EU PROFICIENCY TEST
for the Analysis of Pesticide Residues in Carrots using Single Residue Methods (EUPT-SRM3)
2008

Final Report

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QCG: Quality Control Group
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CONTENTS

3rd CRL-EUROPEAN COMMISSION PROFICIENCY TEST FOR THE DETERMININATION OF PESTICIDES USING SINGLE-RESIDUE METHODS (EUPT-SRM3/2008)

FOREWORD

Regulation 882/2004/EC[1,2] lays down the general tasks and duties of Community Reference Laboratories (CRLs) for Food, Feed and Animal Health. Among these tasks is the organisation of comparative tests, which are performed on an annual basis.

According to Article 28 of Regulation 396/2005/EC on maximum residue levels of pesticides in or on food and feed of plant and animal origin[3], all laboratories analysing samples for the official controls on pesticide residues shall participate in the European Community Proficiency Tests (EUPTs) for pesticide residues facilitated by the Commission. These proficiency tests are carried out on an annual basis, and aim to improve the quality, accuracy and comparability of the residue data reported by EU Member States to the European Commission.

The CRL for pesticides using Single Residue Methods (CRL-SRM) has so far conducted 2 EUPTs within the abovementioned frame; one in collaboration with the CRL for Pesticides in Fruit and Vegetables (CRL-FV) using apple juice as commodity (EUPT-SRM1) and one in collaboration with the CRL for Cereals and Feedingstuff (CRL-CF) using wheat as commodity (EUPT-C1-SRM2). An additional PT in 2008 concerned the analysis of pentachlorophenol (PCP) in guar gum and was organised in cooperation with the CRL for Food of Animal Origin (CRL-AO) in response to a major incident of PCP and dioxins-contaminated guar gum from India entering the EU-market. The present PT (EUPT-SRM3) has been organised in collaboration with the CRL-FV using the same matrix (carrots homogenate) as for the EUPT-FV10 that was launched a few weeks earlier. The CRL-FV organised the purchase of the blank carrots as well as the treatment and shipment of the test material.

At the time of preparation of the EUPT-SRM3, Regulation 396/2005/EC was not fully in force, so participation in this PT was mandatory only for the National Reference Laboratories (NRLs) responsible for the Single Residue Methods. The participation of all other official EU laboratories involved in the determination of pesticide residues in fruit and vegetables for the EU-coordinated or respective national monitoring programmes was highly recommended. Additionally, laboratories from the EFTA states Iceland, Norway and Switzerland were invited to take part in the PT as they also contribute data to the EU-coordinated monitoring programme. Following a request by the Commission all official labs not participating in the test were asked to provide their reasons for non-participation.

This report may be presented to the European Commission Standing Committee for Animal Health and the Food Chain. Furthermore, DG-SANCO has full access to all data of EUPTs including the lab-code/lab-name key.

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[1] Regulation (EC) No 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

[2] Commission Regulation (EC) No 776/2006 of 23 May 2006 - amending Annex VII to Regulation (EC) No 882/2004 of the European Parliament and of the Council as regards Community Reference Laboratories.

[3] Regulation (EC) No 396/2005, published at OJ of the EU L70 of 16.03.2005, as last amended by Regulation 839/2008 published at OJ of the EU L234 of 30.08.2008.

1. INTRODUCTION

On February 1st 2008 all designated NRLs of the EU Member States as well as the official laboratories, as far as their contact data were available, were sent invitations to participate in the 3rd European Commission's Proficiency Test on Pesticides using Single Residue Methods (EUPT-SRM3). To make sure that all relevant official laboratories become aware of the PT, the NRLs were asked to forward the invitation to these laboratories within their countries. Included in this invitation was a list of ten possible pesticides (Possible Pesticides List, see ANNEX IX: Protocol), which could be potentially present in the test material, and their corresponding Minimum Required Reporting Levels (MRRLs).

Following this call, 70 laboratories from 27 countries (25 EU-member states plus two EFTA states) agreed to participate in this PT.

The carrots used as matrix for the PT were grown in Almería, in the south of Spain, and were treated post-harvest, using a microspray technique. Five pesticides were used for the treatments, one as a commercial formulation and the remaining four as a standard mixture dissolved in acetonitrile.

Participating laboratories were provided with 400 g portions of each, 'blank' carrot and 'spiked' carrot homogenates (Test Material). The PT-material was shipped to the participants starting on the 9th of June 2008, and the deadline for submission of results to the organiser was the 11th of July 2008.

The participants were asked to analyse the 'spiked' as well as the 'blank' material, and to determine and report the concentrations of any pesticide from the possible pesticide list that they detected. The 'blank' material was intended to be used for recovery experiments for the pesticides found in the test material and, if necessary, for the preparation of matrix-matched calibration standard solutions. 66 out of the 70 registered laboratories submitted results (see ANNEX I: Compilation of Results)

The medians of the analytical data submitted were used to obtain the assigned (true) concentration for each pesticide residue present. A fit-for-purpose target relative standard deviation (FFP RSD) of 25 % was chosen to calculate the target standard deviations of the assigned values as well as the z-scores for each pesticide present. For information purposes, the robust relative standard deviation (Qn) was also calculated.

As an optional exercise, participants were provided with a mixed standard solution containing all pesticides in the possible pesticide list, with the exception of the dithiocarbamate-component, and asked to determine their concentrations using their own analytical standards. This additional comparative test aimed at estimating to what extent variations in the pesticide concentrations of the standard solutions employed by the participants contribute to the overall variation of the results reported for the PT-test sample. The vials containing the mixed standard solutions were delivered to the laboratories together with the PT-material. The laboratories were instructed to only deliver results for the pesticides that they have previously encountered in the test material. 35 laboratories delivered results for this additional test (see ANNEX VI: Standard Solution Test)

For registration, communication and submission of any results an online toolwas employed in order to simplify all necessary steps.

2. ORGANISATIONAL

2.1 Analytical Methods

The following analytical methods, described briefly below, were employed by the organiser to conduct the homogeneity and stability tests:

For the acidic pesticides (**fluazifop, MCPA**): QuEChERS-method[4], involving extraction with acetonitrile, partitioning after addition of salts, and direct determination by LC-MS/MS in the ESI-neg. mode. LC-MS/MS-conditions see www.crl-pesticides.eu

For **avermectin B1a**: QuEChERS-method[4] involving extraction with acetonitrile, partitioning after addition of salts, and direct determination by LC-MS/MS in the ESI-pos. mode. LC-MS/MS-conditions see www.crl-pesticides.eu

For **propamocarb**: QuEChERS-method[4], involving extraction with acetonitrile, partitioning after addition of salts, and direct determination by LC-MS/MS in the ESI-pos. mode.

For **dithiocarbamates**: in-house-method [see www.crl-pesticides.eu], involving cleavage with HCl/$SnCl_2$, partitioning into isooctane and determination by GC-ECD.

2.2 Selection of Pesticides for the Possible Pesticide List

The pesticides to be included in the possible pesticides list were selected by the Organiser and the scientific committee taking into account the present and upcoming scope of the EU-coordinated monitoring programme, a pesticide priority list ranking the pesticides according to their relevance and risk-potential as well as a list of pesticides relevant for the specific commodity (carrots). The overall capacity and capability of the laboratories within the EU, as assessed from previous PTs and Surveys, was also taken into account. The residue definitions valid for the test were slightly different from those in the legislation to account for analytical difficulties (e.g. in the case of abamectin the laboratories had to report only the B1, a single component, ignoring the minor components B1b and 8,9-Z.

2.3 Preparation of the test material

Before preparing the test material, the pesticides to be spiked and suitable concentration levels for the study were selected following the recommendations made by the Quality Control Group.

Two hundred kilograms of organically grown carrots had their stems removed and were treated with a commercial pesticide formulation of maneb diluted with water, as well as with a mixture of analytical standards of propamocarb, fluazifop, MCPA and abamectin dissolved in acetontrile. In both cases a microspray technique was used for applications. Several units were randomly sampled, homogenized and analysed to check the residue levels present in order to decide whether, or not, additional spraying was necessary. As this was not the case, the entire sample was frozen and chopped using liquid nitrogen and a mincer. The frozen minced carrots were mixed in a constantly spinning container until a homogeneous material was obtained. 400 g portions of the well-mixed homogenate were weighed out into screw-capped polyethylene plastic bottles, sealed, and stored in a freezer at about - 20 °C prior to distribution to participants.

[4] EN-15662; Foods of plant origin - Determination of pesticide residues using GC-MS and/or LC-MS(/MS) following acetonitrile extraction/partitioning and cleanup by dispersive SPE - QuEChERS-method

2.4 Preparation of the 'blank' test material

100 kg of carrots originating from the same field as those used to prepare the spiked test material was employed for the production of the blank test material. The blank-carrot- homogenate was processed in the same way as the treated test material described above.

2.5 Homogeneity test

Ten bottles of treated test material were randomly chosen from those stored in the freezer and analyses were performed on duplicate portions taken from each bottle. The sequence of injection of the 20 extracts analysed by GC and LC was chosen randomly. The quantification was performed using 4-point calibration curves constructed using matrix-matched standard solutions prepared from extracts of the 'blank' carrot material.

The statistical evaluation was performed according to the International Harmonized Protocol published by IUPAC, ISO and AOAC[5]. An overview of the results of the homogeneity test is shown in Table 1. The individual residue data from the homogeneity tests, as well as the results of the statistical analyses, are given in Table 9 in the Annex. The acceptance criterion for the test material to be sufficiently homogenous for the proficiency test was that $Ss/\sigma < 0.3$, where S_s is the standard deviation of the pesticide concentrations determined for the 10 bottles and σ the target fit-for-purpose relative standard deviation (25%) multiplied by the mean concentration of each pesticide.

Table 1: Summary of the homogeneity test

Compound	Mean in mg/kg	Ss/σ	Notes*
Avermectin B1a	0.066	0.291	passed
Dithiocarbamates as CS_2	0.723	0.282	passed
Fluazifop	0.089	0.245	passed
MCPA	0.119	0.275	passed
Propamocarb	0.137	0.257	passed

10 g analytical portions were in all cases except for Dithiocarbamates, where 25 g were used

As all pesticides passed the homogeneity test, the test material was considered to be sufficiently homogenous and suitable for the use in the EUPT-SRM3.

2.6 Stability test

The stability test involved analyses on two occasions, one before the start of the PT exercise, and the other one after its completion. During this period, the test-material was stored at -18°C, as recommended to the participants in the protocol. The analyses were performed using of one randomly chosen bottle, performing quintuplicate extractions on each occasion, and duplicate instrumental analyses of each extract.

The two occasions were:

Day 1: shortly before the sample shipment to the laboratories, 30th May 2008

Day 2: after a period of about two and a half months, 17th July 2008

[5] Thompson M., Ellison S. L. R. and Wood R., The International Harmonized Protocol for the Proficiency Testing of Analytical Chemistry Laboratories. Pure & Appl Chem 78, 145-196 (2006.)

An overview of the results of the stability test is shown in Table 2 and additional Information in Table 10 in the Annex.

Table 2: Summary of the Stability Test

Compound	% Deviation of determined concentrations in Day 1 and Day 2	Notes
Avermectin B1a	- 14%	pass
Dithiocarbamates as CS_2	+ 8%	pass
Fluazifop	+ 5%	pass
MCPA	+ 8%	pass
Propamocarb	+ 4%	pass

With exception of Avermectin B1a, the results of the test indicated good stability of the pesticides. Taking into account analytical variability and the fact that the material was stored considerably longer than the duration of the PT-exercise (7 versus 4 weeks), the stability of avermectin B1a at the suggested conditions was also considered as acceptable throughout the PT.

2.7 Registration, release of documents and result submission

An online submission tool was developed, allowing participants to submit their results via the internet. All participants had to register on the webpage, 'www.eupt.es/crl-srm/eupt-srm3/' (by filling out the Application Form). During the registration process, participants had to give detailed information about their laboratory and - where applicable - to state their reasons for non-participation. After that the participants received login-information (including their lab-code) to get individual access to the restricted area, where they could download specific documents (e.g. protocol, possible pesticides list and calendar) and submit their results. This procedure ensured confidentiality throughout the duration of the EUPT-SRM3.

After logging in to the EUPT-SRM3-webpage, participants were able to access the following forms, as announced in the protocol:

Form 1 : acceptance of the test material,

Form 2 : submission of results and detailed analytical method information and

Form 3 : submission of results of the mixed standard solution test (optional)

2.8 Distribution of the test material

Before shipment of the samples, the laboratories received full instructions (see Protocol in the Annex) for the receipt, storage and analysis of the test materials.

One bottle of treated test material (~400g), one bottle of 'blank' material (~400g), as well as a vial containing a mixed standard solution, encased in a box containing dry ice. The boxes were shipped to the participants starting on the June 9th 2008. Due to strikes in Spain during the week the shipment was planned, several samples had to be delivered with a delay of up-to one week. In order to account for these delays, the deadline for result submission was shifted back by one week from July 4th to July 11th.

The laboratories were encouraged to re-homogenize the entire amount of test-sample contained in the bottle whilst it was still frozen before withdrawing any test portions for analysis, as this was also the procedure followed before performing the stability and homogeneity tests.

3. STATISTICAL METHODS

3.1 False positives and false 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 repeated analysis, are treated as false positives, if they are reported at concentrations at, or above, the Minimum Required Reporting Level (MRRL) stipulated by the Organisers. Results reported that are lower than the MRRL are ignored by the Organisers and not considered as false positives.

3.1.2 False negatives

Results for pesticides reported by the laboratories as not detected (ND), even though they were used by the Organiser to treat the test material and were subsequently detected at, or above, the MRRL by the Organiser (and the majority of participating laboratories) are considered to be false negatives.

3.2 Establishment of the assigned (consensus) values

To establish the assigned values, the median levels of all the reported results, excluding the outliers, were used.

3.3 Target standard deviation

Based on previous experience from EU proficiency tests on fruit and vegetables a fixed fit-for-purpose relative standard deviation (FFP RSD) of 25 % is used. The target standard deviation (σ) for each individual pesticide is calculated by multiplying this FFP RSD by the assigned value. In addition, the robust relative standard deviations Qn are calculated for informative purposes.

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 specific reporting limit (MRRL) of the lab for those labs not having detected the pesticide present in the test material
X is the assigned (consensus) value or true concentration
σ is the fit-for-purpose target standard deviation and equals 25% of the assigned value.

The 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'.

Z-scores for false negatives are determined using the MRRL for calculation.

No calculation of z-score is performed for any false positive result.

4. RESULTS

4.1 Overview of participating labs

70 laboratories from 27 different countries registered to participate this PT, with 66 laboratories from 27 different countries finally reporting results. Among these were 63 laboratories from 25 different EU-Member States and 3 labs from 2 EFTA states. 23 laboratories from 23 countries were designated NRLs for single residue methods (NRL-SRM) in their countries. The EU-Member States where NRL-SRMs did not report results were:

1) LU and MT, which were represented by the NRLs of BE and UK, respectively.

2) FR, ES and RO, where the NRL-SRM-designation was pending or unclear at the time of the test.

3) PL, registered to participate, but later withdrew due to lack of appropriate instrumentation.

An overview of the participating labs is given in Table 3. The names of all participating laboratories can be found in Table 14 in the Annex.

Table 3: Participating laboratories by country

Country	Participating		Sending results		Notes
	Labs	NRL-SRM	Labs	NRL-SRM	
Austria	2	1	2	1	
Belgium	3	1	3	1	Also representing LU
Bulgaria	1	1	1	1	
Cyprus	1	1	1	1	
Czech Republic	2	1	2	1	
Denmark	2	1	2	1	
Estonia	2	1	2	1	
Finland	1	1	1	1	
France	2	0	2	0	FR had not yet assigned any NRL-SRM
Germany	12	1	11	1	
Greece	4	2	3	1	GR has assigned two NRL-SRMs
Hungary	5	1	5	1	
Ireland	1	1	1	1	
Italy	2	1	2	1	
Latvia	1	1	1	1	
Lithuania	1	1	1	1	
Luxemburg	1	0	1	0	Represented by NRL-SRM of BE
Malta	0	0	0	0	Represented by NRL-SRM of UK
Netherlands	2	1	2	1	
Poland	7	1	6	0	
Portugal	4	1	4	1	
Romania	0	0	0	0	Lack of clarity as regards NRL-SRM status
Slovakia	1	1	1	1	
Slovenia	2	1	2	1	
Spain	6	0	5	0	ES had not yet assigned any NRL-SRM
Sweden	1	1	1	1	The NRL-SRM in SE has assigned a private laboratory with this task
United Kingdom	1	1	1	1	Also representing Malta
EU SUM	67	23	63	21	
Norway	1	1	1	1	
Switzerland	2	1	2	1	
OVERALL SUM	70	25	66	23	

4.2 Summary of reported results

An overview of the results can be seen in Table 4. In the Annex the corresponding z-score figures (Figure 6 to Figure 9), as well as the histograms of the result distributions (Figure 1 to Figure 5) can be seen. Method details are also listed in the Annex in Table 12 and Table 13.
Altogether 66 Laboratories reported results. The number of laboratories seeking for the different pesticides varied from 59 for dithiocarbamates (89%) to only 24 for avermectin B1a (36%).

Table 4: Result overview (Pesticides present in test material in bold)

66 Labs in total	No of Labs sought for (% of all)	No of results (% of all)	No of NDs	Median in mg/kg	Qn in %	MRRL in mg/kg
2.4-D	38 (58 %)	0	38	---	---	0.05
Avermectin B1a	24 (36 %)	24 (36 %)	0	0.057	23.4	0.01
Dithiocarbamates as CS_2	59 (89 %)	59 (89 %)	0	0.830	38.4	0.05
Fluazifop	36 (55 %)	35 (55 %)	1*	0.084	26.6	0.05
Haloxyfop	37 (56 %)	0	37	---	---	0.05
MCPA	38 (58 %)	38 (58 %)	0	0.124	27.0	0.05
MCPP	36 (55 %)	0	36	---	---	0.05
Propamocarb	37 (56 %)	37 (56 %)	0	0.118	28.2	0.05

* not considered as false negative as median is too close to MRRL

4.2.1 False positives

No false positive results were reported.

4.2.2 False negatives

One laboratory reported 'ND' (not detected) for fluazifop. However, since the median of all values (0.084 mg/kg) is very close to the MRRL (0.05 mg/kg), this result was not considered to be a false negative.

4.2.3 Distribution of Data

The distributions of the residue levels of the pesticides reported by the laboratories have been plotted as histograms (see Figure 1 to Figure 5 in the Annex). For every pesticide the submitted data produced unimodal distributions with no results being eliminated as outliers.

4.3 Assigned values and target standard deviations

The assigned values for each pesticide were based on the median values calculated using all the reported results. The target standard deviation for each pesticide was obtained using a fixed FFP RSD value of 25 %. In parallel, a robust relative standard deviation (Qn) was calculated for informative purposes. Median values as well as standard deviations are presented in Table 4.
In general the Qn RSD matches satisfactorily with the FFP-RSD with exception of dithiocarbamates.

4.4 Assessment of laboratory performance

4.4.1 General Assessment

The z-scores were calculated using the FFP RSD of 25% for all the pesticides present.

Table 8 in the Annex shows a compilation of the results including the median values for each pesticide as well as the individual z-scores for each laboratory/pesticide combination. The corresponding graphs are shown in Figure 6 to Figure 9 in the Annex.

Table 5 gives an overview on the overall performance (classification) of the laboratories for each of the pesticides. More than 80 % of the laboratories achieved results that can be considered acceptable in all cases. Compared to previous EUPT-SRM, there is a clear improvement.

Table 5: Classification of results

	Acceptable	Questionable	Unacceptable
Avermectin B1a	20 (84 %)	2 (8 %)	2 (8 %)
Dithiocarbamates as CS_2	50 (85 %)	6 (10 %)	3 (5 %)
Fluazifop	28 (80 %)	4 (11 %)	3 (9 %)
MCPA	34 (90 %)	2 (5 %)	2 (5 %)
Propamocarb	34 (92 %)	1 (3 %)	2 (5 %)

The SRM results were treated individually and no laboratory ranking based on the sum of weighted z-scores (SWZ) was calculated.

4.4.2 Comparison NRL-SRM with other laboratories

Table 6 and Table 7 give a comparison of the scope and performance of laboratories acting as NRLs compared to others. As shown in Table 6, NRL-SRM sought on average for about 70 %. Their average scope was thus notably more than other laboratories, that sought only about 50 - 60 % of the possible pesticides.

Table 6: Mean number of analytes sought for by different laboratories

	No of Labs (submitting results)	Mean number of analytes sought for 8 pesticides on potential list
NRL-SRM	21	5.6 (70%)
NRL-other	11	4.4 (55%)
Not NRL-SRM	45	4.2 (53%)
Not NRL	34	4.1 (51%)
All Labs	66	4.6 (58%)

In Table 7 NRL-SRMs are compared to other laboratories as regards the z-scores obtained. In overall, NRL-SRMs obtained lower mean absolute z-scores compared to other laboratories, leading to the conclusion that their results are closer to the assigned (true) value. Interestingly the median values of the NRL-SRMs are in all cases slightly lower than those of other laboratories.

Table 7: Comparison NRL-SRM with others, medians and mean absolute z-scores

	NRL-SRM			Not NRL-SRM			All labs	
	No of results	Median	mean abs z-score	No of results	Median	mean abs z-score	Median	mean abs z-score
Avermectin B1a	7	0.053	0.5	17	0.065	1.4	0.057	1.1
Dithiocarbamates as CS_2	18	0.762	1.0	41	0.840	1.3	0.830	1.2
Fluazifop	12	0.079	0.5	23	0.099	1.4	0.084	1.1
MCPA	17	0.108	0.8	21	0.125	1.0	0.124	0.9
Propamocarb	13	0.110	0.8	24	0.120	1.1	0.118	1.0

4.5 Analytical methods

The analysis of dithiocarbamates as CS_2 involves an initial cleavage using acidic conditions in the presence of $SnCl_2$ and further analysis either by GC, or spectrophotometrically, following a derivatization reaction. GC-based methods involve either liquid-liquid-partitioning (LLP) into a non-polar solvent (in most cases isooctane) followed by GC-analysis or a direct headspace sampling and GC-injection (HS). 28 of the 59 laboratories reporting results for dithiocarbamates indicated that they have employed a method involving GC determination, with 11 of them using the headspace technique, and the rest (17) the liquid-liquid-partitioning approach. 31 laboratories employed the spectrophotometric method (Phot). Interestingly there is a significant difference in the median values of the laboratories using the spectrophotometric method ($median_{Phot}$= 1.02 mg/kg) to those using the GC-based methods ($median_{GC}$= 0.75 mg/kg). The difference between the median values of the LLP ($median_{LLP}$= 0.70) and the HS methods ($median_{HS}$= 0.80) was smaller. Further PTs and experiments should show whether, or not this trend can be confirmed.

For all other compounds present in the test materials the most frequently employed sample preparation approaches were based on the QuEChERS or ChemElut methods. LC-MS/MS was by far the technique most frequently used for determinative analysis. In the case of acidic pesticides laboratories employing the GC-based method involved transformation into GC-amenable derivatives. These were 6 (out of 39) laboratories in the case of MCPA and 4 (out of 35) laboratories in the case of fluazifop. In the case of propamocarb 3 laboratories used GC for analysis and 34 labs LC-MS/MS. In the case of avermectin B1a, which is not amenable to GC, all 24 laboratories reporting results employed LC-MS/MS.

4.6 Conclusions and suggestions for future work

Seventy laboratories agreed to participate in EUPT-SRM3, and 66 of them submitted results.

Compared to the previous two EUPT-SRMs the number of laboratories participating has significantly increased from 27 (from 15 countries) in the EUPT-SRM1 to 31 (from 18 countries) in the EUPT-C1/SRM2 to 70 (from 27 countries) in EUPT-SRM3. The acquisition and increased use of LC-MS/MS instrumentation, is probably one of the main reasons.

For each laboratory/pesticide combination, z-scores based on the FFP RSD of 25% have been calculated. Sub-classification of z-scores into 'acceptable', 'questionable' and 'unacceptable' has also been undertaken.

The overall results with regard to the z-scores for each pesticide present in the test material were good with between 80% and 92% of the results being acceptable. No false positives or false negatives were reported with exception of one case concerning fluazifop. This was not considered to be a false negative as the median value was too close to the MRRL.

