



SUMMARY

1. The test material for FAPAS[®] proficiency test 1970 was dispatched in June 2007. Each participant received a lettuce purée test material to be analysed for pesticide residues. In total, 151 sets of test material were distributed to participants in 31 countries. Of these, 140 participants, i.e. 93%, returned results for some combination of the analytes within the time-scale demanded by the Scheme.
2. From a list of 66 possible pesticide residues, participants were requested to identify and quantify those present in the lettuce purée. The test material contained cypermethrin and methidathion.
3. The assigned value ($\hat{X}$) was calculated from the most appropriate measure of central tendency of participants' results [1, 2, 3].
4. The target standard deviation (σ_p) for each analyte was calculated using the appropriate form of the Horwitz equation [4] and in conjunction with the assigned value ($\hat{X}$) was used to derive a z-score for participants' results. z-Scores are considered satisfactory if $|z| \leq 2$.
5. Results for this proficiency test are summarised as follows:

analyte	assigned value, $\hat{X}$, µg/kg	number of satisfactory scores, $ z \leq 2$	total number of scores	satisfactory %
cypermethrin	602	59	89	66
methidathion	119	99	124	80

6. Surplus test materials are available for sale, see APPENDIX III.
7. Whereas this Report has been produced in good faith and in accordance with best industry practice, neither the Central Science Laboratory 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.

CONTENTS

1. INTRODUCTION	4
1.1. Proficiency Testing	4
2. TEST MATERIAL	4
2.1. Preparation	4
2.2. Homogeneity	5
2.3. Distribution	5
3. RESULTS	5
4. STATISTICAL EVALUATION OF RESULTS	6
4.1. Calculation of the Assigned Value, $\hat{X}$	6
4.2. Target Standard Deviation for the Test, σ_p	6
4.3. Individual z-Scores	7
5. REFERENCES	8
TABLES	
Table 1: Results and z-Scores for Lettuce Purée Test Material	9
Table 2: Additional Pesticide Residues Reported	14
Table 3: Assigned Values and Target Standard Deviations	14
Table 4: Number and Percentage of Satisfactory z-Scores	14
Table 5: Number and Percentage of Participants Correctly Identifying and Obtaining Satisfactory z-Scores for Pesticides Present >30 µg/kg	15
FIGURES	
Figure 1: z-Scores for Cypermethrin (602 µg/kg) in Lettuce Purée Test Material	16
Figure 2: z-Scores for Methidathion (119 µg/kg) in Lettuce Purée Test Material	17
APPENDICES	
APPENDIX I: Homogeneity Data for Lettuce Purée Test Material	18
APPENDIX II: Analytical Methods Used by Participants	19
APPENDIX III: FAPAS® SecureWeb, Reports and Protocol	39

1. INTRODUCTION

1.1. Proficiency Testing

The demand for independent proof of competence from regulatory bodies and customers means that proficiency testing is relevant to all laboratories testing food and feed for quality and safety in every country. Hence, it is a requirement of accreditation to ISO 17025 [5] that the laboratory takes part in a proficiency testing scheme, if a suitable scheme exists. Further, for laboratories entrusted with the official control of food and feeds, Article 12 of EU Regulation (EC) 882/2004 [6] requires such laboratories to be assessed and accredited in accordance with ISO 17025, i.e. proficiency testing is a legal requirement for these laboratories. Thus, together with the use of validated methods, proficiency testing is an essential element of laboratory quality assurance.

The analysis of an external quality check sample as part of a laboratory's routine procedures provides objective standards for individual laboratories to perform against and permits them to compare their analytical results with those from other laboratories. Such standards and comparisons can go beyond the actual chemical analysis. For example, the ability to report results in specified units and within a given time scale are important aspects of quality. Hence, participants in FAPAS® who submit results after the closing date of a proficiency test are only included in the statistical evaluation if there are extenuating circumstances.

It is important to understand the statistical limitations of this external means of quality assessment when gauging the competence of a laboratory. The results of a typical chemical analysis will be normally distributed. That is to say, the majority of results will be centred on a mean value but, inevitably, some results will lie at the extremes of the distribution. The statistics of a normal distribution mean that about 95% of data points will lie between a z-score of -2 and +2. Performance in a FAPAS® proficiency test, therefore, is considered 'satisfactory' if a participant's z-score lies within this range. It follows that if a participant's z-score lies outside $|z| > 2$ there is about a 1 in 20 chance that their result is in fact an acceptable result from the extreme of the distribution. If a participant's z-score lies outside $|z| > 3$ the chance that their result is actually acceptable is only about 1 in 300.

2. TEST MATERIAL

2.1. Preparation

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

Organic lettuces were obtained from a local supplier. They were chopped and homogenised using a Stephan UM44 homogeniser with dry ice and then puréed.

Sub-samples were taken to screen for the possible presence of incurred residues, and the remainder was stored at -20°C. No residues were detected at or above 30 µg/kg.

A standard solution of the spiking pesticides was prepared to give the following approximate final concentrations: cypermethrin 700 µg/kg and methidathion 130 µg/kg.

Ten bags of lettuce purée, each weighing ~3kg, were emptied into a Crypto-Peerless bowl mixer. After each bag a small portion of the standard solution was added and mixed for 10 minutes. The resultant mixture was then stirred for 24 hours.

Samples were distributed into labelled glass bottles with at least 100 g in each.

To prepare the blank samples, the remaining lettuces were homogenised using a Stephan UM44 homogeniser with dry ice, then puréed. The purée was placed in a Crypto-Peerless Bowl Mixer and mixed for 1 hour. Samples were distributed into labelled glass bottles with at least 100 g in each.

Samples were stored at –20°C prior to distribution.

2.2. Homogeneity

Nine randomly selected test materials were analysed in duplicate for both pesticides. The results, together with their statistical evaluation [7], are given in APPENDIX I. These data show sufficient homogeneity, and are not included in the subsequent calculation of the assigned values.

2.3. Distribution

The dispatch date was 18 June 2007. Each participant received an individually numbered lettuce purée test material packed with cooling blocks in an insulated box, together with a covering letter, instructions for electronic submission of results and methods and the results form, for participants with no internet access.

3. RESULTS

Participants were required to report which pesticide residues the lettuce purée had been analysed for, together with a limit of quantification (LoQ). For all pesticides found, the amount present (in µg/kg, uncorrected for recovery) together with the percentage recovery was requested. Results were submitted by 140 participants before the closing date for this proficiency test, 1 August 2007. Most pesticides were to be reported as parent compound only, but some, including cypermethrin, were to be reported as the sum of all isomers.

Each participant was given a laboratory number, assigned in order of receipt of results. The reported results for cypermethrin and methidathion are given in Table 1.

The analytical methods used by each participant for the above pesticide residues are summarised in APPENDIX II.

If a participant failed to identify the presence of cypermethrin and methidathion and their LoQ was *below* the level needed for a satisfactory z-score, then as required by the FAPAS® Protocol [1], the reported result was assigned a zero value.

If a participant failed to identify the presence of cypermethrin and methidathion and their LoQ was *above* the level needed for a satisfactory z-score, then the result was recorded as <LoQ.

Any participant identifying pesticides other than cypermethrin and methidathion at levels >30 µg/kg is listed in Table 2 together with the pesticides reported, level determined, % recovery and LoQ.

