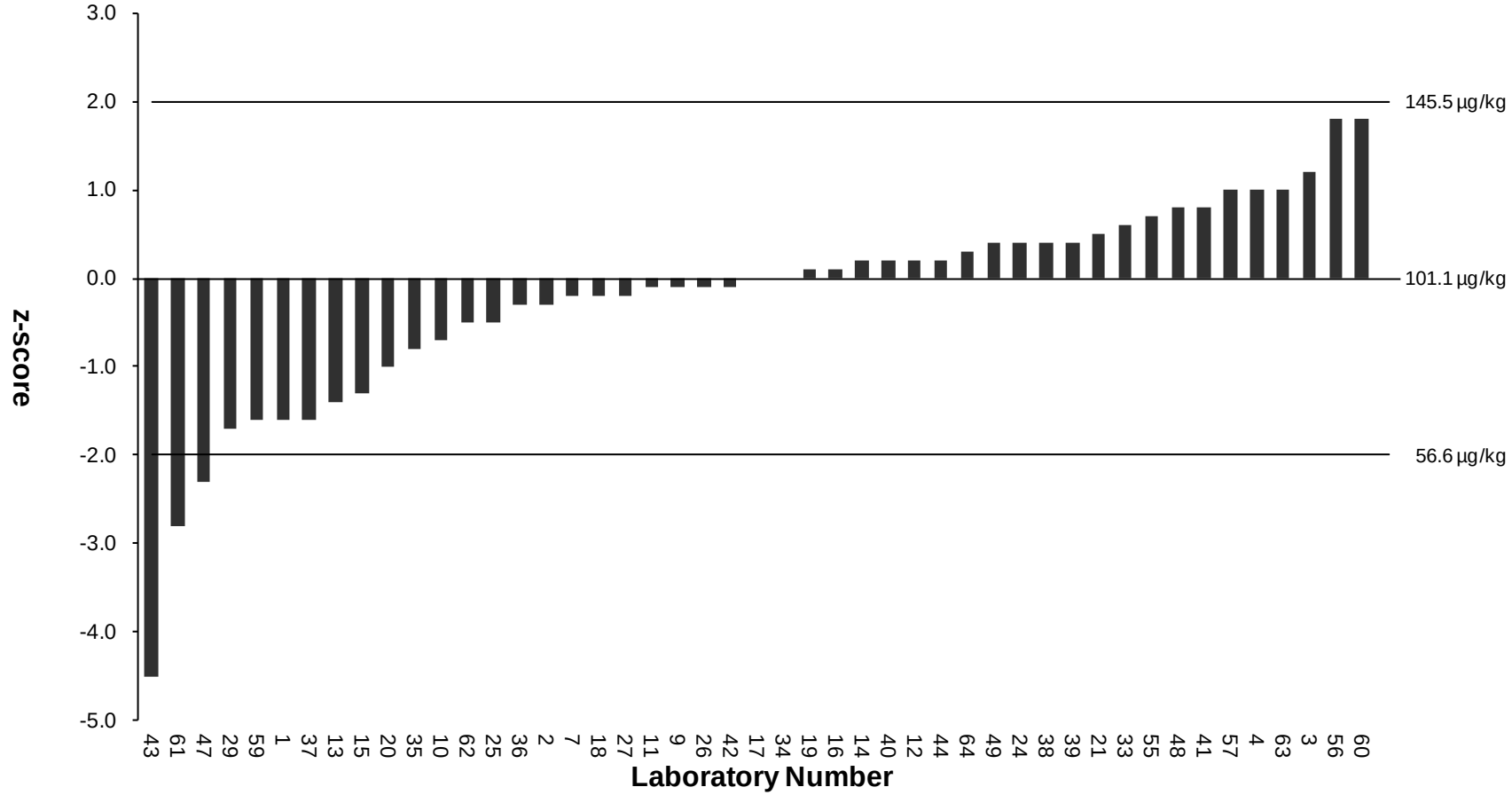

Figure 2: z-Scores for Indoxacarb

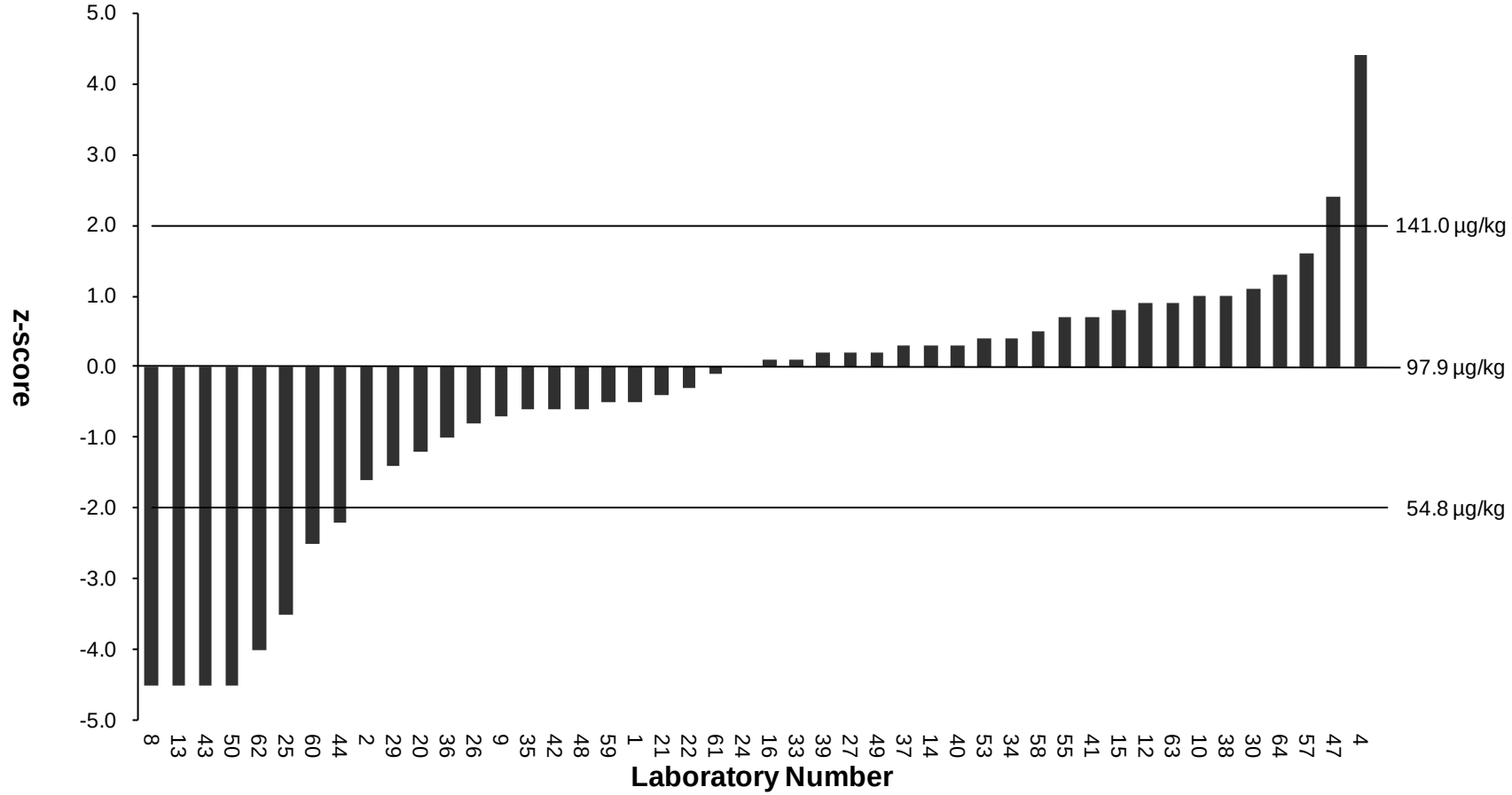

Figure 3: z-Scores for Omethoate

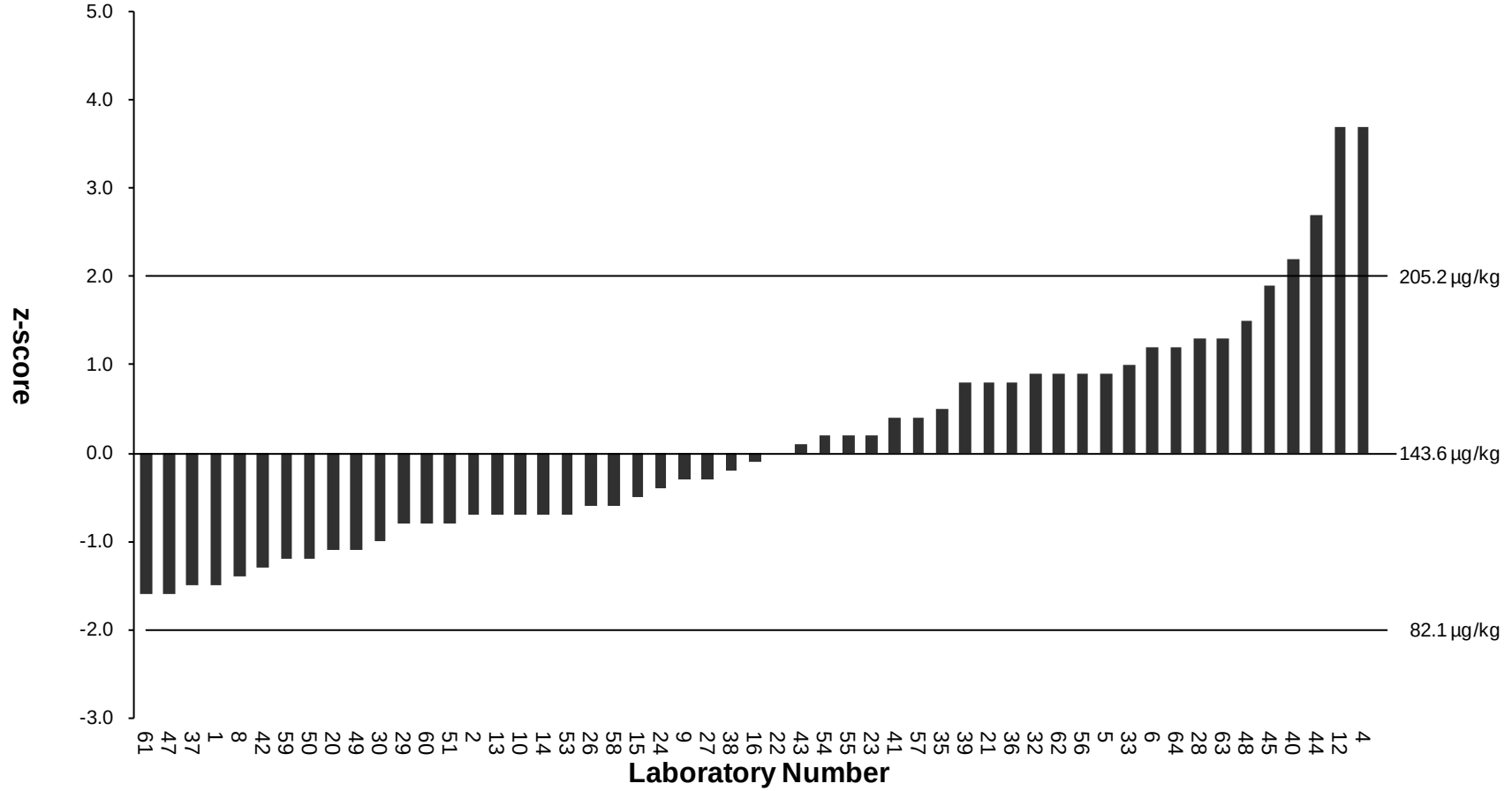

Figure 4: z-Scores for Pirimiphos-methyl

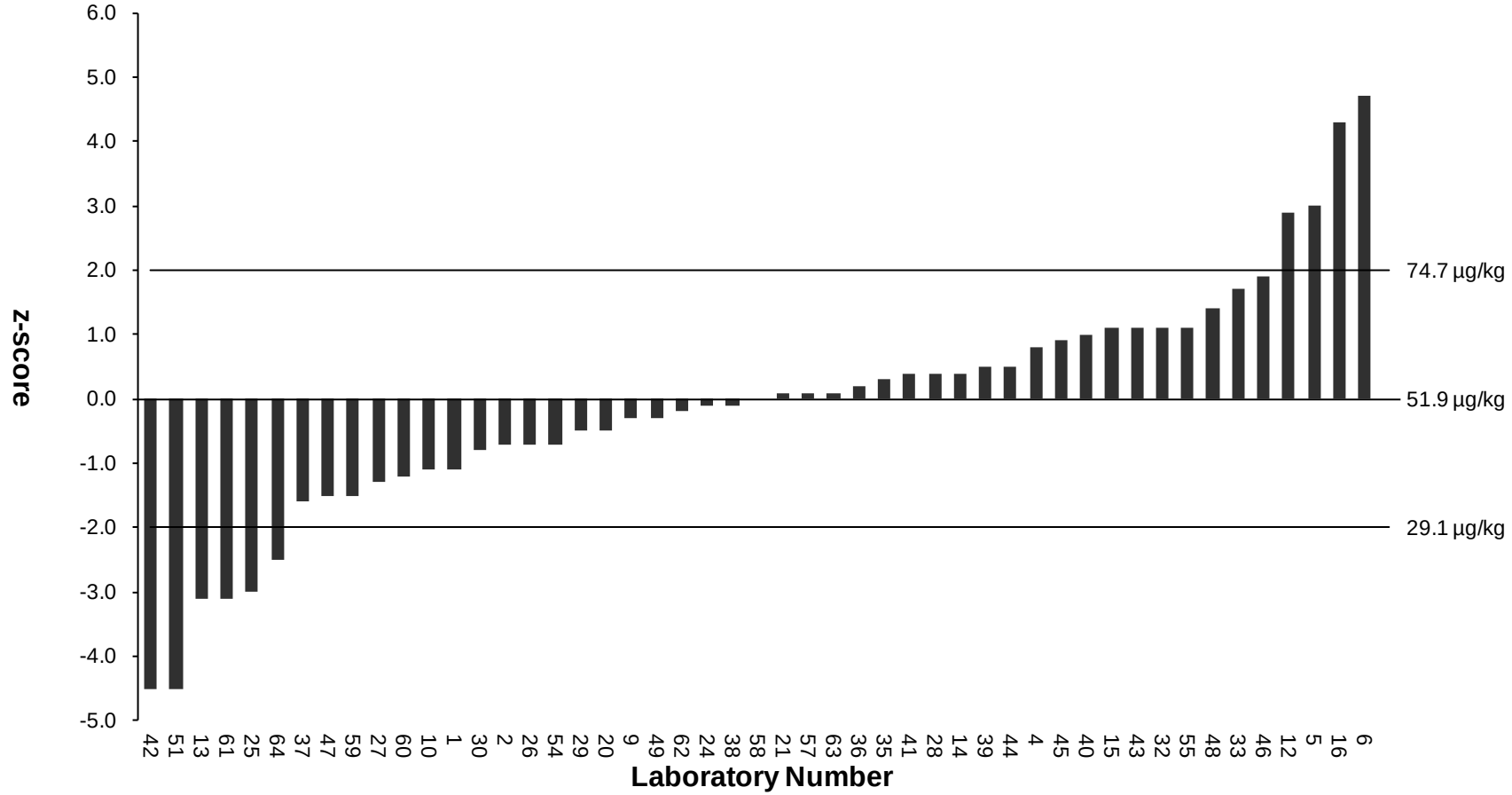

Figure 5: z-Scores for Propiconazole

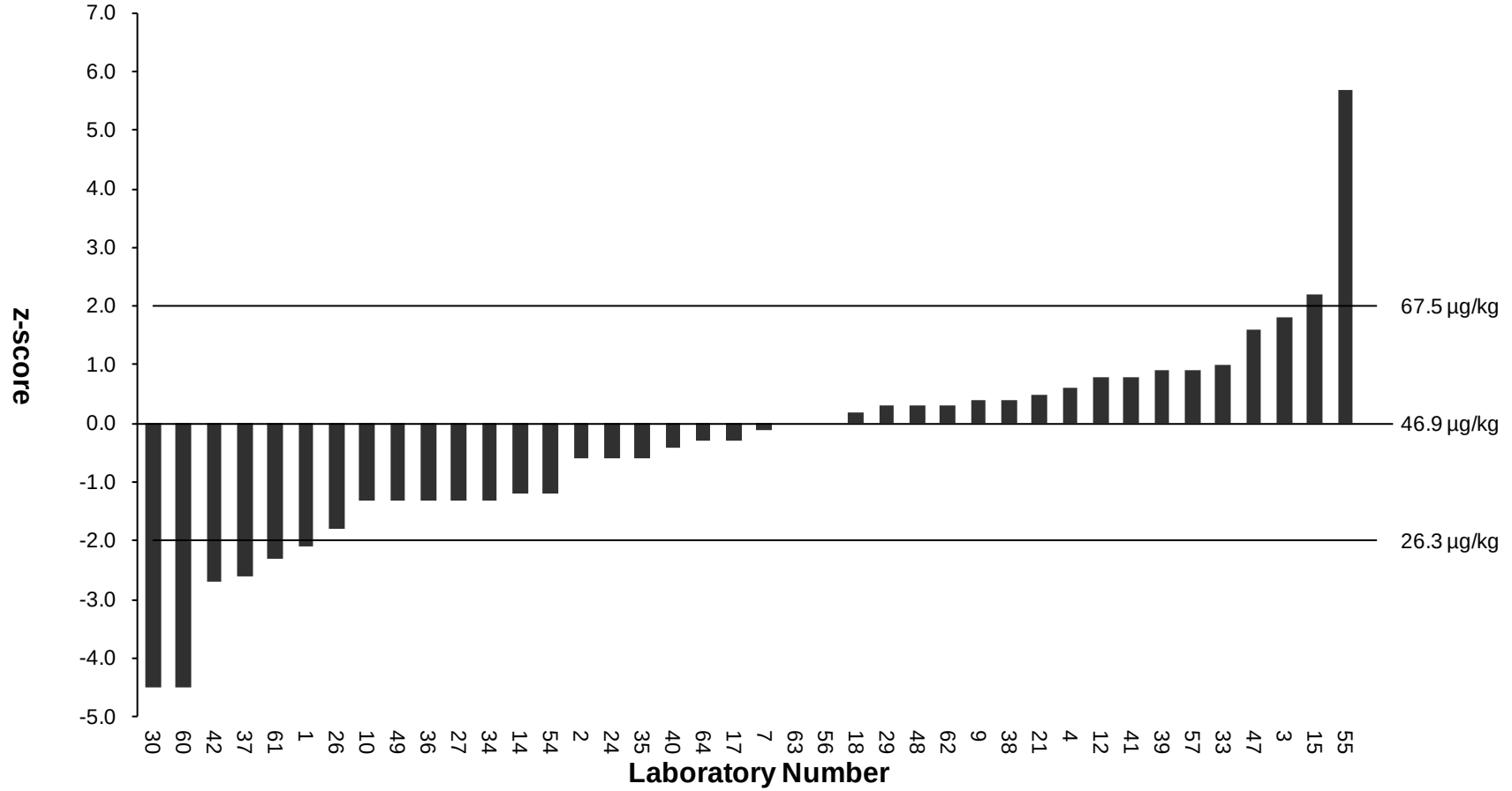

Figure 6: z-Scores for Spinosad

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.

Chlorpyrifos-methyl

Accredited Method Used	laboratory number
yes	001 002 003 006 007 008 009 010 011 013 014 015 017 018 019 020 021 023 025 030 032 033 036 037 038 039 040 041 043 044 047 048 049 053 056 057 060 062 063 064
no	004 012 016 024 027 028 029 031 045 051 058

Sample Weight (g)	laboratory number
≥2 - <5	004 013 030
≥5 - <10	008 012 016 028 040 048 056 060 062
≥10 - <25	002 003 006 007 009 010 011 014 015 017 018 019 020 021 023 024 025 027 029 031 032 036 037 038 039 041 047 049 057 058 063 064
≥25 - <50	001 043 045 053
≥50	033 044 051

Extraction Solvent Components	laboratory number
acetic acid	004 015
acetone	006 033 040 051 053 057 063
acetonitrile	003 004 007 008 009 011 012 014 015 016 017 018 019 020 021 023 024 025 027 028 029 031 032 036 039 041 044 045 049 058 060 062
cyclohexane	040 063 064
dichloromethane	030 053 057

Extraction Solvent Components (continued)	laboratory number
ethyl acetate	001 002 010 013 037 038 040 043 048 063
hexane	047 051
methanol	056
petroleum ether/spirit	057
water	004

Extraction Technique Used	laboratory number
hot solvent extraction at atmospheric pressure (e.g. Soxhlet)	048
cold solvent extraction at atmospheric pressure	001 002 003 004 007 008 009 010 011 012 013 014 016 018 019 020 021 023 024 025 028 029 030 032 033 037 038 040 041 043 044 045 049 056 057 058 060 062 063
