CRL-PROFICIENCY TEST-SRM 1, 2006

Pesticide Residues in Apple Juice Homogenate using Single Residue Methods

Final Report

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QCG: Quality Control Group
AG: Advisory Group

CONTENTS

EUROPEAN COMMISSION CRL-PROFICIENCY TEST 1

ON PESTICIDE RESIDUES USING SINGLE RESIDUE METHODS IN APPLE JUICE HOMOGENATE

2006

The Council Directives 86/362/EEC[1] and 90/642/EEC[2] provide for the organisation and financial support for regular proficiency testing (PT) of those laboratories that perform analyses for their official national monitoring programmes. These proficiency tests are performed in order to ensure the quality, accuracy and comparability of the residue data sent by EU Member States to the European Commission, as well as to the other Member States. All PTs organized so far within this framework have predominantly required the use of multiresidue methods (with a few exceptions). This test, however, focuses on laboratories performing single or group-specific residue methods.

With the recent establishment of Community Reference Laboratories (CRLs) for food, feed and animal health, EU proficiency testing has been given a new broader framework. According to Regulation (EC) No 882/2004[3], which specifies the general responsibilities of the CRLs, the organisation of comparative tests is among the CRL's main tasks. The present Test has been the first organised under the umbrella of the CRL for pesticide residue analysis using Single Residue Methods, the CVUA Stuttgart, and the first purely focusing on compounds that are traditionally not amenable to multiresidue analysis. Participation in this 1st Single Residue Method European Proficiency Test was open to all official national or regional analytical laboratories involved in the determination of pesticide residues in food within the EU.

This report will be presented to the Standing Committee for Animal Health and the Food Chain.

[1] Council Directive 86/362/EEC of 24 July 1986 on the fixing of maximum levels for pesticide residues in and on cereals. Published at OJ of the EU 221, 7.8.1986, p. 37. Directive as last amended by Commission Directive 2006/62/EC (OJ L 206, 27.7.2006, p. 27).

[2] Council Directive 90/642/EEC of 27 November 1990 on the fixing of maximum levels for pesticide residues in and on certain products of plant origin, including fruit and vegetables. Published at OJ L 350, 14.12.1990, p. 71. Directive as last amended by Commission Directive 2006/62/EC.

[3] Regulation (EC) N° 882 /2004 of the European Parliament and of the Council on official controls performed to ensure the verification of compliance with feed and food law, animal health and animal welfare rules. Published at OJ of the EU L191 of 28.05.2004

1. INTRODUCTION

On the 3rd of July 2006, 130 official laboratories as well as the contact points of the EU Member States were sent an invitation to participate in this 1st European Commission's Single Residue Method Proficiency Test. A list of fifteen possible pesticides (Annex 1), which might have been potentially present in the test material, was also included in this invitation. Followng this call, twenty seven laboratories from 15 countries agreed to participate in this PT.

This proficiency test was performed using apple juice homogenate of Spanish origin, that was spiked with three pesticides. Participating laboratories were provided with 400 g portions of each 'blank' apple juice homogenate as well as the spiked apple juice. The test materials were shipped to participants on the 4th of September, 2006 and the deadline for submission of results to the Organiser was the 25th of September 2006. The participants were asked to analyse the spiked test material as well as the `blank` material and report the concentrations of any pesticide residues they found which were included in the list (Annex 1). The 'blank' material was intended to be used by the participants for recovery experiments for the pesticides found in the test material, and if necessary, for the preparation of matrix-matched calibration standards.

The median values of the analytical data submitted were used to obtain the assigned (true) concentrations for each of the pesticide residues present. A fit-for-purpose target relative standard deviation (FFP RSD) of 25%, based on the experience of the Advisory Group, was chosen to calculate the target standard deviations (σ) as well as the z-scores of the compounds present. For informative purposes, the Horwitz Equation was additionally used to calculate target standard deviations and the corresponding z-score values were also calculated.

2. TEST MATERIALS

2.1 Analytical methods

The following analytical methods, described briefly below, were used by the Organisers for the homogeneity and stability tests performed:

- For organotin pesticides: QuEChERS-method (1, 2), involving extraction with acetonitrile, partitioning following addition of salts, dispersive SPE cleanup using PSA sorbent and determination by LC-MS/MS using a gradient containing 1% formic acid.
- For acidic pesticides: QuEChERS-method (1, 2), involving extraction with acetonitrile, partitioning after addition of salts, and direct determination by LC-MS/MS.
- For chlormequat and mepiquat: In-house-method based on (3), involving addition of an isotopically labelled internal standard, extraction with methanol, centrifugation, filtration and direct determinative analysis by LC-MS/MS.

2.2 Preparation of the treated test material

Before preparing the test material, the pesticides and suitable residue levels for the study were selected following recommendations made by the Quality Control Group, which had been specifically appointed for SRM-Proficiency Test 1. Fifty kilograms of apple juice were used in total. Half of the sample, twenty five kilograms, was poured into a large glass beaker,and spiked with the pesticides (chosen by the advisory group), whilst stirring. The mixture was transferred into 5L containers that were rolled over for 24 hours to allow the residues to interact with the matrix. The twenty five liters were then transferred again into the beaker and mixed together intensively using an automatic spinning rotator. A portion was taken and analysed to check the residue levels present in the material. Since the residue levels detected were close to those recommended by the Advisory Group, the test material was sampled. The 400 g samples were weighed out into screw-capped polyethylene plastic bottles, sealed, and stored in a freezer at about - 20 °C prior to distribution to participants.

2.3 Preparation of the 'blank' test material

The apple juice used for the production of the 'blank' test material was subjected to the same treatment as the spiked test material described above.

2.4 Homogeneity test

Ten bottles were randomly chosen from those stored in the freezer and analyses were performed on duplicate portions taken from each bottle. The sequence of analyses was determined using a table of randomly generated numbers. The injection sequence of the 20 extracts was also

randomly chosen in each case. The quantification was performed in each case using a 3-point calibration curve constructed from matrix-matched standards.

The statistical evaluation was performed according to the International Harmonized Protocol published by IUPAC, ISO and AOAC (3). The individual residue data from the homogeneity tests are given in Appendix1. The results of the statistical analyses are given in Table 2.1. The acceptance criteria for the test material to be sufficiently homogenous for the proficiency test were that F critical > F for (p = 0.05), and that $S_s/\sigma < 0.3$, with S_s being the between sampling standard deviation and σ = RSD (25%) x the mean concentration of each pesticide.

Table 2.1. Statistical evaluation of the homogeneity test data (n = 20 analyses)

	Chlormequat (mg/Kg)	**Fenbutatin oxide** (mg/Kg)	**MCPA** (mg/Kg)
Mean (mg/Kg)	0.172	0.490	0.366
F critical	3.02	3.02	3.02
F	2.88	1.06	0.43
S_s/σ	0.10	0.01	0.11
Pass/Fail	Pass	Pass	Pass

Ss: Between Sampling Standard Deviation

2.5 Stability tests

The analytical methods described briefly above (in section 2.1) were also used for the stability tests.

The tests were performed on two occasions. On each occasion, a single bottle stored in the freezer at -20°C was chosen randomly and duplicate analyses were performed.

The two occasions were:

- Day 1: coinciding with the first sample shipment, which took place on 4th September 2006.
- Day 2: shortly after the deadline for reporting results, on 25th September 2006.

The individual results are given in Tables 2.2. In general, these tests did not show any significant decrease in the levels of the three pesticides and demonstrated that the pesticides present in the test material remained stable for the entire duration of the Proficiency Test.

Table 2.2. Statistical test to demonstrate stability

	Chlormequat (mg/Kg)	Fenbutatin oxide (mg/Kg)	MCPA (mg/Kg)
Day 1 (1st sample)	0.167	0.497	0.36
Day 1 (2nd sample)	0.168	0.495	0.363
Mean 1	0.168	0.496	0.362
Day 2 (1st sample)	0.176	0.493	0.39
Day 2 (2nd sample)	0.173	0.487	0.328
Mean 2	0.175	0.490	0.359
(M1-M2)/M1	0.042	0.012	-0.007
%	4.18	1.2	0.69

2.6 Distribution of test material and protocol to participants

One bottle of treated test sample and one bottle of 'blank' material were shipped to each participant in boxes containing dry ice. The samples were sent on the 4th September, 2006.

Following the receipt of the Application Form by each participant a laboratory code was given and all relevant documents (see Annex 1) including forms for reporting the receipt and condition of the samples as well as for reporting the final results and the analytical methods used were sent by e-mail to all participant laboratories. This ensured that confidentiality was maintained throughout the entire duration of this SRM-Proficiency Test. These documents were also uploaded on to the SRM-EUPT 1 web page constructed especially for this Proficiency Test.

