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LABORATORY NUMBER: Available to participants from FAPAS SecureWeb

FAPAS® Proficiency Test 1984

Pesticide Residues at Low Levels in Lettuce Purée

September - October 2008

Report

Prepared on behalf of FAPAS® by

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0009 PT

SUMMARY

1. The test material for FAPAS® proficiency test 1984 was dispatched in September 2008. Each participant received a lettuce purée test material to be analysed for low levels (<30 µg/kg) of pesticide residues. In total, 150 sets of test material were distributed to participants in 29 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 76 possible pesticide residues, participants were requested to identify and quantify those present in the lettuce purée. The test material contained bromopropylate, ethion, γ-HCH (lindane) and myclobutanil.

3. The assigned value ($\hat{X}$) was calculated from the most appropriate measure of central tendency of participants' results [1, 2, 3]. Although myclobutanil was present in the test material, no supporting homogeneity data was available. Therefore, no assigned value has been set for this analyte.

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 $|z| \leq 2$. No z-scores are given for myclobutanil.

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 %
bromopropylate	13.4	60	71	85
ethion	24.4	74	87	85
γ-HCH	21.1	73	97	75

6. No surplus test materials are available for sale.

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.

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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 (~53 kg) were obtained from a local supplier. They were frozen, homogenised using a cryogenic mill with dry ice and transferred to a cold room to slowly thaw.

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 20 µg/kg (50 µg/kg for cypermethrin, deltamethrin, pirimicarb, and profenofos).

30 kg of bulk material was then puréed using a Silverson mixer. A standard solution of the spiking pesticides was prepared to give the following approximate final concentrations: bromopropylate 15 µg/kg, ethion 30 µg/kg, γ-HCH 25 µg/kg and myclobutanil 20µg/kg.

One-third of the bulk purée was transferred to a Crypto-Peerless bowl mixer and one-third of the spiking solution was added directly. The sample was mixed for thirty minutes. A further third of the bulk purée was added along with a further third of the spiking solution. After another thirty minutes' mixing time the remainder of the sample and spiking solution was added. The total bulk was mixed overnight. Samples were measured into labelled glass bottles with at least 100 g in each.

To prepare the blank samples, a portion of milled sample was separated out and puréed separately in the Crypto-Peerless bowl mixer for 1 hour. Sub-samples (at least 100g) of the purée were taken and measured into labelled glass bottles.

The spiked and blank samples were stored at -20°C until distribution.

2.2. Homogeneity

Ten randomly selected test materials were analysed in duplicate for all four pesticides. The results, together with their statistical evaluation [7], are given in APPENDIX I. The statistical tests initially check the data for any widely discrepant pairs using Cochran's test. If found, such data are removed. Thereafter the remaining data are subject to analysis of variance (ANOVA) to estimate the sampling and analytical variances.

The data for bromopropylate, ethion and γ-HCH show sufficient homogeneity, and are not included in the subsequent calculation of the assigned values. For myclobutanil, we were unable to demonstrate sufficient homogeneity.

2.3. Distribution

The dispatch date was 15 September 2008. 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, 31 October 2008.

Each participant was given a laboratory number, assigned in order of receipt of results. The reported results for bromopropylate and ethion are given in Table 1, and for γ-HCH and myclobutanil in Table 2.

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 bromopropylate, ethion or γ-HCH 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 bromopropylate, ethion or γ-HCH 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 bromopropylate, ethion, γ-HCH and myclobutanil at levels >15 µg/kg is listed in Table 3 together with the pesticides reported and level determined.

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) semi-quantitative results e.g. <10,
  - iii) results from participants not quoting a percentage recovery,
  - iv) results from participants whose recovery is outside the range 70 - 120% [9],
  - v) results from participants not quoting an LoQ (limit of quantification),
  - vi) results less than the 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 bromopropylate, ethion and γ-HCH this procedure was straightforward. The robust mean was the best measure of central tendency and chosen as the assigned value.

No assigned value was set for myclobutanil as we could not demonstrate sufficient homogeneity. However, a kernel density plot shows the distribution of the results (Figure 4).

The robust means used to set the assigned values, together with u, n and $\hat{\sigma}$ are shown in Table 4.

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.

The FAPAS® Advisory Committee recently noted that an EU-wide proficiency testing scheme for pesticide residue analysis employs a constant σ_p value of 25% of the mean level. The Committee also noted that this value was not derived from a fully objective source but was a reflection of the observed spread of the results in previous rounds of the proficiency testing scheme. Since this approach to setting the standard deviation for proficiency assessment is not in-keeping with the concept of fitness-for-purpose as set out in the International Harmonised Protocol [8] the Committee recommended that FAPAS® pesticide residue proficiency tests continue to apply a concentration dependant σ_p value derived from the appropriate form of the Horwitz equation [4].

For all 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 ≤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 4.

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 bromopropylate and ethion are given in Table 1, while z-scores for γ-HCH are given in Table 2. Participants' z-scores are shown as histograms in Figures 1 - 3. The number and percentage of z-scores in the satisfactory range, $|z| \leq 2$, for all analytes are given in Table 5.

It is possible for the z-scores published in this report to differ slightly from the z-scores 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 4.

The number and percentage of participants correctly identifying all pesticides, and the number and percentage of participants obtaining satisfactory z-scores for bromopropylate, ethion and γ-HCH are shown in Table 6. In this case, participants identifying other pesticides at or >15 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 Tables 1 and 2 and summarised in Table 5.

5. REFERENCES

1 FAPAS®, 2002, Protocol for the Food Analysis Performance Assessment Scheme, Organisation and Analysis of Data, 6th edition.

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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 31 October 2007. Method Validation and Quality Control Procedures for Pesticide Residues Analysis in Food and Feed. Document No SANCO/2007/3131.

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

laboratory number		analyte							
		bromopropylate assigned value 13.4 µg/kg				ethion assigned value 24.4 µg/kg			
		result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score
001		13		10	-0.1	35		5	2.0
002		10	70	5	-1.1	25	88	10	0.1
003		12	100	10	-0.5	19	100	10	-1.0
004		7.4*	98	10	-2.0	17	105	10	-1.4
005		15.0	116	5	0.5	30.0	91	5	1.1
006		14	98	5	0.2	29	95	5	0.9
007	♠	<10	104	10		16.4	107	10	-1.5
008		<LOQ		10		26.7		10	0.4
009		12	85	10	-0.5	#			
010		12	95	10	-0.5	38	80	10	**2.5**
011		12	85	10	-0.5	22	78	10	-0.4
012		12*	80	30	-0.5	28	105	20	0.7
013		#				#			
014		15	79	10	0.5	18	77	10	-1.2
015		12	96	10	-0.5	25	102		0.1
016		<LOQ		10		26	98.7	5	0.3
017		#				#			
018		#				#			
019		18	108	10	1.6	34	107	10	1.8
020		#				#			
021		#				#			
022		#				#			
023		#				34.9	94.1	10	2.0
024		#				#			
025		#				#			

z-scores outside the satisfactory range, i.e. $|z|>2$, are shown in **bold** LoQ = limit of quantification
= pesticide not analysed * = Result is less than LoQ
Participant comments: ♠ = Bromopropylate detected at <10ug/kg (5.8 extrapolated)

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

laboratory number	analyte							
	bromopropylate assigned value 13.4 µg/kg				ethion assigned value 24.4 µg/kg			
	result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score
026	#				#			
027	9*	90	10	-1.5	18	90	10	-1.2
028	13.0	105	10	-0.1	30.5	88	10	1.1
029	#				#			
030	#				#			
031	#				#			
032	#				#			
033	7.0	100	5	**-2.2**	13.8	100	10	-2.0
034	9*		10	-1.5	29			0.9
035	#				#			
036	13	85	10	-0.1	26	95	10	0.3
037	17	88.1	0.0051	1.2	25	98.4	0.0051	0.1
038	#				#			
039	#				#			
040	#				#			
041	#				#			
042	#				#			
043	#				#			
044	#				#			
045	#				#			
046	#				#			
047	#				#			
048	11.0	102.2	10	-0.8	23.5	96.2	10	-0.2
049	10	108	5	-1.1	25	102	5	0.1
050	15.2	97.7	10	0.6	32.0	119.4	10	1.4

z-scores outside the satisfactory range, i.e. $|z|>2$, are shown in **bold**
# = pesticide not analysed
LoQ = limit of quantification
* = Result is less than LoQ

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

laboratory number		analyte							
		bromopropylate assigned value 13.4 µg/kg				ethion assigned value 24.4 µg/kg			
		result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score
051		#				18	77	10	-1.2
052		#				#			
053		10	73.21	0.01 ppm	-1.1	20		0.01 ppm	-0.8
054		<LOQ		10		26.3	86.41	10	0.4
055		#				#			
056		10	78	5	-1.1	28	83	5	0.7
057		16	90	10	0.9	29	90	10	0.9
058		<LOQ		50		19	90	10	-1.0
059		<LOQ		50		<LOQ		100	
060		9*	111	10	-1.5	20	114	10	-0.8
061		<LOQ		10		0		10	**-4.5**
062		#				#			
063		16	103	10	0.9	29	117	10	0.9
064		16	123	10	0.9	28	114	10	0.7
065		#				#			
066		10.5	109	10	-1.0	20.6	109	10	-0.7
067		#				#			
068		<LOQ	90	10		21	90	10	-0.6
069	♣	0			**-4.5**	0			**-4.5**
070		12	82		-0.5	26	87		0.3
071		10.0		10	-1.1	19.0		10	-1.0
072		15	83	10	0.5	31	78	10	1.2
073		#				#			
074		20	81	10	**2.2**	30	96	10	1.1
075		14	84	10	0.2	25	88	10	0.1

z-scores outside the satisfactory range, i.e. $|z|>2$, are shown in **bold** LoQ = limit of quantification
= pesticide not analysed * = Result is less than LoQ
Participant comments: ♣ = None of posible pesticides is confirmed by GC-MS (sic)