The Qn-RSD was close to the FFP-RSD of 25% in all cases except for the dithiocarbamates (as $CS_{2)}$ (38%). In the latter case, a trend of obtaining higher values when using the spectrophotometric method was observed compared to the GC-based methods. Further experiments and proficiency tests are necessary to elucidate the reasons for this discrepancy.

Overall the scope of the laboratories did improve compared to previous tests, but there is still a need for further significant improvement. Abamectin, fluazifop, propamocarb and MCPA were only covered by 36%, 55%, 56% and 59% of the participating labs, respectively. In the case of dithiocarbamates as CS_2 the coverage rate was 89%, and thus highly satisfactory.

In general the NRL-SRMs performed better than the other laboratories both in terms of scope, as well as regards accuracy as reflected by the z-scores obtained.

In future PTs, the selection of pesticides will continue to concentrate on increasing the scope of the monitoring programmes and the relevance of the pesticides. In order to encourage laboratories to increase their scope and decrease their reporting levels the Scientific Committee suggested that the MRRLs should be lowered and that important pesticides from the Possible Pesticide List should be marked with an asterisk. Non-coverage of these pesticides by the labs should be reflected in the overall ranking or classification.

The Scientific Committee furthermore strongly recommends laboratories to be equipped with LC-MS/MS as many very important pesticides can only be analysed by liquid chromatography. The aim is that laboratories continue to increase their scope of analytes, in order to be able to fully enforce EU legislation and to improve their overall performance, both in terms of correctly detecting the pesticides present as well as determining the residue levels accurately.

5. STANDARD SOLUTION TEST

5.1 Introduction

Proficiency Tests can play a very important role in detecting performance problems and errors in routine analytical procedures. Their role in analytical quality control is thus of paramount importance. A comprehensive evaluation of an unsatisfactory result obtained in a Proficiency Test can lead to the detection of an inappropriate analytical standard solution, problems in the extraction procedure, etc. However, the reasons for inaccurate results cannot always easily be identified due to the many possible sources of errors and their relative contributions.

With the aim of identifying possible sources of error contributing to the overall uncertainty, it was decided to conduct, in parallel to the PT, a Ring Test where the laboratories have to compare their standard solutions with a solution provided by the Organiser s containing the pesticides of the possible pesticide list of the EUPT-SRM3. The objective of this Ring Test was to find out to what extent the variability of the analytical standard concentrations between laboratories, contributed to the overall variability in EUPT-SRM3.

An additional benefit would be to help laboratories to detect possible inaccuracies with their standards and/or related working solutions.

A vial containing a solution of all pesticides in the possible pesticide list (with the exception of the dithiocarbamate-component) was sent to the laboratories that participated in EUPT-SRM3 together with the test material. Participation in this Ring Test was optional for the laboratories. The intention was that laboratories should determine the concentrations of the compounds in the standard solution using the same standard solutions as they used in EUPT-SRM3.

The relevant details that were provided to the participants were the:

(i) volume of standard solution supplied (5 mL)

(ii) solvent used to prepare the solution (acetonitrile)

(iii) concentration range of each compound present (20-90 mg/L).

Laboratories were asked to use the same determination techniques as they used in EUPT-SRM3. Results could be submitted by using Form 3 in the restricted area of the EUPT-website. Deadline for submitting results was 20th March 2008.

5.2 Standard solution preparation

Individual stock solutions in acetonitrile were prepared by weighing out suitable amounts of each of the reference standards. Aliquots of these stock solutions of the individual compounds were then taken to make up a mixed standard stock solution. This procedure was performed by three analysts, independently. From each mixed solution dilutions were prepared at concentrations within the working range of the appropriate detection system in order to measure the relative responses. The RSD of the average response from the three independent solutions had to be <10 % for each standard solutions to be approved. All three mixed stock standard solutions were then mixed.

5 mL of the mixed standard solution (20-90 mg/L) were transferred into screw vials and stored at -20°C until shipment. Only two days had elapsed between preparation and analysis of the solutions and shipment.

5.3 Results and Discussion

Altogether 35 laboratories submitted results for this standard solution test. On the basis of these results an evaluation of the results could be performed.

5.3.1 Statistical treatment

First, new medians (assigned values) and robust relative standard deviation (Qn) were calculated for each pesticide found in the EUPT-SRM3, using only results submitted by laboratories that participated in this standard solution test.
In a second step, the medians (assigned values) of the standard solution test were calculated for each pesticide.
In a third step, a correction factor was calculated for each laboratory/pesticide-combination by dividing the standard solution test results of each laboratory by the corresponding median (assigned value) of the standard solution.
Finally each EUPT-SRM3 result was multiplied with the corresponding correction factor, leading to a new, "corrected result" for each laboratory/pesticide-combination. For these "corrected EUPT-SRM3-results" a new "corrected robust relative standard deviation (Qn)" was calculated to check, whether the distribution of the results has improved.
A summary of these evaluations can be seen in Table 11 in the Annex.

5.3.2 Discussion

Most of the calculated correction factors were close to '1.0', which means that the results of the standard solution test were close to the overall median values. However, in some cases (MCPA, Propamocarb) single correction factors (e.g. > 3) were observed, which, of course, led to a significant 'correction' of the laboratory's result.
One major aspect was to see, whether this "result correction" leads to an improved data distribution (with smaller Qn values). This, however, could not be observed. On the contrary, the "corrected results" led to even worse Qn values in all cases. Thus, no clear correlation trend between the results in the EUPT-SRM3 and the standard solution test results could be found.
Nevertheless, the Scientific Committee is of the opinion, that this Ring Test is useful for the laboratories quality control and thus worthwhile repeating in the future.

6. ACKNOWLEDGEMENTS

The Organising Team wishes to warmly thank all members of the Scientific Committee for their valuable advice. The Organizer also wishes to especially thank the team at the University of Almería for their great assistance and support.

7. ANNEXES

ANNEX I: COMPILATION OF RESULTS

Table 8: Compilation of individual results

	NRL	No of analytes sought for	Avermectin B1a		Dithiocarbamates as CS_2		Fluazifop (free acid)		MCPA (free acid)		Propamocarb		Notes
Median (mg/kg)			0.057		0.830		0.084		0.124		0.118		
MRRL (mg/kg)			0.01		0.05		0.05		0.05		0.05		
Qn RSD (%)			23.4		38.4		26.6		27.0		28.2		
			Result mg/kg	z-score FFP-RSD	Result mg/kg	z-score FFP-RSD	Result mg/kg	z-score FFP-RSD	Result mg/kg	z-score FFP-RSD	Result mg/kg	z-score FFP-RSD	
Lab 01	SRM	5	NA		1.22	1.9	NA		0.139	0.5	0.172	1.8	
Lab 02	other	1	NA		0.211	-3.0	NA		NA		NA		
Lab 03	SRM	8	0.052	-0.4	0.54	-1.4	0.086	0.1	0.117	-0.2	0.133	0.5	
Lab 04	other	1	NA		0.446	-1.9	NA		NA		NA		
Lab 05		8	0.101	3.1	0.65	-0.9	0.103	0.9	0.121	-0.1	0.122	0.1	
Lab 06		1	NA		1.27	2.1	NA		NA		NA		
Lab 07		1	NA		1.15	1.5	NA		NA		NA		
Lab 08	other	5	0.091	2.4	0.48	-1.7	0.106	1.1	NA		0.148	1.0	
Lab 09	SRM	7	0.045	-0.8	0.649	-0.9	NA		0.132	0.3	0.038	-2.7	
Lab 10	SRM	1	NA		0.66	-0.8	NA		NA		NA		Sample received at ambient temp. and thus not accepted
Lab 11		5	NA		NA		0.101	0.8	0.131	0.2	0.099	-0.6	
Lab 12	SRM	8	0.063	0.4	0.523	-1.5	0.082	-0.1	0.103	-0.7	0.133	0.5	
Lab 13	SRM	8	0.065	0.6	0.746	-0.4	0.188	5.0	0.155	1.0	0.168	1.7	
Lab 14		1	NA		0.89	0.3	NA		NA		NA		
Lab 15	SRM	7	NA		0.85	0.1	0.082	-0.1	0.136	0.4	0.135	0.6	
Lab 16		7	0.051	-0.4	NA		0.103	0.9	0.133	0.3	0.132	0.5	
Lab 17	other	1	NA		0.773	-0.3	NA		NA		NA		

	NRL	No of analytes sought for	Avermectin B1a		Dithiocarbamates as CS_2		Fluazifop (free acid)		MCPA (free acid)		Propamocarb		Notes
Median (mg/kg)			0.057		0.830		0.084		0.124		0.118		
MRRL (mg/kg)			0.01		0.05		0.05		0.05		0.05		
Qn RSD (%)			23.4		38.4		26.6		27.0		28.2		
			Result mg/kg	z-score FFP-RSD	Result mg/kg	z-score FFP-RSD	Result mg/kg	z-score FFP-RSD	Result mg/kg	z-score FFP-RSD	Result mg/kg	z-score FFP-RSD	
Lab 18	SRM	4	NA		NA		NA		0.161	1.2	NA		
Lab 19	other	8	0.125	4.8	0.218	-2.9	0.026	-2.8	0.057	-2.2	0.169	1.7	
Lab 20	SRM	6	NA		0.614	-1.0	0.022	-2.9	0.027	-3.1	NA		
Lab 21		8	0.057	0.0	1.21	1.8	0.039	-2.1	0.106	-0.6	0.104	-0.5	
Lab 22		1	NA		0.548	-1.4	NA		NA		NA		
Lab 23		7	NA		0.54	-1.4	0.081	-0.1	0.098	-0.8	0.1	-0.6	
Lab 24	SRM	No results submitted											
Lab 25	SRM	No results submitted											
Lab 26	SRM	8	0.057	0.0	0.825	0.0	0.073	-0.5	0.1	-0.8	0.107	-0.4	
Lab 27		1	NA		0.99	0.8	NA		NA		NA		
Lab 28	other	7	NA		0.745	-0.4	0.079	-0.2	0.106	-0.6	0.118	0.0	
Lab 29	other	8	0.078	1.5	0.8	-0.1	0.101	0.8	0.124	0.0	0.112	-0.2	
Lab 30	SRM	7	NA		0.834	0.0	0.066	-0.8	0.101	-0.7	0.097	-0.7	
Lab 31		8	0.075	1.3	1.021	0.9	0.133	2.4	0.14	0.5	0.142	0.8	
Lab 32	SRM	8	0.053	-0.3	0.159	-3.2	0.085	0.1	0.085	-1.2	0.133	0.5	
Lab 33		6	NA		1.61	3.8	NA		0.364	5.0	0.162	1.5	
Lab 34		8	0.052	-0.4	NA		0.082	-0.1	0.154	1.0	0.13	0.4	
Lab 35		1	NA		0.495	-1.6	NA		NA		NA		
Lab 36	other	8	0.08	1.6	0.801	-0.1	0.106	1.1	0.132	0.3	0.225	3.6	
Lab 37		7	NA		1.0	0.8	0.068	-0.7	0.091	-1.1	0.093	-0.8	
Lab 38		7	0.063	0.4	1.101	1.3	0.093	0.5	0.123	0.0	0.098	-0.7	
Lab 39	SRM	5	NA		NA		NA		0.108	-0.5	0.11	-0.3	
Lab 40		5	0.05	-0.5	0.85	0.1	0.096	0.6	NA		0.154	1.2	
Lab 41	other	6	NA		NA		0.117	1.6	0.204	2.6	0.094	-0.8	

	NRL	No of analytes sought for	Avermectin B1a		Dithiocarbamates as CS_2		Fluazifop (free acid)		MCPA (free acid)		Propamocarb		Notes
Median (mg/kg)			0.057		0.830		0.084		0.124		0.118		
MRRL (mg/kg)			0.01		0.05		0.05		0.05		0.05		
Qn RSD (%)			23.4		38.4		26.6		27.0		28.2		
			Result mg/kg	z-score FFP-RSD	Result mg/kg	z-score FFP-RSD	Result mg/kg	z-score FFP-RSD	Result mg/kg	z-score FFP-RSD	Result mg/kg	z-score FFP-RSD	
Lab 42		8	0.048	-0.6	1.17	1.6	0.088	0.2	0.124	0.0	0.066	-1.8	
Lab 43		No results submitted											
Lab 44		5	NA		0.703	-0.6	ND	-4.0	0.093	-1.0	NA		
Lab 45	SRM	8	0.047	-0.7	1.083	1.2	0.091	0.4	0.149	0.8	0.136	0.6	
Lab 46		1	NA		0.3195	-2.5	NA		NA		NA		
Lab 47	SRM	8	0.054	-0.2	1.24	2.0	0.068	-0.7	0.123	0.0	0.106	-0.4	
Lab 48		7	0.055	-0.1	1.67	4.0	0.15	3.2	0.145	0.7	0.1	-0.6	
Lab 49		1	NA		0.85	0.1	NA		NA		NA		
Lab 50		8	0.08	1.6	0.85	0.1	0.103	0.9	0.125	0.0	0.152	1.2	
Lab 51		8	0.086	2.0	0.595	-1.1	0.078	-0.3	0.148	0.8	0.081	-1.3	
Lab 52	SRM	6	NA		0.86	0.1	NA		0.1	-0.8	0.103	-0.5	
Lab 53	SRM	1	NA		0.63	-1.0	NA		NA		NA		
Lab 54	SRM	6	NA		0.675	-0.7	0.08	-0.2	0.086	-1.2	NA		
Lab 55		1	NA		1.11	1.3	NA		NA		NA		
Lab 56		1	NA		0.947	0.6	NA		NA		NA		
Lab 57	other	1	NA		0.794	-0.2	NA		NA		NA		
Lab 58		No results submitted											
Lab 59	SRM	1	NA		0.92	0.4	NA		NA		NA		
Lab 60	SRM	1	NA		1.07	1.2	NA		NA		NA		
Lab 61	SRM	6	NA		NA		0.078	-0.3	0.084	-1.3	0.1	-0.6	Sample received in partly unfrozen condition, but accepted
Lab 62		1	NA		1.19	1.7	NA		NA		NA		
Lab 63	other	2	NA		0.83	0.0	NA		NA		0.213	3.2	
Lab 64		1	NA		0.34	-2.4	NA		NA		NA		Sample received in

	NRL	No of analytes sought for	Avermectin B1a		Dithiocarbamates as CS_2		Fluazifop (free acid)		MCPA (free acid)		Propamocarb		Notes
Median (mg/kg)			0.057		0.830		0.084		0.124		0.118		
MRRL (mg/kg)			0.01		0.05		0.05		0.05		0.05		
Qn RSD (%)			23.4		38.4		26.6		27.0		28.2		
			Result mg/kg	z-score FFP-RSD	Result mg/kg	z-score FFP-RSD	Result mg/kg	z-score FFP-RSD	Result mg/kg	z-score FFP-RSD	Result mg/kg	z-score FFP-RSD	
													partly unfrozen condition, but accepted
Lab 65	SRM	6	NA		0.698	-0.6	0.075	-0.4	0.133	0.3	NA		
Lab 66		1	NA		0.79	-0.2	NA		NA		NA		
Lab 67		1	NA		1.19	1.7	NA		NA		NA		
Lab 68		5	0.044	-0.9	0.84	0.0	0.067	-0.8	NA		0.109	-0.3	
Lab 69		1	NA		1.05	1.1	NA		NA		NA		
Lab 70		1	NA		1.35	2.5	NA		NA		NA		

It should be noted that evaporation losses of CS_2 leading to underestimated results are more likely at elevated temperatures

ANNEX II: HOMOGENEITY DATA

Table 9: Homogeneity data for all five pesticides

Sample	Avermectin B1a (using 10 g)		Dithiocarbamates (using 25 g)		Fluazifop (using 10 g)		MCPA (using 10 g)		Propamocarb (using 10 g)	
	Portion 1 mg/kg	Portion 2 mg/kg	Portion 1 mg/kg	Portion 2 mg/kg	Portion 1 mg/kg	Portion 2 mg/kg	Portion 1 mg/kg	Portion 2 mg/kg	Portion 1 mg/kg	Portion 2 mg/kg
1	0.065	0.065	0.636	0.670	0.087	0.088	0.136	0.129	0.143	0.148
2	0.061	0.062	0.621	0.766	0.090	0.092	0.126	0.119	0.133	0.131
3	0.070	0.069	0.584	0.658	0.091	0.094	0.121	0.121	0.142	0.142
4	0.056	0.057	0.788	0.634	0.082	0.083	0.104	0.105	0.115	0.126
5	0.067	0.064	0.716	0.692	0.096	0.100	0.124	0.126	0.147	0.149
6	0.069	0.068	0.807	0.707	0.089	0.092	0.117	0.124	0.138	0.137
7	0.067	0.073	0.686	0.812	0.096	0.095	0.127	0.129	0.142	0.148
8	0.065	0.064	0.689	0.717	0.085	0.083	0.109	0.110	0.129	0.131
9	0.074	0.075	0.758	0.781	0.089	0.086	0.124	0.110	0.142	0.142
10	0.066	0.066	0.884	0.848	0.076	0.084	0.110	0.114	0.120	0.132
Mean in mg/kg	0.066		0.723		0.089		0.119		0.137	
S_s/σ	0.291		0.282		0.245		0.275		0.257	
Pass/Fail	**Pass**		**Pass**		**Pass**		**Pass**		**Pass**	

ANNEX III: STABILITY DATA

Table 10: Stability data for all five pesticides

	Avermectin B1a	Dithiocarbamates	Fluazifop	MCPA	Propamicarb
Day 1 (mean in mg/kg)	0.0549	0.871	0.082	0.130	0.112
			-18°C		
Day 2 (mean in mg/kg)	0.0471	0.805	0.086	0.141	0.116
%	**- 14 %**	**+ 8 %**	**+ 5 %**	**+ 8 %**	**+ 4 %**
Pass/Fail	**Pass**	**Pass**	**Pass**	**Pass**	**Pass**

