European Commission

EURL-SRM

EU Reference Laboratories for Residues of Pesticides

Single Residue Methods

EU Proficiency Test on the Analysis of Pesticides Residues Requiring Single Residue Methods in Strawberry Purée

EUPT–SRM12

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Final Report

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EU PROFICIENCY TEST
EUPT-SRM12, 2017

Residues of Pesticides Requiring Single Residue Methods

Test Item: Strawberry Purée

Final Report

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FOREWORD

Regulation 882/2004/EC [1] defines the general tasks and duties of the EU Reference Laboratories (EURLs) for Food, Feed and Animal Health[1] including the organisation of comparative tests (proficiency tests = PTs). These PTs are carried out on an annual basis and aim to improve the quality, accuracy and comparability of the analytical results generated by EU Member States within the framework of the EU coordinated control programs as well as national monitoring programs. By participating in PTs laboratories can assess and at the same time demonstrate their analytical performance. The attention to details paid by laboratories during PT-analysis, together with the need to identify errors and to take corrective actions in cases of underperformance, typically lead to improvements in the quality of analytical results.

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 [2], all laboratories analysing for pesticide residues within the framework of official controls shall participate in the European Union Comparative Proficiency Tests (EUPTs) for pesticide residues. Each Official Laboratory (OfL) must participate in EUPTs concerning the commodities included in its area of competence.

Since 2006 the EURL for pesticide residues requiring the use of Single Residue Methods, EURL-SRM, has annually conducted one scheduled Proficiency Test. Five of those twelve EUPT-SRMs were conducted in collaboration with the EURL for pesticide residues in Fruits and Vegetables (EURL-FV) with apple juice (EUPT-SRM1, 2006), carrot homogenate (EUPT-SRM3, 2008), apple purée (EUPT-SRM5, 2010), potato homogenate (EUPT-SRM8, 2013) and spinach homogenate (EUPT-SRM11, 2016) as test items. Further four EUPT-SRMs were conducted in collaboration with the EURL for pesticide residues in Cereals and Feeding Stuff (EURL-CF) with wheat flour (EUPT-C1/SRM2, 2007), oat flour (EUPT-C3/SRM4, 2009), rice flour (EUPT-C5/SRM6, 2011) and maize flour (EUPT-C9/SRM10, 2015) as test items. The EUPT-SRM9 was the only EUPT-SRM so far, in which a commodity of animal origin was used. The remaining three EUPT-SRMs, the EUPT-SRM7 (2012) based on milled dry lentils, the EUPT-SRM9 (2014) based on cow's milk and the present one, the EUPT-SRM12 based on strawberry purée were organized by the EURL-SRM unilaterally.

Participation in the respective EUPTs is mandatory for all NRLs for pesticides requiring Single Residue Methods (NRL-SRMs) and for all OfLs analysing pesticide residues within the framework of national or EU control programs in commodities represented by the respective EUPT test item. Laboratories in EU Member States analysing pesticide residues within the frame of import controls according to Reg. 669/2009/EC are also considered as performing official controls in the sense of Reg. 882/2005/EC and 396/2005/EC and are thus also obliged to take part in EUPTs. OfLs from EFTA countries (Iceland, Norway and Switzerland) contributing data to the EU-coordinated community control programs, EU laboratories analysing official organic samples within the frame of Reg. 889/2008/EC, as well as OfLs from EU-acceding or -candidate countries (FYROM, Montenegro, Serbia and Turkey) are also invited to take part. A limited number of laboratories from third countries are allowed to take part in this exercise, too. However, only results submitted by labs from EU and EFTA countries are included in the calculation of the assigned values.

Based on information about the commodity scope and labs' NRL-status a tentative list of EU-labs considered as being obliged to participate in the EUPTs is published at the beginning of each year. The pesticide scope is not taken into account in these lists. NRLs and OfLs listed as being obliged to participate in an EUPT exercise in a given year but deciding not to take part, are always asked to state the reason(s) for their non-participation. The same applies to laboratories originally registering to participate in a certain EUPT but finally not submitting results.

[1] Formerly known as Community Reference Laboratories (CRLs)

DG-SANTE has full access to all data of EUPTs including the lab-code/lab-name key. The same applies to all NRLs as far as laboratories belonging to their own country networks are concerned. Results for this EUPT or a series of EUPTs, evaluated on a country by country basis, may be further presented to the European Commission Standing Committee on Plants, Animals, Food and Feed (PAFF)-Section Pesticides Residues ,or during the EURL-Workshops.

CONTENT

EUPT-SRM12: Supplementary Information on Analytical Methods

http://www.eurl-pesticides.eu/library/docs/srm/EUPT-SRM12_Supplementary_Information.pdf

Evaluation Report on the Feedback Survey on EUPT-SRM12

http://www.eurl-pesticides.eu/library/docs/srm/SRM12_Survey_Statistics_Evaluation.pdf

European Commission –
EU-Proficiency Test on Residues of Pesticides
Requiring Single Residue Methods
Test Item: Strawberry Purée
EUPT-SRM12, 2017

INTRODUCTION

On 16 December, 2016 all relevant National Reference Laboratories (NRLs) of the 28 EU-Member States (MS), as well as all relevant EU-Official Laboratories (OfLs) whose contact details were available to the organisers (EURL-SRM) were invited to participate in the 12th European Commission's Proficiency Test Requiring Single Residue Methods (EUPT-SRM12). The EUPT-SRM12-Website contained links to the Announcement/ Invitation Letter, the Calendar, as well as to the Target Pesticides List (see **Appendix 11**). The Target Pesticides List contained 25 compounds potentially being present in the test item. 13 of them were compulsory compounds and were thus considered in the Category A/B classification (based on scope). The compounds of the Target Pesticides List were selected based on a number of criteria and following consultation with the EUPT-Scientific Committee. For each compound a residue definition valid for the PT and the minimum required reporting level (MRRL) were stipulated. Links to the latest version of the "General Protocol" (see **Appendix 9**) containing information common to all EUPTs and to the "Specific Protocol" (see **Appendix 10**) valid for the current PT were also provided. The laboratories were able to register on-line from 17 January to 10 February, 2017.

Based on their commodity scope (fruit and vegetables) and their NRL-status (NRL-SRMs) a tentative list of the laboratories considered as being obliged to participate in the EUPT-SRM12 was published on the EURL-Website as well as on the CIRCA BC-platform. To ensure that all relevant official laboratories were informed about this EUPT, the NRLs were asked to forward the invitation to all relevant official laboratories within their countries. It was made clear that the list of obliged laboratories prepared by the EURLs was only tentative, and the real obligation to participate was based on Reg. 396/2005/EC and Reg. 882/2004/EC. Obliged labs that did not intend to participate were asked to provide an explanation.

In total 129 laboratories from EU and EFTA countries agreed to participate in the test. Two laboratories from EU-candidate countries and seven laboratories from third countries have also registered for the present EUPT, and all of them have submitted results.

The proficiency test EUPT-SRM12 was conducted using organic strawberry purée purchased from a food processing company. The test item was prepared by spiking the strawberry purée with 17 compounds dissolved in standard solutions. More details are given in **Chapter 1 "Test Materials and Blank Material"**.

1. TEST ITEM AND BLANK MATERIAL

1.1 Selection of PT-Commodity and of Compounds for the Target Pesticides List

In agreement with the EUPT- Scientific Committee strawberry purée was chosen as commodity for the EUPT-SRM12.

The compounds to be included in the Target Pesticides List (**Appendix 11**) were selected by the organiser and the EUPT-Scientific Committee (Advisory Group and Quality Control Group) taking the following points into account: 1) the present and upcoming scope of the EU-coordinated control program; 2) a pesticide priority list, ranking the pesticides according to their risk potential; 3) the relevance of pesticides to the specific commodity; 4) the overall scope and capability of the OfLs as assessed in previous PTs or surveys.

For the production of the test item and the blank material, two batches of organic strawberry purée were purchased from a food processing company and checked for the absence of the analytes on the Target Pesticides List. In one of the batches none of the target pesticides was detected except ***phosphonic acid*** at 0.72 mg/kg. This batch was finally used for the preparation of the blank material. The other batch, in which ***chlorate*** (0.032 mg/kg) and ***phosphonic acid*** (0.10 mg/kg) were detected, was used for the preparation of the test item by spiking with 17 compounds (see **Section 1.5, p. 5**).

The minimum required reporting levels (MRRLs) were set at 0.001 mg/kg for ***carbofuran (part of sum)***; at 0.01 mg/kg for ***2,4-D***, *abamectin*, ***captan (parent)***, ***chlorothalonil***, *cyromazine*, ***fenbutatin oxide***, *fluazifop*, ***folpet (parent)***, ***haloxyfop*** and *propamocarb*; at 0.02 mg/kg for *ethephon*, ***bifenazate (sum)***, ***chlorate***, ***dithianon*** and ***N-acetyl glyphosate***; at 0.03 mg/kg for ***dithiocarbamates***, ***glyphosate*** and *AMPA*, and at 0.05 mg/kg for ***phosphonic acid*** and at 3.0 mg/kg for ***bromide ion***.

1.2 Small Scale Preliminary Investigation on the Behavior of the Analytes during Homogenization

In order to estimate the loss of spiked analytes during the preparation of the test material, several preliminary spiking experiments were performed at a small scale using 100 g of the same organic strawberry purée. The experiments consisted of adding adapted mixtures of analytes to different portions of the pre-cooled purée (4 °C), stirred for 10 min at ambient temperature, to ensure homogeneity, and the withdrawal of a first set of analytical portions (portions 1, n = 2). In order to estimate the loss of spiked analytes during the preparation of the test material, several preliminary spiking experiment were performed at a small scale using various 100 g portions of strawberry purée. The experiments consisted of adding suitable mixtures of analytes to different portions of the pre-cooled purée (4 °C), stirring for 10 min at ambient temperature, to ensure homogeneity, and withdrawing of a first set of analytical portions (portions 1, n = 2). The remaining purée portions were stirred for further 3.5 h at ambient temperature prior to the withdrawal of a second set of analytical portions (portions 2, n = 2). The analytical portions were extracted using a modified QuEChERS method (FA-QuEChERS), QuPPe PO method and the method for dithiocarbamates (see **Table 1-2, p. 7**). FA-QuEChERS method was used to minimize the risk of losses of compounds, which are sensitive to high pH (namely ***dithianon***, ***captan*** and ***folpet***). In a preliminary experiment it was demonstrated that all other target analytes were not negatively affected by this procedure.

By setting the concentration of the analytes in the analytical portions 1 as 100 %, yields are corrected for recovery. **Figure 1-1** shows that except ***captan*** (83 %), ***folpet*** (82 %), ***dithianon*** (69 %) and ***dithiocarbamates*** (66 %), the relative recoveries (calculated against portion 1) of all other analytes were close to 100 % indicating minimal changes. It should be noted that the decline of the concentrations of captan (-17 %) and folpet

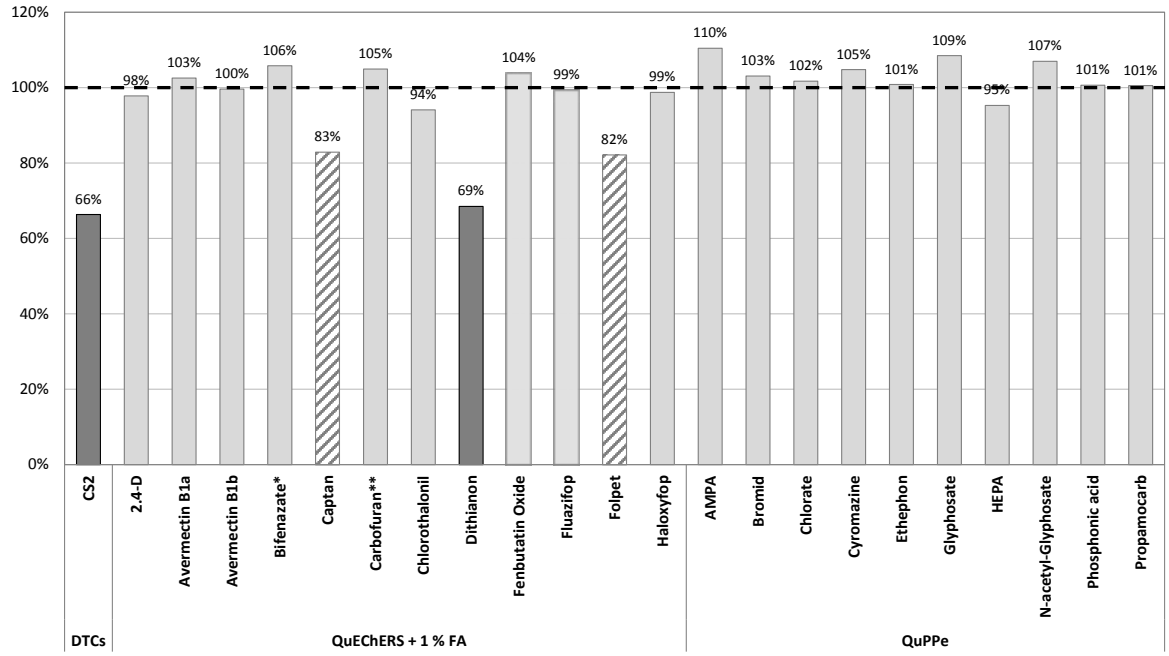


Figure 1-1: Recovery rates of spiked analytes after stirring at ambient temperature for 3.5 h (calculated against the results obtained when stirring stirring for only 10 min = 100 %). Bifenazate*: spiked as bifenazate diazene, converted into bifenazate using ascorbic acid and measured als bifenazate; Carbofuran**: spiked using carbosulfan, converted to carbofuran at acidic conditions and measured as carbofuran

(-18 %) was accompanied by an increase in the concentration of their respective degradation products ***THPI*** (+27 %) and ***phthalimide*** (+15 %). The determined losses of ***captan***, ***folpet***, ***dithianon*** and ***dithiocarbamates*** as well as the increase of the concentration of ***THPI*** and ***phthalimide*** were taken into account in the setting of the final spiking amount of these compounds in order to achieve adequate concentration levels in the final test material.

Analytes interfering each other during analysis (***carbofuran***/benfuracarb/furathiocarb/carbosulfan, ***bifenazate***/bifenazate diazene, ***glyphosate***/***N-acetyl glyphosate***) were studied in separate experiments.

1.2.1 Investigations concerning Carbofuran (sum)

Aiming to better plan the spiking of the material with ***carbofuran*** related components, several experiments were conducted to study the transformation of furathiocarb, benfuracarb and carbosulfan into ***carbofuran*** during the homogenization of strawberry purée. It was further tested whether method SRM23 of the EURL-SRM can be applied for the transformation of these 3 components to ***carbofuran***. Furathiocarb, benfuracarb and carbosulfan were added separately to 100 g portions of strawberry purée, which were all treated the same way as already described.

In case of benfuracarb and carbosulfan the analytical portions withdrawn from the bulk were extracted according to the CEN-QuEChERS approach followed by PSA cleanup and no re-acidification of the cleaned-up extract. In case of furathiocarb, which is resistant to mild acidic conditions, extraction was accomplished via FA-QuEChERS. One set of the extracts was analysed directly by LC-MS/MS and the second one was treated with sulfuric acid at 80 °C for 3 h (acidic hydrolysis) before LC-MS/MS analysis.

Following acidic hydrolysis only ***carbofuran*** could be detected with the recovery rate (calculated against the concentration of ***carbofuran*** equivalents originally spiked) ranging between 81 and 95 % (**Figure 1-2**). Furathiocarb remained intact during the 3.5 h homogenization procedure (110 % recovery rate), but it fully converted to ***carbofuran*** during acidic hydrolysis with recoveries of 95 % – 101 %. Carbosulfan survived at a rate of 23 % during the 3.5 h homogenization procedure with 59 % transforming into ***carbofuran***. Following acidic hydrolysis the determined transformation yield increased to 85 % overall. Benfuracarb completely degraded during the 3.5 h homogenization procedure, transforming into carbofuran at a nearly quantitative rate of 93 % (101 % after acidic hydrolysis). After only 10 min stirring benfuracarb degraded completely, but the transformation to carbofuran was not yet completed, with the transformation rate being only 45 % (and increasing to 88 % after acidic hydrolysis). This confirms the formation of intermediates as described under http://www.crl-pesticides.eu/userfiles/file/EurlSRM/EurlSrm_Observations_Carbofuran.pdf.

These experiments confirmed that the EURL-SRM procedure involving treatment with sulfuric acid at 80 °C for 3 h achieves a nearly quantitative conversion of the three pro-pesticides into carbofuran. It was further

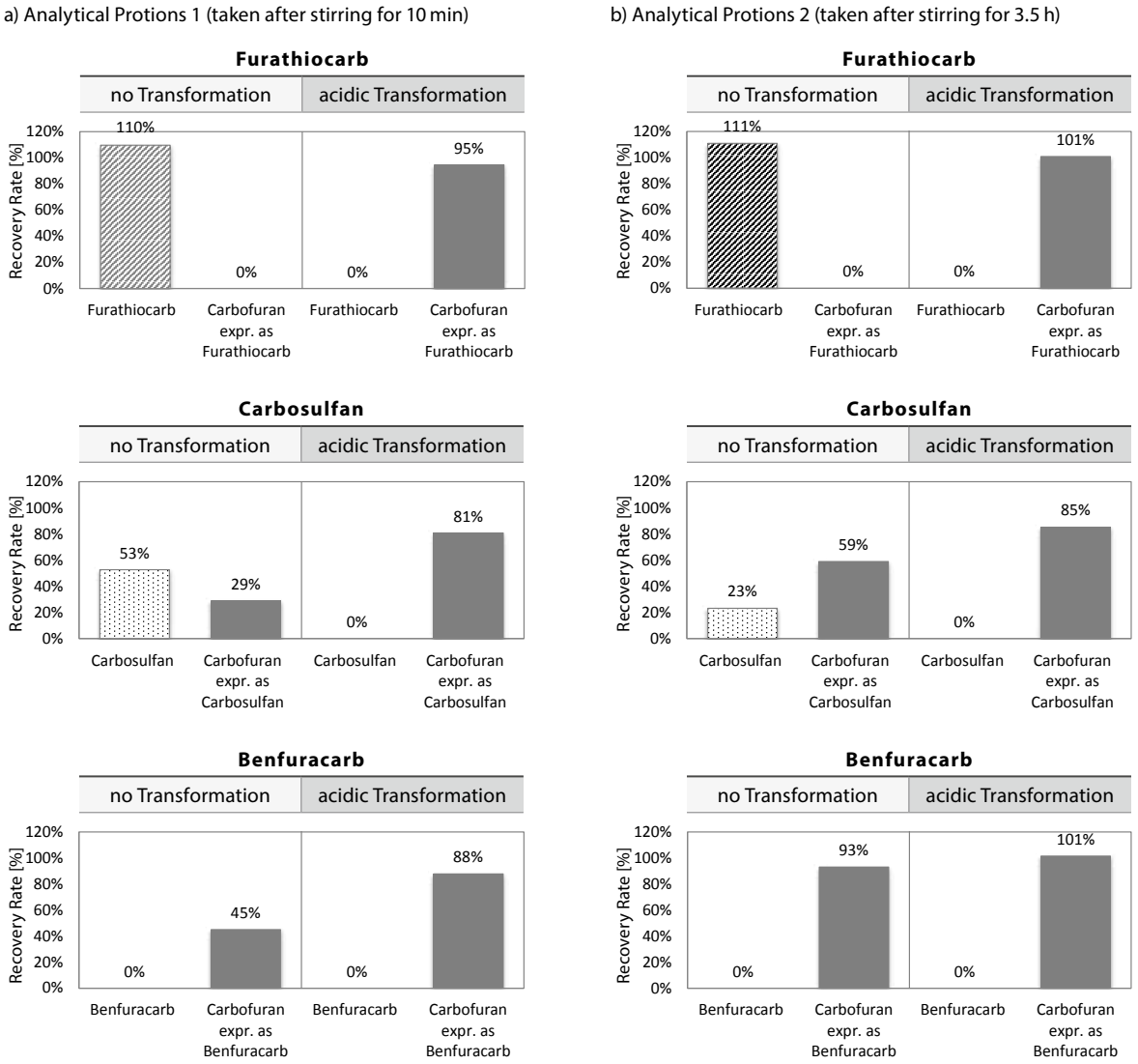


Figure 1-2: Fate of carbosulfan, furathiocarb and benfuracarb in strawberry homogenates during stirring for 10 min and 3.5 h and their transformation to carbofuran both in the strawberry homogenate as well as in the extract via acidic hydrolysis procedure. a) Analytical portions 1 were taken almost immediately after spiking (mixing by stirring for 10 min); b) Analytical portions 2 were taken after stiriing for 3.5 h. The recovery rates were determined using an external matrix-matched calibration setting the concentration of the spiked carbofuran equivalents spiked to the 100 g strawberry homogenate at 100 %.

observed that furathiocarb remains stable during homogenization whereas benfuracarb rapidly degrades with intermediates being formed, which are gradually transformed to carbofuran. Based on the results it was decided to add carbosulfan to the sample as it results in a challenging residue situation where both carbosulfan and carbofuran are present in the sample.

1.2.2 Investigations concerning Bifenazate (sum)

A similar investigation was carried out with bifenazate diazene studying its transformation to ***bifenazate*** (the reduced form).

Bifenazate-diazene and ***bifenazate*** were added to separate strawberry purée portions which were further handled as already described. The analytical portions were extracted according to FA-QuEChERS procedure. One set of extract aliquots was measured directly and a second set was first treated with ascorbic acid and allowed to react for 24 h at room temperature before LC-MS/MS analysis.

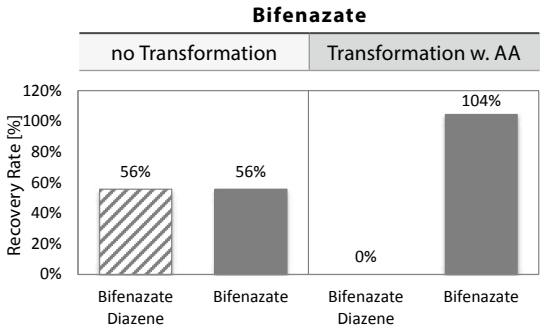
Following spiking of the bulk material and 10 min stirring, about half of the bifenazate-diazene spiked had already transformed into bifenazate (**Figure 1-3**). This is due to the high anti-oxidative potential of strawberry. After 3 hours of stirring, conversion was quantitative. The treatment of the extracts with ascorbic acid resulted in a complete conversion of bifenazate diazene into ***bifenazate*** which was quantified.

Following these experiments it was decided to spike the PT-sample with bifenazate diazene.

1.2.3 Investigation on the Analysis of Glyphosate / N-Acetyl Glyphosate

In the current matrix no transformation of N-acetyl glyphosate to glyphosate and vice versa was detected following a 3 hours of stirring of the spiked bulk strawberry purée.

a) Analytical Protions 1 (taken after stirring for 10 min)



b) Analytical Protions 2 (taken after stirring for 3.5 h)

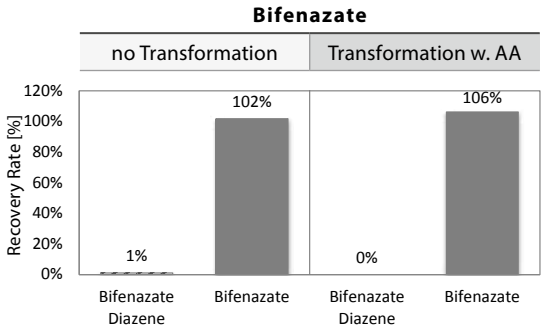


Figure 1-3: Fate of bifenazate-diazene in strawberry homogenate during stirring for 10 min and 3.5 h and its transformation to bifenazate both in the strawberry homogenate as well as in the extract following addition of ascorbic acid. a) Analytical portions 1 were taken almost immediately after spiking (mixing by stirring for 10 min); b) Analytical portions 2 were taken after stiriing for 3.5 h. The recovery rates were determined using an external matrix-matched calibration setting the concentration of the bifenazate equivalents spiked to the 100 g strawberry homogenate at 100 %. AA = ascorbic acid.

1.3 Preliminary Investigation of Homogeneity

On one hand the homogeneity of the sample portions can be reached by long and thorough mixing, on the other hand the procedure of mixing should be kept as short as necessary to keep degradation at tolerable levels. In order to estimate the stirring duration required to achieve satisfactory homogeneity, 75 kg organic strawberry purée were precooled over night at 4 °C. The material was spiked with a mixture of compounds covering a broad polarity range: ***fenbutatin oxide*** (logP 18.4), chlorpyrifos (logP 4.7), dimethoate (logP 0.34), linuron (logP 2.3), thiacloprid (logP 2.1), ***carbofuran*** (logP 2.1) and pyraclostrobin (logP 4.7). After spiking with different pesticides and mixing for 30 min at ambient temperature, two sample portions were withdrawn from each the top, the middle and the bottom layer of the container for analysis. As the results showed satisfactory homogeneity for all compounds already after 30 min of stirring, no further investigations were deemed necessary. Precooling over night at 4 °C and mixing for 60 min was considered appropriate for the preparation of the test item.

1.4 Preparation and Bottling of the Blank Material

Approximately 99 kg organic strawberry purée from 68 packages, each containing 1.5 kg, were pooled in a large plastic vessel, mixed intensively for 30 min using a large mixer and placed in a walk-in refrigerator over night at 4 °C for pre-cooling. On the following day 100 ml of a solvent mixture were slowly added to the cold material, while gently stirring with the mixer. The solvent mixture consisted of 50 ml acetonitrile and 50 ml of water and corresponded both in volume and composition to the spiking solution (see below). Following the addition of the solvent the mixture was gently stirred for 60 min to ensure homogeneity. The mixture was portioned into pressure lock plastic bags and frozen at –20 °C in thin layers over night. The frozen strawberry purée layers were cryogenically milled using dry ice and filled into the bottles in a snow-like state. Approximately 400 g portions of the well-mixed blank strawberry purée were weighed out into labelled and leak-proof screw-capped polyethylene plastic bottles, sealed and stored in a freezer at about –20 °C until distribution to participants.

1.5 Preparation and Bottling of the Test Item

The test item was prepared exactly in the same way as the blank material described above, but instead of adding pure solvent 100 ml of an equally composed mixture containing the target analytes was added. The mixture contained 17 different compounds and was prepared as described in **Table 1-1 (p. 6).** The following steps of homogenisation, portioning and storage were conducted in exactly the same way as for the blank material described above.

1.6 Packaging and Delivery of PT Materials to Participants

On the day of shipment, two frozen bottles, one with test item and the other one with blank material, as well as two vials, one containing isotope labelled chlorate solution and the other one containing isotope labelled phosphonate solution, were packed into thermo-insulated polystyrene boxes, filled-up with dry ice pellets (approx. 2 kg in each box) and shipped by DHL-Express to the laboratories. Where the dry ice transport was not allowed (due to IATA regulations), bigger and thick-walled thermo-insulated polystyrene boxes were used. Sufficient cooling elements were added to the boxes, and the filled packages were deep frozen at –70 °C for three days until shortly before shipment. Once the parcel was picked up by DHL, the recipient received an e-mail from the shipping company entailing the individual tracking number.

Table 1-1: Analytes spiked into 99 kg strawberry purèe for the preparation of the test material

Analytes dissolved in 50 ml ACN		Theor. Conc.	Analytes dissolved in 50 ml H_2O		Theor. Conc.
Compound	Amount	[mg/kg]	Compound	Amount	[mg/kg]
Thiram	59.5 mg	0.601 [1)]	Glyphosate	30.5 mg	0.308
Captan	10.5 mg	0.106	Potassium bromide	2985 mg	30.2 [3)]
Chlorothalonil	15.6 mg	0.158	Sodium chlorate	63.0 mg	0.636 [4)]
Folpet	42.4 mg	0.428	Phosphonic acid	2031 mg	20.5
THPI	10.0 mg	0.101	N-Acetyl glyphosate	10.0 mg [5)]	0.101
Phthalimide	39.5 mg	0.399			
2,4-D (free acid)	8.1 mg	0.082			
Haloxyfop (free acid)	7.2 mg	0.073			
Dithianon	34.9 mg	0.353			
Fenbutatin oxide	10.6 mg	0.107			
Bifenazate-Diazene	29.2 mg	0.295 [2)]			
Carbosulfan (1.0 mg/ml ACN)	0.9 ml	0.0091			

1) as CS_2 0.380 mg/kg; 2) as bifenazate 0.293 mg/kg; 3) as bromide ion 20.3 mg/kg; 4) as chlorate 0.510 mg/kg; 5) one packing unit of 10 mg

Among the 130 shipments to destinations in EU and EFTA countries 110 (85 %) of the packages arrived at the participating labs within 24 hours and 20 (15 %) of the packages within 48 hours. The delivery to countries outside the EU and EFTA zones was accomplished within 48 hours in 5 cases, within 72 hours in 2 cases, and within 4 days in 2 cases. The main reason for the long delivery times were delays at customs clearance. Details on the shipments and the condition of the Test Items upon arrival are shown in **Appendix 2.** 17 laboratories reported that the labels had come off both of the bottles. Except one laboratory, all other 16 laboratories accepted the materials and could distinguish the bottles by a preliminary screening. In another 4 cases the labels had come off one of the bottles. The organisers will re-test the labels to make sure that this problem will not be repeated in the future.

The participants were asked to describe the condition of the test samples upon arrival. The materials in the 110 parcels arriving the participants within one day were fully frozen and embedded in dry ice. The materials in the 18 of the 25 parcels that arrived the participants within two days were in most cases still fully frozen, even in those cases where no more dry ice was left in the boxes or where the parcels were sent with only gel packs. Another 5 participants receiving their parcels within 2 days reported that the material was mostly frozen (in 3 cases) or mostly defrosted (in 2 cases). In the two cases that the parcels arrived the participants within 3 days, the material of one parcel was still mostly frozen and the other one mostly defrosted. In the two cases where the parcels arrived after 4 days, the materials were fully defrosted and even at ambient temperature in one case. However, no noticeable negative influence due to the condition of material on arrival on the results was observed.

At this point organisers would like to appeal to the participants to follow their own parcels via the online tracking tool of the shipping company in order to maintain the ability to take the necessary measures in case of delays, e.g., contacting the customs to ask for an acceleration of the clearance procedure or to place the parcel in a cool place until clearance is granted. The participants are furthermore encouraged to contact the local office of the shipping company to ensure optimal delivery.

1.7 Analytical Methods

The analytical methods used by the organisers to check the homogeneity and storage-stability of the target analytes contained in the test item as well as the absence of target analytes in the blank material are summarized in **Table 1-2**. For more details on the methods used, please refer to the EURL-SRM website: http://www.eurl-pesticides.eu (EURL-SRM-website → Services → Methods).

Table 1-2: Analytical methods used by the organisers to check for the homogeneity and storage-stability of the pesticides present in the test item and to demonstrate the absence of other pesticides in the blank material.

Compound	Extraction	IS	Determinative analysis		Notes
Bifenazate [1)]	**Modified QuEChERS-method** [3] involving: weighing of 10 g strawberry purée into a sealable vessel, addition of IS / ILISs, extraction with ACN + 1 % formic acid (15 min), addition of partitioning salts (4 g $MgSO_4$, 1 g NaCl), 1 min shaking, centrifugation (twice with interval of 30 min and cooling down to 5 °C), and direct determination by LC-MS/MS in the ESI (neg.) and ESI (pos) mode.	**Chlorpyrifos D_{10}**	**LC-MS/MS**	**ESI (pos)**	
Carbofuran [2)]		**Carbofuran D_3**	**LC-MS/MS**	**ESI (pos)**	
Fenbutatin Oxide		**Fenbutatin Oxide D_{30}**	**LC-MS/MS**	**ESI (pos)**	
2,4-D		**2,4-D D_3**	**LC-MS/MS**	**ESI (neg)**	
Dithianon		**Dithianon D_4**	**LC-MS/MS**	**ESI (neg)**	
Haloxyfop		**Haloxyfop D_4**	**LC-MS/MS**	**ESI (neg)**	
Chlorothalonil		**Chlorpyrifos D_{10}**	**GC-MS/MS**	**EI (pos)**	
Captan (parent)		**Captan D_6**	**GC-MS/MS**	**EI (pos)**	
Folpet (parent)		**Folpet D_4**	**GC-MS/MS**	**EI (pos)**	
Phthalimide		**Chlorpyrifos D_{10}**	**GC-MS/MS**	**EI (pos)**	
THPI		**Chlorpyrifos D_{10}**	**GC-MS/MS**	**EI (pos)**	
Captan (sum)		calculated from captan (parent) and THPI			
Folpet (sum)		calculated from folpet (parent) and phthalimide			
*Abamectin**		**Chlorpyrifos D_{10}**	*LC-MS/MS*	*ESI (pos)*	
*Fluazifop**		*BNPU*	*LC-MS/MS*	*ESI (neg)*	
Bromide ion	**QuPPe-PO method** [5] involving: weighing of 10 g strawberry purée into a sealable vessel, addition of ILISs, addition of methanol containing 1 % formic acid, shaking, centrifugation, filtration and direct determination by LC-MS/MS in the ESI (neg.) or ESI (pos.) mode.		**LC-MS/MS**	**ESI (neg)**	**QuPPe M1.4**
Chlorate		**Chlorate $^{18}O_3$**	**LC-MS/MS**	**ESI (neg)**	**QuPPe M1.4**
Glyphosate		**Glyphosate 1,2-$^{13}C_2$, ^{15}N**	**LC-MS/MS**	**ESI (neg)**	**QuPPe M1.3**
N-Acetyl glyphosate		**N-Acetyl glyphosate $^{13}C_2$, ^{15}N**	**LC-MS/MS**	**ESI (neg)**	**QuPPe M1.4**
Phosphonic acid		**Phosphonic acid $^{18}O_3$**	**LC-MS/MS**	**ESI (neg)**	**QuPPe M1.4**
*AMPA**		*AMPA ^{13}C ^{15}N*	*LC-MS/MS*	*ESI (neg)*	*QuPPe M1.3*
*Cyromazine**		*Cyromazine D_4*	*LC-MS/MS*	*ESI (pos)*	*QuPPe M4.2*
*Ethephon**		*Ethephon D_4*	*LC-MS/MS*	*ESI (neg)*	*QuPPe M1.3*
*Propamocarb**		*Propamocarb D_7*	*LC-MS/MS*	*ESI (pos)*	*QuPPe M4.2*
CS_2	**Dithiocarbamate method** involving: weighing of 20 g strawberry purée into a sealable glass vessel, addition of chloroform (as IS), 25 ml iso-octane and 150 ml $SnCl_2$/HCl, followed by cleavage to CS_2 in a shaking water bath for 2 h at 80° C and GC-ECD analysis.	**Chloroform**	**GC-ECD**	–	

* : To check for absence in Blank Material

1) Conversion of bifenazate-diazene, possibly present in the final QuEChERS extract, into bifenazate with ascorbic acid.

2) Conversion of carbosulfan, benfuracarb and furathiocarb, possibly present in the QuEChERS extract, into carbofuran with 5 N H_2SO_4 at 80 °C.

1.8 Homogeneity Test

After filling the test item in the bottles, 10 bottles were randomly chosen for the homogeneity test and two analytical portions were taken from each for analysis. Both the order of sample preparation and the order of extract injection into the analytical instruments were random. Matrix-matched calibration using extract prepared from blank material or procedural calibration using blank material were applied for quantification. Analytical portions of 20 g for ***dithiocarbamates*** and 10 g for all other compounds were used.