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

laboratory number		analyte							
		bromopropylate assigned value 13.4 µg/kg				ethion assigned value 24.4 µg/kg			
		result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score
076		#				25	94.7	10	0.1
077		12.18	94.05	10	-0.4	17.98	87	10	-1.2
078		#				27	98.7	9	0.5
079		21	85	8	**2.6**	28	93	10	0.7
080		#				#			
081		#				40	113	10	**2.9**
082		#				#			
083		#				#			
084		#				#			
085		#				27.760	99.8	10	0.6
086		10	98	10	-1.1	15	100	10	-1.7
087		16	102	10	0.9	22	98	10	-0.4
088		10	95	10	-1.1	22	92	10	-0.4
089		#				#			
090	†	12.6	96.3	5	-0.3	2.2	102	1	**-4.1**
091	†	25.43	95%	20	**4.1**	0			**-4.5**
092		<LOQ		10		24		10	-0.1
093		#				0		10	**-4.5**
094		#				#			
095		13.8	103	9	0.1	22.4	99	7	-0.4
096		<LOQ		10		10		10	**-2.7**
097	†	13.7	95	10	0.1	18.6	112	10	-1.1
098		17		10	1.2	33			1.6
099		12	87	10	-0.5	30	82	10	1.1
100		16.3	105	5	1.0	31.3	103	5	1.3

z-scores outside the satisfactory range, i.e. $|z|>2$, are shown in **bold** LoQ = limit of quantification
† = additional pesticides reported (see Table 3)
# = pesticide not analysed

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

laboratory number	analyte							
	bromopropylate assigned value 13.4 µg/kg				ethion assigned value 24.4 µg/kg			
	result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score
101	20		10	**2.2**	17		10	-1.4
102	#				#			
103	#				#			
104	#				#			
105	30	205	10	**5.6**	23	55	1	-0.3
106	#				#			
107	#				#			
108	#				#			
109	11.5		10	-0.6	26.9		10	0.5
110	11			-0.8	26			0.3
111	0			**-4.5**	#			
112	19	70-110	10	1.9	30	70-110	10	1.1
113	15.0		10	0.5	29.8		10	1.0
114	#				#			
115	8*	91	10	-1.8	17	92	10	-1.4
116	0.014	123	0.01	**-4.5**	0.029	149	0.01	**-4.5**
117	#				0			**-4.5**
118	#				#			
119	12.7	98	0.005	-0.2	25.8	97	0.005	0.3
120	12	96	10	-0.5	29	92	10	0.9
121	<50	86	50		14.6	61	10	-1.8
122	12	100		-0.5	23	101	10	-0.3
123	16	90	10	0.9	30	95	10	1.1
124	#				#			
125	#				#			

z-scores outside the satisfactory range, i.e. $|z|>2$, are shown in **bold**
# = pesticide not analysed
LoQ = limit of quantification
* = Result is less than LoQ

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

laboratory number		analyte							
		bromopropylate assigned value 13.4 µg/kg				ethion assigned value 24.4 µg/kg			
		result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score
126		12	118	5	-0.5	25	87	10	0.1
127		#				#			
128		#				#			
129		11.5	82.0	10	-0.6	26.2	92.5	10	0.3
130		#				0.015		0.005	**-4.5**
131		17	90	10	1.2	28	90	10	0.7
132		16		10	0.9	0		10	**-4.5**
133		13	110	10	-0.1	23	106	10	-0.3
134		15	95	10	0.5	20	102	10	-0.8
135		10	89	10	-1.1	18	90	10	-1.2
136		0			**-4.5**	#			
137		<LOQ		10		7.5	84.8	5	**-3.1**
138		10	106	10	-1.1	18	85	10	-1.2
139	†	4		4	**-3.2**	19	94.2	4	-1.0
140		15		10	0.5	#			

z-scores outside the satisfactory range, i.e. $|z|>2$, are shown in **bold** LoQ = limit of quantification
= pesticide not analysed †= additional pesticides reported (see Table 3)

Table 2: Results and z-Scores for γ-HCH, Results for Myclobutanil in Lettuce Purée Test Material

laboratory number	analyte							
	γ-HCH assigned value 21.1 µg/kg				myclobutanil assigned value not set			
	result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score not set
001	25		2	0.8	20		10	
002	19	84	5	-0.5	16	86	10	
003	26	100	10	1.0	11	100	10	
004	10.5	99	10	**-2.3**	12.5	102	10	
005	25.5	92.6	5	0.9	20.0	117	5	
006	28	96	5	1.5	22	93	5	
007	19.8	95	10	-0.3	13.4	98	10	
008	17.7		10	-0.7	17.7		10	
009	#				#			
010	0			**-4.5**	19	83	10	
011	13	70	10	-1.7	14	85	10	
012	14	70	10	-1.5	16*	110	20	
013	21	108.0	10	0.0	20	98.2	10	
014	#				22	82	10	
015	19	109		-0.5	0		10	
016	28	99.7	5	1.5	17	97.4	5	
017	#				#			
018	#				#			
019	31	94	10	**2.1**	26	105	10	
020	#				#			
021	#				#			
022	#				#			
023	26.7	100.6	10	1.2	0			
024	#				#			
025	#				#			

z-scores outside the satisfactory range, i.e. $|z|>2$, are shown in **bold**
# = pesticide not analysed for
LoQ = limit of quantification
* = Result is less than LoQ

Table 2 (continued): Results and z-Scores for γ-HCH, Results for Myclobutanil in Lettuce Purée Test Material

laboratory number	analyte							
	γ-HCH assigned value 21.1 µg/kg				myclobutanil assigned value not set			
	result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score not set
026	#				#			
027	16	70	10	-1.1	13	100	10	
028	22.2	105	10	0.2	18.1	109	10	
029	#				#			
030	#				#			
031	#				#			
032	#				#			
033	17.8	100	10	-0.7	0		10	
034	22			0.2	0		10	
035	#				#			
036	25	100	10	0.8	19	110	10	
037	22	96.1	0.0051	0.2	14	93.5	0.0051	
038	#				#			
039	#				#			
040	#				#			
041	#				#			
042	#				#			
043	#				#			
044	#				#			
045	#				#			
046	#				#			
047	44	standard addition	10	**4.9**	#			
048	15.5	104.5	10	-1.2	0		10	
049	21	104	1	0.0	20	100	2	
050	40.6	92.2	10	**4.2**	24.2	119.7	10	

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

Table 2 (continued): Results and z-Scores for γ-HCH, Results for Myclobutanil in Lettuce Purée Test Material

laboratory number		analyte							
		γ-HCH assigned value 21.1 µg/kg				myclobutanil assigned value not set			
		result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score not set
051		#				#			
052		#				#			
053		0	69.85	0.1 ppm	**-4.5**	0	62.08	0.1 ppm	
054		23.8	80.98	10	0.6	0		10	
055		#				#			
056		22	74	5	0.2	13	80	5	
057		23	90	10	0.4	24	90	2	
058		15	90	10	-1.3	#			
059		<LOQ		100		0		10	
060		19	78	10	-0.5	19	108	10	
061		0		10	**-4.5**	0		10	
062		#				#			
063		22	93	10	0.2	18	90	10	
064		23	100	10	0.4	14	118	10	
065		#				#			
066		0		10	**-4.5**	13.8	89	10	
067		#				47	82	20	
068		#				25	90	10	
069	♣	0			**-4.5**	0			
070		25	89		0.8	16	86		
071		12.0		10	-2.0	0		10	
072		22	72	10	0.2	0		10	
073		#				#			
074		20	91	10	-0.2	10	93	10	
075		19	85	10	-0.5	0	91	10	

z-scores outside the satisfactory range, i.e. $|z|>2$, are shown in **bold** LoQ = limit of quantification
= pesticide not analysed for
Participant comments: ♣ = None of posible pesticides is confirmed by GC-MS

Table 2 (continued): Results and z-Scores for γ-HCH, Results for Myclobutanil in Lettuce Purée Test Material

laboratory number		analyte							
		γ-HCH assigned value 21.1 µg/kg				myclobutanil assigned value not set			
		result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score not set
076		20	90.5	10	-0.2	17	85.0	10	
077		12.90	96.51	10	-1.8	0	120	10	
078		23	102.3	11	0.4	#			
079		26	85	10	1.0	<LOQ	101	12	
080		#				#			
081		0			**-4.5**	30	104	10	
082		20.603	100.34	6	-0.1	#			
083		21.740*	6	99.7	0.1	#			
084		22.640	100.2	6	0.3	#			
085		21.740	101.5	6	0.1	#			
086		13	91	10	-1.7	0		10	
087		22	95	10	0.2	17	103	10	
088		17	90	10	-0.9	0	84	10	
089		#				#			
090	†	28.8	94.6	5	1.6	14.2	114	1	
091	†	#				0			
092		13		10	-1.7	40		10	
093		0		10	**-4.5**	0		10	
094		21.5	101.2	1	0.1	#			
095		14.5	81	5	-1.4	16.8	90	2	
096		18		10	-0.7	0		10	
097	†	29.3	100	10	1.8	<LOQ	77	30	
098		22			0.2	18			
099		22	96	10	0.2	17	98	10	
100		25.3	104	5	0.9	21.4	101	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 3)
* = Result is less than LoQ

Table 2 (continued): Results and z-Scores for γ-HCH, Results for Myclobutanil in Lettuce Purée Test Material

laboratory number	analyte							
	γ-HCH assigned value 21.1 µg/kg				myclobutanil assigned value not set			
	result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score not set
101	0			**-4.5**	16		10	
102	#				#			
103	#				#			
104	#				#			
105	33	138	10	**2.6**	0	117	5	
106	22	95		0.2	39	216		
107	19.61	98	3	-0.3	#			
108	#				#			
109	20.8		10	-0.1	0		10	
110	22			0.2	0			
111	21.4	-	0.3	0.1	#			
112	23	70-110	10	0.4	23	70-110	10	
113	26.2		10	1.1	19.4		10	
114	#				#			
115	19	98	10	-0.5	13	100	10	
116	0.017	114	0.01	**-4.5**	0.022	105	0.01	
117	#				#			
118	7	>90		**-3.0**	#			
119	22.5	104	0.005	0.3	18.2	98	0.005	
120	25	91	10	0.8	18	93	10	
121	11.9	71	10	-2.0	#			
122	10	82	10	**-2.4**	17	100	10	
123	33	100	10	**2.6**	20	95	10	
124	0.022	75	0.001	**-4.5**	#			
125	14	96	10	-1.5	#			