4. STATISTICAL EVALUATION OF RESULTS

The object of the statistical procedure employed is to obtain a simple and transparent result, which the participant and other interested parties can readily appreciate. Further details, including worked examples, are given in the FAPAS® Protocol [1]. The procedure follows that recommended in the IUPAC/ISO/AOAC International Harmonised Protocol for the Proficiency Testing of (Chemical) Analytical Laboratories [8].

4.1. Calculation of the Assigned Value, $\hat{X}$

The assigned value, $\hat{X}$, i.e. the best estimate of the true concentration of each analyte, was set as the consensus of the results submitted by participants. The procedure used to derive this consensus involved:

- Removing non valid data, i.e.:
 - i) results reported as approximately 10, 100 or 1000 x greater or smaller than the majority of submitted results (as these were considered to be reporting errors),
 - ii) results from participants *not* quoting a percentage recovery,
 - iii) results from participants whose recovery is outside the range 70 – 110% [9],
 - iv) results from participants not quoting an LoQ (limit of quantification),
 - v) results from participants that are lower than their reported LoQ,
- Considering the normality (Kolmogorov-Smirnov test), or otherwise, of the distribution of results.
- Minimising the influence of outliers by the use of a robust statistical procedure to derive the mean [3].
- Assessing the standard uncertainty (u) of the robust mean. For the robust mean:

$$u = \frac{\hat{\sigma}}{\sqrt{n}}$$

where $\hat{\sigma}$ = the standard deviation of the robust mean.

NB this is NOT the target standard deviation for the test (σ_p)

and n = the number of data points used to calculate the robust mean.

For both analytes, this procedure was straightforward. The robust mean was the best measure of central tendency and chosen as the assigned value.

The robust means used to set the assigned values for both analytes, together with u , n and $\hat{\sigma}$ are shown in Table 3.

4.2. Target Standard Deviation for the Test, σ_p

The value of σ_p determines the limits of satisfactory performance in a FAPAS® proficiency test. It is set at a value that reflects best practice for the analyses in question. The standard deviation of reproducibility found in collaborative trials is generally considered an appropriate indicator of the best agreement that can be obtained between laboratories. However, not all analyses have been characterised in this manner. In such cases, the

predictive models of the appropriate form of the Horwitz equation [4] are valuable indicators of best practise.

For both analytes, σ_p was derived from the appropriate form of the Horwitz equation [4]. This equation predicts a standard deviation from a given concentration, c , and requires c to be expressed as a dimensionless mass ratio, e.g. 1 ppm $\equiv 10^{-6}$ or % $\equiv 10^{-2}$. It follows therefore that to express the dimensionless standard deviation predicted by the equation in the original concentration units it must be divided by the relevant mass ratio:

- i) for analyte concentrations <120 ppb

$$\sigma_p = \frac{0.22c}{mr}$$

- ii) for analyte concentrations ≥ 120 ppb and $\leq 13.8\%$

$$\sigma_p = \frac{0.02c^{0.8495}}{mr}$$

- iii) for analyte concentrations >13.8%

$$\sigma_p = \frac{0.01c^{0.5}}{mr}$$

where, in all three cases, c = concentration, i.e. the assigned value, $\hat{X}$, expressed as a dimensionless mass ratio, e.g. 1 ppm $\equiv 10^{-6}$ or % $\equiv 10^{-2}$

and mr = dimensionless mass ratio, e.g. 1 ppm $\equiv 10^{-6}$ or % $\equiv 10^{-2}$

The values for σ_p used to calculate z-scores from the reported results of this test are given in Table 3.

4.3. Individual z-Scores

Participants' z-scores were calculated as:

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

where x = the participant's reported result,

$\hat{X}$ = the assigned value,

and σ_p = the target standard deviation.

Participants' z-scores for all analytes are given in Table 1 and shown as histograms in Figures 1 - 2. The number and percentage of z-scores in the satisfactory range, $|z| \leq 2$, for all analytes are given in Table 4.

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 target standard deviations prior to their publication in Table 3.

The number and percentage of participants correctly identifying both pesticides, and the number and percentage of participants obtaining satisfactory z-scores for cypermethrin and methidathion are shown in Table 5. In this case, participants identifying other pesticides at or >30 µg/kg are not considered satisfactory. This information is given for interest only and is not a measure of satisfactory performance in the proficiency test. Satisfactory performance is indicated in Table 1 and summarised in Table 4.

5. REFERENCES

- 1 FAPAS®, 2002, *Protocol for the Food Analysis Performance Assessment Scheme, Organisation and Analysis of Data*, 6th edition.
- 2 Lowthian, P.J. and Thompson, M., 2002, Bump-hunting for the proficiency tester-searching for multimodality, *Analyst*, **127**, 1359-1364.
- 3 Analytical Methods Committee, 1989, Robust Statistics – How not to reject outliers Part 1. Basic Concepts, *Analyst*, **114**, 1693-1697.
- 4 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.
- 5 ISO/IEC 17025:2005, General requirements for the competence of testing and calibration laboratories.
- 6 Regulation (EC) 882/2004 of the European Parliament and of the Council of 29 April 2004 on official controls performed to ensure the verification of compliance with feed and food law, animal health and animal welfare rules, *Official Journal L* **165**, 30/04/2004, 0001-0141.
- 7 Fearn, T. and Thompson, M., 2001, A new test for sufficient homogeneity, *Analyst*, **126**, 1414-1417.
- 8 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.
- 9 24 March 2006. *Quality Control Procedures for Pesticide Residues Analysis*. Document No SANCO/10232/2006 Available at http://ec.europa.eu/food/plant/resources/qualcontrol_en.pdf. Accessed 26 March 2007.

Table 1: Results and z-Scores for Lettuce Purée Test Material

laboratory number	analyte							
	cypermethrin assigned value 602 µg/kg				methidathion assigned value 119 µg/kg			
	result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score
001	362			-2.3	#			
002	† 815	75		2.1	202	85		3.2
003	460	114	10	-1.4	120	112	20	0.0
004	658	81	10	0.5	135		10	0.6
005	320	80	10	-2.7	130	80	20	0.4
006	676	120	25	0.7	128	105	10	0.4
007	748	78	50	1.4	107	87	50	-0.5
008	† 0	80-115	10	-5.8	#			
009	590	78.3	30	-0.1	160	101.5	20	1.6
010	630	20		0.3	350	20		8.8
011	591	85	10	-0.1	#			
012	831.7	94.5	10	2.2	140.7	100.9	5	0.8
013	553	106	10	-0.5	187	96	10	2.6
014	646.6			0.4	94.8			-0.9
015	849.42		20	2.4	146.07		20	1.0
016	486	82	20.0	-1.1	275	88	20.0	6.0
017	760	111	50	1.5	163	115	10	1.7
018	460	70	20	-1.4	160	70	20	1.6
019	423.0	100.0	25.0	-1.7	129.5	100.0	40.0	0.4
020	736	128	20	1.3	#			
021	838	133	30	2.3	145	76	30	1.0
022	#				100	94	53	-0.7
023	#				97	84	53	-0.8
024	#				99	84	53	-0.8
025	#				98	84	53	-0.8
026	#				90	71	53	-1.1
027	0			-5.8	200	//////	<10	3.1
028	#				227		100	4.1
029	660	105	50-100	0.6	155	98	10	1.4

z-scores outside the satisfactory range, i.e. $|z| > 2$, are shown in **bold** LoQ = limit of quantification
 # = pesticide not analysed for † = additional pesticides reported (see Table 2)