solvent extraction at increased pressure/ PLE	017 051 053
Luke	064
QuEChERS	015 027

Extraction pH Adjusted	laboratory number
yes	001 003 007 010 019 027 028 032 037 038 040 047 049 058 060
no	002 004 006 008 009 011 012 013 014 015 016 017 018 020 021 023 024 025 029 030 031 033 036 039 041 043 044 045 048 051 053 056 057 062 063 064

Sample Clean-up Technique	laboratory number
carbon based column	001 012 017 024 036
extraction	013 015 039
filter	037 057
GPC/HPGPC	010 040
liquid/liquid extraction	003 006 007 016 023 024 033 040 045
NH2/aminopropyl column	012 024 036 044 062
silica column	040
solid phase extraction (SPE) (column/cartridge)	003 007 009 011 018 019 021 023 024 025 032 043 047 048 053 056
solid phase extraction (SPE) (dispersive)	004 008 014 027 028 029 041 049 058 060

Sample Clean-up Technique (continued)	laboratory number
solvent exchange	003
none	002 030 038 063 064
Gr+NH2	020
MgSO4	031

SPE Sorbent Type	laboratory number
C18	021 024 048 056
Envicarb/GCB	009 019 023 043 047
NH2	017 025 044 062
PSA	004 008 014 028 029 031 032 041 049 058 060
silica	040 053
C18/PSA/GCB	027
Carbon/NH2	003 045
ENVICarb(GCB/NH2)	007
ENVI-Carb/LC-NH2	012
Envicarb/NH2	011 018
Gr+NH2	020
PSA and MgSO4	015
QuEChERS Extract tube	016

Certified Standards Used	laboratory number
yes	002 004 006 008 010 011 012 013 014 015 017 018 019 020 021 023 025 028 029 030 031 032 033 036 037 038 039 040 041 043 044 048 049 051 053 056 057 058 060 062 063 064
no	001 003 007 009 016 024 027 045

MS Confirmation	laboratory number
yes	001 002 003 004 007 008 009 010 011 012 013 014 015 017 018 019 020 021 023 024 025 027 028 029 030 031 032 033 036 037 038 039 040 041 043 044 045 047 048 049 051 053 056 057 058 060 062 063 064