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

Table 2 (continued): Results and z-Scores for γ-HCH, Results for Myclobutanil in Lettuce Purée Test Material

laboratory number		analyte							
		γ-HCH assigned value 21.1 µg/kg				myclobutanil assigned value not set			
		result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score not set
126		21	93	5	0.0	18	74	10	
127		#				#			
128		21.64	98.2	4	0.1	#			
129		21.0	100.0	10	0.0	18.7	100.0	10	
130		0.014		0.005	**-4.5**	#			
131		18	90	10	-0.7	20	90	10	
132		0		10	**-4.5**	<LOQ		50	
133		23	102	10	0.4	0		10	
134		25	88	10	0.8	20	98	10	
135		15	67	10	-1.3	13	76	10	
136		0			**-4.5**	0			
137		31		10	**2.1**	10	90	10	
138		29	82		1.7	0		10	
139	†	2	85.3	2	**-4.1**	24	86.7	4	
140		10		3	**-2.4**	15		10	

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 3)

Table 3: Additional Pesticide Residues Reported

laboratory number	pesticide residue > 15 µg/kg	result µg/kg	recovery %	LoQ µg/kg
090	diphenylamine	21.2	94.4	5
091	folpet	21.63	90	200
097	acephate	27.6	76	25
139	lambda-cyhalothrin	17	92.1	4

Table 4: Assigned Values and Target Standard Deviations

analyte	assigned value, µg/kg				target standard deviation, µg/kg	
	data points n	robust mean $\hat{X}$	robust sd $\hat{\sigma}$	uncertainty u	derived from	σ_p
bromopropylate	47	13.4	3.0	0.4	Horwitz*	2.9
ethion	63	24.4	6.2	0.8	Horwitz*	5.4
γ-HCH	63	21.1	5.5	0.7	Horwitz*	4.6

*see page 7 for appropriate form of the Horwitz equation

Table 5: Number and Percentage of Satisfactory z-Scores

analyte	number of satisfactory scores $\|z\| \leq 2$	total number of scores	satisfactory %
bromopropylate	60	71	85
ethion	74	87	85
γ-HCH	73	97	75

Table 6: Number and Percentage of Participants Correctly Identifying and Obtaining Satisfactory z-Scores for Pesticides Present >15 µg/kg

criteria	number of satisfactory participants	total number of participants	satisfactory %
correctly identified all four pesticides	40	140	29
correctly identified all four pesticides and obtained satisfactory z-scores for bromopropylate, ethion and γ-HCH	33	140	24

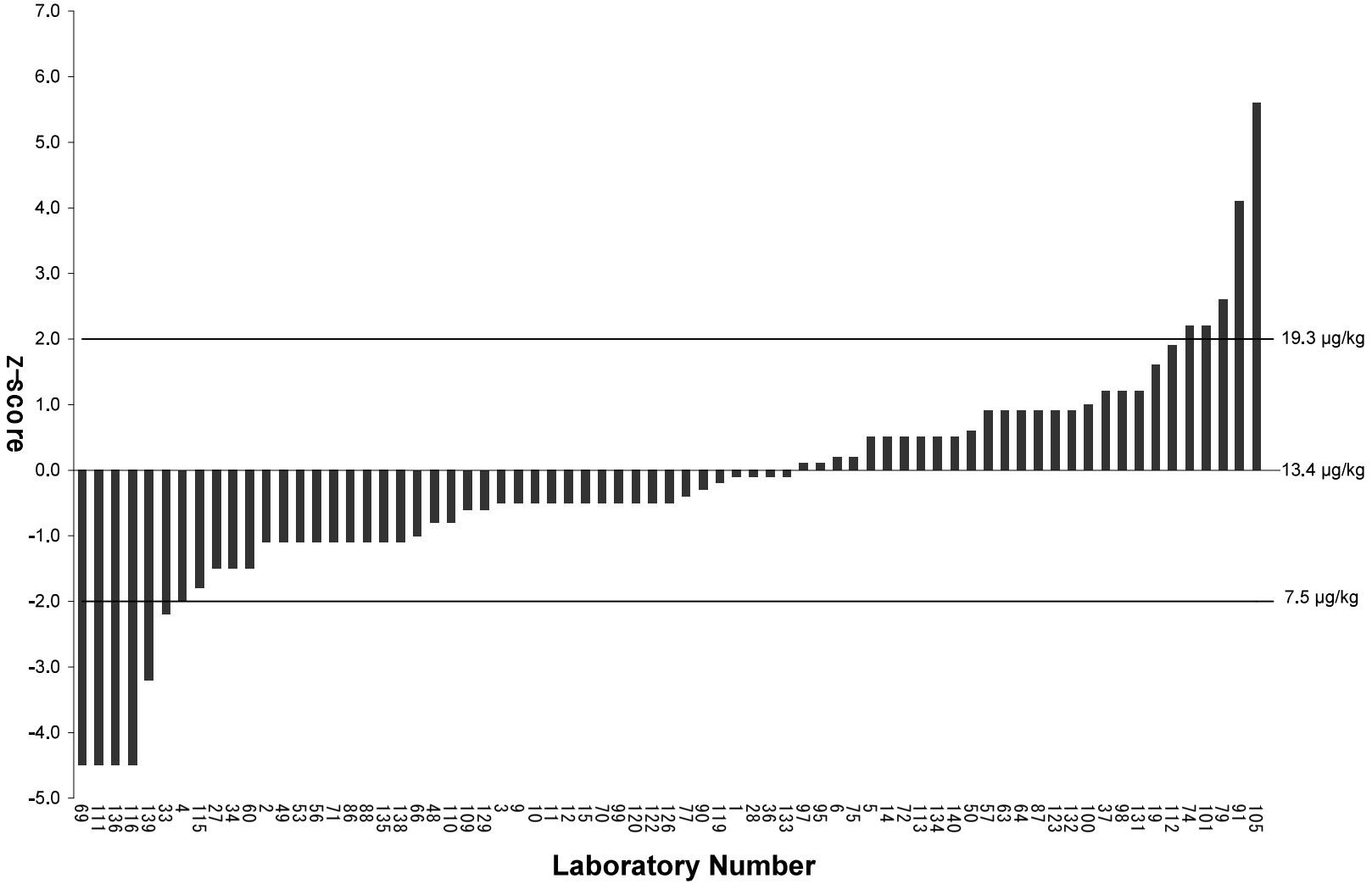

Figure 1: z-Scores for Bromopropylate (13.4 µg/kg) in Lettuce Purée Test Material
participants assigned a result of 0 µg/kg for bromopropylate obtain a z-score of -4.5

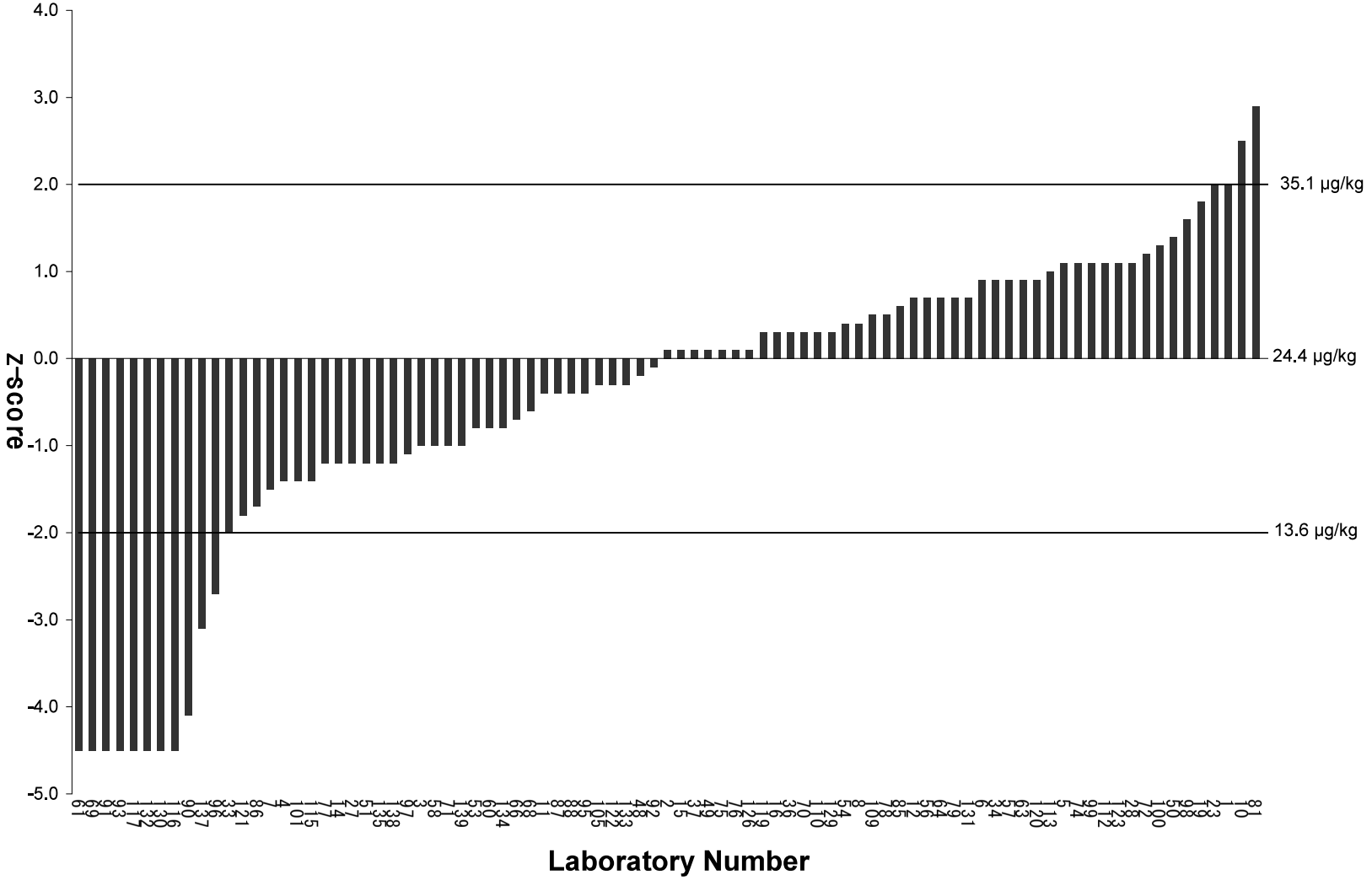

Figure 2: z-Scores for Ethion (24.4 µg/kg) in Lettuce Purée Test Material
participants assigned a result of 0 µg/kg for ethion obtain a z-score of -4.5