ANNEX IV: DATA DISTRIBUTION

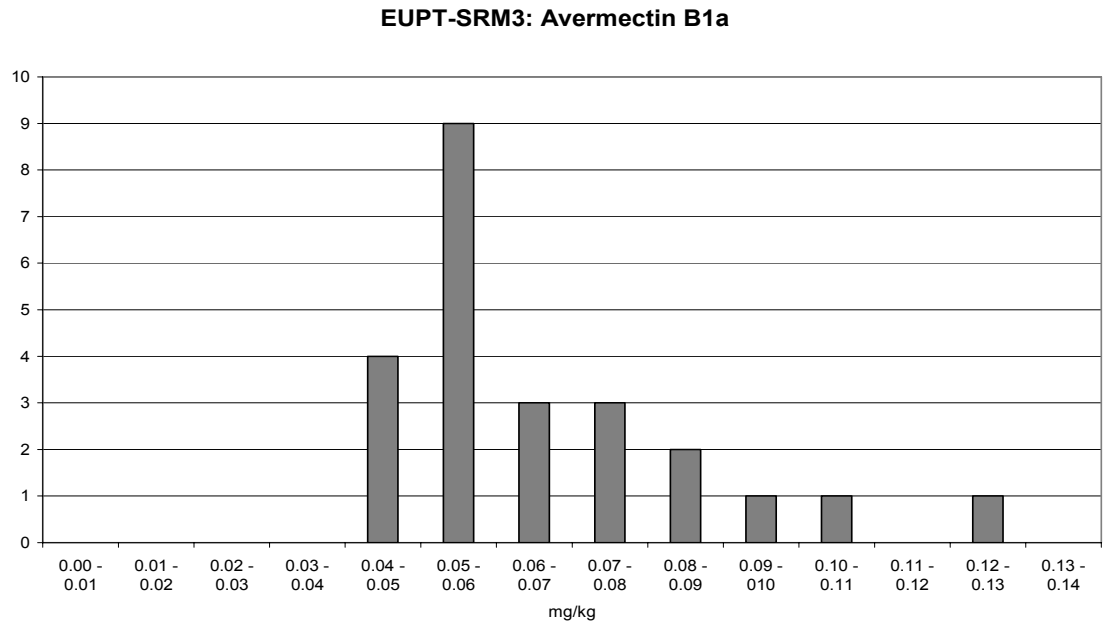

Figure 1: Data distribution for Avermectin B1a

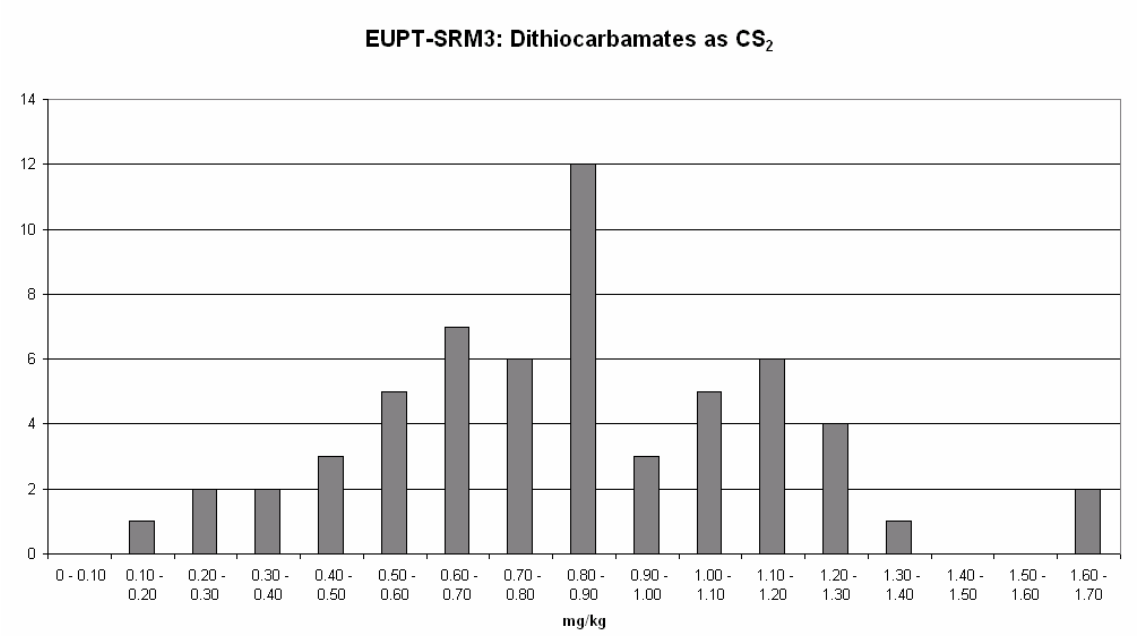

Figure 2: Data distribution for Dithiocarbamates

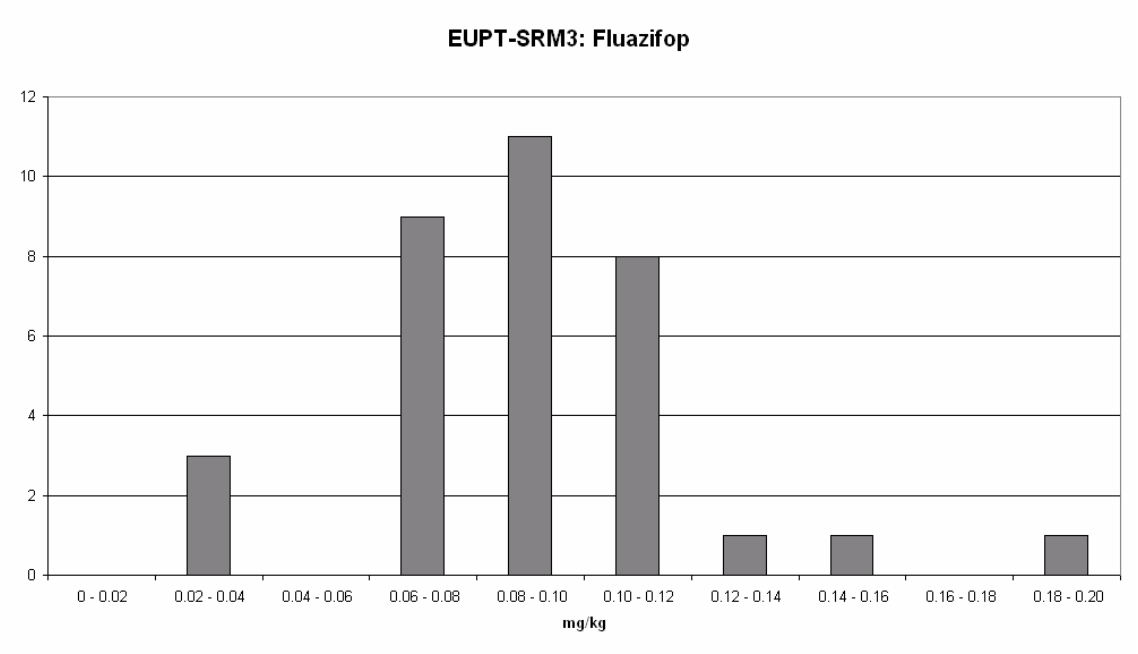

Figure 3: Data distribution for Fluazifop

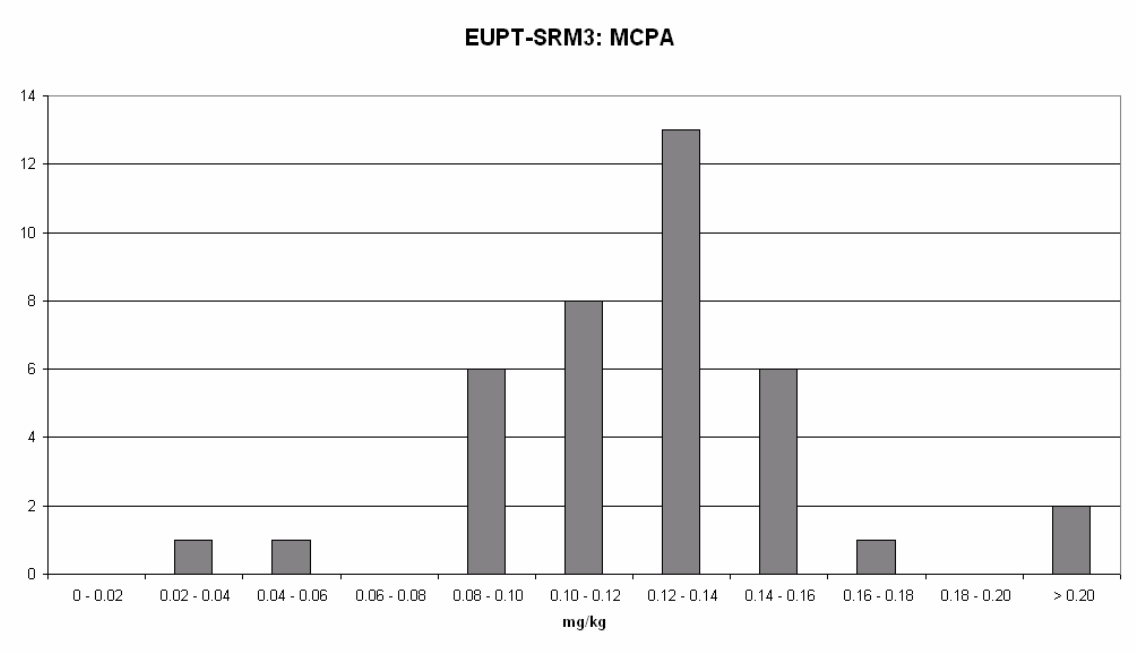

Figure 4: Data distribution for MCPA

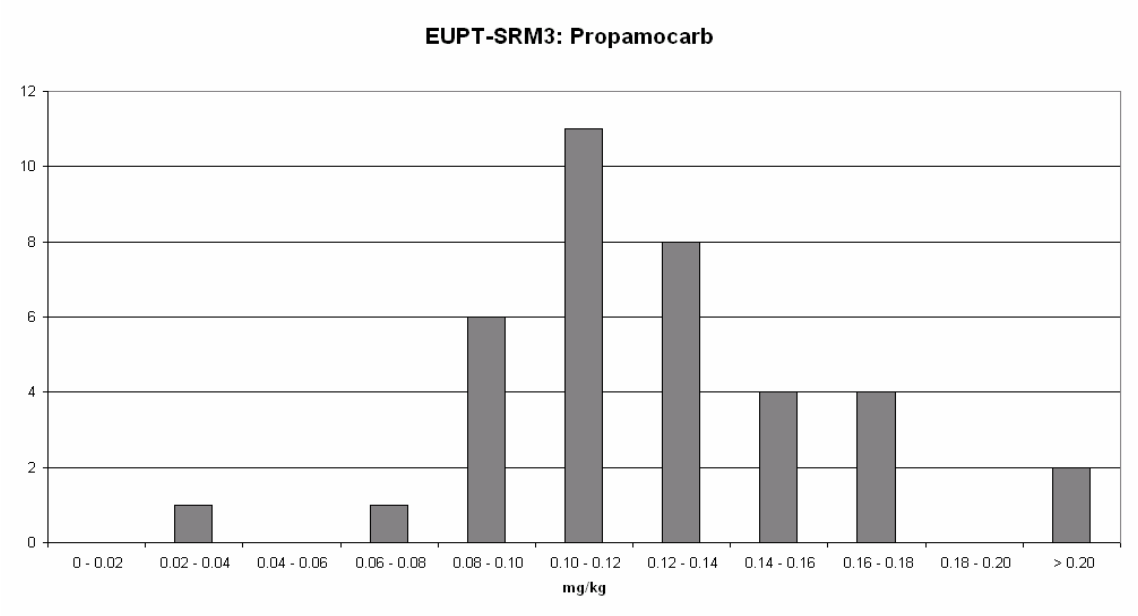

Figure 5: Data distribution for Propamocarb

ANNEX V: z-SCORES

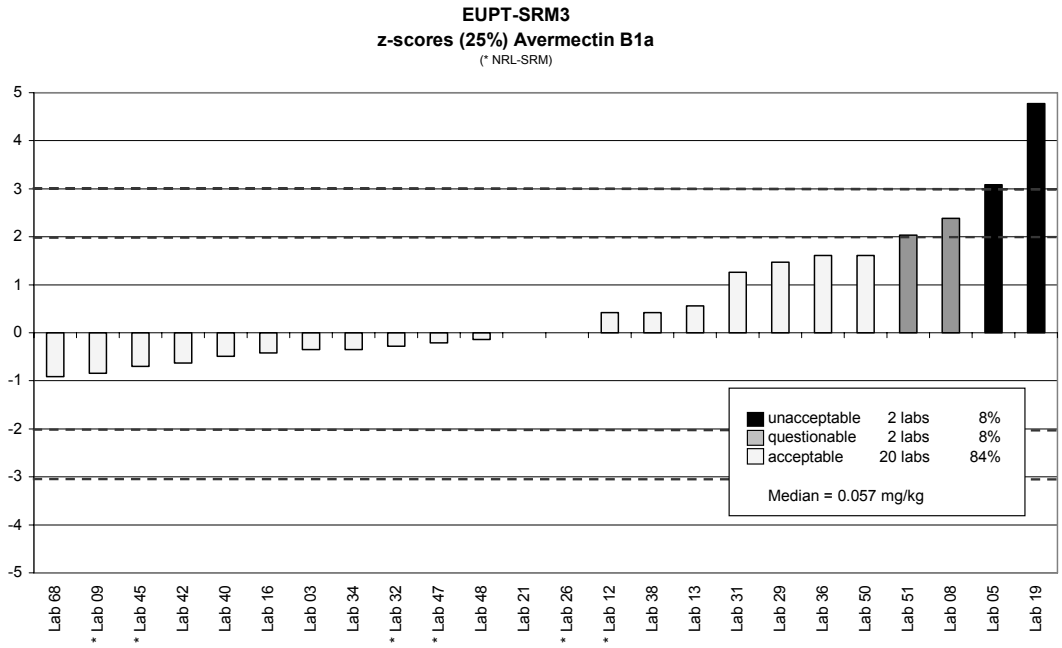

Figure 6: z-scores for Avermectin B1a

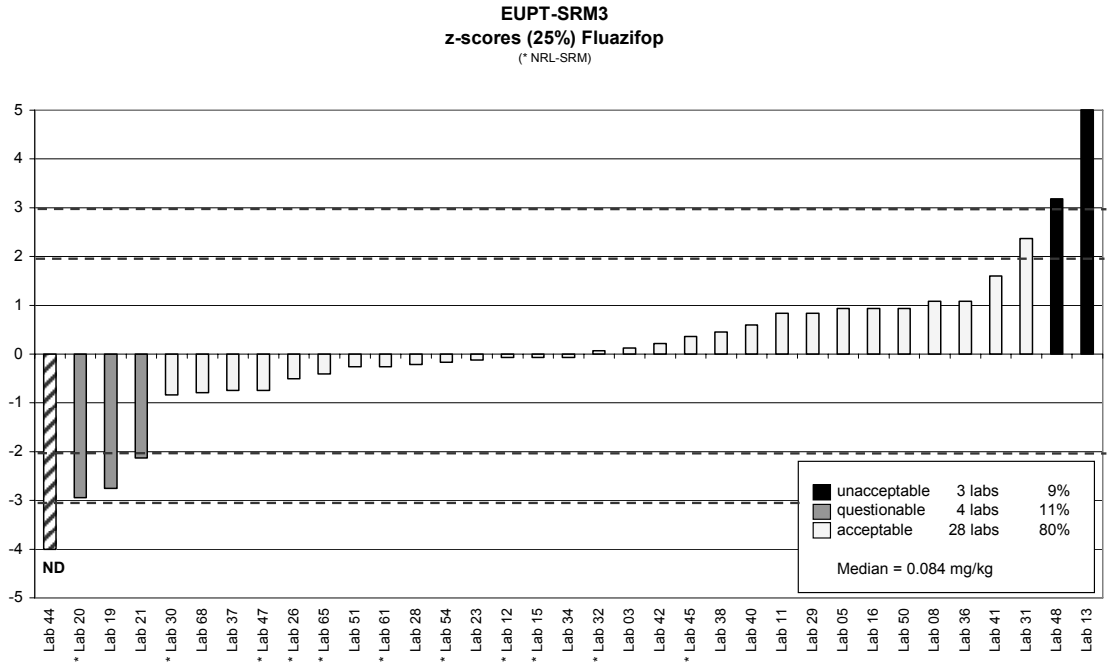

Figure 7: z-scores for Fluazifop

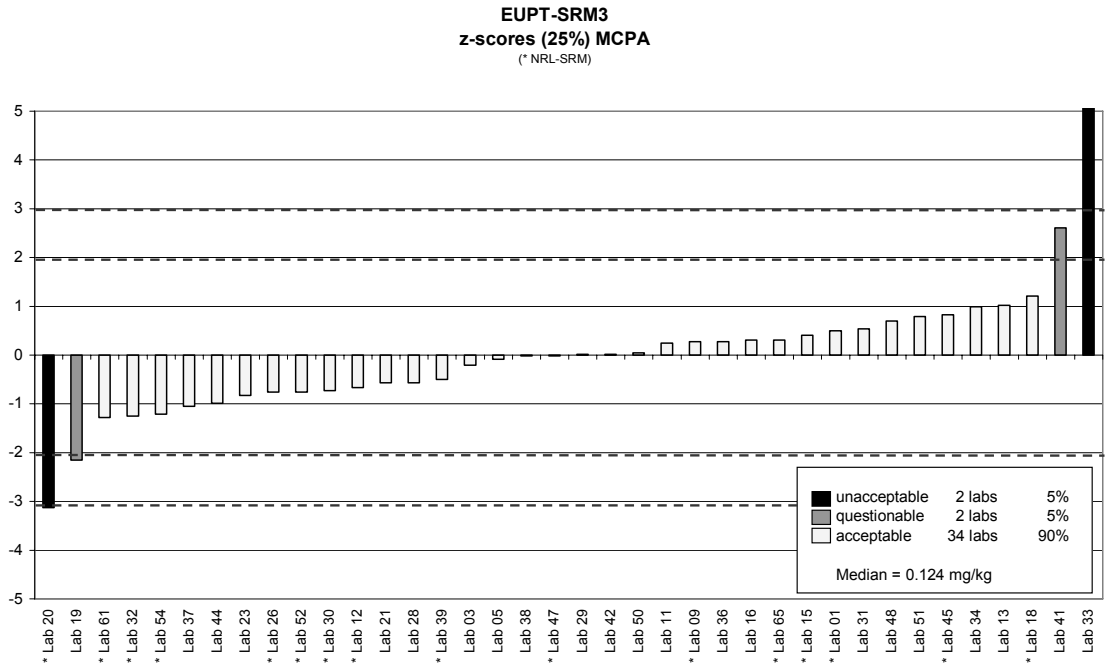

Figure 8: z-scores for MCPA

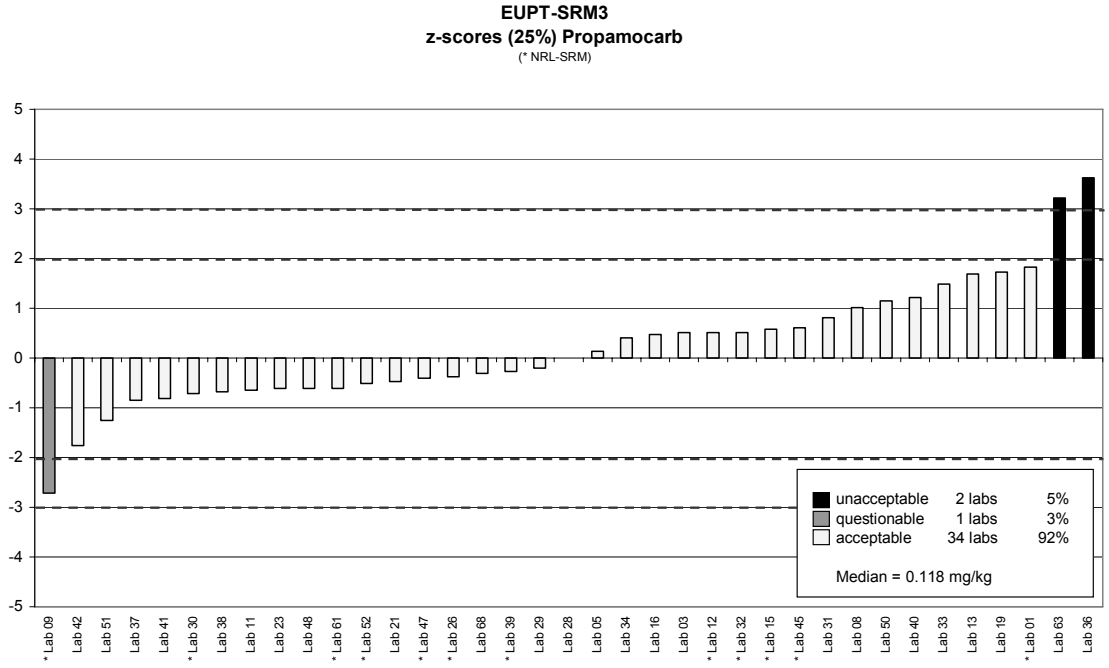

Figure 9: z-scores for Propamocarb

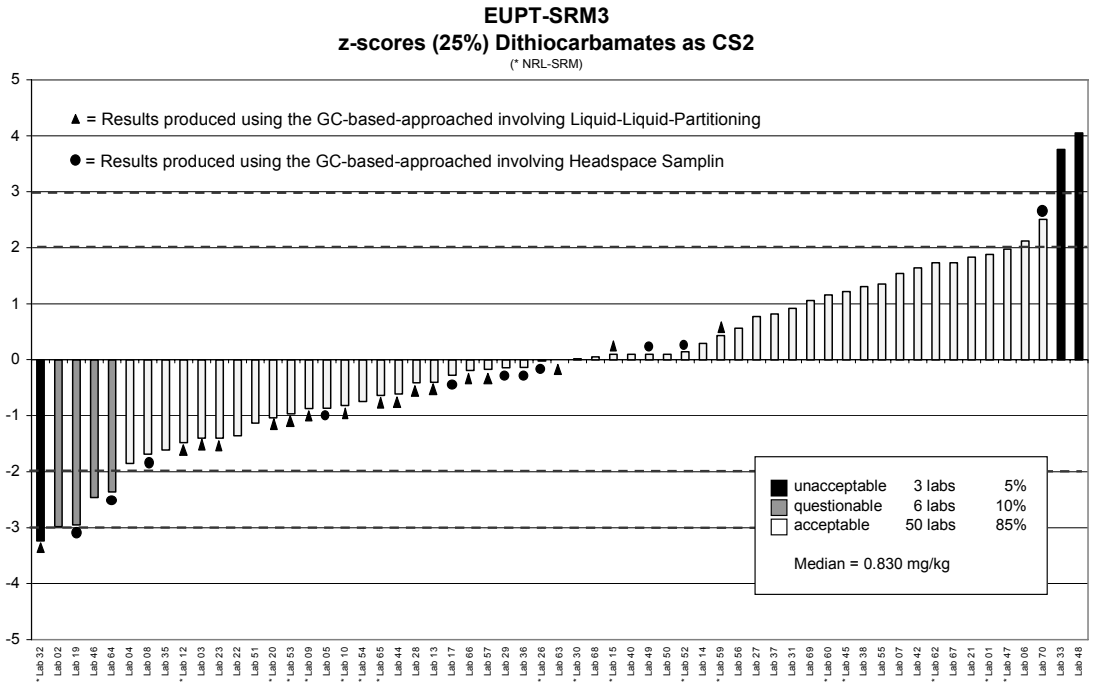

Figure 10: z-scores for Dithiocarbamates

ANNEX VI: STANDARD SOLUTION TEST

Table 11: Standard Solution Test (only labs participating in this Ring Test)

	Avermectin B1a				Fluazifop (free acid)				MCPA (free acid)				Propamocarb			
Original Median (mg/kg)	0.057				0.084				0.124				0.118			
No of Labs	20				28				32				29			
Median (mg/kg)			0.060	0.055			0.084	0.081			0.124	0.127			0.122	0.127
Qn RSD (%)			22.2	28.0			31.9	32.7			27.9	33.3			27.3	36.7
	Standard solution result	Correction factor (SSR/median)	Original PT-result	Corrected result	Standard solution result	Correction factor (SSR/median)	Original PT-result	Corrected result	Standard solution result	Correction factor (SSR/median)	Original PT-result	Corrected result	Standard solution result	Correction factor (SSR/median)	Original PT-result	Corrected result
Lab 01									65.5	0.810	0.139	0.172	17.4	0.584	0.172	0.295
Lab 03	86.6	1.414	0.052	0.037	39.8	0.975	0.086	0.088	89.0	1.100	0.117	0.106	41.2	1.383	0.133	0.096
Lab 05	52.0	0.849	0.101	0.119	65.8	1.613	0.103	0.064	75.0	0.927	0.121	0.131	28.6	0.960	0.122	0.127
Lab 08	39.8	0.650	0.091	0.140	19.7	0.483	0.106	0.219					21.2	0.711	0.148	0.208
Lab 09									61.9	0.765	0.132	0.173				
Lab 12	68.1	1.112	0.063	0.057	24.1	0.591	0.082	0.139	21.8	0.269	0.103	0.383				
Lab 13	83.3	1.360	0.065	0.048	40.0	0.980	0.188	0.192	84.1	1.040	0.155	0.149	35.2	1.181	0.168	0.142
Lab 16	59.7	0.975	0.051	0.052	39.4	0.966	0.103	0.107	67.1	0.829	0.133	0.160	48.4	1.624	0.132	0.081
Lab 18									83.1	1.027	0.161	0.157				
Lab 19	61.5	1.004	0.125	0.125	55.2	1.353	0.026	0.019	92.6	1.145	0.057	0.050	19.7	0.661	0.169	0.256
Lab 20					57.1	1.400	0.022	0.016	97.1	1.200	0.027	0.023				
Lab 21	70.5	1.151	0.057	0.050	40.8	1.000	0.039	0.039	88.3	1.091	0.106	0.097	26.2	0.879	0.104	0.118
Lab 26	61.0	0.996	0.057	0.057	40.5	0.993	0.073	0.074	61.9	0.765	0.100	0.131	26.0	0.872	0.107	0.123
Lab 28					39.4	0.966	0.079	0.082	77.4	0.957	0.106	0.111	26.6	0.893	0.118	0.132
Lab 29	39.0	0.637	0.078	0.122	47.1	1.154	0.101	0.088	78.4	0.969	0.124	0.128	24.3	0.815	0.112	0.137
Lab 30					30.0	0.735	0.066	0.090	79.0	0.977	0.101	0.103	36.0	1.208	0.097	0.080

	Avermectin B1a				Fluazifop (free acid)				MCPA (free acid)				Propamocarb			
Original Median (mg/kg)	0.057				0.084				0.124				0.118			
No of Labs	20				28				32				29			
Median (mg/kg)			0.060	0.055			0.084	0.081			0.124	0.127			0.122	0.127
Qn RSD (%)			22.2	28.0			31.9	32.7			27.9	33.3			27.3	36.7
	Standard solution result	Correction factor (SSR/median)	Original PT-result	Corrected result	Standard solution result	Correction factor (SSR/median)	Original PT-result	Corrected result	Standard solution result	Correction factor (SSR/median)	Original PT-result	Corrected result	Standard solution result	Correction factor (SSR/median)	Original PT-result	Corrected result
Lab 31	50.1	0.818	0.075	0.092	37.3	0.914	0.133	0.146	64.8	0.801	0.140	0.175	22.5	0.755	0.142	0.188
Lab 32	52.0	0.849	0.053	0.062	41.0	1.005	0.085	0.085	81.0	1.001	0.085	0.085	30.0	1.007	0.133	0.132
Lab 33									252.0	3.115	0.364	0.117	30.8	1.034	0.162	0.157
Lab 34	67.4	1.100	0.052	0.047	49.6	1.216	0.082	0.067	98.4	1.216	0.154	0.127	30.6	1.027	0.130	0.127
Lab 36	44.0	0.718	0.080	0.111	53.4	1.309	0.106	0.081	72.1	0.891	0.132	0.148	29.8	1.000	0.225	0.225
Lab 37					37.7	0.924	0.068	0.074	78.4	0.968	0.091	0.094	26.7	0.895	0.093	0.104
Lab 39									93.9	1.161	0.108	0.093	29.3	0.982	0.110	0.112
Lab 40	57.0	0.931	0.050	0.054	43.7	1.071	0.096	0.090					33.0	1.107	0.154	0.139
Lab 41					67.5	1.654	0.117	0.071	104.0	1.286	0.204	0.159	31.0	1.040	0.094	0.090
Lab 42	72.2	1.179	0.048	0.041	40.8	1.000	0.088	0.088	80.8	0.999	0.124	0.124	28.0	0.940	0.066	0.070
Lab 47	70.3	1.148	0.054	0.047	35.2	0.863	0.068	0.079	82.2	1.016	0.123	0.121	26.7	0.896	0.106	0.118
Lab 48	66.8	1.091	0.055	0.050	33.0	0.809	0.150	0.185	70.1	0.867	0.145	0.167	27.7	0.930	0.100	0.108
Lab 50	39.0	0.637	0.080	0.126	55.0	1.348	0.103	0.076	80.0	0.989	0.125	0.126	30.8	1.034	0.152	0.147
Lab 51	119.0	1.943	0.086	0.044	46.0	1.127	0.078	0.069	93.0	1.150	0.148	0.129	35.0	1.174	0.081	0.069
Lab 52									128.0	1.582	0.100	0.063	117.0	3.926	0.103	0.026
Lab 54					19.8	0.485	0.080	0.165	44.2	0.546	0.086	0.158				
Lab 61					44.5	1.091	0.078	0.071	87.5	1.082	0.084	0.078	30.5	1.023	0.100	0.098
Lab 63													30.1	1.010	0.213	0.211
Lab 65					44.3	1.086	0.075	0.069	102.2	1.263	0.133	0.105	17.4	0.584	0.172	0.295