no	006 016

Calibrations	laboratory number
solvent	004 012 028 057
matrix-matched	001 002 006 008 009 010 015 016 017 020 023 024 025 029 030 036 037 038 041 043 044 047 049 058 062
single-level	004 029 037 040 044 051 060
multi-level	002 003 007 008 010 012 013 015 021 024 031 032 033 038 045 049 053 056 057 058 063 064
standard addition	003 007 011 014 018 019 027 039 048 056

Source of Standards	laboratory number
Accu Standard	016 058
Dr Ehrenstorfer	002 004 008 010 012 013 015 028 029 030 031 032 033 037 038 039 040 041 043 047 048 049 053 056 057 060 063 064
Fluka	051 057 062
Sigma/Aldrich	004 039 051 057
Supelco	051
Wako	003 007 009 011 018 019 021 023 045
Cica	017
EPA Research Triangle Park N.C.	044
Hayashi	027 036
HPE	025
Kantoukagaku	020
Neochema	014
Riedel-de Haen	024

Quoted percentage recovery measured in same analytical batch as test material	laboratory number
yes	002 003 004 006 007 010 011 012 014 015 016 018 019 020 021 023 024 028 030 031 032 033 037 038 040 041 043 044 045 047 049 051 053 057 058 062 063 064
no	008 009 013 017 025 027 029 039 048 056 060

If measured when was the spike added	laboratory number
prior to extraction	002 003 004 006 007 009 010 011 012 014 015 016 017 018 019 020 021 023 024 025 028 030 031 032 033 037 038 040 041 043 044 045 047 048 049 051 053 057 058 062 063 064

Level of Spike (µg/kg)	laboratory number
<25	002 010 023 025 030 031 033 037 048 056 064
≥25 - <50	008 012 014 024 048 051 063
≥50 - <100	004 006 009 014 016 021 028 038 040 041 044 047 048 049 053 057 058 062 063
≥100 - <150	014 015 020 043 045 048 063
≥150 - <200	048 063
≥200 - <250	003 007 011 017 018 019 032
≥500	056