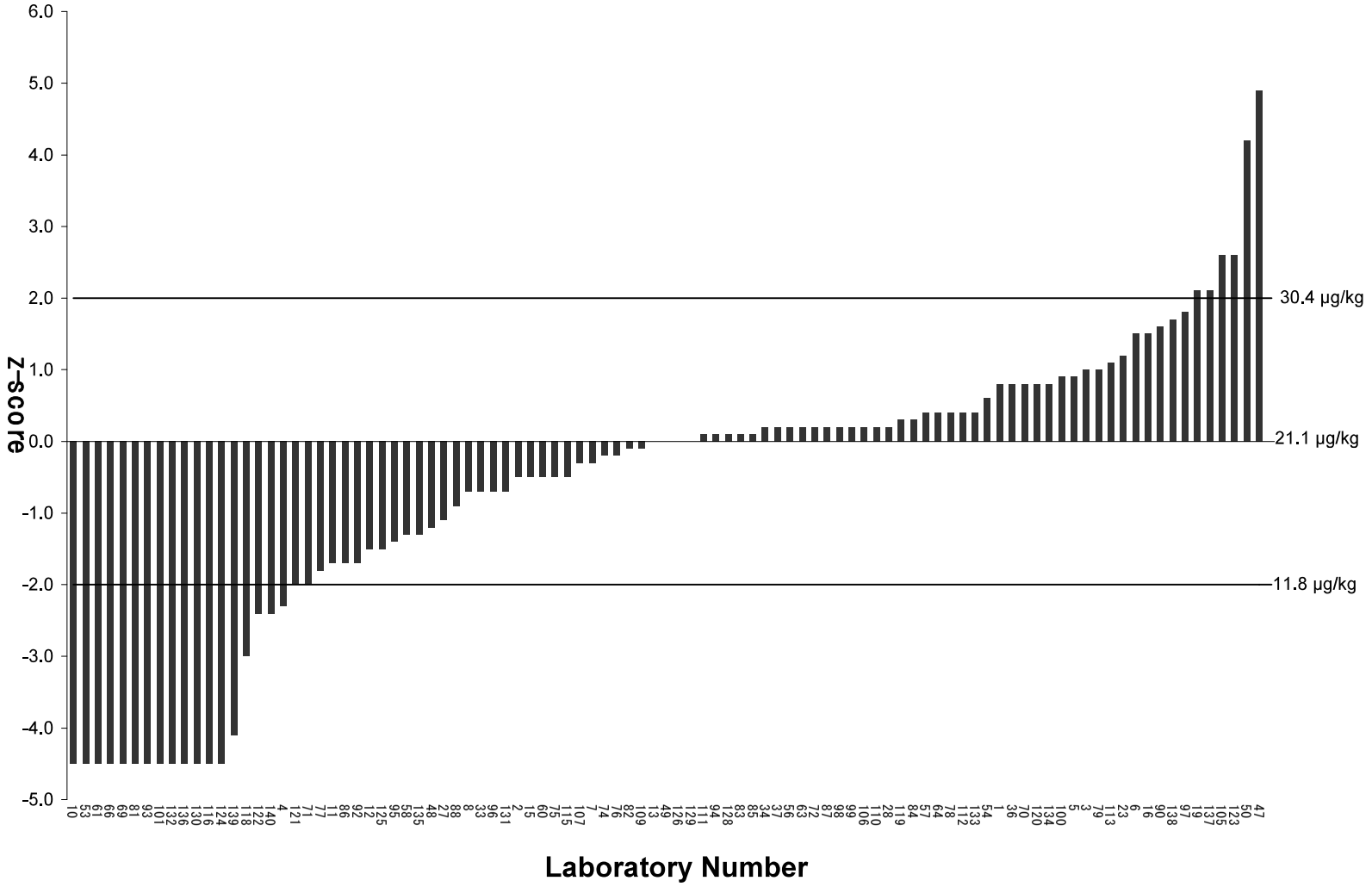

Figure 3: z-Scores for γ-HCH (21.1 µg/kg) in Lettuce Purée Test Material
participants assigned a result of 0 µg/kg for γ-HCH obtain a z-score of –4.5

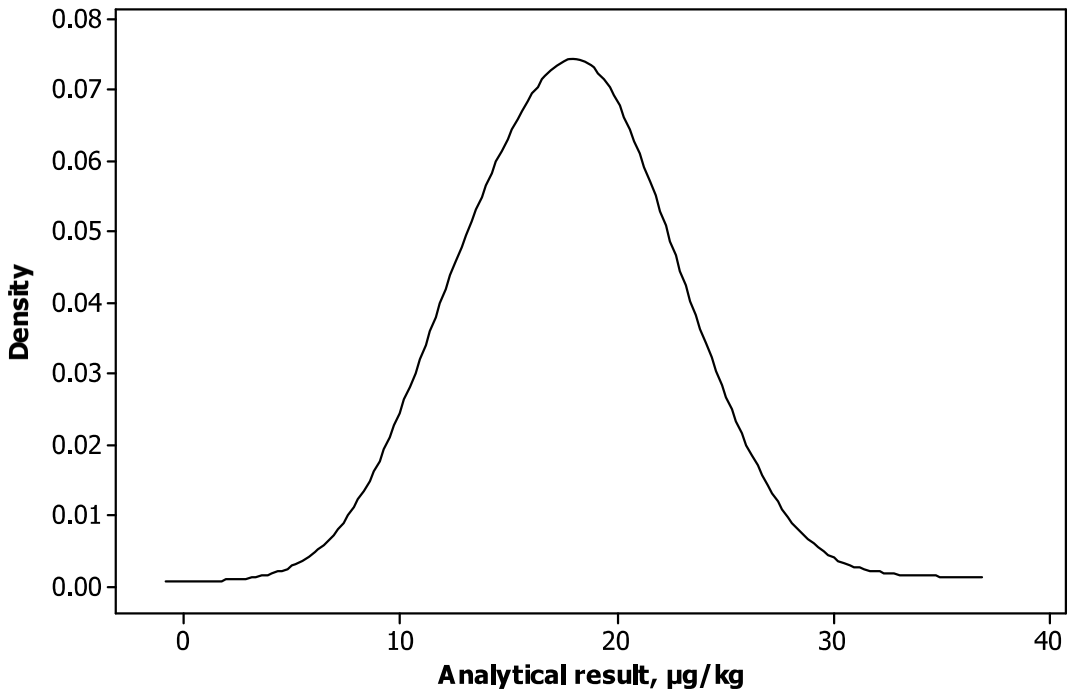

Figure 4: Adaptive Kernel Density Plot for Myclobutanil in Lettuce Purée Test Material

APPENDIX I: Homogeneity Data for Lettuce Purée Test Material

sample id	analyte			
	bromopropylate µg/kg		ethion µg/kg	
	replicate 1	replicate 2	replicate 1	replicate 2
1	10	10	18	21
2	10	11	19	21
3	10	9	19	18
4	10	10	19	22
5	10	9	21	18
6	10	11	19	22
7	9	10	17	19
8	11	12	20	23
9	11	11	23	21
10	11	12	21	21
mean	10		20	
n	20		20	
origin of target sd (σ_p)	Horwitz <120 ppb*		Horwitz <120 ppb*	
RSD%	22.0		22.0	
absolute target sd (σ_p)	2.28		4.42	
s_{an}	0.59		1.70	
s_{sam}^2	0.44		0.15	
σ_{all}^2	0.47		1.76	
critical	1.23		6.24	
s_{sam}^2<critical?	ACCEPT		ACCEPT	

*see page 7 for appropriate form of the Horwitz equation

APPENDIX I (continued): Homogeneity Data for Lettuce Purée Test Material

sample identity	analyte	
	γ-HCH µg/kg	
	replicate 1	replicate 2
1	17	19
2	17	17
3	14	14
4	15	16
5	15	12
6	16	16
7	15	13
8	14	14
9	14	12
10	14	13
mean	15	
n	20	
origin of target sd (σ_p)	Horwitz <120 ppb*	
σ_p as RSD%	22.0	
abs. target sd (σ_p)	3.27	
s_{an}	1.07	
s_{sam}^2	2.26	
σ_{all}^2	0.96	
critical	2.97	
s_{sam}^2 < critical?	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 and Table 2 for this information.
3) Only methods pertinent to bromopropylate, ethion, γ-HCH and myclobutanil have been recorded.