Table 1 (continued): Results and z-Scores for Lettuce Purée Test Material

laboratory number	analyte							
	cypermethrin assigned value 602 µg/kg				methidathion assigned value 119 µg/kg			
	result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score
030	440	85.8	5	-1.6	130	97.1	0.073	0.4
031	325.91	99	2.2	-2.7	318.63	73	11.6	7.6
032	#				#			
033	888.81	106.0	50	2.8	156.17	76.0	20	1.4
034	683	107.60	10	0.8	105	90.29	10	-0.5
035	#				89	80	9	-1.1
036	#				100	88	9	-0.7
037	#				93	74	9	-1.0
038	#				95	84	9	-0.9
039	760		10	1.5	150		10	1.2
040	#				110	89	20	-0.3
041	594	100	30	-0.1	95	100		-0.9
042	410.	56.6	10	-1.8	100.	100	10	-0.7
043	#				86	70	9	-1.3
044	694	108.0	20	0.9	139	100.0	10	0.8
045	#				120	91.4	10	0.0
046	#				110	87	10	-0.3
047	#				110	84	30	-0.3
048	423	112	10	-1.7	33	96.0	10	-3.3
049	#				120	73	30	0.0
050	#				110	113	30	-0.3
051	#				130	84.2	10	0.4
052	#				110	118	28	-0.3
053	750	98.8	50	1.4	120	88.7	10	0.0
054	#				120	90	30	0.0
055	#				110	77	30	-0.3
056	#				110	80	30	-0.3
057	610		50	0.1	130		10	0.4

z-scores outside the satisfactory range, i.e. $|z| > 2$, are shown in **bold**
 # = pesticide not analysed for

LoQ = limit of quantification

Table 1 (continued): Results and z-Scores for Lettuce Purée Test Material

laboratory number	analyte							
	cypermethrin assigned value 602 µg/kg				methidathion assigned value 119 µg/kg			
	result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score
058	#				110	102	30	-0.3
059	0.492	84.3	50	-5.8	0.129	69.4	20	-4.5
060	472	95	10	-1.2	98	97	10	-0.8
061	#				110	81	5.2	-0.3
062	405	76	5	-1.9	125	82	5	0.2
063	710	76	10	1.0	128	94	10	0.4
064	830	99.3	50	2.2	230	80	10	4.3
065	97	95	20	-4.9	148	97	10	1.1
066	1143.50	102.0		5.2	143.44	112.1		0.9
067	#				98	106	5	-0.8
068	442	123	50	-1.5	134	127	10	0.6
069	0.74	100.5	0.01	-5.8	#			
070	#				110	89.4	5.2	-0.3
071	#				110	112	5	-0.3
072	#				100	104	5	-0.7
073	#				120	76.0	5.2	0.0
074	565		10	-0.4	110		20	-0.3
075	947	96	50	3.3	130	81	20	0.4
076	600	86	10	0.0	120	114	10	0.0
077	549	94	10	-0.5	115	98	10	-0.1
078	753	102	20	1.5	122	98	20	0.1
079	#				120	101.8	10	0.0
080	320	110	10	-2.7	100	120	10	-0.7
081	611	96	25	0.1	116	87	10	-0.1
082	#				140	89	5.2	0.8
083	#				#			
084	589		10	-0.1	93		10	-1.0
085	708	92	5	1.0	#			

z-scores outside the satisfactory range, i.e. $|z| > 2$, are shown in **bold**
 # = pesticide not analysed for

LoQ = limit of quantification

Table 1 (continued): Results and z-Scores for Lettuce Purée Test Material

laboratory number	analyte							
	cypermethrin assigned value 602 µg/kg				methidathion assigned value 119 µg/kg			
	result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score
086	#				100	80	5	-0.7
087	#				100	80	4	-0.7
088	#				100	98	4	-0.7
089	#				94	72	4	-0.9
090	#				92	84	10	-1.0
091	470			-1.3	#			
092	2040	77	21	13.8	380	84	6	10.0
093	534.6	92	50	-0.6	112.8	72	20	-0.2
094	#				100	94	4	-0.7
095	562	85.8	2.0	-0.4	221	92.9	10.0	3.9
096	489	101	10	-1.1	94.5	99	5	-0.9
097	#				99	104	5	-0.8
098	#				100	95	5	-0.7
099	#				99	80	10	-0.8
100	787	80	50	1.8	339	85	20	8.4
101	582	106	10	-0.2	87.2	84	10	-1.2
102	† 1600	109	10	9.6	230	88	10	4.3
103	#				#			
104	438.61	>80	0.05	-1.6	168.74	>80	0.02	1.9
105	411	89	70	-1.8	92.5	97	17	-1.0
106	671	100	10	0.7	110	100	10	-0.3
107	653	358		0.5	121	137		0.1
108	#				110	74	4	-0.3
109	#				#			
110	368		10	-2.2	88	78.1	10	-1.2
111	536.31	90.15	20	-0.6	0	99	50	-4.5
112	950	100		3.4	121	100		0.1
113	645	95	10	0.4	272	101	10	5.9

z-scores outside the satisfactory range, i.e. $|z| > 2$, are shown in **bold** LoQ = limit of quantification
= pesticide not analysed for † = additional pesticides reported (see Table 2)

Table 1 (continued): Results and z-Scores for Lettuce Purée Test Material

laboratory number	analyte							
	cypermethrin assigned value 602 µg/kg				methidathion assigned value 119 µg/kg			
	result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score
114	760	151.2	20	1.5	160	124.5	2	1.6
115	760	151.2	20	1.5	100	124.5	2	-0.7
116	681.36	91.50	40	0.8	#			
117	668	96	1	0.6	99	78	1	-0.8
118	684	87		0.8	132	102		0.5
119	1105	95.1	5	4.8	123	106.3	5	0.2
120	450	106.3		-1.5	117	101		-0.1
121	22	100	10	-5.6	147	100	10	1.1
122	#				#			
123	238	82	40	-3.5	30	20	60	-3.4
124	† #				456	85	29	12.9
125	420	96		-1.7	100	104		-0.7
126	730	70-120	10	1.2	120	70-120	50	0.0
127	530	103	50	-0.7	193	128	20	2.8
128	#				162.5	85.0	20	1.7
129	536.4	84	50	-0.6	103.2	88	20	-0.6
130	#				#			
131	† 156.1		10.0	-4.3	0		10.0	-4.5
132	† 891	64	1.5	2.8	293	69	0.6	6.7
133	670			0.7	0			-4.5
134	690	80	10	0.9	120	80	10	0.0
135	0.86			-5.8	215			3.7
136	#				#			
137	200	90.9	10	-3.9	90	88.5	10	-1.1
138	#				#			
139	50			-5.3	56			-2.4
140	† 54	94	1.732	-5.3	2.0	83.7	0.3010	-4.5

z-scores outside the satisfactory range, i.e. $|z| > 2$, are shown in **bold** LoQ = limit of quantification
= pesticide not analysed for † = additional pesticides reported (see Table 2)

Table 2: Additional Pesticide Residues Reported

laboratory number	pesticide residue > 30 µg/kg	result µg/kg	recovery %	LoQ µg/kg
002	chlorpyrifos	42	82	
008	heptenophos	35	80-115	5
102	propargite	43	103	15
124	bromopropylate	31	120	20
131	folpet	180.3		10.0
132	permethrin	48	87	1.5
140	bifenthrin	46		0.204

Table 3: Assigned Values and Target Standard Deviations

analyte	assigned value, µg/kg				target standard deviation, µg/kg	
	data points	robust mean	robust sd	uncertainty	derived from	σ_p
	n	$\hat{X}$	$\hat{\sigma}$	u		
cypermethrin	49	602	194	27.7	Horwitz*	103.9
methidathion	84	119	27.2	2.97	Horwitz*	26.1