ANNEX VII: INFORMATION ABOUT ANALYTICAL METHODS

Table 12: Methods - part 1

Lab-code	Pesticide	Analytical Procedure	Sample weight In g	Extraction Solvent	pH adjustment	Clean up step	Clean up step other specification	RL mg/kg	Determination technique
Lab 01	Dithiocarbamates expressed as CS2	SnCl2/HCl cleavage, CS2-derivatisation/photometric analysis	200					0.05	Photometer
	2,4-D (free acid only)							0.01	
	Fluazifop (free acid only)								
	Haloxyfop (free acid only)								
	MCPA (free acid only)	Extraction with ethanol, l/l-clean-up and derivatization with PFB	20	ethanol		LL		0.01	GC-Q-MS
	MCPP (free acid only)							0.01	
	Abamectin (only Avermectin B1a)								
	Propamocarb	Methanol extraction after addition of some water	20	methanol		NO		0.02	LC-MS
Lab 02	Dithiocarbamates expressed as CS2	SnCl2/HCl cleavage, CS2-derivatisation/photometric analysis	15	HCL	yes, pH 3	NO		0. 05	Photometer
	2,4-D (free acid only)								
	Fluazifop (free acid only)								
	Haloxyfop (free acid only)								
	MCPA (free acid only)								
	MCPP (free acid only)								
	Abamectin (only Avermectin B1a)								
	Propamocarb								
Lab 03	Dithiocarbamates expressed as CS2	M008	50	H2O, HCL, isooctane		NO		0.025	GC-FPD
	2,4-D (free acid only)	M202	75	ethyl acetate	yes, pH 1	NO		0.01	LC-MS/MS
	Fluazifop (free acid only)	M202	75	ethyl acetate	yes, pH 1	NO		0.01	LC-MS/MS
	Haloxyfop (free acid only)	M202	75	ethyl acetate		NO		0.01	LC-MS/MS
	MCPA (free acid only)	M202	75	ethyl acetate	yes, pH 1	NO		0.01	LC-MS/MS
	MCPP (free acid only)	M202	75	ethyl acetate	yes, pH 1	NO		0.01	LC-MS/MS
	Abamectin (only Avermectin B1a)	M200	75	ethyl acetate		NO		0.01	LC-MS/MS
	Propamocarb	M200	75	ethyl acetate		NO		0.01	LC-MS/MS
Lab 04	Dithiocarbamates expressed as CS2	SnCl2/HCl cleavage, CS2-derivatisation/photometric analysis	50					0.05	Photometer
	2,4-D (free acid only)								
	Fluazifop (free acid only)								
	Haloxyfop (free acid only)								
	MCPA (free acid only)								
	MCPP (free acid only)								
	Abamectin (only Avermectin B1a)								
	Propamocarb								
Lab 05	Dithiocarbamates expressed as CS2	SnCl2/HCl cleavage, Headspace-GC of CS2	25			NO		0. 05	GC-FPD
	2,4-D (free acid only)		15	ethyl acetate	yes, pH 2	NO		0. 05	GC-ECD
	Fluazifop (free acid only)		15	ethyl acetate	yes, pH 2	NO		0. 05	GC-ECD
	Haloxyfop (free acid only)		15	ethyl acetate	yes, pH 2	NO		0. 05	GC-ECD
	MCPA (free acid only)		15	ethyl acetate	yes, pH 2	NO		0. 05	GC-ECD
	MCPP (free acid only)		15	ethyl acetate	yes, pH 2	NO		0. 05	GC-ECD
	Abamectin (only Avermectin B1a)	Analytical Methods for Pesticide Residues in Foodstuffs. Ministry of Welfare, health and cultural affairs, Netherlands, Multiresidue Method 1, 3.1.2, 6th Ed, 1996	15	ethyl acetate		NO		0. 05	HPLC-DAD

Lab-code	Pesticide	Analytical Procedure	Sam-ple weight In g	Extraction Solvent	pH adjustment	Clean up step	Clean up step other specifica-tion	RL mg/kg	Determi-nation technique
	Propamocarb	Analytical Methods for Pesticide Residues in Foodstuffs. Ministry of Welfare, health and cultural affairs, Netherlands, Multiresidue Method 1, 3.1.2, 6th Ed, 1996	15	ethyl acetate		NO		0. 05	GC-NPD
Lab 06	Dithiocarbamates expressed as CS2	PN-EN 12396	200			NO		0.25	Photometer
	2,4-D (free acid only)								
	Fluazifop (free acid only)								
	Haloxyfop (free acid only)								
	MCPA (free acid only)								
	MCPP (free acid only)								
	Abamectin (only Avermectin B1a)								
	Propamocarb								
Lab 07	Dithiocarbamates expressed as CS2	PN-EN 12396-3:2002	50	SnCl2x2H2O/HCl/H2O		NO		0.01	Photometer
	2,4-D (free acid only)								
	Fluazifop (free acid only)								
	Haloxyfop (free acid only)								
	MCPA (free acid only)								
	MCPP (free acid only)								
	Abamectin (only Avermectin B1a)								
	Propamocarb								
Lab 08	Dithiocarbamates expressed as CS2	SnCl2/HCl cleavage, Headspace-GC of CS2	50			NO		0. 02	GC-Q-MS
	2,4-D (free acid only)								
	Fluazifop (free acid only)	Klein, J., Alder, L. JAOAC 86, 1015 (2003); prEN 15637, ChemElut-method	10	methanol		SPE		0.01	LC-MS/MS
	Haloxyfop (free acid only)	Klein, J., Alder, L. JAOAC 86, 1015 (2003); prEN 15637, ChemElut-method	10	methanol		SPE		0.01	LC-MS/MS
	MCPA (free acid only)								
	MCPP (free acid only)								
	Abamectin (only Avermectin B1a)	Klein, J., Alder, L. JAOAC 86, 1015 (2003); prEN 15637, ChemElut-method	10	methanol		SPE		0.03	LC-MS/MS
	Propamocarb	Klein, J., Alder, L. JAOAC 86, 1015 (2003); prEN 15637, ChemElut-method	10	methanol		SPE		0.01	LC-MS/MS
Lab 09	Dithiocarbamates expressed as CS2	SnCl2/HCl cleavage, liquid-liquid-partitioning, GC-analysis of CS2	50	ioso-octane		NO		0.05	GC-FPD
	2,4-D (free acid only)								
	Fluazifop (free acid only)								
	Haloxyfop (free acid only)								
	MCPA (free acid only)	in-house	10	acetonitrile	yes, pH 2	SPE		0.02	GC-Q-MS
	MCPP (free acid only)								
	Abamectin (only Avermectin B1a)	prEN 15662, citrate-buffered QuEChERS-method	5	acetonitrile		NO		0.01	LC-MS/MS
	Propamocarb	prEN 15662, citrate-buffered QuEChERS-method	5	acetonitrile		NO		0.01	LC-MS/MS
Lab 10	Dithiocarbamates expressed as CS2	SnCl2/HCl cleavage, isooctane extraction	50	isooctane				0.1	GC-FPD
	2,4-D (free acid only)								
	Fluazifop (free acid only)								
	Haloxyfop (free acid only)								
	MCPA (free acid only)								
	MCPP (free acid only)								
	Abamectin (only Avermectin B1a)								
	Propamocarb								
Lab 11	Dithiocarbamates expressed as CS2								
	2,4-D (free acid only)								
	Fluazifop (free acid only)	Klein, J., Alder, L. JAOAC 86, 1015 (2003); prEN 15637, ChemElut-method, pH 4.5	10.0	methanol		NO	diatomaceous earth	0.01	LC-MS/MS

Lab-code	Pesticide	Analytical Procedure	Sam-ple weight In g	Extraction Solvent	pH adjustment	Clean up step	Clean up step other specifica-tion	RL mg/kg	Determi-nation technique
	Haloxyfop (free acid only)								
	MCPA (free acid only)	Klein, J., Alder, L. JAOAC 86, 1015 (2003); prEN 15637, ChemElut-method,, pH 4.5	10.0	methanol		NO	diatomaceous earth	0.01	LC-MS/MS
	MCPP (free acid only)								
	Abamectin (only Avermectin B1a)								
	Propamocarb	Klein, J., Alder, L. JAOAC 86, 1015 (2003); prEN 15637, ChemElut-method	10.0	methanol		O	diatomaceous earth	0.012	LC-MS/MS
Lab 12	Dithiocarbamates expressed as CS2	SnCl2/HCl cleavage, liquid-liquid-partitioning, GC-analysis of CS2	25	iso octane		NO		0.05	GC-Q-MS
	2,4-D (free acid only)	M. Anastassiades et al JAOAC 86 (2003) original QuEChERS-method	10	acetonitrile		NO		0.01	LC-MS/MS
	Fluazifop (free acid only)	M. Anastassiades et al JAOAC 86 (2003) original QuEChERS-method	10	acetonitrile		NO		0.01	LC-MS/MS
	Haloxyfop (free acid only)	M. Anastassiades et al JAOAC 86 (2003) original QuEChERS-method	10	acetonitrile		NO		0.01	LC-MS/MS
	MCPA (free acid only)	M. Anastassiades et al JAOAC 86 (2003) original QuEChERS-method	10	acetonitrile		NO		0.01	LC-MS/MS
	MCPP (free acid only)	M. Anastassiades et al JAOAC 86 (2003) original QuEChERS-method	10	acetonitrile		NO		0.01	LC-MS/MS
	Abamectin (only Avermectin B1a)	M. Anastassiades et al JAOAC 86 (2003) original QuEChERS-method	10	acetonitrile		NO		0.01	LC-MS/MS
	Propamocarb	M. Anastassiades et al JAOAC 86 (2003) original QuEChERS-method	10	acetonitrile		NO		0.01	LC-MS/MS
Lab 13	Dithiocarbamates expressed as CS2	SnCl2/HCl cleavage, liquid-liquid-partitioning, GC-analysis of CS2	25	iso-octane		LL		0.04	GC-ECD
	2,4-D (free acid only)			methanol				0.05	
	Fluazifop (free acid only)		10			NO		0.05	LC-MS/MS
	Haloxyfop (free acid only)							0.01	
	MCPA (free acid only)		10	methanol		NO		0.1	LC-MS/MS
	MCPP (free acid only)							0.1	
	Abamectin (only Avermectin B1a)			methanol				0.025	LC-MS/MS
	Propamocarb			methanol				0.01	LC-MS/MS
Lab 14	Dithiocarbamates expressed as CS2	SnCl2/HCl cleavage, CS2-derivatisation/photometric analysis	50					0. 01	Photometer
	2,4-D (free acid only)								
	Fluazifop (free acid only)								
	Haloxyfop (free acid only)								
	MCPA (free acid only)								
	MCPP (free acid only)								
	Abamectin (only Avermectin B1a)								
	Propamocarb								
Lab 15	Dithiocarbamates expressed as CS2	SnCl2/HCl cleavage, liquid-liquid-partitioning, GC-analysis of CS2	50	isooctane		NO		0.02	GC-FPD
	2,4-D (free acid only)	in-house method	15	modified 4 (acidified)		NO		0.01	LC-MS/MS
	Fluazifop (free acid only)	in-house method	15	modified 4 (acidified)		NO		0.01	LC-MS/MS
	Haloxyfop (free acid only)	in-house method	15	modified 4 (acidified)		NO		0.05	LC-MS/MS
	MCPA (free acid only)	in-house method	15	modified 4 (acidified)		NO		0.05	LC-MS/MS
	MCPP (free acid only)	in-house method	15	modified 4 (acidified)		NO		0.05	LC-MS/MS
	Abamectin (only Avermectin B1a)								
	Propamocarb	in-house method	15	modified 6		NO		0.01	LC-MS/MS
Lab 16	Dithiocarbamates expressed as CS2								
	2,4-D (free acid only)	M. Anastassiades et al JAOAC 86 (2003) original QuEChERS-method							
	Fluazifop (free acid only)	M. Anastassiades et al JAOAC 86 (2003) original QuEChERS-method	10	acetonitrile		NO		0. 01	LC-MS/MS
	Haloxyfop (free acid only)	M. Anastassiades et al JAOAC 86 (2003) original QuEChERS-method							
	MCPA (free acid only)	M. Anastassiades et al JAOAC 86 (2003) original QuEChERS-method	10	acetonitrile		NO		0. 01	LC-MS/MS
	MCPP (free acid only)	M. Anastassiades et al JAOAC 86 (2003) original QuEChERS-method							
	Abamectin (only Avermectin B1a)	M. Anastassiades et al JAOAC 86 (2003) original QuEChERS-method	10	acetonitrile		NO		0. 01	LC-MS/MS

Lab-code	Pesticide	Analytical Procedure	Sam-ple weight In g	Extraction Solvent	pH adjustment	Clean up step	Clean up step other specifica-tion	RL mg/kg	Determi-nation technique
	Propamocarb	M. Anastassiades et al JAOAC 86 (2003) original QuEChERS-method	10	acetonitrile		NO		0. 01	LC-MS/MS
Lab 17	Dithiocarbamates expressed as CS2	SnCl2/HCl cleavage, Headspace-GC of CS2	25	none		NO		0.01	GC-Q-MS
	2,4-D (free acid only)								
	Fluazifop (free acid only)								
	Haloxyfop (free acid only)								
	MCPA (free acid only)								
	MCPP (free acid only)								
	Abamectin (only Avermectin B1a)								
	Propamocarb								
Lab 18	Dithiocarbamates expressed as CS2								
	2,4-D (free acid only)	Analytical Methods for Pesticide Residues in Foodstuffs. Ministry of Welfare, health and cultural affairs, Netherlands, Multiresidue Method 1, 3.1.2, 6th Ed, 1996	10	acetone followed by dichloro-methane and petroleum ether		NO		0.05	LC-MS/MS
	Fluazifop (free acid only)								
	Haloxyfop (free acid only)	Analytical Methods for Pesticide Residues in Foodstuffs. Ministry of Welfare, health and cultural affairs, Netherlands, Multiresidue Method 1, 3.1.2, 6th Ed, 1996	10	acetone followed by dichloro-methane and petroleum ether		NO		0.05	LC-MS/MS
	MCPA (free acid only)	Analytical Methods for Pesticide Residues in Foodstuffs. Ministry of Welfare, health and cultural affairs, Netherlands, Multiresidue Method 1, 3.1.2, 6th Ed, 1996	10	acetone followed by dichloro-methane and petroleum ether		NO		0.05	LC-MS/MS
	MCPP (free acid only)	Analytical Methods for Pesticide Residues in Foodstuffs. Ministry of Welfare, health and cultural affairs, Netherlands, Multiresidue Method 1, 3.1.2, 6th Ed, 1996	10	acetone followed by dichloro-methane and petroleum ether		NO		0.05	LC-MS/MS
	Abamectin (only Avermectin B1a)								
	Propamocarb								
Lab 19	Dithiocarbamates expressed as CS2	SnCl2/HCl cleavage, Headspace-GC of CS2	5			NO		0.050	GC-ECD
	2,4-D (free acid only)								
	Fluazifop (free acid only)	Janson et al. Journal of Chromatography A 1023 (2004,9, 93-104)	20	ethyl acetate	yes, pH 6	LL		0.005	LC-MS/MS
	Haloxyfop (free acid only)								
	MCPA (free acid only)	Janson et al. Journal of Chromatography A 1023 (2004,9, 93-104)	20	ethyl acetate	yes, pH 6	LL		0.005	LC-MS/MS
	MCPP (free acid only)								
	Abamectin (only Avermectin B1a)		5	acetonitrile		SPE		0.010	LC-MS/MS
	Propamocarb	Janson et al. Journal of Chromatography A 1023 (2004,9, 93-104)	20	ethyl acetate	yes, pH 6	LL		0.010	LC-MS/MS
Lab 20	Dithiocarbamates expressed as CS2	SnCl2/HCl cleavage, liquid-liquid-partitioning, GC-analysis of CS2	50	TMP		NO		0.05	GC-Q-MS
	2,4-D (free acid only)	M. Anastassiades et al JAOAC 86 (2003) original QuEChERS-method	15	acetonitrile	yes, pH 5	DSPE		0.02	LC-MS/MS
	Fluazifop (free acid only)	M. Anastassiades et al JAOAC 86 (2003) original QuEChERS-method	15	acetonitrile	yes, pH 5	DSPE		0.01	LC-MS/MS
	Haloxyfop (free acid only)	M. Anastassiades et al JAOAC 86 (2003) original QuEChERS-method	15	acetonitrile	yes, pH 5	DSPE		0.01	LC-MS/MS
	MCPA (free acid only)	M. Anastassiades et al JAOAC 86 (2003) original QuEChERS-method	15	acetonitrile	yes, pH 5	DSPE		0.01	LC-MS/MS
	MCPP (free acid only)	M. Anastassiades et al JAOAC 86 (2003) original QuEChERS-method	15	acetonitrile	yes, pH 5	DSPE		0.01	LC-MS/MS
	Abamectin (only Avermectin B1a)								
	Propamocarb								
Lab 21	Dithiocarbamates expressed as CS2	SnCl2/HCl cleavage, CS2-derivatisation/photometric analysis	25					0.01	Photometer
	2,4-D (free acid only)								
	Fluazifop (free acid only)	prEN 15662, citrate-buffered QuEChERS-method	10	acetonitrile	yes, pH 5	NO		0.01	LC-MS/MS
	Haloxyfop (free acid only)								
	MCPA (free acid only)	prEN 15662, citrate-buffered QuEChERS-method	10	acetonitrile	yes, pH 5	NO		0.01	LC-MS/MS
	MCPP (free acid only)								
	Abamectin (only Avermectin B1a)	prEN 15662, citrate-buffered QuEChERS-method	10	acetonitrile	yes, pH 5	DSPE		0.01	LC-MS/MS
	Propamocarb	prEN 15662, citrate-buffered QuEChERS-method	10	acetonitrile	yes, pH 5	DSPE		0.01	LC-MS/MS
Lab 22	Dithiocarbamates expressed as CS2	SnCl2/HCl cleavage, CS2-derivatisation/photometric analysis modified by COSTA, unpublished work	50			O	distilation (EN 12396-1)	0.20	Photometer

Lab-code	Pesticide	Analytical Procedure	Sample weight In g	Extraction Solvent	pH adjustment	Clean up step	Clean up step other specification	RL mg/kg	Determination technique
	2,4-D (free acid only)								
	Fluazifop (free acid only)								
	Haloxyfop (free acid only)								
	MCPA (free acid only)								
	MCPP (free acid only)								
	Abamectin (only Avermectin B1a)								
	Propamocarb								
Lab 23	Dithiocarbamates expressed as CS2	EN 12396-2:1998 + Internal Procedure	50	isooctane		NO		0.05	GC-FPD
	2,4-D (free acid only)	M. Anastassiades et al JAOAC 86 (2003) original QuEChERS-method	10	acetonitrile		SPE		0.005	LC-MS/MS
	Fluazifop (free acid only)	M. Anastassiades et al JAOAC 86 (2003) original QuEChERS-method	10	acetonitrile		SPE		0.005	LC-MS/MS
	Haloxyfop (free acid only)	M. Anastassiades et al JAOAC 86 (2003) original QuEChERS-method	10	acetonitrile		SPE		0.005	LC-MS/MS
	MCPA (free acid only)	M. Anastassiades et al JAOAC 86 (2003) original QuEChERS-method	10	acetonitrile		SPE		0.005	LC-MS/MS
	MCPP (free acid only)	M. Anastassiades et al JAOAC 86 (2003) original QuEChERS-method	10	acetonitrile		SPE		0.005	LC-MS/MS
	Abamectin (only Avermectin B1a)								
	Propamocarb	M. Anastassiades et al JAOAC 86 (2003) original QuEChERS-method	10	acetonitrile		SPE		0.005	LC-MS/MS
Lab 26	Dithiocarbamates expressed as CS2	SnCl2/HCl cleavage, Headspace-GC of CS2	10			NO		0. 02	GC-FPD
	2,4-D (free acid only)								
	Fluazifop (free acid only)	prEN 15662, citrate-buffered QuEChERS-method	10	acetonitrile		NO		0. 01	LC-MS/MS
	Haloxyfop (free acid only)								
	MCPA (free acid only)	prEN 15662, citrate-buffered QuEChERS-method	10	acetonitrile		NO		0. 01	LC-MS/MS
	MCPP (free acid only)								
	Abamectin (only Avermectin B1a)	Analytical Methods for Pesticide Residues in Foodstuffs. Ministry of Welfare, health and cultural affairs, Netherlands, Special Methods (Part II, Abamectin), 6th Ed, 1996	10	ethyl acetate		LL		0. 01	LC-MS/MS
	Propamocarb	prEN 15662, citrate-buffered QuEChERS-method	10	acetonitrile		NO		0. 02	LC-MS/MS
Lab 27	Dithiocarbamates expressed as CS2	SnCl2/HCl cleavage, CS2-derivatisation/photometric analysis	10					0. 01	Photometer
	2,4-D (free acid only)								
	Fluazifop (free acid only)								
	Haloxyfop (free acid only)								
	MCPA (free acid only)								
	MCPP (free acid only)								
	Abamectin (only Avermectin B1a)								
	Propamocarb								
Lab 28	Dithiocarbamates expressed as CS2	§ 64 LFGB Nr. L 00.00-34 (DFG-Method) S 19, former § 35 LMBG Nr. L 00.00-34 EN 12396:1998	50	isooctane		NO		0.05	GC-ECD
	2,4-D (free acid only)								
	Fluazifop (free acid only)	Analytical Methods for Pesticide Residues in Foodstuffs. Ministry of Welfare, health and cultural affairs, Netherlands, Multiresidue Method 1, 3.1.2, 6th Ed, 1996 prEN15662:2007	10	acetonitrile		NO		0.02	LC-MS/MS
	Haloxyfop (free acid only)								
	MCPA (free acid only)	Analytical Methods for Pesticide Residues in Foodstuffs. Ministry of Welfare, health and cultural affairs, Netherlands, Multiresidue Method 1, 3.1.2, 6th Ed, 1996 prEN15662:2007	10	acetonitrile		NO		0.02	LC-MS/MS
	MCPP (free acid only)								
	Abamectin (only Avermectin B1a)								