Bromopropylate

Accredited Method Used	laboratory number
yes	001 002 003 004 005 006 010 011 012 014 015 027 028 036 037 048 049 050 053 056 060 063 066 070 071 072 074 077 079 086 088 091 095 099 101 105 109 112 113 115 119 120 122 123 129 131 132 134
no	009 019 033 057 064 075 090 098 100 110 116 121 126

Sample Weight (g)	laboratory number
≥2 -<5	116
≥5 -<10	002 009 015 037 090 109 113 115 131 132 134
≥10 -<25	003 004 005 006 010 011 012 014 019 027 028 033 036 048 049 050 053 056 057 060 064 070 072 074 075 079 086 091 095 098 099 100 101 105 110 112 119 121 122 123 126 129
≥25 -<50	063 071 077 088 097 120
≥50	001 066

Extraction Solvent	laboratory number
petroleum ether/spirit	009 010 072 091
hexane	050
cyclohexane	001 056
dichloromethane	009 010 072 091
ethyl acetate	001 027 033 050 056 057 066 077 086 088 095 115 121 122
acetone	001 003 009 010 012 048 049 050 056 072 074 079 091 097 123 129
acetonitrile	002 004 005 006 011 014 015 019 028 036 037 053 060 063 064 070 071 075 090 098 099 100 101 105 109 110 112 113 116 119 120 126 131 132
methanol	134
water	001 134

Extraction Technique Used	laboratory number
ASE	057
cold solvent extraction at atmospheric pressure	001 003 005 006 009 011 012 014 015 028 033 036 049 056 060 063 064 066 070 072 074 075 079 086 088 090 091 095 097 098 099 100 101 105 109 110 112 115 116 120 121 123 126 129 131 132 134
QUECHERS	071
solvent extraction at increased pressure	077
supercritical fluid extraction	053

Extraction pH Adjusted	laboratory number
yes	002 006 011 014 015 019 028 036 053 056 088 100 101 112 115 119 121 122 131 132
no	001 003 004 005 009 010 012 027 033 037 048 049 050 057 060 063 064 070 071 072 074 075 077 079 086 090 091 095 097 098 099 105 109 113 116 120 123 126 129 134

Sample Clean-up Technique	laboratory number
GPC/HPGPC	001 011 049 056 057 079 088 095 120 121
solid phase extraction (SPE) (column/cartridge)	033 063 064 070 075 077 079 086 105 109 110 115
solid phase extraction (SPE) (dispersive)	005 006 015 028 090 098 099 100 112 131 132
silica column	001
carbon based column	014 019 033 036 064
graphite (GCB-sorbent)	060
NH_2/aminopropyl column	064 105
florisil column	049
PSA	113 126
dispersive sorbent (PSA)	048
QuEChERS (PSA)	101
liquid/liquid extraction	003 015 027 049 053 056 064 074 079 097 116 119 129
Extrelut	134
solvent exchange	064
filter	009 049 064 105
none	012 066 071 072 091 122 123

SPE Column Type	laboratory number
C18	004 110
GCB	014 028 033 063 064 077 079 105 115
Gr	109
Envicarb	014 019 028 033 036 037 053 063 064 077 079 105 115 119
NH_2	004 036 037 053 109 116 119
florisil	074
PSA	011 015 019 070 086 090 099 113 131 134
supelco	075

Certified Standards Used	laboratory number
yes	001 002 003 004 005 006 009 010 011 012 014 015 019 027 028 033 036 037 048 049 050 053 056 057 060 063 066 070 071 072 075 077 079 086 088 090 091 095 097 098 100 101 105 109 110 112 113 115 119 120 121 122 123 126 129 131 134
no	064 074 099 116 132

MS Confirmation	laboratory number
yes	001 002 003 004 005 006 009 010 011 012 014 015 019 027 028 033 036 037 048 049 050 053 056 060 063 064 066 070 071 072 075 077 079 086 088 090 091 095 097 098 100 101 105 109 110 112 113 115 116 119 120 121 122 123 126 129 131 132 134
no	057 074 099

Calibrations	laboratory number
matrix-matched	002 004 005 006 009 010 011 012 027 033 048 050 060 066 070 072 077 086 088 090 095 100 101 109 110 113 115 119 120 121 126 129 131 134
solvent	001 036 049 056 057 064 074 098 099 132
multi-level	005 006 009 010 012 014 015 019 027 033 060 063 071 072 074 098 099 100 101 115 116 119 121 122 123 132
single-level	001 028 048 049 053 056 064 066 075 079 091 097 105 126 129
standard addition	003 005 028 033 037 112

Source of Standards	laboratory number
Chem Service	097
Dr Ehrenstorfer	001 003 005 006 009 010 011 012 015 027 028 033 036 048 056 057 060 066 070 072 077 088 090 091 098 100 101 105 112 115 120 121 123 129 132 134
Eurofins	099

Source of Standards (continued)	laboratory number
Fluka	027 090 112
Hayashi	053 063
Kanto	113
LGC Promochem	049
Neochema	005
qmx	122
Riedel de Haën	037
Sigma/Aldrich	004 006 027 033 071 086 090 095 110 119 126 131
Supelco	004 027
Wako	014 019 064 074 075 079 109

Is quoted percentage recovery measured in same analytical batch as test material?	laboratory number
yes	003 004 005 006 009 011 012 019 027 028 033 037 048 049 050 053 056 060 063 064 066 070 072 074 075 077 086 088 090 095 099 105 109 112 115 116 119 121 122 123 126 129 134
no	001 002 010 014 015 036 057 079 091 097 101 113 120 131 132

If measured in this batch, at what stage was the spike added?	laboratory number
prior to clean up	116
prior to extraction	003 004 006 009 010 011 012 014 019 027 028 033 036 037 048 050 053 056 060 063 064 066 070 072 074 075 077 086 088 090 095 099 100 105 109 115 119 120 121 122 123 126 129 134
prior to instrument measurement	005 112

Level of Spike (µg/kg)	laboratory number
<25	002 005 009 010 011 015 027 028 033 036 048 049 050 056 060 063 064 066 070 086 090 099 115 116 119 121 126 129 134
≥25 -<50	003 005 011 012 028 033 037 088 090 095 097 105 109 112 120 123 134
≥50 -<100	004 033 072 090 100 115 121 122
≥100 -<150	006 014 019 074 075 077 079 099
≥150 -<200	131
≥500	053

Composition of Blank Commodity used for Spiking	laboratory number
lettuce blank provided	005 009 012 014 019 027 028 033 036 048 049 053 056 060 063 064 070 072 075 077 079 086 090 095 105 109 115 116 119 120 121 123 126 129 134
lettuce test material provided	002 003 011 037 099 112 113
lettuce	006 074 097 100
salad	066
blank pear puree	122
in house blank	088 050
strawberry	004

GC Column Type	laboratory number
capillary	002 003 004 005 006 009 010 011 012 014 015 019 027 028 033 036 037 048 053 057 060 063 064 066 070 071 072 074 075 077 086 088 090 091 095 097 098 099 100 101 105 109 112 113 115 116 119 121 123 126 129 131 132 134
megabore	079
narrowbore <0.53 mm id	001 049 050 056 110 122
widebore	120

GC Column Packing	laboratory number
100% methylpolysiloxane	002
95% methyl5% phenylpolysiloxane	001 003 004 005 006 009 011 012 015 019 027 028 033 036 037 048 049 050 053 056 060 063 064 066 070 071 072 074 075 079 086 090 091 095 097 098 099 100 101 105 109 110 112 113 115 119 121 122 123 129 131 132 134
65% methyl35% phenylpolysiloxane	010 049 057 120
50% methyl50% phenylpolysiloxane	088
14% cyanopropylphenyl86% methyl polysiloxane	014 077

GC Injection Volume (µL)	laboratory number
<1	049 126
≥1 -<2	002 003 004 011 028 037 057 071 074 079 086 091 095 101 109 113 115 120 122 129 132
≥2 -<5	010 012 014 019 033 036 048 050 056 063 066 075 077 088 097 105 110 112 116 119 121 123 134
≥5 -<10	001 006 072 098 099 100 131
≥10	005 009 015 027 053 060 070 090

GC Injection Mode	laboratory number
on-column	079
PTV	001 003 005 006 011 015 053 056 070 090 098 100 112 123 126 131 134
split	002
splitless	004 009 010 012 014 019 027 028 033 036 037 049 050 057 063 066 071 072 074 075 077 086 088 091 095 097 101 105 109 110 113 115 116 119 120 121 122 129 132
KAS	099
PTV, solventvent	060
pulsed splitless	048

GC Detector	laboratory number
ECD	002 012 049 056 057 074 121 126 129 134
FID	134
FPD	134
HR-MS	079
ITD	001 098 123
MS	003 005 014 015 019 028 033 037 048 049 053 056 060 063 064 070 071 075 077 086 088 090 091 095 097 099 101 105 109 112 113 115 119 120 122 129 132 134
MS-MS	009 010 011 027 028 036 050 066 072 100 110 112 116 131
NPD	056 121 129 134
TOF-MS	006

HPLC Column Packing	laboratory number
C18	002 028 036 072 090 101 112 129 131
endcapped	090 112 129

HPLC Guard Column Used	laboratory number
yes	028 072 090 116
no	002 014 015 036 112 129 131 132

Mobile Phase Programme	laboratory number
gradient	002 014 015 028 036 072 090 101 112 116 129 131 132

Mobile Phase Components	laboratory number
acetonitrile	002 129 131
methanol	028 036 090 101 112 131 132
acetate	112

Mobile Phase Components (continued)	laboratory number
ammonium formate	090
formic acid	072 090 101 112 129
water	028 090 101 112 129 131 132

HPLC Column Temperature (°C)	laboratory number
ambient	132
>ambient-<50	002 028 036 072 090 112 129 131

HPLC Injection Volume (µL)	laboratory number
<5	129
≥5 -<10	028 036 131 132
≥10 -<25	002 072 090 112
≥25 -<50	101

Mobile Phase Flow Rate (mL/min)	laboratory number
<0.25	002 072 090 131
≥0.25 -<0.75	028 036 101 112 129 132

HPLC Detector Type	laboratory number
MS-MS	002 028 036 072 090 101 112 129 131

Ethion

Accredited Method Used	laboratory number
yes	001 002 003 004 005 006 010 011 012 014 015 016 027 028 036 037 048 049 050 051 053 056 057 060 063 066 070 071 072 074 076 077 078 079 081 085 086 088 095 099 101 105 109 112 113 115 119 120 122 123 129 131 134
no	008 019 033 054 058 064 075 090 098 100 110 116 121 126 130

Sample Weight (g)	laboratory number
≥2 -<5	116
≥5 -<10	002 015 090 109 113 115 131 134
≥10 -<25	003 004 005 006 010 011 012 014 016 019 027 028 033 036 037 048 049 050 053 056 057 058 060 064 070 072 074 075 076 078 079 081 085 086 095 098 099 100 101 105 110 112 119 121 122 123 126 129
≥25 -<50	051 063 071 077 088 097 120 130
≥50	001 054 066

Extraction Solvent	laboratory number
petroleum ether/spirit	010 072
hexane	050 054
cyclohexane	001 056
dichloromethane	010 051 058 072
diethylether	054
ethylacetate	001 027 033 050 054 056 057 066 077 086 088 095 115 121 130
acetone	001 003 010 012 048 049 050 051 054 056 072 074 076 078 079 085 097 101 123 129
acetonitrile	002 004 005 006 008 011 014 015 016 019 028 036 037 053 060 063 064 070 071 075 081 090 098 099 100 105 109 110 112 113 116 119 120 122 126 131