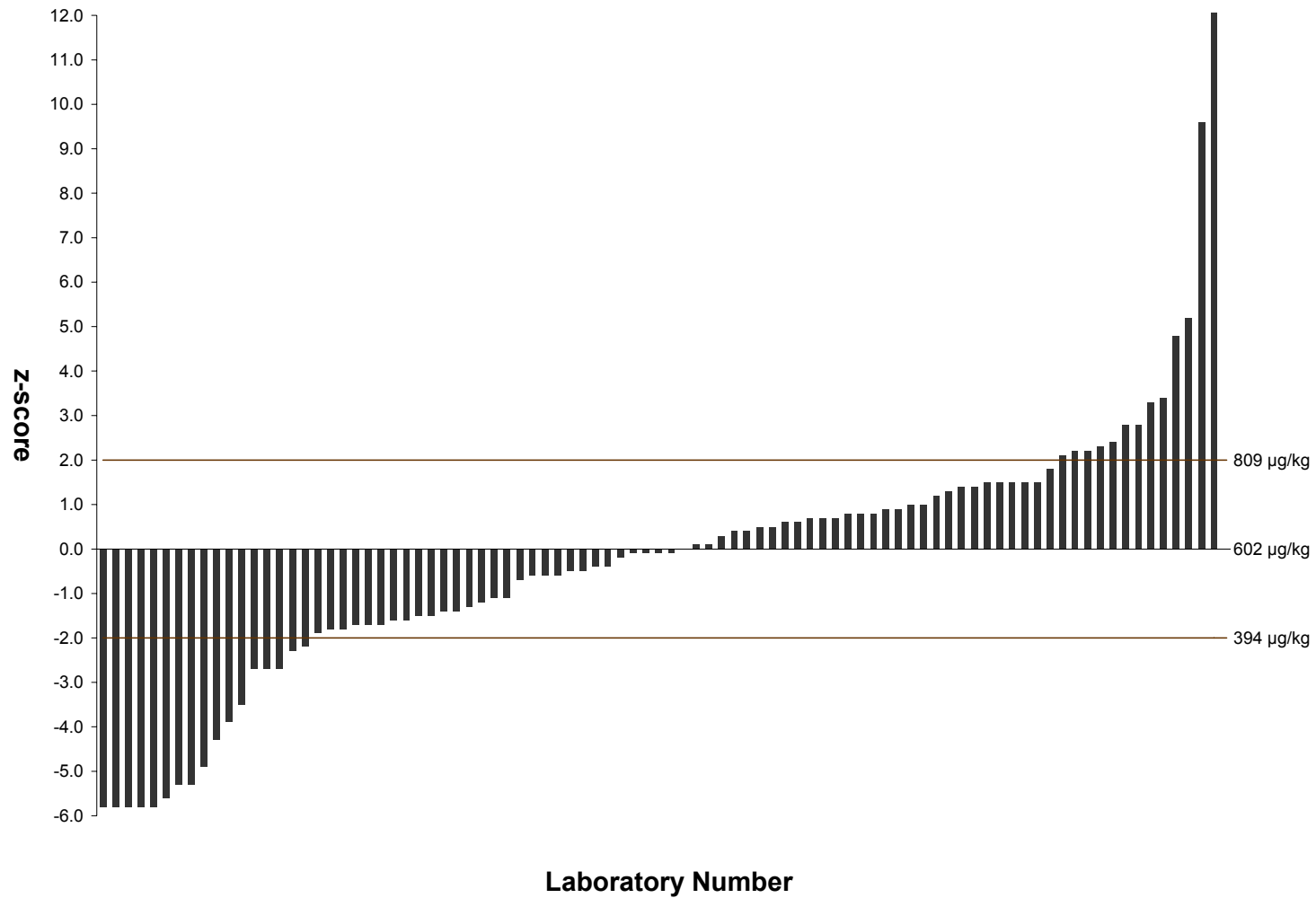
* see page 7 for appropriate form of the Horwitz equation

Table 4: Number and Percentage of Satisfactory z-Scores

analyte	number of satisfactory scores $ z \leq 2$	total number of scores	satisfactory %
cypermethrin	59	89	66
methidathion	99	124	80

Table 5: Number and Percentage of Participants Correctly Identifying and Obtaining Satisfactory z-Scores for Pesticides Present >30 µg/kg

criteria	number of satisfactory participants	total number of participants	satisfactory %
correctly identified both pesticides	72	140	51
correctly identified and obtained satisfactory z-scores for both pesticides	44	140	31