Lab-code	Pesticide	Analytical Procedure	Sam-ple weight In g	Extraction Solvent	pH adjustment	Clean up step	Clean up step other specifica-tion	RL mg/kg	Determi-nation technique
	Propamocarb	Analytical Methods for Pesticide Residues in Foodstuffs. Ministry of Welfare, health and cultural affairs, Netherlands, Multiresidue Method 1, 3.1.2, 6th Ed, 1996 prEN15662:2007	10	acetonitrile		NO		0.02	LC-MS/MS
Lab 29	Dithiocarbamates expressed as CS2	SnCl2/HCl cleavage, Headspace-GC of CS2	50					0.05	GC-FPD
	2,4-D (free acid only)	prEN 15662, citrate-buffered QuEChERS-method	10	acetonitrile	yes, pH 5	DSPE		0.05	LC-MS/MS
	Fluazifop (free acid only)	prEN 15662, citrate-buffered QuEChERS-method	10	acetonitrile	yes, pH 5	DSPE		0.05	LC-MS/MS
	Haloxyfop (free acid only)	prEN 15662, citrate-buffered QuEChERS-method	10	acetonitrile	yes, pH 5	DSPE		0.05	LC-MS/MS
	MCPA (free acid only)	prEN 15662, citrate-buffered QuEChERS-method	10	acetonitrile	yes, pH 5	DSPE		0.05	LC-MS/MS
	MCPP (free acid only)	prEN 15662, citrate-buffered QuEChERS-method	10	acetonitrile	yes, pH 5	DSPE		0.05	LC-MS/MS
	Abamectin (only Avermectin B1a)	prEN 15662, citrate-buffered QuEChERS-method	10	acetonitrile	yes, pH 5	DSPE		0.01	LC-MS/MS
	Propamocarb	prEN 15662, citrate-buffered QuEChERS-method	10	acetonitrile	yes, pH 5	DSPE		0.05	LC-MS/MS
Lab 30	Dithiocarbamates expressed as CS2	Other method (specify below, OBLIGATORY)	49.89			NO		0.4	Photometer
	2,4-D (free acid only)	M. Anastassiades et al JAOAC 86 (2003) original QuEChERS-method	9.27	acetonitrile	yes, pH 2	NO		0.05	
	Fluazifop (free acid only)	M. Anastassiades et al JAOAC 86 (2003) original QuEChERS-method	9.27	acetonitrile	yes, pH 2	NO		0.05	
	Haloxyfop (free acid only)	M. Anastassiades et al JAOAC 86 (2003) original QuEChERS-method	9.27	acetonitrile	yes, pH 2	NO		0.05	
	MCPA (free acid only)	M. Anastassiades et al JAOAC 86 (2003) original QuEChERS-method	9.27	acetonitrile	yes, pH 2	NO		0.05	
	MCPP (free acid only)	M. Anastassiades et al JAOAC 86 (2003) original QuEChERS-method	9.27	acetonitrile	yes, pH 2	NO		0.05	
	Abamectin (only Avermectin B1a)								
	Propamocarb	M. Anastassiades et al JAOAC 86 (2003) original QuEChERS-method	9.87	acetonitrile		NO		0.05	
Lab 31	Dithiocarbamates expressed as CS2	SnCl2/HCl cleavage, CS2-derivatisation/photometric analysis	25					0.05	Photometer
	2,4-D (free acid only)							0.01	
	Fluazifop (free acid only)	prEN 15662, citrate-buffered QuEChERS-method	10	acetonitrile	yes, pH 5	NO		0.01	LC-MS/MS
	Haloxyfop (free acid only)							0.01	
	MCPA (free acid only)	prEN 15662, citrate-buffered QuEChERS-method	10	acetonitrile	yes, pH 5	NO		0.01	LC-MS/MS
	MCPP (free acid only)							0.01	
	Abamectin (only Avermectin B1a)	prEN 15662, citrate-buffered QuEChERS-method	10	acetonitrile	yes, pH 5	DSPE		0.01	LC-MS/MS
	Propamocarb	prEN 15662, citrate-buffered QuEChERS-method	10	acetonitrile	yes, pH 5	DSPE		0.01	LC-MS/MS
Lab 32	Dithiocarbamates expressed as CS2	SnCl2/HCl cleavage, liquid-liquid-partitioning, GC-analysis of CS2	50	SnCl2/HCl/H2O		NO		0.04	GC-Q-MS
	2,4-D (free acid only)	prEN 15662, citrate-buffered QuEChERS-method	10	acetonitrile		NO		0.01	LC-MS/MS
	Fluazifop (free acid only)	prEN 15662, citrate-buffered QuEChERS-method	10	acetonitrile		NO		0.01	LC-MS/MS
	Haloxyfop (free acid only)	prEN 15662, citrate-buffered QuEChERS-method	10	acetonitrile		NO		0.01	LC-MS/MS
	MCPA (free acid only)	prEN 15662, citrate-buffered QuEChERS-method	10	acetonitrile		NO		0.01	LC-MS/MS
	MCPP (free acid only)	prEN 15662, citrate-buffered QuEChERS-method	10	acetonitrile		NO		0.01	LC-MS/MS
	Abamectin (only Avermectin B1a)	Klein, J., Alder, L. JAOAC 86, 1015 (2003); prEN 15637, ChemElut-method	10	methanol		LL		0.01	LC-MS/MS
	Propamocarb	VNORM EN 15055	10	methanol		NO		0.01	LC-MS/MS
Lab 33	Dithiocarbamates expressed as CS2	FP019	100	Cullens				0.1	Photometer
	2,4-D (free acid only)	FP086	10	methanol		O	filter	0.05	LC-MS/MS
	Fluazifop (free acid only)	FP086	10	methanol		O	filter	0.05	LC-MS/MS
	Haloxyfop (free acid only)								
	MCPA (free acid only)	FP086	10	methanol		O	filter	0.05	LC-MS/MS
	MCPP (free acid only)	FP0866	10	methanol		O	filter	0.05	LC-MS/MS
	Abamectin (only Avermectin B1a)								
	Propamocarb	FP086	10	methanol		O	filter	0.05	LC-MS/MS
Lab 34	Dithiocarbamates expressed as CS2								
	2,4-D (free acid only)							0.010	
	Fluazifop (free acid only)	M. Anastassiades et al JAOAC 86 (2003) original QuEChERS-method	10	acetonitrile				0.010	LC-MS/MS

Lab-code	Pesticide	Analytical Procedure	Sam-ple weight In g	Extraction Solvent	pH adjustment	Clean up step	Clean up step other specifica-tion	RL mg/kg	Determi-nation technique
	Haloxyfop (free acid only)							0.005	
	MCPA (free acid only)	M. Anastassiades et al JAOAC 86 (2003) original QuEChERS-method	10	acetonitrile				0.005	LC-MS/MS
	MCPP (free acid only)							0.005	
	Abamectin (only Avermectin B1a)	M. Anastassiades et al JAOAC 86 (2003) original QuEChERS-method	10	acetonitrile				0.010	LC-MS/MS
	Propamocarb	M. Anastassiades et al JAOAC 86 (2003) original QuEChERS-method	10	acetonitrile				0.010	LC-MS/MS
Lab 35	Dithiocarbamates expressed as CS2	SnCl2/HCl cleavage, CS2-derivatisation/photometric analysis	200			NO		0.05	Photometer
	2,4-D (free acid only)								
	Fluazifop (free acid only)								
	Haloxyfop (free acid only)								
	MCPA (free acid only)								
	MCPP (free acid only)								
	Abamectin (only Avermectin B1a)								
	Propamocarb								
Lab 36	Dithiocarbamates expressed as CS2	SnCl2/HCl cleavage, Headspace-GC of CS2	50					0.05	GC-FPD
	2,4-D (free acid only)								
	Fluazifop (free acid only)	M. Anastassiades et al JAOAC 86 (2003) original QuEChERS-method	15	acetonitrile	yes, pH 2	NO		0.05	GC-ITD-MS/MS
	Haloxyfop (free acid only)								
	MCPA (free acid only)	M. Anastassiades et al JAOAC 86 (2003) original QuEChERS-method	15	acetonitrile	yes, pH 2	NO		0.05	GC-ITD-MS/MS
	MCPP (free acid only)								
	Abamectin (only Avermectin B1a)	M. Anastassiades et al JAOAC 86 (2003) original QuEChERS-method	15	acetonitrile		NO		0.05	HPLC-FL
	Propamocarb	MeOH/HCl	10	MeOH/HCl	Yes, twice (acid/base wash)	NO		0.05	GC-ITD-MS/MS
Lab 37	Dithiocarbamates expressed as CS2	SnCl2/HCl cleavage, CS2-derivatisation/photometric analysis	100					0.1	Photometer
	2,4-D (free acid only)	M. Anastassiades et al JAOAC 86 (2003) original QuEChERS-method	10	acetonitrile		NO		0.01	LC-MS/MS
	Fluazifop (free acid only)	M. Anastassiades et al JAOAC 86 (2003) original QuEChERS-method	10	acetonitrile		NO		0.01	LC-MS/MS
	Haloxyfop (free acid only)	M. Anastassiades et al JAOAC 86 (2003) original QuEChERS-method	10	acetonitrile		NO		0.01	LC-MS/MS
	MCPA (free acid only)	M. Anastassiades et al JAOAC 86 (2003) original QuEChERS-method	10	acetonitrile		NO		0.01	LC-MS/MS
	MCPP (free acid only)	M. Anastassiades et al JAOAC 86 (2003) original QuEChERS-method	10	acetonitrile		NO		0.01	LC-MS/MS
	Abamectin (only Avermectin B1a)								
	Propamocarb	M. Anastassiades et al JAOAC 86 (2003) original QuEChERS-method	10	acetonitrile		NO		0.01	LC-MS/MS
Lab 38	Dithiocarbamates expressed as CS2	L00.00-49/3	40						
	2,4-D (free acid only)								
	Fluazifop (free acid only)	Klein, J., Alder, L. JAOAC 86, 1015 (2003); prEN 15637, ChemElut-method Chem Elut pH4.5	25	methanol		LL		0.01	LC-MS/MS
	Haloxyfop (free acid only)								
	MCPA (free acid only)	Klein, J., Alder, L. JAOAC 86, 1015 (2003); prEN 15637, ChemElut-method Chem Elut pH4.5	25	methanol		LL		0.01	LC-MS/MS
	MCPP (free acid only)								
	Abamectin (only Avermectin B1a)	Klein, J., Alder, L. JAOAC 86, 1015 (2003); prEN 15637, ChemElut-method Chem Elut pH4.5	25	methanol		LL		0.01	LC-MS/MS
	Propamocarb	Klein, J., Alder, L. JAOAC 86, 1015 (2003); prEN 15637, ChemElut-method Chem Elut pH4.5	25	methanol		LL		0.01	LC-MS/MS
Lab 39	Dithiocarbamates expressed as CS2								
	2,4-D (free acid only)								

Lab-code	Pesticide	Analytical Procedure	Sam-ple weight In g	Extraction Solvent	pH adjustment	Clean up step	Clean up step other specifica-tion	RL mg/kg	Determi-nation technique
	Fluazifop (free acid only)								
	Haloxyfop (free acid only)								
	MCPA (free acid only)	Other method (specify below, OBLIGATORY)	10	ammonia acetate - acetic acid 20mM in methanol-water (95:5)	NO		0.02	LC-MS/MS	
	MCPP (free acid only)								
	Abamectin (only Avermectin B1a)								
	Propamocarb	Other method (specify below, OBLIGATORY)	10	ammonia acetate - acetic acid 20mM in methanol-water (95:5)	NO		0.015	LC-MS/MS	
Lab 40	Dithiocarbamates expressed as CS2	SnCl2/HCl cleavage, CS2-derivatisation/photometric analysis	100					0.05	Photometer
	2,4-D (free acid only)								
	Fluazifop (free acid only)	Analytical Methods for Pesticide Residues in Foodstuffs. Ministry of Welfare, health and cultural affairs, Netherlands, Multiresidue Method 1, 3.1.2, 6th Ed, 1996	10	acetone followed by dichloro-methane and petroleum ether		NO		0.01	LC-MS/MS
	Haloxyfop (free acid only)	Analytical Methods for Pesticide Residues in Foodstuffs. Ministry of Welfare, health and cultural affairs, Netherlands, Multiresidue Method 1, 3.1.2, 6th Ed, 1996	10	acetone followed by dichloro-methane and petroleum ether				0.01	LC-MS/MS
	MCPA (free acid only)								
	MCPP (free acid only)								
	Abamectin (only Avermectin B1a)	Analytical Methods for Pesticide Residues in Foodstuffs. Ministry of Welfare, health and cultural affairs, Netherlands, Multiresidue Method 1, 3.1.2, 6th Ed, 1996	10	methanol				0.05	LC-MS/MS
	Propamocarb	Analytical Methods for Pesticide Residues in Foodstuffs. Ministry of Welfare, health and cultural affairs, Netherlands, Multiresidue Method 1, 3.1.2, 6th Ed, 1996	10	methanol				0.01	LC-MS/MS
Lab 41	Dithiocarbamates expressed as CS2								
	2,4-D (free acid only)	Quechers with 1% acetic acid and no PSA addition	2.5	Acetonitril with 1% Acetic Acid	NO		0.025	LC-MS/MS	
	Fluazifop (free acid only)	Quechers with 1% acetic acid and no PSA addition	2.5	Acetonitril with 1% Acetic Acid	NO		0.01	LC-MS/MS	
	Haloxyfop (free acid only)	Quechers with 1% acetic acid and no PSA addition	2.5	Acetonitril with 1% Acetic Acid	NO		0.01	LC-MS/MS	
	MCPA (free acid only)	Quechers with 1% acetic acid and no PSA addition	2.5	Acetonitril with 1% Acetic Acid	NO		0.01	LC-MS/MS	
	MCPP (free acid only)	Quechers with 1% acetic acid and no PSA addition	2.5	Acetonitril with 1% Acetic Acid	NO		0.025	LC-MS/MS	
	Abamectin (only Avermectin B1a)								
	Propamocarb	Quechers with 1% acetic acid and no PSA addition	2.5	Acetonitril with 1% Acetic Acid	NO		0.01	LC-MS/MS	
Lab 42	Dithiocarbamates expressed as CS2	' 64 LFGB L00.00-49/3	50					0. 005	Photometer
	2,4-D (free acid only)	Klein, J., Alder, L. JAOAC 86, 1015 (2003); prEN 15637, ChemElut-method	10	methanol		LL		0. 05	LC-MS/MS
	Fluazifop (free acid only)	Klein, J., Alder, L. JAOAC 86, 1015 (2003); prEN 15637, ChemElut-method	10	methanol		LL		0. 01	LC-MS/MS
	Haloxyfop (free acid only)	Klein, J., Alder, L. JAOAC 86, 1015 (2003); prEN 15637, ChemElut-method	10	methanol		LL		0. 02	LC-MS/MS
	MCPA (free acid only)	Klein, J., Alder, L. JAOAC 86, 1015 (2003); prEN 15637, ChemElut-method	10	methanol		LL		0. 005	LC-MS/MS
	MCPP (free acid only)	Klein, J., Alder, L. JAOAC 86, 1015 (2003); prEN 15637, ChemElut-method	10	methanol		LL		0. 02	LC-MS/MS
	Abamectin (only Avermectin B1a)	Klein, J., Alder, L. JAOAC 86, 1015 (2003); prEN 15637, ChemElut-method	10	methanol		LL		0. 02	LC-MS/MS
	Propamocarb	Klein, J., Alder, L. JAOAC 86, 1015 (2003); prEN 15637, ChemElut-method	10	methanol		LL		0. 02	LC-MS/MS
Lab 44	Dithiocarbamates expressed as CS2	Analytical Methods for Pesticide Residues in Foodstuffs. Ministry of Welfare, health and cultural affairs, Netherlands, Multiresidue Method 1, 3.1.2, 6th Ed, 1996	5	acetone followed by cyclohexane and ethyl acetate		NO		0.05	GC-FPD
	2,4-D (free acid only)	§ 64 LFGB Nr. L 00.00-34 (DFG-Method) S 19, former § 35 LMBG Nr. L 00.00-34	10	ethyl acetate		NO		0.01	LC-MS/MS
	Fluazifop (free acid only)	§ 64 LFGB Nr. L 00.00-34 (DFG-Method) S 19, former § 35 LMBG Nr. L 00.00-34	10	ethyl acetate		NO		0.01	LC-MS/MS
	Haloxyfop (free acid only)	§ 64 LFGB Nr. L 00.00-34 (DFG-Method) S 19, former § 35 LMBG Nr. L 00.00-34	10	ethyl acetate		NO		0.01	LC-MS/MS

Lab-code	Pesticide	Analytical Procedure	Sam-ple weight In g	Extraction Solvent	pH adjustment	Clean up step	Clean up step other specifica-tion	RL mg/kg	Determi-nation technique
	MCPA (free acid only)	§ 64 LFGB Nr. L 00.00-34 (DFG-Method) S 19, former § 35 LMBG Nr. L 00.00-34	10	ethyl acetate		NO		0.01	LC-MS/MS
	MCPP (free acid only)								
	Abamectin (only Avermectin B1a)								
	Propamocarb								
Lab 45	Dithiocarbamates expressed as CS2	Spectrometric	200.0	water, acid				0.34	Other
	2,4-D (free acid only)								
	Fluazifop (free acid only)	SOP	10.0	methanol		LL		0.01	LC-MS/MS
	Haloxyfop (free acid only)								
	MCPA (free acid only)	SOP	10.0	methanol		LL		0.01	LC-MS/MS
	MCPP (free acid only)								
	Abamectin (only Avermectin B1a)	SOP	10.0	methanol		LL		0.01	LC-MS/MS
	Propamocarb	SOP	10.0	methanol		LL		0.01	LC-MS/MS
Lab 46	Dithiocarbamates expressed as CS2	SnCl2/HCl cleavage, CS2-derivatisation/photometric analysis	50			NO		0.2	Photometer
	2,4-D (free acid only)								
	Fluazifop (free acid only)								
	Haloxyfop (free acid only)								
	MCPA (free acid only)								
	MCPP (free acid only)								
	Abamectin (only Avermectin B1a)								
	Propamocarb								
Lab 47	Dithiocarbamates expressed as CS2	SnCl2/HCl cleavage, CS2-derivatisation/photometric analysis	200					0.1	Photometer
	2,4-D (free acid only)								
	Fluazifop (free acid only)	prEN 15662, citrate-buffered QuEChERS-method	10	acetonitrile	yes, pH 5	NO		0.01	LC-MS/MS
	Haloxyfop (free acid only)								
	MCPA (free acid only)	prEN 15662, citrate-buffered QuEChERS-method	10	acetonitrile	yes, pH 5	NO		0.01	LC-MS/MS
	MCPP (free acid only)								
	Abamectin (only Avermectin B1a)	prEN 15662, citrate-buffered QuEChERS-method	10	acetonitrile	yes, pH 5	NO		0.01	LC-MS/MS
	Propamocarb	prEN 15662, citrate-buffered QuEChERS-method	10	acetonitrile	yes, pH 5	NO		0.01	LC-MS/MS
Lab 48	Dithiocarbamates expressed as CS2	SnCl2/HCl cleavage, CS2-derivatisation/photometric analysis	50					0.50	Photometer
	2,4-D (free acid only)							0.05	
	Fluazifop (free acid only)		10	acetonitrile	yes, pH 6			0.05	LC-MS/MS
	Haloxyfop (free acid only)								
	MCPA (free acid only)	M. Anastassiades et al JAOAC 86 (2003) original QuEChERS-method	10	acetonitrile	Yes, twice (acid/base wash)			0.05	LC-MS/MS
	MCPP (free acid only)								
	Abamectin (only Avermectin B1a)	M. Anastassiades et al JAOAC 86 (2003) original QuEChERS-method	10	acetonitrile				0.05	LC-MS/MS
	Propamocarb	M. Anastassiades et al JAOAC 86 (2003) original QuEChERS-method	10	acetonitrile				0.05	LC-MS/MS
Lab 49	Dithiocarbamates expressed as CS2	SnCl2/HCl cleavage, Headspace-GC of CS2	50			NO		0. 1	GC-ECD
	2,4-D (free acid only)								
	Fluazifop (free acid only)								
	Haloxyfop (free acid only)								
	MCPA (free acid only)								
	MCPP (free acid only)								
	Abamectin (only Avermectin B1a)								
	Propamocarb								
Lab 50	Dithiocarbamates expressed as CS2	SnCl2/HCl cleavage, CS2-derivatisation/photometric analysis	100	other (specify which)				0.05	Photometer

Lab-code	Pesticide	Analytical Procedure	Sample weight In g	Extraction Solvent	pH adjustment	Clean up step	Clean up step other specification	RL mg/kg	Determination technique
	2,4-D (free acid only)							0.05	
	Fluazifop (free acid only)	prEN 15662, citrate-buffered QuEChERS-method	10	acetonitrile	yes, pH 5	GPC		0.05	GC-ECD
	Haloxyfop (free acid only)							0.05	
	MCPA (free acid only)	prEN 15662, citrate-buffered QuEChERS-method	10	acetonitrile	yes, pH 5	GPC		0.05	GC-ECD
	MCPP (free acid only)							0.05	
	Abamectin (only Avermectin B1a)	prEN 15662, citrate-buffered QuEChERS-method	10	acetonitrile	yes, pH 5	GPC		0.01	HPLC-FL
	Propamocarb	prEN 15662, citrate-buffered QuEChERS-method	10	acetonitrile	yes, pH 5	NO		0.05	GC-NPD
Lab 51	Dithiocarbamates expressed as CS2	SnCl2/HCl cleavage, CS2-derivatisation/photometric analysis	50			NO		0.2	Photometer
	2,4-D (free acid only)	prEN 15662, citrate-buffered QuEChERS-method	10			LL		0.01	LC-MS/MS
	Fluazifop (free acid only)	prEN 15662, citrate-buffered QuEChERS-method	10			LL		0.01	LC-MS/MS
	Haloxyfop (free acid only)	prEN 15662, citrate-buffered QuEChERS-method	10			LL		0.01	LC-MS/MS
	MCPA (free acid only)	prEN 15662, citrate-buffered QuEChERS-method	10			LL		0.01	LC-MS/MS
	MCPP (free acid only)	prEN 15662, citrate-buffered QuEChERS-method	10			LL		0.01	LC-MS/MS
	Abamectin (only Avermectin B1a)	prEN 15662, citrate-buffered QuEChERS-method	15			DSPE	PSA	0.01	LC-MS/MS
	Propamocarb	prEN 15662, citrate-buffered QuEChERS-method	15			DSPE	PSA	0.01	LC-MS/MS
Lab 52	Dithiocarbamates expressed as CS2	SnCl2/HCl cleavage, Headspace-GC of CS2	50	acetone followed by dichloro-methane and petroleum ether		NO		0.05	GC-ECD
	2,4-D (free acid only)	M. Anastassiades et al JAOAC 86 (2003) original QuEChERS-method	10	acetonitrile		DSPE		0.05	LC-MS/MS
	Fluazifop (free acid only)								
	Haloxyfop (free acid only)	M. Anastassiades et al JAOAC 86 (2003) original QuEChERS-method	10	acetonitrile		DSPE		0.05	LC-MS/MS
	MCPA (free acid only)	M. Anastassiades et al JAOAC 86 (2003) original QuEChERS-method	10	acetonitrile		DSPE		0.05	LC-MS/MS
	MCPP (free acid only)	M. Anastassiades et al JAOAC 86 (2003) original QuEChERS-method	10	acetonitrile		DSPE		0.05	LC-MS/MS
	Abamectin (only Avermectin B1a)								
	Propamocarb	M. Anastassiades et al JAOAC 86 (2003) original QuEChERS-method	10	acetonitrile		DSPE		0.05	LC-MS/MS
Lab 53	Dithiocarbamates expressed as CS2		50	SCl2 in 5M HCl	yes, pH 1	LL	conversion to trimethylpentane	0.05	GC-Q-MS
	2,4-D (free acid only)								
	Fluazifop (free acid only)								
	Haloxyfop (free acid only)								
	MCPA (free acid only)								
	MCPP (free acid only)								
	Abamectin (only Avermectin B1a)								
	Propamocarb								
Lab 54	Dithiocarbamates expressed as CS2	SnCl2/HCl cleavage, CS2-derivatisation/photometric analysis	30					0.02	Photometer
	2,4-D (free acid only)	ILM-BASF 80/10055- method ?173	50	methanol	Yes, twice (acid/base wash)	LL		0.05	GC-Q-MS
	Fluazifop (free acid only)	ILM-BASF 80/10055- method ?173	50	methanol	Yes, twice (acid/base wash)	LL		0.05	GC-Q-MS
	Haloxyfop (free acid only)	ILM-BASF 80/10055- method ?173	50	methanol	Yes, twice (acid/base wash)	LL		0.05	GC-Q-MS
	MCPA (free acid only)	ILM-BASF 80/10055- method ?173	50	methanol	Yes, twice (acid/base wash)	LL		0.05	GC-Q-MS

Lab-code	Pesticide	Analytical Procedure	Sam-ple weight In g	Extraction Solvent	pH adjustment	Clean up step	Clean up step other specifica-tion	RL mg/kg	Determi-nation technique
	MCPP (free acid only)	ILM-BASF 80/10055- method ?173	50	methanol	Yes, twice (acid/base wash)	LL		0.05	GC-Q-MS
	Abamectin (only Avermectin B1a)								
	Propamocarb								
Lab 55	Dithiocarbamates expressed as CS2	SnCl2/HCl cleavage, CS2-derivatisation/photometric analysis	200					0.3	Photometer
	2,4-D (free acid only)								
	Fluazifop (free acid only)								
	Haloxyfop (free acid only)								
	MCPA (free acid only)								
	MCPP (free acid only)								
	Abamectin (only Avermectin B1a)								
	Propamocarb								
Lab 56	Dithiocarbamates expressed as CS2	SnCl2/HCl cleavage, CS2-derivatisation/photometric analysis	100			NO		0.1	Photometer
	2,4-D (free acid only)								
	Fluazifop (free acid only)								
	Haloxyfop (free acid only)								
	MCPA (free acid only)								
	MCPP (free acid only)								
	Abamectin (only Avermectin B1a)								
	Propamocarb								
Lab 57	Dithiocarbamates expressed as CS2	SnCl2/HCl cleavage, CS2-derivatisation/photometric analysis	25	toluene		NO		0.03	GC-FPD
	2,4-D (free acid only)								
	Fluazifop (free acid only)								
	Haloxyfop (free acid only)								
	MCPA (free acid only)								
	MCPP (free acid only)								
	Abamectin (only Avermectin B1a)								
	Propamocarb								
Lab 59	Dithiocarbamates expressed as CS2	SnCl2/HCl cleavage, liquid-liquid-partitioning, GC-analysis of CS2	25	isooctane		NO		0.05	GC-ECD
	2,4-D (free acid only)								
	Fluazifop (free acid only)								
	Haloxyfop (free acid only)								
	MCPA (free acid only)								
	MCPP (free acid only)								
	Abamectin (only Avermectin B1a)								
	Propamocarb								
Lab 60	Dithiocarbamates expressed as CS2	EN 12396-1	100			NO		0. 1	Other
	2,4-D (free acid only)								
	Fluazifop (free acid only)								
	Haloxyfop (free acid only)								
	MCPA (free acid only)								
	MCPP (free acid only)								
	Abamectin (only Avermectin B1a)								
	Propamocarb	Klein, J., Alder, L. JAOAC 86, 1015 (2003); prEN 15637, ChemElut-method	5,0	methanol		LL	with dichloro-methane	0.05	LC-MS/MS