Composition of Blank Commodity used for Spiking	laboratory number
blank green bean provided	002 006 008 009 012 014 016 017 018 020 023 024 025 028 030 031 032 037 038 039 041 043 044 045 048 049 051 053 057 058 062 063
green bean test material provided	004 021 040 047
green bean	007 010 011 019 030
Yard long bean	015
a tomato	033
Own blank material	064
Previously analysed sample	056

GC Column Type	laboratory number
capillary	001 002 003 006 008 009 010 011 012 015 017 018 019 020 021 023 024 025 027 028 030 031 032 033 037 038 039 041 043 044 045 047 051 053 056 058 060 062 063 064
narrowbore	014 016 040 048 057

GC Column Packing	laboratory number
100% methyl polysiloxane	016 051
14% cyanopropylphenyl 86%methyl polysiloxane	043 044
50% methyl 50% phenyl polysiloxane	030 045
95% methyl 5% phenyl polysiloxane	001 002 003 007 008 009 010 011 012 014 017 018 019 020 021 023 024 025 027 028 031 032 033 037 038 039 040 041 048 053 056 057 060 062 063 064
DB1701	015
HP-5MS	006
Thermo Pest	058

GC Injection Volume (µL)	laboratory number
<1	028
≥1 - <2	006 008 009 010 012 015 016 020 023 030 033 041 043 044 045 047 048 051 053 056 058 062 063
≥2 - <5	002 003 007 011 017 018 019 024 025 027 032 037 039 040 060
≥5 - <10	013 014 038 057
≥10	001 021 031 064

GC Injection Mode	laboratory number
on-column	010
PTV	001 014 016 021 027 038 039 040 057 060
split	044 064
splitless	003 006 007 008 009 011 012 015 017 018 019 020 023 024 025 028 030 031 032 033 037 043 045 047 048 051 053 056 058 062
KAS	041
LVI	002
solvent vent	063

GC Detector	laboratory number
ECD	033 040 051 053 057
FPD	015 021 045 051 053 060
ITD	002 057 058
MS	003 006 007 008 009 011 012 014 017 018 019 020 028 030 032 033 040 043 044 047 048 056 060 062 063 064
MS-MS	001 010 013 016 023 024 025 027 031 037 038 039 040 041 057
NPD	040

HPLC Column Packing	laboratory number
C18	004 014 016 029 039 047 048 049 057 062
endcapped	014

HPLC Guard Column Used	laboratory number
yes	004 013 014 016 025 029 039 041 047 048 056 057
no	002 031 049 062

Mobile Phase Programme	laboratory number
gradient	004 013 014 016 025 029 039 041 047 048 049 056 057 062

Mobile Phase Components	laboratory number
acetate	014
acetic acid	029 039 041
acetonitrile	014 025 039 048 062
formic acid	049
methanol	004 029 047 049 057
water	004 014 029 048 049 057
ammonium acetate buffer	029
methanol/water	016

HPLC Column Temperature (°C)	laboratory number
>ambient - <50	004 014 016 025 029 039 047 048 049 057
≥50	062

HPLC Injection Volume (µL)	laboratory number
<5	047 062
≥5 - <10	004 016 048 057
≥10 - <25	014 025 049
≥25 - <50	029
≥100 - <150	039

Mobile Phase Flow Rate (mL/min)	laboratory number
<0.25	025 047
≥0.25 - <0.75	004 014 016 039 048 049 057 062
≥0.75 - <1.25	029

HPLC Detector Type	laboratory number
MS-MS	004 014 016 025 029 039 047 049 056 057 062

Indoxacarb

Accredited Method Used	laboratory number
yes	001 002 003 007 009 010 011 013 014 017 018 019 020 021 025 033 037 038 039 040 041 044 047 048 049 056 057 060 062 063 064
no	004 012 015 016 024 027 029 034

Sample Weight (g)	laboratory number
≥2 - <5	004 013
≥5 - <10	012 015 016 034 040 048 056 060 062

Sample Weight (g) (continued)	laboratory number
≥10 - <25	002 003 007 009 010 011 014 018 019 020 021 024 025 027 029 033 037 038 039 041 047 049 057 063 064
≥25 - <50	001 017
≥50	044

Extraction Solvent Components	laboratory number
acetic acid	004 040
acetone	057 063
acetonitrile	003 004 007 009 011 014 015 016 017 018 019 020 021 024 025 027 029 033 034 038 039 040 041 044 049 060 062
cyclohexane	063 064
dichloromethane	057
ethyl acetate	001 002 010 013 037 048 063
hexane	047
methanol	012 056
petroleum ether/spirit	057
water	004

Extraction Technique Used	laboratory number
hot solvent extraction at atmospheric pressure (e.g. Soxhlet)	048
cold solvent extraction at atmospheric pressure	001 002 003 004 007 009 010 011 012 013 014 016 018 019 020 021 024 025 029 033 034 037 038 040 041 044 049 056 057 060 062 063
solvent extraction at increased pressure / PLE	017
QuEChERS	015
Rotavapor	064

Extraction pH Adjusted	laboratory number
yes	003 007 010 019 027 037 038 047 049
no	002 004 009 011 012 013 014 015 016 017 018 020 021 024 025 029 033 034 039 040 041 044 048 056 057 060 062 063 064

Sample Clean-up Technique	laboratory number
carbon based column	017 024
extraction	013 039
filter	012 037 057
Gr+NH2	020
liquid/liquid extraction	003 007 011 016 024
NH2/aminopropyl column	024 044 062
solid phase extraction (SPE) (column/cartridge)	003 007 009 018 019 021 024 025 047 048 056
solid phase extraction (SPE) (dispersive)	004 014 015 027 029 033 034 040 041 049 060
solvent exchange	003
none	002 010 038 063 064

SPE Sorbent Type	laboratory number
C18	021 024 048 056
Envicarb/GCB	009 019 047 060
NH2	017 025 044 062
PSA	004 014 029 033 034 040 041 049
C18/PSA	015
C18/PSA/GCB	027
Carbon/NH2	003
Envicarb(GCB/NH2)	007
Envicarb/NH2	011
Gr+NH2	020
QuEChERS Extract tube	016

Certified Standards Used	laboratory number
yes	001 002 004 010 012 013 014 015 017 018 019 020 021 025 029 033 034 037 038 039 040 041 044 048 049 056 057 060 062 063 064
no	003 007 009 011 016 024 027

MS Confirmation	laboratory number
yes	002 003 004 007 009 010 011 012 013 014 015 017 018 019 020 021 024 025 027 029 033 034 037 038 039 040 041 044 047 048 049 056 057 060 062 063 064
no	016

Calibrations	laboratory number
solvent	004 012 057
matrix-matched	001 002 009 010 015 016 020 024 025 029 034 037 038 041 044 047 049 062
single-level	004 011 029 037 044 060
multi-level	002 003 007 010 012 013 017 018 019 021 024 033 038 040 049 056 057 063 064
standard addition	014 027 039 048 056

Source of Standards	laboratory number
Accu Standard	016
Chem-Serv	044
Cica	017
Dr Ehrenstorfer	002 004 010 013 015 029 033 037 038 039 040 041 047 048 049 056 057 060 063 064
Fluka	057 062
Hayashi Pure Chemical	024
HPE	025
Kantoukagaku	020
Neochema	014
O2SI	034
Sigma/Aldrich	004 039 057
Wako	003 007 009 011 012 018 019 021

Quoted percentage recovery measured in same analytical batch as test material	laboratory number
yes	002 003 004 007 010 011 012 014 015 016 018 019 020 021 024 033 034 037 038 040 041 044 047 049 057 062 063 064
no	009 013 017 025 027 029 039 048 056 060

If measured when was the spike added	laboratory number
prior to extraction	002 003 004 007 009 010 011 012 014 015 016 017 018 019 020 021 024 025 033 034 037 038 040 041 044 047 048 049 057 062 063 064

Level of Spike (µg/kg)	laboratory number
<25	002 010 025 033 048 056 064
≥25 - <50	014 024 048 063
≥50 - <100	004 009 014 015 016 021 037 040 041 047 048 049 057 062 063
≥100 - <150	012 014 020 034 038 044 048 063
≥150 - <200	048 063
≥200 - <250	003 007 011 017 018 019
≥500	056

Composition of Blank Commodity used for Spiking	laboratory number
blank green bean provided	002 009 012 014 015 016 017 018 020 024 025 034 037 038 039 041 044 048 049 057 062 063
green bean test material provided	004 021 040 047
green bean	003 007 010 011 019 033
in house	056 064

GC Column Type	laboratory number
capillary	002 009 020 025 039 044 047 056 060 062 063 064
narrowbore	014 016 048 057