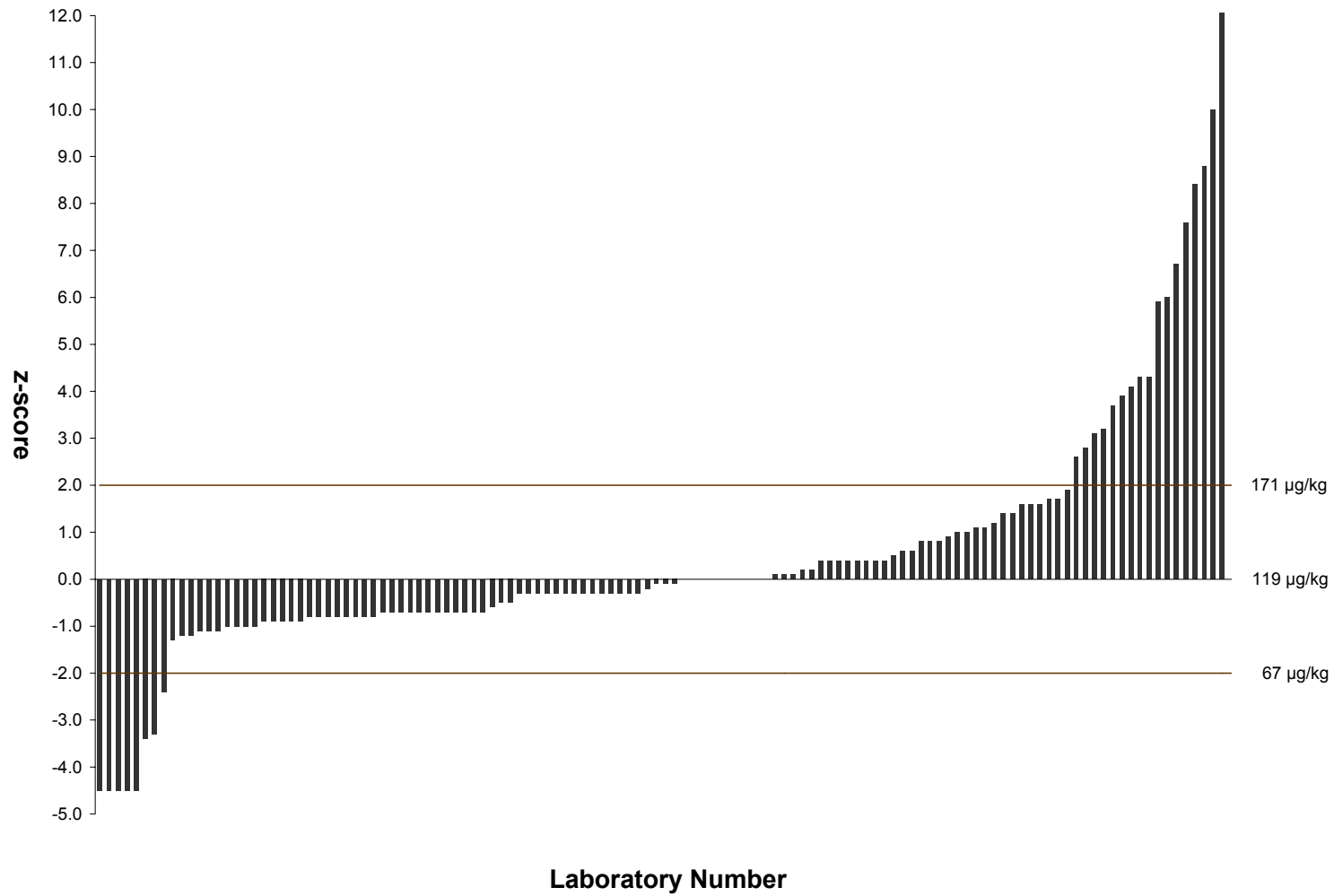


Figure 2: z-Scores for Methidathion (119 µg/kg) in Lettuce Purée Test Material

APPENDIX I: Homogeneity Data for Lettuce Purée Test Material

sample id	analyte			
	cypermethrin µg/kg		methidathion µg/kg	
	replicate 1	replicate 2	replicate 1	replicate 2
1	571.2	609.4	119.4	130.1
2	603.7	589.3	131.0	123.8
3	551.2	449.1	121.5	104.7
4	561.8	589.2	128.8	126.3
5	643.3	661.5	129.1	132.2
6	592.0	660.4	117.8	136.6
7	636.8	692.5	133.1	133.8
8	638.6	636.9	124.8	129.4
9	731.3	707.6	143.7	138.2
mean	618.1		128.0	
<i>n</i>	18		18	
origin of target sd (σ_p)	Horwitz*		Horwitz*	
RSD%	17.20		21.80	
absolute target sd (σ_p)	106.30		27.90	
s_{an}	34.58		6.95	
s_{am}^2	3281.42		30.72	
σ_{all}^2	1016.98		70.07	
<i>critical</i>	3299.92		189.58	
$s_{am}^2 < \text{critical?}$	ACCEPT		ACCEPT	

* see page 7 for appropriate form of the Horwitz equation

APPENDIX II: Analytical Methods Used by Participants

Notes:

- 1) Participants' methods are tabulated according to the information supplied by electronic submission of methods entry. Some responses have been combined or edited for clarity.
- 2) Participants with performance outside the range $|z| \leq 2$ are no longer shown in **bold** within this Appendix. Refer to Table 1 for this information.
- 3) Only methods pertinent to cypermethrin and methidathion have been recorded.

CYPERMETHRIN

Is the Method used Accredited?	laboratory number
yes	003 006 013 016 019 020 021 030 031 033 039 042 044 059 060 063 064 065 066 068 076 077 078 080 081 085 091 092 093 096 101 102 106 110 111 112 113 114 115 116 119 123 125 127 129 132 134 137
no	002 004 005 007 009 011 012 014 015 017 029 034 048 057 062 069 074 075 084 100 104 105 107 117 120 121 131 133 135
Sample Weight (g)	laboratory number
≥1 - <2	019
≥2 - <5	002 102
≥5 - <10	007 009 011 020 030 062 084 085 116 117 119 134
≥10 - <25	003 005 006 012 013 016 017 029 031 033 034 044 048 053 057 059 063 066 068 074 075 076 077 078 080 081 091 092 093 096 105 106 107 110 112 113 114 115 121 123 125 127 129 131 132 135
≥25 - <50	039 041 042 060 064 065 069 100 104 111
≥50	004 015 021 101 120 137

Extraction Solvent	laboratory number
petroleum ether/spirit	005 006 011 013 017 029 064 092 093 116
hexane	009 019 033 080 085 137
isooctane	031
cyclohexane	042
toluene	065
dichloromethane	005 006 007 011 013 014 016 017 029 031 064 075 092 093 132
diethyl ether	009
acetone	005 006 007 011 013 015 016 017 029 031 033 042 064 066 075 076 078 085 092 093 100 104 106 112 116 132 134 137
ethyl acetate	003 004 033 041 042 059 060 066 080 081 085 091 101 105 111 113 120 127 129 131
acetonitrile	002 012 020 021 030 034 039 044 048 053 057 062 063 068 069 074 077 084 096 102 110 114 115 117 119 121 123 125 135
water	021 137
sodium bicarbonate	137

Extraction pH Adjusted?	laboratory number
yes	003 014 041 053 081 101 102 106 113 117 125 131 137
no	002 004 005 006 007 009 011 012 013 015 016 017 019 020 021 029 030 031 033 034 039 042 044 048 057 059 060 062 063 064 065 066 068 069 074 075 076 077 078 080 084 085 091 092 093 096 100 104 105 107 110 111 112 114 115 116 119 120 121 123 127 129 132 134

Sample Clean-up Technique	laboratory number
GPC/HPGPC	015 016 020 031 042 060 074 078 081 101 113
solid phase extraction (SPE)	003 004 007 012 014 021 030 034 039 041 048 062 063 068 074 080 100 102 107 111 114 115 116 117 119 123 129
MSPDE (silica gel)	009
silica column	016 019
carbon based column	053 062 078 110

Sample Clean-up Technique (continued)	laboratory number
graphite-carbon/NH ₂	076
NH ₂ /aminopropyl column	002 021 062 078 110 125
florisil column	033 075 076 085 120
PSA (primary,secondary amine)	020
QuEChERS	121 135
liquid/liquid extraction with Extrelut	044
liquid/liquid extraction or solvent exchange	012 015 033 048 057 065 066 069 076 078 096 104 106 132 137
extraction	012 020 066 084 116 131 137
filter	011 012 020 029 033 062 066
none	005 006 013 017 059 064 077 093 105 112 127
SPE Column Type	laboratory number
C18	030 100 107
silica	120
Envicarb/GCB	021 068 078 110 111 114 115 125 129
EnviCarb/NH ₂ /Silica	039 048 053 069
EnviCarb/NH ₂	003 057 074 092 119
GCB/NH ₂	102
EnviCarb/PSA	044 080 117
GC/PSA	084
NH ₂	123
florisil	004 014 116
SAX	062
SAX/PSA	012
PSA	063
supelco	034

Were Certified Standards Used?	laboratory number
yes	003 004 005 006 007 009 011 013 014 015 016 019 020 021 029 031 033 034 039 041 042 044 053 059 060 062 063 064 065 068 075 077 078 080 081 084 085 091 092 093 096 100 101 102 105 106 110 111 112 114 115 116 121 125 127 129 131 132 134 135 137
no	002 012 017 030 048 057 069 074 076 104 107 113 117 119 120
MS Confirmation?	laboratory number
yes	002 003 004 005 006 011 012 013 014 015 016 017 020 021 029 030 031 034 039 041 042 044 048 053 057 059 060 062 063 064 066 068 069 074 077 078 080 081 084 085 092 093 096 100 101 102 104 105 106 110 111 112 113 114 115 117 119 120 121 125 127 129 131 132 134 135
no	009 019 033 065 075 076 091 116 137
Calibrations	laboratory number
matrix-matched	006 011 013 021 044 059 060 077 081 084 096 101 111 123 129 132 134
solvent	003 005 009 014 016 017 019 020 029 030 033 042 048 062 066 075 076 078 092 100 104 110 137
multi-level	003 004 006 011 012 015 016 020 029 031 033 039 044 057 059 062 065 066 068 069 074 076 077 080 081 085 091 092 093 102 104 107 113 114 115 116 117 119 120 125 127 129 132
single-level	002 007 009 021 030 034 041 042 048 053 063 064 075 078 101 110 131 134 135 137
standard addition	042 105 106 112 121