Lab-code	Pesticide	Analytical Procedure	Sam-ple weight In g	Extraction Solvent	pH adjustment	Clean up step	Clean up step other specifica-tion	RL mg/kg	Determi-nation technique
Lab 61	Dithiocarbamates expressed as CS2						with dichloro-methane		
	2,4-D (free acid only)	Klein, J., Alder, L. JAOAC 86, 1015 (2003); prEN 15637, ChemElut-method use of 3-ml-chem-Elut cartridges pH 4,5	10.0	methanol		LL	with dichloro-methane	0.05	LC-MS/MS
	Fluazifop (free acid only)	Klein, J., Alder, L. JAOAC 86, 1015 (2003); prEN 15637, ChemElut-method use of 3-ml-chem-Elut cartridges pH 4,5	5.0	methanol		LL	with dichloro-methane	0.05	LC-MS/MS
	Haloxyfop (free acid only)	Klein, J., Alder, L. JAOAC 86, 1015 (2003); prEN 15637, ChemElut-method use of 3-ml-chem-Elut cartridges pH 4,5	10.0	methanol		LL	with dichloro-methane	0.05	LC-MS/MS
	MCPA (free acid only)	Klein, J., Alder, L. JAOAC 86, 1015 (2003); prEN 15637, ChemElut-method use of 3-ml-chem-Elut cartridges pH 4,5	5.0	methanol		LL	with dichloro-methane	0.05	LC-MS/MS
	MCPP (free acid only)	Klein, J., Alder, L. JAOAC 86, 1015 (2003); prEN 15637, ChemElut-method use of 3-ml-chem-Elut cartridges pH 4,5	10.0	methanol		LL	with dichloro-methane	0.05	LC-MS/MS
	Abamectin (only Avermectin B1a)								
	Propamocarb	Klein, J., Alder, L. JAOAC 86, 1015 (2003); prEN 15637, ChemElut-method	5.0	methanol		LL	with dichloro-methane	0.05	LC-TOF/MS
Lab 62	Dithiocarbamates expressed as CS2	PN-EN 12396-1:2002	100	SnCl2/HCl		NO		0. 05	Photometer
	2,4-D (free acid only)								
	Fluazifop (free acid only)								
	Haloxyfop (free acid only)								
	MCPA (free acid only)								
	MCPP (free acid only)								
	Abamectin (only Avermectin B1a)								
	Propamocarb								
Lab 63	Dithiocarbamates expressed as CS2	SnCl2/HCl cleavage, liquid-liquid-partitioning, GC-analysis of CS2	25	isooctan		LL		0.05	GC-Q-MS
	2,4-D (free acid only)								
	Fluazifop (free acid only)								
	Haloxyfop (free acid only)								
	MCPA (free acid only)								
	MCPP (free acid only)								
	Abamectin (only Avermectin B1a)								
	Propamocarb	Klein, J., Alder, L. JAOAC 86, 1015 (2003); prEN 15637, ChemElut-method	10	methanol	yes, pH 6	SPE		0.01	LC-MS/MS
Lab 64	Dithiocarbamates expressed as CS2	SnCl2/HCl cleavage, Headspace-GC of CS2	50			NO		0. 05	GC-Q-MS
	2,4-D (free acid only)								
	Fluazifop (free acid only)								
	Haloxyfop (free acid only)								
	MCPA (free acid only)								
	MCPP (free acid only)								
	Abamectin (only Avermectin B1a)								
	Propamocarb								
Lab 65	Dithiocarbamates expressed as CS2	SnCl2/HCl cleavage, liquid-liquid-partitioning, GC-analysis of CS2	30	isooctane		LL		0.01	GC-Q-MS
	2,4-D (free acid only)								
	Fluazifop (free acid only)	prEN 15662, citrate-buffered QuEChERS-method	10	acetonitrile		NO		0.05	LC-MS/MS
	Haloxyfop (free acid only)								
	MCPA (free acid only)	prEN 15662, citrate-buffered QuEChERS-method	10	acetonitrile		NO		0.05	LC-MS/MS
	MCPP (free acid only)								
	Abamectin (only Avermectin B1a)								
	Propamocarb								

Lab-code	Pesticide	Analytical Procedure	Sam-ple weight In g	Extraction Solvent	pH adjustment	Clean up step	Clean up step other specifica-tion	RL mg/kg	Determi-nation technique
Lab 66	Dithiocarbamates expressed as CS2	SnCl2/HCl cleavage, liquid-liquid-partitioning, GC-analysis of CS2	4	isooctane				0.05	GC-FPD
	2,4-D (free acid only)								
	Fluazifop (free acid only)								
	Haloxyfop (free acid only)								
	MCPA (free acid only)								
	MCPP (free acid only)								
	Abamectin (only Avermectin B1a)								
	Propamocarb								
Lab 67	Dithiocarbamates expressed as CS2	PN-EN 12396-1	50,0	SnCl2/HCl		NO		0. 05	Photometer
	2,4-D (free acid only)								
	Fluazifop (free acid only)								
	Haloxyfop (free acid only)								
	MCPA (free acid only)								
	MCPP (free acid only)								
	Abamectin (only Avermectin B1a)								
	Propamocarb								
Lab 68	Dithiocarbamates expressed as CS2	SM01252	102.10	SnCl2	yes, pH 1			0.4	Other
	2,4-D (free acid only)	SM00809	15	acetonitrile				0.010	LC-MS/MS
	Fluazifop (free acid only)	SM00809	15	acetonitrile				0.010	LC-MS/MS
	Haloxyfop (free acid only)								
	MCPA (free acid only)								
	MCPP (free acid only)								
	Abamectin (only Avermectin B1a)	SM00809	15	acetonitrile				0.022	LC-MS/MS
	Propamocarb	SM00809	15	acetonitrile				0.010	LC-MS/MS
Lab 69	Dithiocarbamates expressed as CS2	SnCl2/HCl cleavage, CS2-derivatisation/photometric analysis	100			NO		0.25	Photometer
	2,4-D (free acid only)								
	Fluazifop (free acid only)								
	Haloxyfop (free acid only)								
	MCPA (free acid only)								
	MCPP (free acid only)								
	Abamectin (only Avermectin B1a)								
	Propamocarb								
Lab 70	Dithiocarbamates expressed as CS2	SnCl2/HCl cleavage, Headspace-GC of CS2	1			NO		0.03	GC-FPD
	2,4-D (free acid only)								
	Fluazifop (free acid only)								
	Haloxyfop (free acid only)								
	MCPA (free acid only)								
	MCPP (free acid only)								
	Abamectin (only Avermectin B1a)								
	Propamocarb								

Table 13: Methods - part 2

Lab-code	Pesticide Name	Deri-vati-zation	Derivatization other specification	Quantification using standards	Internal stan-dard	Injection volume (µl)	Injec-tion type	Determination technique	Reporting Level (mg/kg)	Recovery Correction	Recovery (%)	Spiked level (mg/kg)	Recovery from
Lab 01	Dithiocarbamates expressed as CS2			S				Photometer	0.05	no	100.5	0.1	from same batch
	2,4-D (free acid only)								0.01				
	Fluazifop (free acid only)												
	Haloxyfop (free acid only)												
	MCPA (free acid only)	YES	Pentafluorobenzylbromide	S	YES	5	Splitless	GC-Q-MS	0.01	no	87.7	0.05	from same batch
	MCPP (free acid only)								0.01				
	Abamectin (only Avermectin B1a)												
	Propamocarb	NO		M	NO	10		LC-MS	0.02	no	95.4	0.05	from same batch
Lab 02	Dithiocarbamates expressed as CS2	YES		M	NO	-		Photometer	0,05	no			
	2,4-D (free acid only)												
	Fluazifop (free acid only)												
	Haloxyfop (free acid only)												
	MCPA (free acid only)												
	MCPP (free acid only)												
	Abamectin (only Avermectin B1a)												
	Propamocarb												
Lab 03	Dithiocarbamates expressed as CS2	NO		M	NO	5	Splitless	GC-FPD	0.025	no	90	0.360	from same batch
	2,4-D (free acid only)	NO		M	NO	5		LC-MS/MS	0.01	no			
	Fluazifop (free acid only)	NO		M	NO	5		LC-MS/MS	0.01	no	84	0.050	from same batch
	Haloxyfop (free acid only)	NO		M	NO	5		LC-MS/MS	0.01	no			
	MCPA (free acid only)	NO		M	NO	5		LC-MS/MS	0.01	no	94	0.070	from same batch
	MCPP (free acid only)	NO		M	NO	5		LC-MS/MS	0.01	no			
	Abamectin (only Avermectin B1a)	NO		M	NO	5		LC-MS/MS	0.01	no	112	0.025	from same batch
	Propamocarb	NO		M	NO	5		LC-MS/MS	0.01	no	109	0.100	from same batch
Lab 04	Dithiocarbamates expressed as CS2							Photometer	0.05	no	80.5; 78.0	0.5; 1.0	from same batch
	2,4-D (free acid only)												
	Fluazifop (free acid only)												
	Haloxyfop (free acid only)												
	MCPA (free acid only)												
	MCPP (free acid only)												
	Abamectin (only Avermectin B1a)												
	Propamocarb												
Lab 05	Dithiocarbamates expressed as CS2	YES		S	YES	500	On-Column	GC-FPD	0,05	no	115	0. 2	from same batch
	2,4-D (free acid only)	YES		S	NO	1	Splitless	GC-ECD	0,05		91	0. 1	from same batch

Lab-code	Pesticide Name	Deri-vati-zation	Derivatization other specification	Quantification using standards	Internal stan-dard	Injection volume (µl)	Injec-tion type	Determination technique	Reporting Level (mg/kg)	Recovery Correction	Recovery (%)	Spiked level (mg/kg)	Recovery from
	Fluazifop (free acid only)	YES		S	NO	1	Splitless	GC-ECD	0,05	no	90	0. 1	from same batch
	Haloxyfop (free acid only)	YES		S	NO	1	Splitless	GC-ECD	0,05		86	0. 2	from same batch
	MCPA (free acid only)	YES		S	NO	1	Splitless	GC-ECD	0,05	yes, using a recovery rate	50	0. 1	from same batch
	MCPP (free acid only)	YES		S	NO	1	Splitless	GC-ECD	0,05		95	0. 1	from same batch
	Abamectin (only Avermectin B1a)	NO		S	NO	20		HPLC-DAD	0,05	no	110	0. 5	from same batch
	Propamocarb	NO		S	NO	1	Splitless	GC-NPD	0,05	no	79	0. 2	from same batch
Lab 06	Dithiocarbamates expressed as CS2	YES		S	NO			Photometer	0.25	yes, using a recovery rate	83	0.50	from routine QC data
	2,4-D (free acid only)												
	Fluazifop (free acid only)												
	Haloxyfop (free acid only)												
	MCPA (free acid only)												
	MCPP (free acid only)												
	Abamectin (only Avermectin B1a)												
	Propamocarb												
Lab 07	Dithiocarbamates expressed as CS2	NO		S	NO	--		Photometer	0.01	yes, using a recovery rate	88	0.80	from validation data
	2,4-D (free acid only)												
	Fluazifop (free acid only)												
	Haloxyfop (free acid only)												
	MCPA (free acid only)												
	MCPP (free acid only)												
	Abamectin (only Avermectin B1a)												
	Propamocarb												
Lab 08	Dithiocarbamates expressed as CS2			M	YES	500	Split	GC-Q-MS	0,02	no			
	2,4-D (free acid only)												
	Fluazifop (free acid only)			M	YES	25		LC-MS/MS	0.01	no			
	Haloxyfop (free acid only)			M	YES	25		LC-MS/MS	0.01	no			
	MCPA (free acid only)												
	MCPP (free acid only)												
	Abamectin (only Avermectin B1a)			M	YES	25		LC-MS/MS	0.03	no			
	Propamocarb			M	YES	25		LC-MS/MS	0.01	no			
Lab 09	Dithiocarbamates expressed as CS2	NO		S	NO	2	Splitless	GC-FPD	0.05	no	80		
	2,4-D (free acid only)												
	Fluazifop (free acid only)												
	Haloxyfop (free acid only)												
	MCPA (free acid only)	YES		M	YES	10	PTV-LVI	GC-Q-MS	0.02	no	88	0.20	from same batch
	MCPP (free acid only)												
	Abamectin (only Avermectin B1a)	NO		M	NO	10		LC-MS/MS	0.01	yes, using a recovery rate	42	0.04	from same batch

Lab-code	Pesticide Name	Deri-vati-zation	Derivatization other specification	Quantification using standards	Internal stan-dard	Injection volume (µl)	Injec-tion type	Determination technique	Reporting Level (mg/kg)	Recovery Correction	Recovery (%)	Spiked level (mg/kg)	Recovery from
	Propamocarb	NO		M	NO	10		LC-MS/MS	0.01	no	75	0.04	from same batch
Lab 10	Dithiocarbamates expressed as CS2	NO		M	NO	5	Splitless	GC-FPD	0.1	no			from same batch
	2,4-D (free acid only)												
	Fluazifop (free acid only)												
	Haloxyfop (free acid only)												
	MCPA (free acid only)												
	MCPP (free acid only)												
	Abamectin (only Avermectin B1a)												
	Propamocarb												
Lab 11	Dithiocarbamates expressed as CS2												
	2,4-D (free acid only)												
	Fluazifop (free acid only)	NO		M	NO	20		LC-MS/MS	0.01	no	93.8	0.01	from same batch
	Haloxyfop (free acid only)												
	MCPA (free acid only)	NO		M	NO	20		LC-MS/MS	0.01	no	83.8	0.01	from same batch
	MCPP (free acid only)												
	Abamectin (only Avermectin B1a)												
	Propamocarb	NO		M	NO	20		LC-MS/MS	0.012	no	64.7	0.05	from same batch
Lab 12	Dithiocarbamates expressed as CS2	NO		M	NO	2	Splitless	GC-Q-MS	0.05	no	87	0.06	from same batch
	2,4-D (free acid only)	NO		M	YES	3		LC-MS/MS	0.01	no	82	0.01	from same batch
	Fluazifop (free acid only)	NO		M	YES	3		LC-MS/MS	0.01	no	97	0.01	from same batch
	Haloxyfop (free acid only)	NO		M	YES	3		LC-MS/MS	0.01	no	105	0.01	from same batch
	MCPA (free acid only)	NO		M	YES	3		LC-MS/MS	0.01	no	66	0.01	from same batch
	MCPP (free acid only)	NO		M	YES	3		LC-MS/MS	0.01	no	81	0.01	from same batch
	Abamectin (only Avermectin B1a)	NO		M	YES	3		LC-MS/MS	0.01	no	81	0.02	from same batch
	Propamocarb	NO		M	YES	3		LC-MS/MS	0.01	no	113	0.01	from same batch
Lab 13	Dithiocarbamates expressed as CS2	NO		S	NO	1	Splitless	GC-ECD	0.04	no	119	2.5	from same batch
	2,4-D (free acid only)			M	YES	5			0.05				
	Fluazifop (free acid only)			M	YES	5		LC-MS/MS	0.05	no	96	0.1	from same batch
	Haloxyfop (free acid only)								0.01				
	MCPA (free acid only)			M	YES	5		LC-MS/MS	0.1	no	101	0.1	from same batch
	MCPP (free acid only)								0.1				
	Abamectin (only Avermectin B1a)			M	YES	5		LC-MS/MS	0.025	no	122	0.1	from same batch

Lab-code	Pesticide Name	Deri-vati-zation	Derivatization other specification	Quantification using standards	Internal stan-dard	Injection volume (µl)	Injec-tion type	Determination technique	Reporting Level (mg/kg)	Recovery Correction	Recovery (%)	Spiked level (mg/kg)	Recovery from
	Propamocarb			M	YES			LC-MS/MS	0.01	no	107	0.1	from same batch
Lab 14	Dithiocarbamates expressed as CS2							Photometer	0,01	no			
	2,4-D (free acid only)												
	Fluazifop (free acid only)												
	Haloxyfop (free acid only)												
	MCPA (free acid only)												
	MCPP (free acid only)												
	Abamectin (only Avermectin B1a)												
	Propamocarb												
Lab 15	Dithiocarbamates expressed as CS2	NO		S	NO	4	Split	GC-FPD	0.02	no	74	0.2	from same batch
	2,4-D (free acid only)	NO		M	NO	5		LC-MS/MS	0.01	no	95	0.05	from same batch
	Fluazifop (free acid only)	NO		M	NO	5		LC-MS/MS	0.01	no	93	0.05	from same batch
	Haloxyfop (free acid only)	NO		M	NO	5		LC-MS/MS	0.05	no	95	0.05	from same batch
	MCPA (free acid only)	NO		M	NO	5		LC-MS/MS	0.05	no	97	0.05	from same batch
	MCPP (free acid only)	NO		M	NO	5		LC-MS/MS	0.05	no	98	0.01	from same batch
	Abamectin (only Avermectin B1a)												
	Propamocarb	NO		M	NO	5		LC-MS/MS	0.01	no	87	0.05	from same batch
Lab 16	Dithiocarbamates expressed as CS2												
	2,4-D (free acid only)												
	Fluazifop (free acid only)	NO		M	YES	10		LC-MS/MS	0,01	no	87	0. 05	from same batch
	Haloxyfop (free acid only)												
	MCPA (free acid only)	NO		M	YES	10		LC-MS/MS	0,01	no	88	0. 05	from same batch
	MCPP (free acid only)												
	Abamectin (only Avermectin B1a)	NO		M	YES	10		LC-MS/MS	0,01	no	103	0. 05	from same batch
	Propamocarb	NO		M	YES	10		LC-MS/MS	0,01	no	76	0. 05	from same batch
Lab 17	Dithiocarbamates expressed as CS2	NO		S	NO	100	Splitless	GC-Q-MS	0.01	no	88	0.04	from same batch
	2,4-D (free acid only)												
	Fluazifop (free acid only)												
	Haloxyfop (free acid only)												
	MCPA (free acid only)												
	MCPP (free acid only)												
	Abamectin (only Avermectin B1a)												
	Propamocarb												
Lab 18	Dithiocarbamates expressed as CS2												
	2,4-D (free acid only)	NO		M	NO			LC-MS/MS	0.05	no	70	0.05	from same batch

Lab-code	Pesticide Name	Deri-vati-zation	Derivatization other specification	Quantification using standards	Internal stan-dard	Injection volume (µl)	Injec-tion type	Determination technique	Reporting Level (mg/kg)	Recovery Correction	Recovery (%)	Spiked level (mg/kg)	Recovery from
	Fluazifop (free acid only)												
	Haloxyfop (free acid only)	NO		M	NO			LC-MS/MS	0.05	no	65	0.05	from same batch
	MCPA (free acid only)	NO		M	NO			LC-MS/MS	0.05	no	75	0.05	from same batch
	MCPP (free acid only)	NO		M	NO			LC-MS/MS	0.05	no	68	0.05	from same batch
	Abamectin (only Avermectin B1a)												
	Propamocarb												
Lab 19	Dithiocarbamates expressed as CS2	NO		M	NO	250	Splitless	GC-ECD	0.050	no	80	0.2	from routine QC data
	2,4-D (free acid only)												
	Fluazifop (free acid only)	NO		M	NO	10		LC-MS/MS	0.005	yes, via standard addition to aliquots of sample extracts (Art. 48, SANCO/2007/3131)	60	0.1	from routine QC data
	Haloxyfop (free acid only)												
	MCPA (free acid only)	NO		M	NO	10		LC-MS/MS	0.005	yes, via standard addition to aliquots of sample extracts (Art. 48, SANCO/2007/3131)	65	0.1	from routine QC data
	MCPP (free acid only)												
	Abamectin (only Avermectin B1a)	YES		M	NO	10		LC-MS/MS	0.010	yes, via standard addition to aliquots of sample extracts (Art. 48, SANCO/2007/3131)	90	0.1	from routine QC data
	Propamocarb	NO		M	NO	10		LC-MS/MS	0.010	yes, via standard addition to aliquots of sample extracts (Art. 48, SANCO/2007/3131)	100	0.1	from routine QC data
Lab 20	Dithiocarbamates expressed as CS2			M	NO	2	Splitless	GC-Q-MS	0.05	no	48	1.6	from same batch
	2,4-D (free acid only)	NO		M	NO	10	On-Column	LC-MS/MS	0.02				
	Fluazifop (free acid only)	NO		M	NO	10	On-Column	LC-MS/MS	0.01	no	75.5		from same batch
	Haloxyfop (free acid only)	NO		M	NO	10	On-Column	LC-MS/MS	0.01				
	MCPA (free acid only)	NO		M	NO	10	On-Column	LC-MS/MS	0.01				
	MCPP (free acid only)	NO		M	NO	10	On-Column	LC-MS/MS	0.01				
	Abamectin (only Avermectin B1a)												
	Propamocarb												
Lab 21	Dithiocarbamates expressed as CS2			S				Photometer	0.01	no			
	2,4-D (free acid only)												
	Fluazifop (free acid only)	NO		M	YES	20		LC-MS/MS	0.01	yes, automatically via standard addition procedure (Art. 47, SANCO/2007/3131)	102	0.1	from same batch
	Haloxyfop (free acid only)												
	MCPA (free acid only)	NO		M	YES	20		LC-MS/MS	0.01	yes, automatically via standard addition procedure (Art. 47, SANCO/2007/3131)	106	0.1	from same batch
	MCPP (free acid only)												

Lab-code	Pesticide Name	Deri-vati-zation	Derivatization other specification	Quantification using standards	Internal stan-dard	Injection volume (µl)	Injec-tion type	Determination technique	Reporting Level (mg/kg)	Recovery Correction	Recovery (%)	Spiked level (mg/kg)	Recovery from
	Abamectin (only Avermectin B1a)	NO		M	YES	20		LC-MS/MS	0.01	yes, automatically via standard addition procedure (Art. 47, SANCO/2007/3131)	95	0.1	from same batch
	Propamocarb	NO		M	YES	20		LC-MS/MS	0.01	yes, automatically via standard addition procedure (Art. 47, SANCO/2007/3131)	82	0.1	from same batch
Lab 22	Dithiocarbamates expressed as CS2	YES	dietanolamine/copper complex	S	NO	NA		Photometer	0.20	no	NA	NA	
	2,4-D (free acid only)												
	Fluazifop (free acid only)												
	Haloxyfop (free acid only)												
	MCPA (free acid only)												
	MCPP (free acid only)												
	Abamectin (only Avermectin B1a)												
	Propamocarb												
Lab 23	Dithiocarbamates expressed as CS2	NO		S	NO	4	Splitless	GC-FPD	0.05	no	96	0.5	from validation data
	2,4-D (free acid only)	NO		M	NO	5		LC-MS/MS	0.005	no	95	0.01	from same batch
	Fluazifop (free acid only)	NO		M	NO	5		LC-MS/MS	0.005	no	91	0.01	from same batch
	Haloxyfop (free acid only)	NO		M	NO	5		LC-MS/MS	0.005	no	92	0.01	from same batch
	MCPA (free acid only)	NO		M	NO	5		LC-MS/MS	0.005	no	89	0.01	from same batch
	MCPP (free acid only)	NO		M	NO	5		LC-MS/MS	0.005	no	94	0.01	from same batch
	Abamectin (only Avermectin B1a)												
	Propamocarb	NO		M	NO	5		LC-MS/MS	0.005	no	97	0.01	from same batch
Lab 26	Dithiocarbamates expressed as CS2			S	YES	500	On-Column	GC-FPD	0,02	no	80,3	0. 5	from same batch
	2,4-D (free acid only)												
	Fluazifop (free acid only)	NO		M	YES	5		LC-MS/MS	0,01	no	112,1	0. 05	from same batch
	Haloxyfop (free acid only)												
	MCPA (free acid only)	NO		M	YES	5		LC-MS/MS	0,01	no	109,8	0. 05	from same batch
	MCPP (free acid only)												
	Abamectin (only Avermectin B1a)	NO		S	NO	5		LC-MS/MS	0,01	no	109,9	0. 05	from same batch
	Propamocarb	NO		M	YES	5		LC-MS/MS	0,02	no	89,5	0. 05	from same batch
Lab 27	Dithiocarbamates expressed as CS2	YES		S	NO			Photometer	0,01	no			
	2,4-D (free acid only)												
	Fluazifop (free acid only)												
	Haloxyfop (free acid only)												
	MCPA (free acid only)												
	MCPP (free acid only)												