GC Column Packing	laboratory number
100% methyl polysiloxane	016
14% cyanopropylphenyl 86%methyl polysiloxane	044
95% methyl 5% phenyl polysiloxane	002 009 014 020 025 039 048 056 057 060 062 063 064

GC Injection Volume (µL)	laboratory number
≥1 - <2	009 016 020 041 044 047 048 056 060 062 063
≥2 - <5	002 025 039
≥5 - <10	013 014 057
≥10	064

GC Injection Mode	laboratory number
LVI	002
PTV	014 016 027 039 057
solvent vent	063
split	044 064
splitless	009 020 025 047 048 056 060 062

GC Detector	laboratory number
ECD	057
ITD	002 057
MS	009 014 020 044 048 056 060 062 063 064
MS-MS	013 016 025 039 047 057

HPLC Column Packing	laboratory number
C18	001 003 004 007 010 011 012 014 015 017 018 019 021 024 025 027 029 037 038 039 040 048 049 057 062
C8	033 034 041
endcapped	014

HPLC Guard Column Used	laboratory number
yes	001 003 004 007 011 013 014 015 017 019 021 024 025 027 029 033 034 037 038 039 041 048 056 057 060
no	002 010 012 016 018 040 047 049 062

Mobile Phase Programme	laboratory number
gradient	001 003 004 007 010 011 012 013 014 015 016 017 018 019 021 024 025 027 029 033 034 037 038 039 040 041 047 048 049 056 057 060 062

Mobile Phase Components	laboratory number
acetate	010 014 015 027
acetic acid	029 039 041
acetonitrile	012 014 024 025 034 039 048 062
ammonium acetate	003 007 019
ammonium acetate buffer	029
ammonium formate	034 038 040
formic acid	024 034 038 041 049
methanol	001 003 004 007 010 011 012 016 017 018 019 021 027 029 033 037 038 041 049 057
water	001 003 004 007 010 014 016 027 029 033 034 041 048 049 057

HPLC Column Temperature (°C)	laboratory number
ambient	033 040
>ambient - <50	001 003 004 007 010 011 012 014 015 016 017 018 019 021 024 025 027 029 034 039 041 048 049 057
≥50	037 038 062

HPLC Injection Volume (µL)	laboratory number
<5	010 017 034 037 062
≥5 - <10	001 003 004 007 011 012 016 018 019 021 027 038 040 041 048 057
≥10 - <25	014 015 024 025 049

HPLC Injection Volume (µL) (continued)	laboratory number
≥25 - <50	029 033
≥100 - <150	039

Mobile Phase Flow Rate (mL/min)	laboratory number
<0.25	003 007 011 012 017 018 019 021 025 027 040 041
≥0.25 - <0.75	001 004 010 014 015 016 024 033 034 037 038 039 048 049 057 062
≥0.75 - <1.25	029

HPLC Detector Type	laboratory number
MS	003 007 011 018 019 027 033
MS-MS	001 004 010 012 014 015 016 017 021 024 025 029 034 037 038 039 040 041 049 056 057 062

Omethoate

Accredited Method Used	laboratory number
yes	001 002 009 010 014 015 020 021 025 030 033 037 038 039 040 041 044 047 048 049 057 058 060 062 063 064
no	004 012 016 022 024 027 029 034 053

Sample Weight (g)	laboratory number
≥2 - <5	004
≥5 - <10	012 016 034 040 048 060 062 063
≥10 - <25	002 009 010 014 015 020 021 024 025 027 029 030 037 038 039 041 047 049 057 058 064
≥25 - <50	001 022 053
≥50	033 044

Extraction Solvent Components	laboratory number
acetic acid	004 015 040
acetone	022 033 053 057
acetonitrile	004 014 015 016 020 021 024 025 027 029 034 038 039 040 041 044 049 060 062 063
dichloromethane	053 057
ethyl acetate	001 002 010 030 037 048
hexane	047
methanol	009 012 058 064
petroleum ether/spirit	057
water	004 058

Extraction Technique Used	laboratory number
ASE	030
cold solvent extraction at atmospheric pressure	001 002 004 009 010 012 014 016 020 021 024 025 029 033 034 037 038 040 041 044 049 057 058 060 062 063
hot solvent extraction at atmospheric pressure (e.g. Soxhlet)	048
QuEChERS	015 027 064

Extraction pH Adjusted	laboratory number
yes	010 027 037 038 047 049 060
no	002 004 009 012 014 015 016 020 021 022 024 025 029 030 033 034 039 040 041 044 048 053 057 058 062 063 064

Sample Clean-up Technique	laboratory number
carbon based column	024
extraction	015 039
Extrelut	009
filter	012 037 057 058
Gr+NH2	020
liquid/liquid extraction	016 024 033
NH2/aminopropyl column	024 044 062
silica column	053
solid phase extraction (SPE) (column/cartridge)	021 024 025 047 048
solid phase extraction (SPE) (dispersive)	004 014 027 029 034 040 041 049 060 064
none	002 010 030 038 063

SPE Sorbent Type	laboratory number
C18	021 024 048
Envicarb/GCB	047
NH2	025 044 062
PSA	004 014 029 034 040 041 049 060 064
C18/PSA/GCB	027
Gr+NH2	020
PSA and MgSO4	015
QuEChERS Extract tube	016

Certified Standards Used	laboratory number
yes	001 002 004 010 012 014 015 020 021 022 025 029 030 033 034 037 038 039 040 041 044 048 049 053 057 058 060 062 063 064
no	009 016 024 027

MS Confirmation	laboratory number
yes	002 004 009 010 012 014 015 020 021 024 025 027 029 030 033 034 037 038 039 040 041 044 047 048 049 053 057 058 060 062 063 064
no	016 022

Calibrations	laboratory number
matrix-matched	001 002 009 010 015 016 020 022 024 025 029 030 034 037 038 041 044 047 049 053 058 062
multi-level	002 010 012 015 021 024 033 038 040 049 053 057 058 063 064
single-level	004 029 037 044 060
solvent	004 012 057
standard addition	014 027 039 048

Source of Standards	laboratory number
Accu Standard	016 058
Chem-Serv	044
Dr Ehrenstorfer	002 004 010 015 022 029 030 033 037 038 039 040 041 047 048 049 057 060 063 064
Fluka	057 062
HPE	025
Kantoukagaku	020
Neochema	014
O2SI	034
Riedel-de Haen	024
Sigma/Aldrich	004 039 057
Wako	009 012 021 027

Quoted percentage recovery measured in same analytical batch as test material	laboratory number
yes	002 004 010 012 014 015 016 020 021 024 030 033 034 037 038 040 041 044 047 049 057 058 062 063 064
no	009 025 027 029 039 048 053 060

If measured when was the spike added	laboratory number
prior to extraction	002 004 009 010 012 014 015 016 020 021 024 025 030 033 034 037 038 040 041 044 047 048 049 053 057 058 062 063 064

Level of Spike (µg/kg)	laboratory number
<25	002 010 022 025 030 033 048 053 063 064
≥25 - <50	009 014 024 048 063
≥50 - <100	004 014 016 021 040 041 044 047 048 049 057 058 062 063
≥100 - <150	012 014 015 020 034 037 038 048 063
≥150 - <200	048 063

Composition of Blank Commodity used for Spiking	laboratory number
blank green bean provided	002 009 012 014 016 020 022 024 025 034 037 038 039 041 044 048 049 057 058 062 063
green bean test material provided	004 021 030 040 047
green beans	010
yard long bean	015
in house	064
tomato	033

GC Column Type	laboratory number
capillary	002 015 020 022 025 033 039 044 053 060 062
narrowbore	014 016 048 057

GC Column Packing	laboratory number
100% methyl polysiloxane	016
14% cyanopropylphenyl 86%methyl polysiloxane	044
95% methyl 5% phenyl polysiloxane	002 014 020 025 033 039 048 053 057 060 062
DB1701	015

GC Injection Volume (µL)	laboratory number
≥1 - <2	015 016 020 022 033 041 048 053 062
≥2 - <5	002 025 039 044 060
≥5 - <10	014 057

GC Injection Mode	laboratory number
PTV	014 016 027 039 057 060
splitless	015 020 022 025 033 044 048 053 062
LVI	002

GC Detector	laboratory number
ECD	033 057
FPD	015 022 044 053 060
ITD	002 057
MS	014 020 033 048 060 062
MS-MS	016 025 039 057

HPLC Column Packing	laboratory number
C18	001 004 009 010 012 014 016 021 024 025 027 029 037 038 039 040 047 048 049 057 058 062 063 064
C8	034 041
endcapped	014
BEH C18	030

