CERTIFIED DOCUMENT

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LABORATORY NUMBER: Available to participants from FAPAS SecureWeb

FAPAS® Proficiency Test 1978

Pesticide Residues in Cabbage Purée

March - May 2008

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 1978 was dispatched in March 2008. Each participant received a cabbage purée test material to be analysed for pesticide residues. In total, 83 sets of test material were distributed to participants in 25 countries. Of these, 69 participants, i.e. 83%, returned results for some combination of the analytes within the time-scale demanded by the Scheme.

2. From a list of 47 possible pesticide residues, participants were requested to identify and quantify those present in the cabbage purée. The test material contained carbendazim, cyprodinil and methiocarb.

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

4. The target standard deviation (σ_p) for each analyte was calculated using the appropriate form of the Horwitz equation [4] and in conjunction with the assigned value ($\hat{X}$) was used to derive a z-score for participants' results. z-Scores are considered satisfactory if $|z| \leq 2$.

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

analyte	assigned value, $\hat{X}$, µg/kg	number of satisfactory scores, $\|z\| \leq 2$	total number of scores	satisfactory %
carbendazim	265	44	59	75
cyprodinil	151	47	55	85
methiocarb	104	55	66	83

6. Surplus test materials are available for sale, see APPENDIX III.

7. Whereas this Report has been produced in good faith and in accordance with best industry practice, neither the Central Science Laboratory nor the Secretary of State for Environment, Food and Rural Affairs accepts any liability whatsoever as to the application or use of the information contained therein.

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

laboratory number		analyte														
		carbendazim assigned value 265 µg/kg				cyprodinil assigned value 151 µg/kg				methiocarb assigned value 104 µg/kg				methiocarb sulfoxide assigned value not set		
		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
001		#				#				71.30	80	10	-1.4	#		
002		239	74.6	10	-0.5	76	47.9	10	**-2.3**	125	96.0	10	0.9	ND		10
003		550	117	60	**5.5**	160	93	60	0.3	100	96	60	-0.2	ND	105	30
004		239	81	5	-0.5	127	90	5	-0.8	91	117	5	-0.6	trace •		5
005		275	97.2	5	0.2	163	97.1	5	0.4	108	100.2	5	0.2	7.6	98.5	5
006		263		5	0.0	187		5	1.1	111		5	0.3	7		5
007		122		100	**-2.8**	#				#				#		
008		340	94	50	1.4	151	88	10	0.0	160	96	10	**2.4**	#		
009		262	89		-0.1	185	94		1.0	113	77		0.4	#		
010		147	92	10	**-2.3**	175	94	10	0.7	118	105	10	0.6	ND		10
011		150	80–120	5	**-2.2**	118	80–120	5	-1.0	#				#		
012		252	100	10	-0.3	113	100	10	-1.2	116	100	10	0.5	ND		10
013	†	380	105	20	**2.2**	#				140	120	20	1.6	ND	93	20
014		250	83	10	-0.3	190	118	10	1.2	110	100	10	0.3	#		
015		#				464			**9.7**	286			**7.9**	#		

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

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

laboratory number		analyte														
		carbendazim assigned value 265 µg/kg				cyprodinil assigned value 151 µg/kg				methiocarb assigned value 104 µg/kg				methiocarb sulfoxide assigned value not set		
		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
016	†	#				#				0			**-4.5**	#		
017		354	100	10	1.7	161	75	10	0.3	75.0	116	10	-1.3	ND		10
018		204.00	85		-1.2	111.315	88	10	-1.2	78.00	87		-1.1	#		
019		223	105.8	10	-0.8	150	91	10	0.0	105	106.2	10	0.0	ND		50
020		432	100	10	**3.2**	121	100	10	-0.9	137	100	10	1.4	11	100	10
021		398	110	10	**2.6**	248	100	10	**3.0**	150	100	10	2.0	18.2	99	10
022		291	97	10	0.5	175	97	10	0.7	131	103	10	1.2	6	98	10
023		159.5	91	10	-2.0	143	96	10	-0.3	103.5	105	10	0.0	#		
024		252	106	10	-0.3	187	97	10	1.1	120	105	10	0.7	10	102	10
025		275	88		0.2	170	95		0.6	110	95		0.3	ND		20
026		#				140	98	10	-0.4	110	106	10	0.3	ND	86	10
027		#				#				110	88	100	0.3	#		
028	†	191.3	58.2	5	-1.4	144.2	83.4	5	-0.2	92.1	93.6	5	-0.5	4.6	56.2	2
029		354			1.7	122			-0.9	111			0.3	ND		
030		235	92	4	-0.6	178	87	4	0.8	103	95	4	-0.1	7.4	92	4