Lab-code	Pesticide Name	Deri-vati-zation	Derivatization other specification	Quantification using standards	Internal stan-dard	Injection volume (µl)	Injec-tion type	Determination technique	Reporting Level (mg/kg)	Recovery Correction	Recovery (%)	Spiked level (mg/kg)	Recovery from
	Abamectin (only Avermectin B1a)												
	Propamocarb												
Lab 28	Dithiocarbamates expressed as CS2	YES	SnCl2+HCl/H2O	M	NO	3	Split	GC-ECD	0.05		94.7	0.05	
	2,4-D (free acid only)												
	Fluazifop (free acid only)	NO		S	NO	4		LC-MS/MS	0.02		115	0.1	
	Haloxyfop (free acid only)												
	MCPA (free acid only)	NO		S	NO	4		LC-MS/MS	0.02		100	0.1	
	MCPP (free acid only)												
	Abamectin (only Avermectin B1a)												
	Propamocarb	NO		S	NO	4		LC-MS/MS	0.02		109	0.1	
Lab 29	Dithiocarbamates expressed as CS2			S	NO	500	Split	GC-FPD	0.05	no	90	0.5	from same batch
	2,4-D (free acid only)	NO		S	NO	10		LC-MS/MS	0.05	no	105	0.1	from same batch
	Fluazifop (free acid only)	NO		S	NO	10		LC-MS/MS	0.05	no	116	0.1	from same batch
	Haloxyfop (free acid only)	NO		S	NO	10		LC-MS/MS	0.05	no	118	0.1	from same batch
	MCPA (free acid only)	NO		S	NO	10		LC-MS/MS	0.05	no	107	0.1	from same batch
	MCPP (free acid only)	NO		S	NO	10		LC-MS/MS	0.05	no	113	0.1	from same batch
	Abamectin (only Avermectin B1a)	NO		S	NO	10		LC-MS/MS	0.01	no	116	0.1	from same batch
	Propamocarb	NO		S	NO	10		LC-MS/MS	0.05	no	92	0.1	from same batch
Lab 30	Dithiocarbamates expressed as CS2	NO		S	NO			Photometer	0.4	no	95	1	from routine QC data
	2,4-D (free acid only)	NO		S	NO	5			0.05	no	87	0.05	from same batch
	Fluazifop (free acid only)	NO		S	NO	5			0.05	no	82	0.05	from same batch
	Haloxyfop (free acid only)	NO		S	NO	5			0.05	no	85	0.05	from same batch
	MCPA (free acid only)	NO		S	NO	5			0.05	no	85	0.05	from same batch
	MCPP (free acid only)	NO		S	NO	5			0.05	no	88	0.05	from same batch
	Abamectin (only Avermectin B1a)					-							
	Propamocarb	NO		S	NO	5			0.05	no	72	0.05	from same batch
Lab 31	Dithiocarbamates expressed as CS2	YES		S	NO			Photometer	0.05	no			
	2,4-D (free acid only)								0.01				
	Fluazifop (free acid only)	NO		M	YES	10		LC-MS/MS	0.01	no			
	Haloxyfop (free acid only)								0.01				
	MCPA (free acid only)	NO		M	YES	10		LC-MS/MS	0.01	no			
	MCPP (free acid only)								0.01				
	Abamectin (only Avermectin B1a)	NO		M	YES	10		LC-MS/MS	0.01	no			
	Propamocarb	NO		M	YES	10		LC-MS/MS	0.01	no			

Lab-code	Pesticide Name	Deri-vati-zation	Derivatization other specification	Quantification using standards	Internal stan-dard	Injection volume (µl)	Injec-tion type	Determination technique	Reporting Level (mg/kg)	Recovery Correction	Recovery (%)	Spiked level (mg/kg)	Recovery from
Lab 32	Dithiocarbamates expressed as CS2	NO		M	NO	300	Splitless	GC-Q-MS	0.04	no	99	0.25	
	2,4-D (free acid only)	NO		M	NO	55		LC-MS/MS	0.01				
	Fluazifop (free acid only)	NO		M	NO	55		LC-MS/MS	0.01	no	102	0.08	from same batch
	Haloxyfop (free acid only)	NO		M	NO	55		LC-MS/MS	0.01				
	MCPA (free acid only)	NO		M	NO	55		LC-MS/MS	0.01	no	101	0.08	from same batch
	MCPP (free acid only)	NO		M	NO	55		LC-MS/MS	0.01				
	Abamectin (only Avermectin B1a)	NO		M	NO	5		LC-MS/MS	0.01	no			
	Propamocarb	NO		M	NO	5		LC-MS/MS	0.01	no	94	0.08	from same batch
Lab 33	Dithiocarbamates expressed as CS2	NO		S	NO			Photometer	0.1	no	80	0.15	from same batch
	2,4-D (free acid only)	NO		M	YES	10		LC-MS/MS	0.05	no	120	0.1	from same batch
	Fluazifop (free acid only)	NO		M	YES	10		LC-MS/MS	0.05	no	71	0.1	from same batch
	Haloxyfop (free acid only)												
	MCPA (free acid only)	NO		M	YES	10		LC-MS/MS	0.05	no	130	0.1	from same batch
	MCPP (free acid only)	NO		M	YES	10		LC-MS/MS	0.05	no	92	0.1	from same batch
	Abamectin (only Avermectin B1a)												
	Propamocarb	NO		M	YES	10		LC-MS/MS	0.05	no	94	0.1	from same batch
Lab 34	Dithiocarbamates expressed as CS2												
	2,4-D (free acid only)								0.010				
	Fluazifop (free acid only)			M	NO	5		LC-MS/MS	0.010	no	80	0.200	from same batch
	Haloxyfop (free acid only)								0.005				
	MCPA (free acid only)			M	NO	5		LC-MS/MS	0.005	no	95	0.200	from same batch
	MCPP (free acid only)								0.005				
	Abamectin (only Avermectin B1a)			M	NO	5		LC-MS/MS	0.010	no	90	0.200	from same batch
	Propamocarb			M	NO	5		LC-MS/MS	0.010	no	79	0.200	from same batch
Lab 35	Dithiocarbamates expressed as CS2	YES		S	NO			Photometer	0.05	no			from validation data
	2,4-D (free acid only)												
	Fluazifop (free acid only)												
	Haloxyfop (free acid only)												
	MCPA (free acid only)												
	MCPP (free acid only)												
	Abamectin (only Avermectin B1a)												

Lab-code	Pesticide Name	Deri-vati-zation	Derivatization other specification	Quantification using standards	Internal stan-dard	Injection volume (µl)	Injec-tion type	Determination technique	Reporting Level (mg/kg)	Recovery Correction	Recovery (%)	Spiked level (mg/kg)	Recovery from
	Propamocarb												
Lab 36	Dithiocarbamates expressed as CS2	NO		M	NO	250	On-Column	GC-FPD	0.05	no	80.7	1.0	from same batch
	2,4-D (free acid only)												
	Fluazifop (free acid only)	YES		M	NO	1.0	PTV	GC-ITD-MS/MS	0.05	no	87.5	0.25	from same batch
	Haloxyfop (free acid only)												
	MCPA (free acid only)	YES		M	NO	1.0	PTV	GC-ITD-MS/MS	0.05	no	90.9	0.25	from same batch
	MCPP (free acid only)												
	Abamectin (only Avermectin B1a)	NO		S	NO	10	On-Column	HPLC-FL	0.05	no	78.9	0.25	from same batch
	Propamocarb	NO		M	NO	1.0	PTV	GC-ITD-MS/MS	0.05	no	90.0	0.5	from same batch
Lab 37	Dithiocarbamates expressed as CS2	YES	copper(II)acetate	S	NO			Photometer	0.1				
	2,4-D (free acid only)	NO		M	NO	5		LC-MS/MS	0.01	no			
	Fluazifop (free acid only)	NO		M	NO	5		LC-MS/MS	0.01	no	77.6	0.1	from same batch
	Haloxyfop (free acid only)	NO		M	NO	5		LC-MS/MS	0.01	no			
	MCPA (free acid only)	NO		M	NO	5		LC-MS/MS	0.01	no	71.3	0.1	from same batch
	MCPP (free acid only)	NO		M	NO	5		LC-MS/MS	0.01	no			
	Abamectin (only Avermectin B1a)												
	Propamocarb	NO		M	NO	5		LC-MS/MS	0.01	no	67.8	0.1	from same batch
Lab 38	Dithiocarbamates expressed as CS2	YES									95	1.09	from same batch
	2,4-D (free acid only)												
	Fluazifop (free acid only)	NO		M	NO	10		LC-MS/MS	0.01	no	97	0.066	from same batch
	Haloxyfop (free acid only)	NO											
	MCPA (free acid only)	NO		M	NO	10		LC-MS/MS	0.01	no	81	0.149	from same batch
	MCPP (free acid only)												
	Abamectin (only Avermectin B1a)	NO		M	NO	10		LC-MS/MS	0.01	no	85	0.129	from same batch
	Propamocarb	NO		M	NO	10		LC-MS/MS	0.01	no	52	0.078	
Lab 39	Dithiocarbamates expressed as CS2												
	2,4-D (free acid only)												
	Fluazifop (free acid only)												
	Haloxyfop (free acid only)												
	MCPA (free acid only)	NO		M	YES	10		LC-MS/MS	0.02	no	105	0.2	from same batch
	MCPP (free acid only)												
	Abamectin (only Avermectin B1a)												

Lab-code	Pesticide Name	Deri-vati-zation	Derivatization other specification	Quantification using standards	Internal stan-dard	Injection volume (µl)	Injec-tion type	Determination technique	Reporting Level (mg/kg)	Recovery Correction	Recovery (%)	Spiked level (mg/kg)	Recovery from
	Propamocarb	NO		M	YES	10		LC-MS/MS	0.015	no	102	0.2	from same batch
Lab 40	Dithiocarbamates expressed as CS2	YES		S	NO			Photometer	0.05	no	83	0.25	from same batch
	2,4-D (free acid only)												
	Fluazifop (free acid only)	NO		S	NO	5		LC-MS/MS	0.01	no	99.5	0.08	from same batch
	Haloxyfop (free acid only)	NO		S	NO	5		LC-MS/MS	0.01	no	98	0.01	from same batch
	MCPA (free acid only)												
	MCPP (free acid only)												
	Abamectin (only Avermectin B1a)	NO		S	NO	5		LC-MS/MS	0.05	no	104	0.6	from same batch
	Propamocarb	NO		S	NO	5		LC-MS/MS	0.01	no	93	0.3	from same batch
Lab 41	Dithiocarbamates expressed as CS2												
	2,4-D (free acid only)	NO		M	NO	10		LC-MS/MS	0.025	yes, automatically via standard addition procedure (Art. 47, SANCO/2007/3131)	102	0.05	from same batch
	Fluazifop (free acid only)	NO		M	NO	5		LC-MS/MS	0.01	yes, automatically via standard addition procedure (Art. 47, SANCO/2007/3131)	87	0.1	from same batch
	Haloxyfop (free acid only)	NO		M	NO	5		LC-MS/MS	0.01	yes, automatically via standard addition procedure (Art. 47, SANCO/2007/3131)	61	0.1	from same batch
	MCPA (free acid only)	NO		M	NO	10		LC-MS/MS	0.01	yes, automatically via standard addition procedure (Art. 47, SANCO/2007/3131)	102	0.1	from same batch
	MCPP (free acid only)	NO		M	NO	10		LC-MS/MS	0.025	yes, automatically via standard addition procedure (Art. 47, SANCO/2007/3131)	104	0.05	from same batch
	Abamectin (only Avermectin B1a)												from routine QC data
	Propamocarb	NO		M	NO	5		LC-MS/MS	0.01	yes, automatically via standard addition procedure (Art. 47, SANCO/2007/3131)	115	0.1	from same batch
Lab 42	Dithiocarbamates expressed as CS2	NO		S	NO			Photometer	0,005	no	91,4	0. 16	from same batch
	2,4-D (free acid only)	NO		M	YES	20		LC-MS/MS	0,05	no	99,5	0. 1	from same batch
	Fluazifop (free acid only)	NO		M	YES	20		LC-MS/MS	0,01	no	111	0. 1	from same batch
	Haloxyfop (free acid only)	NO		M	YES	20		LC-MS/MS	0,02	no	100	0. 1	from same batch
	MCPA (free acid only)	NO		M	YES	20		LC-MS/MS	0,005	no	98,3	0. 1	from same batch
	MCPP (free acid only)	NO		M	YES	20		LC-MS/MS	0,02	no	103,1	0. 1	from same batch
	Abamectin (only Avermectin B1a)	NO		M	YES	20		LC-MS/MS	0,02	no	101	0. 1	from same batch

Lab-code	Pesticide Name	Deri-vati-zation	Derivatization other specification	Quantification using standards	Internal stan-dard	Injection volume (µl)	Injec-tion type	Determination technique	Reporting Level (mg/kg)	Recovery Correction	Recovery (%)	Spiked level (mg/kg)	Recovery from
	Propamocarb	NO		M	YES	20		LC-MS/MS	0,02	no	48,3	0. 1	from same batch
Lab 44	Dithiocarbamates expressed as CS2	NO		S	NO	10	Splitless	GC-FPD	0.05				
	2,4-D (free acid only)	NO		M	NO	10		LC-MS/MS	0.01				
	Fluazifop (free acid only)	NO		M		10		LC-MS/MS	0.01				
	Haloxyfop (free acid only)	NO		M		10		LC-MS/MS	0.01				
	MCPA (free acid only)	NO		M		10		LC-MS/MS	0.01				
	MCPP (free acid only)												
	Abamectin (only Avermectin B1a)												
	Propamocarb												
Lab 45	Dithiocarbamates expressed as CS2	NO		S				Other	0.34	yes, using a recovery rate	50.6	0.25	from same batch
	2,4-D (free acid only)												
	Fluazifop (free acid only)	NO		M	NO	10		LC-MS/MS	0.01	no	92.6	0.05	from same batch
	Haloxyfop (free acid only)												
	MCPA (free acid only)	NO		M	NO	10		LC-MS/MS	0.01	no	72.3	0.05	from same batch
	MCPP (free acid only)												
	Abamectin (only Avermectin B1a)	NO		M	NO	10		LC-MS/MS	0.01	no	92.4	0.05	from same batch
	Propamocarb	NO		M	NO	10		LC-MS/MS	0.01	no	97.6	0.05	from same batch
Lab 46	Dithiocarbamates expressed as CS2	YES		S	NO			Photometer	0.2	no	105	2	from routine QC data
	2,4-D (free acid only)												
	Fluazifop (free acid only)												
	Haloxyfop (free acid only)												
	MCPA (free acid only)												
	MCPP (free acid only)												
	Abamectin (only Avermectin B1a)												
	Propamocarb												
Lab 47	Dithiocarbamates expressed as CS2			S	NO			Photometer	0.1	no	95	0.5	from same batch
	2,4-D (free acid only)												
	Fluazifop (free acid only)	NO		M	NO	10		LC-MS/MS	0.01	no	91	0.1	from same batch
	Haloxyfop (free acid only)												
	MCPA (free acid only)	NO		M	NO	10		LC-MS/MS	0.01	no	97	0.1	from same batch
	MCPP (free acid only)												
	Abamectin (only Avermectin B1a)	NO		M	NO	10		LC-MS/MS	0.01	no	93	0.1	from same batch
	Propamocarb	NO		M	NO	10		LC-MS/MS	0.01	no	88	0.1	from same batch
Lab 48	Dithiocarbamates expressed as CS2							Photometer	0.50	no	70	1	from same batch
	2,4-D (free acid only)								0.05				

Lab-code	Pesticide Name	Deri-vati-zation	Derivatization other specification	Quantification using standards	Internal stan-dard	Injection volume (µl)	Injec-tion type	Determination technique	Reporting Level (mg/kg)	Recovery Correction	Recovery (%)	Spiked level (mg/kg)	Recovery from
	Fluazifop (free acid only)			M	YES	20		LC-MS/MS	0.05	no	103	0.05	from same batch
	Haloxyfop (free acid only)												
	MCPA (free acid only)			M	YES	20		LC-MS/MS		no	87	0.05	
	MCPP (free acid only)											0.05	
	Abamectin (only Avermectin B1a)			M		20		LC-MS/MS	0.05	no	91	0.05	from same batch
	Propamocarb			M	YES	20		LC-MS/MS	0.05	no	93	0.05	from same batch
Lab 49	Dithiocarbamates expressed as CS2	NO		S	NO	200	Split	GC-ECD	0,1	no	84	1. 0	from same batch
	2,4-D (free acid only)												
	Fluazifop (free acid only)												
	Haloxyfop (free acid only)												
	MCPA (free acid only)												
	MCPP (free acid only)												
	Abamectin (only Avermectin B1a)												
	Propamocarb												
Lab 50	Dithiocarbamates expressed as CS2			S	NO	-		Photometer	0.05	no	80	0.1	from same batch
	2,4-D (free acid only)								0.05				
	Fluazifop (free acid only)	YES		S	NO	1.0	Splitless	GC-ECD	0.05	no	84.5	0.1	from same batch
	Haloxyfop (free acid only)								0.05				
	MCPA (free acid only)	YES		S	NO	1.0	Splitless	GC-ECD	0.05	no	83.0	0.1	from same batch
	MCPP (free acid only)								0.05				
	Abamectin (only Avermectin B1a)	NO		S	NO	20		HPLC-FL	0.01	no	86.0	0.1	from same batch
	Propamocarb	NO		S	NO	1		GC-NPD	0.05	no	75.0	0.2	from same batch
Lab 51	Dithiocarbamates expressed as CS2	YES	Sn2Cl	S	NO	10		Photometer	0.2	no			
	2,4-D (free acid only)			M	NO	10		LC-MS/MS	0.01	no	100	0.1	from same batch
	Fluazifop (free acid only)			M	NO	10		LC-MS/MS	0.01	no	120	0.01	from same batch
	Haloxyfop (free acid only)			M	NO	10		LC-MS/MS	0.01	no	100	0.01	from same batch
	MCPA (free acid only)			M	NO	10		LC-MS/MS	0.01	no	99	0.1	from same batch
	MCPP (free acid only)			M	NO	10		LC-MS/MS	0.01	no	60	0.01	from same batch
	Abamectin (only Avermectin B1a)			M	NO	10		LC-MS/MS	0.01	no	153	0.1	from same batch
	Propamocarb			M	NO	10		LC-MS/MS	0.01	no	56	0.1	from same batch
Lab 52	Dithiocarbamates expressed as CS2			M	NO	250	On-Column	GC-ECD	0.05	no			
	2,4-D (free acid only)	NO		M	NO	50		LC-MS/MS	0.05				

Lab-code	Pesticide Name	Deri-vati-zation	Derivatization other specification	Quantification using standards	Internal stan-dard	Injection volume (µl)	Injec-tion type	Determination technique	Reporting Level (mg/kg)	Recovery Correction	Recovery (%)	Spiked level (mg/kg)	Recovery from
	Fluazifop (free acid only)												
	Haloxyfop (free acid only)	NO		M	NO	50		LC-MS/MS	0.05				
	MCPA (free acid only)	NO		M	NO	50		LC-MS/MS	0.05	yes, using a recovery rate	92	0.100	from same batch
	MCPP (free acid only)	NO		M	NO	50		LC-MS/MS	0.05				
	Abamectin (only Avermectin B1a)												
	Propamocarb	NO		M	NO	50		LC-MS/MS	0.05	yes, using a recovery rate	58	0.100	from same batch
Lab 53	Dithiocarbamates expressed as CS2	YES	degradation to CS2	S	NO	2	Splitless	GC-Q-MS	0.05		80	0.1	from validation data
	2,4-D (free acid only)												
	Fluazifop (free acid only)												
	Haloxyfop (free acid only)												
	MCPA (free acid only)												
	MCPP (free acid only)												
	Abamectin (only Avermectin B1a)												
	Propamocarb												
Lab 54	Dithiocarbamates expressed as CS2	YES	KOH in methanol	S	NO			Photometer	0.02	no	74	0.05	from same batch
	2,4-D (free acid only)	YES	TMSH - Trimethyl sulfonium hidroxide solution - 0.25M in methanol	M	NO	2	Splitless	GC-Q-MS	0.05	no	76	0.05	from same batch
	Fluazifop (free acid only)	YES	TMSH - Trimethyl sulfonium hidroxide solution - 0.25M in methanol	M	NO	2	Splitless	GC-Q-MS	0.05	no	75	0.05	from same batch
	Haloxyfop (free acid only)	YES	TMSH - Trimethyl sulfonium hidroxide solution - 0.25M in methanol	M	NO	2	Splitless	GC-Q-MS	0.05	no	98	0.05	from same batch
	MCPA (free acid only)	YES	TMSH - Trimethyl sulfonium hidroxide solution - 0.25M in methanol	M	NO	2	Splitless	GC-Q-MS	0.05	no	77	0.05	from same batch
	MCPP (free acid only)	YES	TMSH - Trimethyl sulfonium hidroxide solution - 0.25M in methanol	M	NO	2	Splitless	GC-Q-MS	0.05	no	72	0.05	from same batch
	Abamectin (only Avermectin B1a)												
	Propamocarb												
Lab 55	Dithiocarbamates expressed as CS2			M	NO			Photometer	0.3	no	94	1	from same batch
	2,4-D (free acid only)												
	Fluazifop (free acid only)												
	Haloxyfop (free acid only)												
	MCPA (free acid only)												
	MCPP (free acid only)												
	Abamectin (only Avermectin B1a)												
	Propamocarb												
Lab 56	Dithiocarbamates expressed as CS2	YES		S	NO			Photometer	0.1	no			
	2,4-D (free acid only)												
	Fluazifop (free acid only)												

Lab-code	Pesticide Name	Deri-vati-zation	Derivatization other specification	Quantification using standards	Internal stan-dard	Injection volume (µl)	Injec-tion type	Determination technique	Reporting Level (mg/kg)	Recovery Correction	Recovery (%)	Spiked level (mg/kg)	Recovery from
	Haloxyfop (free acid only)												
	MCPA (free acid only)												
	MCPP (free acid only)												
	Abamectin (only Avermectin B1a)												
	Propamocarb												
Lab 57	Dithiocarbamates expressed as CS2	NO		S	NO	2	Split	GC-FPD	0.03	no	80	1	from routine QC data
	2,4-D (free acid only)												
	Fluazifop (free acid only)												
	Haloxyfop (free acid only)												
	MCPA (free acid only)												
	MCPP (free acid only)												
	Abamectin (only Avermectin B1a)												
	Propamocarb												
Lab 59	Dithiocarbamates expressed as CS2	YES		S		1ul	On-Column	GC-ECD	0.05	no	82	1	from same batch
	2,4-D (free acid only)												
	Fluazifop (free acid only)												
	Haloxyfop (free acid only)												
	MCPA (free acid only)												
	MCPP (free acid only)												
	Abamectin (only Avermectin B1a)												
	Propamocarb												
Lab 60	Dithiocarbamates expressed as CS2	YES		S	NO	-		Other	0,1	no	71	2 (tirame)	from same batch
	2,4-D (free acid only)												
	Fluazifop (free acid only)												
	Haloxyfop (free acid only)												
	MCPA (free acid only)												
	MCPP (free acid only)												
	Abamectin (only Avermectin B1a)												
	Propamocarb												
Lab 61	Dithiocarbamates expressed as CS2												
	2,4-D (free acid only)	NO		M	NO	8		LC-MS/MS	0.05	no	80	0.05	from same batch
	Fluazifop (free acid only)	NO		M	NO	8		LC-MS/MS	0.05	no	91	0.05	from same batch
	Haloxyfop (free acid only)	NO		M	NO	8		LC-MS/MS	0.05	no	79	0.05	from same batch
	MCPA (free acid only)	NO		M	NO	8		LC-MS/MS	0.05	no	83	0.05	from same batch
	MCPP (free acid only)	NO		M	NO	8		LC-MS/MS	0.05	no	80	0.05	from same batch
	Abamectin (only Avermectin B1a)												
	Propamocarb	NO		M	NO	8		LC-TOF/MS	0.05	no	73	0.05	from same batch
Lab 62	Dithiocarbamates expressed as CS2	NO		S	NO			Photometer	0,05	no			
	2,4-D (free acid only)												
	Fluazifop (free acid only)												

