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LABORATORY NUMBER: Available to participants from FAPAS SecureWeb

FAPAS® Proficiency Test 0961

Pesticide Residues in Wheat Flour

October - November 2009

Report

Prepared on behalf of FAPAS® by

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UKAS PROFICIENCY TESTING 0009 PT

SUMMARY

1. The test material for FAPAS® Proficiency Test 0961 was dispatched in October 2009. Each participant received a wheat flour test material to be analysed for pesticide residues. In total, 123 test materials were distributed to participants from 33 countries. Of these, 113 participants, i.e. 92%, returned results for some combination of the analytes within the time-scale demanded by the Scheme.

2. From a list of 46 possible pesticide residues, participants were requested to identify and quantify those present in the wheat flour. The test material contained fenitrothion, pirimiphos-methyl, tebuconazole and trifloxystrobin.

3. The assigned value ($\hat{X}$) for each analyte was calculated from the most appropriate measure of central tendency of participants' results [1, 2].

4. The target standard deviations (σ_p) were calculated using the appropriate form of the Horwitz equation [3] and in conjunction with the assigned values ($\hat{X}$) were used to derive z-scores for participants' results. z-Scores are considered satisfactory if $|z| \leq 2$.

5. Results for this test are summarised as follows:

analyte	assigned value $\hat{X}$, µg/kg	number of satisfactory scores $\|z\| \leq 2$	total number of scores	satisfactory %
fenitrothion	69.1	68	95	72
pirimiphos-methyl	304	91	106	86
tebuconazole	229	69	80	86
trifloxystrobin	277	62	75	83

6. Surplus test materials from this proficiency test 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 Food and Environment Research Agency nor the Secretary of State for Environment, Food and Rural Affairs accepts any liability whatsoever as to the application or use of the information contained therein.

CONTENTS

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 [4] 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 [5] 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.

Full details of the FAPAS® proficiency testing scheme is available via our protocols [6, 7].

2. TEST MATERIAL

2.1. Preparation

The test material was prepared by a laboratory contracted to do so by FAPAS®.

Approximately 13 kg of commercially available organic wheat flour was used in the preparation of this test material.

A portion of the wheat flour was screened for the presence of residues given in the FAPAS® list. No residues were found at or above 25 µg/kg:

A standard solution was prepared to give the following approximate final pesticide concentrations in the wheat flour:

fenitrothion	100 µg/kg
pirimiphos-methyl	400 µg/kg
tebuconazole	250 µg/kg
trifloxystrobin	300 µg/kg

The standard solution was added, drop-wise, to a 500 g portion of wheat flour, whilst mixing in a small mixer. This was left in a fume cupboard overnight, to vent any remaining solvent.

The spiked wheat flour was added to the remaining blank wheat flour. This was then mixed in a tumble mixer overnight. The prepared wheat flour was stored in a fridge for approximately 4 weeks before remixing (~ 19 hours).

Individual sub-samples (at least 50 g) were weighed into plastic bags. Each bag was individually numbered and stored at +4°C prior to distribution.

2.2. Homogeneity

Ten randomly selected test materials were analysed in duplicate for each analyte by a laboratory contracted to do so by FAPAS®. 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 results, together with their statistical evaluation [8], 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 8 October 2009. Each participant received an individually numbered wheat flour test material packed in a padded envelope together with a covering letter, instructions for electronic submission and the results form, for participants without access to the internet.

3. RESULTS

Participants were required to report their data in µg/kg for those analytes that they detected, uncorrected for recovery, together with the percentage recovery and limit of quantification (LoQ).

Results were submitted by 113 participants before the closing date for this test, 19 November 2009. Each participant was given a laboratory number, assigned in order of receipt of results. The results reported for fenitrothion, pirimiphos-methyl, tebuconazole and trifloxystrobin are given in Table 1.

If a participant failed to identify the presence of fenitrothion, pirimiphos-methyl, tebuconazole or trifloxystrobin and their LoQ was *below* the level needed for a satisfactory z-score then, as required by the FAPAS® Protocol, the reported result was assigned a value of zero.

If a participant failed to identify the presence of fenitrothion, pirimiphos-methyl, tebuconazole or trifloxystrobin and their LoQ was *above* the level needed for a satisfactory z-score, then the result was recorded as <LoQ.

Any participants identifying pesticides other than fenitrothion, pirimiphos-methyl, tebuconazole and trifloxystrobin at levels at or greater than 25 µg/kg are listed in Table 2 together with the pesticides reported and the levels determined.

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

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. The procedure follows that recommended in the IUPAC/ISO/AOAC International Harmonised Protocol for the Proficiency Testing of (Chemical) Analytical Laboratories [9].

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) participants reporting "not detected" and subsequently assigned a result of 0 µg/kg,
  - ii) results from participants not quoting a percentage recovery,
  - iii) results from participants whose recovery is outside the range 70 – 120% [10],
  - iv) results from participants not quoting an LoQ.
  - v) results reported as approximately 10, 100 or 1000 × greater or smaller than the majority of submitted results (as these were considered to be reporting errors).
- Minimising the influence of outliers by the use of a robust statistical procedure to derive the mean [2].
- Considering the normality (Kolmogorov-Smirnov test), or otherwise, of the distribution of the selected results.
- Assessing the uncertainty (u) of the robust mean:

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

where $\hat{\sigma}$ = the robust standard deviation [2]
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 all four pesticides this procedure was straightforward. The robust mean was considered to give the best measure of central tendency and was therefore used to set the assigned values.

The assigned values ($\hat{X}$) for each pesticide, together with *n, u* and $\hat{\sigma}$ are shown in Table 3.

4.2. Target Standard Deviation for the Round, 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 [3] are valuable indicators of best practice.

For all four pesticides, σ_p was derived from the appropriate form of the Horwitz equation [3]. 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 ≡ 10^{-6} or % ≡ 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 ≡ 10^{-6} or % ≡ 10^{-2}

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

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

In October 2008, the FAPAS® Advisory Committee noted that an EU wide PT 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 PT 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 [9] the Committee recommended that FAPAS® pesticide residue PTs continue to apply a concentration dependant σ_p value derived from the appropriate form of the Horwitz equation [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 each pesticde are given in Table 1 and shown as histograms in Figures 1-4.

The number and percentage of z-scores in the satisfactory range, $|z| \leq 2$, for each pesticide 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.

Table 5 shows the number and percentage of participants correctly identifying and obtaining satisfactory z-scores for all four pesticides. This information is not a measure of satisfactory performance in the test. Satisfactory performance is indicated in Table 1 and summarised in Table 4.

5. REFERENCES

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9 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(1)**, pp. 145-196.

10 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 in Wheat Flour Test Material

laboratory number		analyte															
		fenitrothion assigned value 69.1 µg/kg				pirimiphos-methyl assigned value 304 µg/kg				tebuconazole assigned value 229 µg/kg				trifloxystrobin assigned value 277 µg/kg			
		result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score
001		85	130	10	1.0	334	110	10	0.5	#				310	128	10	0.6
002		81	98	10	0.8	310	103	10	0.1	235	124	10	0.1	269	119	10	-0.1
003		80	100	20	0.7	310	100	20	0.1	#				#			
004		60	75	20	-0.6	270	71	20	-0.6	180	78	20	-1.1	180	81	20	-1.8
005		431.0	126.0	10	**23.8**	911.0	102.0	10	**10.4**	#				648.0	78.0	10	**6.9**
006		290	112	20	**14.5**	300	100	20	-0.1	290	113	20	1.3	330	97	60	1.0
007		67.16	91.25		-0.1	350.25	110.01	10	0.8	239.50	68.54	10	0.2	304.66	101.57	10	0.5
008		68	79	10	-0.1	291	87	10	-0.2	279	109	10	1.1	340	106	10	1.2
009		67	64	5	-0.1	300	59	2	-0.1	230	86	1	0.0	310	73	6	0.6
010		72		10	0.2	185		5	-2.0	116		10	**-2.5**	351		10	1.4
011		#				#				#				#			
012		60	95	10	-0.6	0			**-5.2**	180	99	10	-1.1	#			
013		60	174	10	-0.6	270	99	10	-0.6	160	27	10	-1.5	210	83	10	-1.2
014		72.9	91	10	0.2	345	96	10	0.7	231	89	10	0.0	269	94	10	-0.1
015	†	0		<10	**-4.5**	218.5		<10	-1.5	#				#			

z-scores outside the satisfactory range, i.e |z| >2, are shown in **bold** # = pesticide not analysed for LoQ = limit of quantification
† = additional pesticides identified >25 µg/kg

Table 1 (Continued): Results and z-Scores in Wheat Flour Test Material

laboratory number	analyte															
	fenitrothion assigned value 69.1 µg/kg				pirimiphos-methyl assigned value 304 µg/kg				tebuconazole assigned value 229 µg/kg				trifloxystrobin assigned value 277 µg/kg			
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016	54	86	10	-1.0	180	82	10	**-2.1**	155	80	10	-1.6	175	79	10	-1.9
017	#				280	93.7	10	-0.4	#				#			
018	#				#				#				#			
019	#				381	85	10	1.3	#				342	108	10	1.2
020	62	99.02	10	-0.5	276	96.56	10	-0.5	247	120.05	10	0.4	#			
021	51.9	98.5	10	-1.1	301.1	100.6	10	0.0	203.1	94.6	5	-0.6	274.5	99.9	10	0.0
022	70	95.2	10	0.1	340	89.0	10	0.6	270	88.8	10	0.9	310	94.7	10	0.6
023	70.9	107	20	0.1	302	110	20	0.0	208	96	20	-0.5	322	109	20	0.8
024	80	101	10	0.7	310	102	10	0.1	210	94	10	-0.4	300	97	10	0.4
025	73			0.3	342			0.7	200			-0.6	206			-1.3
026	#				#				#				#			
027	#				380	98	10	1.3	278	88	10	1.1	320	97	10	0.8
028	91	95	10	1.4	312	89	10	0.1	267	101	10	0.8	250	97	10	-0.5
029	0.008	70	0.005	**-4.5**	#				#				#			
030	62.0	98.8	20	-0.5	293.9	102.4	20	-0.2	#				#			

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Table 1 (Continued): Results and z-Scores in Wheat Flour Test Material

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		result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score
031		47*	81	50	-1.5	230	88	10	-1.3	#				#			
032		54	108	10	-1.0	274	98	50	-0.5	284	112	100	1.2	262	94	50	-0.3
033		109	106	0.01	**2.6**	380	103	0.01	1.3	268	103	0.01	0.8	327	104	0.01	0.9
034		66.3	97	5	-0.2	312.0	95	5	0.1	211.4	98	5	-0.4	261.3	97	5	-0.3
035		56.9	87	10	-0.8	238.6	81	10	-1.1	239	95.9	10	0.2	278	97.0	10	0.0
036		96.32	165.23	20	1.8	372.13	116.06	2	1.2	209.48	97.44	4	-0.4	264.60	102.37	1	-0.2
037		0		10	**-4.5**	240	100	10	-1.1	260	100	50	0.7	260	100	10	-0.3
038		61	95	10	-0.5	269	94	10	-0.6	234	120	10	0.1	226	102	10	-0.9
039		0	105.2	0.02	**-4.5**	355	86.0	0.02	0.9	290	104.7	0.01	1.3	281	108.1	0.01	0.1
040		0	75	9	**-4.5**	386.5	85	4	1.4	#				#			
041		110	104	10	**2.7**	310	105	10	0.1	178	91	10	-1.1	440	119	10	**3.0**
042		#				392	100	10	1.5	266	100	10	0.8	309	100	10	0.6
043	†	#				#				#				#			
044		56		5	-0.9	254		5	-0.9	200		10	-0.6	276		10	0.0
045		65		20	-0.3	336		20	0.6	227		40	-0.1	288		20	0.2

z-scores outside the satisfactory range, i.e |z| >2, are shown in **bold** # = pesticide not analysed for LoQ = limit of quantification
† = additional pesticides identified >25 µg/kg * = result is <LoQ