3. STATISTICAL METHODS

3.1 False positives and negatives

3.1.1 False positives

In principle, results indicating the presence of pesticides that were included in the pesticide list, and which were (i) not used in the preparation of the test material, (ii) and not detected by the organiser, even following a repeat analysis, were treated as false positives, if they were reported at concentrations at or above the MRPL stipulated by the Organiser. Results reported that were lower than 0.01mg/Kg were ignored by the Organiser and not considered as false positives. No z-score value was calculated for these results.

3.1.2 False negatives

Results for pesticides that were not reported by the laboratories, although they were used by the Organiser to treat the test material and were subsequently detected at, or above, the MRPL by the Organiser (and the majority of participating laboratories) were considered to be false negatives. z-Scores were not only calculated for all pesticides detected at levels exceeding the MRPL but also for the false negatives, in the latter case using the MRPL for calculation.

3.2 Estimation of the assigned values

To establish the assigned values, the median levels of all the reported results, excluding the outliers, were used. Individual results without any absolute values reported, such as detected (D), were ignored.

3.3 Fixed target standard deviation (fit-for-purpose, FFP)

To assign the target standard deviations for each individual pesticide, a fixed relative standard deviation (fit-for-purpose, FFP) was used. Based on previous experience and recommendations by the Advisory Group and also as a conclusion from the discussion session on proficiency testing at EPRW 2004 in Stockholm, Sweden, the fixed relative standard deviation (FFP RSD) was considered to be 25 %. The target standard deviation (σ) for each individual pesticide was calculated by multiplying this FFP RSD by the assigned value. In addition, the concentration dependent Horwitz standard deviation was also calculated for informative purposes. This value was multiplyed by the median value to obtain the (Horwitz) target standard deviation.

3.4 z-Scores

A z-score for each laboratory/pesticide combination is calculated according to the following equation:

$$z = (x-X) / \sigma \qquad \text{Eq.1}$$

Where:

- x is the result reported by the participant or the MRPL for those labs not having detected the pesticide present in the sample
- X is the assigned value or true concentration
- σ is the target standard deviation obtained by multiplying the median by the FFP RSD of 25%
- σ_H is the target standard deviation calculated using the Horwitz equation

z-Score classification is as follows:

$\|z\| \leq 2$	Acceptable
$2 < \|z\| \leq 3$	Questionable
$\|z\| > 3$	Unacceptable

- Any z-score values of $|z| > 5$ is reported as '+5', or '-5'.
- No calculation of z-score is performed for any false positive result.
- For false negatives, the MRPL is used to calculate the z-score and whether it should be included or not in a graphical representation is being considered.

4. RESULTS

4.1 Summary of results reported

Twenty seven laboratories agreed to participate in this proficiency test and three of them did not submit results.
The results of these participating laboratories are presented in this report.
A summary of the results reported can be seen below in Table 4.1.

Table 4.1 Summary of Results

Pesticides	No. of Reported Results	No. of Reported NA	No. of Reported ND (False negatives)	% of the Total Reported Results *
Chlormequat	23	1	0	96
Fenbutatin oxide	5	19	0	21
MCPA	10	14	0	42

* The % of the total results has been calculated using the number of reported results from the total number of laboratories submitting results.

NA = Not analysed
ND = Not detected

The laboratories that agreed to participate are listed in Annex 2. All data reported by the participants is shown in the appendices. The analytical results reported can be seen in Appendix 3 and 7, the recoveries achieved and the analytical methods used are shown in Appendix 7. For an explanation of the symbols used in these tables, see Annex 1.

4.1.1 False positives

No false positives were reported.

4.1.2 False negatives

Pesticides actually present in the test material but reported as not detected (ND), would have been considered to be false negatives. No false negatives were reported.

4.2 Assigned values and target standard deviations

To establish the assigned values, the medians of all the reported results were used. A statistical programme was used to calculate the medians. In the case of MCPA one value was excluded from the median calculation since it was very distant from the second largest result reported. However, the median would not have been significantly changed even if this outlier had been included. All median values for all pesticides can be seen in Table 4.2.

There were not enough results reported for fenbutatin oxide to allow a statistical treatment. Therefore no z-score values were calculated for the laboratories reporting this pesticide.
The target standard deviation was obtained using a fixed FFP RSD value of 25%. In parallel, a robust standard deviation (Qn) as well as the concentration dependent Horwitz RSDs were also calculated for informative purposes. These RSDs can be seen in Table 4.2.

Table 4.2 Median values and RSDs for all pesticides present in the test material

Pesticides	MPRL (mg/Kg)	Median (mg/Kg)	FFP RSD (%)	Horwitz RSD (%)	Qn RSD (%)
Chlormequat	0.05	0.171	25	21	16
Fenbutatin Oxide*	0.05	-	-	-	-
MCPA	0.05	0.315	25	19	23

*** no calculations performed because of too few results reported**

4.3 Assessment of laboratory performance

4.3.1 z-Scores

z-Scores have been calculated for chlormequat and MCPA in two different ways;

1) Using the FFP RSD of 25%; Appendix 3 shows the individual z-scores together with the median for each laboratory and pesticide, and Appendix 4 - the corresponding graphs.
2) Using Horwitz Equation; Appendix 5 shows the individual z-scores together with the median for each laboratory and pesticide and Appendix 6 - the corresponding graphs.

Each compound was treated individually and, unlike the PTs for multiresidue methods, no laboratory ranking based on Weighted Summed z-Scores (WSZ) were calculated.

5. CONCLUSIONS

As this was the first proficiency test for single residue methods, it was decided to keep the list of pesticides to be sought (pesticide list) small and to spike the test sample with only a few compounds at relatively high concentrations. Also, the test material chosen, apple juice, was considered to be of low analytical difficulty.

In total, twenty seven laboratories from 15 countries applied to participate in this test and twenty four of them submited results.

The pesticide list contained analytes from 3 different classesof pesticides (quarternary ammonium pesticides, organotin-pesticides and phenoxyacid pesticides) each potentially requiring separateextraction/clean-up procedures. The test material was spiked with one compound per group namely chlormequat, fenbutatin oxide, and MCPA.

Chlormequat, which is included in the EU-monitoring programme was analysed by 23 of the 24 participating laboratories. Ten laboratories submitted results for MCPA, but only 5 for fenbutatin oxide (plus one reporting a semi-quantitative result). No median values or z-scores were calculated for the latter due to the small number of results reported.

The participaton in the proficiency test and the number of reported results may have been rather low, but the overall accuracy of the reported results was outstanding compared with previous PTs involving multiresidue analysis. Irrespective of which RSD was used for calculation (FFP RSD of 25 % or Horwitz RSD), all results for chlormequat, and all but one for MCPA, were within the satisfactory range (z< +/-2). No false positive and no false negative results were reported.

The relatively low participation rate in this proficiency test indicates that the use of single residue or group specific methods in the EU member states may still be rather limited. This is surely related to the fact that such methods often involvee laborious and troublesome sample preparation steps or require special instrumentation which in many laboratories is either non-existent or is fully occupieded with other analyses. If at all, single residue methods are often only performed when they are specifically required (e.g. when these pesticides are included in the monitoring list). The fact that this PT was organized at short notice (just two months before sending the samples) has also contributed to the low participation, as laboratories did not have time to establish the methods required.

In any case it is obvious, that appropriate measures should be taken to ensure that compounds currently not amenable to multiresidue methods are also satisfactorily monitored. Such measures may include the development and validation of simple-to-use, fast and cheap methodologies for such compounds.

6. SUGGESTIONS FOR FUTURE WORK

Proficiency tests for pesticide residues using single residue methods should continue to be performed in the future for various important pesticides and commodities. In future PTs the list of pesticides to be sought for should be distributed to the labs well in advance of the test, so that laboratories have time to include new pesticides in their scope.

In the 4th revision the Quality Control Procedures for Pesticide Residue Analysis (SANCO 10232/2006), labs are allowed to use a default expanded uncertainty value of 50% for results obtained with multiresidue methods (derived from the FFP RSD of 25% and using a 95% confidence level). The document, however, also provides for the use of lower expanded uncertainty values for results obtained using single residue methods (in particular if stable isotopically labelled internal standards are used), and if this is supported by interlaboratory reproducibility RSDs that are lower than 25%. The current results for chlormequat (where isotopically labelled internal standards were mainly used) showed an interlaboratory RSD (Qn-RSD) of 16%, which indicates that the setting of a lower default expanded uncertainty value would indeed be feasible. On the other hand, it should be taken into consideration that these results were generated by very experienced laboratories which are not necessarily truly representive of all official laboratories within the EU.. Additional proficiency tests with chlormequat and other compounds, as well as with other types of commodities, should be performed in the future in order to collect enough data to reinforce this conclusion.