The statistical evaluation of the homogeneity test data was performed according to the International Harmonized Protocols published by IUPAC, ISO and AOAC [4, 6]. An overview of the statistical evaluations of the homogeneity test is shown in **Table 1-3**. The individual residue data of the homogeneity test is given in **Appendix 3**.

The acceptance criterion for the test item to be sufficiently homogeneous for the Proficiency Test was that $s_{sam}{}^2$ is smaller than c with s_{sam} being the between-bottle sampling standard deviation and $c = F_1 \times \sigma_{all}{}^2 + F_2 \times s_{an}{}^2$, F_1 and F_2 being constants with values of 1.88 and 1.01, respectively, and applying when duplicate samples are taken from 10 bottles. $\sigma_{all}{}^2 = 0.3 \times$ FFP-RSD (25 %) $\times$ the analytical sampling mean of the analyte, and s_{an} is the estimate of the analytical standard deviation.

As all target compounds passed the homogeneity test, the test item was considered to be sufficiently homogenous and suitable for the EUPT-SRM12.

1.9 Storage Stability Test

In the Specific Protocol laboratories were recommended storing the samples in the freezer until analysis. The stability test samples were thus also stored under the same conditions. Shortly after the shipment of the samples to the participants, three of the spare test item bottles were chosen randomly and all analytical portions necessary for all three stability tests were weighed into the vessels in which the analysis was to be conducted. The portions of stability tests 1 were extracted immediately and those of stability tests 2 and 3 were placed in the freezer at –20 °C until analysis as described in **Section 1.7 (p. 7)**. The extracts of all stability tests corresponding to one method were stored in the freezer at –20 °C and measured isochronically (within the same sequence) at a day suitable for the laboratory.

Stability test 1 (extraction shortly after shipment):
- 24 March 2017 (analytes via QuPPe-Methods)
- 29 March 2017 (analytes via QuEChERS-Methods)
- 06 April 2017 (***dithiocarbamates***)

Stability test 2 (extraction five weeks after shipment):
- 13 April 2017 (analytes via QuPPe-Methods)
- 19 April 2017 (analytes via QuEChERS-Methods)
- 27 April 2017 (***dithiocarbamates***)

Stability test 3 (extraction four weeks after deadline for results submission):
- 12 May 2017 (analytes via QuPPe-Methods)
- 11 May 2017 (analytes via QuEChERS-Methods)
- 18 May 2017 (***dithiocarbamates***)

Table 1-3: Statistical evaluation of homogeneity test data (n = 20), details please see **Appendix 3**.

COMPULSORY COMPOUNDS								
	2,4-D	Captan (parent)	Chlorothalonil	Dithiocarbamates	Fenbutatin oxide	Folpet (parent)	Glyphosate	Haloxyop
Analytical portion size [g]	10	10	10	20	10	10	10	10
Mean [mg/kg]	0.079	0.094	0.135	0.261	0.096	0.395	0.305	0.074
s_{sam}^2	8.72×10^{-7}	0.00	0.00	0.00	0.00	0.00	2.66×10^{-4}	6.44×10^{-7}
c	6.76×10^{-5}	1.02×10^{-4}	2.34×10^{-4}	1.23×10^{-3}	1.01×10^{-4}	1.72×10^{-4}	1.64×10^{-3}	6.20×10^{-5}
Passed/Failed	passed	passed	passed	passed	passed	passed	passed	passed
OPTIONAL COMPOUNDS								
	Bifenazate (sum)	Bromide ion	Carbofuran (part of sum)	Chlorate	Dithianon	Phosphonic acid	N-Acetyl-glyphosate	
Analytical portion size [g]	10	10	10	10	10	10	10	
Mean [mg/kg]	0.275	21.0	0.0043	0.489	0.314	18.5	0.088	
s_{sam}^2	3.78×10^{-7}	0.00	9.44×10^{-10}	1.18×10^{-4}	3.87×10^{-5}	0.00	1.49×10^{-6}	
c	8.89×10^{-4}	5.96	2.13×10^{-7}	2.74×10^{-3}	1.13×10^{-3}	3.99	9.89×10^{-5}	
Passed/Failed	passed	passed	passed	passed	passed	passed	passed	
ADDITIONAL COMPOUNDS								
	Captan (sum)	Folpet (sum)	Phthalimide	THPI				
Analytical portion size [g]	10	10	10	10				
Mean [mg/kg]	0.288	1.196	0.099	0.396				
s_{sam}^2	0.00	2.86×10^{-5}	0.00	0.00				
c	9.63×10^{-4}	1.70×10^{-2}	1.25×10^{-4}	2.18×10^{-3}				
Passed/Failed	passed	passed	passed	passed				

s_{sam}^2 : sampling variance; *c*: critical value

A target compound is considered to be adequately stable if $|y_i - y| \leq 0.3 \times \sigma_{pt}$, where y_i is the mean value of the last period of the stability test, y is the mean value of the first period of the stability test and σ_{pt} the standard deviation used for proficiency assessment, typically 25 % of the assigned value. With the exception of ***phthalimide*** and ***THPI***, all other analytes contained in the test item showed a stability within the acceptable limits when stored under the recommended conditions (–18 °C) within a period exceeding the duration of the exercise by two weeks (**Table 1-4, p. 10**). For the compounds passing the test it is assumed that, if the recommended storage conditions were followed, the influence of sample storage on the results of these analytes was negligible at least throughout the duration of the EUPT.

Table 1-4: Results of storage stability test (storage at -18 °C). Please see the text or **Appendix 4** for the dates of analysis for each analytes.

COMPULSORY COMPOUNDS								
	2,4-D	Captan (parent)	Chlorothalonil	Dithiocarbamates	Fenbutatin oxide	Folpet (parent)	Glyphosate	Haloxyop
Storage at –18 °C (mean values in mg/kg)								
Analysis 1 24. + 29.03.2017 + 06.04.2017	0.079	0.087	0.141	0.258	0.097	0.398	0.306	0.075
Analysis 2 13. + 19. + 27.04.2017	0.079	0.093	0.134	0.260	0.098	0.393	0.292	0.074
Analysis 3 11. + 12. + 18.05.2017	0.078	0.088	0.137	0.244	0.099	0.388	0.287	0.073
Deviation [mg/kg] ([%]) Analysis 3 vs. Analysis 1	0.001 (-0,8 %)	0.001 (1.3 %)	0.004 (-3.4 %)	0.014 (-5.6 %)	0.002 (2.0 %)	0.010 (-2.5 %)	0.019 (-6.3 %)	0.001 (-1,8 %)
$0.3 \times \sigma_{pt}$ [mg/kg]	0.006	0.006	0.009	0.020	0.006	0.025	0.023	0.005
Passed/Failed	passed	passed	passed	passed	passed	passed	passed	passed

OPTIONAL COMPOUNDS								
	Bifenazate (sum)	Bromide ion	Carbofuran (part of sum)	Chlorate	Dithianon	Phosphonic acid	N-Acetyl-glyphosate	
Storage at –18 °C (mean values in mg/kg)								
Analysis 1 24. + 29.03.2017	0.290	20.1	0.0043	0.431	0.322	18.4	0.087	
Analysis 2 13. + 19.04.2017	0.284	20.0	0.0042	0.451	0.318	19.0	0.087	
Analysis 3 11. + 12.05.2017	0.283	19.8	0.0045	0.434	0.325	18.4	0.085	
Deviation [mg/kg] ([%]) Analysis 3 vs. Analysis 1	0.007 (-2.4 %)	0.383 (-1.9 %)	0.0002 (4.0 %)	0.002 (0.5 %)	0.003 (0,9 %)	0.057 (-0.3 %)	0.001 (-1.7 %)	
$0.3 \times \sigma_{pt}$ [mg/kg]	0.020	1.433	0.0002	0.037	0.022	1.448	0.008	
Passed/Failed	passed	passed	passed	passed	passed	passed	passed	

ADDITIONAL COMPOUNDS								
	Captan (sum)	Folpet (sum)	Phthalimide	THPI				
Storage at –18 °C (mean values in mg/kg)								
Analysis 1 29.03.2017	0.286	1.182	0.389	0.100				
Analysis 2 19.04.2017	0.293	1.199	0.400	0.101				
Analysis 3 11.05.2017	0.268	1.081	0.344	0.090				
Deviation [mg/kg] ([%]) Analysis 3 vs. Analysis 1	0.018 (-6.3 %)	0.101 (-8.5 %)	0.045 (-11.6 %)	0.010 (-9.7 %)				
$0.3 \times \sigma_{pt}$ [mg/kg]	0.023	0.090	0.033	0.008				
Passed/Failed	passed	**failed**	**failed**	**failed**				

In the case of ***phthalimide*** and ***THPI*** the determined concentrations in day 3 were by 11.6 and 9.7 %, respectively, lower than those determined in day 1. This decline was a bit higher than the tolerance of 8.725 %. Based on experience with these compounds, however, the more likely scenario would have actually been the degradation of the parents with a parallel increase of the ***THPI*** and ***phthalimide*** concentrations (see also preliminary test under **Section 1.2**). It should be emphasized, however, that the analysis of ***THPI*** and ***phthalimide*** is quite challenging as these compounds are additionally formed during the thermal degradation of ***captan*** and ***folpet*** in the GC-injector. The analytical results of ***THPI*** and ***phthalimide*** are thus associated with a considerable uncertainty and it is not unlikely that the determined deviations are more related to spurious analytical errors rather than to degradation. The higher concentration in day 2 compared to day 1 also points towards this direction. As ***THPI*** and ***phthalimide*** are only shown for informative purposes in the PT ("additional compounds"), no further measures were deemed necessary.

The results of all analyses conducted within the framework of the stability test are shown in **Table 1-4** and **Appendix 4**.

1.10 Transport Stability Test

With the exception of 4 laboratories where the shipments were retarded due to customs clearance delays or remote location, all other 135 laboratories (97 %) received their test items within 48 hours. Among these 135 laboratories 110 received the parcels within one day, all of them reporting that the material was embedded in dry ice. 18 among the 25 laboratories receiving the parcels between 24 and 48 hours reported that the material was received in fully frozen state, 5 laboratories reported that it was mostly frozen, and only 2 laboratories reported that it was mostly defrosted.

As the vast majority of the laboratories received their samples in frozen state, the organisers decided not to conduct the transport stability test in the current PT.

1.11 Organisational Aspects

1.11.1 Preparation and Distribution of a Tentative List of Obliged Laboratories

A tentative list of laboratories (NRLs and OfLs) obliged to participate in the current EUPT was compiled based on available information on NRL-status and commodity scope as recorded in the EURL-DataPool. The available information on the pesticide scope covered by the laboratories was not considered when drafting this list due to concerns that it might not be up-to-date and/or not applicable to the present commodity (strawberry). The tentative list was distributed to the OfLs and the NRLs so that all laboratories could check their own data including their status and contact information and report any errors. The reported errors were corrected, and a new version was released. The NRLs were reminded of their responsibility for their network and were prompted to carefully check the status, commodity scope and contact data of the OfLs within their network. They were also asked to amend and complement the list, if necessary, and to ensure that all obliged OfLs within their network were informed of this EUPT. It was made clear to all NRLs and OfLs that the list of obliged laboratories was tentative and that the real obligation for participation is deriving from Art. 28 of Reg. 396/2005/EC (for OfLs) and Art. 33 of Reg. 882/2004/EC (for NRL-SRMs). Following DG-SANTE instructions, obliged labs that were not intending to participate in the EUPT-SRM12 were instructed to provide explanations for their non-participation.

1.11.2 Announcement / Invitation and EUPT-SRM12-Website

Within the EURL-Web-Portal an EUPT-SRM12-Website was constructed with links to all documents relevant to this EUPT (i.e., Announcement/Invitation Letter, Calendar, Target Pesticides List, Specific Protocol and General EUPT Protocol). These documents were uploaded to the EURL-Web-Portal and the CIRCA BC.

The Announcement/Invitation Letter for the EUPT-SRM12 was published on the EUPT-SRM12-Website in December 2016 and was sent to all NRL-SRMs, all OfLs analysing pesticide residues in food and feeding stuff within the framework of official controls, all laboratories performing import controls according to Reg. 669/2009/EC, as far as they were tracked in the EURL-DataPool, as well as to EU laboratories analysing official organic samples within the frame of Reg. 889/2008/EC. The latter laboratories were considered eligible but not obliged to participate. It was indicated to the OfLs that their obligation to participate in EUPTs arises from Reg. 396/2005/EC, irrespective of the content of the tentative list of obliged laboratories. NRLs and OfLs from EFTA and EU-candidate countries were also invited if their contact data was available. A number of laboratories from third countries were also invited to take part in this exercise. The acceptance of their registration was decided, however, on a case by case basis, and the laboratories were informed individually of the acceptance or rejection of their registration.

1.11.3 Registration and Confidentiality

For the first time the participants were able to register for the EUPT via a website connected to the EURL-DataPool. It is intended to apply this concept to all EUPTs organized by the 4 EURLs dealing with pesticides in order to reduce the burden of labs participating in more than one EUPT per year and to avoid the administrative effort of crosschecking and updating data between different databases. All laboratories listed in the tentative list as being obliged to participate in the current EUPT, regardless of whether they were intending to participate in this exercise or not, were requested to either register or to state their reasons for non-participation using the same website.

Upon registration or change of registration status, the labs received an electronic confirmation about their participation or non-participation in the current PT. On the day of sample shipment, participating labs were provided via e-mail with a unique laboratory code as well as with unique, automatically generated login data to access the online Result-Submission-Website. This ensured confidentiality throughout the entire duration of the PT.

For further information on confidentiality please refer to the General EUPT Protocol (**Appendix 9**).

1.11.4 Distribution of the Test Items and the Blank Material

One bottle of test item (approx. 400 g), one bottle of blank material (approx. 400 g) and two vials containing isotope labelled internal standards (ILISs) of (***chlorate***/***perchlorate*** and ***phosphonic acid***) were shipped on 13 March, 2017 to each participant in thermo-insulated polystyrene boxes with dry ice. The packages for laboratories in countries where according to IATA Dangerous Goods Regulations shipments with dry ice were not allowed contained cooling elements instead of dry ice.

Three days prior to the shipment, detailed instructions on how to treat the test item and blank material upon receipt were provided to the participating laboratories in the Specific Protocol (**Appendix 10**). The participants were also informed on how the ILISs enclosed in the parcel could be employed in the analysis.

1.11.5 Submission of Results and Additional Information

An online submission tool allowed participants to submit their results via the Internet. Using their individual login data, all participants had access to the Result-Submission-Website from a week after the sample shipment until the result submission deadline (21 April, 2017). Participants were asked not only to report their analytical results but also to state whether the compounds on the Target Pesticides List were part of their routine scope and to indicate their experience with the analysis of these compounds. In addition, laboratories had to provide details about the methods applied and to state their own reporting limits (RLs) for each target compound they had analysed. The participants had furthermore the possibility to make statements as regards the condition of the material received. This information could be submitted from the day of shipment onwards.

1.11.6 Actions following Results Submission and Distribution Preliminary Report

Where information on analytical methods or results was inconsistent, laboratories were contacted. One laboratory, that had originally registered to participate in the current PT but finally did not submit any results, was asked to provide explanations. On 11 May, 2017, the preliminary report on the EUPT-SRM12 with the preliminary assigned values was released and sent to the participants. Laboratories having submitted false positive or negative results were asked to provide information on the methods used for analysing those compounds. In addition, participants were asked to investigate the reasons for results with $|\text{z-score}| > 2$ and to report them. In order to have the complete and correct data for the evaluation, a reminder was sent to the participants again to fill in all the data requested on the submission page for the methodological information.

In order to obtain feedback from the participants and to improve the service quality in the future, parallel to the release of the preliminary report the organisers invited the participants to participate in a survey on EUPT-SRM12. The survey contained 5 questions on the organisation (general, registration, information and instruction provided, shipment/delivery, test item, blank material, ILIS standards provided and results submission pages), on the relevance of the used matrix (strawberry) to the routine work, on the assigned values of the analytes, as well as on the preliminary report and wishes as regards the commodities and/or analytes to be included in the upcoming two EUPT-SRMs. 127 of 139 participants (91 %) took part in the survey. The evaluation and compilation of comments was published on 27 July and can be downloaded via http://www.eurl-pesticides.eu/library/docs/srm/SRM12_Survey_Statistics_Evaluation.pdf.

2. EVALUATION RULES

2.1 False Positives and Negatives

2.1.1 False Positives (FPs)

Any reported result with a concentration at or above the Minimum Required Reporting Level (MRRL) of an analyte in the Target Pesticides List which was (a) not detected by the organiser, even following repetitive analysis, and/or (b) not detected by the overwhelming majority (e.g. > 95 %) of the participants that analysed for this compound, is treated as a false positive result. Results of an analyte absent in the test item but with a value lower than the MRRL are excluded by the organiser and not considered as false positives. No z-scores are calculated for false positive results.

2.1.2 False Negatives (FNs)

These are results of target analytes reported as "analysed" but without reporting numerical values, although they were used by the organiser to prepare the test item and were detected, at or above the MRRL, by the organiser and the overwhelming majority of the participating laboratories. In accordance with the General Protocol z-scores for false negatives are calculated using the MRRL as the result, or using the lab's reporting-limit (RL), if this is lower. Any RLs that are higher than the MRRL are not taken into account. Following the General Protocol, results reported as "< RL" without providing a numerical value are also judged as false negatives if the RL exceeds the MRRL.

2.2 Assigned Values (x_{pt}) and Calculation of the Respective Uncertainties ($u(x_{pt})$)

In accordance with EUPT-General Protocol (**Appendix 8**) the assigned values x_{pt} of each pesticide in the PT is established using the mean value of robust statistics using Algorithms A (x^*) [6] of all reported results from EU and EFTA countries. Results associated with obvious mistakes and gross errors may be excluded from the population for the establishment of the assigned values. The add-in "RobStat" provided by Royal Society of Chemistry was used to calculate the assigned values with the convergence criterion = 10^{-6}.

The uncertainty of the assigned values of each analyte is calculated according to ISO 13528:2015 [6] using the following equation:

$$u(x_{pt}) = 1.25 \times [(s^*)/\sqrt{p}\,]$$

Where $u(x_{pt})$ is the uncertainty of the assigned value in mg/kg, s^* is the robust standard deviation estimate in mg/kg and p is the number of data points considered (= the number of results used to calculate the assigned value). The factor 1.25 is based on the standard deviation of the median, or the efficiency of the median as an estimate of the mean, in a large set of results drawn from a normal distribution.

The tolerance for the uncertainty of the assigned value of each pesticide is calculated as $0.3 \times FFP\text{-}\sigma_{pt}$, where $FFP\text{-}\sigma_{pt}$ is the target standard deviation of the assigned value derived using a fixed standard deviation of 25 % (see **Section 2.3**). If $u(x_{pt}) < 0.3 \times FFP\text{-}\sigma_{pt}$, is met, then the uncertainty of the assigned value is considered to be negligible and not needed to be considered in the interpretation of the proficiency test results.

2.3 Fixed Target Standard Deviation using FFP-Approach ($FFP\text{-}\sigma_{pt}$)

Based on experience from previous EU Proficiency Tests on fruit and vegetables and cereals, the EUPT-Scientific Committee agreed to apply a fixed fit-for-purpose relative standard deviation (FFP-RSD) of 25 % for calculating the z-scores. The fixed target standard deviation using the fit-for-purpose approach ($FFP\text{-}\sigma_{pt}$), for each individual target analyte is calculated by multiplying the assigned value by the FFP-RSD of 25 %. In addition, the robust relative standard deviation of the assigned value (CV^*) is calculated for informative purposes.

2.4 z-Scores

For each combination of laboratory and target analyte a z-score is calculated according to the following equation:

$$z_i = (x_i - x_{pt}) / FFP\text{-}\sigma_{pt}$$

Where

- x_i is the result for the target analyte (i) as reported by the participant
  (For results considered as false negatives, x_i is set as equal to the respective minimum required reporting level (MRRL) or the laboratory reporting level (RL), if RL < MRRL.)
- x_{pt} is the assigned value for the target analyte (i)
- $FFP\text{-}\sigma_{pt}$ is the standard deviation for proficiency assessment using the fit-for-purpose approach (see above).

  Any z-scores > 5 are set at 5 in calculations of combined z-scores (see **2.5.2**).

The z-scores are classified as follows:

$\|z\| \leq 2$	acceptable
$2 < \|z\| < 3$	questionable
$\|z\| \geq 3$	unacceptable

For results considered as false negatives, z-scores are calculated using the MRRL or the RL, if RL < MRRL. No z-scores are allocated to false positive results.

2.5 Laboratory Classification

2.5.1 Category A and B classification

Based on the scope of target analytes covered by the laboratories in this exercise, laboratories are subdivided into Categories (A and B) in accordance with the rules in the General Protocol (**Appendix 8**). To be classified into Category A a laboratory should

a) have analysed at least 90 % of the compulsory pesticides on the Target Pesticides List,
b) have correctly reported concentration values for at least 90 % of the compulsory pesticides present in the test item,
c) not have reported any false positive results.

2.5.2 Combined z-Scores

For informative purposes and to allow comparison of the overall performance of the laboratories the Average of the Absolute z-Scores (AAZ) is calculated for laboratories with 5 or more z-scores. **Combined z-scores are, however, considered to be of lesser importance than the individual z-scores**.

Average of the Absolute z-Scores (AAZ)

The AAZ is calculated using the following formula:

$$AAZ = \frac{\sum_{i=1}^{n} |z_i|}{n}$$

where "n" is the number of each laboratory's z-scores that are considered in this formula. This includes z-scores assigned for false negative results.
For the calculation, any z-score > 5 is set at 5.

3. PARTICIPATION

139 laboratories from 39 countries (28 EU-Member States, 3 EFTA- countries, 1 EU-candidate country and 7 third countries) originally registered for participation in the EUPT-SRM12. Out of those laboratories only one EU-Member State laboratory failed to submit any results, reporting after the PT that the analytes on the Target Pesticides List were out of its routine scope and due to a lack of analytical standards the two analytes detected in a screening could not be quantified. This lab was therefore retroactively regarded as non-participating. An overview of the participating laboratories and countries is given in **Table 3-1**.

A list of all individual laboratories that registered for this EUPT is presented in **Appendix 1**. Croatia was the only EU-country not represented by an NRL-SRM. Malta was represented by its proxy-NRL-SRM based in the United Kingdom.

All 13 laboratories from non-EU countries submitted results (4 from EFTA countries, 2 from one EU-candidate country and 7 from third countries). For the first time one OfL from Iceland has participated in an EUPT-SRM. The results submitted by the laboratories based in Serbia (EU candidate country) and by the 7 laboratories based in third countries were not taken into account when calculating the assigned values.

In total, 174 EU-OfLs (including NRL-SRMs) were originally considered as being obliged to participate in the present EUPT and were included on a tentative list of obliged labs that was distributed to the labs of the network prior to the registration period for this EUPT. The list included all NRL-SRMs, regardless of their commodity scope, and all EU-OfLs analysing for pesticide residues in cereals or feed.

All labs tentatively considered as obliged to participate were invited to log in the registration page and register for their participation in the current PT or to provide an explanation for their non-participation.

26 obliged laboratories explained their non-participation with the fact that the matrix (strawberry) or the SRM12 target pesticides or both were out of their routine scope, partly due to a lack of required instruments. Excluding those 26 laboratories that provided sufficient explanations, the number of EU-laboratories considered as being obliged decreased to 148. Out of the 110 obliged laboratories that have registered for this PT 109 laboratories finally submitted result. The laboratory which failed to report any results was retrospectively classified as not obliged to participate, since the analytes were reportedly out of its routine scope. The number of EU-laboratories considered as being obliged thus decreased to 147. Out of the 147 obliged OfLs 38 (26 %) did neither register for the PT nor provide any explanation for non-participation. These laboratories originated from 12 countries as follows: BG 1, HR 2, FR 2, DE 3, IT 5, NL 1, PL 6, PT 1, RO 2, SK 1, ES 11 and UK 3.

Table 3-1: Number of laboratories listed as being obliged to participate in the EUPT-SRM12, labs that registered to participate, and labs that finally submitted results (grouped by contracting country)

EU: NRLs and OfLs										
Contracting Country [1)]	Labs originally considered as obliged (*based on scope)	Labs providing sufficient expl. for non-participation		Finally considered as obliged	Registered for Participation		Submitted Results		Obliged labs non particip. w/o giving expl.	Notes
		Prior to PT	During the PT		All	NRL-SRMs	All	NRL-SRMs		
AT	2	0	0	2	2 + [1]	1	2 + [1]	1		
BE	6	0	0	6	6 + [1]	1	6 + [1]	1		
BE/NL	1	0	0	1	1	0	1	0		
BE/BG/FR/LU	1	0	0	1	1	0	1	0		
BG	3	1	0	2	1	1	1	1	1	
HR	9	2	0	7	5	0	5	0	2	HR has not yet established an NRL-SRM.
CY	1	0	0	1	1	1	1	1		
CZ	2	0	0	2	2 + [1]	1	2 + [1]	1		
DK	1	0	0	1	1	1	1	1		
EE	2	0	0	2	2	1	2	1		
FI	3	1	0	2	2	1	2	1		
FR	10	0	0	10	8 + [1]	1	8 + [1]	1	2	
DE	21	2	0	19	16 + [2]	1	16 + [2]	1	3	
DE/MT	1	0	0	1	1	0	1			
GR	3	1	0	2	2 + [1]	2	2 + [1]	2		GR has appointed two NRL-SRMs.
HU	5	0	0	5	5	1	5	1		
IE	1	0	0	1	1	1	1	1		
IT	20	5	1	14	9 + {1}	1	9	1	5	
IT/MT	1	0	0	1	1	0	1	0		
LV	1	0	0	1	1	1	1	1		
LT	1	0	0	1	1 + [1]	1	1 + [1]	1		
LU	1	0	0	1	1	1	1	1		
MT	0*	0*	0*	0*	0*	0*	0*	0*		*MT-NRL-SRM represented by proxy by the UK-NRL-SRM; MT subcontracted routine analysis to an OfLs in DE and IT
NL	2	0	0	2	1	1	1	1	1	
PL	11	1	0	10	4 + [4]	1	4 + [4]	1	6	
PT	4	0	0	4	3	1	3	1	1	
RO	5	1	0	4	2 + [1]	1	2 + [1]	1	2	
SK	2	0	0	2	1	1	1	1	1	
SI	3	0	0	3	3	1	3	1		
ES	41	10	0	31	20 + [2]	2	20 + [2]	2	11	ES has appointed two NRL-SRMs
ES/MT	1	10	0	1	1 + [2]	0	1 + [2]	0		
SE	2	0	0	2	2	1	2	1		
UK/MT	1	0	0	1	1	1	1	1		UK-NRL-SRM represents also MT
UK	4	0	0	4	1 + [1]		1 + [1]		3	
EU-total	**172**	**24**	**1**	**147**	**110 + [16]**	**28**	**109 + [16]**	**28**	**38**	

Table 3-1 (cont.): Number of laboratories listed as being obliged to participate in the EUPT-SRM12, labs that registered to participate, and labs that finally submitted results (grouped by contracting country)

EFTA										
Contracting Country [1)]	Labs originally considered as obliged (*based on scope)	Labs providing sufficient expl. for non-participation		Finally considered as obliged	Registered for Participation		Submitted Results		Obliged labs non particip. w/o giving expl.	Notes
		Prior to PT	During the PT		All	NRL-SRMs	All	NRL-SRMs		
NO					[1]	1	[1]	1		
IS					[1]	–	[1]	–		
CH					[2]	–	[2]	–		
EU+EFTA Total					110+[20]	29	109+[20]	29		
Third Countries / EU candidate country										
SR					2	–	2	–		
CA					1	–	1	–		
CR					1	–	1	–		
EG					1	–	1	–		
HK					1	–	1	–		
MY					1	–	1	–		
KR					1	–	1	–		
TW					1	–	1	–		
Third Countries / EU candidate country Total					9		9			
Overall Sum				147	139	29	138	29		

4. RESULTS

4.1 Overview of Results

An overview of the percentage of laboratories having targeted each of the analytes present in the Target Pesticides List is shown in **Table 4-1**.

Table 4-2 (p. 24) gives an overview of all results submitted by each laboratory. The individual numerical results reported by the laboratories are shown in **Table 4-8 (p. 42), Table 4-9 (p. 48)** and **Table 4-10 (p. 56)** for compulsory, optional and additonal compounds, respectively. Originally, four analytes, ***captan (sum)***, ***folpet (sum)***, ***THPI*** and ***phthalimid***, were considered for data collection only and regarded as "additional compounds". Since the number and the quality of the submitted results was high, finally, the organizers decided to proceed with a laboratory-based evaluation of the assiged values, z-scores, FNs and FPs also for these four analytes and to show this data for informative purposes only. Detailed information about

Table 4-1: Percentage of EU and EFTA laboratories that have analysed for the compounds in the Target Pesticides List

Compounds		Present in test item	Labs analysed for the compound			
			EU [1]- and EFTA-Labs		EU obliged Labs only	
			No. [2]	% (based on *n* = 129 [3])	No. [2]	% (based on *n* = 147 [4])
Compulsory Compounds	**2,4-D**	**Yes**	98	76 %	82	56 %
	Abamectin	No	97	75 %	81	55 %
	Captan (parent)	**Yes**	93	72 %	77	52 %
	Chlorothalonil	**Yes**	111	86 %	94	64 %
	Cyromazine	No	97	75 %	82	56 %
	Dithiocarbamates	**Yes**	107	83 %	90	61 %
	Ethephon	No	76	59 %	64	44 %
	Fenbutatin Oxide	**Yes**	82	64 %	70	48 %
	Fluazifop	No	99	77 %	83	56 %
	Folpet (parent)	**Yes**	98	76 %	81	55 %
	Glyphosate	**Yes**	86	67 %	72	49 %
	Haloxyfop	**Yes**	97	75 %	82	56 %
	Propamocarb	No	109	84 %	92	63 %
Optional Compounds	**AMPA**	No	58	45 %	48	33 %
	Bifenazate (sum)	**Yes**	54	42 %	42	29 %
	Bromide ion	**Yes**	52	40 %	44	30 %
	Carbofuran (part of sum)	**Yes**	74	57 %	62	42 %
	Chlorate	**Yes**	60	47 %	47	32 %
	Dithianon	**Yes**	64	50 %	52	35 %
	Phosphonic acid	**Yes**	50	39 %	39	27 %
	N-Acetyl glyphosate	**Yes**	16	12 %	13	9 %
Additional Compounds	**Captan (sum)**	**Yes**	65	50 %	52	35 %
	Folpet (sum)	**Yes**	66	51 %	53	36 %
	THPI	**Yes**	67	52 %	54	37 %
	Phthalimide	**Yes**	67	52 %	54	37 %

1) Including official laboratories participating on voluntary basis
2) Laboratories representing more than one country were counted only once.
3) 129 is the number of participating OfLs from EU and EFTA countries (including NRLs and official laboratories participating on voluntary basis) having registered for the present PT and submitted at least one result.
4) 147 is the number of OfLs (including NRLs) from EU countries, which were finally considered as obliged to participate in the EUPT-SRM12 (taking into account any explanations for non-participation).

the analytical methods used by the laboratories is shown in the web under "EUPT-SRM12 - Supplementary Information" accessible via the the link: http://www.eurl-pesticides.eu/library/docs/srm/EUPT-SRM12_Supplementary_Information.pdf.

Table 4-2: Scope and categorization of participating laboratories (including third country laboratories and laboratories that have not submitted results)

Compulsory Compounds															
Compulsory Compound listed in Target List			**2,4-D**	**Abamectin**	**Captan (parent)**	**Chlorothalonil**	**Cyromazine**	**Dithiocarbamates**	**Ethephon**	**Fenbutatin Oxide**	**Fluazifop**	**Folpet (parent)**	**Glyphosate**	**Haloxyfop**	**Propamocarb**	**Analysed / correctly found among COMPULSORY compounds (max. 13 / 8)**
within MACP [1]			**Reg.**	**Reg.**	**Reg.**	**Reg.**	**Reg.**	**Reg.**	**Reg.**	**Reg.**	**Reg.**	**Reg.**	**Reg.**	**Reg.**	**Reg.**	
present in Test Item			**Yes**	No	**Yes**	**Yes**	No	**Yes**	No	**Yes**	No	**Yes**	**Yes**	**Yes**	No	
evaluated in this PT			**Yes**	No	**Yes**	**Yes**	No	**Yes**	No	**Yes**	No	**Yes**	**Yes**	**Yes**	No	
Lab-Code SRM12-	**NRL-SRM**	**Cat.** [2]														
1		A	V	ND	V	V	ND	V	ND	V	ND	V	V	V	ND	13 / 8
2		A	V	ND	V	V	ND	V	ND	**FN**	ND	V	V	V	ND	13 / 7
3	x	A	V	ND	V	V	ND	V	ND	V	ND	V	V	V	ND	13 / 8
4		B	V	ND	V	V	ND	V		V	ND	V		V	ND	11 / 7
5		B						V								1 / 1
6	x	A	V	ND	V	V	ND	V	ND	V	ND	V	**FN**	V	ND	13 / 7
7		A	V	ND	V	V	ND	V	ND	V	ND	V	V	V	ND	13 / 8
8		B	V	ND			ND	V	ND		ND		V	V	ND	9 / 4
9		A	V	ND	V	V	ND	V	ND	V	ND	V	V	V	ND	13 / 8
10		B	V			V	ND	V	ND		ND		V	V	ND	9 / 5
11		B	V	ND		V					ND			V	ND	6 / 3
12		A	V	ND	V	V	ND	V	ND	V	ND	V	V	V	ND	13 / 8
13	x	A	V	ND	V	V	ND	V	ND	V	ND	V	V	V	ND	13 / 8
14		A	V	ND	V	V	ND	V	ND	V	ND	V	V	V	ND	13 / 8
15		B	V	ND	V	V	ND	V			ND	V		V	ND	10 / 6
16	x	B	V	ND		V	ND	V	ND	V	ND		V	V	ND	11 / 6
17	x	A	V	ND	V	V	ND	V	ND	V	ND	V	V	V	ND	13 / 8
18		A	V	ND	V	V	ND	V	ND	V	ND	V	V	V	ND	13 / 8
19	x	A	V	ND	V	V	ND	V	ND	V	ND	V	V	V	ND	13 / 8
20		A	V	ND	V	V	ND	V	ND	V	ND	V	V	V	ND	13 / 8
21		A	V	ND	V	V	ND	V	ND	V	ND	V	V	V	ND	13 / 8
22		A	V	ND	V	V	ND	V	ND	V	ND	V	V	V	ND	13 / 8

1) MACP = EU Multiannual Control Program; Reg.: MACP Regulation; WD: MACP Working Document ("Working document on pesticides to be considered for inclusion in the national control programmes to ensure compliance with maximum residue levels of pesticides residues in and on food of plant and animal origin")

2) Category A/B classification (Cat A was assigned to laboratories that have analysed at least 12 out of the 13 compulsory compounds on the Target Pesticides List, correctly detected 7 or more out of the 8 compulsory compounds present in the test item and have not reported any false positive result, see **Section 4.4.4, p. 59**)

V = analysed for and submitted concentration Value > "MRRL" for a pesticide present in the test item; **ND** = analysed for and correctly reported as "Not Detected"; **Empty cells**: not analysed; **FN** = analysed for but falsely not detected (False Negative result); **FN*** = analysed for a compound present in the test material and reported not detected due to lab's RL being > assigned value, therefore judged as FN; **FP** = false positive result (FP): Result reported as "≤ MRRL" and, therefore, not regarded as FP.