Source of Standards	laboratory number
Accu Standard	003 021 084
Chem Service	064 066
Cluzeau	014
Dr Ehrenstorfer	003 004 006 009 011 013 015 016 017 019 021 029 060 062 063 065 066 068 075 077 081 092 093 100 102 104 105 106 111 114 115 116 120 121 123 127 131 132 134 135 137
Fluka	002 029 031 123
Hayashi	034039 048 053 080
HPC	044 096
Neochema	042
original	057
R-Biopharm Rhone	123
Restek	020
Riedel de Haen	006
Sigma/Aldrich	021 029 030 033 085 113 123
Supelco	123
Wako	069 074 076 078 084 107 110 117 119 125
Is quoted percentage recovery measured in same analytical batch as test material?	laboratory number
yes	003 006 011 012 013 015 016 017 019 020 021 029 030 033 034 041 042 044 048 053 060 062 063 064 065 066 068 069 074 076 077 078 080 081 084 092 096 101 102 104 105 106 110 111 112 113 114 115 116 117 119 120 121 127 129 132 134 135
no	002 004 005 009 031 039 057 059 075 085 091 093 100 107 123 125 131 137

If measured in this batch, at what stage was the spike added?	laboratory number
prior to extraction	003 006 007 009 011 012 013 015 016 017 020 021 029 030 033 034 041 044 048 053 059 060 062 064 065 066 068 069 076 077 078 080 081 092 096 101 102 104 105 106 110 111 112 113 114 115 116 117 119 120 121 123 127 129 132 135
prior to clean up	019 042 074
prior to instrument measurement	134

Ratio of Isomers	laboratory number
1.6:1.5:1.1:1.0	034
1:0.8:0.8:1	041
2.8:1.7:2.0:1.0	020
2:2:3:3	119
23.4:28.7:20.6:27.3	012
29:40:25:33	080
3:3:2:2	003 062
50:50	064
mixture	102
sum of all isomers	044
unknown	076

Level Of Spike (µg/kg)	laboratory number
<25	011 015 016 033 048 059 060 085 096 132 135
≥25 - <50	029 065 081 104 113 114 115 119
≥50 - <100	006 007 009 030 041 042 062 064 068 074 101 102 116 120 127 129
≥100 - <150	003 004 012 017 019 034 044 069 076 078 080 105 110 111 117 121
≥150 - <200	066 077
≥200 - <250	016 075 123 129
≥250 - <300	106
≥400 - <500	019
≥500	020 053 112 134

Composition Of Blank Commodity Used For Spiking	laboratory number
blank lettuce provided	006 011 017 019 021 029 033 034 044 048 064 066 068 069 074 077 078 080 092 096 102 104 111 112 113 114 115 116 117 121 125 127 129 132
spiked lettuce test material provided	007 012 053 063 105 106 110 119 135
lettuce	003 009 015 016 062 076 081 085 101 120
green soybeans	030
potato	075
strawberry	123
tomato	059
clear sample	065
in house blank	020 060

GC Column Type	laboratory number
capillary	002 003 005 006 007 011 012 013 015 016 019 020 021 031 033 034 039 041 042 044 048 053 057 059 060 062 063 064 065 066 068 069 074 076 077 080 081 092 093 096 100 102 104 105 106 107 110 111 112 113 114 115 117 119 120 121 123 125 129 131 134 135 137
megabore	014 078 132
narrowbore	017 030 075 085 116 127
widebore	009

GC Column Packing	laboratory number
100% methyl polysiloxane	048 064 137
95% methyl 5% phenyl polysiloxane	002 003 005 006 007 009 011 012 013 014 015 016 017 020 030 031 034 039 041 042 044 048 053 057 059 062 063 065 068 074 077 078 080 081 096 100 102 104 105 106 107 110 112 113 114 117 119 121 125 127 129 131 132 134 135
95 %polydimethylsiloxane 5% polysilarylilene	093
65% methyl 35% phenyl polysiloxane	019 076 120
50% methyl 50% phenyl polysiloxane	004 009 060 069 075 116
14% cyanopropylphenyl 86%methyl polysiloxane	021 065 085 092 100 111 115
EPA method 608 specific column	066

GC Injection Volume (µL)	laboratory number
<1	085 107
≥1 - <2	002 004 009 014 015 016 019 020 021 033 048 057 063 064 066 068 069 075 078 081 093 100 104 105 106 113 116 119 120 127 129 131 132 134 135
≥2 - <5	005 006 007 012 017 030 031 034 039 044 060 062 065 074 076 077 080 096 102 111 112 114 115 117 121 125
≥5 - <10	003 029 137
≥10	011 013 042 053 059 092 110

GC Injection Mode	laboratory number
on-column	014 064 078 081
PTV	003 029 042 053 059 077 092 106 110 121 134
split	011 013 135
splitless	002 004 005 006 007 009 012 015 016 017 019 020 021 030 031 033 034 039 044 048 060 062 063 065 066 068 069 074 075 076 080 085 093 096 100 102 104 105 107 111 112 113 114 115 116 117 119 120 125 127 129 131 132 137
pulsed splitless	057

GC Detector	laboratory number
ECD	002 003 004 005 006 007 009 012 014 015 016 017 019 031 033 048 065 066 069 074 075 076 078 085 100 115 116 120 131 135 137
ELCD	004 064
FID	135
FPD	002 004 019 064 085 093
ITD	004 020 029 059 105 112
MSD	005 012 015 017 021 030 031 034 039 041 042 044 048 053 057 060 062 063 066 080 092 093 096 100 102 104 106 107 110 111 113 114 117 119 120 121 123 125 127 129 131 132 134 135
MS-MS	011 013 068 077 081 110

NPD

003 004 015 017 065 085

HPLC Column Packing	laboratory number
C18	004 029 101 114 115
HPLC Guard Column Used?	laboratory number
yes	004 029 101 114
no	064 085 093 115 134
Mobile Phase Programme	laboratory number
gradient	004 029 101 114 115
Mobile Phase Components	laboratory number
acetonitrile	114 115
methanol	004 029 101
acetate	029
formate	101
water	004 029
HPLC Column Temperature (°C)	laboratory number
ambient	029 101
>ambient - <50	004 114 115
HPLC Injection Volume (µL)	laboratory number
≥5 - <10	029 101
≥10 - <25	004 114 115
Mobile Phase Flow Rate (mL/min)	laboratory number
≥0.25 - <0.75	004 029 101 114 115
HPLC Detector Type	laboratory number
MS-MS	004 101 114 115
PAD	029