Table 1 (Continued): Results and z-Scores in Wheat Flour Test Material

laboratory number	analyte															
	fenitrothion assigned value 69.1 µg/kg				pirimiphos-methyl assigned value 304 µg/kg				tebuconazole assigned value 229 µg/kg				trifloxystrobin assigned value 277 µg/kg			
	result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score
046	0		20	**-4.5**	346	91	8	0.7	247	85	8	0.4	314	87	8	0.7
047	73	98	10	0.3	354	96	10	0.9	237	97	10	0.2	289	105	10	0.2
048	96.8		10	1.8	539.6		10	**4.1**	562.5		20	**7.3**	505.0		10	**4.3**
049	78.7	101	10	0.6	283.3	90	10	-0.4	235.9	69	10	0.1	234.2	80	10	-0.8
050	71.9	96	10	0.2	328.7	96	10	0.4	243.6	82	10	0.3	423.1	88	10	**2.7**
051	0			**-4.5**	118	95	5	**-3.2**	0			**-5.0**	90	92	5	**-3.5**
052	63.1	96	10	-0.4	301.4	92	10	0.0	199.6	93	20	-0.7	238.5	94	20	-0.7
053	134	98	10	**4.3**	324	107	10	0.3	257	94	10	0.6	334	105	10	1.1
054	61	102	10	-0.5	258	87	10	-0.8	193	78	10	-0.8	247	100	10	-0.6
055	48	81	10	-1.4	263	73	10	-0.7	#				#			
056	0		25.0	**-4.5**	284.0		2.5	-0.3	#				88.0		25.0	**-3.5**
057	50	96.3	10	-1.3	230	106.5	10	-1.3	210	79.5	10	-0.4	320	80.9	10	0.8
058	74	106	10	0.3	320	106	10	0.3	230	89	10	0.0	270	87	10	-0.1
059	67.4	82.1	20	-0.1	332.0	102.4	20	0.5	189.1	89.4	50	-0.9	226	71.9	50	-0.9
060	91.6	101	25	1.5	468	102	25	**2.8**	#				#			

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 in Wheat Flour Test Material

laboratory number		analyte															
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		result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score
061		102	108	12	**2.2**	217	102	12	-1.5	#				#			
062		48	96	5	-1.4	307	100	5	0.1	205	95	5	-0.5	364	129	2	1.6
063		104	139		**2.3**	343	110		0.7	279	135		1.1	308	140		0.6
064		50.7	81.8	10	-1.2	207.6	80.8	10	-1.7	195.7	74.5	10	-0.7	285.1	82.5	10	0.2
065		0			**-4.5**	396	82	10	1.6	395	117	10	**3.6**	425	63	10	**2.8**
066		88	80	10	1.2	291	105	10	-0.2	245	95	10	0.3	315	110	10	0.7
067	†	<LOQ	40	100		880	0	100	**9.9**	#				#			
068		#				355.7	90	3	0.9	#				#			
069		77	92.4	10	0.5	320	93.0	10	0.3	#				#			
070		#				267	86		-0.6	200	88		-0.6	#			
071		#				279	86	10	-0.4	190	88	10	-0.9	#			
072		72	103	5	0.2	318	97	5	0.2	231	99	10	0.0	272	104	10	-0.1
073		81.19	100.7	10	0.8	285.83	105.8	10	-0.3	237.14	106.6	10	0.2	177.84	80.1	10	-1.8
074	†	<LOQ	79	50		264	92	20	-0.7	154	82	10	-1.6	#			
075		50	93	10	-1.3	333	98	10	0.5	#				#			

z-scores outside the satisfactory range, i.e |z| >2, are shown in **bold** # = pesticide not analysed for LoQ = limit of quantification
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076		<LOQ		50		410	134	50	1.8	#				#			
077		45.0		10	-1.6	200.0		10	-1.8	150.0		10	-1.7	#			
078		65			-0.3	292			-0.2	0			**-5.0**	#			
079		108	114	10	**2.6**	349	95	10	0.8	277	115	10	1.0	339	100	10	1.2
080		#				321.3	88.7	50	0.3	#				#			
081		72	95	20	0.2	321	93	20	0.3	212	89	20	-0.4	278	94	20	0.0
082		83	104.0	10	0.9	320	102.4	10	0.3	#				#			
083		72			0.2	349			0.8	0			**-5.0**	123			**-2.9**
084		#				300	111	5	-0.1	240	105	5	0.2	#			
085		13.59			**-3.7**	0			**-5.2**	0			**-5.0**	0			**-5.2**
086		74	100	10	0.3	250	100	10	-0.9	260	95	10	0.7	310	80	10	0.6
087		38	109	20	-2.0	0	76	50	**-5.2**	186	85	10	-0.9	#			
088		490	105	10	**27.7**	390	105	10	1.5	220	95	10	-0.2	310	90	10	0.6
089		0	84	10	**-4.5**	235	109	10	-1.2	0	102	10	**-5.0**	0	110	10	**-5.2**
090	‡	104	82		**2.3**	269	73		-0.6	#				203	68		-1.4

z-scores outside the satisfactory range, i.e |z| >2, are shown in **bold** # = pesticide not analysed for LoQ = limit of quantification
particpant comment submitted with results: ‡ = tebuconazole identified but not quantified

Table 1 (Continued): Results and z-Scores in Wheat Flour Test Material

laboratory number		analyte															
		fenitrothion assigned value 69.1 µg/kg				pirimiphos-methyl assigned value 304 µg/kg				tebuconazole assigned value 229 µg/kg				trifloxystrobin assigned value 277 µg/kg			
		result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score
091		80	95	10	0.7	340	95	10	0.6	270	95	10	0.9	300	95	10	0.4
092		0	88	0.01	**-4.5**	0	85	0.01	**-5.2**	0	86	0.01	**-5.0**	0	86	0.01	**-5.2**
093	†	#				250	96.0	0.01	-0.9	#				#			
094		83		5	0.9	290		10	-0.2	160		10	-1.5	237		5	-0.7
095		79	114	10	0.6	305	93	10	0.0	315	111	10	1.9	350	101	10	1.4
096		57	94	0,01	-0.8	264	98	0,01	-0.7	232	119	0,01	0.1	291	107	0,01	0.3
097		68.97			0.0	266.35			-0.6	145.87			-1.8	#			
098		66	75	10	-0.2	347	108	10	0.7	253	97	20	0.5	252	85	10	-0.5
099		18	93	10	**-3.4**	183	95	10	**-2.1**	#				#			
100		41	70	<10	-1.8	172	83	<10	**-2.3**	174	72	<10	-1.2	172	71	<10	-1.9
101		79	95	10	0.6	348	100	10	0.8	240	84	10	0.2	290	90	10	0.2
102		66	120	10	-0.2	406	120	10	1.8	#				#			
103		72	98	5	0.2	214	98	5	-1.5	151	97	5	-1.7	183	98	5	-1.7
104		56	70-120	10	-0.9	285	120	10	-0.3	233	95	10	0.1	182	115	10	-1.8
105		0		10	**-4.5**	174	86	10	**-2.2**	#				#			

z-scores outside the satisfactory range, i.e |z| >2, are shown in **bold** # = pesticide not analysed for LoQ = limit of quantification
† = additional pesticides identified >25 µg/kg

Table 1 (Continued): Results and z-Scores in Wheat Flour Test Material

laboratory number		analyte															
		fenitrothion assigned value 69.1 µg/kg				pirimiphos-methyl assigned value 304 µg/kg				tebuconazole assigned value 229 µg/kg				trifloxystrobin assigned value 277 µg/kg			
		result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score	result µg/kg	recovery %	LoQ µg/kg	z-score
106		45	100	10	-1.6	#				159	100	10	-1.5	209	100	10	-1.3
107		0.131			**-4.5**	0.227			**-5.2**	0.233			**-5.0**	0.355			**-5.1**
108		95.4		3	1.7	284		3	-0.3	167		3	-1.4	172		3	-1.9
109		46.56	78	10.00	-1.5	226.34	75	10.00	-1.3	0	59	10.00	**-5.0**	173.36	74	10.00	-1.9
110	†	#				#				#				#			
111		0		10	**-4.5**	47		10	**-4.4**	180		50	-1.1	64		50	**-4.0**
112		0	124	10	**-4.5**	393	81	10	1.5	#				#			
113		54	73	10	-1.0	261	70	10	-0.7	#				#			

z-scores outside the satisfactory range, i.e |z| >2, are shown in **bold** # = pesticide not analysed for LoQ = limit of quantification
† = additional pesticides identified >25 µg/kg

Table 2: Additional Pesticides Identified in Wheat Flour Test Material

laboratory number	additional pesticides (>25 µg/kg)	result µg/kg	recovery %	LoQ µg/kg
015	chlorpyrifos-methyl	72.4		
043	alpha-endosulfan	80	81	4.4
110	alpha-endosulfan	80	111	40
110	beta-endosulfan	60	111	40
074	fenpropimorph	69	80	10
093	parathion	90	84.7	0.01
067	permethrin	27.8	109	10

Table 3: Assigned Values and Target Standard Deviations

analyte	assigned value, $\hat{X}$ µg/kg				target standard deviation, µg/kg	
	data points *n*	robust mean, $\hat{X}$	robust standard deviation, $\hat{\sigma}$	uncertainty, *u*	derived from	σ_p
fenitrothion	59	69.1	18.3	2.39	Horwitz*	15.2
pirimiphos-methyl	81	304	61.0	6.77	Horwitz*	58.1
tebuconazole	55	229	42.7	5.76	Horwitz*	45.8
trifloxystrobin	56	277	59.4	7.93	Horwitz*	53.7

*see page 7 for the 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 %
fenitrothion	68	95	72
pirimiphos-methyl	91	106	86
tebuconazole	69	80	86
trifloxystrobin	62	75	83

Table 5: Number and Percentage of Participants Correctly Identifying and Obtaining Satisfactory z-Scores for all Pesticides Present >25 µg/kg

criteria	number of satisfactory participants	total number of participants	satisfactory %
correctly identified all four pesticides present	56	113	50
correctly identified and obtained satisfactory z-scores for all four pesticides present	43	113	38