Table 4-2 (cont.): Scope and categorization of participating laboratories (including third country laboratories and laboratories that have not submitted results)

			Optional Compounds									Total	Additional Compounds				
Optional / Additional Compound listed in Target List			AMPA	Bifenazate	Bromide ion	Carbofuran	Chlorate	Dithianon	Phosphonic acid	N-Acetyl glyphosate	Analysed / correctly found among OPTIONAL compounds (max. 8 / 7)	Analysed / correctly found among COMPULSORY and OPTIONAL compounds (max. 21 / 15)	Captan (sum)	Folpet (sum)	THPI	Phthalimide	Analysed / correctly found among ADDITIONAL compounds within the EUPT-Target Pesticides List (max. 4 / 4)
within MACP [1]			WD	WD	Reg.	Reg.	WD	Reg.	Reg.	WD			Reg.	Reg.	Reg.	Reg.	
present in Test Item			No	Yes	Yes	Yes	Yes	Yes	Yes	Yes			Yes	Yes	Yes	Yes	
evaluated in this PT			No	Yes	Yes	No	Yes	Yes	Yes	Yes			Yes	Yes	Yes	Yes	
Lab-Code SRM12-	NRL-SRM	Cat. [2]															
1		A	ND	V	V	V	V	V	V		7 / 6	20 / 14	V	V	V	V	4 / 4
2		A	ND	V	V	**FN**	V	V	V		7 / 5	20 / 12	V	V	V	V	4 / 4
3	x	A	ND	V		V		V			4 / 3	17 / 11					0 / 0
4		B				V		V			2 / 2	13 / 9	V	V	V	V	4 / 4
5		B									0 / 0	1 / 1					0 / 0
6	x	A	ND	V		**FN***	V	V	V	**FN**	7 / 4	20 / 11					0 / 0
7		A	ND	V	V		V		V		5 / 4	18 / 12	V	V	V	V	4 / 4
8		B	ND		V	V	V		V	V	6 / 5	15 / 9					0 / 0
9		A				V	V		V		3 / 3	16 / 11	V		V		2 / 2
10		B	ND			V	V		V		4 / 3	13 / 8					0 / 0
11		B									0 / 0	6 / 3					0 / 0
12		A	ND	V	V	**FN***	V	V	V		7 / 5	20 / 13					0 / 0
13	x	A	ND		V	V		V			4 / 3	17 / 11		V		V	2 / 2
14		A	ND				V	V	V		4 / 3	17 / 11	V	V	V	V	4 / 4
15		B			V						1 / 1	11 / 7					0 / 0
16	x	B	ND		V						2 / 1	13 / 7					0 / 0
17	x	A		V		V					2 / 2	15 / 10	V	V	V	V	4 / 4
18		A	ND	V	V	V	V	V	V		7 / 6	20 / 14	V	V	V	V	4 / 4
19	x	A			V		V	V			3 / 3	16 / 11	V	V	V	V	4 / 4
20		A				V	V	V		V	4 / 4	17 / 12	V	V	V	V	4 / 4
21		A	ND	V		V	V	V	V		6 / 5	19 / 13	V	V	V	V	4 / 4
22		A			V	V	V	V	V		5 / 5	18 / 13	V	V	V	V	4 / 4

1) MACP = EU Multiannual Control Program; Reg.: MACP Regulation; WD: MACP Working Document ("Working document on pesticides to be considered for inclusion in the national control programmes to ensure compliance with maximum residue levels of pesticides residues in and on food of plant and animal origin")

2) Category A/B classification (Cat A was assigned to laboratories that have analysed at least 9 out of the 11 compulsory compounds on the Target Pesticides List, correctly detected 7 or more out of the 8 compulsory compounds present in the test item and have not reported any false positive result, see **Section 4.4.4, p. 59**)

V = analysed for and submitted concentration Value > "MRRL" for a pesticide present in the test item; **ND** = analysed for and correctly reported as "Not Detected"; **Empty cells**: not analysed; **FN** = analysed for but falsely not detected (False Negative result); **FN*** = analysed for a compound present in the test material and reported not detected due to lab's RL > assigned value, therefore judged as FN; **FP** = false positive result (FP): Result reported as "≤ MRRL" and, therefore, not regarded as FP.

Table 4-2 (cont.): Scope and categorization of participating laboratories (including third country laboratories and laboratories that have not submitted results)

Compulsory Compounds																
Compulsory Compound listed in Target List			2,4-D	Abamectin	Captan (parent)	Chlorothalonil	Cyromazine	Dithiocarbamates	Ethephon	Fenbutatin Oxide	Fluazifop	Folpet (parent)	Glyphosate	Haloxyfop	Propamocarb	Analysed / correctly found among COMPULSORY compounds (max. 13 / 8)
within MACP [1]			Reg.	Reg.	Reg.	Reg.	Reg.	Reg.	Reg.	Reg.	Reg.	Reg.	Reg.	Reg.	Reg.	
present in Test Item			Yes	No	Yes	Yes	No	Yes	No	Yes	No	Yes	Yes	Yes	No	
evaluated in this PT			Yes	No	Yes	Yes	No	Yes	No	Yes	No	Yes	Yes	Yes	No	
Lab-Code SRM12-	NRL-SRM	Cat. [2]														
23		B		ND	V	V	ND		ND		ND	V	V	V	ND	10 / 5
24		B	V	ND	V	V	ND	V		V	ND	V		V	ND	11 / 7
25		A	V	ND	V	V	ND	V	ND	V	ND	V	V	V	ND	13 / 8
26		A	V	ND	V	V	ND	V	ND	V	ND	V	V	V	ND	13 / 8
27		B	V			**FN**	ND				ND	V			ND	6 / 2
28	x	B	V	ND		V	ND	V		V	ND		V	V	ND	10 / 6
29	x	B	V	ND	V	V	ND			V	ND	V		V	ND	10 / 6
30	x	A	V	ND	V	V	ND	V	ND	V	ND	V	V	V	ND	13 / 8
31		B			V	V		V				V				4 / 4
32		B	V		V	V	ND	V	ND		ND	V	V	V	ND	11 / 7
33	x	A	V	ND	V	V	ND	V	ND	V	ND	V	V	V	ND	13 / 8
34		B	V	ND		V	ND	V	ND	V	ND		V	V	ND	11 / 6
35	x	A	V	ND	V	V	ND	V	ND	**FN**	ND	V	V	V	ND	13 / 7
37		B		ND	V	V		V				V			ND	6 / 4
38		B	V	ND	V	V		V				V	V		ND	8 / 6
39		A	V	ND	V	V	ND	V	ND	V	ND	V	V	V	ND	13 / 8
40		B		ND		V	ND	V		V	ND	V	V		ND	9 / 5
41		A	V	ND	V	V	ND	V	ND	V	ND	V	V	V	ND	13 / 8
42		A	V	ND	V	V	ND	V	ND	V	ND	V	V	V	ND	13 / 8
43		B						V					V			2 / 2
44	x	B	**FN**	ND	V	V		V			ND	V		**FN**		8 / 4
45		A	V	ND	V	V	ND	V	ND	V	ND	V	V	V	ND	13 / 8
46		A	V	ND	V	V	ND	V	ND	V	ND	V	V	V	ND	13 / 8
47		A	V	ND	V	V	ND	V	ND	V	ND	V	V	V	ND	13 / 8
48		A	V	ND	V	V	ND	V	ND		ND	V	V	V	ND	12 / 7
49	x	A	V	ND	V	V	ND	V	ND	V	ND	V	V	V	ND	13 / 8

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2) Category A/B classification (Cat A was assigned to laboratories that have analysed at least 12 out of the 13 compulsory compounds on the Target Pesticides List, correctly detected 7 or more out of the 8 compulsory compounds present in the test item and have not reported any false positive result, see **Section 4.4.4, p. 59**)

V = analysed for and submitted concentration Value > "MRRL" for a pesticide present in the test item; **ND** = analysed for and correctly reported as "Not Detected"; **Empty cells**: not analysed; **FN** = analysed for but falsely not detected (False Negative result); **FN*** = analysed for a compound present in the test material and reported not detected due to lab's RL being > assigned value, therefore judged as FN; **FP** = false positive result (FP): Result reported as "≤ MRRL" and, therefore, not regarded as FP.

Table 4-2 (cont.): Scope and categorization of participating laboratories (including third country laboratories and laboratories that have not submitted results)

			Optional Compounds								Total	Additional Compounds				
Optional / Additional Compound listed in Target List			AMPA	Bifenazate	Bromide ion	Carbofuran	Chlorate	Dithianon	Phosphonic acid	N-Acetyl glyphosate	Analysed / correctly found among OPTIONAL compounds (max. 8 / 7)	Analysed / correctly found among COMPULSORY and OPTIONAL compounds (max. 21 / 15)	Captan (sum)	Folpet (sum)	THPI	Phthalimide	Analysed / correctly found among ADDITIONAL compounds within the EUPT-Target Pesticides List (max. 4 / 4)
within MACP [1]			WD	WD	Reg.	Reg.	WD	Reg.	Reg.	WD			Reg.	Reg.	Reg.	Reg.	
present in Test Item			No	Yes	Yes	Yes	Yes	Yes	Yes	Yes			Yes	Yes	Yes	Yes	
evaluated in this PT			No	Yes	Yes	No	Yes	Yes	Yes	Yes			Yes	Yes	Yes	Yes	
Lab-Code SRM12-	NRL-SRM	Cat. [2]															
23		B									0 / 0	10 / 5					0 / 0
24		B			V			V			2 / 2	13 / 9					0 / 0
25		A	ND	V		V		V		V	5 / 4	18 / 12	V	V	V	V	4 / 4
26		A	ND	V	V	V	V	V	V		7 / 6	20 / 14	V	V	V	V	4 / 4
27		B		V		**FN***					2 / 1	8 / 3		V	V		2 / 2
28	x	B						V			1 / 1	11 / 7					0 / 0
29	x	B		V		V					2 / 2	12 / 8	V	V	V	V	4 / 4
30	x	A	ND			V		V			3 / 2	16 / 10					0 / 0
31		B									0 / 0	4 / 4				V	1 / 1
32		B									0 / 0	11 / 7					0 / 0
33	x	A	ND	V	V	V	V	V	V	V	8 / 7	21 / 15	V	V	V	V	4 / 4
34		B	ND	V	V	V	V	V	V		7 / 6	18 / 12			V	V	2 / 2
35	x	A	ND	V	V	V	V	V	V	V	8 / 7	21 / 14	V	V	V	V	4 / 4
37		B									0 / 0	6 / 4					0 / 0
38		B				V	V				2 / 2	10 / 8					0 / 0
39		A	ND	V	V	**FN***	V	V		V	7 / 5	20 / 13	V	V	V	V	4 / 4
40		B	ND			V		V			3 / 2	12 / 7					0 / 0
41		A	ND	V	V	V		V	V		6 / 5	19 / 13	V	V	V	V	4 / 4
42		A	ND	V	V	V	V		V		6 / 5	19 / 13	V	V	V	V	4 / 4
43		B	ND				V				2 / 1	4 / 3					0 / 0
44	x	B									0 / 0	8 / 4					0 / 0
45		A		V	V	V	V				4 / 4	17 / 12					0 / 0
46		A	ND				V	V			3 / 2	16 / 10	V	V	V	V	4 / 4
47		A	ND	V	V		V	V	V		6 / 5	19 / 13	V	V	V	**FN**	4 / 3
48		A	ND	V	V	V	V				5 / 4	17 / 11	V	V	V	V	4 / 4
49	x	A			V	V					2 / 2	15 / 10	V	V	V	V	4 / 4

1) MACP = EU Multiannual Control Program; Reg.: MACP Regulation; WD: MACP Working Document ("Working document on pesticides to be considered for inclusion in the national control programmes to ensure compliance with maximum residue levels of pesticides residues in and on food of plant and animal origin")

2) Category A/B classification (Cat A was assigned to laboratories that have analysed at least 9 out of the 11 compulsory compounds on the Target Pesticides List, correctly detected 7 or more out of the 8 compulsory compounds present in the test item and have not reported any false positive result, see **Section 4.4.4, p. 59**)

V = analysed for and submitted concentration Value > "MRRL" for a pesticide present in the test item; **ND** = analysed for and correctly reported as "Not Detected"; **Empty cells**: not analysed; **FN** = analysed for but falsely not detected (False Negative result); **FN*** = analysed for a compound present in the test material and reported not detected due to lab's RL > assigned value, therefore judged as FN; **FP** = false positive result
(FP): Result reported as "≤ MRRL" and, therefore, not regarded as FP.

Table 4-2 (cont.): Scope and categorization of participating laboratories (including third country laboratories and laboratories that have not submitted results)

Compulsory Compounds																
Compulsory Compound listed in Target List			2,4-D	Abamectin	Captan (parent)	Chlorothalonil	Cyromazine	Dithiocarbamates	Ethephon	Fenbutatin Oxide	Fluazifop	Folpet (parent)	Glyphosate	Haloxyfop	Propamocarb	Analysed / correctly found among COMPULSORY compounds (max. 13 / 8)
within MACP [1]			Reg.	Reg.	Reg.	Reg.	Reg.	Reg.	Reg.	Reg.	Reg.	Reg.	Reg.	Reg.	Reg.	
present in Test Item			Yes	No	Yes	Yes	No	Yes	No	Yes	No	Yes	Yes	Yes	No	
evaluated in this PT			Yes	No	Yes	Yes	No	Yes	No	Yes	No	Yes	Yes	Yes	No	
Lab-Code SRM12-	NRL-SRM	Cat. [2]														
50		B	V	ND	V	V	ND	V		V	ND	V		V	ND	11 / 7
51		B	V	ND		V	ND			V	ND			V	ND	8 / 4
52		A	V	ND	V	V	ND	V	ND	V	ND	V	V	V	ND	13 / 8
53		A	V	ND	V	V	ND	V	ND	V	ND	V	V	V	ND	13 / 8
54		B				V	ND				ND	V		V	ND	6 / 3
55		B						V							ND	2 / 1
56		B		ND	V	V		V			ND	V			ND	7 / 4
57		A	V	ND	V	V	ND	V	ND	V	ND	V	V	V	ND	13 / 8
58		A	V	ND	V	V	ND	V	ND	V	ND	V	V	V	ND	13 / 8
59		B						V								1 / 1
60		B						V	ND				V			3 / 2
61		B	V		V						ND	V		V	ND	6 / 4
62	x	B	V		V	V		V			ND	V	V	**FN**	ND	9 / 6
63		A	V	ND	V	V	ND	V	ND	V	ND	V		V	ND	12 / 7
64		B	V			V	ND	V	ND		ND	V	V	V	ND	10 / 6
65		B						V								1 / 1
66	x	A	V	ND	V	V	ND	V	ND	V	ND	V	V	V	ND	13 / 8
67		A	V	ND	V	V	ND	V	ND	V	ND	V	V	V	ND	13 / 8
68		B	V	ND			ND	V			ND		V	V	ND	8 / 4
69		B	V	ND	V	V	ND	V			ND	V		V	ND	10 / 6
70		A	V	ND	V	V	ND	V	ND	V	ND	V	V	V	ND	13 / 8
71		B	V	ND		V	ND	V	ND	V	ND		V	V	ND	11 / 6
72		A	V	ND	V	V	ND	V	ND	V	ND	V	V	V	ND	13 / 8
73	x	B		ND	V	V	ND	V				V	V		ND	8 / 5
74		A	V	ND	V	V	ND	V	ND	V	ND	V	V	V	ND	13 / 8
75	x	A	V	ND	V	V	ND	V	ND	V	ND	V	V	V	ND	13 / 8

1) MACP = EU Multiannual Control Program; Reg.: MACP Regulation; WD: MACP Working Document ("Working document on pesticides to be considered for inclusion in the national control programmes to ensure compliance with maximum residue levels of pesticides residues in and on food of plant and animal origin")

2) Category A/B classification (Cat A was assigned to laboratories that have analysed at least 12 out of the 13 compulsory compounds on the Target Pesticides List, correctly detected 7 or more out of the 8 compulsory compounds present in the test item and have not reported any false positive result, see **Section 4.4.4, p. 59**)

V = analysed for and submitted concentration Value > "MRRL" for a pesticide present in the test item; **ND** = analysed for and correctly reported as "Not Detected"; **Empty cells**: not analysed; **FN** = analysed for but falsely not detected (False Negative result); **FN*** = analysed for a compound present in the test material and reported not detected due to lab's RL being > assigned value, therefore judged as FN; **FP** = false positive result (FP): Result reported as "≤ MRRL" and, therefore, not regarded as FP.

Table 4-2 (cont.): Scope and categorization of participating laboratories (including third country laboratories and laboratories that have not submitted results)

			Optional Compounds									Total	Additional Compounds				
Optional / Additional Compound listed in Target List			AMPA	Bifenazate	Bromide ion	Carbofuran	Chlorate	Dithianon	Phosphonic acid	N-Acetyl glyphosate	Analysed / correctly found among OPTIONAL compounds (max. 8 / 7)	Analysed / correctly found among COMPULSORY and OPTIONAL compounds (max. 21 / 15)	Captan (sum)	Folpet (sum)	THPI	Phthalimide	Analysed / correctly found among ADDITIONAL compounds within the EUPT-Target Pesticides List (max. 4 / 4)
within MACP [1]			WD	WD	Reg.	Reg.	WD	Reg.	Reg.	WD			Reg.	Reg.	Reg.	Reg.	
present in Test Item			No	Yes	Yes	Yes	Yes	Yes	Yes	Yes			Yes	Yes	Yes	Yes	
evaluated in this PT			No	Yes	Yes	No	Yes	Yes	Yes	Yes			Yes	Yes	Yes	Yes	
Lab-Code SRM12-	NRL-SRM	Cat. [2]															
50		B		V	V	V		V			4 / 4	15 / 11	V	V	V	V	4 / 4
51		B						V			1 / 1	9 / 5			V	V	2 / 2
52		A	ND	V	V	**FN***	V	V	V	V	8 / 6	21 / 14	V	V	V	V	4 / 4
53		A	ND	V	V	V	V	V	V		7 / 6	20 / 14	V	V	V	V	4 / 4
54		B									0 / 0	6 / 3					0 / 0
55		B									0 / 0	2 / 1					0 / 0
56		B									0 / 0	7 / 4					0 / 0
57		A				V	V	V	V		4 / 4	17 / 12	V	V	V	V	4 / 4
58		A	ND	V	V	V	V	V	V		7 / 6	20 / 14	V	V	V	V	4 / 4
59		B									0 / 0	1 / 1					0 / 0
60		B									0 / 0	3 / 2					0 / 0
61		B									0 / 0	6 / 4	V	V	V	V	4 / 4
62	x	B	ND			**FN***					2 / 0	11 / 6					0 / 0
63		A		V	V	V	V	V	V		6 / 6	18 / 13					0 / 0
64		B						V	V		2 / 2	12 / 8	V	V	V	V	4 / 4
65		B									0 / 0	1 / 1					0 / 0
66	x	A	ND		V	V	V				4 / 3	17 / 11	V	V	V	V	4 / 4
67		A	ND			V	V	V	V		5 / 4	18 / 12					0 / 0
68		B	ND								1 / 0	9 / 4					0 / 0
69		B			V	V					2 / 2	12 / 8					0 / 0
70		A	ND	V		V	V	V	V		6 / 5	19 / 13	V	V	V	V	4 / 4
71		B	ND	V	V	V	V	V			6 / 5	17 / 11	V	V	V	V	4 / 4
72		A	ND	V		V	V	V	V		6 / 5	19 / 13	V	V	V	V	4 / 4
73	x	B				**FN***	V				2 / 1	10 / 6	V	V	V	V	4 / 4
74		A	ND	V	V	V		V			5 / 4	18 / 12	V	V	V	V	4 / 4
75	x	A				**FN***		V			2 / 1	15 / 9					0 / 0

1) MACP = EU Multiannual Control Program; Reg.: MACP Regulation; WD: MACP Working Document ("Working document on pesticides to be considered for inclusion in the national control programmes to ensure compliance with maximum residue levels of pesticides residues in and on food of plant and animal origin")

2) Category A/B classification (Cat A was assigned to laboratories that have analysed at least 9 out of the 11 compulsory compounds on the Target Pesticides List, correctly detected 7 or more out of the 8 compulsory compounds present in the test item and have not reported any false positive result, see **Section 4.4.4, p. 59**)

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(FP): Result reported as "≤ MRRL" and, therefore, not regarded as FP.

Table 4-2 (cont.): Scope and categorization of participating laboratories (including third country laboratories and laboratories that have not submitted results)

Compulsory Compounds																
Compulsory Compound listed in Target List			2,4-D	Abamectin	Captan (parent)	Chlorothalonil	Cyromazine	Dithiocarbamates	Ethephon	Fenbutatin Oxide	Fluazifop	Folpet (parent)	Glyphosate	Haloxyfop	Propamocarb	Analysed / correctly found among COMPULSORY compounds (max. 13 / 8)
within MACP [1]			Reg.	Reg.	Reg.	Reg.	Reg.	Reg.	Reg.	Reg.	Reg.	Reg.	Reg.	Reg.	Reg.	
present in Test Item			Yes	No	Yes	Yes	No	Yes	No	Yes	No	Yes	Yes	Yes	No	
evaluated in this PT			Yes	No	Yes	Yes	No	Yes	No	Yes	No	Yes	Yes	Yes	No	
Lab-Code SRM12-	NRL-SRM	Cat. [2]														
76		A	V	ND	V	V	ND	V	ND	V	ND	V	V	V	ND	13 / 8
77		B		ND	V	V		V		V		V				6 / 5
78		A	V	ND	V	V	ND	V	ND	V	ND	V	V	V	ND	13 / 8
79		B	V	ND		V	ND	V	ND	V	ND		V	V	ND	11 / 6
80		B				V										1 / 1
81	x	B	V	ND		V	ND	V	ND	V	ND		V	V	ND	11 / 6
82		B						V								1 / 1
83	x	A	V	ND	V	V	ND		ND	V	ND	V	V	V	ND	12 / 7
84		A	V	ND	V	V	ND	V	ND	V	ND	V	V	V	ND	13 / 8
85	x	B				V	ND								ND	3 / 1
86		B	V	ND	V	V	ND	V			ND	V	V	V	ND	11 / 7
87		B						V								1 / 1
88		A	V	ND	V	V	ND	V	ND	V	ND	V	V	V	ND	13 / 8
89		B	V	ND		V	ND			V			V		ND	7 / 4
90		A	V	ND	V	V	ND	V	ND	V	ND	V	V	V	ND	13 / 8
91	x	B	V	ND	V	V	ND	V		V	ND	V		V	ND	11 / 7
92	x	A	V	ND	V	V	ND	V	ND	V	ND	V	V	V	ND	13 / 8
93		B	V	ND	V	V	ND			V	ND	V		V	ND	10 / 6
94		A	V	ND	V	V	ND	V	ND	V	ND	V	V	V	ND	13 / 8
95		A	V	ND	V	V	ND	V	ND	V	ND	V	V	V	ND	13 / 8
96	x	A	V	ND	V	V	ND	V	ND	V	ND	V	V	V	ND	13 / 8
97		A	V	ND	V	V	ND	V	ND	V	ND	V	V	V	ND	13 / 8
98	x	A	V	ND	V	V	ND	V	ND	V	ND	V	V	V	ND	13 / 8
99		A	V	ND	V	V	ND	V	ND	**FN**	ND	V	V	V	ND	13 / 7
100		B						V								1 / 1
101		B				V										1 / 1

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2) Category A/B classification (Cat A was assigned to laboratories that have analysed at least 12 out of the 13 compulsory compounds on the Target Pesticides List, correctly detected 7 or more out of the 8 compulsory compounds present in the test item and have not reported any false positive result, see **Section 4.4.4, p. 59**)

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Table 4-2 (cont.): Scope and categorization of participating laboratories (including third country laboratories and laboratories that have not submitted results)

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Optional / Additional Compound listed in Target List			AMPA	Bifenazate	Bromide ion	Carbofuran	Chlorate	Dithianon	Phosphonic acid	N-Acetyl glyphosate	Analysed / correctly found among OPTIONAL compounds (max. 8 / 7)	Analysed / correctly found among COMPULSORY and OPTIONAL compounds (max. 21 / 15)	Captan (sum)	Folpet (sum)	THPI	Phthalimide	Analysed / correctly found among ADDITIONAL compounds within the EUPT-Target Pesticides List (max. 4 / 4)
within MACP [1]			WD	WD	Reg.	Reg.	WD	Reg.	Reg.	WD			Reg.	Reg.	Reg.	Reg.	
present in Test Item			No	Yes	Yes	Yes	Yes	Yes	Yes	Yes			Yes	Yes	Yes	Yes	
evaluated in this PT			No	Yes	Yes	No	Yes	Yes	Yes	Yes			Yes	Yes	Yes	Yes	
Lab-Code SRM12-	NRL-SRM	Cat. [2]															
76		A	ND	V	V	V	V	V	V	V	8 / 7	21 / 15	V	V	V	V	4 / 4
77		B									0 / 0	6 / 5					0 / 0
78		A		V	V	V	V		V		5 / 5	18 / 13	V	V	V	V	4 / 4
79		B	ND		V	V	V	V	V		6 / 5	17 / 11	V	V			2 / 2
80		B									0 / 0	1 / 1					0 / 0
81	x	B			V	V	V	V	V	V	6 / 6	17 / 12	V	V	V	V	4 / 4
82		B									0 / 0	1 / 1					0 / 0
83	x	A									0 / 0	12 / 7					0 / 0
84		A	ND	V	V	V	V	V	V		7 / 6	20 / 14	V	V	V	V	4 / 4
85	x	B									0 / 0	3 / 1					0 / 0
86		B	ND	V			V		V		4 / 3	15 / 10	V	V	V	V	4 / 4
87		B									0 / 0	1 / 1					0 / 0
88		A					V				1 / 1	14 / 9	V	V	V	V	4 / 4
89		B		V				V			2 / 2	9 / 6					0 / 0
90		A	ND			V	V	V	V		5 / 4	18 / 12	V	V	V	V	4 / 4
91	x	B			V			V			2 / 2	13 / 9					0 / 0
92	x	A		V		V	V	V	V		5 / 5	18 / 13	V	V	V	V	4 / 4
93		B									0 / 0	10 / 6					0 / 0
94		A			V		V	V	V		4 / 4	17 / 12	V	V	V	V	4 / 4
95		A	ND	V	V	V	V	**FN**	V		7 / 5	20 / 13	V	V	V	V	4 / 4
96	x	A									0 / 0	13 / 8					0 / 0
97		A	ND	V	V	**FN***	V	V	V	V	8 / 6	21 / 14	V	V	V	V	4 / 4
98	x	A			V	**FN**	V	V	V		5 / 4	18 / 12	V	V	V	V	4 / 4
99		A	ND	V	V	V	V		V	V	7 / 6	20 / 13	V	V	V	V	4 / 4
100		B									0 / 0	1 / 1					0 / 0
101		B									0 / 0	1 / 1					0 / 0

1) MACP = EU Multiannual Control Program; Reg.: MACP Regulation; WD: MACP Working Document ("Working document on pesticides to be considered for inclusion in the national control programmes to ensure compliance with maximum residue levels of pesticides residues in and on food of plant and animal origin")

2) Category A/B classification (Cat A was assigned to laboratories that have analysed at least 9 out of the 11 compulsory compounds on the Target Pesticides List, correctly detected 7 or more out of the 8 compulsory compounds present in the test item and have not reported any false positive result, see **Section 4.4.4, p. 59**)

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Table 4-2 (cont.): Scope and categorization of participating laboratories (including third country laboratories and laboratories that have not submitted results)

Compulsory Compounds																
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within MACP [1]			Reg.	Reg.	Reg.	Reg.	Reg.	Reg.	Reg.	Reg.	Reg.	Reg.	Reg.	Reg.	Reg.	
present in Test Item			Yes	No	Yes	Yes	No	Yes	No	Yes	No	Yes	Yes	Yes	No	
evaluated in this PT			Yes	No	Yes	Yes	No	Yes	No	Yes	No	Yes	Yes	Yes	No	
Lab-Code SRM12-	NRL-SRM	Cat. [2]														
102		A	V	ND	V	V	ND	V	ND	V	ND	**FN**	V	V	ND	13 / 7
103	x	A	V	ND	V	V	ND	V	ND	V	ND	V	V	V	ND	13 / 8
104		A	V	ND	V	V	ND	V	ND	V	ND	V	V	V	ND	13 / 8
105	x	B			V	V	ND	V				V			ND	6 / 4
106		B	V	ND	**FN**	**FN**	ND	V	ND	**FN**	ND	**FN**	V	V	ND	13 / 4
107	x	A	V	ND	V	V	ND	V	ND	V	ND	V	V	V	ND	13 / 8
108		B	V	ND	V	V	ND	V			ND	V	V	V	ND	11 / 7
109		A	V	ND	V	V	ND	V	ND	V	ND	V	V	V	ND	13 / 8
110		B			V	V						V				3 / 3
111		B						V								1 / 1
112		B			V	V						V				3 / 3
113		A	V	ND	V	V	ND	V		V	ND	V	V	V	ND	12 / 8
114		B						V								1 / 1
115		A	V	ND	V	V	ND	V	ND	V	ND	V	V	V	ND	13 / 8
116		A	V	ND	V	V	ND	V	ND	V	ND	V	V	V	ND	13 / 8
117		B	V	ND	V	V	ND				ND	V		V	ND	9 / 5
118		B				V		V				V		V	**FP**	5 / 4
119		B						V								1 / 1
120		B		ND	**FN***	V						V			ND	5 / 2
121		B				V	ND								ND	3 / 1
122		B						V	ND				V			3 / 2
123		B								V					ND	2 / 1
124	x	B	V	ND	V	V		V			ND	V		V	ND	9 / 6
125		A	V	ND	V	V	ND		ND	V	ND	V	V	V	ND	12 / 7
126		B	V		V	V				V	ND	V	V	V	ND	9 / 7
127		A	V	ND	V	V	ND	V	ND	V	ND	V	V	V	ND	13 / 8

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Table 4-2 (cont.): Scope and categorization of participating laboratories (including third country laboratories and laboratories that have not submitted results)

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within MACP [1]			WD	WD	Reg.	Reg.	WD	Reg.	Reg.	WD			Reg.	Reg.	Reg.	Reg.	
present in Test Item			No	Yes	Yes	Yes	Yes	Yes	Yes	Yes			Yes	Yes	Yes	Yes	
evaluated in this PT			No	Yes	Yes	No	Yes	Yes	Yes	Yes			Yes	Yes	Yes	Yes	
Lab-Code SRM12-	NRL-SRM	Cat. [2]															
102		A	ND	V	V	V	V	V	V		7 / 6	20 / 13	V	V	V	V	4 / 4
103	x	A	ND	V	V	V	V	V	V	V	8 / 7	21 / 15	V	V	V	V	4 / 4
104		A	ND	V				V			3 / 2	16 / 10	V	V	V	V	4 / 4
105	x	B									0 / 0	6 / 4	V	V	V	V	4 / 4
106		B	ND	V		**FN***					3 / 1	16 / 5	V	V	V	V	4 / 4
107	x	A		V		**FN**		V			3 / 2	16 / 10					0 / 0
108		B									0 / 0	11 / 7					0 / 0
109		A	ND	V		V	V	V	V	V	7 / 6	20 / 14	V	V	V	V	4 / 4
110		B									0 / 0	3 / 3					0 / 0
111		B									0 / 0	1 / 1					0 / 0
112		B									0 / 0	3 / 3					0 / 0
113		A	ND	V	V	V		V			5 / 4	17 / 12	V	V	V	V	4 / 4
114		B									0 / 0	1 / 1					0 / 0
115		A	ND	V	V	V	V	V	V	V	8 / 7	21 / 15	V	V	V	V	4 / 4
116		A	ND		V		V	V	V		5 / 4	18 / 12	V	V	V	V	4 / 4
117		B			V						1 / 1	10 / 6					0 / 0
118		B		V	V	V					3 / 3	8 / 7					0 / 0
119		B									0 / 0	1 / 1					0 / 0
120		B				**FN***					1 / 0	6 / 2					0 / 0
121		B				V					1 / 1	4 / 2					0 / 0
122		B	ND								1 / 0	4 / 2					0 / 0
123		B				V					1 / 1	3 / 2					0 / 0
124	x	B				**FN***					1 / 0	10 / 6					0 / 0
125		A	ND	V	V	V	V	V	V	V	8 / 7	20 / 14	V	V	V	V	4 / 4
126		B		V		**FN***					2 / 1	11 / 8	V	V	V	V	4 / 4
127		A	ND	V		V	V	V	V		6 / 5	19 / 13	V	V	V	V	4 / 4

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Table 4-2 (cont.): Scope and categorization of participating laboratories (including third country laboratories and laboratories that have not submitted results)

Compulsory Compounds																
Compulsory Compound listed in Target List			**2,4-D**	**Abamectin**	**Captan (parent)**	**Chlorothalonil**	**Cyromazine**	**Dithiocarbamates**	**Ethephon**	**Fenbutatin Oxide**	**Fluazifop**	**Folpet (parent)**	**Glyphosate**	**Haloxyfop**	**Propamocarb**	**Analysed / correctly found among COMPULSORY compounds (max. 13 / 8)**
within MACP [1)]			**Reg.**	**Reg.**	**Reg.**	**Reg.**	**Reg.**	**Reg.**	**Reg.**	**Reg.**	**Reg.**	**Reg.**	**Reg.**	**Reg.**	**Reg.**	
present in Test Item			**Yes**	No	**Yes**	**Yes**	No	**Yes**	No	**Yes**	No	**Yes**	**Yes**	**Yes**	No	
evaluated in this PT			**Yes**	No	**Yes**	**Yes**	No	**Yes**	No	**Yes**	No	**Yes**	**Yes**	**Yes**	No	
Lab-Code SRM12-	**NRL-SRM**	**Cat. [2)]**														
128		B	V													1 / 1
129		A	V	ND	V	V	ND	V	ND	V	ND	V		V	ND	12 / 7
130		A	V	ND	V	V	ND	V	ND	V	ND	V	V	V	ND	13 / 8
3rd-131		B	V	*(FP)*		V	ND	V					V		ND	7 / 4
3rd-132		B	V	**FP**	V	V	ND	V	ND			V	V	V	ND	11 / 7
3rd-133		A	V	ND	V	V	ND		ND	V	ND	V	V	V	ND	12 / 7
3rd-134		B				V		V		V						3 / 3
3rd-135		B				V	**FP**	V							ND	4 / 2
3rd-136		A	V	ND	V	V	ND	V	ND	V	ND	V	V	V	ND	13 / 8
3rd-137		B	V			V							V		*(FP)*	4 / 3
3rd-138		B			V	V		V	ND	V		V				6 / 5
3rd-139		A	V	ND	V	V	ND	V	ND	V	ND	V	V	V	ND	13 / 8

1) MACP = EU Multiannual Control Program; Reg.: MACP Regulation; WD: MACP Working Document ("Working document on pesticides to be considered for inclusion in the national control programmes to ensure compliance with maximum residue levels of pesticides residues in and on food of plant and animal origin")

2) Category A/B classification (Cat A was assigned to laboratories that have analysed at least 12 out of the 13 compulsory compounds on the Target Pesticides List, correctly detected 7 or more out of the 8 compulsory compounds present in the test item and have not reported any false positive result, see **Section 4.4.4, p. 59**)

V = analysed for and submitted concentration Value > "MRRL" for a pesticide present in the test item; **ND** = analysed for and correctly reported as "Not Detected"; **Empty cells**: not analysed; **FN** = analysed for but falsely not detected (False Negative result); **FN*** = analysed for a compound present in the test material and reported not detected due to lab's RL being > assigned value, therefore judged as FN; **FP** = false positive result
(FP): Result reported as "≤ MRRL" and, therefore, not regarded as FP.