7. REFERENCES

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8. ACKNOWLEDGEMENTS

The Organiser is grateful to the European Commission for funding this 1st Single Residue Method European Proficiency Test.

The Organiser wishes to thank the members of the Scientific Committee for their invaluable and knowledgeable advice.

The Organiser wishes to give a special thank-you to Almeria University.

APPENDIX 1. Homogeneity Data

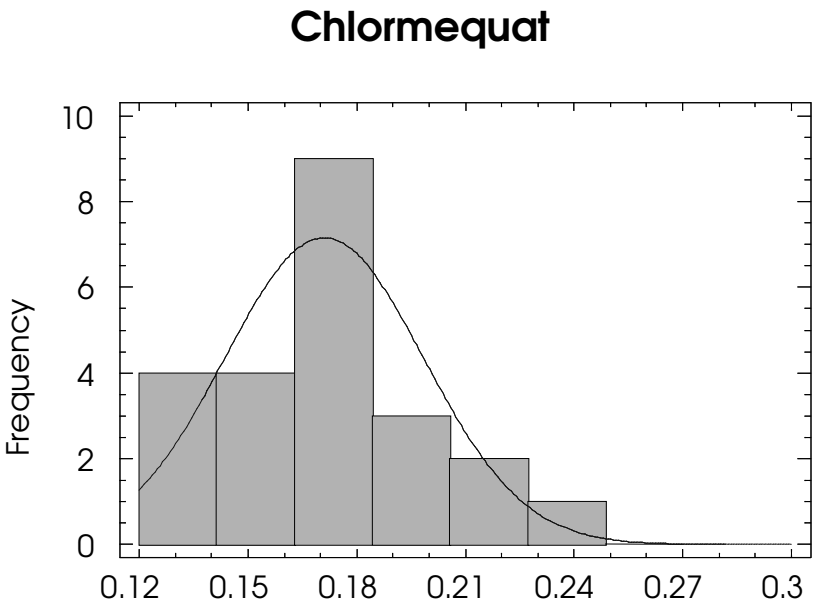
Chlormequat (mg/Kg)		
Sample	Portion 1	Portion 2
1	0.167	0.168
2	0.181	0.178
3	0.165	0.171
4	0.169	0.179
5	0.162	0.166
6	0.176	0.183
7	0.167	0.174
8	0.178	0.167
9	0.169	0.167
10	0.165	0.168

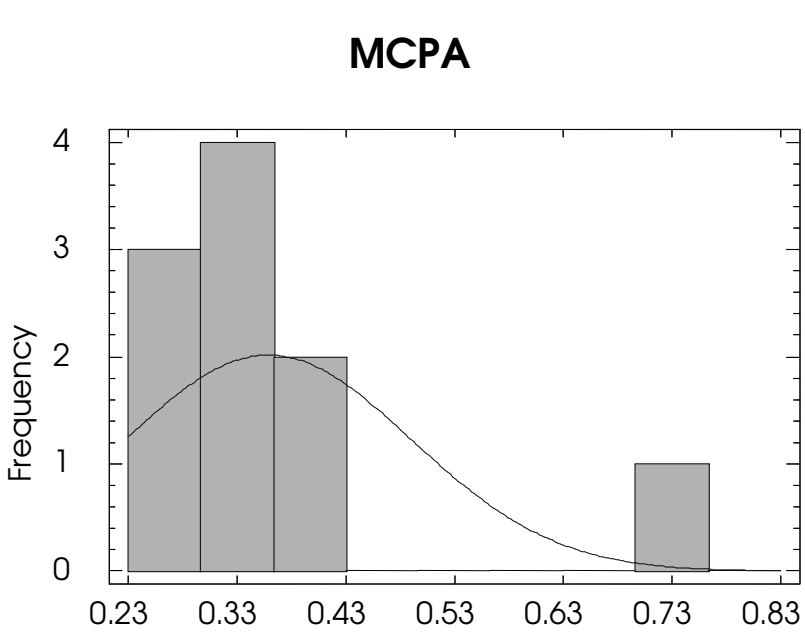
Fenbutatin Oxide (mg/Kg)		
Sample	Portion 1	Portion 2
1	0.500	0.500
2	0.481	0.498
3	0.500	0.485
4	0.492	0.479
5	0.483	0.475
6	0.499	0.500
7	0.494	0.477
8	0.504	0.477
9	0.481	0.485
10	0.492	0.499

MCPA (mg/Kg)		
Sample	Portion 1	Portion 2
1	0.390	0.358
2	0.361	0.364
3	0.372	0.387
4	0.364	0.336
5	0.387	0.379
6	0.369	0.346
7	0.365	0.387
8	0.344	0.381
9	0.392	0.358
10	0.360	0.363

APPENDIX 2. Histograms of residue data for each pesticide for all the laboratories.

Results presented as histograms:



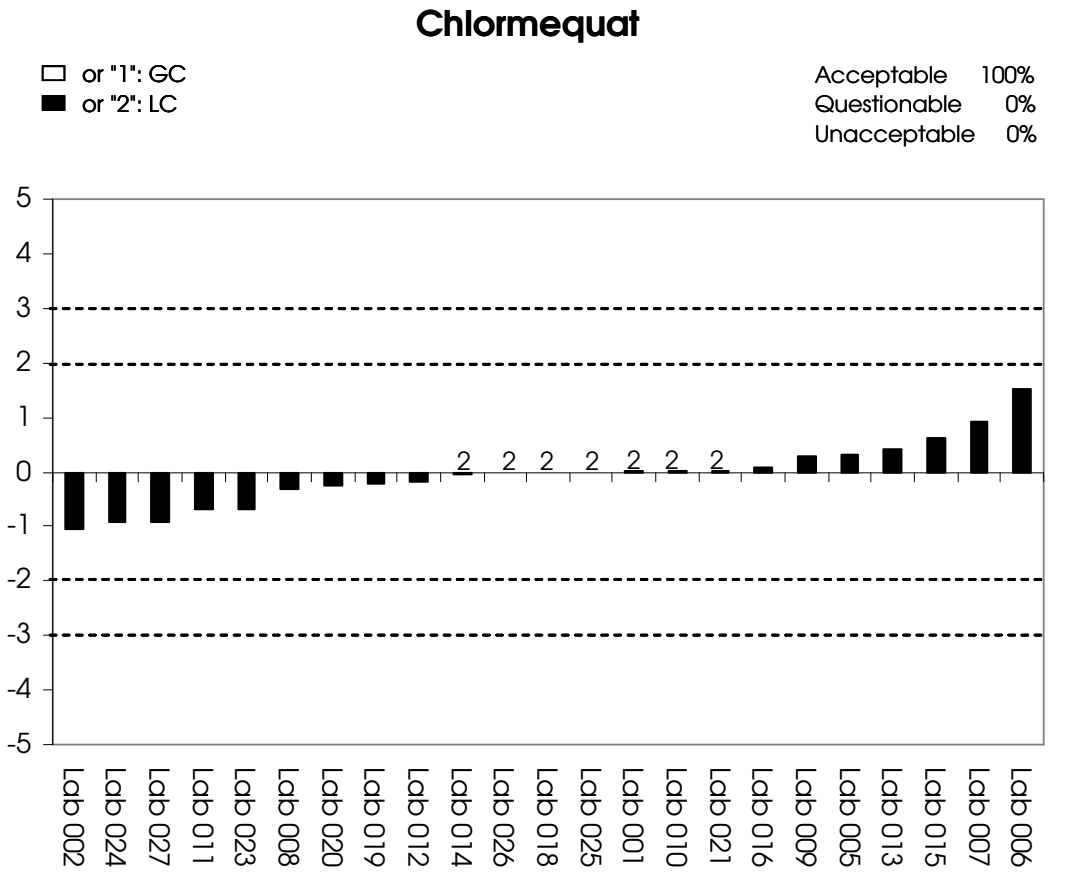


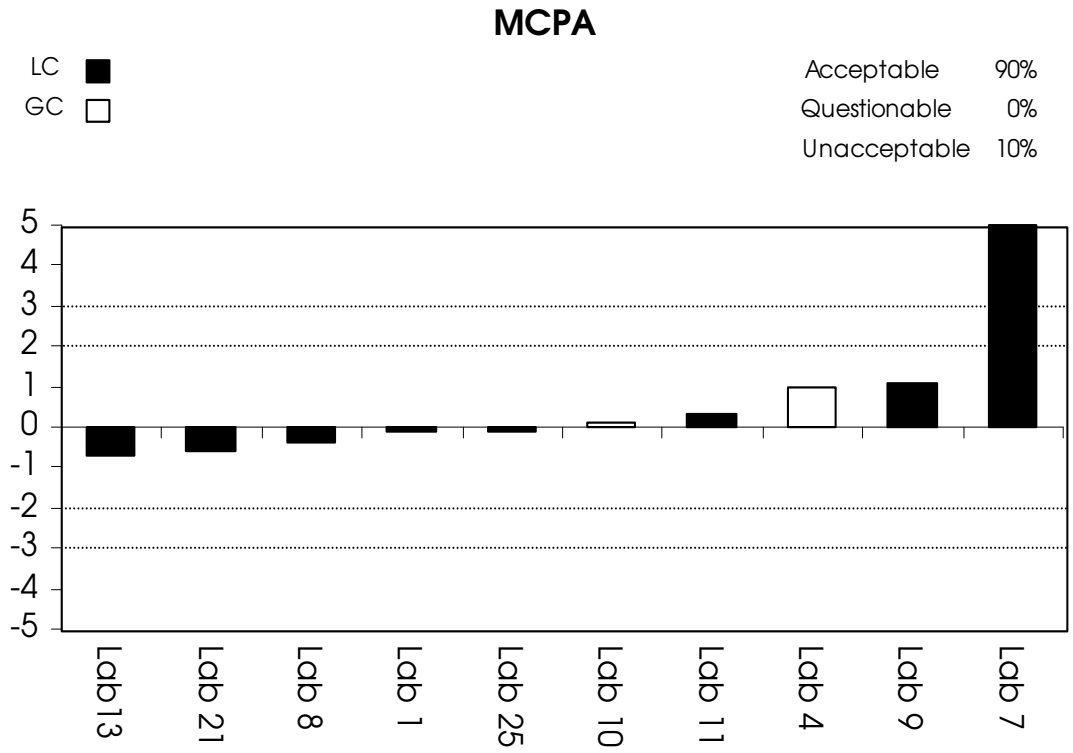
APPENDIX 3. Results (mg/kg) and z-scores for FFP RSD (25%)

Results given by the laboratories (mg/kg) and their calculated z-score value using FFP RSD 25%

Lab Code	Chlormequat	z-Score (FFP RSD 25%)	Fenbutatin oxide	No z-Score calculation	MCPA	z-Score (FFP RSD 25%)
MRPL (mg/kg)	0.05		0.05		0.05	
Median (mg/kg)	0.171		-		0.315	
Spiked Level (mg/kg)	0.156		0.490		0.361	
1	0.172	0.0	0.482		0.306	-0.1
2	0.126	-1.1	NA		NA	
3	No Results Reported					
4	NA		NA		0.390	1.0
5	0.185	0.3	NA		NA	
6	0.236	1.5	NA		NA	
7	0.210	0.9	NA		0.710	5.0
8	0.158	-0.3	0.464		0.286	-0.4
9	0.183	0.3	0.490		0.402	1.1
10	0.172	0.0	NA		0.324	0.1
11	0.141	-0.7	NA		0.337	0.3
12	0.163	-0.2	NA		NA	
13	0.189	0.4	0.400		0.257	-0.7
14	0.169	0.0	NA		NA	
15	0.197	0.6	NA		NA	
16	0.174	0.1	NA		NA	
17	No Results Reported					
18	0.171	0.0	NA		NA	
19	0.162	-0.2	0.610		NA	
20	0.160	-0.3	NA		NA	
21	0.172	0.0	NA		0.271	-0.6
22	No Results Reported					
23	0.142	-0.7	NA		NA	
24	0.131	-0.9	NA		NA	
25	0.171	0.0	NA		0.305	-0.1
26	0.170	0.0	NA		NA	
27	0.132	-0.9	NA		NA	

APPENDIX 4. Graphical Representation of z-scores for FFP RSD (25%).



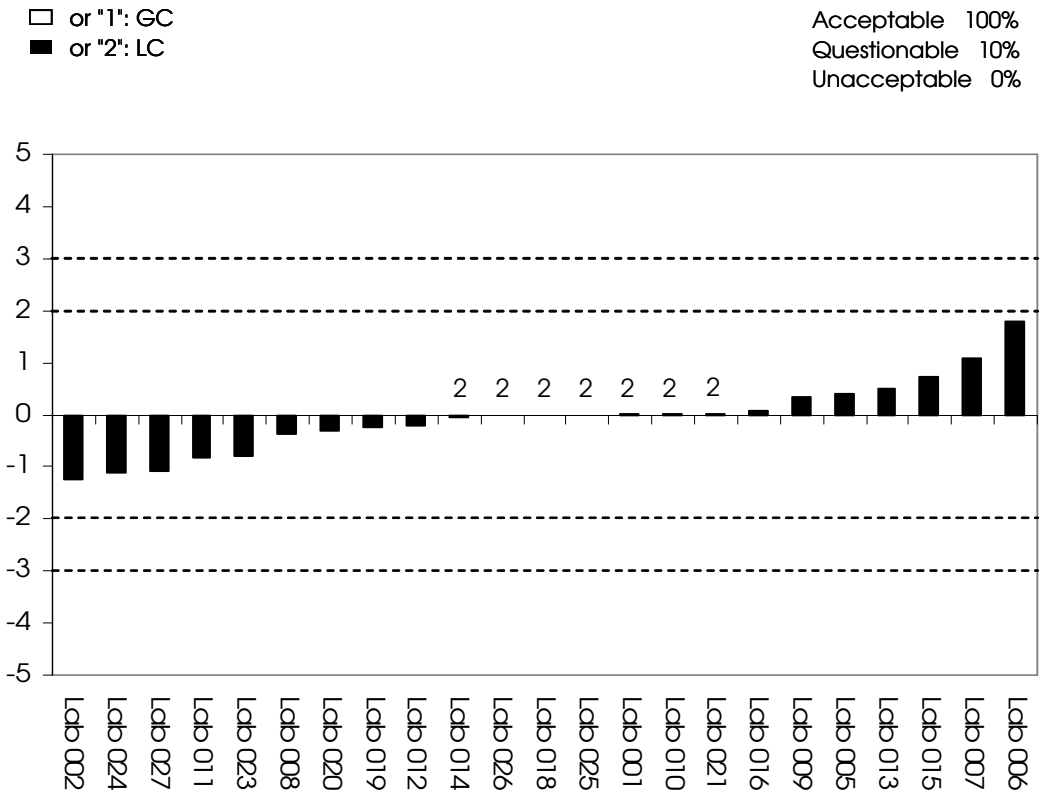


APPENDIX 5. Results (mg/kg) and z-scores using Horwitz Equation

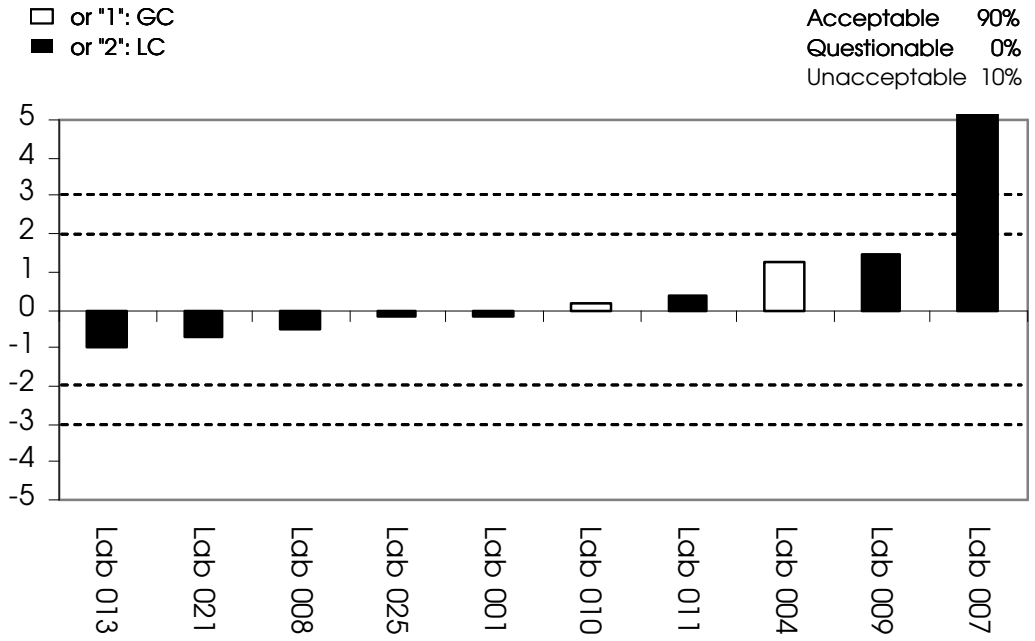
Results given by the laboratories (mg/kg) and their calculated z-score value using Horwitz Equation