z-scores outside the satisfactory range, i.e. |z| >2, are shown in **bold** LoQ = limit of quantification ND = not detected
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Table 1 (continued): Results and z-Scores for Cabbage Purée 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
031		234	81	10	-0.6	136	81.0	10	-0.5	79.0	85	10	-1.1	<LOQ		10
032		289.2	115	20	0.5	145.0	80.0	20	-0.2	92.1	93	20	-0.5	14.6	116	10
033		218		10	-0.9	173		10	0.7	80		10	-1.1	< 10		10
034		200		10	-1.3	203		10	1.6	106		10	0.1	#		
035		303	80	10	0.7	#				79	92	10	-1.1	10	109	10
036		275.6	90	10	0.2	203.63	82	10	1.6	112.17	89	10	0.3	9.6 *	79	10
037	†	410	96	20	**2.8**	#				780	78	10	**29.5**	10	120	10
038		#				278		0.01	**3.9**	71		0.01	-1.4	#		
039		249	78	10	-0.3	155	88	10	0.1	105	90	10	0.0	ND	95	10
040		280	80	10	0.3	70	80	10	**-2.5**	55	80	10	**-2.1**	#		
041		182	74	10	-1.6	104	81	10	-1.5	328	96	10	**9.8**	#		
042		219	71.8	10	-0.9	150	71.2	15	0.0	77	74.7	4	-1.2	5	68.1	4
043		164	88	20	-2.0	102	88	20	-1.5	72	83	20	-1.4	ND	84	20
044		#				#				319.2	70–120		**9.4**	85.6	70–120	
045		144	76	10	**-2.3**	177	82	10	0.8	#				#		

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

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

laboratory number	analyte														
	carbendazim assigned value 265 µg/kg				cyprodinil assigned value 151 µg/kg				methiocarb assigned value 104 µg/kg				methiocarb sulfoxide assigned value not set		
	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
046	260	112.9	10	-0.1	160	94.8	10	0.3	100	104.0	10	-0.2	91	102.2	10
047	487	89	10	**4.3**	152	95	10	0.0	104	98	25	0.0	ND		20
048	252	90	1	-0.3	#				101	105	1	-0.1	10	94	1
049	250	120	10	-0.3	191	89	10	1.2	111	95	4	0.3	#		
050	49	91	1	**-4.2**	30	102	3	**-3.8**	19	96	1	**-3.7**	3	105	1
051	#				#				80.00		20	-1.1	#		
052	246.06	105	30	-0.4	#				111.35	99.2	30	0.3	#		
053	313	83	10	0.9	200	109	10	1.5	144	81	10	1.7	#		
054	174.8	75	10	-1.7	#				124.2	94	10	0.9	10	76	10
055	207	110	5	-1.1	126	97	5	-0.8	93	109	5	-0.5	#		
056	627.3	82.5	5	**7.0**	146.8	86.2	5	-0.1	84.8	86.6	5	-0.8	#		
057	241	95	5	-0.5	180	91	5	0.9	97	103	5	-0.3	▲		10
058	305	93.43	10	0.8	157	83.44	11	0.2	104	59.58	14	0.0	#		
059	513	86	10	**4.8**	250	75	10	**3.1**	157	72	10	**2.3**	12	70	10
060	221	80	10	-0.8	192	80	10	1.3	85	80	10	-0.8	10	80	10

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

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

laboratory number		analyte														
		carbendazim assigned value 265 µg/kg				cyprodinil assigned value 151 µg/kg				methiocarb assigned value 104 µg/kg				methiocarb sulfoxide assigned value not set		
		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
061		318	96	10	1.0	91	96	10	-1.9	108	96	10	0.2	#		
062		259	100	20	-0.1	99	100	10	-1.6	87	89	30	-0.7	20*	100	30
063		529		5	**5.1**	123		10	-0.9	93		10	-0.5	6		5
064		250	100	10	-0.3	170	90	10	0.6	87	100	10	-0.7	11	97	10
065	†	#				#				0		13	**-4.5**	#		
066		#				104		10	-1.5	130		10	1.1	#		
067		26	100	7	**-4.6**	#				14	95	11	**-3.9**	#		
068		242	80	10	-0.4	104	60	10	-1.5	97	80	10	-0.3	10	100	10
069		280	70	50	0.3	0			**-4.7**	79	70	50	-1.1	#		

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

Table 2: Additional Pesticide Residues Reported

laboratory number	pesticide residue ≥ 30 µg/kg	result µg/kg	recovery %	LoQ µg/kg
013	metamitron	63	110	20
016	carbaryl	1625		
016	methomyl	35000		
016	oxamyl	12389		
016	propoxur	51657		
028	disulfoton	31.4	82	5
037	thiabendazole	110	80	10
065	ethiofencarb	175		

Table 3: 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
carbendazim	49	265	87.3	12.5	Horwitz*	51.8
cyprodinil	42	151	37.8	5.8	Horwitz*	32.2
methiocarb	50	104	26.4	3.7	Horwitz*	22.9

* see page 7 for appropriate form of the Horwitz equation

Table 4: Number and Percentage of Satisfactory z-Scores

analyte	number of satisfactory scores $\|z\| \leq 2$	total number of scores	satisfactory %
carbendazim	44	59	75
cyprodinil	47	55	85
methiocarb	55	66	83

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

criteria	number of satisfactory participants	total number of participants	satisfactory %
correctly identified all three pesticides	47	69	68
correctly identified and obtained satisfactory z-scores for all three pesticides	34	69	49