4.2 Analysis of Blank Material

As described in **Section 1.2 (p. 1)** the organiser has detected ***phosphonic acid*** in the blank material at the level of 0.72 mg/kg. 27 out of the 50 laboratories analysing for ***phosphonic acid*** reported the detection of this compound in the blank material with 24 of the laboratories reporting a numerical value (**Table 4-3**), two of them reporting < MRRL (0.05 mg/kg) and one of them reporting > 1 mg/kg. The assigned value of ***phosphonic acid*** (19.3 mg/kg) was, however, at least 17 times higher than the highest finding (1.12 mg/kg) in the blank material. The organisers thus concluded that the use of the blank material for calibration had only a negligible influence on the results of the laboratories.

Table 4-2 (cont.): Scope and categorization of participating laboratories (including third country laboratories and laboratories that have not submitted results)

			Optional Compounds									Total	Additional Compounds				
Optional / Additional Compound listed in Target List			AMPA	Bifenazate	Bromide ion	Carbofuran	Chlorate	Dithianon	Phosphonic acid	N-Acetyl glyphosate	Analysed / correctly found among OPTIONAL compounds (max. 8 / 7)	Analysed / correctly found among COMPULSORY and OPTIONAL compounds (max. 21 / 15)	Captan (sum)	Folpet (sum)	THPI	Phthalimide	Analysed / correctly found among ADDITIONAL compounds within the EUPT-Target Pesticides List (max. 4 / 4)
within MACP [1]			WD	WD	Reg.	Reg.	WD	Reg.	Reg.	WD			Reg.	Reg.	Reg.	Reg.	
present in Test Item			No	Yes	Yes	Yes	Yes	Yes	Yes	Yes			Yes	Yes	Yes	Yes	
evaluated in this PT			No	Yes	Yes	No	Yes	Yes	Yes	Yes			Yes	Yes	Yes	Yes	
Lab-Code SRM12-	NRL-SRM	Cat. [2]															
128		B									0 / 0	1 / 1					0 / 0
129		A		V	V		V	V	V		5 / 5	17 / 12	V	V	V	V	4 / 4
130		A	ND	V		V	V	V	V		6 / 5	19 / 13	V	V	V	V	4 / 4
3rd-131		B	ND					V			2 / 1	9 / 5					0 / 0
3rd-132		B			V	**FN***					2 / 1	13 / 8			V	V	2 / 2
3rd-133		A	ND		V						2 / 1	14 / 8					0 / 0
3rd-134		B		V		V					2 / 2	7 / 5					0 / 0
3rd-135		B				**FN***					1 / 0	5 / 2					0 / 0
3rd-136		A									0 / 0	13 / 8					0 / 0
3rd-137		B									0 / 0	4 / 3					0 / 0
3rd-138		B		V	V				V		3 / 3	9 / 8					0 / 0
3rd-139		A									0 / 0	13 / 8					0 / 0

1) MACP = EU Multiannual Control Program; Reg.: MACP Regulation; WD: MACP Working Document ("Working document on pesticides to be considered for inclusion in the national control programmes to ensure compliance with maximum residue levels of pesticides residues in and on food of plant and animal origin")

2) Category A/B classification (Cat A was assigned to laboratories that have analysed at least 9 out of the 11 compulsory compounds on the Target Pesticides List, correctly detected 7 or more out of the 8 compulsory compounds present in the test item and have not reported any false positive result, see **Section 4.4.4, p. 59**)

V = analysed for and submitted concentration Value > "MRRL" for a pesticide present in the test item; **ND** = analysed for and correctly reported as "Not Detected"; **Empty cells**: not analysed; **FN** = analysed for but falsely not detected (False Negative result); **FN*** = analysed for a compound present in the test material and reported not detected due to lab's RL > assigned value, therefore judged as FN; **FP** = false positive result (FP): Result reported as "≤ MRRL" and, therefore, not regarded as FP.

Among the other target analytes there were further three cases where participants reported detections in the blank material at levels at or above the MRRL (**Table 4-3, p. 36)**. These were two cases of ***dithiocarbamates*** (MRRL = 0.03 mg/kg) at 0.134 mg/kg (SRM12-44) and 0.04 mg/kg (SRM12-40), and two cases of ***glyphosate*** (MRRL = 0.03 mg/kg) at 0.1 mg/kg (SRM12-40) and 0.03 mg/kg (SRM12-83). Since the organisers and all other laboratories having analysed for these compounds did not detect them in the blank material, these findings were regarded as analytical errors. The affected laboratories are encouraged to find the reasons behind these errors. Interestingly, none of these three laboratories reporting "false positive" results in the blank reported any false positive in the test item.

Table 4-3: Concentration of analytes in the blank material reported by the participating laboratories

Compound	MRRL [mg/kg]	Conc. in Blank Material [mg/kg]	Reported by
2,4-D	0.01	<0.01	SRM12-9
		<0.01	SRM12-75
		<0.01	SRM12-3rd-131
Captan (parent)	0.01	<0.01	SRM12-9
		<0.01	SRM12-75
Chlorothalonil	0.01	<0.01	SRM12-9
		<0.01	SRM12-75
		<0.01	SRM12-121
		<0.01	SRM12-3rd-131
Dithiocarbamates	0.03	0.0005	SRM12-2
		0.024	SRM12-48
		0.04	SRM12-40
		0.134	SRM12-44
		<0.01	SRM12-9
		<0.01	SRM12-3rd-131
		<0.03	SRM12-75
		<0.05	SRM12-114
		<0.050	SRM12-55
Fenbutatin Oxide	0.01	<0.01	SRM12-9
		<0.01	SRM12-40
		<0.01	SRM12-75
Folpet (parent)	0.01	<0.01	SRM12-9
		<0.01	SRM12-75
Glyphosate	0.03	0.006	SRM12-18
		0.0072	SRM12-72
		0.009	SRM12-14
		0.03	SRM12-83
		0.1	SRM12-40
		<0.01	SRM12-125
		<0.01	SRM12-12
		<0.01	SRM12-3rd-131
		<0.03	SRM12-75
		<0.05	SRM12-9
Haloxyfop	0.01	<0.01	SRM12-9
		<0.01	SRM12-75
Captan (sum)	–	<0.01	SRM12-9
THPI	–	<0.01	SRM12-9
Phthalimide	–	0.017	SRM12-51
		<0.01	SRM12-71

Compound	MRRL [mg/kg]	Conc. in Blank Material [mg/kg]	Reported by
Bromide ion	3	0.05	SRM12-125
		0.115	SRM12-53
		0.16	SRM12-48
		<10.0	SRM12-71
Carbofuran	0.001	0.0005	SRM12-8
		<0.001	SRM12-9
		<0.001	SRM12-40
		<0.001	SRM12-121
Chlorate	0.02	0.0019	SRM12-45
		0.003	SRM12-14
		0.003	SRM12-18
		0.003	SRM12-53
		0.005	SRM12-76
		0.007	SRM12-90
		<0.01	SRM12-9
		<0.01	SRM12-125
Dithianon	0.02	<0.01	SRM12-40
		<0.010	SRM12-3rd-131
		<0.02	SRM12-75
Phosphonic acid	0.05	0.025	SRM12-125
		0.029	SRM12-67
		0.071	SRM12-76
		0.117	SRM12-129
		0.383	SRM12-14
		0.438	SRM12-130
		0.461	SRM12-1
		0.479	SRM12-90
		0.5	SRM12-78
		0.5	SRM12-84
		0.531	SRM12-92
		0.58	SRM12-12
		0.617	SRM12-53
		0.62	SRM12-10
		0.63	SRM12-109
		0.665	SRM12-72
		0.67	SRM12-9
		0.67	SRM12-127
		0.674	SRM12-18
		0.679	SRM12-79
		0.686	SRM12-33
		0.734	SRM12-103
		0.777	SRM12-94
		0.81	SRM12-21
		1.12	SRM12-34
		>1	SRM12-98
		Not quantified	SRM12-42

4.3 Assigned Values and Target Standard Deviations

The assigned value (x_{pt}) of each analyte present in the test item was established as the mean of robust statistics (x^*) of all numerical results submitted by laboratories from EU and EFTA countries calculated using Algorithm A [6, **Appendix 8**]. Results from third country laboratories were not taken into account. Based on these assigned values, z-scores were calculated for all submitted results using the FFP-approach (**Section 4.4.3, p. 41**), and a preliminary report was released on 11 May, 2017. The uncertainties ($u(x_{pt})$) of the assigned values were calculated as described under **Section 2.2, p. 15**.

In the case of ***carbofuran (part of sum)*** the very wide distribution of participants' results (CV^* 47.1 %) resulted in the robust mean being assosiated with a statistical uncertainty exceeding the tolerance (**Table 4-5, p. 38**). The Scientific Committee therefore decided to evaluate the robust mean and z-scores for ***carbofuran (part of sum)*** using different scenarios and for informative purposes only.

The CV^*-value of ***phosphonic acid*** (27.0 %)**,** ***captan (parent)*** (28.1 %) and ***THPI*** (30.5 %) was higher than the FFP-RSD of 25 %, but all of them met the criterion for the statistical certainty. The CV^*-values of all other compulsory analytes were lower or just slightly higher than 25 %. The average CV^*s of compulsory analytes based on the entire population of EU-and EFTA-laboratories was 21.2 %, and the average CV^*s of optional analytes based on the entire population excluding ***carbofuran (part of sum)*** was 21.6 %. Both were clearly lower than the FFP-RSD of 25 %.

Originally ***captan (parent)***, ***folpet (parent)***, ***phthalimide*** and ***THPI*** were considered only for data collection. Due to high quality of the results submitted by the participants these four addtional analytes were finally evaluated, incl. assigned values, CV^* and z-scores as well as FNs and FPs, but for infromative purpose only.

4.4 Assessment of Laboratory Performance

4.4.1 False Positives

Among EU- and EFTA-laboratories only one laboratory reported one numerical result for an analyte (*propamocarb*) on the Target Pesticides List but not present in the test material. Two other false positive results (*abamectin* and *cyromazine*) were reported by two laboratories from third countries. *Propamocarb*, *abamectin* and *cyromazine* were neither detected by the organisers nor by the overwhelming majority of the participants (**Table 4-4**). These three results exceeded the laboratories' reporting limits for these compounds, were higher than the respective MRRLs in the Target Pesticides List, and were, therefore, judged as false positives.

One laboratory (SRM-3rd-131) reported in one case a numerical result for *abamectin* (0.0045 mg/kg) that was lower than the MRRL. Another laboratory reported "< MRRL" for *propamocarb*. Following the rules in the General Protocol these two results were not judged as false positives.

Table 4-4: Overview of false positive and potentially false positive results reported by participating laboratories

Compound	PT-Code	Analysed	Reported Result [mg/kg]	RL [mg/kg]	MRRL [mg/kg]	Judgement
Abamectin	SRM12-3rd-132	Yes	0.023	0.01	0.01	FP
	SRM12-3rd-131	Yes	0.0045	0.01	0.01	–
Cyromazine	SRM12-3rd-135	Yes	0.03	0.01	0.01	FP
Propamocarb	SRM12-118	Yes	0.034	0.01	0.01	FP
	SRM12-3rd-137	Yes	< RL (= MRRL)	0.01	0.01	–

Table 4-5: Assigned values, uncertainties of assigned values and CV^* values calculated for all compounds present in the test item

Assigned Value and CV^* Based on the Entire Population of Results from EU and EFTA Laboratories								
	Compound	No. of FNs	No. of numerical results (EU+EFTA)	Assigned Value [mg/kg]	$u(x_{pt})$ [1)] [mg/kg]	$u(x_{pt})$ Tolerance [mg/kg]	Judgement for UAV-test	CV^* [2)] [%]
Compulsory Compounds	2,4-D	1	97	0.079	+/- 0.00133	0.0059	passed	13.3
	Captan (parent)	1 [5)]+1	91	0.085	+/- 0.00313	0.0064	passed	28.1
	Chlorothalonil	2	109	0.125	+/- 0.00378	0.0094	passed	25.2
	Dithiocarbamates	0	107	0.267	+/- 0.00717	0.0200	passed	22.2
	Fenbutatin Oxide	4	78	0.086	+/- 0.00255	0.0064	passed	21.0
	Folpet (parent)	2	96	0.334	+/- 0.01066	0.0251	passed	25.0
	Glyphosate	1	85	0.306	+/- 0.00867	0.0230	passed	20.9
	Haloxyfop	2	95	0.070	+/- 0.00125	0.0053	passed	13.9
	Average [3)] CV^*							**21.2**
Optional Compounds	Bifenazate (sum)	0	54	0.270	+/- 0.01013	0.0202	passed	22.1
	Bromide ion	0	52	19.1	+/- 0.53064	1.4337	passed	16.0
	Carbofuran (part of sum) [4)]	13 [5)]+3	58	0.0030	+/- 0.00023	0.0023	**failed**	47.1
	Chlorate	0	60	0.490	+/- 0.01283	0.0367	passed	16.2
	Dithianon	1	63	0.294	+/- 0.01170	0.0220	passed	25.3
	Phosphonic acid	0	50	19.2	+/- 0.9180	1.4443	passed	27.0
	N-Acetyl-glyphosate	1	15	0.100	+/- 0.00751	0.00753	passed	23.2
	Average [3)] CV^*							**21.6**
Additional Compounds	Captan (sum)	0	65	0.302	+/- 0.0181	0.0226	passed	25.2
	Folpet (sum)	0	66	1.195	+/- 0.03871	0.0896	passed	21.1
	THPI	0	67	0.110	+/- 0.00515	0.0083	passed	30.5
	Phthalimide	1	66	0.446	+/- 0.01485	0.0334	passed	21.6
	Average [3)] CV^*							**24.6**

1: $u(x_{pt})$**:** Uncertainty of assigned value calculated as shown under **Section 2.2 (p. 15)**
2: CV^***:** Relative standard deviation based on robust statistics
3: The average CV^* is given for information purposes only. CV^*s of individual compounds or average CV^*s of individual compounds or related compounds over many PTs are more meaningfull and conclusive.
4: Excluded from the calculation of the average CV^*s and the assigned values were calculated for informative purpose only.
5: Laboratories had a reporting limit higher than the assigned value and reported "not detected". Following the General Protocol, these results were judged as "false positive".

4.4.2 False Negatives

Among the compulsory compounds there were 14 cases (4× ***fenbutatin oxide***, 2× ***captan (parent)***, 2× ***folpet (parent),*** 2× ***chlorothalonil,*** 2× ***haloxyfop,*** 1× ***glyphosate*** and 1× ***2,4-D***) where the participants reported "analysed, but not detected" for target compounds which were spiked to the test item and detected by the majority of the laboratories targeting them (**Table 4-6**). All these results were reported by laboratories from EU and EFTA countries. As the assigned values for these seven analytes were sufficiently distant from the MRRLs, these results were judged as false negatives. In one case of ***captan (parent)*** the "false negative" judgement resulted from the fact that the laboratory had a higher reporting limit than the assigned value, as this is the rule stated in the General Protocol. These 14 false negative results represented 1.8 % of the total 772 results reported by the EU/EFTA laboratories for compulsory target compounds present in the test item and 1.7 % of the total 819 results from all participating laboratories.

Table 4-6: Overview of false negative results reported by participating laboratories (including 3rd country laboratories)

	Compound	PT-Code	Analysed	Detected	RL [mg/kg]	MRRL [mg/kg]	Assigned Value [mg/kg]	Judgement
Compulsory Compounds	2,4-D	SRM12-44	Yes	No	0.05	0.01	0.079	False Negative
	Captan (parent)	SRM12-106	Yes	No	0.01	0.01	0.085	False Negative
		SRM12-120	Yes	No	0.2			False Negative
	Chlorothalonil	SRM12-27	Yes	No	0.01	0.01	0.125	False Negative
		SRM12-106	Yes	No	0.01			False Negative
	Fenbutatin Oxide	SRM12-2	Yes	No	0.01	0.01	0.086	False Negative
		SRM12-35	Yes	No	0.01			False Negative
		SRM12-99	Yes	No	0.01			False Negative
		SRM12-106	Yes	No	0.01			False Negative
	Folpet (parent)	SRM12-102	Yes	No	0.01	0.01	0.334	False Negative
		SRM12-106	Yes	No	0.01			False Negative
	Glyphosate	SRM12-6	Yes	No	0.1	0.03	0.306	False Negative
	Haloxyfop	SRM12-44	Yes	No	0.05	0.01	0.070	False Negative
		SRM12-62	Yes	No	0.05			False Negative
Optional Compounds	Carbofuran	SRM12-62	Yes	No	0.05	0.001	0.0030	False Negative
		SRM12-6	Yes	No	0.01			False Negative
		SRM12-12	Yes	No	0.01			False Negative
		SRM12-27	Yes	No	0.01			False Negative
		SRM12-39	Yes	No	0.01			False Negative
		SRM12-52	Yes	No	0.01			False Negative
		SRM12-73	Yes	No	0.01			False Negative
		SRM12-97	Yes	No	0.01			False Negative
		SRM12-106	Yes	No	0.01			False Negative
		SRM12-120	Yes	No	0.01			False Negative
		SRM12-124	Yes	No	0.01			False Negative
		SRM12-126	Yes	No	0.01			False Negative
		SRM12-3rd-132	Yes	No	0.01			False Negative
		SRM12-3rd-135	Yes	No	0.01			False Negative
		SRM12-75	Yes	No	0.005			False Negative
		SRM12-2	Yes	No	0.001			False Negative
		SRM12-98	Yes	No	0.001			False Negative
		SRM12-107	Yes	No	0.001			False Negative
	Dithianon	SRM12-95	Yes	No	0.01		0.294	False Negative
	N-Acetyl glyphosate	SRM12-6	Yes	No	0.1		0.100	False Negative
	Phthalimide	SRM12-47	Yes	No	0.01	–	0.446	False Negative

Among the optional compounds there were 20 cases (18× ***carbofuran***, 1× ***dithianon***, 3× ***N-acetyl glyphosate***) where the participants reported "analysed, but not detected" for target compounds that were spiked to the test item and detected by the majority of the laboratories targeting them (**Table 4-6, p. 39**). All of these false negative results but two, concerning ***carbofuran (part of sum)***, were reported by participants from EU and EFTA countries. In 15 among the 18 cases of false negatives concerning ***carbofuran (part of sum)*** the reporting limits were higher than the assigned value. According to the rule in the General Protocol, these results were still judged as fase negatives.

The 18 false negative results reported by EU/EFTA laboratories accounted for 4.9 % of the total 370 results reported by the EU/EFTA laboratories for optional target compounds. The 20 false negative results reported in total represented 5.3 % of the results reported by all participating labs for optional compounds.

Among the additional compounds there was only one false negative result, concerning ***phthalimide***, which was reported by an EU/EFTA-laboratory. That accounted for 0.8 % of the total 265 results reported by the EU/EFTA laboratories for additional target compounds present in the test item.

Table 4-7: Overall performance based on z-score classification

		EU and EFTA laboratories				
	Compound	No. of results [1]	Acceptable No. (%)	Questionable No. (%)	Unacceptable [1] No. (%)	FNs No.
Compulsory Compounds	2,4-D	98	93 (95 %)	2 (2 %)	3 (3 %)	1
	Captan (parent)	93	77 (83 %)	6 (6 %)	10 (11 %)	2
	Chlorothalonil	111	97 (87 %)	5 (5 %)	9 (8 %)	2
	Dithiocarbamates	107	101 (94 %)	2 (2 %)	4 (4 %)	
	Fenbutatin Oxide	82	72 (88 %)	3 (4 %)	7 (9 %)	4
	Folpet (parent)	98	82 (84 %)	6 (6 %)	10 (10 %)	2
	Glyphosate	86	77 (90 %)	3 (3 %)	6 (7 %)	1
	Haloxyfop	97	89 (92 %)	3 (3 %)	5 (5 %)	2
	Subtotal (average)	**772**	**688 (89 %)**	**30 (4 %)**	**54 (7 %)**	**14**
Optional Compounds [2]	Bifenazate	54	52 (96 %)	1 (2 %)	1 (2 %)	
	Bromide ion	52	47 (90 %)	4 (8 %)	1 (2 %)	
	Chlorate	60	54 (90 %)	3 (5 %)	3 (5 %)	
	Dithianon	64	54 (84 %)	2 (3 %)	8 (13 %)	1
	Phosphonic acid	50	43 (86 %)	4 (8 %)	3 (6 %)	
	N-Acetyl glyphosate	16	15 (94 %)	(0 %)	1 (6 %)	1
	Subtotal (average)	**296**	**265 (90 %)**	**14 (5 %)**	**17 (6 %)**	**2**
Additional Compounds	Captan (sum)	65	61 (94 %)	3 (5 %)	1 (2 %)	
	Folpet (sum)	66	58 (88 %)	4 (6 %)	4 (6 %)	
	THPI	67	58 (87 %)	4 (6 %)	5 (7 %)	
	Phthalimide	67	61 (91 %)	(0 %)	6 (9 %)	1
	Subtotal (average)	**265**	**238 (90 %)**	**11 (4 %)**	**16 (6 %)**	**1**
Overall EU/EFTA (Average)		**1333**	**1191 (89 %)**	**55 (4 %)**	**87 (7 %)**	**17**

1) including false negatives (FNs)
2) excluding carbofuran

4.4.3 Laboratory Performance Based on z-Scores

All individual z-scores were calculated using the FFP-RSD of 25 % and the assigned values derived from the entire population of results received from EU/EFTA laboratories. **Table 4-7** shows the overall classification of z-scores achieved by all laboratories for compulsory, optional and additional compounds. The respective rules are shown in **Section 2.4 (p. 16)**. Among the laboratories from EU and EFTA countries "Acceptable" z-scores were achieved by 83 – 95 % (89 % on average) of the labs in the case of compulsory compounds, by 84 – 96 % (90 % on average) in the case of optional compounds excluding ***carbofuran (part of sum)*** and by 87 – 94 % (90 % on average) for additional compounds. Overall and excluding carbofuran, 89 % of the results submitted by EU- and EFTA-countries were acceptable, 4 % questionable and 7 % unacceptable (including false negatives). The respective overall figures of 3rd country labs were 71 %, 7 % and 21 %. Deviations of the sum from 100 % are due to rounding errors. False positive results were not counted.

A compilation of all individual results and z-scores for each laboratory is shown in **Table 4-8 (p. 42), Table 4-9 (p. 48)** and **Table 4-10 (p. 56)** for compulsory, optional and additional compounds, respectively. The corresponding kernel density histograms showing the distribution of the reported results are shown in **Appendix 5**. A graphic representation of the z-score distribution of each target analyte present in the test item can be seen in **Appendix 6**.

Table 4-7 (cont.): Overall performance based on z-score classification

	3rd country laboratories					
	Compound	No. of results [1]	Acceptable No. (%)	Questionable No. (%)	Unacceptable [1] No. (%)	FNs No.
Compulsory Compounds	2,4-D	6	5 (83 %)	–	1 (17 %)	
	Captan (parent)	5	4 (80 %)	–	1 (20 %)	
	Chlorothalonil	9	7 (78 %)	1 (11 %)	1 (11 %)	
	Dithiocarbamates	7	5 (71 %)	–	2 (29 %)	
	Fenbutatin Oxide	5	4 (80 %)	–	1 (20 %)	
	Folpet (parent)	5	2 (40 %)	1 (20 %)	2 (40 %)	
	Glyphosate	6	5 (83 %)	1 (17 %)	–	
	Haloxyfop	4	2 (50 %)	1 (25 %)	1 (25 %)	
	Subtotal (average)	**47**	**34 (72 %)**	**4 (9 %)**	**9 (19 %)**	
Optional Compounds [2]	Bifenazate	2	1 (50 %)		1 (50 %)	
	Bromide ion	3	3 (100 %)			
	Chlorate					
	Dithianon	1	1 (100 %)			
	Phosphonic acid	1	1 (100 %)			
	N-Acetyl glyphosate					
	Subtotal (average)	**7**	**6 (86 %)**	**0 (0 %)**	**1 (14 %)**	
Additional Compounds	Captan (sum)					
	Folpet (sum)					
	THPI	1			1 (100 %)	
	Phthalimide	1			1 (100 %)	
	Subtotal (average)	**2**			**2 (100 %)**	
Overall 3rd country (Average)		**56**	**40 (71 %)**	**4 (7 %)**	**12 (21 %)**	**0**

1) including false negatives (FNs)
2) excluding carbofuran

Table 4-8: Results reported and z-scores achieved by all participating laboratories for COMPULSORY compounds

COMPULSORY Compound				2,4-D		Captan (parent)		Chlorothalonil		Dithiocarbamates	
MRRL [mg/kg]				0.01		0.01		0.01		0.03	
Assigned Value [mg/kg]				0.079		0.085		0.125		0.267	
CV^*				13.3 %		28.1 %		25.2 %		22.2 %	
Lab code SRM12-	NRL-SRM	Analysed / corr. found, max. 13 / 8	Cat.*	Conc. [mg/kg]	z-Score§ (FFP-RSD = 25 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)
1		13 / 8	A	0.068	-0.5	0.055	-1.4	0.086	-1.3	0.330	0.9
2		13 / 7	A	0.0692	-0.5	0.0881	0.1	0.120	-0.2	0.234	-0.5
3	x	13 / 8	A	0.082	0.2	0.066	-0.9	0.129	0.1	0.196	-1.1
4		11 / 7	B	0.0742	-0.2	0.0723	-0.6	0.0956	-0.9	0.330	0.9
5		1 / 1	B							0.244	-0.3
6	x	13 / 7	A	0.080	0.1	0.115	1.4	0.122	-0.1	0.239	-0.4
7		13 / 8	A	0.073	-0.3	0.079	-0.3	0.137	0.4	0.279	0.2
8		9 / 4	B	0.089	0.5					0.225	-0.6
9		13 / 8	A	0.0581	-1.0	0.011	-3.5	0.315	6.1	0.341	1.1
10		9 / 5	B	0.067	-0.6			0.290	5.3	0.382	1.7
11		6 / 3	B	0.043	-1.8			0.494	11.8		
12		13 / 8	A	0.075	-0.2	0.077	-0.4	0.151	0.8	0.193	-1.1
13	x	13 / 8	A	0.064	-0.7	0.131	2.2	0.089	-1.2	0.286	0.3
14		13 / 8	A	0.063	-0.8	0.080	-0.2	0.115	-0.3	0.228	-0.6
15		10 / 6	B	0.075	-0.2	0.081	-0.2	0.146	0.7	0.280	0.2
16	x	11 / 6	B	0.056	-1.2			0.147	0.7	0.210	-0.9
17	x	13 / 8	A	0.0807	0.1	0.0872	0.1	0.118	-0.2	0.303	0.5
18		13 / 8	A	0.074	-0.2	0.098	0.6	0.164	1.2	0.270	0.0
19	x	13 / 8	A	0.088	0.5	0.090	0.2	0.099	-0.8	0.246	-0.3
20		13 / 8	A	0.0746	-0.2	0.0858	0.0	0.148	0.7	0.300	0.5
21		13 / 8	A	0.080	0.1	0.084	0.0	0.141	0.5	0.211	-0.8
22		13 / 8	A	0.090	0.6	0.09	0.2	0.134	0.3	0.337	1.0
23		10 / 5	B			0.025	-2.8	0.030	-3.0		
24		11 / 7	B	0.083	0.2	0.062	-1.1	0.119	-0.2	0.313	0.7
25		13 / 8	A	0.080	0.1	0.086	0.0	0.123	-0.1	0.216	-0.8
26		13 / 8	A	0.079	0.0	0.075	-0.5	0.130	0.2	0.180	-1.3
27		6 / 2	B	0.0772	-0.1			FN	-3.7		
28	x	10 / 6	B	0.071	-0.4			0.107	-0.6	0.305	0.6
29	x	10 / 6	B	0.0785	0.0	0.062	-1.1	0.106	-0.6		
30	x	13 / 8	A	0.076	-0.1	0.103	0.8	0.121	-0.1	0.167	-1.5
31		4 / 4	B			0.0904	0.3	0.0987	-0.8	0.289	0.3
32		11 / 7	B	0.081	0.1	0.094	0.4	0.101	-0.8	0.296	0.4
33	x	13 / 8	A	0.080	0.1	0.082	-0.1	0.132	0.2	0.348	1.2
34		11 / 6	B	0.077	-0.1			0.125	0.0	0.318	0.8
35	x	13 / 7	A	0.0846	0.3	0.104	0.9	0.143	0.6	0.819	8.3
37		6 / 4	B			0.083	-0.1	0.120	-0.2	0.260	-0.1
38		8 / 6	B	0.072	-0.3	0.063	-1.0	0.110	-0.5	0.200	-1.0
39		13 / 8	A	0.086	0.4	0.090	0.2	0.142	0.5	0.310	0.6
40		9 / 5	B					0.161	1.1	0.250	-0.3
41		13 / 8	A	0.081	0.1	0.050	-1.6	0.105	-0.6	0.140	-1.9
42		13 / 8	A	0.077	-0.1	0.080	-0.2	0.105	-0.6	0.143	-1.9
43		2 / 2	B							0.203	-1.0

* Category A/B classification (Cat A was assigned to laboratories that have correctly analysed at least 12 of 13 compulsory compounds on the Target Pesticides List, corretly detected 7 or more out of the 8 compulsory compounds and that have not reported any false positive results)

Table 4-8 (cont.): Results reported and z-scores achieved by all participating laboratories for COMPULSORY compounds

	COMPULSORY Compound			Fenbutatin Oxide		Folpet (parent)		Glyphosate		Haloxyfop	
	MRRL [mg/kg]			0.01		0.01		0.03		0.01	
	Assigned Value [mg/kg]			0.086		0.334		0.306		0.070	
	*CV**			21.0 %		25.0 %		20.9 %		13.9 %	
Lab code SRM12-	NRL-SRM	Analysed / corr. found, max. 13 / 8	Cat.*	Conc. [mg/kg]	z-Score§ (FFP-RSD = 25 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)
1		13 / 8	A	0.076	-0.4	0.190	-1.7	0.629	4.2	0.056	-0.8
2		13 / 7	A	FN	-3.5	0.349	0.2	0.317	0.1	0.058	-0.7
3	x	13 / 8	A	0.111	1.2	0.292	-0.5	0.320	0.2	0.070	0.0
4		11 / 7	B	0.0576	-1.3	0.295	-0.5			0.0641	-0.3
5		1 / 1	B								
6	x	13 / 7	A	0.089	0.2	0.317	-0.2	FN	-3.6	0.080	0.6
7		13 / 8	A	0.083	-0.1	0.286	-0.6	0.355	0.6	0.070	0.0
8		9 / 4	B					0.394	1.1	0.082	0.7
9		13 / 8	A	0.0624	-1.1	0.172	-1.9	0.779	6.2	0.0688	-0.1
10		9 / 5	B					0.312	0.1	0.066	-0.2
11		6 / 3	B							0.041	-1.7
12		13 / 8	A	0.11	1.1	0.420	1.0	0.3	-0.1	0.070	0.0
13	x	13 / 8	A	0.081	-0.2	0.292	-0.5	0.290	-0.2	0.064	-0.4
14		13 / 8	A	0.080	-0.3	0.355	0.2	0.311	0.1	0.069	-0.1
15		10 / 6	B			0.290	-0.5			0.060	-0.6
16	x	11 / 6	B	0.091	0.3			0.240	-0.9	0.064	-0.4
17	x	13 / 8	A	0.089	0.2	0.298	-0.4	0.331	0.3	0.0763	0.3
18		13 / 8	A	0.104	0.9	0.382	0.6	0.299	-0.1	0.064	-0.4
19	x	13 / 8	A	0.080	-0.3	0.413	0.9	0.291	-0.2	0.068	-0.1
20		13 / 8	A	0.0793	-0.3	0.401	0.8	0.299	-0.1	0.0706	0.0
21		13 / 8	A	0.087	0.1	0.349	0.2	0.279	-0.4	0.073	0.2
22		13 / 8	A	0.091	0.3	0.386	0.6	0.360	0.7	0.054	-0.9
23		10 / 5	B			0.040	-3.5	0.180	-1.6	0.040	-1.7
24		11 / 7	B	0.068	-0.8	0.333	0.0			0.065	-0.3
25		13 / 8	A	0.077	-0.4	0.449	1.4	0.370	0.8	0.072	0.1
26		13 / 8	A	0.092	0.3	0.950	7.4	0.250	-0.7	0.072	0.1
27		6 / 2	B			0.0759	-3.1				
28	x	10 / 6	B	0.120	1.6			0.259	-0.6	0.061	-0.5
29	x	10 / 6	B	0.0875	0.1	0.163	-2.1			0.0678	-0.1
30	x	13 / 8	A	0.134	2.3	0.453	1.4	0.299	-0.1	0.074	0.2
31		4 / 4	B			0.321	-0.2				
32		11 / 7	B			0.373	0.5	0.297	-0.1	0.072	0.1
33	x	13 / 8	A	0.097	0.5	0.340	0.1	0.289	-0.2	0.076	0.3
34		11 / 6	B	0.085	0.0			0.314	0.1	0.058	-0.7
35	x	13 / 7	A	FN	-3.5	0.559	2.7	0.268	-0.5	0.0726	0.1
37		6 / 4	B			0.320	-0.2				
38		8 / 6	B			0.320	-0.2	0.130	-2.3		
39		13 / 8	A	0.083	-0.1	0.360	0.3	0.282	-0.3	0.060	-0.6
40		9 / 5	B	0.0845	-0.1	0.619	3.4	0.199	-1.4		
41		13 / 8	A	0.082	-0.2	0.230	-1.2	0.320	0.2	0.072	0.1
42		13 / 8	A	0.080	-0.3	0.260	-0.9	0.275	-0.4	0.076	0.3
43		2 / 2	B					0.246	-0.8		

* Category A/B classification (Cat A was assigned to laboratories that have correctly analysed at least 12 of 13 compulsory compounds on the Target Pesticides List, corretly detected 7 or more out of the 8 compulsory compounds and that have not reported any false positive results)

Table 4-8 (cont.): Results reported and z-scores achieved by all participating laboratories for COMPULSORY compounds