Lab Code	Chlormequat	z-Score (21%)	Fenbutatin oxide	No z-Score calculation	MCPA	z-Score (19%)
MRPL (mg/kg)	0.05		0.05		0.05	
Median (mg/kg)	0.171		-		0.315	
Spiked Level (mg/kg)	0.156		0.490		0.361	
1	0.172	0.0	0.482		0.306	-0.2
2	0.126	-1.3	NA		NA	
3	No Results Reported					
4	NA		NA		0.390	1.3
5	0.185	0.4	NA		NA	
6	0.236	1.8	NA		NA	
7	0.210	1.1	NA		0.710	6.6
8	0.158	-0.4	0.464		0.286	-0.5
9	0.183	0.3	0.490		0.402	1.5
10	0.172	0.0	NA		0.324	0.2
11	0.141	-0.8	NA		0.337	0.4
12	0.163	-0.2	NA		NA	
13	0.189	0.5	0.400		0.257	-1.0
14	0.169	-0.1	NA		NA	
15	0.197	0.7	NA		NA	
16	0.174	0.1	NA		NA	
17	No Results Reported					
18	0.171	0.0	NA		NA	
19	0.162	-0.3	0.610		NA	
20	0.160	-0.3	NA		NA	
21	0.172	0.0	NA		0.271	-0.7
22	No Results Reported					
23	0.142	-0.8	NA		NA	
24	0.131	-1.1	NA		NA	
25	0.171	0.0	NA		0.305	-0.2
26	0.170	0.0	NA		NA	
27	0.132	-1.1	NA		NA	

Chlormequat (21%)



MCPA (19%)



APPENDIX 7. Methods used by participants for determining pesticides.

CHLORMEQUAT	Lab Code	1	2	3	4	5	6	7	8	9
	Quantification Using Standards in Solvent or in Matrix	M	SI	No Results Reported	NA	SI	S	M	MI	SI
	Confirmation Method	LC-MS/MS	LC-MS/MS			LC-MS/MS	LC-MS	LC-MS/MS	LC-MS/MS	LC-MS/MS
	RL(mg/kg)	0.05	0.01			0.03	0.02	0.05	0.01	0.005
	Recovery (%)						0.61		106	97
	Sample Weight (g)	15	20			20	5	10	10	20
	Extraction Solvent	6	6			Methanol	Water-methanol 50/50	Methanol	6	6
	Clean-Up Step									
	Derivatization Step									
	Internal standard		D_4			D_4		D_4	D_4	D_4
	Injection Volume (µl)	40	100			20	10	50	10	5
	Injection Type						standard			
	Determination	LC-MS/MS	LC-MS/MS			LC-MS/MS	LC-MS	LC-MS/MS	LC-MS/MS	LC-MS/MS (ESI +)
	Reference Method	Dilution and direct injection	CEN/TC 275 EN 15054 (2005)			ASU L 00.00-76 v.December 2002	NF EN 15054	CEN prEN 15055:2006:E	Method by Lutz Alder	DIN EN 15055:2006

APPENDIX 7. Methods used by participants for determining pesticides.

CHLORMEQUAT	Lab Code	10	11	12	13	14	15	16	17	18
	Quantification Using Standards in Solvent or in Matrix	M	SI	S	M	MI	M	S	No Results Reported	SI
	Confirmation Method	LC-MS	LC-MS	LC-MS/MS	LC/MS/MS	LC-MS/MS	LC-MS/MS	LC-MS/MS		LC-MS
	RL(mg/kg)	0.05	0.01	0.01	0.05	0.02	0.01	0.015		0.02
	Recovery (%)	118	99	102		127		110		
	Sample Weight (g)	20	10	20	8	20	20	50		10
	Extraction Solvent	6	Acetone/ water	Methanol/ Water	6	6	6	6		Methanol
	Clean-Up Step		LL							
	Derivatization Step									
	Internal standard		D_4	D_4		D_4		D_4		D_4
	Injection Volume (µl)	10	10	20	20	40	5	10		100
	Injection Type	Loop								
	Determination	LC-MS	LC-MS	LC-MS/MS	LC/MS/MS	LC-MS/MS	LC-MS/MS	LC-MS/MS		LC-MS
	Reference Method	prEN15054/ Internal Method M39	Juhler, Vahl, J. AOAC Int. 82 (2), 331 ff, (1999), modified	ASU § 64 LFGB L 00.00-76		In house	In house	Methodensammlung § 64 LFGB (ehem. § 35 LMBG) L 00.00-76		§ 35 LMBG, §64 LFGB, 00.00-75

APPENDIX 7. Methods used by participants for determining pesticides.

CHLORMEQUAT	Lab Code	19	20	21	22	23	24	25	26	27
	Quantification Using Standards in Solvent or in Matrix	SI	M	SI	No Results Reported	SI	MI	M	MI	M
	Confirmation Method	LC-MS/MS	LC-MS/MS	LC-MS/MS		LC-MS-MS	LC-MS/MS	LC-MS/MS	LC-MS	LC-MS/MS
	RL(mg/kg)	0.01	0.01	0.01		0.005	0.05	0.02	0.05	0.004
	Recovery (%)	105	107	103			90	91	85	95.3
	Sample Weight (g)	10	10	25		20	5	5	0.5	6.3
	Extraction Solvent	Methanol/ Water	Methanol/ water/ acetic acid (75:24:1 v/v)	6		6	Water/ Methanol		6 (diluted, 1:1)	6
	Clean-Up Step		SPE (C18)							
	Derivatization Step									
	Internal standard	D_4	^{13}C-chlormequat-chlorid	D_4		D_4	yes		D_4	
	Injection Volume (µl)	15	20	10		5	10	50	10	10
	Injection Type			Loop		Sample loop	Loop	Partial	Partial loop filling	
	Determination	LC-MS/MS	LC-MS/MS	LC-MS/MS		LC-MS/MS	LC-MS/MS	LC-MS/MS	LC-MS (ITD)	LC-MS/MS
	Reference Method	SLV M030		prEN 15054		Method § 64 LFBG L 00.00-76	In house	In house	CEN Method	KM 36

APPENDIX 7. Methods used by participants for determining pesticides.

FENBUTATIN OXIDE	Lab Code	1	2	3	4	5	6	7	8	9	10	11	12
	Quantification Using Standards in Solvent or in Matrix	M		No Results Reported					M	M			
	Confirmation Method	LC-MS/MS							LC-MS/MS	LC-MS/MS			
	RL(mg/kg)	0.05							0.01	0.01			
	Recovery (%)								103	96			
	Sample Weight (g)	15							10	10			
	Extraction Solvent	6							5	5			
	Clean-Up Step								O				
	Derivatization Step												
	Internal standard									Triphenyl phosphate			
	Injection Volume (µl)	40							10	9			
	Injection Type												
	Determination	LC-MS/MS							LC-MS/MS	LC-MS/MS (ESI +)			
	Reference Method	Dilution and direct injection							QuEChERS Method	M. Anastassiades, S.J. Lehotay, D. Stajnbaher and F.J. Schenck, J AOAC Int 86 (2003) 412; QuEChERS A Mini-Multiresidue Method for the Analysis of Pesticide Residues in Low-Fat Products, www.quechers.com			

APPENDIX 7. Methods used by participants for determining pesticides.

FENBUTATIN OXIDE														
Lab Code	**Quantification Using Standards in Solvent or in Matrix**	**Confirmation Method**	**RL(mg/kg)**	**Recovery (%)**	**Sample Weight (g)**	**Extraction Solvent**	**Clean-Up Step**	**Derivatization Step**	**Internal standard**	**Injection Volume (µl)**	**Injection Type**	**Determination**	**Reference Method**
13	M	LC/MS/MS	0.05		8	5				20		LC/MS/MS	
14	NA												
15	NA												
16	NA												
17	No Results Reported												
18	NA												
19	S	GC-FPD	0.05	119	25	Isooctane/ Acetic acid/ Water	O	MeMgCl		2	Splitless	GC-FPD	SLV M021
20	NA												
21	NA												
22	No Results Reported												
23	NA												
24	NA												
25	NA												
26	NA												
27	NA												

APPENDIX 7. Methods used by participants for determining pesticides.