HPLC Guard Column Used	laboratory number
yes	001 004 009 014 016 021 024 025 027 029 034 037 038 039 041 047 048 057 064
no	002 010 012 030 040 049 058 062 063

Mobile Phase Programme	laboratory number
gradient	001 004 009 010 012 014 016 021 024 025 027 029 030 034 037 038 039 040 041 047 048 049 057 058 062 063 064

Mobile Phase Components	laboratory number
acetate	010 014 027
acetic acid	029 039 041
acetonitrile	012 014 024 025 034 039 048 062 063

Mobile Phase Components (continued)	laboratory number
ammonium acetate buffer	029
ammonium formate	034 038 040
formic acid	009 024 034 038 041 049
methanol	001 004 009 010 012 016 021 027 029 037 038 041 047 049 057 058
water	001 004 010 014 016 027 029 030 034 041 048 049 057 058 063 064

HPLC Column Temperature (°C)	laboratory number
ambient	040 063
>ambient - <50	001 004 009 010 012 014 016 021 024 025 027 029 030 034 039 041 047 048 049 057 064
≥50	037 038 058 062

HPLC Injection Volume (µL)	laboratory number
<5	010 034 037 047 062
≥5 - <10	001 004 012 016 021 027 030 038 040 041 048 057 058
≥10 - <25	009 014 024 025 049 063
≥25 - <50	029
≥50 - <100	064
≥100 - <150	039

Mobile Phase Flow Rate (mL/min)	laboratory number
<0.25	009 012 021 025 027 040 041 047 064
≥0.25 - <0.75	001 004 010 014 016 024 030 034 037 038 039 048 049 057 058 062 063
≥0.75 - <1.25	029

HPLC Detector Type	laboratory number
MS	027 053
MS-MS	001 004 009 010 012 014 016 021 024 025 029 030 034 037 038 039 040 041 047 049 057 058 062 063 064

Pirimiphos-methyl

Accredited Method Used	laboratory number
yes	001 002 005 006 008 009 010 013 014 015 020 021 022 023 030 032 033 037 038 039 040 041 043 044 047 048 049 050 053 056 057 060 062 063 064
no	004 012 016 024 027 028 029 045 051 058

Sample Weight (g)	laboratory number
≥2 - <5	004 013 030
≥5 - <10	008 012 016 028 040 048 050 060 062
≥10 - <25	002 005 006 009 010 014 015 020 021 023 024 027 029 032 037 038 039 041 047 049 057 058 063 064
≥25 - <50	001 022 043 045 053
≥50	033 044 051 056

Extraction Solvent Components	laboratory number
acetic acid	004 006 015 056
acetone	022 033 040 051 053 057 063
acetonitrile	004 005 006 008 009 012 014 015 016 020 021 023 024 027 028 029 032 039 041 044 045 049 056 058 060 062
cyclohexane	040 063 064
dichloromethane	030 050 053 057
ethyl acetate	001 002 010 013 037 038 040 043 048 063
hexane	047 050 051
petroleum ether/spirit	057
water	004

Extraction Technique Used	laboratory number
hot solvent extraction at atmospheric pressure (e.g. Soxhlet)	048
cold solvent extraction at atmospheric pressure	001 002 004 005 008 009 010 012 013 014 016 020 021 023 024 028 029 030 032 033 037 038 040 041 043 044 045 049 050 056 057 058 060 062 063
solvent extraction at increased pressure / PLE	051 053
Luke	064
QuEChERS	015 027

Extraction pH Adjusted	laboratory number
yes	001 010 027 028 032 037 038 040 047 049 058 060
no	002 004 005 006 008 009 012 013 014 015 016 020 021 022 023 024 029 030 033 039 041 043 044 045 048 050 051 053 056 057 062 063 064

Sample Clean-up Technique	laboratory number
carbon based column	001 012 024
extraction	013 015 039
filter	037 057
GPC/HPGPC	010 040
Gr+NH2	020
liquid/liquid extraction	006 016 023 024 033 040 045
NH2/aminopropyl column	005 012 024 044 062
silica column	040
solid phase extraction (SPE) (column/cartridge)	009 021 023 024 032 043 047 048 053
solid phase extraction (SPE) (dispersive)	004 008 014 027 028 029 041 049 050 058 060
none	002 030 038 056 063 064

SPE Sorbent Type	laboratory number
C18	021 024 048
C18/PSA/GCB	027
Carbon/NH2	045
diatomaceous earth	050
Envicarb/GCB	005 009 023 043 047
ENVI-Carb/LC-NH2	012
Gr+NH2	020
NH2	044 062
PSA	004 008 014 028 029 032 041 049 058 060
PSA and MgSO4	015
QuEChERS Extract tube	016
silica	040 053

Certified Standards Used	laboratory number
yes	002 004 005 006 008 010 012 013 014 015 020 021 022 023 028 029 030 032 033 037 038 039 040 041 043 044 048 049 050 051 053 056 057 058 060 062 063 064
no	001 009 016 024 027 045

MS Confirmation	laboratory number
yes	001 002 004 005 008 009 010 012 013 014 015 020 021 023 024 027 028 029 030 032 033 037 038 039 040 041 043 044 045 047 048 049 050 051 053 056 057 058 060 062 063 064
no	006 016 022

Calibrations	laboratory number
matrix-matched	001 002 006 008 009 010 015 016 020 022 023 024 029 030 037 038 041 043 044 047 049 050 058 062
multi-level	002 005 008 010 012 013 015 021 024 032 033 038 045 049 053 057 058 063 064
single-level	004 029 037 040 044 051 060
solvent	004 012 028 057
standard addition	014 027 039 048 056

Source of Standards	laboratory number
Accu Standard	016 058
Dr Ehrenstorfer	002 004 008 010 012 013 015 022 028 029 030 032 033 037 038 039 040 041 043 047 048 049 050 053 056 057 060 063 064
Fluka	051 057 062
Hayashi	027
Kanto Reagents	005
Kantoukagaku	020
Neochema	014
Riedel-de Haen	024
Riedel-de-Hocu	044
Sigma/Aldrich	004 039 051 057
Supelco	051
Wako	009 021 023 045

Quoted percentage recovery measured in same analytical batch as test material	laboratory number
yes	002 004 006 010 012 014 015 016 020 021 022 023 024 028 030 032 033 037 038 040 041 043 044 045 047 049 050 051 053 056 057 058 062 063 064
no	005 008 009 013 027 029 039 048 060

If measured when was the spike added	laboratory number
prior to extraction	002 004 006 009 010 012 014 015 016 020 021 023 024 028 030 032 033 037 038 040 041 043 044 045 047 048 049 050 051 053 056 057 058 062 063 064

Level of Spike (µg/kg)	laboratory number
<25	002 006 010 022 023 030 033 048 056 064
≥25 - <50	008 012 014 024 048 051 063
≥50 - <100	004 009 014 016 021 028 037 040 041 047 048 049 053 057 058 062 063
≥100 - <150	014 015 020 043 045 048 050 063
≥150 - <200	038 048 063
≥200 - <250	032 044 056

Composition of Blank Commodity used for Spiking	laboratory number
blank green bean provided	002 008 009 012 014 016 020 022 023 024 028 030 032 037 038 039 041 043 044 045 048 049 051 053 057 058 062 063
green bean test material provided	004 021 040 047
green beans	010 050
yard long bean	015
in house	006 056 064
tomato	033

GC Column Type	laboratory number
capillary	001 002 005 006 008 009 010 012 015 020 021 022 023 024 027 028 030 032 033 037 038 039 043 044 045 049 050 051 053 058 060 062 063 064
narrowbore	014 016 040 048 057

GC Column Packing	laboratory number
100% methyl polysiloxane	016 051
14% cyanopropylphenyl 86%methyl polysiloxane	043 044
50% methyl 50% phenyl polysiloxane	030 045
95% methyl 5% phenyl polysiloxane	001 002 005 008 009 010 012 014 020 021 023 024 027 028 032 033 037 038 039 040 048 049 050 053 057 060 062 063 064
DB1701	015
Thermo Pest	058

GC Injection Volume (µL)	laboratory number
<1	028
≥1 - <2	006 008 009 010 012 015 016 020 022 023 030 033 041 043 044 045 048 049 050 051 053 058 062 063
≥2 - <5	002 005 024 027 032 037 039 040 060
≥5 - <10	013 014 038 057
≥10	001 021 064