COMPULSORY Compound				2,4-D		Captan (parent)		Chlorothalonil		Dithiocarbamates	
MRRL [mg/kg]				0.01		0.01		0.01		0.03	
Assigned Value [mg/kg]				0.079		0.085		0.125		0.267	
CV^*				13.3 %		28.1 %		25.2 %		22.2 %	
Lab code SRM12-	NRL-SRM	Analysed / corr. found, max. 13 / 8	Cat.*	Conc. [mg/kg]	z-Score§ (FFP-RSD = 25 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)
44	x	8 / 4	B	FN	-3.5	0.085	0.0	0.067	-1.9	0.323	0.8
45		13 / 8	A	0.0691	-0.5	0.107	1.0	0.171	1.5	0.236	-0.5
46		13 / 8	A	0.085	0.3	0.0748	-0.5	0.130	0.2	0.203	-1.0
47		13 / 8	A	0.076	-0.1	0.063	-1.0	0.170	1.4	0.306	0.6
48		12 / 7	A	0.084	0.3	0.0745	-0.5	0.131	0.2	0.284	0.3
49	x	13 / 8	A	0.0812	0.1	0.0436	-1.9	0.0952	-1.0	0.259	-0.1
50		11 / 7	B	0.085	0.3	0.080	-0.2	0.192	2.1	0.314	0.7
51		8 / 4	B	0.0725	-0.3			0.230	3.3		
52		13 / 8	A	0.069	-0.5	0.137	2.5	0.132	0.2	0.500	3.5
53		13 / 8	A	0.073	-0.3	0.072	-0.6	0.119	-0.2	0.298	0.5
54		6 / 3	B					0.069	-1.8		
55		2 / 1	B							0.227	-0.6
56		7 / 4	B			0.055	-1.4	0.105	-0.6	0.250	-0.3
57		13 / 8	A	0.069	-0.5	0.073	-0.6	0.094	-1.0	0.270	0.0
58		13 / 8	A	0.0617	-0.9	0.106	1.0	0.144	0.6	0.271	0.1
59		1 / 1	B							0.256	-0.2
60		3 / 2	B							0.278	0.2
61		6 / 4	B	0.075	-0.2	0.0815	-0.2				
62	x	9 / 6	B	0.071	-0.4	0.062	-1.1	0.087	-1.2	0.208	-0.9
63		12 / 7	A	0.082	0.2	0.050	-1.6	0.171	1.5	0.219	-0.7
64		10 / 6	B	0.082	0.2			0.151	0.8	0.255	-0.2
65		1 / 1	B							0.259	-0.1
66	x	13 / 8	A	0.0765	-0.1	0.0842	0.0	0.154	0.9	0.268	0.0
67		13 / 8	A	0.102	1.2	0.096	0.5	0.106	-0.6	0.287	0.3
68		8 / 4	B	0.080	0.1					0.280	0.2
69		10 / 6	B	0.089	0.5	0.0843	0.0	0.0995	-0.8	0.308	0.6
70		13 / 8	A	0.056	-1.2	0.020	-3.1	0.120	-0.2	0.270	0.0
71		11 / 6	B	0.791	36.2			0.0995	-0.8	0.247	-0.3
72		13 / 8	A	0.094	0.8	0.137	2.5	0.175	1.6	0.265	0.0
73	x	8 / 5	B			0.131	2.2	0.126	0.0	0.342	1.1
74		13 / 8	A	0.0836	0.2	0.0741	-0.5	0.133	0.2	0.372	1.6
75	x	13 / 8	A	0.128	2.5	0.122	1.7	0.172	1.5	0.476	3.1
76		13 / 8	A	0.084	0.3	0.100	0.7	0.133	0.2	0.376	1.6
77		6 / 5	B			0.080	-0.2	0.105	-0.6	0.191	-1.1
78		13 / 8	A	0.073	-0.3	0.092	0.3	0.122	-0.1	0.211	-0.8
79		11 / 6	B	0.070	-0.4			0.089	-1.2	0.092	-2.6
80		1 / 1	B					0.050	-2.4		
81	x	11 / 6	B	0.0769	-0.1			0.125	0.0	0.251	-0.2
82		1 / 1	B							0.210	-0.9
83	x	12 / 7	A	0.980	45.8	0.080	-0.2	0.010	-3.7		
84		13 / 8	A	0.075	-0.2	0.080	-0.2	0.150	0.8	0.290	0.3
85	x	3 / 1	B					0.212	2.8		
86		11 / 7	B	0.083	0.2	0.193	5.1	0.096	-0.9	0.420	2.3
87		1 / 1	B							0.231	-0.5

* Category A/B classification (Cat A was assigned to laboratories that have correctly analysed at least 12 of 13 compulsory compounds on the Target Pesticides List, corretly detected 7 or more out of the 8 compulsory compounds and that have not reported any false positive results)

Table 4-8 (cont.): Results reported and z-scores achieved by all participating laboratories for COMPULSORY compounds

COMPULSORY Compound				Fenbutatin Oxide		Folpet (parent)		Glyphosate		Haloxyfop	
MRRL [mg/kg]				0.01		0.01		0.03		0.01	
Assigned Value [mg/kg]				0.086		0.334		0.306		0.070	
*CV**				21.0 %		25.0 %		20.9 %		13.9 %	
Lab code SRM12-	NRL-SRM	Analysed / corr. found, max. 13 / 8	Cat.*	Conc. [mg/kg]	z-Score § (FFP-RSD = 25 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)
44	x	8 / 4	B			0.305	-0.4			FN	-3.4
45		13 / 8	A	0.0788	-0.3	0.462	1.5	0.301	-0.1	0.0857	0.9
46		13 / 8	A	0.0738	-0.6	0.326	-0.1	0.235	-0.9	0.064	-0.4
47		13 / 8	A	0.114	1.3	0.260	-0.9	0.323	0.2	0.063	-0.4
48		12 / 7	A			0.329	-0.1	0.239	-0.9	0.067	-0.2
49	x	13 / 8	A	0.0796	-0.3	0.225	-1.3	0.396	1.2	0.0749	0.3
50		11 / 7	B	0.087	0.1	0.379	0.5			0.070	0.0
51		8 / 4	B	0.104	0.9					0.0655	-0.3
52		13 / 8	A	0.036	-2.3	0.862	6.3	0.388	1.1	0.075	0.3
53		13 / 8	A	0.111	1.2	0.326	-0.1	0.280	-0.3	0.074	0.2
54		6 / 3	B			0.269	-0.8			0.084	0.8
55		2 / 1	B								
56		7 / 4	B			0.304	-0.4				
57		13 / 8	A	0.103	0.8	0.270	-0.8	1.300	13.0	0.071	0.0
58		13 / 8	A	0.0695	-0.8	0.271	-0.8	0.255	-0.7	0.078	0.4
59		1 / 1	B								
60		3 / 2	B					0.313	0.1		
61		6 / 4	B			0.385	0.6			0.070	0.0
62	x	9 / 6	B			0.377	0.5	0.256	-0.7	FN	-4.0
63		12 / 7	A	0.231	6.8	0.085	-3.0			0.067	-0.2
64		10 / 6	B			0.350	0.2	0.418	1.5	0.067	-0.2
65		1 / 1	B								
66	x	13 / 8	A	0.0785	-0.3	0.440	1.3	0.281	-0.3	0.0649	-0.3
67		13 / 8	A	0.068	-0.8	0.286	-0.6	0.485	2.3	0.0743	0.2
68		8 / 4	B					0.055	-3.3	0.072	0.1
69		10 / 6	B			0.362	0.3			0.073	0.2
70		13 / 8	A	0.065	-1.0	0.330	-0.1	0.283	-0.3	0.066	-0.2
71		11 / 6	B	0.0679	-0.8			0.326	0.3	0.748	38.6
72		13 / 8	A	0.108	1.0	0.434	1.2	0.325	0.2	0.073	0.2
73	x	8 / 5	B			0.365	0.4	0.359	0.7		
74		13 / 8	A	0.105	0.9	0.344	0.1	0.228	-1.0	0.0772	0.4
75	x	13 / 8	A	0.288	9.5	0.371	0.4	0.288	-0.2	0.121	2.9
76		13 / 8	A	0.072	-0.6	0.360	0.3	0.268	-0.5	0.070	0.0
77		6 / 5	B	0.076	-0.4	0.296	-0.5				
78		13 / 8	A	0.107	1.0	0.377	0.5	0.510	2.7	0.070	0.0
79		11 / 6	B	0.074	-0.5			0.270	-0.5	0.060	-0.6
80		1 / 1	B								
81	x	11 / 6	B	0.0695	-0.8			0.266	-0.5	0.0665	-0.2
82		1 / 1	B								
83	x	12 / 7	A	0.020	-3.1	0.440	1.3	0.170	-1.8	0.810	42.2
84		13 / 8	A	0.100	0.7	0.320	-0.2	0.280	-0.3	0.070	0.0
85	x	3 / 1	B								
86		11 / 7	B			0.231	-1.2	0.456	2.0	0.066	-0.2
87		1 / 1	B								

* Category A/B classification (Cat A was assigned to laboratories that have correctly analysed at least 12 of 13 compulsory compounds on the Target Pesticides List, corretly detected 7 or more out of the 8 compulsory compounds and that have not reported any false positive results)

Table 4-8 (cont.): Results reported and z-scores achieved by all participating laboratories for COMPULSORY compounds

COMPULSORY Compound				2,4-D		Captan (parent)		Chlorothalonil		Dithiocarbamates	
MRRL [mg/kg]				0.01		0.01		0.01		0.03	
Assigned Value [mg/kg]				0.079		0.085		0.125		0.267	
CV^*				13.3 %		28.1 %		25.2 %		22.2 %	
Lab code SRM12-	NRL-SRM	Analysed / corr. found, max. 13 / 8	Cat.*	Conc. [mg/kg]	z-Score§ (FFP-RSD = 25 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)
88		13 / 8	A	0.075	-0.2	0.088	0.1	0.122	-0.1	0.255	-0.2
89		7 / 4	B	0.102	1.2			0.148	0.7		
90		13 / 8	A	0.086	0.4	0.090	0.2	0.127	0.1	0.566	4.5
91	x	11 / 7	B	0.120	2.1	0.088	0.1	0.140	0.5	0.280	0.2
92	x	13 / 8	A	0.0905	0.6	0.0852	0.0	0.106	-0.6	0.281	0.2
93		10 / 6	B	0.0722	-0.3	0.0471	-1.8	0.111	-0.5		
94		13 / 8	A	0.076	-0.1	0.078	-0.3	0.135	0.3	0.295	0.4
95		13 / 8	A	0.078	0.0	0.149	3.0	0.116	-0.3	0.212	-0.8
96	x	13 / 8	A	0.071	-0.4	0.099	0.7	0.103	-0.7	0.307	0.6
97		13 / 8	A	0.087	0.4	0.086	0.0	0.173	1.5	0.348	1.2
98	x	13 / 8	A	0.069	-0.5	0.0213	-3.0	0.0353	-2.9	0.247	-0.3
99		13 / 7	A	0.079	0.0	0.188	4.9	0.116	-0.3	0.282	0.2
100		1 / 1	B							0.330	0.9
101		1 / 1	B					0.080	-1.4		
102		13 / 7	A	0.064	-0.7	0.089	0.2	0.310	5.9	0.240	-0.4
103	x	13 / 8	A	0.0782	0.0	0.105	0.9	0.138	0.4	0.263	-0.1
104		13 / 8	A	0.0907	0.6	0.0818	-0.1	0.102	-0.7	0.296	0.4
105	x	6 / 4	B			0.120	1.7	0.160	1.1	0.260	-0.1
106		13 / 4	B	0.100	1.1	FN	-3.5	FN	-3.7	0.181	-1.3
107	x	13 / 8	A	0.112	1.7	0.063	-1.0	0.161	1.1	0.147	-1.8
108		11 / 7	B	0.087	0.4	0.450	17.2	0.114	-0.4	0.270	0.0
109		13 / 8	A	0.075	-0.2	0.076	-0.4	0.145	0.6	0.302	0.5
110		3 / 3	B			0.313	10.7	0.167	1.3		
111		1 / 1	B							0.240	-0.4
112		3 / 3	B			0.042	-2.0	0.037	-2.8		
113		12 / 8	A	0.085	0.3	0.063	-1.0	0.094	-1.0	0.340	1.1
114		1 / 1	B							0.290	0.3
115		13 / 8	A	0.059	-1.0	0.090	0.2	0.135	0.3	0.280	0.2
116		13 / 8	A	0.0833	0.2	0.0792	-0.3	0.131	0.2	0.253	-0.2
117		9 / 5	B	0.0918	0.7	0.126	1.9	0.102	-0.7		
118		5 / 4	B					0.086	-1.3	0.150	-1.8
119		1 / 1	B							0.269	0.0
120		5 / 2	B			FN	-3.5	0.111	-0.5		
121		3 / 1	B					0.121	-0.1		
122		3 / 2	B							0.274	0.1
123		2 / 1	B								
124	x	9 / 6	B	0.0766	-0.1	0.095	0.5	0.131	0.2	0.221	-0.7
125		12 / 7	A	0.094	0.8	0.137	2.5	0.137	0.4		
126		9 / 7	B	0.059	-1.0	0.072	-0.6	0.131	0.2		
127		13 / 8	A	0.073	-0.3	0.104	0.9	0.154	0.9	0.336	1.0
128		1 / 1	B	0.0658	-0.7						
129		12 / 7	A	0.086	0.4	0.069	-0.8	0.101	-0.8	0.142	-1.9
130		13 / 8	A	0.101	1.1	0.089	0.2	0.105	-0.6	0.181	-1.3

* Category A/B classification (Cat A was assigned to laboratories that have correctly analysed at least 12 of 13 compulsory compounds on the Target Pesticides List, corretly detected 7 or more out of the 8 compulsory compounds and that have not reported any false positive results)

Table 4-8 (cont.): Results reported and z-scores achieved by all participating laboratories for COMPULSORY compounds

	COMPULSORY Compound			Fenbutatin Oxide		Folpet (parent)		Glyphosate		Haloxyfop	
	MRRL [mg/kg]			0.01		0.01		0.03		0.01	
	Assigned Value [mg/kg]			0.086		0.334		0.306		0.070	
	*CV**			21.0 %		25.0 %		20.9 %		13.9 %	
Lab code SRM12-	NRL-SRM	Analysed / corr. found, max. 13 / 8	Cat.*	Conc. [mg/kg]	z-Score § (FFP-RSD = 25 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)
88		13 / 8	A	0.080	-0.3	0.301	-0.4	0.312	0.1	0.056	-0.8
89		7 / 4	B	0.076	-0.4			0.322	0.2		
90		13 / 8	A	0.095	0.4	0.253	-1.0	0.271	-0.5	0.080	0.6
91	x	11 / 7	B	0.079	-0.3	0.360	0.3			0.054	-0.9
92	x	13 / 8	A	0.0842	-0.1	0.355	0.2	0.346	0.5	0.0803	0.6
93		10 / 6	B	0.085	0.0	0.287	-0.6			0.0545	-0.9
94		13 / 8	A	0.079	-0.3	0.346	0.1	0.364	0.8	0.072	0.1
95		13 / 8	A	0.073	-0.6	0.751	5.0	0.350	0.6	0.076	0.3
96	x	13 / 8	A	0.080	-0.3	0.117	-2.6	0.284	-0.3	0.064	-0.4
97		13 / 8	A	0.104	0.9	0.424	1.1	0.775	6.1	0.087	1.0
98	x	13 / 8	A	0.0561	-1.4	0.141	-2.3	0.301	-0.1	0.0515	-1.1
99		13 / 7	A	FN	-3.5	0.375	0.5	0.200	-1.4	0.077	0.4
100		1 / 1	B								
101		1 / 1	B								
102		13 / 7	A	0.059	-1.2	FN	-3.9	0.290	-0.2	0.032	-2.2
103	x	13 / 8	A	0.0951	0.4	0.440	1.3	0.295	-0.1	0.0727	0.1
104		13 / 8	A	0.0765	-0.4	0.328	-0.1	0.341	0.5	0.0753	0.3
105	x	6 / 4	B			0.450	1.4				
106		13 / 4	B	FN	-3.5	FN	-3.9	0.350	0.6	0.120	2.8
107	x	13 / 8	A	0.107	1.0	0.307	-0.3	0.212	-1.2	0.0856	0.9
108		11 / 7	B			0.350	0.2	0.193	-1.5	0.047	-1.3
109		13 / 8	A	0.086	0.0	0.351	0.2	0.260	-0.6	0.070	0.0
110		3 / 3	B			1.23	10.7				
111		1 / 1	B								
112		3 / 3	B			0.132	-2.4				
113		12 / 8	A	0.087	0.1	0.285	-0.6	0.300	-0.1	0.076	0.3
114		1 / 1	B								
115		13 / 8	A	0.075	-0.5	0.347	0.2	0.270	-0.5	0.071	0.0
116		13 / 8	A	0.107	1.0	0.246	-1.1	0.351	0.6	0.0752	0.3
117		9 / 5	B			0.258	-0.9			0.0949	1.4
118		5 / 4	B			0.292	-0.5			0.134	3.6
119		1 / 1	B								
120		5 / 2	B			0.330	-0.1				
121		3 / 1	B								
122		3 / 2	B					0.345	0.5		
123		2 / 1	B	0.080	-0.3						
124	x	9 / 6	B			0.323	-0.1			0.0728	0.1
125		12 / 7	A	0.102	0.8	0.526	2.3	0.442	1.8	0.082	0.7
126		9 / 7	B	0.036	-2.3	0.295	-0.5	0.291	-0.2	0.047	-1.3
127		13 / 8	A	0.078	-0.4	0.375	0.5	0.371	0.8	0.068	-0.1
128		1 / 1	B								
129		12 / 7	A	0.072	-0.6	0.259	-0.9			0.067	-0.2
130		13 / 8	A	0.123	1.7	0.389	0.7	0.371	0.8	0.087	1.0

* Category A/B classification (Cat A was assigned to laboratories that have correctly analysed at least 12 of 13 compulsory compounds on the Target Pesticides List, corretly detected 7 or more out of the 8 compulsory compounds and that have not reported any false positive results)

Table 4-8 (cont.): Results reported and z-scores achieved by all participating laboratories for COMPULSORY compounds

COMPULSORY Compound				2,4-D		Captan (parent)		Chlorothalonil		Dithiocarbamates	
MRRL [mg/kg]				0.01		0.01		0.01		0.03	
Assigned Value [mg/kg]				0.079		0.085		0.125		0.267	
*CV**				13.3 %		28.1 %		25.2 %		22.2 %	
Lab code SRM12-	NRL-SRM	Analysed / corr. found, max. 13 / 8	Cat.*	Conc. [mg/kg]	z-Score § (FFP-RSD = 25 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)
3rd-131		7 / 4	B	0.085	0.3			0.135	0.3	0.235	-0.5
3rd-132		11 / 7	B	0.083	0.2	0.089	0.2	0.070	-1.8	0.290	0.3
3rd-133		12 / 7	A	0.0797	0.1	0.0844	0.0	0.133	0.2		
3rd-134		3 / 3	B					0.110	-0.5	0.270	0.0
3rd-135		4 / 2	B					0.090	-1.1	1.96	25.4
3rd-136		13 / 8	A	0.077	-0.1	0.111	1.2	0.0422	-2.7	0.232	-0.5
3rd-137		4 / 3	B	0.087	0.4			0.285	5.1		
3rd-138		6 / 5	B			0.265	8.5	0.095	-1.0	0.304	0.6
3rd-139		13 / 8	A	0.208	6.6	0.0775	-0.4	0.118	-0.2	0.473	3.1

* Category A/B classification (Cat A was assigned to laboratories that have correctly analysed at least 12 of 13 compulsory compounds on the Target Pesticides List, corretly detected 7 or more out of the 8 compulsory compounds and that have not reported any false positive results)

Table 4-9: Results reported and z-scores achieved by all participating laboratories for OPTIONAL compounds

OPTIONAL Compound				Bifenazate		Bromide ion		Chlorate		Dithianon	
MRRL [mg/kg]				0.02		3		0.02		0.02	
Assigned Value [mg/kg]				0.270		19.1		0.490		0.294	
*CV**				22.1 %		16.0 %		16.2 %		25.3 %	
Lab code SRM12-	NRL-SRM	Analysed / corr. found max. 8 / 7	Cat.*	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)
1		7 / 6	A	0.172	-1.5	22.0	0.6	0.452	-0.3	0.160	-1.8
2		7 / 5	A	0.198	-1.1	17.3	-0.4	0.470	-0.2	0.268	-0.3
3	x	4 / 3	A	0.201	-1.0					0.104	-2.6
4		2 / 2	B							0.284	-0.1
5		0 / 0	B								
6	x	7 / 4	A	0.184	-1.3			0.700	1.7	0.275	-0.3
7		5 / 4	A	0.385	1.7	13.2	-1.2	0.504	0.1		
8		6 / 5	B			21.2	0.4	0.790	2.5		
9		3 / 3	A					0.525	0.3		
10		4 / 3	B					0.546	0.5		
11		0 / 0	B								
12		7 / 5	A	0.220	-0.7	21.0	0.4	0.470	-0.2	0.290	0.0
13	x	4 / 3	A			28.5	2.0			0.280	-0.2
14		4 / 3	A					0.525	0.3	0.306	0.2
15		1 / 1	B			18.0	-0.2				
16	x	2 / 1	B			20.2	0.2				
17	x	2 / 2	A	0.308	0.6						
18		7 / 6	A	0.249	-0.3	17.1	-0.4	0.490	0.0	0.287	-0.1

* Category A/B classification (Cat A was assigned to laboratories that have correctly analysed at least 12 of 13 compulsory compounds on the Target Pesticides List, corretly detected 7 or more out of the 8 compulsory compounds and that have not reported any false positive result)

Table 4-8 (cont.): Results reported and z-scores achieved by all participating laboratories for COMPULSORY compounds

	COMPULSORY Compound			Fenbutatin Oxide		Folpet (parent)		Glyphosate		Haloxyfop	
	MRRL [mg/kg]			0.01		0.01		0.03		0.01	
	Assigned Value [mg/kg]			0.086		0.334		0.306		0.070	
	*CV**			21.0 %		25.0 %		20.9 %		13.9 %	
	Lab code SRM12-	NRL-SRM	Analysed / corr. found, max. 13 / 8	Cat.*	Conc. [mg/kg]	z-Score§ (FFP-RSD = 25 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)
	3rd-131		7 / 4	B					0.204	-1.3		
	3rd-132		11 / 7	B			0.394	0.7	0.098	-2.7	0.032	-2.2
	3rd-133		12 / 7	A	0.0893	0.2	0.355	0.2	0.299	-0.1	0.0733	0.2
	3rd-134		3 / 3	B	0.090	0.2						
	3rd-135		4 / 2	B								
	3rd-136		13 / 8	A	0.0899	0.2	0.665	4.0	0.315	0.1	0.0658	-0.3
	3rd-137		4 / 3	B					0.179	-1.7		
	3rd-138		6 / 5	B	0.110	1.1	0.745	4.9				
	3rd-139		13 / 8	A	0.249	7.6	0.0953	-2.9	0.317	0.1	0.016	-3.1

* Category A/B classification (Cat A was assigned to laboratories that have correctly analysed at least 12 of 13 compulsory compounds on the Target Pesticides List, corretly detected 7 or more out of the 8 compulsory compounds and that have not reported any false positive results)

Table 4-9 (cont.): Results reported and z-scores achieved by all participating laboratories for OPTIONAL compounds

	OPTIONAL Compound			Phosphonic acid		N-Acetyl glyphosate		*Carbofuran´ (z-scores for information only)*		
	MRRL [mg/kg]			0.05		0.02		*0.001*		
	Assigned Value [mg/kg]			19.3		0.100			*0.0030* (based on entire population, 58 numerical results)	*0.0039* (based on 13 results with acidic transformation)
	*CV**			27.0 %		23.2 %			*47.1 %*	*50.2 %*
	Lab code SRM12-	NRL-SRM	Analysed / corr. found max. 8 / 7	Cat.*	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)	Conc. [mg/kg]	z-score (FFP-RSD = 25 %)	z-score (FFP-RSD = 25 %)
	1		7 / 6	A	15.9	-0.7			*0.0027*	*-0.4*	*-1.2*
	2		7 / 5	A	17.9	-0.3			*FN*	*-2.7*	*-3.0*
	3	x	4 / 3	A					*0.0020*	*-1.3*	*-2.0*
	4		2 / 2	B					*0.00163*	*-1.8*	*-2.3*
	5		0 / 0	B							
	6	x	7 / 4	A	20.9	0.3	FN	-3.2	*FN*	*-2.7*	*-3.0*
	7		5 / 4	A	20.34	0.2					
	8		6 / 5	B	16.7	-0.5	0.093	-0.3	*0.0027*	*-0.4*	*-1.2*
	9		3 / 3	A	51.7	6.7			*0.0034*	*0.5*	*-0.5*
	10		4 / 3	B	27.9	1.8			*0.0067*	*4.9*	*2.8*
	11		0 / 0	B							
	12		7 / 5	A	21.0	0.4			*FN*	*-2.7*	*-3.0*
	13	x	4 / 3	A					*0.0019*	*-1.5*	*-2.1*
	14		4 / 3	A	17.78	-0.3					
	15		1 / 1	B							
	16	x	2 / 1	B							
	17	x	2 / 2	A					*0.0057*	*3.6*	*1.8*
	18		7 / 6	A	20.1	0.2			*0.0018*	*-1.6*	*-2.2*

* Category A/B classification (Cat A was assigned to laboratories that have correctly analysed at least 12 of 13 compulsory compounds on the Target Pesticides List, corretly detected 7 or more out of the 8 compulsory compounds and that have not reported any false positive result)

Table 4-9 (cont.): Results reported and z-scores achieved by all participating laboratories for OPTIONAL compounds

OPTIONAL Compound				Bifenazate		Bromide ion		Chlorate		Dithianon	
MRRL [mg/kg]				0.02		3		0.02		0.02	
Assigned Value [mg/kg]				0.270		19.1		0.490		0.294	
*CV**				22.1 %		16.0 %		16.2 %		25.3 %	
Lab code SRM12-	NRL-SRM	Analysed / corr. found max. 8 / 7	Cat.*	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)
19	x	3 / 3	A			20.2	0.2	0.551	0.5	0.277	-0.2
20		4 / 4	A					0.545	0.5	0.305	0.2
21		6 / 5	A	0.241	-0.4			0.524	0.3	0.390	1.3
22		5 / 5	A			18.9	0.0	0.510	0.2	0.340	0.6
23		0 / 0	B								
24		2 / 2	B			18.2	-0.2			0.329	0.5
25		5 / 4	A	0.320	0.7					0.320	0.4
26		7 / 6	A	0.300	0.4	18.0	-0.2	0.570	0.7	0.330	0.5
27		2 / 1	B	0.239	-0.5						
28	x	1 / 1	B							0.267	-0.4
29	x	2 / 2	B	0.218	-0.8						
30	x	3 / 2	A							0.748	6.2
31		0 / 0	B								
32		0 / 0	B								
33	x	8 / 7	A	0.204	-1.0	17.3	-0.4	0.527	0.3	0.307	0.2
34		7 / 6	B	0.326	0.8	10.1	-1.9	1.17	5.6	0.242	-0.7
35	x	8 / 7	A	0.316	0.7	18.0	-0.2	0.447	-0.3	0.306	0.2
37		0 / 0	B								
38		2 / 2	B					0.510	0.2		
39		7 / 5	A	0.280	0.1	18.0	-0.2	0.560	0.6	0.410	1.6
40		3 / 2	B							0.325	0.4
41		6 / 5	A	0.280	0.1	16.9	-0.5			1.40	15.1
42		6 / 5	A	0.275	0.1	0.160	-4.0	0.529	0.3		
43		2 / 1	B					0.340	-1.2		
44	x	0 / 0	B								
45		4 / 4	A	0.377	1.6	29.6	2.2	0.248	-2.0		
46		3 / 2	A					0.490	0.0	0.0407	-3.4
47		6 / 5	A	0.285	0.2	19.5	0.1	0.492	0.0	0.297	0.0
48		5 / 4	A	0.295	0.4	20.79	0.4	0.348	-1.2		
49	x	2 / 2	A			19.0	0.0				
50		4 / 4	B	0.285	0.2	20.3	0.2			0.323	0.4
51		1 / 1	B							0.380	1.2
52		8 / 6	A	0.257	-0.2	21.5	0.5	0.488	0.0	0.024	-3.7
53		7 / 6	A	0.234	-0.5	21.483	0.5	0.494	0.0	0.317	0.3
54		0 / 0	B								
55		0 / 0	B								
56		0 / 0	B								
57		4 / 4	A					0.547	0.5	0.107	-2.5
58		7 / 6	A	0.423	2.3	27.4	1.7	0.102	-3.2	0.189	-1.4
59		0 / 0	B								
60		0 / 0	B								
61		0 / 0	B								

* Category A/B classification (Cat A was assigned to laboratories that have correctly analysed at least 12 of 13 compulsory compounds on the Target Pesticides List, corretly detected 7 or more out of the 8 compulsory compounds and that have not reported any false positive result)

Table 4-9 (cont.): Results reported and z-scores achieved by all participating laboratories for OPTIONAL compounds

	OPTIONAL Compound			Phosphonic acid		N-Acetyl glyphosate		*Carbofuran´ (z-scores for information only)*		
	MRRL [mg/kg]			0.05		0.02		*0.001*		
	Assigned Value [mg/kg]			19.3		0.100			*0.0030* (based on entire population, 58 numerical results)	*0.0039* (based on 13 results with acidic transformation)
	*CV**			27.0 %		23.2 %			*47.1 %*	*50.2 %*
Lab code SRM12-	NRL-SRM	Analysed / corr. found max. 8 / 7	Cat.*	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)	Conc. [mg/kg]	z-score (FFP-RSD = 25 %)	z-score (FFP-RSD = 25 %)
19	x	3 / 3	A							
20		4 / 4	A			0.069	-1.2	*0.0024*	*-0.8*	*-1.6*
21		6 / 5	A	16.4	-0.6			*0.0031*	*0.1*	*-0.8*
22		5 / 5	A	22.4	0.7			*0.0053*	*3.1*	*1.4*
23		0 / 0	B							
24		2 / 2	B							
25		5 / 4	A			0.093	-0.3	*0.0061*	*4.1*	*2.2*
26		7 / 6	A	25.3	1.3			*0.0020*	*-1.3*	*-2.0*
27		2 / 1	B					*FN*	*-2.7*	*-3.0*
28	x	1 / 1	B							
29	x	2 / 2	B					*0.0034*	*0.5*	*-0.5*
30	x	3 / 2	A					*0.0016*	*-1.9*	*-2.4*
31		0 / 0	B							
32		0 / 0	B							
33	x	8 / 7	A	17.3	-0.4	0.134	1.3	*0.0039*	*1.2*	*0.0*
34		7 / 6	B	67.6	10.0			*0.0021*	*-1.2*	*-1.9*
35	x	8 / 7	A	19.9	0.1	0.125	1.0	*0.0047*	*2.3*	*0.8*
37		0 / 0	B							
38		2 / 2	B					*0.0018*	*-1.6*	*-2.2*
39		7 / 5	A			0.121	0.8	*FN*	*-2.7*	*-3.0*
40		3 / 2	B					*0.0090*	*8.0*	*5.2*
41		6 / 5	A	25.0	1.2			*0.0025*	*-0.7*	*-1.5*
42		6 / 5	A	18.85	-0.1			*0.00435*	*1.8*	*0.4*
43		2 / 1	B							
44	x	0 / 0	B							
45		4 / 4	A					*0.0023*	*-0.9*	*-1.7*
46		3 / 2	A							
47		6 / 5	A	14.2	-1.1					
48		5 / 4	A					*0.0039*	*1.2*	*0.0*
49	x	2 / 2	A					*0.0012*	*-2.4*	*-2.8*
50		4 / 4	B					*0.0015*	*-2.0*	*-2.5*
51		1 / 1	B							
52		8 / 6	A	15.3	-0.8	0.139	1.5	*FN*	*-2.7*	*-3.0*
53		7 / 6	A	21.026	0.4			*0.0040*	*1.3*	*0.1*
54		0 / 0	B							
55		0 / 0	B							
56		0 / 0	B							
57		4 / 4	A	5.14	-2.9			*0.0046*	*2.1*	*0.7*
58		7 / 6	A	28.0	1.8			*0.0041*	*1.5*	*0.2*
59		0 / 0	B							
60		0 / 0	B							
61		0 / 0	B							

* Category A/B classification (Cat A was assigned to laboratories that have correctly analysed at least 12 of 13 compulsory compounds on the Target Pesticides List, corretly detected 7 or more out of the 8 compulsory compounds and that have not reported any false positive result)

Table 4-9 (cont.): Results reported and z-scores achieved by all participating laboratories for OPTIONAL compounds

OPTIONAL Compound				Bifenazate		Bromide ion		Chlorate		Dithianon	
MRRL [mg/kg]				0.02		3		0.02		0.02	
Assigned Value [mg/kg]				0.270		19.1		0.490		0.294	
*CV**				22.1 %		16.0 %		16.2 %		25.3 %	
Lab code SRM12-	NRL-SRM	Analysed / corr. found max. 8 / 7	Cat.*	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)
62	x	2 / 0	B								
63		6 / 6	A	0.304	0.5	31.0	2.5	0.221	-2.2	0.252	-0.6
64		2 / 2	B							0.299	0.1
65		0 / 0	B								
66	x	4 / 3	A			19.7	0.1	0.695	1.7		
67		5 / 4	A					0.402	-0.7	0.317	0.3
68		1 / 0	B								
69		2 / 2	B			19.4	0.1				
70		6 / 5	A	0.630	5.3			0.128	-3.0	0.350	0.8
71		6 / 5	B	0.245	-0.4	29.3	2.1	0.411	-0.6	0.179	-1.6
72		6 / 5	A	0.187	-1.2			0.554	0.5	0.384	1.2
73	x	2 / 1	B					0.391	-0.8		
74		5 / 4	A	0.340	1.0	19.9	0.2			0.277	-0.2
75	x	2 / 1	A							0.019	-3.7
76		8 / 7	A	0.227	-0.6	16.35	-0.6	0.480	-0.1	0.285	-0.1
77		0 / 0	B								
78		5 / 5	A	0.245	-0.4	18.1	-0.2	0.471	-0.2		
79		6 / 5	B			10.6	-1.8	0.501	0.1	0.868	7.8
80		0 / 0	B								
81	x	6 / 6	B			19.7	0.1	0.400	-0.7	0.282	-0.2
82		0 / 0	B								
83	x	0 / 0	A								
84		7 / 6	A	0.290	0.3	20.0	0.2	0.540	0.4	0.280	-0.2
85	x	0 / 0	B								
86		4 / 3	B	0.239	-0.5			0.468	-0.2		
87		0 / 0	B								
88		1 / 1	A					0.485	0.0		
89		2 / 2	B	0.397	1.9					0.238	-0.8
90		5 / 4	A					0.482	-0.1	2.57	31.0
91	x	2 / 2	B			14.8	-0.9			0.260	-0.5
92	x	5 / 5	A	0.293	0.3			0.507	0.1	0.313	0.3
93		0 / 0	B								
94		4 / 4	A			11.9	-1.5	0.549	0.5	0.405	1.5
95		7 / 5	A	0.237	-0.5	21.2	0.4	0.433	-0.5		-3.9
96	x	0 / 0	A								
97		8 / 6	A	0.350	1.2	24.4	1.1	0.502	0.1	0.300	0.1
98	x	5 / 4	A			17.8	-0.3	0.441	-0.4	0.299	0.1
99		7 / 6	A	0.201	-1.0	16.8	-0.5	0.562	0.6		
100		0 / 0	B								
101		0 / 0	B								
102		7 / 6	A	0.230	-0.6	18.1	-0.2	0.730	2.0	0.250	-0.6

* Category A/B classification (Cat A was assigned to laboratories that have correctly analysed at least 12 of 13 compulsory compounds on the Target Pesticides List, corretly detected 7 or more out of the 8 compulsory compounds and that have not reported any false positive result)

Table 4-9 (cont.): Results reported and z-scores achieved by all participating laboratories for OPTIONAL compounds