MCPA									
Lab Code	1	2	3	4	5	6	7	8	9
Quantification Using Standards in Solvent or in Matrix	M			M			M	M	M
Confirmation Method	LC-MS/MS			GC-MS-der.			LC-MS/MS	LC-MS/MS	LC-MS/MS
RL(mg/kg)	0.05			0.02			0.05	0.05	0.01
Recovery (%)				87				91	101
Sample Weight (g)	15			5			10	10	10
Extraction Solvent	6			SPE/ acetone elution			Acetonitrile	5	5
Clean-Up Step								O	
Derivatization Step		NA	No Results Reported	diazomethane	NA	NA			
Internal standard				2,4-DP-D6					Bis-nitrophenyl urea (nicarbazin)
Injection Volume (µl)	40			2			50	10	5
Injection Type				splitless					
Determination	LC-MS/MS			GC-MS single quad			LC-MS/MS	LC-MS/MS	LC-MS/MS (ESI -)
Reference Method	Dilution and direct injection			in-house method			Analysis of Acidic Pesticides using the QuEChERS Method and LC-MS/MS	QuEChERS Method	M. Anastassiades, S.J. Lehotay, D. Stajnbaher and F.J. Schenck, J AOAC Int 86 (2003) 412; QuEChERS- A Mini-Multiresidue Method for the Analysis of Pesticide Residues in Low-Fat Products, www.quechers.com

APPENDIX 7. Methods used by participants for determining pesticides.

MCPA	Lab Code	10	11	12	13	14	15	16	17	18	19	20	21
	Quantification Using Standards in Solvent or in Matrix	S with fenoprop as intern. stand.	M		M								M
	Confirmation Method	GC-MS after derivat.	LC-MS/MS		LC/MS/MS								LC-MS/MS
	RL(mg/kg)	0.01	0.01		0.05								0.05
	Recovery (%)	99	97										85
	Sample Weight (g)	20	10		8								10
	Extraction Solvent	Dichloromethane	5		5								5
	Clean-Up Step	O: pH adjustment	DSPE										
	Derivatization Step	Pentafluorobenzyl bromide		NA		NA	NA	NA	No Results Reported	NA	NA	NA	
	Internal standard	Fenoprop	Nicarbazin										
	Injection Volume (µl)	2	10		20								10
	Injection Type	Splitless											Loop
	Determination	GC-MS	LC-MS/MS		LC/MS/MS								LC-MS/MS
	Reference Method	Internal method M19	M. Anastassiades et al., J. AOAC Int. 86 (2), 412-431, (2003), modified (QuEChERS)										QuEChERS

APPENDIX 7. Methods used by participants for determining pesticides.

MCPA													
Lab Code	Quantification Using Standards in Solvent or in Matrix	Confirmation Method	RL(mg/kg)	Recovery (%)	Sample Weight (g)	Extraction Solvent	Clean-Up Step	Derivatization Step	Internal standard	Injection Volume (µl)	Injection Type	Determination	Reference Method
22	No Results Reported												
23	NA												
24	NA												
25	M	LC-MS/MS	0.02	98	5					20	Partial	LC-MS/MS	In house
26	NA												
27	NA												

Protocol

Introduction

Only laboratories that are involved in providing residue data for their national monitoring programmes, and/or the EU co-ordinated monitoring programme are invited to participate in this 1st European Proficiency Test for Pesticide Residue Analysis using Single Residue Methods.

To participate, each laboratory will have to send by e-mail the Application Form to the Organiser. The laboratories will then receive confirmation of acceptance of their participation by e-mail together with a Laboratory Code, which must always be used in communications with the Organiser. Any e-mail without this code will not be answered. The Laboratory Code is confidential and will only be known by the participant, the Organiser, and the Commission. In the Final Report there will not be any correlation between the code and the laboratory name. However, some results may need to be presented on a country basis to the Standing Committee on the Food Chain and Animal Health, and a link between codes and laboratories may become indirectly possible, especially if there are only a few laboratories in one country.

Each participant will receive 3 Laboratory Reporting Forms (1-3) which must be filled-in by the participant and returned to the Organiser. These forms concern:

- Form 1: Confirmation of Sample Receipt and Sample Acceptance
- Form 2: Submission of Results
- Form 3: Submission of Method Parameters

Please ensure that you strictly adhere to the deadlines shown in the EUPT-SRM01 Schedule. On receipt of each form, the Organiser will respond with a confirmatory e-mail.

The Pesticide List includes all the possible pesticides that could be present in the test material and that should be targeted by the participating labs. MRPL values (minimum required performance levels) for each pesticide are also given. These values are the levels that the laboratories should be able to attain.

Payment: Only laboratories that have paid the transport costs will receive the test materials. If laboratories need more time to pay, they must send by fax a justification to verify that the payment procedure has started. Invoices for the sample transporting will be sent to the laboratories upon request (see Application Form).

ANNEX 1. Protocol and Instructions. List of pesticides to be sought.

The official language used in this Proficiency Test will be English.

The interchange of results between participating laboratories during the test is not allowed!

General Characteristics

Objectives

The objective of this proficiency test is to obtain information about the quality, accuracy and comparability of the pesticide residue data sent to the European Commission within the framework of the EU and national pesticide monitoring programmes. Participating laboratories will be provided with an assessment of their own analytical performance and the reliability of their data compared to other laboratories.

Steps to Follow

The Proficiency Test is made up of the following 8 steps that are essential for the generation of satisfactory results:

1. Invitation to the participating laboratories and distribution of the Application Form, Pesticide List and Protocol.
2. Preparation of the test materials. Homogeneity and stability testing performed by the Organiser.
3. Confirmation of the receipt of the participants Application Form and communication of the Laboratory Codes and the Laboratory Reporting Forms 1-3.
4. Payment in advance for the shipment of the test materials, or receipt of a fax demonstrating that the payment procedure has started.
5. Shipment of the test material, together with the blank.
6. The participant laboratories will be responsible for reporting their data to the Organiser using the Laboratory Reporting Forms supplied, by the stipulated deadlines.
7. The Organiser will evaluate the results at the end of the proficiency test, once the deadline for receipt of results has passed.
8. The Organiser will send a copy of the Final Report to each participant laboratory. This report will include information regarding the design of the test, the homogeneity and stability test results, a record of the shipped samples, a statistical evaluation of the participant's results, graphical displays of the results and conclusions. Any other relevant information considered of value will also be included.

Background Information

This proficiency test concerns pesticide residues analysis in apple juice.

The test material will be frozen (using liquid nitrogen), chopped, homogenized and sub-sampled into polyethylene bottles that have previously been coded.

Ten of these bottles, containing the test material, will be chosen randomly and analysed by an independent laboratory to check for homogeneity.

The test material will be stored frozen (–20ºC) prior to shipment to participants.

Two bottles, again chosen randomly, will be analysed over a period of time to confirm the stability of the pesticides in the test material (firstly when the test materials are shipped, and then a few days after the deadline for receipt of results from the participants). These results will not be included in the statistical analysis of the proficiency test.

The aim is only to check the stability during the shipping process and the proficiency test.

Schedule

The following table shows the program for this EUPT-SRM 01

Activity	Data/Deadline	Contact
- Deadline for receiving Application Form from invited laboratories	15th of July 2006	pmedina@ual.es
- Sample Treatment, Homogenisation,. - Storage/Stability Test	July/August/September 2006	
- Deadline for the Payment of Shipping Costs and/or demonstration that payment procedure has been initiated	1st of September 2006	Fax: # 34 950015645 or pmedina@ual.es
- Sample Distribution.	4th of September 2006	
- Deadline for receiving Laboratory Reporting Form 1	6th of September 2006	pmedina@ual.es
- Deadline for receiving results and method parameters: Laboratory Reporting Forms 2 and 3	25th of September 2006	pmedina@ual.es
- Final Report	14th of October 2006	

Participating Laboratories

It is up to the contact points/authorities/organisations responsible for the official monitoring of pesticide residues in each country to select the laboratories that should participate, although it is a requirement that a laboratory must be active in contributing results to the national monitoring

programme and/or the EU co-ordinated programme. It is up to the participants to fill in and return the Application Form so the Organiser has all their details before the deadline.

Amount of Sample

Approximately 400g of apple juice test material will be shipped together with 400g of 'blank' surrounded with dry ice and packed in boxes. The courier costs are charged and must be paid by the participants before shipment of the samples. There will only be a limited amount of test material and laboratories should not ask for more than they require to perform the analysis.