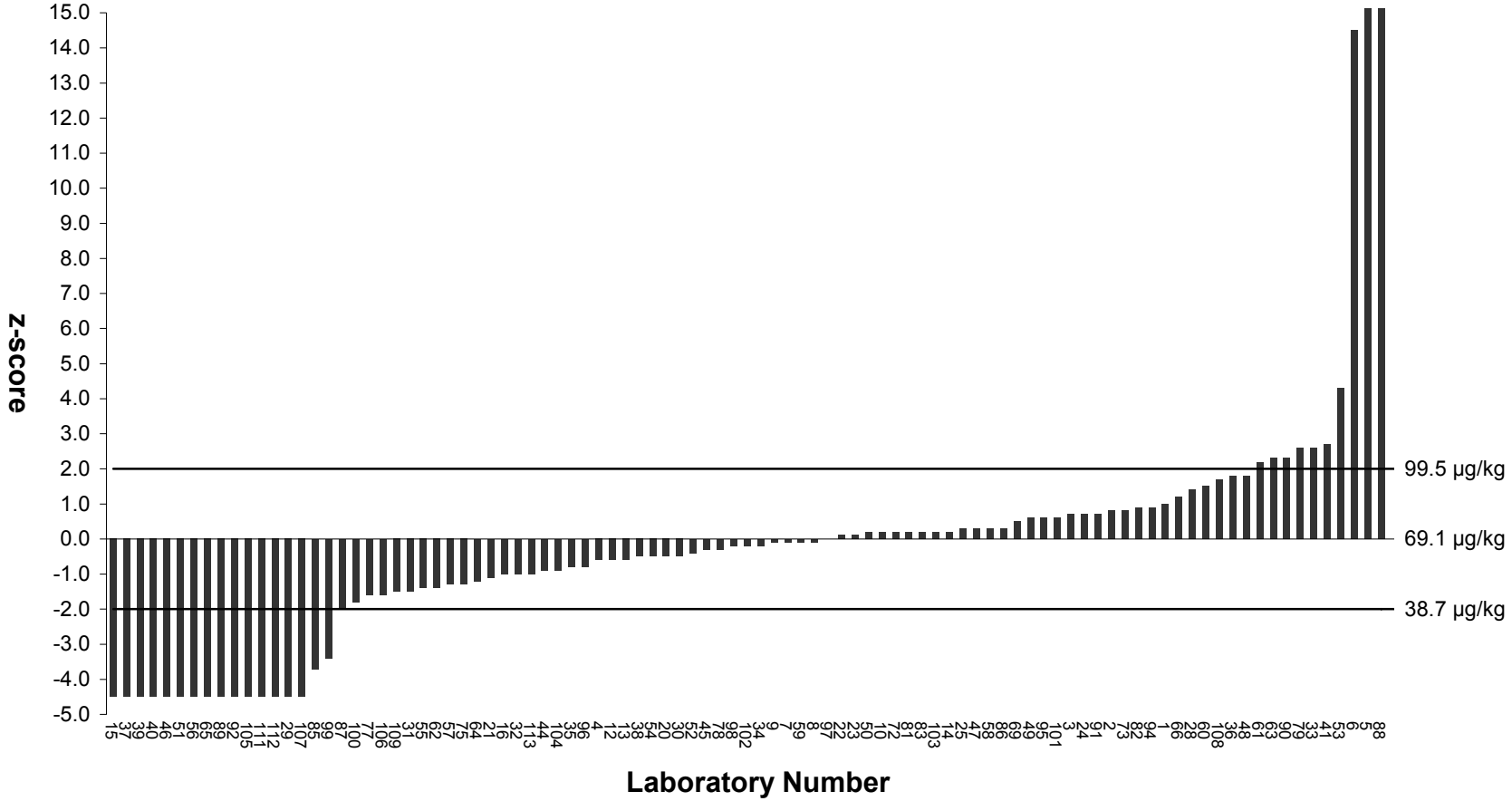

Figure 1: z-Scores for Fenitrothion (69.1 µg/kg) in Wheat Flour Test Material

participants assigned a result of 0 µg/kg for fenitrothion obtain a z-score of –4.5

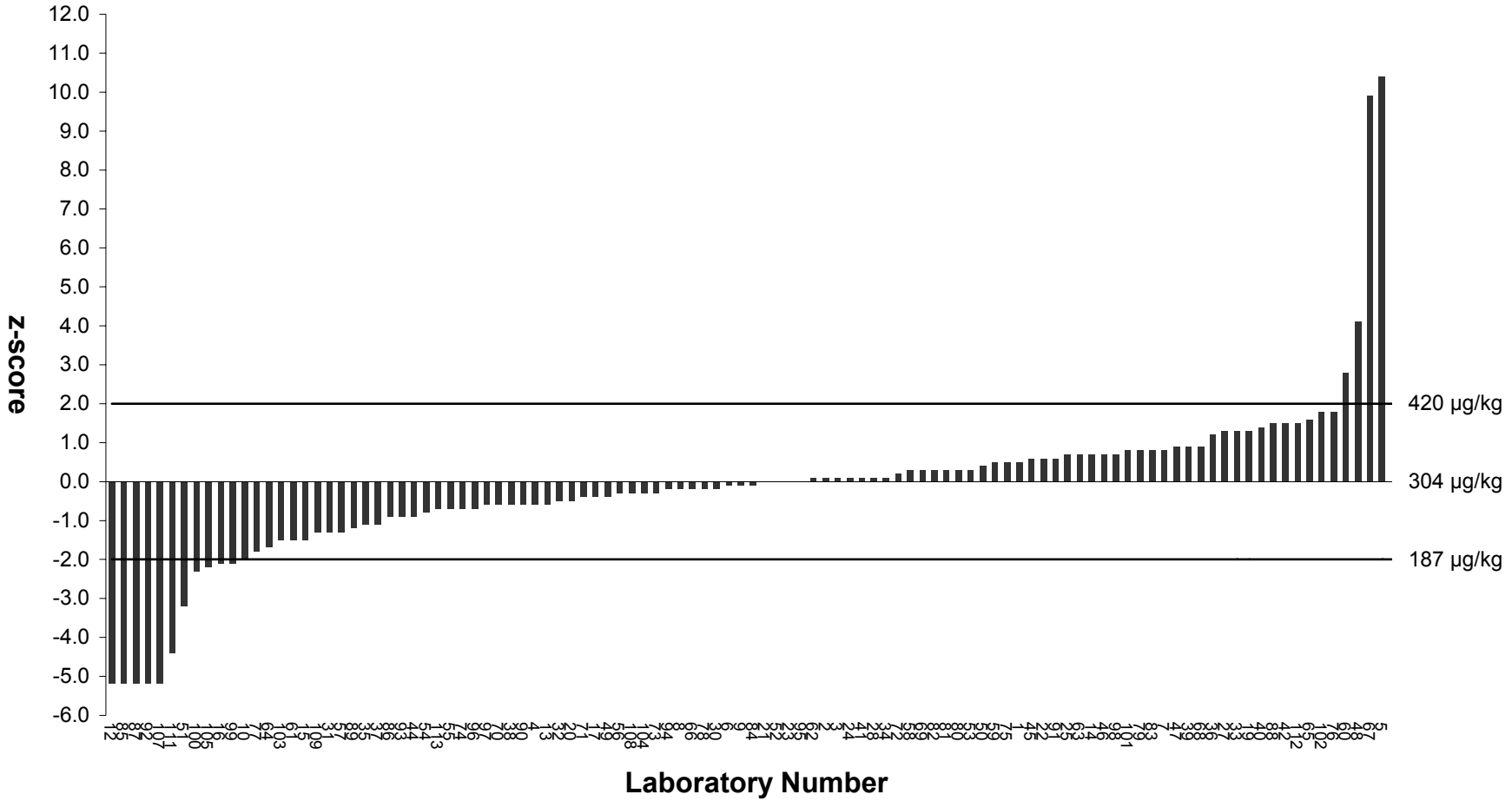

Figure 2: z-Scores for Pirimiphos-Methyl (304 µg/kg) in Wheat Flour Test Material

participants assigned a result of 0 µg/kg for pirimiphos-mehtyl obtain a z-score of –5.2

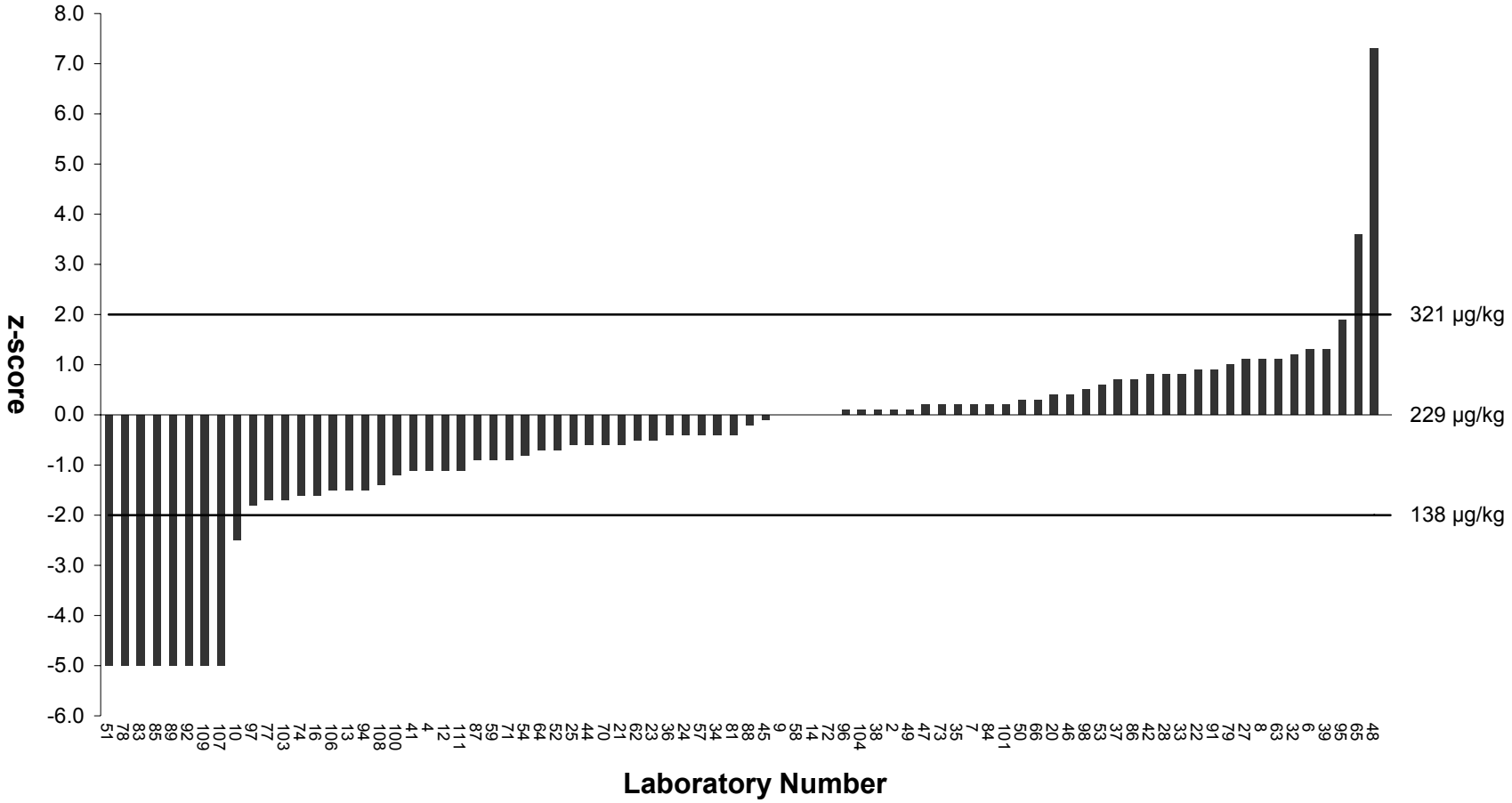

Figure 3: z-Scores for Tebuconazole (229 µg/kg) in Wheat Flour Test Material

participants assigned a result of 0 µg/kg for tebuconazole obtain a z-score of –5.0