	OPTIONAL Compound				Phosphonic acid		N-Acetyl glyphosate		*Carbofuran´ (z-scores for information only)*		
	MRRL [mg/kg]				0.05		0.02		*0.001*		
	Assigned Value [mg/kg]				19.3		0.100			*0.0030* (based on entire population, 58 numerical results)	*0.0039* (based on 13 results with acidic transformation)
	*CV**				27.0 %		23.2 %			*47.1 %*	*50.2 %*
	Lab code SRM12-	NRL-SRM	Analysed / corr. found max. 8 / 7	Cat.*	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)	Conc. [mg/kg]	z-score (FFP-RSD = 25 %)	z-score (FFP-RSD = 25 %)
	62	x	2 / 0	B					*FN*	*-2.7*	*-3.0*
	63		6 / 6	A	0.185	-4.0			*0.0090*	*8.0*	*5.2*
	64		2 / 2	B	19.1	0.0					
	65		0 / 0	B							
	66	x	4 / 3	A					*0.0026*	*-0.5*	*-1.4*
	67		5 / 4	A	13.7	-1.2			*0.0025*	*-0.7*	*-1.5*
	68		1 / 0	B							
	69		2 / 2	B					*0.0021*	*-1.2*	*-1.9*
	70		6 / 5	A	9.10	-2.1			*0.0051*	*2.8*	*1.2*
	71		6 / 5	B					*0.0028*	*-0.3*	*-1.1*
	72		6 / 5	A	18.95	-0.1			*0.0018*	*-1.6*	*-2.2*
	73	x	2 / 1	B					*FN*	*-2.7*	*-3.0*
	74		5 / 4	A					*0.0029*	*-0.1*	*-1.0*
	75	x	2 / 1	A					*FN*	*-2.7*	*-3.0*
	76		8 / 7	A	18.05	-0.3	0.093	-0.3	*0.0021*	*-1.2*	*-1.9*
	77		0 / 0	B							
	78		5 / 5	A	17.4	-0.4			*0.00411*	*1.5*	*0.2*
	79		6 / 5	B	23.03	0.8			*0.0020*	*-1.3*	*-2.0*
	80		0 / 0	B							
	81	x	6 / 6	B	21.0	0.4	0.106	0.2	*0.0027*	*-0.4*	*-1.2*
	82		0 / 0	B							
	83	x	0 / 0	A							
	84		7 / 6	A	22.0	0.6			*0.0049*	*2.5*	*1.0*
	85	x	0 / 0	B							
	86		4 / 3	B	14.5	-1.0					
	87		0 / 0	B							
	88		1 / 1	A							
	89		2 / 2	B							
	90		5 / 4	A	9.95	-1.9			*0.0038*	*1.1*	*-0.1*
	91	x	2 / 2	B							
	92	x	5 / 5	A	20.0	0.2			*0.0024*	*-0.8*	*-1.6*
	93		0 / 0	B							
	94		4 / 4	A	22.7	0.7					
	95		7 / 5	A	14.6	-1.0			*0.0007*	*-3.1*	*-3.3*
	96	x	0 / 0	A							
	97		8 / 6	A	20.9	0.3	0.093	-0.3	*FN*	*-2.7*	*-3.0*
	98	x	5 / 4	A	21.2	0.4			*FN*	*-2.7*	*-3.0*
	99		7 / 6	A	7.91	-2.4	0.084	-0.7	*0.0019*	*-1.5*	*-2.1*
	100		0 / 0	B							
	101		0 / 0	B							
	102		7 / 6	A	20.5	0.3			*0.0017*	*-1.7*	*-2.3*

* Category A/B classification (Cat A was assigned to laboratories that have correctly analysed at least 12 of 13 compulsory compounds on the Target Pesticides List, corretly detected 7 or more out of the 8 compulsory compounds and that have not reported any false positive result)

Table 4-9 (cont.): Results reported and z-scores achieved by all participating laboratories for OPTIONAL compounds

OPTIONAL Compound				Bifenazate		Bromide ion		Chlorate		Dithianon	
MRRL [mg/kg]				0.02		3		0.02		0.02	
Assigned Value [mg/kg]				0.270		19.1		0.490		0.294	
*CV**				22.1 %		16.0 %		16.2 %		25.3 %	
Lab code SRM12-	NRL-SRM	Analysed / corr. found max. 8 / 7	Cat.*	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)
103	x	8 / 7	A	0.278	0.1	18.4	-0.1	0.415	-0.6	0.323	0.4
104		3 / 2	A	0.302	0.5					0.271	-0.3
105	x	0 / 0	B								
106		3 / 1	B	0.250	-0.3						
107	x	3 / 2	A	0.239	-0.5					0.188	-1.4
108		0 / 0	B								
109		7 / 6	A	0.310	0.6			0.535	0.4	0.346	0.7
110		0 / 0	B								
111		0 / 0	B								
112		0 / 0	B								
113		5 / 4	A	0.310	0.6	23.0	0.8			0.300	0.1
114		0 / 0	B								
115		8 / 7	A	0.250	-0.3	20.0	0.2	0.490	0.0	0.280	-0.2
116		5 / 4	A			18.3	-0.2	0.516	0.2	0.438	2.0
117		1 / 1	B			19.1	0.0				
118		3 / 3	B	0.210	-0.9	21.1	0.4				
119		0 / 0	B								
120		1 / 0	B								
121		1 / 1	B								
122		1 / 0	B								
123		1 / 1	B								
124	x	1 / 0	B								
125		8 / 7	A	0.322	0.8	18.0	-0.2	0.570	0.7	0.354	0.8
126		2 / 1	B	0.159	-1.6						
127		6 / 5	A	0.333	0.9			0.268	-1.8	0.261	-0.4
128		0 / 0	B								
129		5 / 5	A	0.256	-0.2	5.66	-2.8	0.232	-2.1	0.233	-0.8
130		6 / 5	A	0.258	-0.2			0.497	0.1	0.210	-1.1
3rd-131		2 / 1	B							0.340	0.6
3rd-132		2 / 1	B			9.50	-2.0				
3rd-133		2 / 1	A			21.2	0.4				
3rd-134		2 / 2	B	0.500	3.4						
3rd-135		1 / 0	B								
3rd-136		0 / 0	A								
3rd-137		0 / 0	B								
3rd-138		3 / 3	B	0.219	-0.8	20.3	0.2				
3rd-139		0 / 0	A								

* Category A/B classification (Cat A was assigned to laboratories that have correctly analysed at least 12 of 13 compulsory compounds on the Target Pesticides List, corretly detected 7 or more out of the 8 compulsory compounds and that have not reported any false positive result)

Table 4-9 (cont.): Results reported and z-scores achieved by all participating laboratories for OPTIONAL compounds

	OPTIONAL Compound				Phosphonic acid		N-Acetyl glyphosate		*Carbofuran´ (z-scores for information only)*		
	MRRL [mg/kg]				0.05		0.02		*0.001*		
	Assigned Value [mg/kg]				19.3		0.100			*0.0030* (based on entire population, 58 numerical results)	*0.0039* (based on 13 results with acidic transformation)
	*CV**				27.0 %		23.2 %			*47.1 %*	*50.2 %*
	Lab code SRM12-	NRL-SRM	Analysed / corr. found max. 8 / 7	Cat.*	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)	Conc. [mg/kg]	z-score (FFP-RSD = 25 %)	z-score (FFP-RSD = 25 %)
	103	x	8 / 7	A	20.4	0.2	0.094	-0.3	*0.0032*	*0.3*	*-0.7*
	104		3 / 2	A							
	105	x	0 / 0	B							
	106		3 / 1	B					*FN*	*-2.7*	*-3.0*
	107	x	3 / 2	A					*FN*	*-2.7*	*-3.0*
	108		0 / 0	B							
	109		7 / 6	A	22.5	0.7	0.09	-0.4	*0.0031*	*0.1*	*-0.8*
	110		0 / 0	B							
	111		0 / 0	B							
	112		0 / 0	B							
	113		5 / 4	A					*0.0032*	*0.3*	*-0.7*
	114		0 / 0	B							
	115		8 / 7	A	29.0	2.0	0.071	-1.2	*0.0017*	*-1.7*	*-2.3*
	116		5 / 4	A	17.4	-0.4					
	117		1 / 1	B							
	118		3 / 3	B					*0.033*	*40.0*	*29.6*
	119		0 / 0	B							
	120		1 / 0	B					*FN*	*-2.7*	*-3.0*
	121		1 / 1	B					*0.0023*	*-0.9*	*-1.7*
	122		1 / 0	B							
	123		1 / 1	B					*0.0013*	*-2.3*	*-2.7*
	124	x	1 / 0	B					*FN*	*-2.7*	*-3.0*
	125		8 / 7	A	18.0	-0.3	0.104	0.1	*0.0027*	*-0.4*	*-1.2*
	126		2 / 1	B					*FN*	*-2.7*	*-3.0*
	127		6 / 5	A	20.1	0.2			*0.0027*	*-0.4*	*-1.2*
	128		0 / 0	B							
	129		5 / 5	A	8.41	-2.3					
	130		6 / 5	A	26.5	1.5			*0.0022*	*-1.1*	*-1.8*
	3rd-131		2 / 1	B							
	3rd-132		2 / 1	B					*FN*	*-2.7*	*-3.0*
	3rd-133		2 / 1	A							
	3rd-134		2 / 2	B					*0.0030*	*0.0*	*-0.9*
	3rd-135		1 / 0	B					*FN*	*-2.7*	*-3.0*
	3rd-136		0 / 0	A							
	3rd-137		0 / 0	B							
	3rd-138		3 / 3	B	17.4	-0.4					
	3rd-139		0 / 0	A							

* Category A/B classification (Cat A was assigned to laboratories that have correctly analysed at least 12 of 13 compulsory compounds on the Target Pesticides List, corretly detected 7 or more out of the 8 compulsory compounds and that have not reported any false positive result)

Table 4-10: Results reported and z-scores achieved by all participating laboratories for ADDITIONAL compounds

ADDITIONAL Compound				Captan (sum)		Folpet (sum)		THPI		Phthalimide	
MRRL [mg/kg]				–		–		–		–	
Assigned Value [mg/kg]				0.302		1.195		0.110		0.446	
*CV**				25.2 %		21.1 %		30.5 %		21.6 %	
Lab code SRM12-	NRL-SRM	Analysed / corr. found max. 8 / 7	Cat.*	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)
1		4 / 4	A	0.252	-0.7	0.961	-0.8	0.099	-0.4	0.382	-0.6
2		4 / 4	A	0.277	-0.3	1.12	-0.3	0.0946	-0.6	0.382	-0.6
3	x	0 / 0	A								
4		4 / 4	B	0.246	-0.7	1.21	0.1	0.0874	-0.8	0.454	0.1
5		0 / 0	B								
6	x	0 / 0	A								
7		4 / 4	A	0.243	-0.8	0.973	-0.7	0.083	-1.0	0.341	-0.9
8		0 / 0	B								
9		2 / 2	A	0.137	-2.2			0.0636	-1.7		
10		0 / 0	B								
11		0 / 0	B								
12		0 / 0	A								
13	x	2 / 2	A			1.93	2.5			0.813	3.3
14		4 / 4	A	0.328	0.3	1.24	0.2	0.125	0.5	0.441	0.0
15		0 / 0	B								
16	x	0 / 0	B								
17	x	4 / 4	A	0.251	-0.7	1.19	0.0	0.0823	-1.0	0.441	0.0
18		4 / 4	A	0.313	0.2	1.255	0.2	0.108	-0.1	0.433	-0.1
19	x	4 / 4	A	0.351	0.7	1.54	1.2	0.131	0.7	0.558	1.0
20		4 / 4	A	0.324	0.3	1.196	0.0	0.119	0.3	0.397	-0.4
21		4 / 4	A	0.313	0.2	1.30	0.4	0.115	0.2	0.470	0.2
22		4 / 4	A	0.390	1.2	1.37	0.6	0.153	1.5	0.488	0.4
23		0 / 0	B								
24		0 / 0	B								
25		4 / 4	A	0.478	2.3	1.736	1.8	0.196	3.1	0.637	1.7
26		4 / 4	A	0.200	-1.3	2.00	2.7	0.062	-1.8	0.550	0.9
27		2 / 2	B			0.0759	-3.7	0.0738	-1.3		
28	x	0 / 0	B								
29	x	4 / 4	B	0.315	0.2	1.16	-0.1	0.127	0.6	0.497	0.5
30	x	0 / 0	A								
31		1 / 1	B							0.516	0.6
32		0 / 0	B								
33	x	4 / 4	A	0.267	-0.5	1.12	-0.3	0.093	-0.6	0.386	-0.5
34		2 / 2	B					0.283	6.3	0.602	1.4
35	x	4 / 4	A	0.341	0.5	1.44	0.8	0.119	0.3	0.437	-0.1
37		0 / 0	B								
38		0 / 0	B								
39		4 / 4	A	0.404	1.4	2.58	4.6	0.158	1.7	1.50	9.5
40		0 / 0	B								
41		4 / 4	A	0.316	0.2	0.754	-1.5	0.134	0.9	0.260	-1.7
42		4 / 4	A	0.336	0.5	1.074	-0.4	0.129	0.7	0.403	-0.4
43		0 / 0	B								

* Category A/B classification (Cat A was assigned to laboratories that have correctly analysed at least 12 of 13 compulsory compounds on the Target Pesticides List, corretly detected 7 or more out of the 8 compulsory compounds and that have not reported any false positive result)

Table 4-10 (cont.): Results reported and z-scores achieved by all participating laboratories for ADDITIONAL compounds

ADDITIONAL Compound				Captan (sum)		Folpet (sum)		THPI		Phthalimide	
MRRL [mg/kg]				–		–		–		–	
Assigned Value [mg/kg]				0.302		1.195		0.110		0.446	
*CV**				25.2 %		21.1 %		30.5 %		21.6 %	
Lab code SRM12-	NRL-SRM	Analysed / corr. found max. 8 / 7	Cat.*	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)
44	x	0 / 0	B								
45		0 / 0	A								
46		4 / 4	A	0.282	-0.3	1.114	-0.3	0.104	-0.2	0.390	-0.5
47		4 / 3	A	0.405	1.4	0.260	-3.1	0.172	2.2	FN	-3.9
48		4 / 4	A	0.254	-0.6	1.347	0.5	0.0901	-0.7	0.505	0.5
49	x	4 / 4	A	0.155	-1.9	0.805	-1.3	0.0561	-2.0	0.288	-1.4
50		4 / 4	B	0.263	-0.5	1.236	0.1	0.092	-0.7	0.425	-0.2
51		2 / 2	B					0.128	0.6	0.455	0.1
52		4 / 4	A	0.338	0.5	1.74	1.8	0.101	-0.3	0.436	-0.1
53		4 / 4	A	0.209	-1.2	1.131	-0.2	0.069	-1.5	0.399	-0.4
54		0 / 0	B								
55		0 / 0	B								
56		0 / 0	B								
57		4 / 4	A	0.185	-1.5	1.10	-0.3	0.093	-0.6	0.414	-0.3
58		4 / 4	A	0.565	3.5	2.11	3.1	0.231	4.4	0.911	4.2
59		0 / 0	B								
60		0 / 0	B								
61		4 / 4	B	0.310	0.1	1.33	0.5	0.115	0.2	0.470	0.2
62	x	0 / 0	B								
63		0 / 0	A								
64		4 / 4	B	0.290	-0.2	1.181	0.0	0.113	0.1	0.416	-0.3
65		0 / 0	B								
66	x	4 / 4	A	0.209	-1.2	0.579	-2.1	0.0628	-1.7	0.444	0.0
67		0 / 0	A								
68		0 / 0	B								
69		0 / 0	B								
70		4 / 4	A	0.148	-2.0	0.96	-0.8	0.064	-1.7	0.310	-1.2
71		4 / 4	B	0.158	-1.9	0.828	-1.2	0.0794	-1.1	0.412	-0.3
72		4 / 4	A	0.294	-0.1	1.188	0.0	0.079	-1.1	0.374	-0.6
73	x	4 / 4	B	0.340	0.5	1.15	-0.2	0.105	-0.2	0.389	-0.5
74		4 / 4	A	0.284	-0.2	1.13	-0.2	0.143	1.2	0.561	1.0
75	x	0 / 0	A								
76		4 / 4	A	0.299	0.0	1.247	0.2	0.100	-0.4	0.440	-0.1
77		0 / 0	B								
78		4 / 4	A	0.289	-0.2	1.208	0.0	0.099	-0.4	0.412	-0.3
79		2 / 2	B	0.273	-0.4	1.19	0.0				
80		0 / 0	B								
81	x	4 / 4	B	0.306	0.1	1.09	-0.4	0.107	-0.1	0.518	0.6
82		0 / 0	B								
83	x	0 / 0	A								
84		4 / 4	A	0.289	-0.2	1.146	-0.2	0.105	-0.2	0.410	-0.3
85	x	0 / 0	B								

* Category A/B classification (Cat A was assigned to laboratories that have correctly analysed at least 12 of 13 compulsory compounds on the Target Pesticides List, corretly detected 7 or more out of the 8 compulsory compounds and that have not reported any false positive result)

Table 4-10 (cont.): Results reported and z-scores achieved by all participating laboratories for ADDITIONAL compounds

ADDITIONAL Compound				Captan (sum)		Folpet (sum)		THPI		Phthalimide	
MRRL [mg/kg]				–		–		–		–	
Assigned Value [mg/kg]				0.302		1.195		0.110		0.446	
*CV**				25.2 %		21.1 %		30.5 %		21.6 %	
Lab code SRM12-	NRL-SRM	Analysed / corr. found max. 8 / 7	Cat.*	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)
86		4 / 4	B	0.426	1.6	0.697	-1.7	0.117	0.2	0.231	-1.9
87		0 / 0	B								
88		4 / 4	A	0.279	-0.3	1.27	0.3	0.096	-0.5	0.482	0.3
89		0 / 0	B								
90		4 / 4	A	0.283	-0.2	0.963	-0.8	0.097	-0.5	0.352	-0.8
91	x	0 / 0	B								
92	x	4 / 4	A	0.383	1.1	1.07	-0.4	0.150	1.4	0.349	-0.9
93		0 / 0	B								
94		4 / 4	A	0.291	-0.1	1.27	0.3	0.107	-0.1	0.457	0.1
95		4 / 4	A	0.421	1.6	1.61	1.4	0.137	1.0	0.425	-0.2
96	x	0 / 0	A								
97		4 / 4	A	0.287	-0.2	1.242	0.2	0.101	-0.3	0.406	-0.4
98	x	4 / 4	A	0.256	-0.6	1.44	0.8	0.117	0.2	0.645	1.8
99		4 / 4	A	0.411	1.4	1.31	0.4	0.112	0.1	0.465	0.2
100		0 / 0	B								
101		0 / 0	B								
102		4 / 4	A	0.420	1.6	1.07	-0.4	0.170	2.2	0.440	-0.1
103	x	4 / 4	A	0.331	0.4	1.11	-0.3	0.123	0.5	0.506	0.5
104		4 / 4	A	0.342	0.5	1.26	0.2	0.131	0.7	0.468	0.2
105	x	4 / 4	B	0.330	0.4	1.11	-0.3	0.110	0.0	0.330	-1.0
106		4 / 4	B	0.300	0.0	1.00	-0.7	0.300	6.9	1.00	5.0
107	x	0 / 0	A								
108		0 / 0	B								
109		4 / 4	A	0.301	0.0	1.26	0.2	0.113	0.1	0.451	0.0
110		0 / 0	B								
111		0 / 0	B								
112		0 / 0	B								
113		4 / 4	A	0.288	-0.2	1.21	0.1	0.110	0.0	0.460	0.1
114		0 / 0	B								
115		4 / 4	A	0.183	-1.6	1.43	0.8	0.047	-2.3	0.500	0.5
116		4 / 4	A	0.322	0.3	1.12	-0.3	0.122	0.4	0.431	-0.1
117		0 / 0	B								
118		0 / 0	B								
119		0 / 0	B								
120		0 / 0	B								
121		0 / 0	B								
122		0 / 0	B								
123		0 / 0	B								
124	x	0 / 0	B								
125		4 / 4	A	0.248	-0.7	1.023	-0.6	0.056	-2.0	0.248	-1.8
126		4 / 4	B	0.231	-0.9	0.960	-0.8	0.08	-1.1	0.33	-1.0
127		4 / 4	A	0.446	1.9	1.62	1.4	0.171	2.2	0.612	1.5

* Category A/B classification (Cat A was assigned to laboratories that have correctly analysed at least 12 of 13 compulsory compounds on the Target Pesticides List, corretly detected 7 or more out of the 8 compulsory compounds and that have not reported any false positive result)

Table 4-10 (cont.): Results reported and z-scores achieved by all participating laboratories for ADDITIONAL compounds

ADDITIONAL Compound				Captan (sum)		Folpet (sum)		THPI		Phthalimide	
MRRL [mg/kg]				–		–		–		–	
Assigned Value [mg/kg]				0.302		1.195		0.110		0.446	
CV^*				25.2 %		21.1 %		30.5 %		21.6 %	
Lab code SRM12-	NRL-SRM	Analysed / corr. found max. 8 / 7	Cat.*	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)	Conc. [mg/kg]	z-Score (FFP-RSD = 25 %)
128		0 / 0	B								
129		4 / 4	A	0.506	2.7	1.97	2.6	0.22	4.0	0.851	3.6
130		4 / 4	A	0.288	-0.2	0.960	-0.8	0.101	-0.3	0.285	-1.4
3rd-131		0 / 0	B								
3rd-132		2 / 2	B					0.71	21.7	1.56	10.0
3rd-133		0 / 0	A								
3rd-134		0 / 0	B								
3rd-135		0 / 0	B								
3rd-136		0 / 0	A								
3rd-137		0 / 0	B								
3rd-138		0 / 0	B								
3rd-139		0 / 0	A								

* Category A/B classification (Cat A was assigned to laboratories that have correctly analysed at least 12 of 13 compulsory compounds on the Target Pesticides List, corretly detected 7 or more out of the 8 compulsory compounds and that have not reported any false positive result)

4.4.4 Laboratory Classification Based on Scope

All participating laboratories having reported at least one result were classified into categories A or B according to the rules stated in **Section 2.5 (p. 16)**. Following the rules defined in the General Protocol (7th Edition, see **Appendix 8**), a laboratory had to fulfill the following conditions in order to be classified into Category A in the present PT: a) analysis of at least twelve out of the thirteen compulsory pesticides on the Target Pesticides List; b) correct detection of at least seven out of the eight compulsory pesticides present in the test item, and c) no false positive results.

A total of 64 EU and EFTA laboratories (50 %) were classified into Category A and 65 (50 %) into Category B. Three out of the 9 EU candidate and third-country laboratories were classified into Category A. Considering only the compulsory compounds the laboratories from EU and EFTA countries classified into Category A achieved an overall AAZ of 0.8 (n = 507), whereas those classified into Category B achieved an overall AAZ of 1.0 (n = 256). The AAZ values remain the same, when including laboratories from EU candidate and third countries (at n = 530 for the compulsory compounds and n = 289 for the optional compounds).

Table 4-11 and **Table 4-12 (p. 62)** show the details of laboratories classified into Category A and B, respectively. For informative purposes, the overall AAZ was calculated for laboratories with 5 or more individual z-scores among the compulsory compounds. For the AAZ calculation any z-scores > 5 were set at 5.

Table 4-11: Category A laboratories ordered by lab-codes

COMPULSORY Compounds			2,4-D	Captan (parent)	Chlorotha-lonil	Dithiocar-bamates	Fenbutatin Oxide	Folpet (parent)	Glypho-sate	Haloxyfop	
MRRL [mg/kg]			0.01	0.01	0.01	0.03	0.01	0.01	0.03	0.01	
Assigned Value [mg/kg]			0.079	0.085	0.125	0.267	0.086	0.334	0.306	0.070	
*CV**			13.3 %	28.1 %	25.2 %	22.2 %	21.0 %	25.0 %	20.9 %	13.9 %	
Lab code SRM12-	NRL-SRM	Analysed / corr. found[1)]	z-Scores	z-Scores	z-Scores	z-Scores	z-Scores	z-Scores	z-Scores	z-Scores	AAZ[2)]
1		13 / 8	-0.5	-1.4	-1.3	0.9	-0.4	-1.7	4.2	-0.8	**1.4**
2		13 / 7	-0.5	0.1	-0.2	-0.5	-3.5 FN	0.2	0.1	-0.7	**0.7**
3	x	13 / 8	0.2	-0.9	0.1	-1.1	1.2	-0.5	0.2	0.0	**0.5**
6	x	13 / 7	0.1	1.4	-0.1	-0.4	0.2	-0.2	-3.6 FN	0.6	**0.8**
7		13 / 8	-0.3	-0.3	0.4	0.2	-0.1	-0.6	0.6	0.0	**0.3**
9		13 / 8	-1.0	-3.5	6.1	1.1	-1.1	-1.9	6.2	-0.1	**2.3**
12		13 / 8	-0.2	-0.4	0.8	-1.1	1.1	1.0	-0.1	0.0	**0.6**
13	x	13 / 8	-0.7	2.2	-1.2	0.3	-0.2	-0.5	-0.2	-0.4	**0.7**
14		13 / 8	-0.8	-0.2	-0.3	-0.6	-0.3	0.2	0.1	-0.1	**0.3**
17	x	13 / 8	0.1	0.1	-0.2	0.5	0.2	-0.4	0.3	0.3	**0.3**
18		13 / 8	-0.2	0.6	1.2	0.0	0.9	0.6	-0.1	-0.4	**0.5**
19	x	13 / 8	0.5	0.2	-0.8	-0.3	-0.3	0.9	-0.2	-0.1	**0.4**
20		13 / 8	-0.2	0.0	0.7	0.5	-0.3	0.8	-0.1	0.0	**0.3**
21		13 / 8	0.1	0.0	0.5	-0.8	0.1	0.2	-0.4	0.2	**0.3**
22		13 / 8	0.6	0.2	0.3	1.0	0.3	0.6	0.7	-0.9	**0.6**
25		13 / 8	0.1	0.0	-0.1	-0.8	-0.4	1.4	0.8	0.1	**0.5**
26		13 / 8	0.0	-0.5	0.2	-1.3	0.3	7.4	-0.7	0.1	**1.0**
30	x	13 / 8	-0.1	0.8	-0.1	-1.5	2.3	1.4	-0.1	0.2	**0.8**
33	x	13 / 8	0.1	-0.1	0.2	1.2	0.5	0.1	-0.2	0.3	**0.3**
35	x	13 / 7	0.3	0.9	0.6	8.3	-3.5 FN	2.7	-0.5	0.1	**1.7**
39		13 / 8	0.4	0.2	0.5	0.6	-0.1	0.3	-0.3	-0.6	**0.4**
41		13 / 8	0.1	-1.6	-0.6	-1.9	-0.2	-1.2	0.2	0.1	**0.7**
42		13 / 8	-0.1	-0.2	-0.6	-1.9	-0.3	-0.9	-0.4	0.3	**0.6**
45		13 / 8	-0.5	1.0	1.5	-0.5	-0.3	1.5	-0.1	0.9	**0.8**
46		13 / 8	0.3	-0.5	0.2	-1.0	-0.6	-0.1	-0.9	-0.4	**0.5**
47		13 / 8	-0.1	-1.0	1.4	0.6	1.3	-0.9	0.2	-0.4	**0.7**
48		12 / 7	0.3	-0.5	0.2	0.3		-0.1	-0.9	-0.2	**0.4**
49	x	13 / 8	0.1	-1.9	-1.0	-0.1	-0.3	-1.3	1.2	0.3	**0.8**
52		13 / 8	-0.5	2.5	0.2	3.5	-2.3	6.3	1.1	0.3	**1.9**
53		13 / 8	-0.3	-0.6	-0.2	0.5	1.2	-0.1	-0.3	0.2	**0.4**
57		13 / 8	-0.5	-0.6	-1.0	0.0	0.8	-0.8	13.0	0.0	**1.1**
58		13 / 8	-0.9	1.0	0.6	0.1	-0.8	-0.8	-0.7	0.4	**0.7**
63		12 / 7	0.2	-1.6	1.5	-0.7	6.8	-3.0		-0.2	**1.7**

1) Referring to compulsory compounds only (max. 13/8)
2) AAZ: Average of Absolute z-scores, is given for informative purposes. It was calculated using all z-scores of each laboratory using assigned values based on the entire population.
For the calculation of the AAZ the value "5" was applied where the z-score was higher than 5 (shown in square brackets).
FN = false negative results

Table 4-11 (cont.): Category A laboratories ordered by lab-codes

COMPULSORY Compounds			2,4-D	Captan (parent)	Chlorotha-lonil	Dithiocar-bamates	Fenbutatin Oxide	Folpet (parent)	Glypho-sate	Haloxyfop	
MRRL [mg/kg]			0.01	0.01	0.01	0.03	0.01	0.01	0.03	0.01	
Assigned Value [mg/kg]			0.079	0.085	0.125	0.267	0.086	0.334	0.306	0.070	
*CV**			13.3 %	28.1 %	25.2 %	22.2 %	21.0 %	25.0 %	20.9 %	13.9 %	
Lab code SRM12-	NRL-SRM	Analysed / corr. found [1]	z-Scores	z-Scores	z-Scores	z-Scores	z-Scores	z-Scores	z-Scores	z-Scores	AAZ [2]
66	x	13 / 8	-0.1	0.0	0.9	0.0	-0.3	1.3	-0.3	-0.3	**0.4**
67		13 / 8	1.2	0.5	-0.6	0.3	-0.8	-0.6	2.3	0.2	**0.8**
70		13 / 8	-1.2	-3.1	-0.2	0.0	-1.0	-0.1	-0.3	-0.2	**0.8**
72		13 / 8	0.8	2.5	1.6	0.0	1.0	1.2	0.2	0.2	**0.9**
74		13 / 8	0.2	-0.5	0.2	1.6	0.9	0.1	-1.0	0.4	**0.6**
75	x	13 / 8	2.5	1.7	1.5	3.1	9.5	0.4	-0.2	2.9	**2.2**
76		13 / 8	0.3	0.7	0.2	1.6	-0.6	0.3	-0.5	0.0	**0.5**
78		13 / 8	-0.3	0.3	-0.1	-0.8	1.0	0.5	2.7	0.0	**0.7**
83	x	12 / 7	45.8	-0.2	-3.7		-3.1	1.3	-1.8	42.2	**2.9**
84		13 / 8	-0.2	-0.2	0.8	0.3	0.7	-0.2	-0.3	0.0	**0.3**
88		13 / 8	-0.2	0.1	-0.1	-0.2	-0.3	-0.4	0.1	-0.8	**0.3**
90		13 / 8	0.4	0.2	0.1	4.5	0.4	-1.0	-0.5	0.6	**1.0**
92	x	13 / 8	0.6	0.0	-0.6	0.2	-0.1	0.2	0.5	0.6	**0.4**
94		13 / 8	-0.1	-0.3	0.3	0.4	-0.3	0.1	0.8	0.1	**0.3**
95		13 / 8	0.0	3.0	-0.3	-0.8	-0.6	5.0	0.6	0.3	**1.3**
96	x	13 / 8	-0.4	0.7	-0.7	0.6	-0.3	-2.6	-0.3	-0.4	**0.8**
97		13 / 8	0.4	0.0	1.5	1.2	0.9	1.1	6.1	1.0	**1.4**
98	x	13 / 8	-0.5	-3.0	-2.9	-0.3	-1.4	-2.3	-0.1	-1.1	**1.5**
99		13 / 7	0.0	4.9	-0.3	0.2	-3.5 FN	0.5	-1.4	0.4	**1.4**
102		13 / 7	-0.7	0.2	5.9	-0.4	-1.2	-3.9 FN	-0.2	-2.2	**1.7**
103	x	13 / 8	0.0	0.9	0.4	-0.1	0.4	1.3	-0.1	0.1	**0.4**
104		13 / 8	0.6	-0.1	-0.7	0.4	-0.4	-0.1	0.5	0.3	**0.4**
107	x	13 / 8	1.7	-1.0	1.1	-1.8	1.0	-0.3	-1.2	0.9	**1.1**
109		13 / 8	-0.2	-0.4	0.6	0.5	0.0	0.2	-0.6	0.0	**0.3**
113		12 / 8	0.3	-1.0	-1.0	1.1	0.1	-0.6	-0.1	0.3	**0.6**
115		13 / 8	-1.0	0.2	0.3	0.2	-0.5	0.2	-0.5	0.0	**0.4**
116		13 / 8	0.2	-0.3	0.2	-0.2	1.0	-1.1	0.6	0.3	**0.5**
125		12 / 7	0.8	2.5	0.4		0.8	2.3	1.8	0.7	**1.3**
127		13 / 8	-0.3	0.9	0.9	1.0	-0.4	0.5	0.8	-0.1	**0.6**
129		12 / 7	0.4	-0.8	-0.8	-1.9	-0.6	-0.9		-0.2	**0.8**
130		13 / 8	1.1	0.2	-0.6	-1.3	1.7	0.7	0.8	1.0	**0.9**
3rd-133		12 / 7	0.1	0.0	0.2		0.2	0.2	-0.1	0.2	**0.1**
3rd-136		13 / 8	-0.1	1.2	-2.7	-0.5	0.2	4.0	0.1	-0.3	**1.1**
3rd-139		13 / 8	6.6	-0.4	-0.2	3.1	7.6	-2.9	0.1	-3.1	**2.5**

1) Referring to compulsory compounds only (max. 13/8)
2) AAZ: Average of Absolute z-scores, is given for informative purposes. It was calculated using all z-scores of each laboratory using assigned values based on the entire population.
For the calculation of the AAZ the value "5" was applied where the z-score was higher than 5 (shown in square brackets).
FN = false negative results

Table 4-12: Category B laboratories ordered by lab-codes

COMPULSORY Compounds			2,4-D	Captan (parent)	Chlorotha-lonil	Dithiocar-bamates	Fenbutatin Oxide	Folpet (parent)	Glypho-sate	Haloxyfop	
Assigned Value [mg/kg]			0.01	0.01	0.01	0.03	0.01	0.01	0.03	0.01	
MRRL [mg/kg]			0.079	0.085	0.125	0.267	0.086	0.334	0.306	0.070	
*CV**			13.3 %	28.1 %	25.2 %	22.2 %	21.0 %	25.0 %	20.9 %	13.9 %	
Lab code SRM12-	NRL-SRM	Analysed / corr. found [1)]	z-Scores	z-Scores	z-Scores	z-Scores	z-Scores	z-Scores		z-Scores	AAZ [2)]
4		11 / 7	-0.2	-0.6	-0.9	0.9	-1.3	-0.5		-0.3	0.7
5		1 / 1				-0.3					
8		9 / 4	0.5			-0.6			1.1	0.7	
10		9 / 5	-0.6		5.3	1.7			0.1	-0.2	1.5
11		6 / 3	-1.8		11.8					-1.7	
15		10 / 6	-0.2	-0.2	0.7	0.2		-0.5		-0.6	0.4
16	x	11 / 6	-1.2		0.7	-0.9	0.3		-0.9	-0.4	0.7
23		10 / 5		-2.8	-3.0			-3.5	-1.6	-1.7	2.5
24		11 / 7	0.2	-1.1	-0.2	0.7	-0.8	0.0		-0.3	0.5
27		6 / 2	-0.1		-3.7 FN			-3.1			
28	x	10 / 6	-0.4		-0.6	0.6	1.6		-0.6	-0.5	0.7
29	x	10 / 6	0.0	-1.1	-0.6		0.1	-2.1		-0.1	0.7
31		4 / 4		0.3	-0.8	0.3		-0.2			
32		11 / 7	0.1	0.4	-0.8	0.4		0.5	-0.1	0.1	0.3
34		11 / 6	-0.1		0.0	0.8	0.0		0.1	-0.7	0.3
37		6 / 4		-0.1	-0.2	-0.1		-0.2			
38		8 / 6	-0.3	-1.0	-0.5	-1.0		-0.2	-2.3		0.9
40		9 / 5			1.1	-0.3	-0.1	3.4	-1.4		1.3
43		2 / 2				-1.0			-0.8		
44	x	8 / 4	-3.5 FN	0.0	-1.9	0.8		-0.4		-3.4 FN	1.7
50		11 / 7	0.3	-0.2	2.1	0.7	0.1	0.5		0.0	0.6
51		8 / 4	-0.3		3.3		0.9			-0.3	
54		6 / 3			-1.8			-0.8		0.8	
55		2 / 1				-0.6					
56		7 / 4		-1.4	-0.6	-0.3		-0.4			
59		1 / 1				-0.2					
60		3 / 2				0.2			0.1		
61		6 / 4	-0.2	-0.2				0.6		0.0	
62	x	9 / 6	-0.4	-1.1	-1.2	-0.9		0.5	-0.7	-4.0 FN	1.3
64		10 / 6	0.2		0.8	-0.2		0.2	1.5	-0.2	0.5
65		1 / 1				-0.1					
68		8 / 4	0.1			0.2			-3.3	0.1	
69		10 / 6	0.5	0.0	-0.8	0.6		0.3		0.2	0.4
71		11 / 6	36.2		-0.8	-0.3	-0.8		0.3	38.6	2.0
73	x	8 / 5		2.2	0.0	1.1		0.4	0.7		0.9

1) Referring to compulsory compounds only (max. 13/7)

2) AAZ: Average of Absolute z-scores, is given for informative purposes for participants having reported at least 5 results for compulsory analytes. It was calculated using all z-scores of each lab using assigned values based on the entire population.
For the calculation of the AAZ the value "5" was applied where the z-score was higher than 5 (shown in square brackets).