Application Form

The participating laboratories must complete the application form and return it by e-mail to the Organiser. In the Application From you should also fill in information that is essential for us to prepare the official invoice. The Application Form must be sent to the Organiser by **15th July 2006**, at the latest.

Shipping of Samples

The shipment of the test materials will be carried on the 4th of September. A Reminder-Message will be sent out a week before shipment, and laboratories must make arrangements for the reception of the test materials and their proper storage until analysis can commence. The Participants should let the Organiser know of any possible public holidays in their country/city during the delivery time mentioned in the calendar and make every effort to receive the shipment even if the laboratory is closed.

Sample Manipulation Advises

Once received, the test material should be stored frozen until it is to be analysed.

Be sure to mix the contents of the bottle thoroughly, to ensure homogeneity of the test material, before taking the analytical portion(s).

ANNEX 1. Protocol and Instructions. List of pesticides to be sought.

Contact Information

Please do not hesitate to contact the Organisers if you need any clarification!

The official postal addresses, phone numbers, fax numbers and e-mail-addresses of the Organisers are as follows:

CRL for Pesticide Residue Analysis using Single Residue Methods:

CVUA Stuttgart
Schaflandstrasse 3/2
D-70736 Fellbach - Germany
Phone Numbers: #49-711-3426-1124 or -1128 or -1141
Fax Number: #49-711-588176
Email: Michelangelo.Anastassiades@cvuas.bwl.de

CRL for Pesticide Residue Analysis in Fruits and Vegetables:

Universidad de Almería
Edificio Químicas CITE I
Ctra. Sacramento s/n
04120 Almería - Spain
Phone Numbers: #34 950015034 or # -645
Fax Number: #34 950015645
E-mail: pmedina@ual.es
THIS IS THE ADDRESS WHERE ALL THE FORMS MUST BE SENT

Web-Site

All documents as well as the latest information can be found at our web-site, the address of which will be communicated soon.

Laboratory Reporting Form 1 (Sample Receipt and Acceptance)

Once the laboratory has received the test materials they must complete Form 1, filling-in the date of receipt, the condition of the test material, and its acceptance and send it to the Organiser via e-mail (pmedina@ual.es). The deadline for returning the completed Form 1 to the Organiser is the 6th of September 2006. If the laboratory does not respond before this deadline the Organiser will assume that the laboratory has received and accepted the test material.

Important Note: Do not forget to fill-in the laboratory code assigned to you on this form.

Laboratory Reporting Form 2 (Results)

General Remarks

The test material contains a certain number of pesticides from the Pesticide List. Please read carefully the list in Form 2 since the residue definitions are not given (see the Pesticide List).

It should not be assumed that only pesticides registered for use on apple juice will be present.

The analytical procedures used for each pesticide sought must also be reported in Form 2 in a coded form.

Form 2 must be sent to the Organiser by 25th of September 2006, at the latest. Results received after this date will not be included in the statistical treatment, or in the final report. The laboratories are responsible for reporting their results to the Organiser. The Organiser will acknowledge receipt of the results by e-mail.

Important Note: Do not forget to fill-in the laboratory code assigned to you on this form.

Reporting of Results

Each laboratory must report only one result for each of the pesticides present in the test material, using their normal routine analytical procedure(s). More than one method may be used to cover all the compounds targeted. The results (concentration levels of the pesticides detected) should be, expressed in mg/kg, and must be accompanied by the laboratories reporting level (RL) for each pesticide. This level will only serve for information and documentation purposes.

Significant Figures:

- Concentrations <0.100 mg/kg, to be expressed to two significant figures (three decimals places, i.e. 0.058 mg/kg).
- Concentrations > 0.100 mg/kg, to be expressed to three significant figures, i.e. 0.156, 1.64, 10.3 mg/kg.

In cases where a pesticide was not detected, it should be recorded as <RL (smaller than the reporting limit of the laboratory); if a residue was found then add the concentration of it. The results/concentrations must be reported as numbers. Any other form of data will not be considered. In the column "Scope of your Method" fill-in A if the pesticide was sought, and NA if this was not the case.

Correction of Results for Recovery

Where stable-isotopically labelled internal standards are used, correction of recovery is inherent to the procedure and thus allowed. Otherwise results are not to be corrected using recovery correction factors. If such factors are usually employed by the laboratory, they should provide them to the Organiser as informative data only. This information must be sent together with the results in Form 2.

Laboratory Reporting Form 3 (Analytical Procedures Used)

A brief summary of the analytical procedure(s) used is required from each laboratory on Form 3.

If more than one method has been used, please label them with different letters or codes in Form 2, and use as many copies of Form 3 as are needed (one for each method).

The Organiser must receive Form 3 by mail by 25th of September 2006, at the latest.

Important Note: Do not forget to fill-in the laboratory code assigned to you on this form.

Evaluation of the Results

The statistics used for the treatment and assessment of the data will be described in detail in the Final Report. A short summary of how the results will be treated is given below.

– ***False Positives***

These are the results that show the presence of pesticides which are listed in the pesticide list and which are (i) not used in the sample treatment, (ii) and not detected by the Organiser even in a repeated analysis. However, if a number of laboratories detect the same additional pesticide, or if the concentration is close to the MPRL, then a decision as to whether or not this should be considered to be a false positive result will be made on a case-by-case basis.

Any results reported that are lower than 0.01mg/Kg will not be considered as false positives.

– ***False Negatives***

These are results for pesticides that were not reported by the laboratories although they were used by the Organiser to treat the test material and are detected at, or above, the MRPL.

– ***Establishing the true concentration (μ)***

The true concentration in all cases will be determined by the median of all the results. The median value for every pesticide present will thus be generated.

– ***Establishing the assigned value for the standard deviation***

The assigned value for the standard deviation (δ) will be fixed by the Organiser.

$$\text{Where } \delta = b_i * \mu_i \qquad \text{being } b_i = \%FFP/100\%$$

An assigned value will be established based on the Fit-For-Purpose (FFP) Standard Deviation model. An average fixed value of 25% has already been chosen. However, the Organiser may increase this value for certain difficult pesticide-crop-concentration combinations, after

consultation with the committee of experts, and based on experience gained from previous Proficiency Tests.

– ***z-Scores***

This parameter is calculated using this formula:

$$z_i = (x_i - \mu_i) / \delta_i$$

Where x_i is the value reported by the laboratories, μ_i the assigned value and δ_i the standard deviation at that level, for each pesticide ($_i$).

Any z-score values of /z/ > 5 will be reported as '+5', or '-5'.

z-Score values will be interpreted in the following way:

$|z| \leq 2$ Acceptable

$2 < |z| \leq 3$ Questionable

$|z| > 3$ Unacceptable

For the values considered to be false negative results, z-scores will be calculated using the MPRL values as the value for x_i.

However, a z-score will not be assigned to any false positive results.

The Organiser will consider whether, or not, these values should appear in the histograms.

ANNEX 1. Protocol and Instructions. List of pesticides to be sought.

Form 1

Laboratory Code: *EUPT-SRM 01-Lab-*____

Date of receipt: _____/_____/*2006*

Test material codes *(Check the bottles of the sample and the 'blank')*:

EUPT-SRM 01-blank:____

EUPT-SRM 01-sample:___

Loses: YES____ NO____

Frozen: YES____ NO____

X I accept the test material. I do not need more.

Please, fill in this form and send it back by e-mail (pmedina@ual.es) as soon as you have received the test material but not later than on the 6th of September 2006.

Signature (only if sent by fax or ordinary mail):

If no form is received by the Organiser, it will be assumed that the test material has been accepted by the laboratory.

ANNEX 1. Protocol and Instructions. List of pesticides to be sought.

Form 2 (Results)

Laboratory Code:_____ Date:_____

Test material Code: _____ Blank code: _____

Pesticide	Scope of your Method (1)	Analytical Procedure (2)	Conc. (mg/kg) (3)	Quantification Using Standards in Solvents or Matrix (4)	Confirmation Method (5)	RL (mg/kg) (6)	Recovery (7)
2,4-D							
Dichloroprop							
2-Naphthoxyacetic acid							
4-CPA							
Chlormequat							
Cyhexatin							
Dicamba							
Fenbutatin oxide							
Fentin							
Fluazifop							
MCPA							
Mecoprop							
Mepiquat							
Quizalofop							

(1) If the pesticide is not included in your analysis, fill NA. If the pesticide is detected, fill D. If the pesticide is NOT detected, fill ND.
(2) Write the same code as you use in Form 3 for the analytical method used, e.g. A, B, C…
(3) Concentration, report only one result. Record the concentrations for all pesticides according to the residue definition given in the Pesticide List.
(4) Standards: S = standard/calibration in pure solvent; M = standard/calibration in matrix extract; SI = standard/calibration in pure solvent using an isotopically labelled target compound as Internal Standard; MI = standard/calibration in matrix extract using an isotopically labelled target compound as Internal Standard.