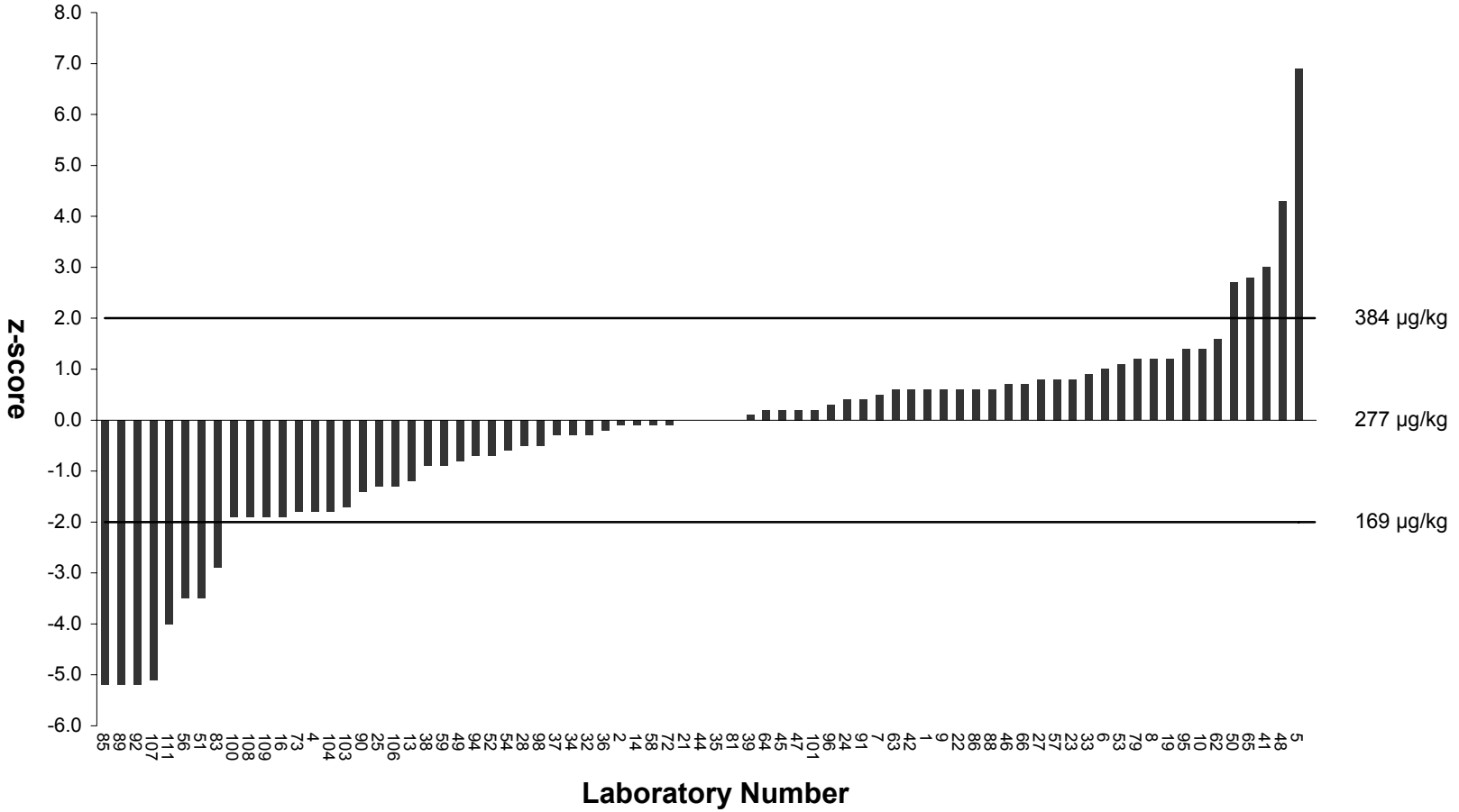

Figure 4: z-Scores for Trifloxystrobin (277 µg/kg) in Wheat Flour Test Material

participants assigned a result of 0 µg/kg for trifloxystrobin obtain a z-score of –5.2

APPENDIX I: Homogeneity Data for Wheat Flour Test Material

sample identity	analyte							
	fenitrothion µg/kg		pirimiphos-methyl µg/kg		tebuconazole µg/kg		trifloxystrobin µg/kg	
	replicate 1	replicate 2	replicate 1	replicate 2	replicate 1	replicate 2	replicate 1	replicate 2
1	78	83	283	307	184	197	235	244
2	82	85	307	309	189	192	237	240
3	80	78	293	289	184	189	236	236
4	80	82	297	305	187	193	239	241
5	80	80	295	300	186	187	245	241
6	76	83	282	299	193	182	241	226
7	83	71	304	259	196	172	251	216
8	74	70	274	260	179	180	230	233
9	73	75	274	276	181	181	224	234
10	77	80	288	295	181	187	233	236
mean, n	79	20	290	20	186	20	236	20
origin of target sd (σ_p)	Horwitz*		Horwitz*		Horwitz*		Horwitz*	
abs. target sd (σ_p) & as RSD%	17.3	22.0	55.9	19.3	38.3	20.6	46.9	19.9
s_{an}	3.63		12.7		6.98		9.16	
s_{sam}^2	4.90		69.4		0		0	
σ_{all}^2	26.8		281		132		198	
critical	63.8		692		298		457	
s_{sam}^2<*critical?*	**ACCEPT**		**ACCEPT**		**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 submitted electronically, but some responses may have been combined or edited for clarity.
2. Only methods relating to fenitrothion, pirimiphos-methyl, tebuconazole and trifloxystrobin are tabulated.
3. Participants with performance outside the satisfactory range, i.e. |z| >2, are no longer shown in **bold** in this section but their z-scores are now shown in **bold** in Table 1.

Fenitrothion

Accredited Method Used	laboratory number
yes	002 004 005 007 008 010 012 013 014 016 021 022 023 024 025 029 032 033 034 035 036 038 044 047 048 050 058 059 061 062 064 066 069 072 073 077 081 082 086 088 090 091 094 096 097 098 100 103 104 106 107 108
no	003 006 009 020 028 031 045 049 052 053 055 057 060 063 075 083 095 099 101 102 109

Sample Weight (g)	laboratory number
≥2 - <5	007 031 049 057 061 062 073 091 096 107
≥5 - <10	013 014 021 023 028 033 035 036 045 048 050 052 053 055 058 064 075 077 083 086 088 090 095 097 100 101 102 104 109
≥10 - <25	002 004 009 012 016 020 022 024 025 029 032 034 038 047 059 060 063 066 069 072 082 099 103 106
≥25 - <50	003 005 008 081 094 098 108
≥50	006 044 085

Water Added Prior to Extraction (SANCO/2007/3131)	laboratory number
yes	002 004 008 014 020 021 022 024 025 028 032 033 034 035 036 038 047 049 053 055 057 058 061 062 063 064 066 073 077 081 082 086 088 090 091 098 099 100 102 104 107
no	005 006 007 009 023 029 044 048 050 052 059 075 083 094 095 097 106 108 109

Extraction Solvent	laboratory number
acetic acid	013 052
acetone	004 005 006 010 016 020 023 025 029 032 035 038 047 050 055 058 059 069 072 075 081 088 094 095 098 099 103 104 107 108 109
acetonitrile	002 003 007 009 012 013 014 021 022 024 028 031 033 034 036 045 049 052 053 057 061 062 063 064 066 073 077 082 083 085 086 090 091 094 102
cyclohexane	025 038 055 060 097 099 103
dichloromethane	004 006 023 059 095 109
ethyl acetate	008 025 035 044 048 055 060 096 097 099 100 103 104
hexane	004 029 035 050 059 101
methanol	005 081 106
petroleum ether/spirit	059 072 095
water	004 010 038 059 095 103

Extraction Technique Used	laboratory number
cold solvent extraction at atmospheric pressure	002 003 004 005 007 008 009 010 014 016 021 024 025 028 029 032 033 035 036 038 044 045 047 049 050 053 057 058 060 062 064 066 069 072 073 075 077 081 082 086 088 090 091 096 097 098 099 100 101 102 104 106 107 108
ASE	023 048
bath sonication	109
liquid/liquid partition-Kuderna Danish concentration	059
shaker	006 022
solvent extraction	020
Vortex	052

Extraction pH Adjusted	laboratory number
yes	007 012 014 021 049 082 086 097 098
no	002 003 004 005 006 008 009 010 013 016 020 022 023 024 025 028 029 031 032 033 034 035 036 038 044 045 047 048 050 052 053 055 057 058 059 060 061 062 063 064 066 069 072 073 075 077 081 083 088 090 091 094 095 096 099 100 101 102 103 104 106 107 108 109

Sample Clean-up Technique	laboratory number
carbon based column	021 047
C18 SPE column/cartridge	022
extraction	025 028 049 062 091 108
Extrelut	066
filter	005 010
florisil column	003 016 059 061 069 077 101
GPC	060
GPC/HPGPC	004 005 006 023 024 025 032 038 048 055 066 073 075 081 094 096 098 107 108
liquid/liquid extraction / solvent exchange	002 005 013 021 029 034 058 059 061 062 082 088 095 108
NH_2/aminopropyl column	002 012 021 047
silica column	020 094 099
solid phase extraction (SPE) (column/cartridge)	009 021 022 023 031 036 050 057 059 062 063 064 082 106
solid phase extraction (SPE) (dispersive)	007 014 033 045 053 086 090 097 102 103
none	008 044 072 083 100

SPE Column Type	laboratory number
BONDESil - NH_2	095
C18	002 007 009 033 062 063 106
C18/Amino	023
Envi-Carb/GCB	012 036 047 059 082
Envi-Carb/NH_2	022 034
graphite-carbon/NH2	058 062 088
PSA	014 021 031 052 053 057 062 064 090
silica	050 060 094

Certified Standards Used	laboratory number
yes	002 004 005 006 007 008 012 013 014 016 021 022 023 024 025 028 029 031 032 033 034 035 036 038 044 045 047 048 049 050 052 053 055 059 060 061 062 064 066 069 072 073 075 077 081 082 083 086 091 094 095 096 097 098 099 100 101 102 103 104 106 107 109
no	003 009 010 020 057 058 063 088 090 108

MS Confirmation	laboratory number
yes	002 004 005 006 007 008 009 010 012 014 016 020 021 022 023 024 028 031 032 033 034 035 036 045 047 048 049 050 052 053 057 062 064 066 069 072 073 075 077 081 082 083 086 090 091 094 096 097 098 100 102 103 104 106 107 109
no	003 013 025 029 038 044 055 058 059 060 061 063 088 095 099 101

Calibrations	laboratory number
matrix-matched	004 008 013 014 016 022 023 024 025 032 033 034 035 047 048 050 053 057 059 072 075 081 090 091 094 096 097 098 103
multi-level	002 004 007 012 013 014 016 020 021 023 028 029 033 034 035 044 045 047 049 050 055 058 059 060 062 069 073 077 088 090 094 096 097 098 100 102 104 108 109
single-level	003 006 008 009 010 025 031 036 038 052 061 066 075 086 095 099 101 107
solvent	005 009 021 028 038 045 058 062 063 082 088 107 108
standard addition	083 096 097 106