Extraction Solvent (continued)	laboratory number
methanol	134
water	001 101 134

Extraction Technique Used	laboratory number
ASE	057
cold solvent extraction at atmospheric pressure	001 003 005 006 008 011 012 014 015 016 028 033 036 049 056 060 063 064 066 070 072 074 075 078 079 081 086 088 090 095 097 098 099 100 105 109 110 112 115 116 120 121 123 126 129 131 134
maceration with solvent	130
QUECHERS	071
solvent extraction	076
solvent extraction at increased pressure	085
supercritical fluid extraction	053

Extraction pH Adjusted?	laboratory number
yes	002 006 011 014 015 016 019 028 036 053 056 066 081 088 100 112 115 119 121 131
no	001 003 004 005 008 010 012 027 033 037 048 049 050 051 054 057 058 060 063 064 070 071 072 074 075 076 077 078 079 085 086 090 095 097 098 099 101 105 109 113 116 120 122 123 126 129 130 134

Sample Clean-up Technique	laboratory number
GPC/HPGPC	001 008 011 049 056 057 079 088 095 120 121
solid phase extraction (SPE) (column/cartridge)	033 063 064 070 075 077 078 079 081 086 105 109 110 115
solid phase extraction (SPE) (dispersive)	005 006 015 028 090 098 099 100 112 131
silica column	001 051 076
carbon based column	014 019 033 036 064 130
graphite (GCB-sorbent)	060

Sample Clean-up Technique (continued)	laboratory number
NH_2/aminopropyl column	064 105
florisil column	049 054 101 130
PSA	016 113 126
dispersive sorbent (PSA)	048
liquid/liquid extraction	003 015 027 049 053 056 064 074 079 085 097 101 116 119 129
solvent exchange	064
Extrelut	134
filter	049 064 105
none	012 066 071 072 123

SPE Column Type	laboratory number
C18	004 110
silica	054
GCB	014 028 033 063 064 077 079 081 105 115
Gr	109
Envicarb	008 014 019 028 033 036 037 053 063 064 077 079 081 105 115 119 122 130
NH_2	004 008 036 037 053 109 116 119 122
florisil	074 078 130
PSA	011 015 019 070 086 090 099 113 131 134
supelco	075

Were Certified Standards Used	laboratory number
yes	001 002 003 004 005 006 008 010 011 012 014 015 016 019 027 028 033 036 048 049 050 051 053 056 057 058 060 063 066 070 071 072 075 077 078 079 081 085 086 088 090 095 097 098 100 101 105 109 110 112 113 115 119 120 121 122 123 126 129 130 131 134
no	037 054 064 074 076 099 116

MS Confirmation	laboratory number
yes	001 002 003 004 005 006 008 010 011 012 014 015 016 019 027 028 033 036 037 048 049 050 051 053 054 056 058 060 063 064 066 070 071 072 075 076 077 078 079 081 085 086 088 090 095 097 098 100 105 109 110 112 113 115 116 119 120 121 122 123 126 129 131 134
no	057 074 099 101 130

Calibrations	laboratory number
matrix-matched	002 004 005 006 010 011 012 016 027 033 048 050 058 060 066 070 072 077 086 088 090 095 100 109 110 113 115 119 120 121 126 129 131 134
solvent	001 036 049 056 057 064 074 098 099 101 130
multi-level	005 006 008 010 012 014 015 019 027 033 051 054 060 063 071 072 074 076 078 081 085 098 099 100 115 116 119 121 122 123
single-level	001 028 048 049 053 056 064 066 075 079 097 101 105 126 129
standard addition	003 005 028 033 037 112

Source of Standards	laboratory number
Chem Service	054 097 130
Dr Ehrenstorfer	001 003 005 006 010 011 012 015 016 027 028 033 036 037 048 051 056 057 060 066 070 072 077 078 085 088 090 098 100 105 112 115 120 121 123 129 134

Source of Standards (continued)	laboratory number
Eurofins	099
Fluka	027 090 112
Hayashi	053 063
Kanto	113
LGC Promochem	049
Neochema	005
Sigma/Aldrich	004 006 027 033 071 086 090 095 110 119 126 131
Supelco	004 027
Wako	008 014 019 064 074 075 076 079 081 109

Is quoted percentage recovery measured in same analytical batch as test material?	laboratory number
yes	003 004 005 006 008 011 012 016 019 027 028 033 037 048 049 050 051 053 056 060 063 064 066 070 072 074 075 076 077 078 081 085 086 088 090 095 099 101 105 109 112 115 116 119 121 122 123 126 129 130 134
no	001 002 010 014 015 036 054 057 079 097 113 120 131

If measured in this batch, at what stage was the spike added?	laboratory number
prior to clean up	008 116
prior to extraction	003 004 006 010 011 012 014 016 019 027 028 033 036 037 048 050 051 053 056 058 060 063 064 066 070 072 074 075 076 077 078 081 085 086 088 090 095 099 100 101 105 109 115 119 120 121 122 123 126 129 130 134
prior to instrument measurement	005 112

Level of Spike (µg/kg)	laboratory number
<25	002 005 010 011 012 015 016 027 028 033 036 050 051 056 060 063 064 066 070 076 078 086 090 099 115 116 119 121 126 129 130 134
≥25 -<50	003 005 011 028 033 048 049 058 088 090 095 097 105 109 112 120 123 134
≥50 -<100	004 008 033 072 085 090 100 115 121 122
≥100 -<150	006 014 019 037 074 075 077 079 099
≥150 -<200	131
≥200 -<250	081
≥500	053

Composition of Blank Commodity used for Spiking	laboratory number
lettuce blank provided	005 008 012 014 016 019 027 028 033 036 048 049 053 056 058 060 063 064 070 072 075 076 077 078 079 081 085 086 090 095 101 105 109 115 116 119 120 121 122 123 126 129 130 134
lettuce test material provided	002 003 011 051 099 112 113
lettuce	006 074 097 100
salad	066
strawberry	004
solvent	037
in house blank	088 050

GC Column Type	laboratory number
capillary	002 003 004 005 006 010 011 012 014 015 016 019 027 028 033 036 037 048 051 053 054 057 058 060 063 064 070 071 072 074 075 077 078 081 085 086 088 090 095 097 098 099 100 101 109 112 113 115 116 119 121 126 129 130 131 134
megabore	076 079

GC Column Type (continued)	laboratory number
narrowbore <0.53 mm id	001 049 050 056 110 122
widebore	120

GC Column Packing	laboratory number
100% methylpolysiloxane	002 048 076
95% methyl5% phenylpolysiloxane	001 003 004 005 006 008 011 012 015 019 027 028 033 036 037 049 050 053 054 056 060 063 064 070 071 072 075 078 081 085 086 090 095 097 098 099 100 101 109 110 112 113 115 119 121 122 129 130 131 134
65% methyl35% phenylpolysiloxane	010 049 057 120
50% methyl50% phenylpolysiloxane	088 101
5% methyl95% phenylpolysiloxane	016
14% cyanopropylphenyl86% methyl polysiloxane	014 077 079
trifluoropropylmethylpolysiloxane	074

GC Injection Volume (µL)	laboratory number
<1	049 126
≥1 -<2	002 003 004 011 016 028 037 054 057 071 078 079 085 086 095 101 109 113 115 120 122 129 130
≥2 -<5	008 010 012 014 019 033 036 048 050 051 056 063 074 076 077 081 088 097 110 112 116 119 121 134
≥5 -<10	001 006 058 072 098 099 100 131
≥10	005 015 027 053 060 070 075 090

GC Injection Mode	laboratory number
on-column	079
PTV	001 003 005 006 011 015 016 053 056 070 090 098 100 112 126 131 134
split	002 085

GC Injection Mode (continued)	laboratory number
splitless	004 008 010 012 014 019 027 028 033 036 037 048 049 050 051 054 057 058 063 071 072 074 075 076 077 078 081 086 088 095 097 101 109 110 113 115 116 119 120 121 122 129 130
KAS	099
PTV, solventvent	060

GC Detector	laboratory number
ECD	002 051 056 085 121 129 130 134
FID	134
FPD	008 012 048 049 051 054 057 074 075 076 079 130 134
ITD	001 098
MS	003 005 014 015 016 019 028 033 037 049 053 056 060 063 064 070 071 081 086 088 090 095 097 099 109 112 113 115 119 120 122 129 134
MS-MS	010 011 027 028 036 050 058 072 100 110 112 116 131
NPD	049 056 078 101 121 126 129 134
PFPD	077
TOF-MS	006

HPLC Column Packing	laboratory number
C18	002 028 036 066 072 090 105 123 131 112 129
C8	015
endcapped	090 112 129

HPLC Guard Column Used	laboratory number
yes	015 028 066 072 090 105 116
no	002 014 036 112 123 129 131

Mobile Phase Programme	laboratory number
gradient	002 014 015 028 036 066 072 090 105 112 116 123 129 131

Mobile Phase Components	laboratory number
acetonitrile	002 105 129 131
methanol	015 028 036 066 090 112 123 131
acetate	112
ammonium formate	066 090
formic acid	072 090 105 112 129
water	015 028 090 105 112 129 131

HPLC Column Temperature (°C)	laboratory number
ambient	066
>ambient-<50	002 015 028 036 072 090 105 112 123 129 131

HPLC Injection Volume (µL)	laboratory number
<5	015 123 129
≥5 -<10	028 036 066 131
≥10 -<25	002 072 090 105 112

Mobile Phase Flow Rate (mL/min)	laboratory number
<0.25	002 015 072 090 131
≥0.25 -<0.75	028 036 066 105 112 123 129

HPLC Detector Type	laboratory number
MS-MS	002 015 028 036 066 072 090 105 112 123 129 131

γ-HCH

Accredited Method Used	laboratory number
yes	001 002 003 004 005 006 011 012 015 016 027 028 036 037 047 048 049 050 056 057 060 063 070 071 072 074 076 077 078 079 082 083 084 085 086 088 095 099 105 107 109 112 113 115 119 120 122 123 125 128 129 131 134 135
no	008 013 019 033 054 058 064 075 090 098 100 106 110 111 116 118 121 124 126 130