FN = false negative results

Table 4-12 (cont.): Category B laboratories ordered by lab-codes

COMPULSORY Compounds			2,4-D	Captan (parent)	Chlorotha-lonil	Dithiocar-bamates	Fenbutatin Oxide	Folpet (parent)	Glypho-sate	Haloxyfop	
Assigned Value [mg/kg]			0.01	0.01	0.01	0.03	0.01	0.01	0.03	0.01	
MRRL [mg/kg]			0.079	0.085	0.125	0.267	0.086	0.334	0.306	0.070	
*CV**			13.3 %	28.1 %	25.2 %	22.2 %	21.0 %	25.0 %	20.9 %	13.9 %	
Lab code SRM12-	NRL-SRM	Analysed / corr. found [1]	z-Scores	z-Scores	z-Scores	z-Scores	z-Scores	z-Scores		z-Scores	AAZ [2]
77		6 / 5		-0.2	-0.6	-1.1	-0.4	-0.5			0.6
79		11 / 6	-0.4		-1.2	-2.6	-0.5		-0.5	-0.6	1.0
80		1 / 1			-2.4						
81	x	11 / 6	-0.1		0.0	-0.2	-0.8		-0.5	-0.2	0.3
82		1 / 1				-0.9					
85	x	3 / 1			2.8						
86		11 / 7	0.2	5.1	-0.9	2.3		-1.2	2.0	-0.2	1.7
87		1 / 1				-0.5					
89		7 / 4	1.2		0.7		-0.4		0.2		
91	x	11 / 7	2.1	0.1	0.5	0.2	-0.3	0.3		-0.9	0.6
93		10 / 6	-0.3	-1.8	-0.5		0.0	-0.6		-0.9	0.7
100		1 / 1				0.9					
101		1 / 1			-1.4						
105	x	6 / 4		1.7	1.1	-0.1		1.4			
106		13 / 4	1.1	-3.5 FN	-3.7 FN	-1.3	-3.5 FN	-3.9 FN	0.6	2.8	2.6
108		11 / 7	0.4	17.2	-0.4	0.0		0.2	-1.5	-1.3	1.3
110		3 / 3		10.7	1.3			10.7			
111		1 / 1				-0.4					
112		3 / 3		-2.0	-2.8			-2.4			
114		1 / 1				0.3					
117		9 / 5	0.7	1.9	-0.7			-0.9		1.4	1.1
118		5 / 4			-1.3	-1.8		-0.5		3.6	
119		1 / 1				0.0					
120		5 / 2		-3.5 FN	-0.5			-0.1			
121		3 / 1			-0.1						
122		3 / 2				0.1			0.5		
123		2 / 1					-0.3				
124	x	9 / 6	-0.1	0.5	0.2	-0.7		-0.1		0.1	0.3
126		9 / 7	-1.0	-0.6	0.2		-2.3	-0.5	-0.2	-1.3	0.9
128		1 / 1	-0.7								
3rd-131		7 / 4	0.3		0.3	-0.5			-1.3		
3rd-132		11 / 7	0.2	0.2	-1.8	0.3		0.7	-2.7	-2.2	1.2
3rd-134		3 / 3			-0.5	0.0	0.2				
3rd-135		4 / 2			-1.1	25.4					
3rd-137		4 / 3	0.4		5.1				-1.7		
3rd-138		6 / 5		8.5	-1.0	0.6	1.1	4.9			2.5

1) Referring to compulsory compounds only (max. 13/7)
2) AAZ: Average of Absolute z-scores, is given for informative purposes for participants having reported at least 5 results for compulsory analytes. It was calculated using all z-scores of each lab using assigned values based on the entire population.
For the calculation of the AAZ the value "5" was applied where the z-score was higher than 5 (shown in square brackets).
FN = false negative results

4.4.5 Laboratory Feedback in Case of Poor Results

As a follow-up measure to this EUPT, all participating laboratories having achieved questionable ($2 < |z\text{-score}| < 3$) or unacceptable ($|z\text{-score}| \geq 3$) results were asked to investigate the reasons for their poor performance and to report them to the organisers. The aim of this measure is to sensibilize the laboratories to investigate the sources of errors. A compilation of the feedback received by the laboratories is given in **Appendix 7**. With this compilation it is intended to make all participanting labs aware of common and potential error sources so that they can be avoided or eliminated in the future. This information also provides input to NRLs on how to better assist OfLs within the network in improving their performance.

In the current PT, excluding ***carbofuran (part of sum)*** that showed an unacceptble uncertainty of the assigned value, in total 744 results were reported by 147 participants. 161 results by 73 laboratories were allocated with $|z| > 2$, and thereof 102 results by 53 laboratories with $|z| \geq 3$ (see **Table 4-7, p. 40**). Among EU and EFTA laboratories, $|z| > 2$ was assigned to 143 results by 66 laboratories, and $|z| \geq 3$ to 88 results by 46 laboratories. All these laboratories were asked to provide a feedback. Overall, 57 laboratories responded to the organisers with (possible) reasons for their poor performance in 123 cases. In 20 of those cases the real reasons for generating biased results could not be clarified, inspite of intensive investigation. The most frequently reported error sources were "error in the concentration of analytical standards or calibration solutions" (24 cases), "lack of experience" (14 cases) and "transcription or administrative errors" (14 cases). Compared to the previous PTs the rate of "transcription or administrative errors" was high in the current PT (e.g., only 1 case in EUPT-SRM10 and EUPT-SRM11).

Other error sources commonly reported were: "Use of inappropriate procedure" (9 cases), "error in the evaluation or in the interpretation of measurement data" (9 cases), "application of inappropriate calibration" (9 cases), "technical problems with measurement instrumentation" like poor sensitivity (8 cases), "matrix effect not properly compensated" (6 cases) and "result not corrected for low or high recovery" (5 cases). Concerning ***captan (parent)*** and ***THPI*** as well as ***folpet (parent)*** and ***phthalimide***, in 4 cases the laboratories blamed the conversion the parent compounds into the metabolites as a source of bias for the individual compounds with the sum concentration being acceptable.

The other reasons for poor performace were: "procedure not properly conducted" (4 cases), "strong chromatographic interferences" (3 cases), "detection signals strongly interferred by matrix components" and "degradation during sample preparation or measurement" (each 2 cases) as well as "misunderstanding of the definition of the analyte" (1 case). The responses from the other labratories are pending.

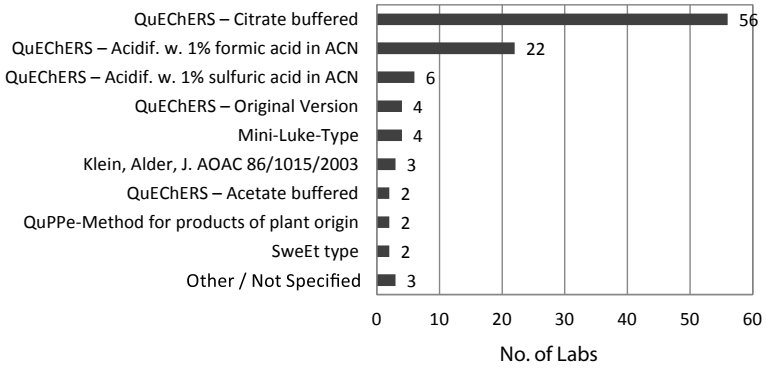
4.5 Methodological Information

4.5.1 Analytical methods used

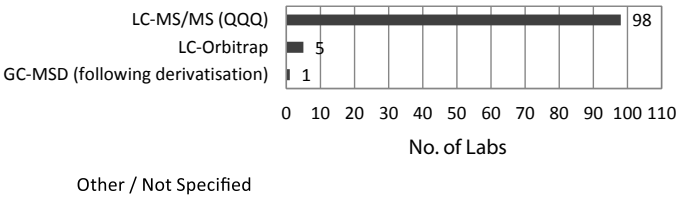
An overview of the methods used by all participating laboratories for sample preparation and determination for each analyte present in the test item can be seen in **Figure 4-1**. Detailed information about the analytical methods used by the laboratories for each of the analytes can be found online under **"EUPT-SRM12 - Supplementary Information"** accessible using the the link: http://www.eurl-pesticides.eu/library/docs/srm/EUPT-SRM12_Supplementary_Information.pdf.

COMPULSORY COMPOUNDS

2,4-D (free acid): Sample preparation

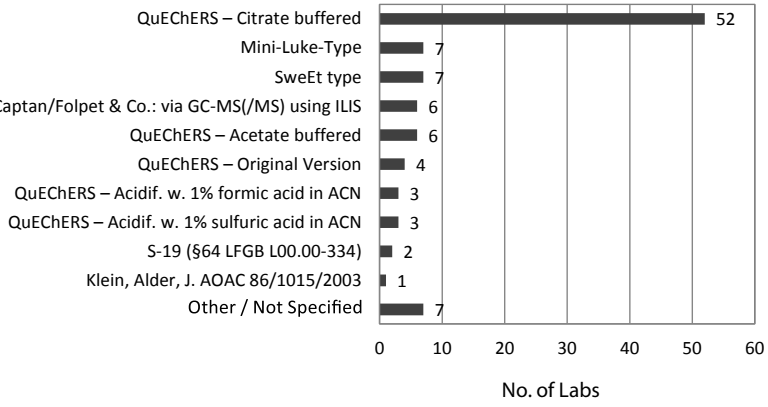


2,4-D (free acid): Determination technique



Other / Not Specified

Captan (parent): Sample preparation



Captan (parent): Determination technique

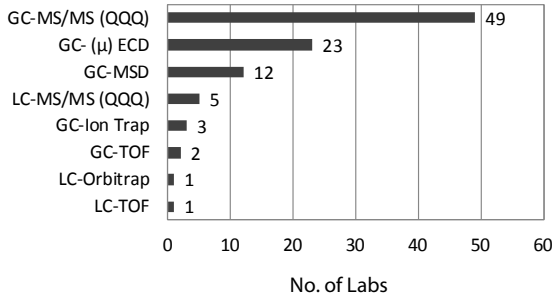
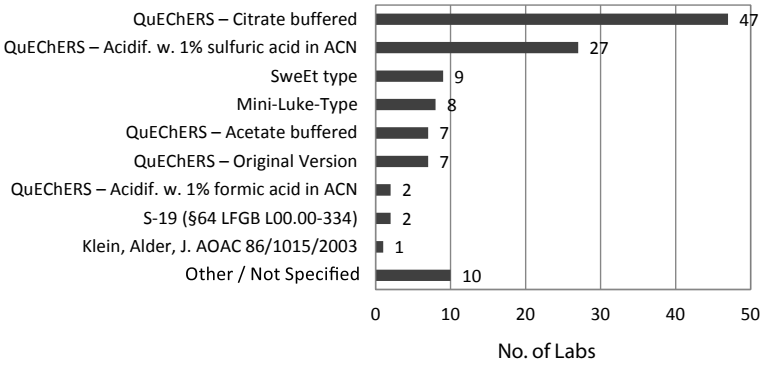


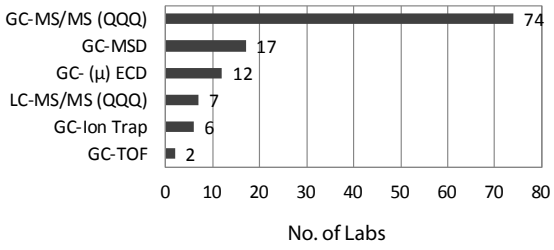
Figure 4-1: Sample preparation and determination techniques applied by laboratories as reported

COMPULSORY COMPOUNDS

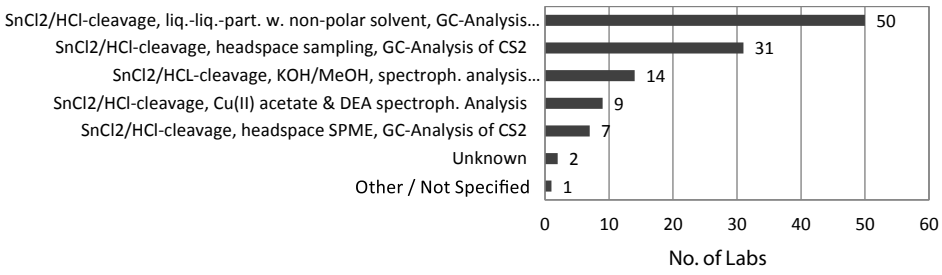
Chlorothalonil: Sample preparation



Chlorothalonil: Determination technique



Dithiocarbamates: Sample preparation



Dithiocarbamates: Determination technique

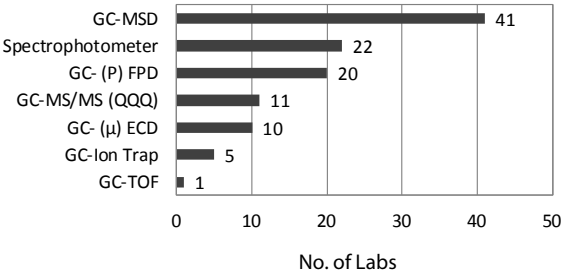
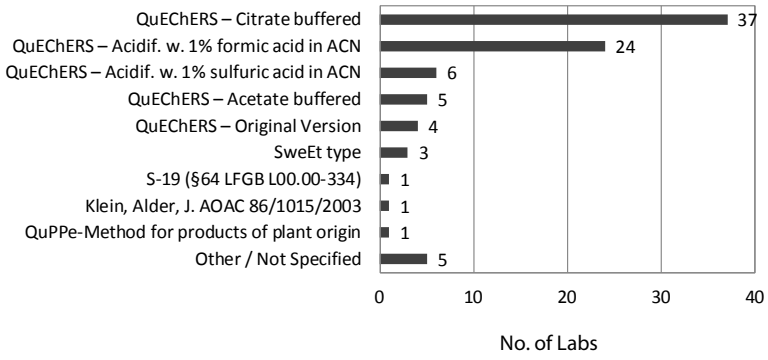


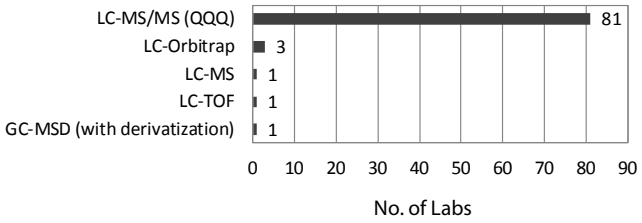
Figure 4-1 (cont.): Sample preparation and determination techniques applied by laboratories as reported

COMPULSORY COMPOUNDS

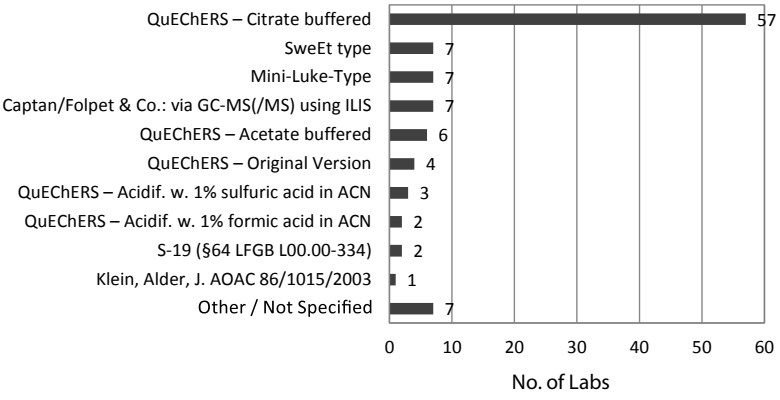
Fenbutatin Oxide: Sample preparation



Fenbutatin Oxide: Determination technique



Folpet (parent): Sample preparation



Folpet (parent): Determination technique

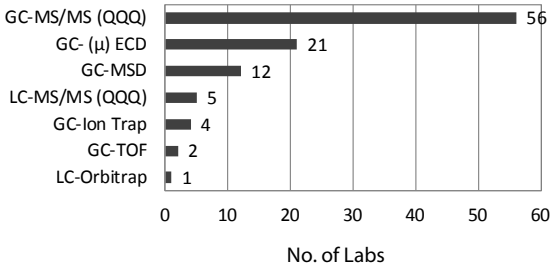
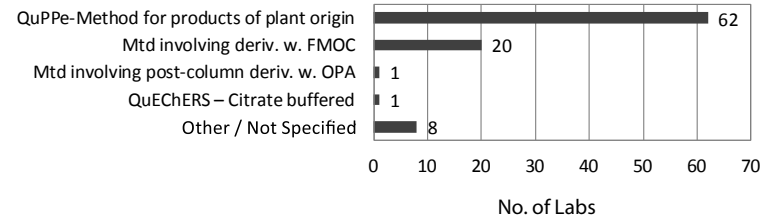


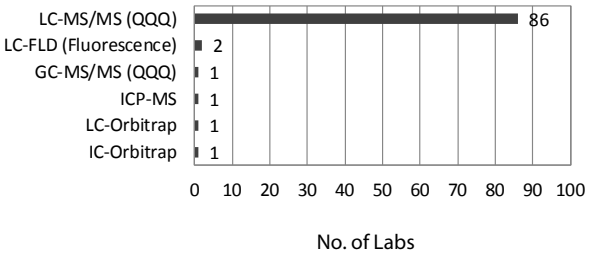
Figure 4-1 (cont.): Sample preparation and determination techniques applied by laboratories as reported

COMPULSORY COMPOUNDS

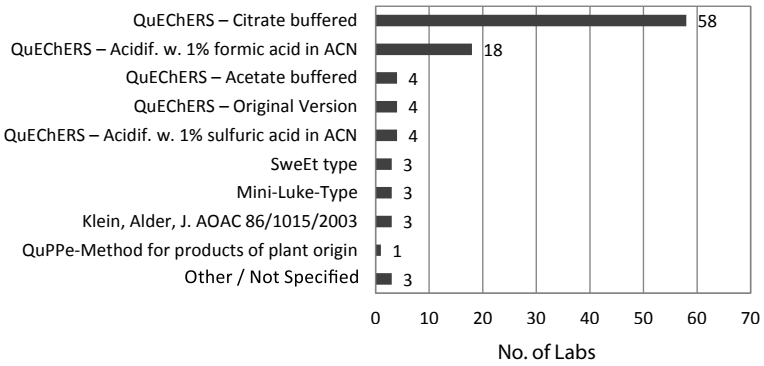
Glyphosate: Sample preparation



Glyphosate: Determination technique



Haloxyfop: Sample preparation



Haloxyfop: Determination technique

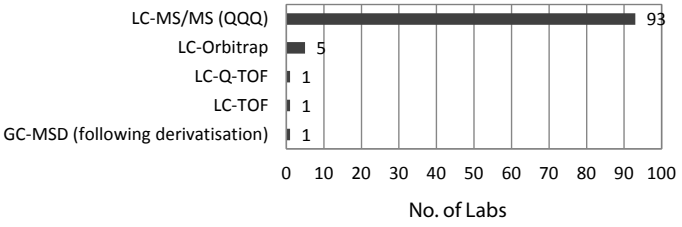
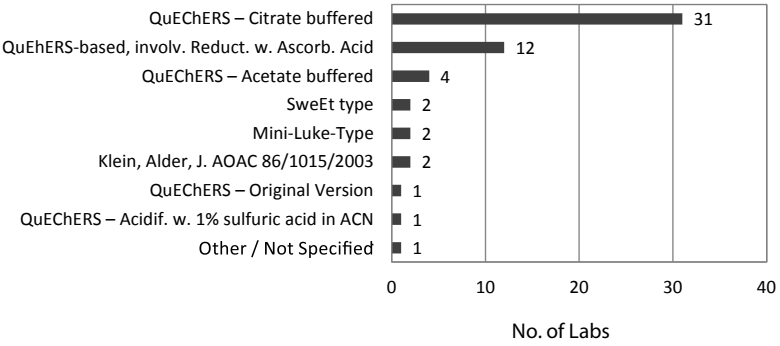


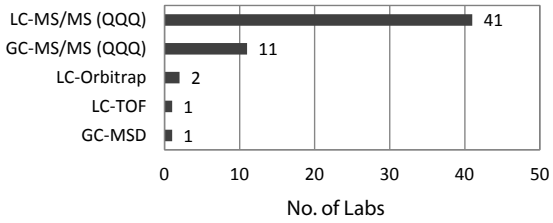
Figure 4-1 (cont.): Sample preparation and determination techniques applied by laboratories as reported

OPTIONAL COMPOUNDS

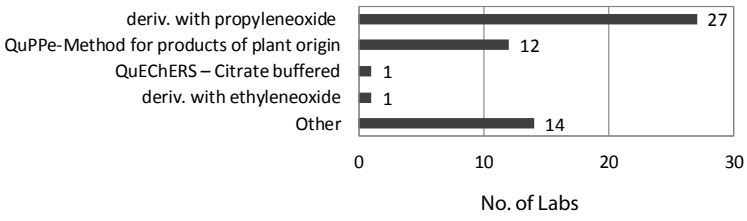
Bifenazate (sum): Sample preparation



Bifenazate (sum): Determination technique



Bromide ion: Sample preparation



Bromide ion: Determination technique

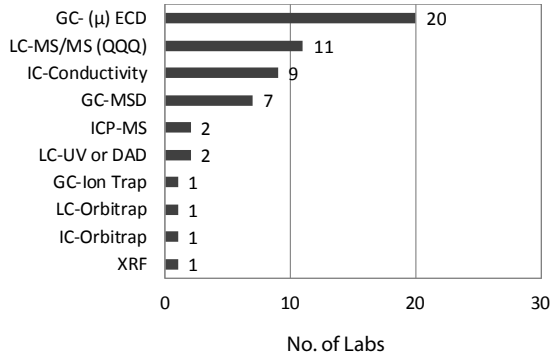
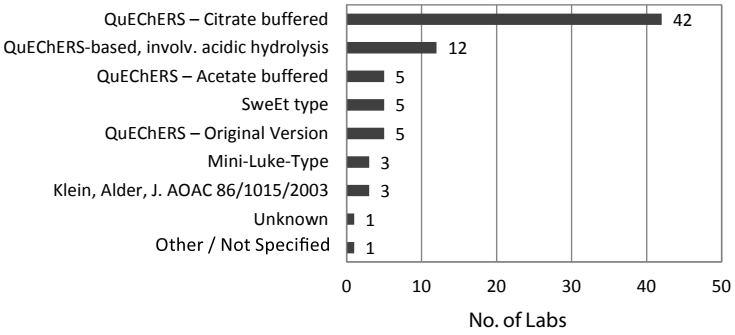


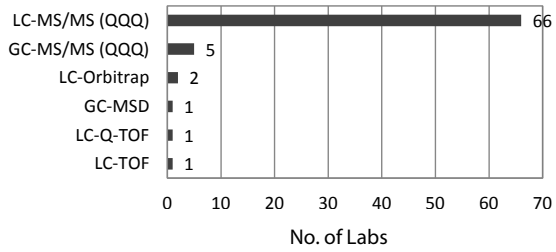
Figure 4-1 (cont.): Sample preparation and determination techniques applied by laboratories as reported

OPTIONAL COMPOUNDS

Carbofuran (part of sum): Sample preparation



Carbofuran (part of sum): Determination technique



Chlorate: Sample preparation

Chlorate: Determination technique

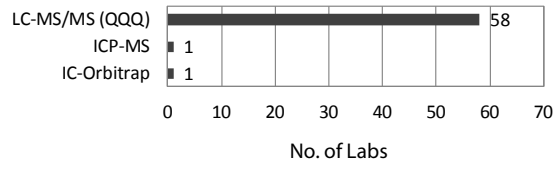
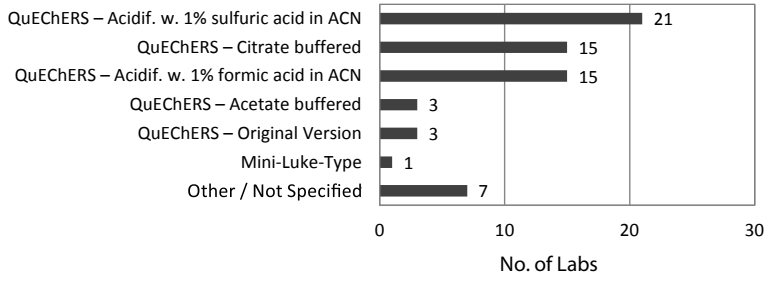


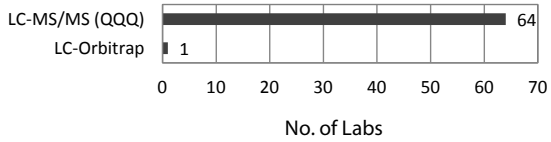
Figure 4-1 (cont.): Sample preparation and determination techniques applied by laboratories as reported

OPTIONAL COMPOUNDS

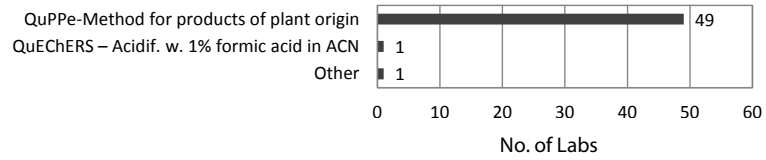
Dithianon: Sample preparation



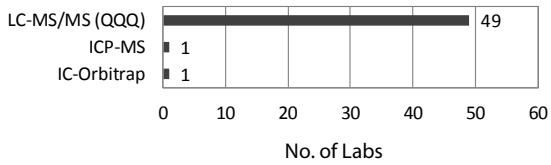
Dithianon: Determination technique



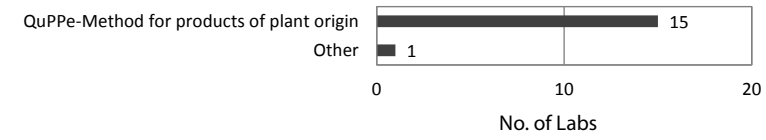
Phosphonic acid: Sample preparation



Phosphonic acid: Determination technique



N-Acetyl glyphosate: Sample preparation



N-Acetyl glyphosate: Determination technique

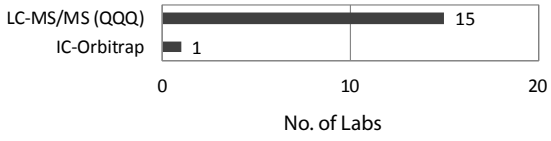
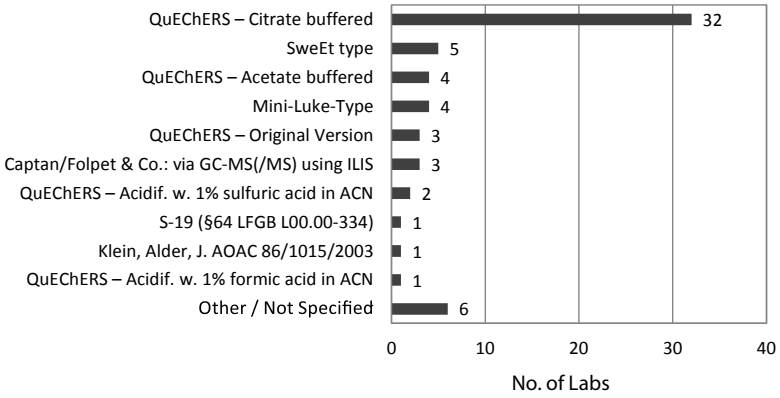


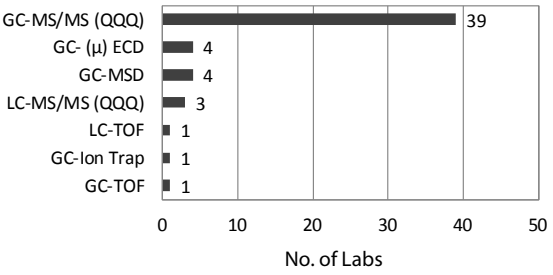
Figure 4-1 (cont.): Sample preparation and determination techniques applied by laboratories as reported

ADDITIONAL COMPOUNDS

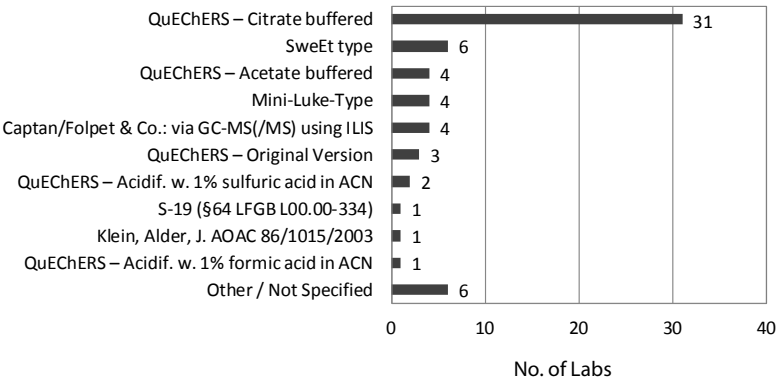
Captan (sum): Sample preparation



Captan (sum): Determination technique



Folpet (sum): Sample preparation



Folpet (sum): Determination technique

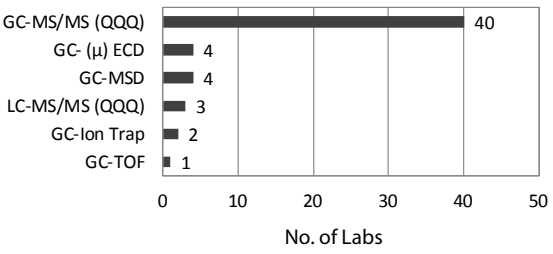
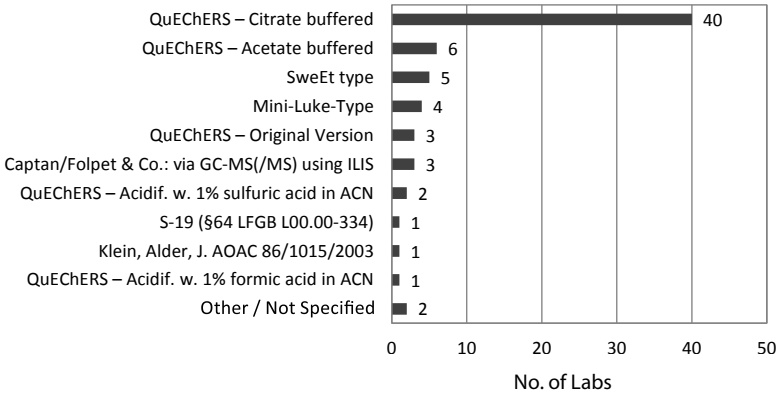


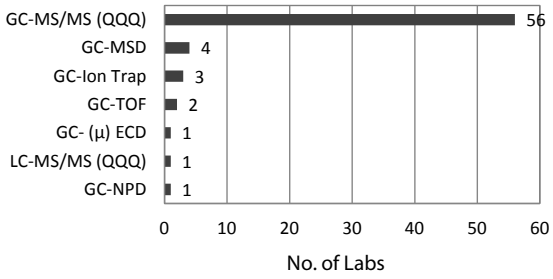
Figure 4-1 (cont.): Sample preparation and determination techniques applied by laboratories as reported

ADDITIONAL COMPOUNDS

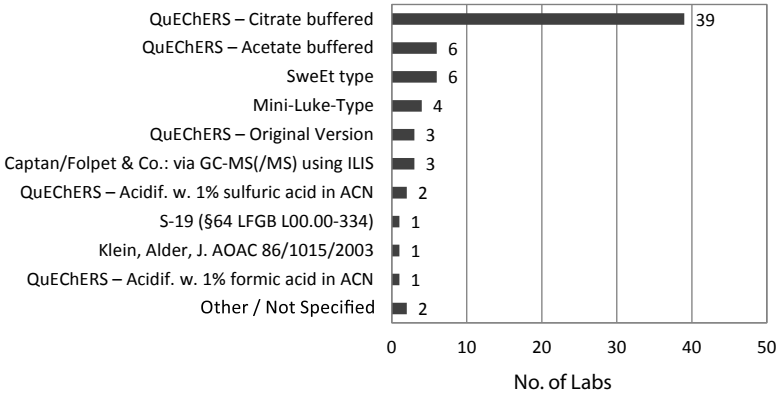
THPI: Sample preparation



THPI: Determination technique



Phthalimide: Sample preparation



Phthalimide: Determination technique

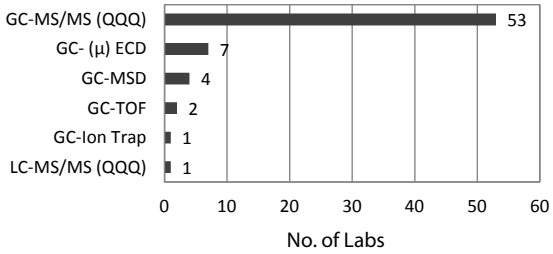


Figure 4-1 (cont.): Sample preparation and determination techniques applied by laboratories as reported

Table 4-13: Calibration approaches and internal standrads employed (aggregation by internal standards)