Source of Standards	laboratory number
Accu Standard	077
Chem Service	006 059 109
Dr Ehrenstorfer	004 007 008 010 012 016 021 023 024 025 028 032 033 035 036 044 045 047 048 049 050 052 053 055 060 063 064 066 072 073 075 081 086 090 094 096 097 098 099 100 101 102 103 104 106 107 108
Fluka	014 053 063 090
Kanto	062
Restek	003
Riedel-de Haen	020
Sigma/Aldrich	005 009 014 028 031 034 053 061 063 077 083 098
Sumitomo	038
Supelco	053
Wako	002 022 057 058 063 069 082 088

Quoted Percentage Recovery Measured in Same Analytical Batch as Test Material	laboratory number
yes	002 003 004 005 006 008 009 012 013 014 016 020 021 022 023 024 028 032 033 035 036 038 044 047 048 049 050 052 055 057 058 059 060 061 062 064 066 069 072 075 081 082 088 090 091 094 095 096 097 098 099 100 101 102 104 109
no	007 010 025 031 034 045 053 063 073 077 086 103 106 107 108

Stage the Spike was Added	laboratory number
prior to extraction	002 004 005 006 008 009 012 013 014 016 020 022 023 024 028 031 032 033 034 035 036 038 044 047 048 049 050 052 055 057 058 059 060 061 062 066 069 072 073 075 081 082 088 090 097 098 099 100 101 102 104 107 109
prior to clean up	003 064 094 095
prior to instrument measurement	021 096

Level of Spike (µg/kg)	laboratory number
<25	003 004 006 013 014 020 023 033 048 050 053 057 069 082 099 106 109
≥25 - <50	032 047 050 059 090 096 097 107
≥50 - <100	004 008 009 016 022 024 028 034 035 038 049 052 055 060 061 064 066 072 075 081 096 100 101 102 104
≥100 - <150	033 036 044 058 062 066 073 088 094 095 096 104 106
≥150 - <200	023 091 096 104
≥200 - <250	002 007 012 014 031 063 096 104 106
≥250 - <300	021 032
≥300 - <400	096
≥400 - <500	032 096
≥500	005 098

Composition of Blank Commodity used for Spiking	laboratory number
blank provided	002 004 007 009 012 014 016 021 022 028 032 034 038 044 047 048 050 057 061 062 063 064 066 069 073 082 094 095 101 102 104 107
test material provided	024 036 049 052 055 072 096 097 099 109
blank ground wheat	006
flour	003
in house blank	013 035 075 081 090 100
in house cereal sample	005
previously analysed sample	106
rice	020
wheat	008
wheat flour	023 053 059 098

GC Column Type	laboratory number
capillary	002 003 004 006 007 008 010 012 013 014 016 020 021 022 023 025 029 031 032 033 034 035 036 044 045 047 048 049 050 052 053 055 057 058 060 061 062 063 064 069 073 075 077 081 082 083 085 086 088 094 095 096 097 098 099 101 102 103 104 106 107 108 109
megabore	059 072 090
narrowbore <0.53 mm id	005 009 038 066 100
widebore	024

GC Column Packing	laboratory number
100% methyl polysiloxane	059 061 066 072 099 106
14% cyanopropylphenyl 86%methyl polysiloxane	010 025 038 060 075 090 101
50% cyanopropylphenyl 50% methyl	059
50% methyl 50% phenyl polysiloxane	003 005 032 059 062 072 081
50% trifluoropropyl - 50%methyl	020
65% methyl 35% phenyl polysiloxane	029 048
8% Phenyl-polysiloxane-carborane	007
95% methyl 5% phenyl polysiloxane	002 003 004 006 008 009 012 013 014 016 021 022 023 024 031 032 033 034 035 036 044 045 047 049 050 052 053 055 057 062 063 064 066 073 077 083 086 094 096 097 098 100 102 103 104 107 108 109
trifluoropropyl methylpolysiloxane	058 069 082 088

GC Injection Volume (µL)	laboratory number
<1	057
≥1 - <2	003 004 006 013 021 023 025 029 032 035 044 047 048 050 052 059 060 072 075 077 082 090 094 095 097 098 099 100 101 102 103 104 106 107 108 109
≥2 - <5	005 008 009 012 014 020 022 024 033 034 036 038 055 058 061 062 063 066 069 073 081 086 088
≥5 - <10	002 016 045 053 096
≥10	007 010 031 049 064 083

GC Injection Mode	laboratory number
on-column	010 059 077
PTV	004 014 031 033 045 053 086 096
split	005 007 029 032 049 073 083 107
splitless	002 003 008 009 012 013 016 020 021 022 023 024 025 034 035 036 038 044 047 048 050 052 055 057 058 060 061 062 063 064 066 069 072 075 081 082 088 090 094 095 097 098 099 100 101 102 103 104 106 108 109
pulsed splitless	006

GC Detector	laboratory number
ECD	003 004 007 029 032 059 060 085 094 101 107 109
ELCD-X	059
FPD	004 020 025 038 044 058 059 060 061 062 066 069 075 082 086 088 090 095 096 099
ITD	045 081
MS	002 004 005 010 013 021 033 034 036 049 050 062 063 064 077 096
MSD	006 012 014 022 024 032 047 048 066 073 086 094 098 100 104 106 107 108 109
MS-MS	008 009 016 023 031 035 052 053 057 072 083 102 103
MS-X-MS	097
NPD	032 055 072 094 107 109

HPLC Column Packing	laboratory number
C18	016 021 028 047 053 063 102
C8	049

HPLC Guard Column Used	laboratory number
yes	016 021 047 053 063 102
no	028 029 049 064 077 082 095 103 106 109

Mobile Phase Programme	laboratory number
gradient	016 021 028 047 049 053 063 077 102 103

Mobile Phase Components	laboratory number
acetic acid	021
acetonitrile	028
formic acid	016 028 102
methanol	021 047 049 053 063
water	021 053

HPLC Column Temperature (°C)	laboratory number
ambient	021
>ambient - <50	016 028 047 049 053 063 102

HPLC Injection Volume (µL)	laboratory number
≥5 - <10	028 049 063 102
≥10 - <25	016 021 047 053

Mobile Phase Flow Rate (mL/min)	laboratory number
<0.25	016 021
≥0.25 - <0.75	028 047 049 053 063 102

HPLC Pre Column Derivatisation	laboratory number
none	047 049 063

HPLC Post Column Derivatisation	laboratory number
none	047 049 063

HPLC Detector Type	laboratory number
MS-MS	016 021 028 047 049 053 063 102

Pirimiphos-methyl

Accredited Method Used	laboratory number
yes	002 004 005 007 008 010 013 014 016 017 021 022 023 024 025 027 030 032 033 034 035 036 037 038 039 040 046 047 048 050 051 058 059 061 062 064 065 066 068 069 070 071 072 073 074 077 081 082 086 088 089 090 091 094 096 097 098 100 101 103 104 107 108
no	003 006 009 019 020 028 031 042 044 045 049 052 053 055 056 057 060 063 067 075 076 080 083 084 093 095 099 102 105 109

Sample Weight (g)	laboratory number
≥2 - <5	007 019 031 040 046 049 057 061 062 068 073 091 093 096 107
≥5 - <10	014 021 023 028 030 033 035 036 037 039 045 048 050 051 052 053 055 056 058 064 065 075 077 080 083 084 086 088 089 090 095 097 100 101 102 104 109
≥10 - <25	002 004 009 013 016 017 020 022 024 025 027 032 034 038 042 047 059 060 063 066 067 069 070 071 072 074 076 082 099 103
≥25 - <50	003 005 008 081 098 105 108
≥50	006 044 094

Water Added Prior to Extraction (SANCO/2007/3131)	laboratory number
yes	002 004 008 014 017 019 020 021 022 024 025 027 028 032 033 034 035 036 037 038 039 046 047 049 051 053 055 056 057 058 061 062 063 064 065 066 070 071 073 074 076 077 080 081 082 084 086 088 089 090 091 098 099 100 102 104 107
no	005 006 007 009 023 030 040 044 048 050 052 059 068 075 083 093 094 095 097 105 108 109

Extraction Solvent	laboratory number
acetic acid	013 052
acetone	004 005 006 010 016 017 020 023 025 032 035 038 047 050 051 055 058 059 065 068 069 072 075 076 081 088 093 094 095 098 099 103 104 107 108 109
acetonitrile	002 003 007 009 013 014 019 021 022 024 027 028 031 033 034 036 039 042 045 046 049 051 052 053 056 057 061 062 063 064 065 066 070 071 073 074 077 080 082 083 084 086 089 090 091 094 101 102
cyclohexane	025 038 055 060 094 097 099 103
dichloromethane	004 006 023 051 059 067 068 076 095 109
ethyl acetate	008 025 035 044 048 055 060 094 096 097 099 100 103 104 105
hexane	004 035 050 059 093
methanol	005 030 037 074 081
petroleum ether/spirit	040 051 059 072 076 095
water	003 004 010 038 042 051 059 095 103

Extraction Technique Used	laboratory number
ASE	023 048
bath sonication	109
cold solvent extraction at atmospheric pressure	002 003 004 005 007 008 009 010 014 016 021 024 025 028 030 032 033 035 036 038 040 044 045 046 047 049 050 051 053 056 057 058 060 062 064 066 067 068 069 070 071 072 073 074 075 076 077 080 081 082 086 088 089 090 091 096 097 098 099 100 101 102 104 105 107 108
hot solvent extraction at atmospheric pressure (e.g. soxhlet)	093
liquid/liquid partition	059
QuEChERS	084
shaker	006 022 042
solvent extraction	020
SPE	037
Vortex	052

Extraction pH Adjusted	laboratory number
yes	007 014 019 021 037 039 049 082 084 086 089 094 097 098
no	002 003 004 005 006 008 009 010 013 016 017 020 022 023 024 025 027 028 030 031 032 033 034 035 036 038 040 042 044 045 046 047 048 050 051 052 053 055 056 057 058 059 060 061 062 063 064 065 066 067 068 069 070 071 072 073 074 075 076 077 080 081 083 088 090 091 093 095 096 099 100 101 102 103 104 105 107 108 109

Sample Clean-up Technique	laboratory number
C18 SPE column/cartridge	022
carbon based column	021 047
dilute	042
extraction	025 028 049 062 091 108
Extrelut	066
filter	005 010 030 042
florisil column	003 016 051 059 061 069 077
GPC	060
GPC/HPGPC	004 005 006 023 024 025 032 038 048 055 066 068 073 075 081 089 094 096 098 107 108
liquid/liquid extraction / solvent exchange	002 003 005 013 017 021 034 058 059 061 062 070 071 082 088 093 095 108
NH_2/aminopropyl column	002 021 047 065
QuEChERS	080
silica column	017 020 037 099
solid phase extraction (SPE) (column/cartridge)	009 019 021 022 023 031 036 050 057 059 062 063 064 067 070 071 082
solid phase extraction (SPE) (dispersive)	007 014 033 039 045 053 056 084 086 090 097 101 102 103
none	008 044 046 072 074 076 083 100 105

SPE Column Type	laboratory number
alumina	093
BONDESil - NH_2	095
C18	002 007 009 033 039 062 063 080
C18/Amino	023
Envi-Carb /GCB	036 047 059 082
Envi-Carb /NH_2	022 034 070 071
florisil	067
graphite-carbon/NH_2	058 062 088
PSA	014 021 031 039 052 053 056 057 062 064 080 084 090
silica	050 060 094
strata X 200 mg	037