Lab-code	Pesticide Name	Deri-vati-zation	Derivatization other specification	Quantification using standards	Internal stan-dard	Injection volume (µl)	Injec-tion type	Determination technique	Reporting Level (mg/kg)	Recovery Correction	Recovery (%)	Spiked level (mg/kg)	Recovery from
	Haloxyfop (free acid only)												
	MCPA (free acid only)												
	MCPP (free acid only)												
	Abamectin (only Avermectin B1a)												
	Propamocarb												
Lab 63	Dithiocarbamates expressed as CS2	NO		S	NO	1	Splitless	GC-Q-MS	0.05	yes, using a recovery rate	70	1.6	from routine QC data
	2,4-D (free acid only)												
	Fluazifop (free acid only)												
	Haloxyfop (free acid only)												
	MCPA (free acid only)												
	MCPP (free acid only)												
	Abamectin (only Avermectin B1a)												
	Propamocarb	NO		M	NO	35		LC-MS/MS	0.01	no	110	0.2	
Lab 64	Dithiocarbamates expressed as CS2	NO		M	NO	200	Split	GC-Q-MS	0,05	no	82,0	2	from routine QC data
	2,4-D (free acid only)												
	Fluazifop (free acid only)												
	Haloxyfop (free acid only)												
	MCPA (free acid only)												
	MCPP (free acid only)												
	Abamectin (only Avermectin B1a)												
	Propamocarb												
Lab 65	Dithiocarbamates expressed as CS2	NO		S	NO	1	Splitless	GC-Q-MS	0.01	no	82	1	from validation data
	2,4-D (free acid only)												
	Fluazifop (free acid only)	NO		M	YES	2		LC-MS/MS	0.05	no	92	0.050	from same batch
	Haloxyfop (free acid only)												
	MCPA (free acid only)	NO		M	YES	2		LC-MS/MS	0.05	no	90	0.050	from same batch
	MCPP (free acid only)												
	Abamectin (only Avermectin B1a)												
	Propamocarb												
Lab 66	Dithiocarbamates expressed as CS2	NO		S	NO	2	Splitless	GC-FPD	0.05	no	95	0.05	from validation data
	2,4-D (free acid only)												
	Fluazifop (free acid only)												
	Haloxyfop (free acid only)												
	MCPA (free acid only)												
	MCPP (free acid only)												
	Abamectin (only Avermectin B1a)												
	Propamocarb												
Lab 67	Dithiocarbamates expressed as CS2	NO		S	NO	-		Photometer	0,05	no	-	-	
	2,4-D (free acid only)												
	Fluazifop (free acid only)												
	Haloxyfop (free acid only)												

Lab-code	Pesticide Name	Deri-vati-zation	Derivatization other specification	Quantification using standards	Internal stan-dard	Injection volume (µl)	Injec-tion type	Determination technique	Reporting Level (mg/kg)	Recovery Correction	Recovery (%)	Spiked level (mg/kg)	Recovery from
	MCPA (free acid only)												
	MCPP (free acid only)												
	Abamectin (only Avermectin B1a)												
	Propamocarb												
Lab 68	Dithiocarbamates expressed as CS2			S	NO			Other	0.4		97.2	0.5	
	2,4-D (free acid only)	NO		S	NO			LC-MS/MS	0.010		98.6	0.050	
	Fluazifop (free acid only)	NO		S	NO			LC-MS/MS	0.010		93.1	0.050	
	Haloxyfop (free acid only)												
	MCPA (free acid only)												
	MCPP (free acid only)												
	Abamectin (only Avermectin B1a)	NO		S	NO			LC-MS/MS	0.022		89.1	0.050	
	Propamocarb	NO		S	NO			LC-MS/MS	0.010		0.075	0.050	
Lab 69	Dithiocarbamates expressed as CS2	YES		S	NO			Photometer	0.25	no	79.72	1.00	from same batch
	2,4-D (free acid only)												
	Fluazifop (free acid only)												
	Haloxyfop (free acid only)												
	MCPA (free acid only)												
	MCPP (free acid only)												
	Abamectin (only Avermectin B1a)												
	Propamocarb												
Lab 70	Dithiocarbamates expressed as CS2	NO		M		500	Split	GC-FPD	0.03	yes, automatically via standard addition procedure (Art. 47, SANCO/2007/3131)			
	2,4-D (free acid only)												
	Fluazifop (free acid only)												
	Haloxyfop (free acid only)												
	MCPA (free acid only)												
	MCPP (free acid only)												
	Abamectin (only Avermectin B1a)												
	Propamocarb												

ANNEX VIII: LIST OF PARTICIPATING LABORATORIES

Table 14: List of participating laboratories

Laboratory Name	Country	City	NRL-Status
Austrian Agency for Food and Health Safety (AGES) Analytical Competence Center for Plant Protection Products	AT	Innsbruck	NRL-SRM
Competence Centre for Residue Analysis, Austrian Agency for Health and Food Safety,	AT	Vienna	NRL-Other
Fytolab	BE	Zwijnaarde - Gent	No NRL
Lovap NV	BE	Geel	No NRL
Scientific Institute of Public Health	BE	Bruxelles	NRL-SRM
Central Laboratory for Chemical Testing and Control	BG	Sofia	NRL-SRM
Kantonales Labor Zurich	CH	Zurich	No NRL
Service de la Consommation et des Affaires Vitirinaires (SCAV)	CH	Geneve	NRL-Other
Pesticide Residues Laboratory of State General Laboratory	CY	Nicosia	NRL-SRM
Czech Agriculture and Food Inspection Authority	CZ	Brno	NRL-SRM
Institute of Chemical Technology, Prague, Dept. of Food Chemistry and Analysis	CZ	Prague	No NRL
Bayerisches Landesamt für Gesundheit und Lebensmittelsicherheit Erlangen	DE	Berlin	No NRL
BBGes-ILAT	DE	Berlin	No NRL
CVUA Münster	DE	Münster	No NRL
Federal Office of Consumer Protection and Food Safety (BVL)	DE	Berlin	NRL-SRM
Institut für Hygiene und Umwelt	DE	Hamburg	No NRL
Landesamt für Landwirtschaft, Lebensmittelsicherheit und Fischerei MV	DE	Berlin	No NRL
Landesamt für Verbraucherschutz Sachsen-Anhalt	DE	Berlin	No NRL
Landesbetrieb Hessisches Landeslabor	DE	Kassel	No NRL
Landeslabor Schleswig-Holstein	DE	Neumünster	No NRL
Landesuntersuchungsamt	DE	Speyer	No NRL
LSGV (Landesamt für Soziales, Gesundheit und Verbraucherschutz)	DE	Saarbrücken	No NRL
Niedersaechsisches Landesamt für Verbraucherschutz und Lebensmittelsicherheit, Lebensmittelinstitut Oldenburg	DE	Berlin	No NRL
Danish Vet. and Food Adm. Region East	DK	Ringsted	No NRL
National Food Institute	DK	Soeborg	NRL-SRM
Laboratory for Residues and Contaminants, Agricultural Research Centre	EE	SAKU	NRL-Other
Tartu Laboratory of Health Protection Inspectorate	EE	Tartu	NRL-SRM
Instituto Tecnologico de Canarias	ES	Santa Lucia - Las Palmas	No NRL
Lab. Produccion y Sanidad Vegetal	ES	Jaen	No NRL
Laboratorio Agrario Regional - Junta de Castilla y León	ES	Burgos	NRL-Other
Laboratorio Agroalimentario de la Generalitat Valenciana	ES	BURJASSOT, Valencia	No NRL
Laboratorio Agroambiental (Diputación Foral de Gipuzkoa)	ES	Zizurkil	No NRL
Laboratorio Arbitral Agoalimentatio	ES	Madrid	NRL-Other
Finnish Customs Laboratory	FI	Espoo	NRL-SRM
Laboratory SCL - Rennes	FR	Rennes	No NRL
SCL Sevice commun des laboratoires Illkirch	FR	Illkirch	No NRL
General Chemical State Laboratory	GR	Athens	NRL-SRM
Pesticide Residues Laboratory, Benaki Phytopathological Institute	GR	Kiphissia-Athens	NRL-SRM

Laboratory Name	Country	City	NRL-Status
Regional Center of Plant Protection and Quality Control of Achaia, Lab. of Pesticide Residues	GR	Patra	No NRL
Regional Centre Of Plant Protection & Quality Control Of Iraklion	GR	Iraklion, Crete	No NRL
Agricultural Office of BAZ County Plant Protection and Soil Conservation Directorate, Pesticide Residue Laboratory	HU	Miskolc	NRL-SRM
Agricultural Office of County Csongrad, Directorate of Plant protection and Soil Conservation, Pesticide Residue Analytical Laboratory	HU	Szeged	No NRL
Agricultural Office of County Fejer, Plant Protection and Soil Conservation Directorate, Pesticide Residue Analytical Laboratory	HU	Velence	NRL-Other
Plant Protection and Soil Conservation Directorate of Jasz-Nagykun-Szolnok County	HU	Szolnok	No NRL
PPSCD of Somogy County, Pesticide Analytical Laboratory	HU	Budapest	NRL-Other
Pesticide Control laboratory	IE	Co. Kildare	NRL-SRM
ARPA Sicilia DAP Ragusa	IT	Ragusa	No NRL
Istituto Superiore di Sanita - Rep. Antiparassitari	IT	Rome	NRL-SRM
National Veterinary Laboratory	LT	Vilnius	NRL-SRM
Laboratoire National de Santé	LU	Luxembourg	NRL-Other
National Diagnostic Centre	LV	Riga	NRL-SRM
RIKILT Institute of Food Safety	NL	Wageningen	NRL-Other
VWA - Food and Consumer Product Safety Authority	NL	Amsterdam	NRL-SRM
Bioforsk, Plant Health and Plant Protection, Pesticide Chemistry	NO	Aas	NRL-SRM
Department of Pesticide Residue Research, Institute of Plant Protection	PL	Poznan	NRL-Other
Food Safety Laboratory, Research Institute of Pomology & Floriculture	PL	Skierniewice	No NRL
Laboratory of Department of Environmental Toxicology, National Institute of Hygiene	PL	Warsaw	NRL-SRM
Wojewodzka Stacja Sanitarno - Epidemiologiczna w Krakowie	PL	Krakow	No NRL
Wojewodzka Stacja Sanitarno - Epidemiologiczna we Wroclawiu, Dzial Laboratoryjny	PL	Wroclaw	No NRL
Wojewsdzka Stacja Sanitarno - Epidemiologiczna w Rzeszowie,Oddzial Laboratoryjny w Przemyslu	PL	Rzeszsw	No NRL
WSSE Opole - Oddzial Laboratoryjny w Kluczborku	PL	Opole	no NRL
Direção Regional de Agricultura do Algarve	PT	Faro	No NRL
INIA - Laboratório de Resíduos de Pesticidas	PT	Lisboa	NRL-Other
Laboratório de Qualidade Agrícola	PT	Camacha	No NRL
Laboratório de Qualidade Alimentar da DRAP-N	PT	Senhora da Hora-Matosinhos	No NRL
Eurofins Food/Agro Sweden AB	SE	Lidköping	NRL-SRM (delegated)
Institute of Public Health Maribor	SI	Maribor	NRL-SRM
Institute of public health of the republic of Slovenia	SI	Ljubljana	NRL-Other
State Veterinary and Food Institute	SK	Bratislava	NRL-SRM
Central Science Laboratory	UK	York	NRL-SRM

ANNEX IX: PROTOCOL

Main characteristics

The aim of this proficiency test is to obtain information about the quality, accuracy and comparability of the residue data of pesticides that are typically analysed using single/selective residue methods within the framework of the EU and national pesticide monitoring programmes. Participating laboratories will be provided with an assessment of both their own analytical performance and the reliability of their data - compared to other laboratories.

The Quality Control Group for this proficiency test plays a vital role in the organisation of the test and the evaluation of its results. It is an independent group, approved by the Organiser and DG SANCO and each member has proven experience over many years, working in quality control for their own national accreditation bodies. The Quality Control Group's role is to help the Organiser make decisions concerning test quality, pesticide selection, spiking levels, MRRLs and statistical treatments of the results.

The Organiser would like to point out that the results may be presented to the Standing Committee on the Food Chain and Animal Health on a country-to-country basis. It is therefore possible that a link between codes and laboratories could be made, especially for those Member States where only one lab participates. The performance data of the individual labs may also be forwarded to institutions within DG-SANCO upon request.

The official language used in this Proficiency Test is English.

Communication between participating laboratories during the test on matters concerning this PT exercise is not permitted.

Steps to follow

This Proficiency Test is made up of the following 7 essential steps:

1. To participate, each laboratory must complete the Application Form, available on-line at the CRL-SRM Web page (www.crl-pesticides.eu), before the deadline stipulated on the Calendar. It is recommended that laboratories download the Possible Pesticide List from this web site. Laboratories should carefully read the Pesticide List, where important information about the reporting of the results, as well as the MRRLs, is given. Labs should note that the pesticide residue definitions within this exercise do not always follow Regulation 396/2005.
2. Laboratories will then receive an e-mail confirming their participation in this exercise and assigning them a unique Laboratory Code only known by the participant, the Organiser s and the Commission upon request. Laboratories will be able to access the restricted area containing the Protocol and Forms (shown in the following steps) using their login information consisting of their e-mail address and their password as chosen on the application form.
3. The payment procedure must have started before the 30th April. An e-mail showing the bank transfer confirmation, or similar, has to be sent beforehand. Please note that this year the bank account has changed. Payments without a laboratory code to identify them will not be considered as paid.
4. When the participant laboratories receive the test material (and not before), they must enter the restricted area and submit Form 1 on-line to inform the Organiser if they have accepted

the test material. They will then receive a confirmation message. This Form has a deadline that must be met. If no test material has been received by the 11th June, please contact the Organiser by e-mail (crl-pesticides@cvuas.bwl.de and pmedina@ual.es)

5. The participant laboratories must meet the deadline for submitting the results and method information- using Form 2 on-line.

6. The laboratories participating in the optional standard-solution test should submit their results online via Form 3 until 11th July. The standard solutions will be shipped together with the test material to all participants. It is strongly recommended to participate in this exercise, because it helps to compare individual results and might be useful for identifying possible deviations in the concentration of the standard solutions used by the laboratories.

7. The Organiser will evaluate the results at the end of the proficiency test, once the deadline for receipt of the results has passed. The Organiser will send a hard 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 statistical evaluation of the participant's results as well as graphical displays of the results and conclusions. Any other relevant information considered of value will also be included. There will be no link between the Laboratory Codes and the laboratory's identity in the Final Report.

Calendar

The following table shows the programme for this EUPT-SRM3

Table 1: Calendar of the EUPT-SRM3

ACTIVITY	WHO?	DATE
- Publication of the Pesticide List and Calendar on the Web page (Pesticide List has been updated on March)	CRL-SRM	January 2008
- Submission of the Application Form (on-line) by invited laboratories (Remember your password)	Invited Laboratories	8th April 2008
- Sample Treatment, Homogenisation, and Storage. Homogeneity & Stability Tests.	CRL-FV (on behalf of CRL-SRM)	April 2008
- Deadline for payment and sending proof of payment	Participating Laboratories	9th May 2008
Reminder of imminent sample delivery	CRL-SRM	2nd June 2008
- Distribution of test material and standard solutions	CRL-FV (on behalf of CRL-SRM)	9th June 2008
- Submission of Form1 (on-line) (regarding receipt of sample)	Participating Laboratories	20th June 2008
- Submission of Form2 (on-line) (test results and method information)	Participating Laboratories	11th July 2008
- Submission of Form3 (on-line) (results of standard solutions)	Participating Laboratories	11th July 2008
- Preliminary Report: only results, no statistical treatment.	CRL-SRM	July 2008
- Evaluation Meeting	Organiser s, Commission, QC Group	October 2008
- Final Report to the Laboratories	CRL-SRM	December 2008

Possible Pesticide List

The Pesticide List can be accessed at the CRL-SRM website (www.crl-pesticides.eu) from January 2008. This list includes all the possible pesticides that might be present in the test material. The distribution of this list well before the receipt of the test materials gives the labs plenty of time to purchase any standards that they may require and to validate their methods.

Laboratories should carefully read the Pesticide List, where important information about the reporting of the results, as well as the MRRLs is given. Labs should note that the pesticide residue definitions within this exercise do not always follow the Regulation 396/2005.

Each pesticide and the relevant compounds included in the residue definitions have been given an MRRL value (Minimum Required Reporting Level). The MRRL values are the levels that laboratories are expected to attain. They were established by the Quality Control Group and the Organiser and will be used for the calculation of false negative z-scores.

Table 2: List of potential pesticides

Compound	Residue definition within this PT	MRRL* (mg/kg)
2.4-D	free acid only	0.05
Abamectin	only avermectin B1a	0.01
Dithiocarbamates as CS_2	Dithiocarbamates expressed as CS2, (incl. maneb, mancozeb, metiram, propineb, thiram and ziram)	0.05
Fluazifop	free acid only	0.05
Haloxyfop	free acid only	0.05
MCPA	free acid only	0.05
MCPP	free acid only	0.05
Propamocarb	propamocarb	0.05

Participating Laboratories

It is the duty of the administrative bodies responsible for the official monitoring of pesticide residues in each country to select the laboratories that should participate. According to provisions in Regulation 882/2004 (article 32 and 33) NRLs must participate in PTs organised by the CRL. Furthermore, according to Regulation 396/2005, participation of official laboratories (OFLs) providing residue data for their national and/or the EU coordinated monitoring programmes also becomes compulsory from 1st September 2008, provided that the PT exercise wholly or partly overlaps with their scope within the monitoring programmes.

For this PT, the Commission wishes that all NRLs and OFLs fill-in the application form regardless of whether, or not, they decide to participate. Laboratories that decide not to participate must state their reasons in the Application Form, as well as informing their NRL. If a laboratory initially decides to participate, but later on withdraws again, it must also give the reasons to the NRL and the PT-Organiser . These reasons will be communicated to the European Commission.

It is up to the participants to complete the Application Form on-line so that the Organiser has all their details before the deadline. The Organiser will not be responsible if a laboratory does not receive notice of the web page address necessary to take part in the test.

Application Form

On the CRL-SRM web site (www.crl-pesticides.eu) the participating laboratories must complete the Application Form on-line.

It is important that laboratories' details are updated and that laboratories make sure their e-mail system is working throughout the duration of the test.

On the Application Form, there is also information that must be provided, in order to be able to issue an official invoice. The Application Form must be submitted on-line by 28th March 2008 at the latest.

Laboratories must be aware that some fields in this Form are OBLIGATORY and are marked with red asterisks. If they are not filled-in, they will not be able to continue.

Methodology

This proficiency test is based on pesticide residues analysis of carrot. The carrots were grown in Almería, Spain.
The pesticide treatments will be carried out post-harvest either using commercial formulations in micro-spray solutions or using standard solutions. 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 to check for homogeneity.
The test material will be stored frozen (–20°C) 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 solely to check stability during the shipping process and for the duration of the proficiency test.

Amount of test material

Approximately 300g of carrot test material will be shipped together with 300g of 'blank' carrot, surrounded with dry ice and packed in boxes. The courier costs will be charged to, and must be paid by, the participants before shipment of the test materials. There will only be a limited amount of test material and laboratories should not ask for more than they require in order to perform the analysis. Laboratories ordering a second portion of the material will have to pay double the price for this PT.

Shipping of test material

The shipment of the test materials will be carried out over a one-week period. The Organisers will try to ensure that all the shipments arrive at once. A warning message will be sent out a week before the shipment. Laboratories must make their own arrangements for the reception of the test materials. They must inform the Organiser of any public holidays in their country/city during the delivery period given in the calendar, as well as make the necessary arrangements to receive the shipment even if the laboratory is closed.

Advice on test material handling

Once received, the test material must be stored frozen until it is to be analysed.
The contents of the bottle should be mixed thoroughly, to ensure homogeneity of the test material, before taking the analytical portion(s).

Form 1, acceptance of test material

Once the laboratory has received the test materials, they must complete Form 1 on-line: filling in the date of receipt, the condition of the test material, and acknowledging its' acceptance. Form

1 has a deadline, so if the form has not been filled-in by the 20th June 2008, laboratories may no longer be able to submit the form on-line.
Please note that on every form there are OBLIGATORY fields that must be filled-in otherwise the form cannot be submitted. Form 1 will be available in the restricted area from the time the shipment process starts.
If any laboratory has not received the test material by the 19th June, it must inform the Organiser immediately by e-mail (crl-pesticides@cvuas.bwl.de and pmedina@ual.es).

Form 2, results and analytical method information

Once the laboratory has analysed the test material and is ready to submit their data, they must enter the restricted zone and complete Form 2 on–line. Most of the fields are OBLIGATORY and have to be filled in otherwise it will not be possible to submit results.

Test Material Analysed

The test material contains a certain number of pesticides from the Possible Pesticide List. The residue definitions valid for this exercise are presented in the Possible Pesticide List.
Each laboratory must report the result for each of the pesticide residues that they found in the test material - reporting only one analytical procedure for each residue.
The results, expressed as residue levels in mg/kg, must also be reported, together with the laboratories' reporting level (RL) for each pesticide. This level will be used for information purposes only.

Residue Levels - Significant Figures

The results must be expressed in mg/kg in the following way:
Residue levels < 0.100 mg/kg, to be expressed to two significant figures (three decimals places: i.e. 0.058 mg/kg).
Residue levels ≥ 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 sought, but not detected, it should be recorded as 'ND' and the reporting level should be written in the appropriate column. If it was not sought, it should be recorded as 'NA'.
The results/residue levels must be reported as numbers.

Correction of Results

According to Method Validation and Quality Control Procedures for Pesticide Residues Analysis in Food And Feed, Document No. SANCO/2007/3131, residues data do not have to be adjusted for recovery, when the mean recovery is in the range of 70-120%. If residues data are adjusted for recovery, then this must be stated. Therefore laboratories will be required to report whether their results were adjusted for recovery, the recovery factor (if adjusted for recovery), the spiking level of the recovery, and if it was determined in the same analytical batch as the test material. No recovery factors are required where recovery adjustments resulted from using the 'standard addition' approach, or from the use of isotopically labelled internal standards (with spiking of the test material in both cases).

Method information

The laboratories will also be asked to give a reference and provide details of the analytical procedure(s) used. A drop-down box option containing the most common analytical methods will

be available. If none of these methods are applicable, laboratories should select the option 'OTHER' typing the reference (e.g. publication in a scientific journal) for the analytical method in the corresponding field.
Form 2 must be sent to the Organiser by 11th July 2008, at the latest. No results will be accepted after this date. The laboratories will be responsible for reporting their results to the Organiser. The Organiser will acknowledge receipt of the results by sending back a confirmation message.

Form 3, standard solution (optional exercise)

Together with the test material the Organiser will send to each participating laboratory. a vial with the standard solution mixture containing all compounds from "Possible Pesticide List" with exception of dithiocarbamates.
The laboratories are asked to determine the concentration of those pesticides in the standard solution mixture, that they have encountered in the test material and report them to the Organiser s via Form3. The purpose of this optional exercise will be to check for the deviations of the standard solutions that were used by the laboratory for the sample analysis. Multiple injections are recommended to mach for instrument variability.
The relevant details of this standard solution are:
(i) The Laboratories will receive screw capped vials containing 5 mL aliquot of the standard solution mixture. These vials should be stored in the freezer until dilution and measurement. For the sake of efficiency, the solutions will be shipment together with the test materials. The standard solution mixture will contain all pesticides from the possible pesticides list. Nevertheless, the laboratories are asked to report only those pesticides found in the sample material.
(ii) The solvent used to prepare the solution will be acetonitrile.
(iii) The concentration range of each compound present will be 20 - 90 mg/L. Please note that the concentration of each pesticide in the standard mixture has no connection to the concentration in the sample material.

Preparation of the standard solution mixture by the Organiser : Individual stock solutions in acetonitrile will be prepared by weighing out suitable amounts of each of the reference standards that were freshly produced for this test. Aliquots of these stock solutions of the individual compounds will then be combined to prepare the standard solution mixture within the range 20 - 90 mg/L.
The participant will be able to report the results of this exercise in Form 3 in the restricted area, which will be accessible shortly after Form 1 and 2.

ANNEX X: ORGANISATION ADDRESS

The official postal address, phone number, fax number and e-mail address of the Organiser are as follows:

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