METHIDATHION

Is the Method used Accredited?	laboratory number
yes	003 006 013 016 019 021 022 023 024 025 026 028 030 031 033 035 037 038 039 042 043 044 045 046 047 049 050 051 052 054 055 056 058 059 060 061 063 064 065 066 068 070 071 073 076 077 078 080 081 082 086 087 088 089 090 093 094 096 097 098 099 101 102 106 108 110 112 113 114 115 119 123 125 127 128 129 132 134 137
no	002 004 005 007 009 012 014 015 017 029 034 036 040 048 057 062 067 072 074 075 079 100 104 105 107 117 120 121 124 135
Sample Weight (g)	laboratory number
≥1 - <2	019
≥2 - <5	002 102
≥5 - <10	006 007 009 030 062 117 119 128 134
≥10 - <25	003 005 012 013 016 017 022 023 024 025 026 029 031 033 034 035 036 037 038 040 043 044 045 046 047 048 049 050 051 052 053 054 055 056 057 058 059 061 063 066 067 068 070 071 072 073 074 075 076 077 078 079 080 081 082 086 087 088 089 090 093 094 096 097 098 099 105 106 107 108 110 112 113 114 115 121 123 125 127 129 132 135
≥25 - <50	039 041 042 060 064 065 100 104
≥50	004 015 021 028 101 120 137
Extraction Solvent	laboratory number
petroleum ether / spirit	005 006 013 017 029 064 093
hexane	019 080 137
isooctane	031
cyclohexane	042
dichloromethane	005 006 007 009 013 014 016 017 029 031 064 075 080 093 132
acetone	005 006 007 009 013 015 016 017 028 029 031 033 042 064 066 075 076 078 093 100

104 106 112 124 132 134 137

Extraction Solvent (continued)	laboratory number
ethyl acetate	003 004 033 041 042 059 060 065 066 081 101 105 113 120 127 128 129
acetonitrile	002 012 021 022 023 024 025 026 030 034 035 036 037 038 039 040 043 044 045 046 047 048 049 050 051 052 053 054 055 056 057 058 061 062 067 068 070 071 072 073 074 077 082 086 087 088 089 090 094 096 097 098 099 102 108 110 114 115 117 119 121 123 125 135
methanol	063
water	021 137
sodium bicarbonate	137
sodium sulphate	006

Extraction pH Adjusted?	laboratory number
yes	014 040 041 045 046 049 050 051 052 053 054 055 058 070 071 073 079 081 082 086 087 088 089 090 094 097 098 101 102 106 108 113 117 125 137
no	002 004 005 006 007 009 012 013 015 016 017 019 021 022 023 024 025 026 028 029 030 031 033 034 035 036 037 038 039 042 043 044 047 048 056 057 059 060 062 063 064 065 066 067 068 072 074 075 076 077 078 080 093 096 099 100 104 105 107 110 112 114 115 119 120 121 123 124 127 128 129 132 134

Sample Clean-up Technique	laboratory number
GPC/HPGPC	015 016 031 042 060 074 078 081 101 113 124
solid phase extraction (SPE)	003 004 007 012 014 021 022 023 024 025 026 030 034 035 036 037 038 039 040 041 043 047 048 049 050 051 052 054 055 056 058 061 062 068 070 071 073 074 079 080 082 086 087 088 089 090 094 097 098 099 100 102 107 108 114 115 117 119 123 129
MSPDE (silica gel)	009
silica column	016 019 028
carbon based column	053 061 062 067 070 072 073 078 082 110
NH ₂ /aminopropyl column	002 021 061 062 070 073 078 082 110 125

florisil column

028 033 075 076

Sample Clean-up Technique (continued)	laboratory number
alumina column	028
QuEChERS	121 135
liquid/liquid extraction with Extrelut	044 063
liquid/liquid extraction or solvent exchange	012 015 022 023 024 025 026 033 036 037 038 043 045 046 047 048 057 061 066 070 073 076 078 082 090 096 104 106 108 132 137
extraction	012 055 066 099 108 137
filter	012 029 033 061 062 066 070 073 082 108
none	005 006 013 017 059 064 065 077 093 105 112 120 127

SPE Column Type	laboratory number
C18	030 079 100 107
EnviCarb/GCB	003 021 040 067 068 071 072 078 086 097 098 110 114 115 125 129
EnviCarb/NH ₂ /Silica	039 048 053
EnviCarb/NH ₂	022 023 024 025 026 035 036 037 038 043 045 047 049 050 052 054 055 056 057 058 061 070 073 074 082 090 096 099 108 119
NH ₂ /GCB	046 051 102
EnviCarb/PSA	044 080 117
NH ₂	087 088 089 094 123
Florisil	004 014
SAX/PSA	012
SAX	062
supelco	034

Were Certified Standards Used?	laboratory number
yes	003 004 005 006 007 009 013 014 015 016 019 021 029 031 033 034 035 036 037 038 039 041 042 043 044 049 050 052 053 054 055 056 058 059 060 062 063 064 065 067 068 070 072 073 075 077 078 080 081 082 086 087 088 089 093 094 096 097 098 100 101 102 105 106 108 110 112 114 115 121 124 125 127 128 129 132 134 135 137
no	002 012 017 022 023 024 025 026 028 030 040 045 046 047 048 051 057 061 071 074 076 079 090 099 104 107 113 117 119 120

MS Confirmation?	laboratory number
yes	002 003 004 005 006 012 013 014 015 016 017 021 022 023 024 025 026 029 030 031 034 035 036 037 038 039 040 041 042 043 044 045 046 047 048 049 050 051 052 053 054 055 056 057 058 059 060 061 062 063 064 066 067 068 070 071 072 073 074 077 078 079 080 081 082 086 087 088 089 090 093 094 096 097 098 099 100 101 102 104 105 106 108 110 112 113 114 115 117 119 120 125 127 128 129 132 134 135
no	009 019 028 033 065 075 076 121 124 137
Calibrations	laboratory number
matrix-matched	006 013 021 044 059 060 061 070 073 077 081 082 096 101 123 124 129 132 134
solvent	003 005 009 014 016 017 019 029 030 033 042 048 062 066 075 076 078 100 104 110 137
multi-level	003 004 006 012 015 016 028 029 031 033 037 038 039 040 043 044 045 046 051 055 057 059 061 062 063 065 066 068 070 071 073 074 076 077 079 080 081 082 086 093 097 098 102 104 107 108 113 114 115 117 119 120 125 127 128 129 132
single-level	002 007 009 021 030 034 041 042 048 053 064 075 078 101 110 134 135 137
standard addition	022 023 024 025 026 035 036 037 038 040 042 043 047 049 050 052 054 055 056 058 061 067 070 071 072 073 082 086 087 088 089 090 094 097 098 099 105 106 108 112 121
Source of Standards	laboratory number
Accu Standard	003 021
Chem Service	064 066
Cluzeau	014
Dr Ehrenstorfer	003 004 006 009 013 015 016 017 019 021 028 029 060 062 063 065 066 068 075 077 081 093 100 104 105 106 114 115 119 120 121 123 124 127 128 132 134 135 137
Fluka	002 029 031 123
Hayashi	034 039 053 080
HPC	044 096

Source of Standards (continued)	laboratory number
Neochema	042
original	057
R-Biopharm Rhone	123
Riedel de Haen	006 030
Sigma/Aldrich	021 029 033 113 123
Supelco	123
Wako	022 023 024 025 026 035 036 037 038 043 045 046 048 049 050 051 052 054 055 056 058 061 067 070 071 072 073 074 076 078 079 082 086 087 088 089 090 094 097 098 099 102 107 108 110 117 125
Is quoted percentage recovery measured in same analytical batch as test material?	laboratory number
yes	003 006 012 013 016 017 019 021 022 023 024 025 026 029 030 033 034 035 036 037 038 040 041 042 043 044 045 046 047 048 049 050 051 052 053 054 055 056 058 060 061 062 063 064 065 066 067 068 070 071 072 073 074 076 077 078 079 080 081 082 086 087 088 089 090 094 096 097 098 099 101 102 104 105 106 108 110 112 113 114 115 117 119 120 121 124 127 128 129 132 134 135
no	002 004 005 009 015 028 031 057 059 075 084 093 100 107 123 125 137
If measured in this batch, at what stage was the spike added?	laboratory number
prior to extraction	003 006 007 009 012 013 016 017 021 022 023 024 025 026 029 030 033 034 035 036 037 038 040 041 043 044 045 046 047 048 049 050 051 052 053 054 055 056 058 059 060 061 062 063 064 065 066 067 068 070 071 072 073 076 077 078 079 080 081 082 086 087 088 089 090 094 096 097 098 099 101 102 104 105 106 108 110 112 113 114 115 117 119 120 121 123 127 128 129 132 135
prior to clean up	019 042 074
prior to instrument measurement	124 134