Certified Standards Used	laboratory number
yes	002 004 005 006 007 008 013 014 016 017 019 021 022 023 024 025 027 028 030 031 032 033 034 035 036 037 038 039 040 042 044 045 046 047 048 049 050 051 052 053 055 056 059 060 061 062 064 066 067 068 069 070 071 072 073 074 075 076 077 080 081 082 083 084 086 089 091 093 094 095 096 097 098 099 100 101 102 103 104 105 107 109
no	003 009 010 020 057 058 063 065 088 090 108

MS Confirmation	laboratory number
yes	002 004 005 006 007 008 009 010 014 016 017 019 020 021 022 023 024 027 028 031 032 033 034 035 036 037 039 042 045 046 047 048 049 050 051 052 053 056 057 062 064 066 067 069 070 071 072 073 074 075 077 080 081 082 083 084 086 089 090 091 094 096 097 098 100 102 103 104 105 107 109
no	003 013 025 030 038 040 044 055 058 059 060 061 063 065 068 076 088 095 099 101

Calibrations	laboratory number
matrix-matched	004 008 013 014 016 022 023 024 025 030 032 033 034 035 037 042 046 047 048 050 053 057 059 065 070 071 072 074 075 077 080 081 090 091 096 097 098 101 103
multi-level	002 004 007 014 016 017 020 021 023 027 028 030 033 034 035 040 042 044 045 047 049 050 051 055 056 058 059 060 062 068 069 070 071 073 077 080 084 088 090 093 094 096 097 098 100 102 104 105 108 109
single-level	003 006 008 009 010 025 031 036 038 052 061 066 067 075 076 086 094 095 099 107
solvent	005 009 017 019 021 028 038 042 045 058 062 063 074 082 088 107 108
standard addition	039 042 083 089 096 097

Source of Standards	laboratory number
Accu Standard	040 051 077 084
Chem Service	006 059 109
Dr Ehrenstorfer	004 007 008 010 016 021 023 024 025 027 028 032 033 035 036 037 038 040 042 045 047 048 049 050 052 053 055 058 060 063 064 066 068 070 071 072 073 075 076 081 086 088 089 090 094 096 097 098 099 100 101 102 103 104 107 108
Fluka	014 053 063 080 090 093
GB	067
Greyhound	030
Kanto	062 065
LGC	042 074
QMx	074
Restek	003
Riedel-de Haën	022
Sigma/Aldrich	005 009 014 017 028 030 031 034 039 044 046 053 056 061 063 077 083 094 098 105
Supelco	053
Wako	002 019 020 057 063 069 082

Quoted Percentage Recovery Measured in Same Analytical Batch as Test Material	laboratory number
yes	002 003 004 005 006 008 009 013 014 016 017 019 020 021 022 023 024 027 028 030 032 033 035 036 037 038 039 042 044 046 047 048 049 050 051 052 055 056 057 058 059 060 061 062 064 065 066 067 068 069 070 071 072 074 075 080 081 082 084 088 089 090 091 093 095 096 097 098 099 100 101 102 104 105 109
no	007 010 025 031 034 040 045 053 063 073 076 077 086 094 103 107 108

Stage the Spike was Added	laboratory number
prior to extraction	002 003 004 005 006 008 009 013 014 016 017 019 020 022 023 024 027 028 030 031 032 033 034 035 036 037 038 039 044 046 047 048 049 050 051 052 055 056 057 058 059 060 061 062 065 066 067 068 069 070 071 072 073 074 075 080 081 082 084 088 089 090 093 094 095 097 098 099 100 101 102 104 107 109
prior to clean up	064
prior to instrument measurement	021 042 096

Level of Spike (µg/kg)	laboratory number
<25	003 004 006 013 014 017 020 023 027 033 046 048 050 053 057 069 070 071 082 084 099 109
≥25 - <50	032 039 047 050 059 076 090 096 097 107
≥50 - <100	009 016 019 022 034 035 049 060 061 064 065 068 075 081 096 100 101 102 104
≥100 - <150	004 033 036 044 056 058 062 067 073 074 088 095 096 104
≥150 - <200	023 055 096 104
≥200 - <250	002 007 008 014 031 037 063 066 089 096 104
≥250 - <300	021 032 038 080
≥300 - <400	004 024 028 042 052 066 096
≥400 - <500	032 072 096
≥500	005 042 091 094 098

Composition of Blank Commodity used for Spiking	laboratory number
blank provided	002 004 007 009 014 016 019 021 022 028 032 034 038 044 046 047 048 050 051 057 061 062 063 064 065 066 067 068 069 070 071 073 076 082 094 095 101 102 104 107
test material provided	024 036 039 042 049 052 055 072 089 096 097 099 109
blank ground wheat	006
flour	037
in house blank	013 027 035 075 081 090 100
in house cereal sample	005
previously analysed sample	074
rice	017 020
wheat	008 053
wheat flour	003 031 033 056 059 080 084 093 098
wholemeal flour	030

GC Column Type	laboratory number
capillary	002 003 004 006 008 010 013 014 016 019 020 021 022 023 025 027 028 031 032 033 034 035 036 037 039 040 044 045 047 048 049 050 051 052 053 055 056 057 058 060 061 062 063 064 065 067 068 069 070 071 073 074 075 076 077 081 082 083 086 088 089 093 094 095 096 097 098 099 101 102 103 104 105 107 108 109
megabore	017 059 072 090
narrowbore <0.53 mm id	005 009 038 066 100
widebore	024 030

GC Column Packing	laboratory number
100% methyl polysiloxane	059 061 066 072 094 099
14% cyanopropylphenyl 86%methyl polysiloxane	003 010 025 027 038 060 075 090
5% methyl 95% dimethylpolysiloxane	074
50% cyanopropylphenyl 50% methyl	059
50% methyl 50% phenyl polysiloxane	005 028 032 059 062 072 081
65% methyl 35% phenyl polysiloxane	048 051 076

GC Column Packing (continued)	laboratory number
95% methyl 5% phenyl polysiloxane	002 003 004 006 008 009 014 016 019 021 022 023 024 027 030 031 032 033 034 035 036 037 039 040 044 045 047 049 050 052 053 055 056 057 062 063 064 065 066 067 068 070 071 073 077 083 086 089 093 096 097 098 100 102 103 104 105 107 108 109
trifluoropropyl methylpolysiloxane	058 069 082 088
50% trifluoropropyl - 50%methyl	017 020

GC Injection Volume (µL)	laboratory number
<1	057
≥1 - <2	003 004 006 013 019 021 023 025 027 030 032 035 037 039 040 044 047 048 050 051 052 059 060 062 065 067 068 070 071 072 075 077 082 089 090 093 094 095 097 098 099 100 102 103 104 105 107 108 109
≥2 - <5	005 008 009 014 017 020 022 024 028 033 034 036 038 055 056 058 061 063 066 069 073 076 081 086 088
≥5 - <10	002 016 045 053 074 096
≥10	010 031 049 064 083

GC Injection Mode	laboratory number
on-column	010 059 077
PTV	004 014 028 031 033 045 053 056 074 086 096
split	005 032 049 051 073 083 093 107
splitless	002 003 008 009 013 016 017 019 020 021 022 023 024 025 027 030 034 035 036 038 039 040 044 047 048 050 052 055 057 058 060 061 062 063 064 065 066 067 068 069 070 071 072 075 076 081 082 088 089 090 094 095 097 098 099 100 102 103 104 105 108 109
pulsed splitless	006

GC Detector	laboratory number
ECD	004 027 032 039 059 060 094 107 109
ELCD-X	059
FPD	003 004 017 020 025 027 030 038 044 058 059 060 061 062 066 069 075 082 086 088 090 095 096 099
ITD	045
MSD	002 004 005 006 010 013 014 019 021 022 024 028 032 033 034 036 037 039 047 048 049 050 051 056 062 063 064 066 067 073 077 081 086 089 093 094 096 098 100 104 105 107 108 109
MS-MS	008 009 016 023 031 035 052 053 057 065 070 071 072 074 083 102 103
MS-X-MS	097
NPD	032 037 039 040 055 068 072 076 094 107 109
XSD	095

HPLC Column Packing	laboratory number
C18	007 016 021 027 039 046 047 053 063 077 084 089 102
C18 polar embedded	080
C18 X-Terra type	042
C8	049
endcapped	084

HPLC Guard Column Used?	laboratory number
yes	007 016 021 039 047 053 063 089 101 102
no	027 042 046 049 064 065 068 077 080 082 084 095 103 109

Mobile Phase Programme	laboratory number
gradient	007 016 021 027 039 042 046 047 049 053 063 077 080 084 089 101 102 103

Mobile Phase Components	laboratory number
acetate	042
acetic acid	021
acetonitrile	007 039 077 101
ammonium acetate	084
ammonium formate buffer	080
formic acid	016 102
methanol	007 021 027 042 046 047 049 053 063 080 089
water	021 027 042 046 053 077 089 101

HPLC Column Temperature (°C)	laboratory number
ambient	021 027 101
>ambient - <50	007 016 039 042 046 047 049 053 063 077 080 084 089 102

HPLC Injection Volume (µL)	laboratory number
<5	007
≥5 - <10	046 049 063 102
≥10 - <25	016 021 027 039 047 053 077 080 101
≥25 - <50	084 089
≥100 - <150	042

Mobile Phase Flow Rate (mL/min)	laboratory number
<0.25	016 021 089
≥0.25 - <0.75	007 027 042 046 047 049 053 063 077 080 084 102
≥0.75 - <1.25	039

HPLC Pre Column Derivatisation	laboratory number
none	027 046 047 049 063 080 084 089

HPLC Post Column Derivatisation	laboratory number
none	027 046 047 049 063 080 084 089

HPLC Detector Type	laboratory number
MS-MS	007 016 021 027 039 042 046 047 049 053 063 077 080 084 089 101 102

Tebuconazole

Accredited Method Used	laboratory number
yes	002 007 008 012 013 014 016 021 022 023 024 025 027 032 033 034 035 036 037 038 039 046 047 048 050 058 059 062 064 065 066 070 071 072 073 074 077 081 086 088 091 094 096 097 098 100 101 103 104 106 107 108
no	006 009 010 020 028 042 044 045 049 052 053 057 063 084 095

Sample Weight (g)	laboratory number
≥2 - <5	007 046 049 057 062 073 091 096 107
≥5 - <10	004 014 021 023 028 033 036 037 039 045 048 050 052 053 058 064 065 077 084 086 088 095 097 100 101 104
≥10 - <25	002 008 009 012 013 016 020 022 024 025 027 032 034 035 038 042 044 047 059 063 070 071 072 074 103 106
≥25 - <50	066 081 094 098 108
≥50	006

Water Added Prior to Extraction (SANCO/2007/3131)	laboratory number
yes	002 004 008 014 020 021 022 024 025 027 028 032 033 034 035 036 037 038 039 046 047 049 053 057 058 062 063 064 065 066 070 071 073 074 077 081 084 086 088 091 098 100 104 107
no	006 007 009 023 044 048 050 052 059 094 095 097 108