GC Injection Mode	laboratory number
LVI	002
on-column	010
PTV	001 014 016 021 027 038 039 040 057 060
solvent vent	063
split	044 064
splitless	005 006 008 009 012 015 020 022 023 024 028 030 032 033 037 043 045 048 049 050 051 053 058 062

GC Detector	laboratory number
ECD	033 040 051 053 057
FPD	015 022 045 051 053 060
ITD	002 057 058
MS	005 006 008 009 012 014 020 028 030 032 033 040 043 044 048 050 060 062 063 064
MS-MS	001 010 013 016 021 023 024 027 037 038 039 040 049 056 057
NPD	040

HPLC Column Packing	laboratory number
C18	004 014 016 029 039 047 048 056 057 062
C8	041
endcapped	014

HPLC Guard Column Used	laboratory number
yes	004 013 014 016 029 039 041 047 048 056 057
no	002 049 062

Mobile Phase Programme	laboratory number
isocratic	049
gradient	004 013 014 016 029 039 041 047 048 056 057 062

Mobile Phase Components	laboratory number
acetate	014
acetic acid	029 039 041
acetonitrile	014 039 048 056 062
ammonium acetate buffer	029
formic acid	041 056
methanol	004 016 029 041 047 057
water	004 014 016 029 041 048 056 057

HPLC Column Temperature (°C)	laboratory number
>ambient - <50	004 014 016 029 039 041 047 048 056 057
≥50	062

HPLC Injection Volume (µL)	laboratory number
<5	047 062
≥5 - <10	004 016 041 048 056 057
≥10 - <25	014
≥25 - <50	029
≥100 - <150	039

Mobile Phase Flow Rate (mL/min)	laboratory number
<0.25	041 047
≥0.25 - <0.75	004 014 016 039 048 056 057 062
≥0.75 - <1.25	029

HPLC Detector Type	laboratory number
MS-MS	004 014 016 029 039 041 047 056 057 062

Propiconazole

Accredited Method Used	laboratory number
yes	001 002 005 006 009 010 013 014 020 021 025 030 032 033 037 038 039 040 041 043 044 047 048 049 057 058 060 062 063 064
no	004 012 015 016 024 027 028 029 045 046

Sample Weight (g)	laboratory number
≥2 - <5	004 013 030
≥5 - <10	012 016 028 040 048 060 062
≥10 - <25	002 005 006 009 010 014 015 020 021 024 025 027 029 032 033 037 038 039 041 046 047 049 057 058 063 064
≥25 - <50	001 043 045
≥50	044

Extraction Solvent Components	laboratory number
acetic acid	004 015 046
acetone	006 040 057 063
acetonitrile	004 005 009 012 014 015 016 020 021 024 025 027 028 029 032 033 039 041 044 045 046 049 060 062
cyclohexane	040 063 064
dichloromethane	030 057
ethyl acetate	001 002 010 013 037 038 040 043 048 063
hexane	047
methanol	058
petroleum ether/spirit	057
water	004 058

Extraction Technique Used	laboratory number
hot solvent extraction at atmospheric pressure (e.g. Soxhlet)	048
cold solvent extraction at atmospheric pressure	001 002 004 005 009 010 012 013 014 016 020 021 024 025 028 029 030 032 033 037 038 040 041 043 044 045 046 049 057 058 060 062 063
Luke	064
QuEChERS	015 027

Extraction pH Adjusted	laboratory number
yes	001 010 027 028 032 037 038 040 046 047 049
no	002 004 005 006 009 012 013 014 015 016 020 021 024 025 029 030 033 039 041 043 044 045 048 057 058 060 062 063 064

Sample Clean-up Technique	laboratory number
carbon based column	001 012 024
extraction	013 039
filter	037 057 058
GPC/HPGPC	040
Gr+NH2	020
liquid/liquid extraction	006 016 024 040 045
NH2/aminopropyl column	005 012 024 044 062
silica column	040
solid phase extraction (SPE) (column/cartridge)	009 021 024 025 032 043 047 048
solid phase extraction (SPE) (dispersive)	004 014 015 027 028 029 033 041 046 049 060
none	002 010 030 038 063 064

SPE Sorbent Type	laboratory number
C18	021 024 048
C18/PSA/GCB	027
Carbon/NH2	045
Envicarb/GCB	005 009 043 047 060

SPE Sorbent Type (continued)	laboratory number
ENVI-Carb/LC-NH2	012
Gr+NH2	020
NH2	025 044 062
PSA	004 014 015 028 029 032 033 041 046 049
QuEChERS Extract tube	016
silica	040

Certified Standards Used	laboratory number
yes	002 004 005 006 010 012 013 014 015 020 021 025 028 029 030 032 033 037 038 039 040 041 043 044 046 048 049 057 058 060 062 063 064
no	001 009 016 024 027 045

MS Confirmation	laboratory number
yes	001 002 004 005 009 010 012 013 014 015 020 021 024 025 027 028 029 030 032 033 037 038 039 040 041 043 044 045 046 047 048 049 057 058 060 062 063 064
no	006 016

Calibrations	laboratory number
matrix-matched	001 002 006 009 010 015 016 020 024 025 029 030 037 038 041 043 044 046 047 049 058 062
multi-level	002 005 010 012 013 015 021 024 032 033 038 045 049 057 058 063 064
single-level	004 029 037 040 044 060
solvent	004 012 028 057
standard addition	014 027 039 048

Source of Standards	laboratory number
Accu Standard	016 058
Ciba-Geigy	044
Dr Ehrenstorfer	002 004 010 012 013 015 028 029 030 032 033 037 038 039 040 041 043 046 047 048 049 057 060 063 064

Source of Standards (continued)	laboratory number
Fluka	057 062
Hayashi	027
Hayashi Pure Chemical	024
HPE	025
Kanto Reagents	005
Kantoukagaku	020
Neochema	014
Sigma/Aldrich	004 039 057
Wako	009 021 045

Quoted percentage recovery measured in same analytical batch as test material	laboratory number
yes	002 004 006 010 012 014 015 016 020 021 024 028 030 032 033 037 038 040 041 043 044 045 046 047 049 057 058 062 063 064
no	005 009 013 025 027 029 039 048 060

If measured when was the spike added	laboratory number
prior to extraction	002 004 006 009 010 012 014 015 016 020 021 024 025 028 030 032 033 037 038 040 041 043 044 045 046 047 048 049 057 058 062 063 064

Level of Spike (µg/kg)	laboratory number
<25	002 010 025 030 033 048 064
≥25 - <50	012 014 024 037 048 063
≥50 - <100	004 006 009 014 016 021 028 038 040 041 044 046 047 048 049 057 058 062 063
≥100 - <150	014 015 020 043 045 048 063
≥150 - <200	048 063
≥200 - <250	032

Composition of Blank Commodity used for Spiking	laboratory number
blank green bean provided	002 006 009 012 014 015 016 020 024 025 028 030 032 037 038 039 041 043 044 045 046 048 049 057 058 062 063
green bean test material provided	004 021 040 047
green bean	010 033
in house	064

GC Column Type	laboratory number
capillary	001 002 005 006 009 012 015 020 021 025 027 028 030 032 038 039 041 043 044 045 046 049 060 062 063 064
narrowbore	014 016 040 048 057

GC Column Packing	laboratory number
100% methyl polysiloxane	016
14% cyanopropylphenyl 86%methyl polysiloxane	043 044
50% methyl 50% phenyl polysiloxane	030
95% methyl 5% phenyl polysiloxane	001 002 005 009 012 014 015 020 021 025 027 028 032 038 039 040 041 045 046 048 049 057 060 062 063 064
HP-5MS	006

GC Injection Volume (µL)	laboratory number
<1	028
≥1 - <2	006 009 012 016 020 030 041 043 044 045 046 048 049 060 062 063
≥2 - <5	002 005 025 027 032 039 040
≥5 - <10	013 014 015 038 057
≥10	001 021 064

GC Injection Mode	laboratory number
KAS	041
LVI	002
PTV	001 014 015 016 021 027 038 039 040 057

GC Injection Mode (continued)	laboratory number
solvent vent	063
split	044 064
splitless	005 006 009 012 020 025 028 030 032 043 045 046 048 049 060 062

GC Detector	laboratory number
ECD	040 046 057
ITD	002 057
MS	005 006 009 012 014 015 020 028 030 032 040 043 044 048 060 062 063 064
MS-MS	001 013 016 021 025 027 038 039 040 041 049 057
NPD	040 045

HPLC Column Packing	laboratory number
C18	004 010 014 016 024 025 029 037 039 047 048 057 058 062
C8	033
endcapped	014