(5) Give the instrumental analysis technique used for confirmation e.g. GC-FPD, HPLC-UV, GC-MS, GC-MS after derivatization, LC-MS, LC-MS/MS.

(6) RL Your Reporting Level, must be given for all pesticides. For pesticides with metabolites/degradation products included in the MRL definition, give the "Reporting Level" for the global compound (see residue definition in the pesticide list).
(7) The concentration/results reported in (3) must not be corrected using recovery factors even if the laboratory usually corrects them. Nevertheless, you may give the correction factor for each pesticide as informative data.

I agree to be responsible for completing and returning this form to the Organiser on the 20th of September at the latest. In case of no e-mail confirmation of reception of this document (in 3 or 4 days), I will contact the Organiser as soon as possible.

Signature (only if fax or ordinary mail is used):

Laboratories should fill in this form and send it to the following e-mail: pmedina@ual.es

Form 3 (Analytical Procedures Used)

Laboratory Code:_____ Date:_____

Complete one of these forms for every different analytical procedure used

Analytical Procedure (1):_____

Sample Weight (g):_____

Extraction solvent/s (2):_____

Cleanup step (3):_____

Derivatization step (in any) (4):_____

Internal standard (if any):_____

Injection Volume: _____ Injection Type: _____

Determination Technique (5): _____

Reference Method (Obligatory):_____
Signature (only if the form is send by Fax or ordinary mail):

[X] I agree to be responsible for delivering this form to the Organiser. In case of no e-mail confirmation of receipt of this form (in 3 or 4 days), I will contact the Organiser as soon as possible.

Please return this Form not later than the 25th of September 2006

(1) Write the same code as you use in Form 2 for the analytical method used, e.g. A, B, C…

(2) Denoted as 1 = ethyl acetate, 2 = acetone followed by cyclohexane and ethyl acetone, 3 = acetone followed by dichloromethane, 4 = acetone followed by dichloromethane and petroleum ether, 5 = acetonitrile, 6 = methanol, 7 = other (specify which).

(3) Clean-up: GPC = gel permeation chromatography, SPE = solid phase extraction, LL = liquid-liquid partition, NO = no clean-up, O = other clean-up method
(4) Derivatization step: e.g. Pentafluorobenzylbromide/Na_2CO_3

(5) Determination Technique: e.g. GC-ECD, GC-NPD, GC-FPD, GC-MS (single-quad), GC-ITD, HPLC-FL, HPLC-UV, HPLC-DAD, LC-MS, LC-MS/MS (specify the one used for each pesticide determination)

Laboratories should fill-in this form and send it to the following e-mail: pmedina@ual.es

ANNEX 1. Protocol and Instructions. List of pesticides to be sought.

PESTICIDES LIST FOR THE EUPT-SRM 01

Pesticide	MRPL (mg/Kg)
2,4-D (2,4-Dichlorophenoxy acetic acid) (free acid)	0.05
Dichloroprop (2,4-DP, 2,4- Dichlorophenoxy propionic acid) (free acid, including Dichlorprop-P)	0.05
2-Naphthoxyacetic acid	0.05#
4-CPA (4-Chlorophenoxy acetic acid)	0.05#
Azocyclotin (see Cyhexatin)	See cyhexatin
Chlormequat (expressed as Chlormequat cation)	0.05
Cyhexatin (Azocyclotin + Cyhexatin, expressed as Cyhexatin)	0.05
Dicamba (Free acid)	0.05#
Fenbutatin oxide	0.05
Fentin (Fentin hydroxide+ Fentin acetate, expressed as Fentin)	0.05
Fluazifop (free acid, Fluazifop + Fluazifop-P)	0.05#
MCPA (Free acid)	0.05#
Mecoprop (MCPP) (free acid, Mecoprop + Mecoprop-P)	0.05
Mepiquat (expressed as Mepiquat cation)	0.05#
Quizalofop (free acid, Quizalofop + Quizalofop-P)	0.05#

MRPLs were chosen to be the lowest EU harmonized MRLs (disregarding baby food directive). For non-harmonized compounds the MRPL was chosen by the Organisers and the figure is followed by #

ANNEX 2. List of laboratories invited to participate in SRM 01.

COUNTRY	CITY	LABORATORY NAME	REPORTED RESULTS
AUSTRIA	VIENNA	AUSTRIAN AGENCY FOR HEALTH AND FOOD SAFETY, COMPETENCE CENTRE RESIDUE ANALYSIS VIENNA	YES
AUSTRIA	INNSBRUCK	AUSTRIAN AGENCY FOR FOOD AND HEALTH SAFETY (AGES) ANALYTICAL COMPETENCE FOR PLANT PROTECTION PRODUCTS	YES
CZECH REPUBLIC	PRAGUE	CZECH AGRICULTURE AND FOOD INSPECTORATE	YES
CZECH REPUBLIC	PRAGUE	INSTITUTE OF CHEMICAL TECHNOLOGY, PRAGUE DEPARTMENT OF FOOD CHEMISTRY AND ANALYSIS	YES
DENMARK	RINGSTED	DANISH FOOD AND VETERINARY ADMINISTRATION, REGIONAL PESTICIDE LABORATORY	NO
DENMARK	SOEBORG	DANISH INSTITUTE FOR FOOD AND VETERINARY RESEARCH	YES
FINLAND	ESPOO	FINNISH CUSTOMS LABORATORY	YES
FRANCE	RENNES	LABORATOIRE DGCCRF-RENNES	NO
FRANCE	PESSAC	DGCCRF LABORATOIRE DE BORDEAUX	YES
GERMANY	TRIER	LANDESUNTERSUCHUNGSAMT-INSTITUT FÜR LEBENSMITTELCHEMIE TRIER	YES
GERMANY	ERLANGEN	BAYERISCHES LANDESAMT FÜR GESUNDHEIT UND LEBENSMITTELSICHERHEIT	YES
GERMANY	OLDENBURG	LAVES LEBENSMITTELINSTITUT OLDENBURG	YES
GERMANY	HAGEN	CHEMISCHES UNTERSUCHUNGSAMT DER STADT HAGEN	YES
GERMANY	MÜNSTER	CHEMISCHES LANDES- UND STAATLICHES VETERINÄRUNTERSUCHUNGSAMT	YES
GERMANY	BERLIN	BBGES-ILAT, FB 26	YES
GERMANY	SPEYER	LANDESUNTERSUCHUNGSAMT, INSTITUT FUR LEBENSMITTELCHEMIE SPEYER	YES

ANNEX 2. List of laboratories invited to participate in SRM 01.

COUNTRY	CITY	LABORATORY NAME	REPORTED RESULTS
GERMANY	ROSTOCK	LANDESAMT FÜR LANDWIRTSCHAFT, LEBENSMITTEL SICHERHEIT UND FISCHEREI MECKLENBURG-VORPOMMERN	YES
ITALY	RAGUSA	AUSL N7 RAGUSA DAP RAGUSA ARPA SICILIA L.I.P. SEZIONE CHIMICA	YES
LATVIA	RIGA	NATIONAL DIAGNOSTIC CENTRE	YES
NORWAY	AAS	NORWEGIAN INSTITUTE FOR AGRICULTURAL AND ENVIRONMENTAL RESEARCH, BIOFORSK LABORATORY	YES
SLOVAKIA	BRATISLAVA	STATE VETERINARY AND FOOD INSTITUTE BRATISLAVA	NO
SLOVENIA	MARIBOR	PUBLIC HEALTH INSTITUTE, ENVIRONMENTAL PROTECTION INSTITUTE	YES
SPAIN	BURJASSOT (VALENCIA)	AGROALIMENTARIO GENERALITAT VALENCIANA	YES
SWEDEN	LIDKÖPING	ANALYCEN NORDIC AB	YES
THE NETHERLANDS	AMSTERDAM	VWA-FOOD AND CONSUMER PRODUCT SAFETY AUTHORITY	YES
UNITED KINGDOM	TEDDINGTON, MIDDLESEX	LABORATORY OF THE GOVERNMENT CHEMIST LIMITED	YES
UNITED KINGDOM	YORK	CENTRAL SCIENCE LABORATORY	YES