Extraction Solvent	laboratory number
acetic acid	008 052 106
acetone	006 010 013 016 020 023 025 032 035 038 047 050 058 059 065 072 074 081 088 094 095 098 103 104 107 108
acetonitrile	002 004 007 008 009 012 014 021 022 024 027 028 033 034 036 039 042 045 046 049 052 053 057 062 063 064 065 066 070 071 073 077 084 086 091 094 101 106
cyclohexane	025 038 097 103
dichloromethane	006 023 035 059 095
ethyl acetate	025 048 097 100 103 104
hexane	044 050 059
methanol	037 074 081 096
petroleum ether/spirit	035 059 072 095
water	010 038 042 059 077 095 096 103

Extraction Technique Used	laboratory number
ASE	023 048
cold solvent extraction at atmospheric pressure	002 004 007 008 009 010 014 016 021 024 025 028 032 033 035 036 038 044 045 046 047 049 050 053 057 058 062 064 066 070 071 072 073 074 077 081 086 088 091 096 097 098 100 101 104 106 107 108
liquid/liquid partition	059
QuEChERS	084
shaker	006 022(30min) 042
solvent extraction	020
SPE	037
Vortex	052

Extraction pH Adjusted	laboratory number
yes	007 008 012 014 021 037 039 049 084 086 097 098 106
no	002 004 006 009 010 013 016 020 022 023 024 025 027 028 032 033 034 035 036 038 042 044 045 046 047 048 050 052 053 057 058 059 062 063 064 065 066 070 071 072 073 074 077 081 088 091 094 095 096 100 101 103 104 107 108

Sample Clean-up Technique	laboratory number
carbon based column	021 047
extraction	025 028 049 062 091 108
Extrelut	066 096
filter	008 010 042
florisil column	016 020 059
GPC/HPGPC	006 023 024 025 032 038 048 073 081 094 098 107 108
liquid/liquid extraction / solvent exchange	002 013 021 034 058 059 062 070 071 088 095 108
NH_2/aminopropyl column	002 012 021 047 065
silica column	037 094
solid phase extraction (SPE) (column/cartridge)	009 021 022 023 036 050 057 059 062 063 064 070 071
solid phase extraction (SPE) (dispersive)	004 007 014 033 039 045 053 084 086 097 101 103 106
C18 SPE column/cartridge	022
dilute	042
none	044 046 072 074 100

SPE Column Type	laboratory number
BONDESil - NH_2	095
C18	002 007 009 033 039 062 063
C18/Amino	023
Envi-Carb /GCB	012 036 047 059
Envi-Carb /NH_2	022 034 070 071
graphite-carbon/NH_2	058 062 088
PSA	004 014 021 039 052 053 057 062 064 084 106
silica	050 094
strata X 200 mg	037

Certified Standards Used	laboratory number
yes	002 004 006 007 008 012 013 014 016 021 022 023 024 025 027 028 032 033 034 035 036 037 038 039 042 045 046 047 048 049 050 052 053 059 062 064 066 070 071 072 073 074 077 081 084 086 091 094 095 096 097 098 100 101 103 104 106 107
no	009 010 020 044 057 058 063 065 088 108

MS Confirmation	laboratory number
yes	002 004 006 007 008 009 010 012 014 016 020 021 022 023 024 027 028 032 033 034 035 036 037 038 039 042 044 045 046 047 048 049 050 052 053 057 062 064 066 070 071 072 073 074 077 081 084 086 091 094 095 096 097 098 100 103 104 106 107
no	013 025 058 059 063 065 088 101

Calibrations	laboratory number
matrix-matched	004 008 013 014 016 022 023 024 025 032 033 034 037 042 046 047 048 050 053 057 059 065 070 071 072 074 077 081 091 094 096 097 098 101 103
multi-level	002 007 009 012 014 016 020 021 023 027 028 033 034 035 042 045 047 049 050 058 059 062 070 071 073 077 084 088 094 096 097 098 100 104 108
single-level	006 008 010 025 036 038 044 052 086 095 107
solvent	009 021 028 035 038 042 045 058 062 063 074 088 107 108
standard addition	039 042 066 096 097 106

Source of Standards	laboratory number
Accu Standard	077 084
Chem Service	006 059 073
Dr Ehrenstorfer	004 007 008 010 012 016 021 023 024 025 027 028 032 033 035 036 037 038 042 044 045 047 048 049 050 052 053 063 064 066 070 071 072 081 086 094 096 097 098 100 101 103 104 106 107 108
Fluka	014 046 053 063

Source of Standards (continued)	laboratory number
Kanto	062 065
LGC	042 074
QMx	074
Sigma/Aldrich	014 028 034 039 053 063 077 098
Supelco	053
Wako	002 020 022 057 058 063 088

Quoted Percentage Recovery Measured in Same Analytical Batch as Test Material	laboratory number
yes	002 004 006 008 009 012 013 014 016 020 021 022 023 024 027 028 032 033 035 036 037 038 039 042 044 046 047 048 049 050 052 057 058 059 062 064 065 066 070 071 072 074 081 084 088 091 094 095 096 097 098 100 101 104
no	007 010 025 034 045 053 063 073 077 086 103 106 107 108

Stage the Spike was Added	laboratory number
prior to extraction	002 004 006 008 009 012 013 014 016 020 022 023 024 027 028 032 033 034 035 036 037 038 039 044 046 047 048 049 050 052 057 058 059 062 065 066 070 071 072 073 074 081 084 088 095 097 098 100 101 104 107
prior to clean up	064 094
prior to instrument measurement	021 042 096

Level of Spike (µg/kg)	laboratory number
<25	004 006 013 014 020 023 027 033 046 048 050 053 057 070 071 084
≥25 - <50	032 039 047 050 059 097 107
≥50 - <100	009 016 022 028 034 035 049 064 065 081 100 101 104 106
≥100 - <150	033 036 058 062 073 074 088 094 095 096 104 106
≥150 - <200	023 044 104 106
≥200 - <250	002 004 007 012 014 024 037 038 052 063 066 072 096 104

Level of Spike (µg/kg) (continued)	laboratory number
≥250 - <300	008 021 032 096
≥300 - <400	042 066
≥400 - <500	032 096
≥500	042 091 096 098

Composition of Blank Commodity used for Spiking	laboratory number
blank provided	002 004 007 009 012 013 014 016 021 022 028 032 034 038 044 046 047 048 050 057 062 063 064 065 066 070 071 073 094 095 101 104 107
test material provided	024 036 039 042 049 052 072 096 097
blank ground wheat	006
flour	037
in house blank	027 035 081 100
previously analysed sample	074 106
rice	020
wheat	008 053
wheat flour	023 059 084 098

GC Column Type	laboratory number
capillary	002 006 010 012 013 014 016 020 021 022 023 025 027 032 033 034 037 039 044 045 047 048 049 050 052 053 057 062 063 064 065 070 071 072 073 074 081 086 094 095 097 098 101 103 104 107 108
megabore	059
narrowbore <0.53 mm id	038 100
widebore	024

GC Column Packing	laboratory number
100% methyl polysiloxane	059
14% cyanopropylphenyl 86%methyl polysiloxane	010 027
50% methyl 50% phenyl polysiloxane	032 059 081
65% methyl 35% phenyl polysiloxane	048

GC Column Packing (continued)	laboratory number
95% methyl 5% phenyl polysiloxane	002 006 012 013 014 016 020 021 022 023 024 025 027 032 033 034 037 038 039 045 047 049 050 052 053 057 062 063 064 065 070 071 072 073 086 094 097 098 100 103 104 107 108
5% methyl 95% dimethylpolysiloxane	074
50% Cyanopropylphenyl 50% methyl	059

GC Injection Volume (µL)	laboratory number
<1	057
≥1 - <2	006 013 021 023 025 027 032 037 039 047 048 050 052 059 062 065 070 071 072 094 095 097 098 100 103 104 107 108
≥2 - <5	012 014 020 022 024 033 034 038 063 073 081 086
≥5 - <10	002 016 045 053 074
≥10	010 049 064

GC Injection Mode	laboratory number
on-column	010 059
PTV	014 033 038 045 053 072 074 086
split	032 049 073 107
splitless	002 012 013 016 020 021 022 023 024 025 027 034 039 047 048 050 052 057 062 063 064 065 070 071 081 094 095 097 098 100 103 104 108
pulsed splitless	006

GC Detector	laboratory number
ECD	027 032 039 059 094 107
ELCD-X	059
FPD	027 059
ITD	045
MSD	002 006 010 012 013 014 020 021 022 024 025 032 033 034 037 038 039 047 048 049 050 062 063 064 073 081 086 094 095 098 100 104 107 108

GC Detector (continued)	laboratory number
MS-MS	016 023 025 052 053 057 065 070 071 072 074 103
MS-X-MS	097
NPD	032 037 039 094 107

HPLC Column Packing	laboratory number
C18	004 007 008 009 016 021 027 028 035 036 039 044 046 047 053 058 063 066 077 084 088 096 106
C18 X-Terra type	042
C8	049
endcapped	035 084

HPLC Guard Column Used?	laboratory number
yes	007 008 009 016 021 035 039 047 053 063 066 096 101 106
no	004 027 028 036 042 044 046 049 058 064 065 077 084 088 095 103

Mobile Phase Programme	laboratory number
isocratic	044
gradient	004 007 008 009 016 021 027 028 035 036 039 042 046 047 049 053 058 063 066 077 084 088 096 101 103 106

Mobile Phase Components	laboratory number
acetate	042
acetic acid	021 036 106
acetic acid ammonium	058 088
acetonitrile	007 009 028 035 036 039 044 077 101 106
ammonium acetate	084 096
ammonium formate	008
formic acid	009 016 028 044
methanol	004 021 027 042 046 047 049 053 058 063 066 088
water	004 007 021 027 035 042 044 046 053 066 077 101 106

077 101 106

HPLC Column Temperature (°C)	laboratory number
ambient	008 021 027 044 096 101
>ambient - <50	004 007 009 016 028 035 036 039 042 046 047 049 053 058 063 066 077 084 088 106

HPLC Injection Volume (µL)	laboratory number
<5	007 035 058 066 088
≥5 - <10	028 046 049 063 096
≥10 - <25	004 008 016 021 027 036 039 047 053 077 101 106
≥25 - <50	009 084
≥50 - <100	044
≥100 - <150	042

Mobile Phase Flow Rate (mL/min)	laboratory number
<0.25	016 021 058 088
≥0.25 - <0.75	004 007 008 009 027 028 035 036 042 046 047 049 053 063 066 077 084 096 106
≥0.75 - <1.25	039 044

HPLC Pre Column Derivatisation	laboratory number
none	027 035 036 044 046 047 049 063 066 084 096

HPLC Post Column Derivatisation	laboratory number
none	027 035 036 044 046 047 049 063 066 084 096

HPLC Detector Type	laboratory number
MS-MS	004 007 008 009 016 021 027 028 035 036 039 042 044 046 047 049 053 058 063 066 077 084 088 096 101 106

Trifloxystrobin

Accredited Method Used	laboratory number
yes	001 002 004 005 007 008 013 014 016 021 022 023 024 025 027 032 033 034 035 036 037 038 039 046 047 048 050 051 058 059 062 064 065 066 072 073 081 086 088 090 091 094 096 098 100 101 103 104 106 107 108
no	006 009 010 019 028 042 044 045 049 052 053 056 057 063 083 095 109

Sample Weight (g)	laboratory number
≥2 - <5	007 019 046 049 057 062 073 091 096 107
≥5 - <10	013 014 021 023 028 033 036 037 039 045 048 050 051 052 053 056 058 064 065 083 086 088 090 095 100 101 104 109
≥10 - <25	001 002 004 008 009 016 022 024 025 027 032 034 035 038 042 044 047 059 063 072 103 106
≥25 - <50	005 010 066 081 094 098 108
≥50	006