Sample Weight (g)	laboratory number
≥2 -<5	116
≥5 -<10	002 015 037 090 109 113 115 122 131 134
≥10 -<25	003 004 005 006 011 012 016 019 027 028 033 036 047 048 049 050 056 057 058 060 064 070 072 074 075 076 078 079 082 083 084 085 086 095 098 099 100 105 106 107 110 112 119 121 123 124 125 126 128 129
≥25 -<50	013 063 071 077 088 097 120 130 135
≥50	001 054 111 118

Extraction Solvent	laboratory number
petroleum ether/spirit	072 124
hexane	050 054 111 128
cyclohexane	001 056 122
dichloromethane	058 072 124
diethylether	054
ethylacetate	001 027 033 047 050 054 056 057 077 086 088 095 115 118 121 122 130 135
acetone	001 003 012 048 049 050 054 056 072 074 076 078 079 082 083 084 085 097 107 111 123 124 128 129
acetonitrile	002 004 005 006 008 011 013 015 016 019 028 036 037 060 063 064 070 071 075 090 098 099 100 105 106 109 110 112 113 116 119 120 125 126 131

Extraction Solvent (continued)	laboratory number
methanol	134
water	001 111 134

Extraction Technique Used	laboratory number
ASE	057
cold solvent extraction at atmospheric pressure	001 003 005 006 008 011 012 013 015 016 028 033 036 047 049 056 060 063 064 070 072 074 075 078 079 086 088 090 095 097 098 099 100 105 106 109 110 111 112 115 116 120 121 123 124 125 126 128 129 131 134
maceration with solvent	130
QUECHERS	071
solvent extraction	076
solvent extraction at increased pressure	077 082 083 084 085 107

Extraction pH Adjusted	laboratory number
yes	002 006 011 015 016 019 028 036 047 056 088 100 112 115 119 121 131 135
no	001 003 004 005 008 012 013 027 033 037 048 049 050 054 057 058 060 063 064 070 071 072 074 075 076 077 078 079 082 083 084 085 086 090 095 097 098 099 105 106 107 109 111 113 116 118 120 122 123 124 125 126 128 129 130 134

Sample Clean-up Technique	laboratory number
GPC/HPGPC	001 008 011 049 056 057 079 088 095 120 121 122
solid phase extraction (SPE) (column/cartridge)	033 047 063 064 070 075 077 078 079 086 105 106 109 110 115 125 128
solid phase extraction (SPE) (dispersive)	005 006 015 028 090 098 099 100 112 131
silica column	001
carbon based column	019 033 036 064 106 130
graphite (GCB-sorbent)	060
NH_2/aminopropyl column	064 105
florisil column	049 054 076 118 130
PSA	016 113 126
dispersive sorbent (PSA)	048
extraction	124
liquid/liquid extraction	003 013 015 027 049 056 064 074 079 082 083 084 085 097 107 116 119 129
solvent exchange	064
Extrelut	134
filter	049 064 105 106
none	012 071 072 111 123

SPE Column Type	laboratory number
C18	110 004
silica	054 013
Envicarb	008 036 037 119 130 013 028 033 047 063 064 077 079 105 115 125 019
GCB	028 033 047 063 064 077 079 105 115 125
Gr	109
NH_2	106 116 004 008 036 037 119 013 109
florisil	074 078 082 128 130 107 (FLO)
FL	083 084
PSA	011 015 070 086 090 099 113 131 134 019
supelco	075

Were Certified Standards Used	laboratory number
yes	001 002 003 004 005 006 008 011 012 015 016 019 027 028 033 036 037 047 048 049 050 056 057 058 060 063 070 071 072 075 077 078 079 082 083 084 085 086 088 090 095 097 098 100 105 107 109 110 111 112 113 115 119 120 121 122 123 124 125 126 128 129 130 131 134 135
no	013 054 064 074 076 099 106 116 118

MS Confirmation	laboratory number
yes	001 002 003 004 005 006 008 011 012 013 015 016 019 027 028 033 036 037 047 048 049 050 054 056 058 060 063 064 070 071 072 075 076 077 078 079 082 083 084 085 086 088 090 095 097 098 100 105 106 107 109 110 111 112 113 115 116 118 119 120 121 122 123 124 125 126 128 129 131 134 135
no	057 074 099 130

Calibrations	laboratory number
matrix-matched	002 004 005 006 011 012 016 027 033 047 048 050 058 060 070 072 077 086 088 090 095 100 109 110 113 115 119 120 121 126 128 129 131 134
solvent	001 036 049 056 057 064 074 098 099 111 130
multi-level	005 006 008 012 013 015 019 027 033 054 060 063 071 072 074 076 078 082 083 084 085 098 099 100 106 107 115 116 118 119 121 122 123 124 125 128
single-level	001 028 048 049 056 064 075 079 097 105 111 126 129 135
standard addition	003 005 028 033 037 112

Source of Standards	laboratory number
Chem Service	054 097 130
Dr Ehrenstorfer	001 003 005 006 011 012 015 016 027 028 033 036 048 056 057 060 070 072 076 077 078 082 083 084 085 088 090 095 098 100 105 111 112 115 118 120 121 122 123 124 128 129 134
Eurofins	099
Fluka	027 090 111 112
Hayasi	063
Kanto	113
LGC Promochem	049
Neochema	005
Riedel-de Haën	107
Sigma/Aldrich	004 006 027 033 047 071 086 090 110 111 119 126 131
Supelco	004 027 111
Wako	008 013 019 037 064 074 075 079 106 109 125

Is quoted percentage recovery measured in same analytical batch as test material?	laboratory number
yes	003 004 005 006 008 011 012 013 016 019 027 028 033 037 047 048 049 050 056 060 063 064 070 072 074 075 076 077 078 082 083 084 085 086 088 090 095 099 105 106 107 109 112 115 116 118 119 121 122 123 125 126 128 129 130 134
no	001 002 015 036 054 057 079 097 111 113 120 124 131 135

If measured in this batch, at what stage was the spike added?	laboratory number
prior to clean up	008 116
prior to extraction	003 004 006 011 012 013 016 019 027 028 033 036 037 047 048 050 056 058 060 063 064 070 072 074 075 076 077 078 082 083 084 085 086 088 090 095 099 100 105 106 107 109 111 115 119 120 121 122 123 124 125 126 128 129 130 134

If measured in this batch, at what stage was the spike added? (continued)	laboratory number
prior to instrument measurement	005 112 118

Level of Spike (µg/kg)	laboratory number
<25	002 005 011 012 015 016 027 028 033 036 048 049 050 056 060 063 064 070 076 078 086 090 099 107 115 116 119 121 124 126 128 129 130 134
≥25 -<50	003 005 011 028 033 037 058 088 090 095 097 105 109 112 120 123 128 134 135
≥50 -<100	004 008 033 047 072 074 082 083 084 085 090 100 106 115 121 122
≥100 -<150	006 013 019 075 077 079 099 111 125
≥150 -<200	131

Composition of Blank Commodity used for Spiking	laboratory number
lettuce blank provided	005 008 012 013 016 019 027 028 033 036 048 049 056 058 060 063 064 070 072 075 076 077 078 079 082 083 084 085 086 090 095 105 106 109 111 115 116 119 120 121 123 125 126 128 129 130 134
lettuce test material provided	002 003 011 037 047 099 112 113 124
lettuce	006 074 097 100
peach	135
strawberry	004
in house blank	050 088 122

GC Column Type	laboratory number
capillary	002 003 004 005 006 011 012 013 015 016 019 027 028 033 036 037 048 054 057 058 060 063 064 070 071 072 074 075 076 077 078 082 083 084 085 086 088 090 095 097 098 099 100 105 106 107 109 111 112 113 115 116 118 119 121 123 124 126 128 129 130 131 134 135
megabore	079
narrowbore <0.53 mm id	001 047 049 050 056 110 122 125
widebore	120

GC Column Packing	laboratory number
100% methylpolysiloxane	002 076
95% methyl5% phenylpolysiloxane	001 003 004 005 006 008 011 012 015 019 027 028 033 036 037 049 050 054 056 060 063 064 070 071 072 075 078 079 082 083 084 085 086 090 095 097 098 099 100 105 106 107 109 110 112 113 115 119 121 122 123 124 125 128 129 130 131 134 135
12% phenyl88% methylpolysiloxane	111
14% phenyl86% methylpolysiloxane	047
65% methyl35% phenylpolysiloxane	049 057 120
50% methyl50% phenylpolysiloxane	013 088 118
5% methyl95% phenylpolysiloxane	016
14% cyanopropylphenyl86% methyl polysiloxane	048 074 077 107 111 128

GC Injection Volume (µL)	laboratory number
<1	049 126
≥1 -<2	002 003 004 011 013 016 028 037 047 048 054 057 071 074 078 079 082 083 084 085 086 095 107 109 111 113 115 118 120 122 124 128 129 130
≥2 -<5	008 012 019 033 036 050 056 063 075 076 077 088 097 105 106 110 112 116 119 121 123 125 134

GC Injection Volume (µL) (continued)	laboratory number
≥5 - <10	001 006 058 072 098 099 100 131
≥10	005 015 027 060 070 090 135

GC Injection Mode	laboratory number
KAS	099
on-column	079
PTV	001 003 005 006 011 015 016 056 070 090 098 100 112 123 126 131 134 135
PTV, solventvent	060
pulsed splitless	048
split	002 047 078 082 083 084 085 107 128
splitless	004 008 012 013 019 027 028 033 036 037 049 050 054 057 058 063 071 072 074 075 076 077 086 088 095 097 105 106 109 110 111 113 115 116 118 119 120 121 122 124 125 129 130

GC Detector	laboratory number
ECD	002 008 012 013 049 054 056 057 074 076 078 082 083 084 085 107 111 121 124 126 128 129 130 134
FID	134
FPD	130 134
HR-MS	079
ITD	001 098 123
MS	003 005 015 016 019 028 033 037 047 049 056 060 063 064 070 071 075 077 086 088 090 095 097 099 105 106 109 112 113 115 118 119 120 122 125 128 129 134 135
MS-MS	011 027 028 036 050 058 072 100 106 110 112 116 131
NPD	056 121 129 134
TOF-MS	006
XSD	048