HPLC Guard Column Used	laboratory number
yes	004 013 014 016 024 025 029 033 037 039 041 047 048 057 060
no	002 010 049 058 062

Mobile Phase Programme	laboratory number
isocratic	049
gradient	004 010 013 014 016 024 025 029 033 037 039 041 047 048 057 058 060 062

Mobile Phase Components	laboratory number
acetate	010 014
acetic acid	029 039 041
acetonitrile	014 024 025 039 048 062

Mobile Phase Components (continued)	laboratory number
ammonium acetate buffer	029
ammonium formate	037
formic acid	024
methanol	004 010 016 029 033 037 047 057 058
water	004 010 014 016 029 033 048 057 058

HPLC Column Temperature (°C)	laboratory number
ambient	033
>ambient - <50	004 010 014 016 024 025 029 039 047 048 057
≥50	037 058 062

HPLC Injection Volume (µL)	laboratory number
<5	010 037 047 062
≥5 - <10	004 016 048 057 058
≥10 - <25	014 024 025
≥25 - <50	029 033
≥100 - <150	039

Mobile Phase Flow Rate (mL/min)	laboratory number
<0.25	025 047
≥0.25 - <0.75	004 010 014 016 024 033 037 039 048 057 058 062
≥0.75 - <1.25	029

HPLC Detector Type	laboratory number
MS	033
MS-MS	004 010 014 016 024 025 029 037 039 047 057 058 062

Spinosad

Accredited Method Used	laboratory number
yes	001 002 003 007 009 010 014 017 018 021 033 034 037 038 039 040 041 047 048 049 056 057 062 063 064
no	004 012 015 024 027 029

Sample Weight (g)	laboratory number
≥2 - <5	004
≥5 - <10	012 015 034 040 048 062 063
≥10 - <25	002 003 007 009 010 014 018 021 024 027 029 033 037 038 039 041 047 049 057 064
≥25 - <50	001 017
≥50	056

Extraction Solvent Components	laboratory number
acetic acid	004 040 056
acetone	057
acetonitrile	002 003 004 007 014 015 017 018 021 024 027 029 033 034 038 039 040 041 049 056 062 063
dichloromethane	057
ethyl acetate	001 010 037 048
hexane	047
methanol	009 012 064
petroleum ether/spirit	057
water	004

Extraction Technique Used	laboratory number
hot solvent extraction at atmospheric pressure (e.g. Soxhlet)	048
cold solvent extraction at atmospheric pressure	001 002 003 004 007 009 010 012 014 018 021 024 029 033 034 037 038 040 041 049 056 057 062 063
solvent extraction at increased pressure / PLE	017
QuEChERs	064
QuEChERS	015

Extraction pH Adjusted	laboratory number
yes	001 003 007 010 027 037 038 047 049
no	002 004 009 012 014 015 017 018 021 024 029 033 034 039 040 041 048 056 057 062 063 064

Sample Clean-up Technique	laboratory number
carbon based column	001 017 024
extraction	039
Extrelut	009
filter	012 037 057
liquid/liquid extraction	003 007 024
NH2/aminopropyl column	024 062
PSA	002
solid phase extraction (SPE) (column/cartridge)	003 007 018 021 024 047 048
solid phase extraction (SPE) (dispersive)	004 014 015 027 029 033 034 040 041 049 064
solvent exchange	003
none	010 038 056 063

SPE Sorbent Type	laboratory number
C18	021 024 048
Envicarb/GCB	047
NH2	017 062
PSA	002 004 014 029 033 034 040 041 049 064
C18/PSA	015

SPE Sorbent Type (continued)	laboratory number
C18/PSA/GCB	027
Carbon/NH2	003
ENVICarb(GCB/NH2)	007

Certified Standards Used	laboratory number
yes	002 004 010 012 014 015 017 018 021 029 033 034 037 038 039 040 041 048 049 056 057 062 063 064
no	001 003 007 009 024 027

MS Confirmation	laboratory number
yes	001 002 003 004 007 009 010 012 014 015 017 018 021 024 027 029 033 034 037 038 039 040 041 047 048 049 056 057 062 063 064

Calibrations	laboratory number
matrix-matched	001 002 009 010 015 024 029 034 037 038 041 047 049 062
multi-level	002 003 007 010 012 017 018 021 024 033 038 040 049 057 063 064
single-level	004 029 037
solvent	004 012 057
standard addition	014 027 039 048 056

Source of Standards	laboratory number
Cica	017
Dr Ehrenstorfer	002 004 010 015 029 033 037 038 039 040 041 047 048 049 056 057 063 064
Fluka	057 062
Hayashi	027
Hayashi Pure Chemical	024
Neochema	014
O2SI	034
Sigma/Aldrich	004 039 057
Wako	003 007 009 012 018 021

Quoted percentage recovery measured in same analytical batch as test material	laboratory number
yes	002 003 004 007 010 012 014 015 018 021 024 033 034 037 038 040 041 047 049 056 057 062 063 064
no	009 017 027 029 039 048

If measured when was the spike added	laboratory number
prior to extraction	002 003 004 007 009 010 012 014 015 017 018 021 024 033 034 037 038 040 041 047 048 049 056 057 062 063 064

Ratio of Isomers	laboratory number
80:20	009

Level of Spike (µg/kg)	laboratory number
<25	002 010 033 048 056 063 064
≥25 - <50	009 014 024 037 048 063
≥50 - <100	004 014 015 021 040 041 047 048 049 057 062 063
≥100 - <150	012 014 034 038 048 063
≥150 - <200	048 063
≥200 - <250	003 007 017 018 056

Composition of Blank Commodity used for Spiking	laboratory number
blank green bean provided	002 009 012 014 015 017 018 024 034 037 038 039 041 048 049 057 062 063
green bean test material provided	004 021 040 047
green bean	003 007 010 033
in house	056 034

GC Column Type	laboratory number
capillary	001 062
narrowbore	014 048 057

GC Column Packing	laboratory number
95% methyl 5% phenyl polysiloxane	001 014 048 057 062

GC Injection Volume (µL)	laboratory number
≥1 - <2	041 048 062
≥5 - <10	014 057
≥10	001

GC Injection Mode	laboratory number
PTV	001 014 027 057
splitless	048 062

GC Detector	laboratory number
ECD	057
ITD	057
MS	014 048 062 014 048 062
MS-MS	001 056 057

HPLC Column Packing	laboratory number
C18	002 003 004 007 009 010 012 014 015 017 018 021 024 027 029 037 038 039 040 047 048 049 056 057 062 063 064
C8	033 034 041
endcapped	014

HPLC Guard Column Used	laboratory number
yes	002 003 004 007 009 014 015 017 021 024 027 029 033 034 037 038 039 041 047 048 056 057 064
no	010 012 018 040 049 062 063

Mobile Phase Programme	laboratory number
gradient	002 003 004 007 009 010 012 014 015 017 018 021 024 027 029 033 034 037 038 039 040 041 047 048 049 056 057 062 063 064

Mobile Phase Components	laboratory number
acetate	010 014 015 027
acetic acid	029 039 041
acetonitrile	002 012 014 024 034 039 048 056 062 063
ammonium acetate	003 007
ammonium acetate buffer	029
ammonium formate	034 038 040
formic acid	002 009 024 034 038 041 049 056
methanol	003 004 007 009 010 012 017 018 021 027 029 033 037 038 041 047 049 057
water	002 003 004 007 010 014 027 029 033 034 041 048 049 056 057 063 064

HPLC Column Temperature (°C)	laboratory number
ambient	033 040 063
>ambient - <50	002 003 004 007 009 010 012 014 015 017 018 021 024 027 029 034 039 041 047 048 049 056 057 064
≥50	037 038 062

HPLC Injection Volume (µL)	laboratory number
<5	010 017 034 037 047 062
≥5 - <10	003 004 007 012 018 021 027 038 040 041 048 056 057
≥10 - <25	002 009 014 015 024 049 063
≥25 - <50	029 033
≥50 - <100	064
≥100 - <150	039

Mobile Phase Flow Rate (mL/min)	laboratory number
<0.25	002 003 007 009 012 017 018 021 027 040 041 047 064
≥0.25 - <0.75	004 010 014 015 024 033 034 037 038 039 048 049 056 057 062 063
≥0.75 - <1.25	029

HPLC Detector Type	laboratory number
MS	003 007 018 027 033
MS-MS	002 004 009 010 012 014 015 017 021 024 029 034 037 038 039 040 041 047 049 056 057 062 063 064

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.
- 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.

3. PROTOCOL

The Protocols [2, 3] 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 17043:2010

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

5. CONTACT DETAILS

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