Ratio of Isomers	laboratory number
1	119
3:2:2:2	062
50:50	064
mixture	102

Level Of Spike (µg/kg)	laboratory number
<25	009 016 033 035 047 048 059 060 061 063 070 073 079 082 096 108 132 135
≥25 - <50	029 055 065 081 099 104 113 114 115 119 129
≥50 - <100	006 007 030 036 037 038 041 042 043 062 064 067 068 072 074 087 088 089 090 102 120 124 127 128
≥100 - <150	004 012 017 019 034 044 066 076 078 080 105 110 117
≥150 - <200	129
≥200 - <250	016 052 075 112 121 123
≥250 - <300	022 023 024 025 026 040 045 046 049 050 051 054 056 058 071 086 094 097 098 106
≥400 - <500	101
≥500	053 077 134

Composition Of Blank Commodity Used For Spiking	laboratory number
blank lettuce provided	006 017 019 021 029 033 034 040 044 047 048 049 050 052 054 055 056 058 064 066 067 068 072 073 074 077 078 079 080 090 096 099 102 104 112 113 114 115 117 121 124 125 127 128 129 132
spiked lettuce test material provided	007 012 045 046 051 053 063 084 105 106 110 119 135
lettuce	003 035 036 037 038 043 076 101 016 061 070 082 087 088 089 094 108 062 081 071 086 097 098 009 024 022 023 026 120
green soybeans	030
potato	075
strawberry	123

Composition Of Blank Commodity Used For Spiking (continued)	laboratory number
tomato	059
clear sample	065
in house blank	025 060
GC Column Type	laboratory number
capillary	002 003 005 007 012 013 015 016 019 021 022 023 024 025 026 028 031 033 034 035 036 037 038 039 040 041 042 043 044 047 048 049 050 052 053 054 055 056 057 058 059 060 061 062 064 065 066 067 068 070 071 072 073 074 076 077 079 080 081 082 084 086 087 088 089 090 093 094 096 097 098 099 100 102 104 105 106 107 108 110 112 113 114 115 117 119 120 121 123 124 125 128 134 135 137
megabore	014 078 132
narrowbore	017 045 046 051 075 127
widebore	009
GC Column Packing	laboratory number
100% methyl polysiloxane	048 064 076 137
95% methyl 5% phenyl polysiloxane	002 005 007 009 012 013 014 015 016 017 022 023 024 025 026 028 034 035 036 037 038 039 040 041 042 043 044 045 046 047 048 049 050 051 052 053 054 055 056 057 058 059 061 062 065 067 068 070 071 072 073 077 079 080 081 082 084 086 087 088 089 090 094 096 097 098 099 100 102 104 105 106 107 108 110 112 113 114 117 119 125 127 128 132 134
95 %polydimethylsiloxane 5% polysilarylilene	093
65% methyl 35% phenyl polysiloxane	019 120
50% methyl 50% phenyl polysiloxane	004 009 060 075 135
14% cyanopropylphenyl 86%methyl polysiloxane	021 031 065 078 100 121
trifluoropropyl methylpolysiloxane	115
EPA method 608 specific column	066
Low Bleed Column (XLB)	074

GC Injection Volume (µL)	laboratory number
<1	107 128
≥1 - <2	002 004 009 014 015 016 019 021 033 048 052 057 064 066 068 070 073 074 075 081 082 084 093 100 104 105 106 113 119 120 124 127 132 134 135
≥2 - <5	003 005 007 012 017 022 023 024 025 026 031 034 035 036 037 038 039 040 043 044 045 046 047 049 050 051 054 055 056 058 060 061 062 065 067 071 072 076 077 078 079 080 086 087 088 089 090 094 096 097 098 099 102 108 112 114 115 117 121 125
≥5 - <10	028 029 137
≥10	013 042 053 059 110
GC Injection Mode	laboratory number
on-column	014 064 081
PTV	003 028 029 042 053 059 077 106 110 134
split	013 024 135
splitless	002 004 005 007 009 012 015 016 017 019 021 022 023 025 026 031 033 034 035 036 037 038 039 040 043 044 045 046 047 048 049 050 051 052 054 055 056 058 060 061 062 065 066 067 068 070 071 072 073 074 075 076 078 079 080 082 084 086 087 088 089 090 093 094 096 097 098 099 100 102 104 105 107 108 112 113 114 115 117 119 120 121 124 125 127 132 137
pulsed splitless	057
GC Detector	laboratory number
ECD	002 003 004 005 007 009 014 015 017 028 065 100 124 135 137
ELCD	004 064
FID	135
FPD	002 004 012 019 028 033 039 064 066 074 076 078 093 115 120 121
ITD	004 029 059 105 112

GC Detector (continued)	laboratory number
MSD	005 012 015 016 017 021 022 023 024 025 026 031 034 035 036 037 038 040 041 042 043 044 045 046 047 048 049 050 051 052 053 054 055 056 057 058 060 061 062 067 070 071 072 073 079 080 082 084 086 087 088 089 090 093 094 096 097 098 099 100 102 104 106 107 108 110 113 114 117 119 120 123 125 127 128 132 134 135
MS-MS	013 068 077 081 110
NPD	003 004 009 015 017 031 065 075 137
HPLC Column Packing	laboratory number
C18	004 006 029 030 063 101 114 115 129
endcapped	030
HPLC Guard Column Used?	laboratory number
yes	004 006 029 063 101 114 129
no	030 064 093 115 134
Mobile Phase Programme	laboratory number
gradient	004 006 029 030 063 101 114 115 129
Mobile Phase Components	laboratory number
acetonitrile	030 114 115
methanol	004 006 029 063 101 129
acetate	029
formic acid	030 063
formate	006 101
water	004 006 029 063 129
HPLC Column Temperature (°C)	laboratory number
ambient	029 101 129
>ambient - <50	004 006 030 063 114 115

HPLC Injection Volume (µL)	laboratory number
≥5 - <10	006 029 063 101
≥10 - <25	004 030 114 115 129
Mobile Phase Flow Rate (mL/min)	laboratory number
<0.25	030 063 129
≥0.25 - <0.75	004 006 029 101 114 115
HPLC Detector Type	laboratory number
conductivity	084
MS-MS	004 006 030 063 101 114 115 129
PAD	029

APPENDIX III: FAPAS® SecureWeb, Reports and Protocol

1. FAPAS® SECUREWEB

Access to the secure area of our web site 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 is priced £35 if ordered at the same time as the proficiency test or £50 if ordered subsequently.

3. PROTOCOL

The Protocol [1] sets out how FAPAS® is organised. It gives full details of the statistical procedures used and includes worked examples. Copies can be downloaded from our website.

4. CONTACT DETAILS

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