Water Added Prior to Extraction (SANCO/2007/3131)	laboratory number
yes	002 004 008 014 019 021 022 024 025 027 028 032 033 034 035 036 037 038 039 046 047 049 051 053 056 057 058 062 063 064 065 066 073 081 086 088 090 091 098 100 104 107
no	001 005 006 007 009 023 044 048 050 052 059 083 094 095 106 108 109

Extraction Solvent	laboratory number
acetic acid	008 013 052
acetone	004 005 006 010 016 023 025 032 035 038 044 047 050 051 058 059 065 072 081 088 094 095 098 103 104 107 108 109
acetonitrile	001 002 007 008 009 013 014 019 021 022 024 027 028 033 034 036 039 042 045 046 049 051 052 053 056 057 062 063 064 065 066 073 083 086 090 091 094 101

Extraction Solvent (continued)	laboratory number
cyclohexane	025 038 103
dichloromethane	004 006 023 035 044 051 059 095 109
ethyl acetate	025 048 096 100 103 104
hexane	004 050 059
methanol	005 037 081 106
petroleum ether/spirit	035 051 059 072 095
water	004 010 038 042 051 059 095 103

Extraction Technique Used	laboratory number
ASE	023 048
bath Sonication	109
cold solvent extraction at atmospheric pressure	001 002 004 005 007 008 009 010 014 016 021 024 025 028 032 033 035 036 038 044 045 046 047 049 050 051 053 056 057 058 062 064 066 072 073 081 086 088 090 091 096 098 100 101 104 106 107 108
liquid/liquid partition	059
shaker	006 022 042
SPE	037
Vortex	052

Extraction pH Adjusted	laboratory number
yes	007 008 014 019 021 037 039 049 086 098
no	001 002 004 005 006 009 010 013 016 022 023 024 025 027 028 032 033 034 035 036 038 042 044 045 046 047 048 050 051 052 053 056 057 058 059 062 063 064 065 066 072 073 081 083 088 090 091 094 095 096 100 101 103 104 106 107 108 109

Sample Clean-up Technique	laboratory number
C18 SPE column/cartridge	022
carbon based column	021 047
dilute	042
extraction	025 028 049 062 091 108

Sample Clean-up Technique (continued)	laboratory number
Extrelut	066
filter	005 008 010 042
florisil column	016 051 059
GPC/HPGPC	004 005 006 023 024 025 032 038 048 073 081 094 096 098 107 108
liquid/liquid extraction / solvent exchange	002 005 013 021 034 058 059 062 088 095 108
NH_2/aminopropyl column	001 002 021 047 065
silica column	037 094
solid phase extraction (SPE) (column/cartridge)	009 019 021 022 023 036 044 050 057 059 062 063 064 106
solid phase extraction (SPE) (dispersive)	007 014 033 039 045 053 056 086 090 101 103
none	046 072 083 100

SPE Column Type	laboratory number
BONDESil - NH_2	095
C18	002 007 009 033 039 062 063 106
C18/Amino	023
Envi-carb/GCB	036 047 059
Envi-Carb/NH_2	022 034
graphite-carbon/NH_2	058 062 088
PSA	001 014 021 039 052 053 056 057 062 064 090
silica	044 050 094
strata X 200 mg	037

Certified Standards Used	laboratory number
yes	001 002 004 005 006 007 008 013 014 016 019 021 022 023 024 025 027 028 032 033 034 035 036 037 038 039 042 044 045 046 047 048 049 050 051 052 053 056 059 062 064 066 072 073 081 083 086 091 094 095 096 098 100 101 103 104 106 107 109
no	009 010 057 058 063 065 088 090 108

MS Confirmation	laboratory number
yes	001 002 004 005 006 007 008 009 010 014 016 019 021 022 023 024 027 028 032 033 034 035 036 037 038 039 042 044 045 046 047 048 049 050 051 052 053 056 057 062 064 066 072 073 081 083 086 090 091 094 095 096 098 100 103 104 106 107 109
no	013 025 058 059 063 065 088 101

Calibrations	laboratory number
matrix-matched	004 008 013 014 016 022 023 024 025 032 033 034 037 042 046 047 048 050 053 057 059 065 072 081 090 091 094 096 098 101 103
multi-level	001 002 004 007 013 014 016 021 023 027 028 033 034 035 042 045 047 049 050 051 056 058 059 062 073 088 090 094 096 098 100 104 108 109
single-level	006 008 009 010 025 036 038 044 052 086 095 107
solvent	005 009 019 021 028 035 038 042 045 058 062 063 088 107 108
standard addition	039 042 066 083 096 106

Source of Standards	laboratory number
Accu Standard	051
Chem Service	006 059 109
Dr Ehrenstorfer	001 004 007 008 010 016 021 023 024 025 027 028 032 033 035 036 037 038 042 045 047 048 049 050 052 053 062 063 064 066 072 081 086 090 094 096 098 100 101 103 104 106 107 108
Fluka	014 053 063 090
Kanto	062 065
LGC	042
Riedel-deHaen	073
Sigma/Aldrich	005 009 014 028 034 039 044 046 053 056 063 083 098
Supelco	053
Wako	002 019 022 057 058 063 088

Quoted Percentage Recovery Measured in Same Analytical Batch as Test Material	laboratory number
yes	001 002 004 005 006 008 009 013 014 016 019 021 022 023 024 027 028 032 033 035 036 037 038 039 042 044 046 047 048 049 050 051 052 056 057 058 059 062 064 065 066 072 081 088 090 091 094 095 096 098 100 101 104 109
no	007 010 025 034 045 053 063 073 086 103 106 107 108

Stage the Spike was Added	laboratory number
prior to extraction	001 002 004 005 006 008 009 013 014 016 019 022 023 024 027 028 032 033 034 035 036 037 038 039 044 046 047 048 049 050 051 052 056 057 058 059 062 065 066 072 073 081 088 090 095 098 100 101 104 107 109
prior to clean up	064 094
prior to instrument measurement	021 042 096

Level of Spike (µg/kg)	laboratory number
<25	004 006 013 014 023 027 033 046 048 050 053 057 106 109
≥25 - <50	032 039 047 050 059 090 096 107
≥50 - <100	009 016 019 022 034 035 049 064 065 081 096 100 101 104
≥100 - <150	001 004 033 036 056 058 062 073 088 094 095 096 104 106
≥150 - <200	023 044 096 104
≥200 - <250	002 007 014 037 052 063 072 096 104 106
≥250 - <300	008 021 032
≥300 - <400	004 024 028 038 042 066 096
≥400 - <500	032 066 096
≥500	005 042 091 098

Composition of Blank Commodity used for Spiking	laboratory number
blank provided	001 002 004 007 009 014 016 019 021 022 028 032 034 038 044 046 047 048 050 051 057 062 063 064 065 066 073 094 095 101 104 107
test material provided	024 036 039 042 049 052 072 096 109
blank ground wheat	006
flour	037
in house blank	013 027 035 081 090 100
in house cereal sample	005
previously analysed sample	106
wheat	008 053
wheat flour	023 059 098

GC Column Type	laboratory number
capillary	001 002 004 006 013 014 016 019 021 022 023 025 027 028 032 033 034 037 039 044 047 048 049 050 051 053 056 062 063 064 065 081 083 086 090 094 095 096 098 101 103 104 106 107 108 109
megabore	059 072
narrowbore <0.53 mm id	005 009 038 100
widebore	024

GC Column Packing	laboratory number
100% methyl polysiloxane	059 072 106
14% cyanopropylphenyl 86%methyl polysiloxane	027
50% methyl 50% phenyl polysiloxane	005 028 032 059 072 081
65% methyl 35% phenyl polysiloxane	048 051
95% methyl 5% phenyl polysiloxane	001 002 004 006 009 013 014 016 019 021 022 023 024 025 027 032 033 034 037 038 039 044 047 049 050 053 056 062 063 064 065 083 086 090 094 096 098 100 103 104 107 108 109
50% Cyanopropylphenyl 50% methyl	059

GC Injection Volume (µL)	laboratory number
≥1 - <2	001 004 006 013 019 021 023 025 027 032 037 039 044 047 048 050 051 059 062 065 072 090 094 095 098 100 103 104 106 107 108 109
≥2 - <5	005 009 014 022 024 028 033 034 038 056 063 081 086
≥5 - <10	002 016 053 096
≥10	049 064 083

GC Injection Mode	laboratory number
on-column	059
PTV	004 014 028 033 038 053 056 086 096
split	005 032 049 051 083 107
splitless	001 002 009 013 016 019 021 022 023 024 025 027 034 039 044 047 048 050 052 062 063 064 065 072 081 090 094 095 098 100 103 104 106 108 109
pulsed splitless	006

GC Detector	laboratory number
ECD	004 027 032 037 039 044 059 072 090 094 107 109
ELCD-X	059
FPD	004 027 059
MSD	001 002 004 005 006 013 014 019 021 022 024 025 028 032 033 034 037 038 039 047 048 049 050 051 056 062 063 064 081 086 094 095 096 098 100 104 106 107 108 109
MS-MS	009 016 023 053 065 072 083 103
NPD	032 037 039 094 107 109

HPLC Column Packing	laboratory number
C18	007 008 010 016 021 027 035 036 039 046 047 052 053 058 062 063 066 073 088
C18 X-Terra type	001 042
C8	049
endcapped	010 035 045

HPLC Guard Column Used	laboratory number
yes	007 008 016 021 035 039 045 047 052 053 057 062 063 066 073 101
no	010 027 036 042 046 049 058 064 065 088 095 103 106 109

Mobile Phase Programme	laboratory number
gradient	007 008 010 016 021 027 035 036 039 042 045 046 047 049 052 053 057 058 062 063 066 073 088 101 103

Mobile Phase Components	laboratory number
acetate	042
acetic acid	021 036
acetic acid ammonium	058 088
acetonitrile	007 010 035 036 039 052 101
ammonium acetate	062
ammonium formate	008 045 073
formic acid	010 016 052
methanol	021 027 042 045 046 047 049 053 057 058 062 063 066 073 088
water	007 010 021 027 035 042 045 046 053 062 066 101

HPLC Column Temperature (°C)	laboratory number
ambient	008 021 027 101
>ambient - <50	007 010 016 035 036 039 042 045 046 047 049 052 053 057 058 062 063 066 073 088

HPLC Injection Volume (µL)	laboratory number
<5	007 035 058 066 088
≥5 - <10	010 045 046 049 062 063
≥10 - <25	008 016 021 027 036 039 047 052 053 057 073 101
≥100 - <150	042

Mobile Phase Flow Rate (mL/min)	laboratory number
<0.25	016 021 045 052 057 058 088
≥0.25 - <0.75	007 008 010 027 035 036 042 046 047 049 053 062 063 066 073
≥0.75 - <1.25	039

HPLC Pre Column Derivatisation	laboratory number
none	010 027 035 036 046 047 049 057 062 063 066

HPLC Post Column Derivatisation	laboratory number
none	010 027 035 036 046 047 049 057 062 063 066

HPLC Detector Type	laboratory number
MS-MS	007 008 010 016 021 027 035 036 039 042 045 046 047 049 052 053 057 058 062 063 066 073 088 101

APPENDIX III: FAPAS® Secure Web, 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 Protocols [6, 7] set 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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