HPLC Column Packing	laboratory number
C18	002 028 036 072 090 131 112 129
endcapped	090 112 129

HPLC Guard Column Used	laboratory number
yes	028 072 090 106 116 135
no	002 015 036 084 112 129 131

Mobile Phase Programme	laboratory number
gradient	002 015 028 036 072 090 112 116 129 131 135
isocratic	084 106

Mobile Phase Components	laboratory number
acetonitrile	002 129 131
methanol	028 036 090 112 131
acetate	112
ammonium formate	090
formic acid	072 090 112 129
water	028 090 112 129 131

HPLC Column Temperature (°C)	laboratory number
>ambient-<50	002 028 036 072 090 112 129 131

HPLC Injection Volume (µL)	laboratory number
<5	129
≥5 -<10	028 036 131
≥10 -<25	002 072 090 112

Mobile Phase Flow Rate (mL/min)	laboratory number
<0.25	002 072 090 131
≥0.25 -<0.75	028 036 112 129

HPLC Detector Type	laboratory number
MS-MS	002 028 036 072 090 112 129 131

Myclobutanil

Accredited Method Used	laboratory number
yes	001 002 003 004 005 006 010 011 012 014 016 027 028 036 049 050 056 060 063 066 070 074 076 081 095 099 101 112 113 115 119 120 122 123 129 131 134 135
no	008 013 019 037 057 064 067 090 098 100 106 116 126

Sample Weight (g)	laboratory number
≥2 -<5	116
≥5 -<10	002 090 113 115 131 134
≥10 -<25	003 004 005 006 010 011 012 014 016 019 027 028 036 037 049 050 056 057 060 064 067 070 074 076 081 095 098 099 100 101 106 112 119 122 123 126 129
≥25 -<50	013 063 120 135
≥50	001 066

Extraction Solvent	laboratory number
petroleum ether/spirit	010 050
hexane	050
cyclohexane	001 056
dichloromethane	010
ethyl acetate	001 027 056 057 066 067 115 122 135
acetone	001 003 010 012 049 050 056 074 123 129
acetonitrile	002 004 005 006 008 011 013 014 016 019 028 036 037 060 063 064 070 076 081 090 095 098 099 100 101 106 112 113 116 119 120 126 131
methanol	134
water	001 134

Extraction Technique Used	laboratory number
cold solvent extraction at atmospheric pressure	001 003 005 006 008 011 012 013 014 016 028 036 049 056 060 063 064 066 067 070 074 081 090 095 098 099 100 106 112 115 116 120 123 126 129 131 134
ASE	057
solvent extraction	076

Extraction pH Adjusted	laboratory number
yes	002 006 011 014 016 019 028 036 056 066 081 100 101 112 115 119 131 135
no	001 003 004 005 008 010 012 013 027 037 049 050 057 060 063 064 067 070 074 076 090 095 098 099 106 113 116 120 122 123 126 129 134

Sample Clean-up Technique	laboratory number
GPC/HPGPC	001 008 011 037 049 056 057 067 120
solid phase extraction (SPE)	037
solid phase extraction (SPE) (column/cartridge)	063 064 070 076 081 106 115
solid phase extraction (SPE) (dispersive)	005 006 028 090 098 099 100 101 112 131

Sample Clean-up Technique (continued)	laboratory number
silica column	001
carbon based column	014 019 036 064 106
graphite (GCB-sorbent)	060
NH_2/aminopropyl column	064
PSA	016 101 113 126
liquid/liquid extraction	003 013 027 049 056 064 074 116 119 129
solvent exchange	064
Extrelut	134
filter	049 064 106
none	012 066 095 122 123

SPE Column Type	laboratory number
C18	004
Silica	013
Envicarb	008 013 014 019 028 036 037 063 064 081 115 119
graphite-carbon	074
GCB	014 028 063 064 081 115
NH_2	004 008 013 036 037 074 106 116 119
PSA	011 019 070 090 099 113 131 134

Certified Standards Used	laboratory number
yes	001 002 003 004 005 006 008 010 011 012 014 016 019 027 028 036 049 050 056 057 060 063 066 070 081 090 095 098 100 101 112 113 115 119 120 122 123 126 129 131 134 135
no	013 037 064 067 074 076 099 106 116

MS Confirmation	laboratory number
yes	001 002 003 004 005 006 008 010 011 012 013 014 016 019 027 028 036 037 049 050 056 060 063 064 066 067 070 076 081 090 095 098 100 101 106 112 113 115 116 119 120 122 123 126 129 131 134 135
no	057 074 099

Calibrations	laboratory number
matrix-matched	002 004 005 006 010 011 012 016 027 050 060 066 067 070 090 095 100 113 115 119 120 126 129 131 134 135
solvent	001 036 049 056 057 064 074 098 099
multi-level	005 006 008 010 012 013 014 019 027 060 063 074 076 081 098 099 100 101 106 115 116 119 122 123
single-level	001 028 049 056 064 066 126 129 135
standard addition	003 005 028 037 112

Source of Standards	laboratory number
Dr Ehrenstorfer	001 003 005 006 010 011 012 016 027 028 036 056 057 060 066 067 070 074 090 095 098 100 101 112 115 120 122 123 129 134
Eurofins	099
Fluka	027 090 112
Hayasi	063
Kanto	113
LGC Promochem	049
Neochema	005
Sigma/Aldrich	004 006 027 090 119 126 131
Supelco	004 027
Wako	008 013 014 019 037 064 076 081 106

Is quoted percentage recovery measured in same analytical batch as test material?	laboratory number
yes	003 004 005 006 008 011 012 013 016 019 027 028 037 049 050 056 060 063 064 066 070 074 076 081 090 095 099 101 106 112 115 116 119 122 123 126 129 134
no	001 002 010 014 036 057 067 113 120 131 135

If measured in this batch, at what stage was the spike added?	laboratory number
prior to extraction	003 004 006 010 011 012 013 014 016 019 027 028 036 037 050 056 060 063 064 066 070 074 076 081 090 095 099 100 101 106 115 119 120 122 123 126 129 134 135
prior to clean up	008 116
prior to instrument measurement	005 112

Level of Spike (µg/kg)	laboratory number
<25	002 005 010 011 012 016 027 028 036 049 056 060 063 064 066 067 070 076 090 095 099 101 115 116 119 120 126 129 134
≥25 -<50	003 005 011 028 090 112 123 134
≥50 -<100	004 008 050 090 100 106 115 122 135
≥100 -<150	006 013 014 019 037 074 099
≥150 -<200	131
≥200 -<250	081

Composition of Blank Commodity used for Spiking	laboratory number
blank provided	005 008 012 013 014 016 019 027 028 036 049 056 060 063 064 070 076 081 090 095 106 115 116 119 120 122 123 126 129 134
test material provided	002 003 011 099 112 113
lettuce	006 074 100
salad	066
other tomato	135

Composition of Blank Commodity used for Spiking (continued)	laboratory number
strawberry	004 101
own blank	050

GC Column Type	laboratory number
capillary	002 003 004 005 006 010 011 012 013 014 016 019 027 028 036 037 060 063 064 067 070 076 081 090 098 100 106 112 113 115 116 119 126 129 131 134 135
narrowbore <0.53 mm id	001 049 056 122
widebore	120

GC Column Packing	laboratory number
100% methylpolysiloxane	002 067
95% methyl5% phenylpolysiloxane	001 003 004 005 006 008 011 012 019 027 028 036 037 049 056 060 063 064 070 076 081 090 098 100 106 112 113 115 119 122 129 131 134 135
65% methyl35% phenylpolysiloxane	010 120
50% methyl50% phenylpolysiloxane	013
5% methylsiloxane 95% phenylpolysiloxane	016
14% cyanopropylphenyl86% methyl polysiloxane	014

GC Injection Volume (µL)	laboratory number
<1	126
≥1 -<2	002 003 004 011 013 016 028 037 057 067 113 115 120 122 129
≥2 -<5	008 010 012 014 019 036 049 056 063 076 081 106 112 116 119 134
≥5 -<10	001 006 098 100 131
≥10	005 027 060 070 090 135

GC Injection Mode	laboratory number
PTV	001 003 005 006 011 016 049 056 060 070 090 098 100 112 126 131 134
solventvent	060
split	002
splitless	004 008 010 012 013 014 019 027 028 036 037 050 057 063 067 076 081 106 113 115 116 119 120 122 129

GC Detector	laboratory number
ECD	002 008 012 056 067 126 129 134
FID	134
FPD	134
ITD	001 098
MS	003 005 014 016 019 028 037 049 056 060 063 064 070 076 081 090 106 112 113 119 120 122 129 134 135
MS-MS	010 011 027 028 036 100 106 112 115 116 131
NPD	013 056 129 134
TOF-MS	006

HPLC Column Packing	laboratory number
C18	002 016 028 036 050 057 066 074 090 095 099 101 123 131 112 129
endcapped	090 112 129

HPLC Guard Column Used	laboratory number
yes	016 028 050 057 066 090 099 106 116
no	002 014 036 074 095 101 112 123 129 131

Mobile Phase Programme	laboratory number
gradient	002 014 016 028 036 050 057 066 074 090 095 099 101 112 116 123 129 131
isocratic	106

Mobile Phase Components	laboratory number
acetonitrile	002 050 057 129 131
methanol	016 028 036 066 074 090 095 099 101 112 123 131
acetate	112
acetic acid	099
acetic acid ammonium	074
ammonium formate	066 090
formic acid	050 090 112 129
water	016 028 050 090 095 101 112 129 131

HPLC Column Temperature (°C)	laboratory number
ambient	016 066
>ambient-<50	002 028 036 050 057 074 090 095 099 101 112 123 129 131

HPLC Injection Volume (µL)	laboratory number
<5	074 123 129
≥5 -<10	016 028 036 050 066 095 131
≥10 -<25	002 057 090 099 112
≥25 -<50	101

Mobile Phase Flow Rate (mL/min)	laboratory number
<0.25	002 074 090 131
≥0.25 -<0.75	016 028 036 050 057 066 095 099 101 112 123 129

HPLC Detector Type	laboratory number
MS-MS	002 016 028 036 050 057 066 074 090 095 099 101 112 123 129 131

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.
- 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

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

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