		COMPULSORY COMPOUNDS									OPTIONAL COMPOUNDS							
Internal Standards (IS) used		2,4-D	Captan (parent)	Chlorothalonil	Dithiocarbamates	Fenbutatin Oxide	Folpet (parent)	Glyphosate	Haloxyfop	Sum of Compulsory Compounds	Bifenazate	Bromide ion	Carbofuran	Chlorate	Dithianon	Phosphonic acid	N-Acetyl glyphosate	Sum of Optional Compounds
Yes, ILISs	**Sum**	**3 (3 %)**	**20 (20 %)**	**– (0 %)**	**3 (3 %)**	**1 (1 %)**	**16 (16 %)**	**55 (60 %)**	**– (0 %)**	**98 (12 %)**	**– (0 %)**	**– (0 %)**	**2 (3 %)**	**38 (63 %)**	**12 (18 %)**	**31 (61 %)**	**2 (13 %)**	**85 (22 %)**
Procedural calibr.			5				3	10 (11 %)		**18 (2 %)**				6 (10 %)		7 (14 %)		**13 (3 %)**
Std add. to sample PORTIONS		1	1				1	1		**4 (0.5 %)**			1	2	2	3	1	**9 (2 %)**
Std add. to extract ALIQUOTS		1	5				5	4		**15 (2 %)**			1	2	1			**4 (1 %)**
MATRIX based: same matrix / other matrix [not specified]			5 / 0 [0]				5 / 0 [1]	19 / 1 [1] (23 %)		**32 (4 %)**				14 / 0 [0] (23 %)	3 / 1 [0]	9 / 0 [0] (18 %)		**27 (7 %)**
Pure SOLVENT based		1	3		3	1	1	17 (18 %)		**26 (3 %)**				13 (22 %)	4	11 (22 %)	1	**29 (8 %)**
No Data on calibration			1					2		**3 (0.4 %)**				1	1	1		**3 (1 %)**
Yes, other IS	**Sum**	**46 (44 %)**	**44 (45 %)**	**72 (60 %)**	**17 (15 %)**	**35 (40 %)**	**50 (49 %)**	**– (0 %)**	**48 (48 %)**	**312 (38 %)**	**30 (54 %)**	**14 (25 %)**	**34 (44 %)**	**7 (12 %)**	**21 (32 %)**	**4 (8 %)**	**3 (19 %)**	**113 (30 %)**
Procedural calibr.		4	2	8	2	4	4		4	**28 (3 %)**	4	5 (9 %)	4		4			**17 (4 %)**
Std add. to sample PORTIONS		2	4	2	1	3	3		3	**18 (2 %)**	3		3		1			**7 (2 %)**
Std add. to extract ALIQUOTS		5	3	6		5	3		6	**28 (3 %)**	2		2		3	1		**8 (2 %)**
MATRIX based: same matrix / other matrix [not specified]		22 / 2 [2] (25 %)	22 / 5 [2] (30 %)	38 / 5 [5] (40 %)	3 / 0 [2]	17 / 2 [1] (23 %)	26 / 5 [1] (31 %)		20 / 3 [2] (25 %)	**185 (23 %)**	13 / 4 [1] (32 %)	1 / 0 [0]	18 / 4 [1] (30 %)	4 / 2 [0] (10 %)	5 / 2 [2] (14 %)	1 / 1 [0]	3 / 0 [0]	**62 (16 %)**
Pure SOLVENT based		9	6	7	8	3	6		9	**48 (6 %)**	3	7 (13 %)	2	1	4	1		**18 (5 %)**
No Data				1	1		2		1	**5 (1 %)**		1						**1 (0 %)**
No	**Sum**	**49 (47 %)**	**27 (28 %)**	**39 (33 %)**	**80 (70 %)**	**43 (49 %)**	**30 (29 %)**	**33 (36 %)**	**47 (47 %)**	**348 (42 %)**	**21 (38 %)**	**35 (64 %)**	**32 (42 %)**	**12 (20 %)**	**25 (38 %)**	**13 (25 %)**	**10 (63 %)**	**148 (39 %)**
Procedural calibr.		2		1	6	4		6	2	**21 (3 %)**	2	3	2	2	3	1	4	**17 (4 %)**
Std add. to sample PORTIONS				2	5		2	2	3	**14 (2 %)**	1	3		1	1	2		**8 (2 %)**
Std add. to extract ALIQUOTS		3	2	3		4	2	3	3	**20 (2 %)**	3	1	3	3	2	4		**16 (4 %)**
MATRIX based: same matrix / other matrix [not specified]		37 / 2 [2] (39 %)	18 / 1 [4] (23 %)	25 / 3 [3] (26 %)	13 / 2 [0] (13 %)	25 / 2 [1] (32 %)	19 / 1 [3] (22 %)	18 / 0 [1] (21 %)	33 / 2 [1] (36 %)	**216 (26 %)**	9 / 1 [2] (21 %)	7 / 0 [1] (15 %)	19 / 2 [2] (30 %)	5 / 0 [1] (10 %)	19 / 0 [0] (29 %)	4 / 0 [1] (10 %)	4 / 0 [1] (31 %)	**78 (21 %)**
Pure SOLVENT based		2	2	1	51 (45 %)	7	3	1	2	**69 (8 %)**	3	18 (33 %)	4			1	1	**27 (7 %)**
Other		1			1			1	1	**4 (0.5 %)**		1						**1 (0.3 %)**
No Data				1	2			1		**4 (0.5 %)**		1						**1 (0.3 %)**
No data on IS	**Sum**	**6 (6 %)**	**7 (7 %)**	**9 (8 %)**	**14 (12 %)**	**8 (9 %)**	**7 (7 %)**	**4 (4 %)**	**6 (6 %)**	**61 (7 %)**	**5 (9 %)**	**6 (11 %)**	**9 (12 %)**	**3 (5 %)**	**7 (11 %)**	**3 (6 %)**	**1 (6 %)**	**34 (9 %)**
Procedural calibr.		2	1	1	1	1	1		2	**9 (1 %)**	1	1	2		2			**6 (2 %)**
Std add. to sample PORTIONS		2	3	3		2	3	1	2	**16 (2 %)**	2	2	2	2	2	2	1	**13 (3 %)**
MATRIX based: same matrix / other matrix [not specified]		[1]	[2]	3 / 0 [1]	1 / 0 [2]	2 / 0 [1]	2 / 0 [0]	1 / 0 [0]	1 / 0 [0]	**17 (2 %)**	1 / 0 [0]	0 / 0 [1]	2 / 0 [1]	1 / 0 [0]	2 / 0 [1]	1 / 0 [0]		**10 (3 %)**
Pure SOLVENT based		1	1	1	7	1	1	2	1	**15 (2 %)**	1	2	1					**4 (1 %)**
Other																		
No Data					3	1				**4 (0.5 %)**			1					**1 (0.3 %)**
Overall		**104**	**98**	**120**	**114**	**87**	**103**	**92**	**101**	**819**	**56**	**55**	**77**	**60**	**65**	**51**	**16**	**380**
Corrected for matrix effects		**83 (80 %)**	**75 (77 %)**	**92 (77 %)**	**35 (31 %)**	**68 (78 %)**	**81 (79 %)**	**86 (93 %)**	**79 (78 %)**	**599 (73 %)**	**41 (73 %)**	**23 (42 %)**	**59 (77 %)**	**56 (93 %)**	**50 (77 %)**	**47 (92 %)**	**14 (88 %)**	**290 (76 %)**

Percentages in parentheses for each of the compounds based on the number of laboratories analysed for these compounds; percentages in parentheses for each group of compounds based on the sum of total number of results in each group

		ADDITIONAL COMPOUNDS					SUM
Internal Standards (IS) used		Captan (sum)	Folpet (sum)	THPI	Phthalimide	Sum of Additional Compounds	
Yes, ILISs	**Sum**	**9 (14 %)**	**8 (12 %)**	**5 (7 %)**	**3 (4 %)**	**25 (9 %)**	**208 (14 %)**
Procedural calibr.		1				**1 (0.4 %)**	**32 (2 %)**
Std add. to sample PORTIONS		1	1			**2 (1 %)**	**15 (1 %)**
Std add. to extract ALIQUOTS		2	2	3	2	**9 (3 %)**	**28 (2 %)**
MATRIX based: same matrix / other matrix [not specified]		4 / 0 [0]	3 / 0 [1]	2 / 0 [0]	0 / 0 [1]	**11 (4 %)**	**70 (5 %)**
Pure SOLVENT based		1	1			**2 (1 %)**	**57 (4 %)**
No Data on calibration							**6 (0 %)**
Yes, other IS	**Sum**	**28 (43 %)**	**29 (44 %)**	**42 (62 %)**	**44 (65 %)**	**143 (54 %)**	**568 (39 %)**
Procedural calibr.		3	4	6	6	**19 (7 %)**	**64 (4 %)**
Std add. to sample PORTIONS		1	1	3	3	**8 (3 %)**	**33 (2 %)**
Std add. to extract ALIQUOTS				2	3	**5 (2 %)**	**41 (3 %)**
MATRIX based: same matrix / other matrix [not specified]		17 / 4 [2] (35 %)	17 / 4 [1] (33 %)	18 / 5 [2] (37 %)	20 / 5 [1] (38 %)	**96 (36 %)**	**343 (23 %)**
Pure SOLVENT based		1	2	5	5	**13 (5 %)**	**79 (5 %)**
No Data				1	1	**2 (1 %)**	**8 (1 %)**
No	**Sum**	**10 (15 %)**	**11 (17 %)**	**18 (26 %)**	**17 (25 %)**	**56 (21 %)**	**552 (38 %)**
Procedural calibr.							**38 (3 %)**
Std add. to sample PORTIONS				1	1	**2 (1 %)**	**24 (2 %)**
Std add. to extract ALIQUOTS				2	2	**4 (1 %)**	**40 (3 %)**
MATRIX based: same matrix / other matrix [not specified]		8 / 0 [1] (14 %)	9 / 0 [1] (15 %)	14 / 0 [1] (22 %)	12 / 0 [1] (19 %)	**47 (18 %)**	**341 (23 %)**
Pure SOLVENT based					1	**1 (0.4 %)**	**97 (7 %)**
Other							**5 (0 %)**
No Data		1	1			**2 (1 %)**	**7 (0 %)**
No data on IS	**Sum**	**18 (28 %)**	**18 (27 %)**	**3 (4 %)**	**4 (6 %)**	**43 (16 %)**	**138 (9 %)**
Procedural calibr.		2	2			**4 (1 %)**	**19 (1 %)**
Std add. to sample PORTIONS		1	1	1	1	**4 (1 %)**	**33 (2 %)**
MATRIX based: same matrix / other matrix [not specified]		3 / 0 [1]	3 / 0 [1]	1 / 0 [1]	1 / 0 [1]	**12 (4 %)**	**39 (3 %)**
Pure SOLVENT based					1	**1 (0.4 %)**	**20 (1 %)**
Other		1	1			**2 (1 %)**	**2 (0 %)**
No Data		10	10			**20 (7 %)**	**25 (2 %)**
Overall		**65**	**66**	**68**	**68**	**267**	**1466**
Corrected for matrix effects		**44 (68 %)**	**45 (68 %)**	**53 (78 %)**	**52 (76 %)**	**194 (73 %)**	**1083 (74 %)**

4.5.2 Calibration Approaches and Use of Internal Standards

Internal standards (ISs) are typically employed to correct for recovery, volume deviations and/or to compensate for the influence of matrix on measurement or derivatisation. **Table 4-13** and **Table 4-14** give an overview of the use or non-use of ISs as well as on the calibration approaches employed by the participants within this PT.

The use of isotopically labelled internal standards (ILISs), especially if applied already to the sample portion at the beginning of the procedure, is the most effective way to compensate for errors during sample preparation and measurement. In the present proficiency test ILISs were used by the participants in 208 cases (which corresponds to appoximately 14 % of the results overall). In 181 (87 %) out of the 208 cases where ILISs were used, these were added at the beginning or an intermediate stage of the procedure. In 11 % of the cases the ILISs were only added to the final extract (**Table 4-15, p. 76**). If added to the final extracts, ILISs will not correct for losses during extraction and cleanup, but will still correct for matrix effects during measurement that are in many cases the main source of bias and variability.

ILISs were most frequently used for ***chlorate*** (63 % of the laboratories submitting results for this compound), ***phosphonic acid*** (61 %) and ***glyphosate*** (60 %) (**Table 4-13**). In the case of ***chlorate*** and ***phosphonic acid*** ILISs were provided by the organisers. In the case of ***captan (parent and sum)***, ***folpet (parent and sum)***, ***dithianon*** and ***N-acetyl glyphosate*** the ILISs of the corresponding compounds were used by 10 – 20 % of the participating laboratories. For the following analytes ILISs were used only occasionally: ***2,4-D*** (3 laboratories), ***fenbutatin oxide*** (1 laboratory), ***carbofuran*** (2 laboratories), ***THPI*** (5 laboratories) and ***phthalimide*** (3 laboratories) and ***dithiocarbamates*** (3 laboratories using $^{13}CS_2$ as ILIS). No ILISs were used for ***haloxyfop***, ***chlorothalonil***, ***bifenazate*** and ***bromide ion***.

Table 4-14: Calibration approaches employed for the analysis (aggregated by type of calibration)

	COMPULSORY COMPOUNDS									OPTIONAL COMPOUNDS							
Calibration Type	2,4-D	Captan (parent)	Chlorothalonil	Dithiocarbamates	Fenbutatin Oxide	Folpet (parent)	Glyphosate	Haloxyfop	Sum of Compulsory Compounds	Bifenazate	Bromide ion	Carbofuran	Chlorate	Dithianon	Phosphonic acid	N-Acetyl glyphosate	Sum of Optional Compounds
Procedural calibr.	8	8	10	9	9	8	16 (17 %)	8	**76 (9 %)**	7	9 (16 %)	8	8	9	8 (16 %)	4 (25 %)	**53 (14 %)**
Std add. to sample PORTIONS	5	8	7	6	5	9	4	8	**52 (6 %)**	6	5	6	5	6	7 (14 %)	2	**37 (10 %)**
Std add. to extract ALIQUOTS	9	10 (10 %)	9		9 (10 %)	10 (10 %)	7	9	**63 (8 %)**	5	1	6	5	6	5		**28 (7 %)**
MATRIX based	68 (65 %)	59 (60 %)	83 (69 %)	23 (20 %)	51 (59 %)	63 (61 %)	41 (45 %)	62 (61 %)	**450 (55 %)**	31 (55 %)	10	49 (64 %)	27 (45 %)	35 (54 %)	17 (33 %)	8 (50 %)	**177 (47 %)**
Matrix-Matched (= Strawberry as Matrix)	60 (58 %)	47 (48 %)	66 (55 %)	17 (15 %)	44 (51 %)	52 (50 %)	38 (41 %)	54 (53 %)	**378 (46 %)**	23 (41 %)	8 (15 %)	39 (51 %)	24 (40 %)	29 (45 %)	15 (29 %)	7 (44 %)	**145 (38 %)**
Other Matrix	8 (8 %)	12 (12 %)	17 (14 %)	6 (5 %)	7 (8 %)	11 (11 %)	3 (3 %)	8 (8 %)	**72 (9 %)**	8 (14 %)	2 (4 %)	10 (13 %)	3 (5 %)	6 (9 %)	2 (4 %)	1 (6 %)	**32 (8 %)**
Pure SOLVENT based	13	12	9	69 (61 %)	12	11	20 (22 %)	12	**158 (19 %)**	7	27 (49 %)	7	14	8	13	2	**78 (21 %)**
Other	1			1			1	1	**4 (0 %)**		1						**1 (0 %)**
No Data on Calibration		1	2	6	1	2	3	1	**16 (2 %)**		2	1	1	1	1		**6 (2 %)**
Overall	**104**	**98**	**120**	**114**	**87**	**103**	**92**	**101**	**819**	**56**	**55**	**77**	**60**	**65**	**51**	**16**	**380**
The percentages in brackets refer to the overall population stated at the bottom of the table																	

Table 4-15: Stages of the procedure at which the internal standards were used in the EUPT-SRM12 for the analysis

		COMPULSORY COMPOUNDS									OPTIONAL COMPOUNDS			
Q: was IS used?		2,4-D	Captan (parent)	Chlorothalonil	Dithiocarbamates	Fenbutatin Oxide	Folpet (parent)	Glyphosate	Haloxyfop	Overall (Compulsory Compounds)	Bifenazate	Bromide ion	Carbofuran	Chlorate
ISs were added to...														
Yes, ILISs	**Sum**	**3**	**20**	**–**	**3**	**1**	**16**	**55**	**–**	**98**	**–**	**–**	**2**	**38**
1) at the beginning of procedure		3 (100 %)	16 (80 %)	–	3 (100 %)	1 (100 %)	12 (75 %)	47 (85 %)	–	**82 (84 %)**	–	–	2 (100 %)	**34 (89 %)**
2) at an intermediate stage (between 1 and 3)		–	–	–	–	–	–	2 (4 %)	–	**2 (2 %)**	–	–	–	**–**
3) to an aliquot of the final extract		–	4 (20 %)	–	–	–	4 (25 %)	4 (7 %)	–	**12 (12 %)**	–	–	–	**3 (8 %)**
No Data		–	–	–	–	–	–	2 (4 %)	–	**2 (2 %)**	–	–	–	**1 (3 %)**
Yes, other ISs	**Sum**	**46**	**44**	**72**	**17**	**35**	**50**	**– (0 %)**	**48**	**312**	**30**	**14**	**34**	**7**
1) at the beginning of procedure		37 (80 %)	29 (66 %)	55 (76 %)	13 (76 %)	28 (80 %)	35 (70 %)	–	38 (79 %)	**235 (75 %)**	24 (80 %)	7 (50 %)	23 (68 %)	**6 (86 %)**
2) at an intermediate stage (between 1 and 3)		–	3 (7 %)	3 (4 %)	2 (12 %)	–	3 (6 %)	–	–	**11 (4 %)**	–	2 (14 %)	–	**–**
3) to an aliquot of the final extract		9 (20 %)	12 (27 %)	14 (19 %)	2 (12 %)	7 (20 %)	12 (24 %)	–	10 (21 %)	**66 (21 %)**	6 (20 %)	5 (36 %)	11 (32 %)	**1 (14 %)**
No	**Sum**	**48 + 1***	**26 + 1***	**39**	**79 + 1***	**42 + 1***	**30**	**33**	**46 + 1***	**343 + 5***	**21**	**35**	**32**	**12**
No data	**Sum**	**6**	**7**	**9**	**14**	**8**	**7**	**4**	**6**	**61**	**5**	**6**	**9**	**3**
Overall		**104**	**98**	**120**	**114**	**87**	**103**	**92**	**101**	**819**	**56**	**55**	**77**	**60**
Percentages in parentheses for each of the compounds refer to the respective subporpulation. * ISs or ILISs were used only for the purpose of quality control, not used for calculation of results.														

		ADDITIONAL COMPOUNDS					SUM	MAXIMUM OF EACH CALIBRATION TYPE	
	Internal Standards (IS) used	Captan (sum)	Folpet (sum)	THPI	Phthalimide	Sum of Additional Compounds			
	Procedural calibr.	6	6	6	6	**24 (9 %)**	**153 (10 %)**	Procedural calibr.	glyphosate (17 %), bromide (16 %), phosphonic acid (16 %)
	Std add. to sample PORTIONS	3	3	5	5	**16 (6 %)**	**105 (7 %)**	Std add. to sample PORTIONS	phosphonic acid 14 %
	Std add. to extract ALIQUOTS	2	2	7 (10 %)	7 (10 %)	**18 (7 %)**	**109 (7 %)**	Std add. to extract ALIQUOTS	captan, folpet, THPI, phthalimid, fenbutatin oxide (10 % each)
	MATRIX based	40 (62 %)	40 (61 %)	44 (65 %)	42 (62 %)	**166 (62 %)**	**793 (54 %)**	MATRIX-based	chlorothalonil (69 %), 2,4-D (65 %)
	Matrix-Matched (= Strawberry as Matrix)	32 (49 %)	32 (48 %)	35 (51 %)	33 (49 %)	**132 (49 %)**	**655 (45 %)**	MATRIX-Matched	2,4-D (58 %), chlorothalonil (55 %)
	Other Matrix	8 (12 %)	8 (12 %)	9 (13 %)	9 (13 %)	**34 (13 %)**	**138 (9 %)**		
	Pure SOLVENT based	2	3	5	7	**17 (6 %)**	**253 (17 %)**	Pure SOLVENT based	dithiocarbamates (61 %)
	Other	1	1			**2 (1 %)**	**7 (0 %)**		
	No Data on Calibration	11	11	1	1	**24 (9 %)**	**46 (3 %)**		
	Overall	**65**	**66**	**68**	**68**	**267**	**1466**		

			OPTIONAL COMPOUNDS				ADDITIONAL COMPOUNDS					ALL COMPOUNDS
	Q: was IS used?		Dithianon	Phosphonic acid	N-Acetyl glyphosate	Overall (Optional Compounds)	Captan (sum)	Folpet (sum)	THPI	Phthalimide	Overall (Additional Compounds)	Overall
	ISs were added to...											
	Yes, ILISs	**Sum**	**12**	**31**	**2**	**85**	**9**	**8**	**5**	**3**	**25**	**208**
	1) at the beginning of procedure		11 (92 %)	27 (87 %)	2 (100 %)	**76 (89 %)**	8 (89 %)	7 (88 %)	3 (60 %)	2 (67 %)	20 (80 %)	178 (86 %)
	3) at an intermediate stage (between 1 and 2)		–	1 (3 %)	–	**1 (1 %)**	–	–	–	–	–	3 (1 %)
	2) to an aliquot of the final extract		1 (8 %)	2 (6 %)	–	**6 (7 %)**	1 (11 %)	1 (13 %)	2 (40 %)	1 (33 %)	5 (20 %)	23 (11 %)
	No Data		–	1 (3 %)	–	**2 (2 %)**	–	–	–	–	–	4 (2 %)
	Yes, other ISs	**Sum**	**21**	**4**	**3**	**113**	**29**	**30**	**42**	**44**	**145**	**570**
	1) at the beginning of procedure		17 (81 %)	3 (75 %)	3 (100 %)	**83 (73 %)**	19 (66 %)	21 (70 %)	30 (71 %)	31 (70 %)	101 (70 %)	419 (74 %)
	3) at an intermediate stage (between 1 and 2)		–	1 (25 %)	–	**3 (3 %)**	1 (3 %)	1 (3 %)	1 (2 %)	1 (2 %)	4 (3 %)	18 (3 %)
	2) to an aliquot of the final extract		4 (19 %)	–	–	**27 (24 %)**	9 (31 %)	8 (27 %)	11 (26 %)	12 (27 %)	40 (28 %)	133 (23 %)
	No	**Sum**	**24 + 1***	**13**	**10**	**147 + 1***	**10**	**11**	**18**	**17**	**56**	**546 + 6***
	No data	**Sum**	**7**	**3**	**1**	**34**	**17**	**17**	**3**	**4**	**41**	**136**
	Overall		**65**	**51**	**16**	**380**	**65**	**66**	**68**	**68**	**267**	**1466**

Percentages in parentheses for each of the compounds refer to the respective subporpulation.
* ISs or ILISs were used only for the purpose of quality control, not used for calculation of results.

Table 4-16: Impact of ILISs or other ISs on the distribution of results and the average bias (only results from EU and EFTA laboratories were taken into account)

	Glyphosate			Chlorate		
	All Results	Results Obtained Using ILIS	Results Obtained without ILIS	All Results	Results Obtained Using ILIS	Results Obtained without ILIS
Robust Mean [mg/kg]	0.306	0.297	0.329	0.490	0.498	0.469
CV^*	20.9 %	**18.4 %**	**31.1 %**	16.2 %	**13.3 %**	**20.0 %**
AAZ [1] (average bias in %)	0.9 (23 %)	**0.7** (18 %)	1.3 (33 %)	0.7 (18 %)	**0.6** (15 %)	0.7 (18 %)
No. of results	86 [2]	53	29 [2]	60	38	12
No. (%) of acceptable results	77 (90 %)	50 (94 %)	23 (79 %)	54 (90 %)	35 (92 %)	11 (92 %)
No. (%) of questionable results	3 (3 %)	1 (2 %)	2 (7 %)	3 (5 %)	2 (5 %)	1 (8 %)
No. (%) of unacceptable results	6 (7 %) [2]	2 (4 %)	1 (14 %) [2]	3 (5 %)	1 (3 %)	0 (0 %)
	Phosphonic acid					
	All Results	Results Obtained Using ILIS	Results Obtained without ILIS			
Robust Mean [mg/kg]	19.3	19.9	19.6			
CV^*	27.0 %	**19.3 %**	**40.7 %**			
AAZ [1] (average bias)	1.0 (25 %)	**0.7** (18 %)	1.3 (33 %)			
No. of results	50	31	13			
No. (%) of acceptable results	43 (86 %)	29 (94 %)	11 (85 %)			
No. (%) of questionable results	4 (8 %)	2 (6 %)	1 (8 %)			
No. (%) of unacceptable results	3 (6 %)	0 (0 %)	1 (8 %)			

1) z-scores calculated using the robust mean in the corresponding population, "5" was used in case of the z-score was higher than 5. In the case of the porpulation of all results the robust mean is equal to the assigned value.
2) including false negative results

For ***glyphosate***, ***chlorate*** and ***phosphonic acid*** the results generated using ILISs were compared to the results generated not using them (**Table 4-16, p. 78**). Both the distance of the robust means and the variability of the results (reflected in CV^* and AAZ) of the two populations were calculated and compared with the respective figures of the overall population. The robust means of the two subpopulations (with and without ILIS) were overall close to each other. However, the distribution of the results of the laboratories using ILIS was for all three compounds clearly narrower compared to that of laboratories not using ILIS. As can be seen in **Table 4-16**, in the case of ***glyphosate*** the CV^*/AAZ values of the population using ILIS were 18.4 %/0.7 compared to 31.1 %/1.3 of the population not using it. The respective figures for ***chlorate*** were 13.3 %/0.6 vs. 20.0 % /0.7, and for ***phosphonic acid*** 19.3 %/0.7 vs. 40.7 %/1.3. Similar observations as regards the positive impact of ILISs on the quality of the results were made in the previous EUPT-SRMs (6 – 11).

In total 39 % of the results (570 out of 1466) were generated using "other ISs" (**Table 4-14, p. 76**). Other ISs may correct for volumetric errors and spills, and if these ISs show similar analytical behaviour to the target analytes, they may also partly correct for recovery and for sensitivity drifts of instruments. At the same time, however, such IS may also introduce errors. This is often the case in LC-MS/MS measurements if matrix effects are not compensated (e.g. through matrix-matching). Among the 570 cases where other ISs were used the laboratories reported their addition at the beginning of the procedure in 419 cases (74 %). 38 % of the results (552 out of 1466) were generated without using any ISs, and there was no information reported on the use of ISs for 9 % of the results (136 out of 1466).

Solvent-based calibrations were mainly used for ***dithiocarbamates*** (61 % of the cases), followed by ***bromide ion*** (49 %), ***phosphonic acid*** (25 %), ***chlorate*** (23 %) and ***glyphosate*** (22 %) (**Table 4-14, p. 76**).

Matrix-Matched calibration was the most frequently used calibration approach with 655 (45 %) of all results being generated in this way. This approach involved calibration based on blank extracts of the same matrix type, either by using the blank matrix that was provided or using an own blank strawberry. Matrix-matched calibration was used at frequencies ranging between 29 % for ***phosphonic acid*** to 58 % for ***2,4-D***. Among the 655 cases where matrix-matched calibrations were used the blank material provided by the organisers was used and in 621 cases (95 % thereof, 42 % overall) and an own blank strawberry was used in 34 cases (5 % thereof, 2 % overall). In 138 cases (9 % overall) other commodities were used for calibration. In this case matrix effects during measurement are not correctly compensated unless ILIS is used. It is assumed that the laboratories employing other blank commodities for calibration have selected this approach in order to ensure that the PT-results are generated in a way reflecting routine procedures.

Procedural calibration, another form of matrix matched calibration, in which results are automatically corrected for recovery, was used in 10 % of the cases overall. This approach was most frequently used in the case of ***N-acetyl glyphosate*** (25 % of the cases) followed by ***glyphosate*** (17 %), ***phosphonic acid*** (16 %), ***bromide ion*** (16 %), ***dithianon*** (14 %), ***chlorate*** (13 %) and ***bifenazate (sum)*** (13 %).

Standard addition to sample portions was used in 7 % of the cases overall and most frequently for ***phosphonic acid*** (14 % of the cases), ***N-acetyl glyphosate*** (13 %) and ***bifenazate (sum)*** (11 %).

In a wider sense matrix matching is not only accomplished when the analysis involves matrix-matched calibration but also when procedural calibration and standard additions to sample portions or extract aliquots are used. Altogether matrix-matching was employed in 69 % of the cases. In all these cases matrix effects were properly compensated. Matrix effects were further compensated in all cases where ILISs were used by the laboratories. Altogether matrix effects were thus compensated in 74 % of the cases. The compounds, for which matrix effects were compensated the least, were ***bromide ion*** (only 42 % of the cases involving matrix-matching) and ***dithiocarbamates*** (only 31 % of the cases). For all other compounds altogether matrix effects were compensated in more than 79 % of the cases with the glyphosate (93%), chlorate (93%) and phosphonic acid (92%) showing the highest percentages, see also **Table 4-13 (p. 74)**.

4.5.3 Correction for Recovery

Recovery corrections can be accomplished by using ILISs at the beginning or an intermediate stage of the procedure, or by other approaches. The two calibration types "procedural calibration" and "standard addition to sample portions" entail an "automatic" correction for recovery, irrespective whether ILIS is used or not. Another way of correcting for recovery is the use of recovery factors derived from recovery experiments to correct the results. The various approaches employed by the laboratories for recovery correction are compiled in **Table 4-17 (p. 80)**. In many cases different calibration types were combined with the use of ILISs for better accuracy. Overall, correction of results for recovery using various approaches was accomplished in 506 cases corresponding to 34 % of all results (**Table 4-17**).

The compounds for which recovery-based correction was applied most frequently were ***phosphonic acid*** (73 % of the cases), ***chlorate*** (70 %), ***glyphosate*** (66 %), ***N-acetyl glyphosate*** (50 %), ***dithianon*** (42 %), ***bromide ion*** (31 %), ***captan*** (33 %), ***folpet*** (31 %), ***bifenazate (sum)*** (30 %) and ***carbofuran (part of sum)*** (29 %). ***THPI*** (26 %), ***phthalimid***e (25 %), ***fenbutatin oxide*** (22 %), ***haloxyfop*** (21 %), ***dithiocarbamates*** (21 %), ***chlorothalonil*** (19 %) and ***2,4-D*** (19 %) follow. There were also several cases where different approaches correcting for recovery were combined such as ILISs with procedural calibrations in 32 cases (2 %) and with standard additions to sample portions in 15 cases (1 %) (**Table 4-13, p. 74**). ILISs were furthermore combined with standard additions to extract aliquots in 28 cases (2 %) and with matrix-matched calibrations in 70 cases (5 %). The use of matrix-matched calibrations also helps to compensate for matrix effects, especially when the same matrix is used. The use of recovery factors was rather limited (5 % of the cases).

Table 4-17: Overview of approches by the participating laboratories for recovery correction

	COMPULSORY COMPOUNDS									OPTIONAL COMPOUNDS			
Approches	**2,4-D**	**Captan (parent)**	**Chlorothalonil**	**Dithiocarbamates**	**Fenbutatin Oxide**	**Folpet (parent)**	**Glyphosate**	**Haloxyfop**	**Sum (Compulsory Compounds)**	**Bifenazate**	**Bromide ion**	**Carbofuran**	**Chlorate**
No. of Analysed for	104	98	120	114	87	103	92	101	**819**	56	55	77	60
Approches with Compensation for Recovery													
ILIS, added at the beginning	3 (3 %)	16 (16 %)		3 (3 %)	1 (1 %)	12 (12 %)	47 (51 %)		**82 (10 %)**			2 (3 %)	34 (57 %)
ILIS, added at an intermediate step							2 (2 %)		**2 (< 1 %)**				
ILIS + Procedual Calibration							1 (1 %)		**1 (< 1 %)**				
ILIS + Recovery Factor							1 (1 %)		**1 (< 1 %)**				1 (2 %)
Procedual calibration	8 (8 %)	3 (3 %)	10 (8 %)	9 (8 %)	9 (10 %)	5 (5 %)	7 (8 %)	8 (8 %)	**59 (7 %)**	7 (13 %)	9 (16 %)	8 (10 %)	3 (5 %)
Recovery factor	5 (5 %)	6 (6 %)	6 (5 %)	6 (5 %)	5 (6 %)	7 (7 %)		5 (5 %)	**40 (5 %)**	4 (7 %)	3 (5 %)	7 (9 %)	1 (2 %)
Std add. to sample portion	4 (4 %)	7 (7 %)	7 (6 %)	6 (5 %)	4 (5 %)	8 (8 %)	3 (3 %)	8 (8 %)	**47 (6 %)**	6 (11 %)	5 (9 %)	5 (6 %)	3 (5 %)
Sum	**20 (19 %)**	**32 (33 %)**	**23 (19 %)**	**24 (21 %)**	**19 (22 %)**	**32 (31 %)**	**61 (66 %)**	**21 (21 %)**	**232 (28 %)**	**17 (30 %)**	**17 (31 %)**	**22 (29 %)**	**42 (70 %)**

The distribution of the 75 recovery figures that were used to correct the results for recovery is shown in **Figure 4-2**. The recovery rates used in these cases ranged from 10 % to 170 %. In 64 % of the cases, however, these recovery figures were within 70 to 120 % and in 61 % of the cases between 80 and 120 %. In previous EUPT-SRMs the percentage of cases using recovery figures between 80 % and 120 % was 42 % in EUPT-SRM8, 35 % in EUPT-SRM9, 27 % in EUPT-SRM10 and 10 % in EUPT-SRM11. In 71 cases the recovery rates were obtained by using the blank material provided by the organiser, in the other four cases the recovery figures were derived by using other matrices.

In 28 cases the recovery factor was based on only one recovery figure. In 22, 28, and 1 case they were based one, two, three or more than five recovery replicates, respectively (**Table 4-18, p. 82**). Looking at previous EUPT-SRMs. The percentage of cases in which the recovery figures were obtained from only one experiment fluctuated from 28 % (19 out of 67) in EUPT-SRM8 up to 56 % (29 out of 52) in EUPT-SRM9, then down to 28 % (8 out of 29) in EUPT-SRM9 and to 18 % (4 out of 22) in EUPT-SRM11, and up again to 35 % (28 out of 79 cases) in the present PT. As the EURL-SRM has repeatedly emphasized in the EUPT-reports and at the EURL-Workshops, the use of recovery figures to correct for recovery require special attention. Recovery figures derived from one single experiment are particularly critical due to the higher risk of spurious errors.

Approches	OPTIONAL COMPOUNDS: Dithianon	Phosphonic acid	N-Acetyl glyphosate	Sum (Optional Compounds)	ADDITIONAL COMPOUNDS: Captan (sum)	Folpet (sum)	THPI	Phthalimide	Sum (Additional Compounds)	ALL COMPOUNDS: Overall
No. of Analysed for	65	51	16	**380**	65	66	68	68	**267**	**1466**
Approches with Compensation for Recovery										
ILIS, added at the beginning	11 (17 %)	28 (55 %)	2 (13 %)	**77 (20 %)**	8 (12 %)	7 (11 %)	3 (4 %)	2 (3 %)	**20 (7 %)**	**179 (12 %)**
ILIS, added at an intermediate step		1 (2 %)		**1 (< 1 %)**						**3 (< 1 %)**
ILIS + Procedual Calibration										**1 (< 1 %)**
ILIS + Recovery Factor		1 (2 %)		**2 (1 %)**						**3 (< 1 %)**
Procedual calibration	9 (14 %)	2 (4 %)	4 (25 %)	**42 (11 %)**	5 (8 %)	6 (9 %)	6 (9 %)	6 (9 %)	**23 (9 %)**	**124 (8 %)**
Recovery factor	3 (5 %)	1 (2 %)	1 (6 %)	**20 (5 %)**	3 (5 %)	3 (5 %)	4 (6 %)	4 (6 %)	**14 (5 %)**	**74 (5 %)**
Std add. to sample portion	4 (6 %)	4 (8 %)	1 (6 %)	**28 (7 %)**	2 (3 %)	2 (3 %)	5 (7 %)	5 (7 %)	**14 (5 %)**	**89 (6 %)**
Sum	**27 (42 %)**	**37 (73 %)**	**8 (50 %)**	**170 (45 %)**	**18 (28 %)**	**18 (27 %)**	**18 (26 %)**	**17 (25 %)**	**71 (27 %)**	**473 (32 %)**

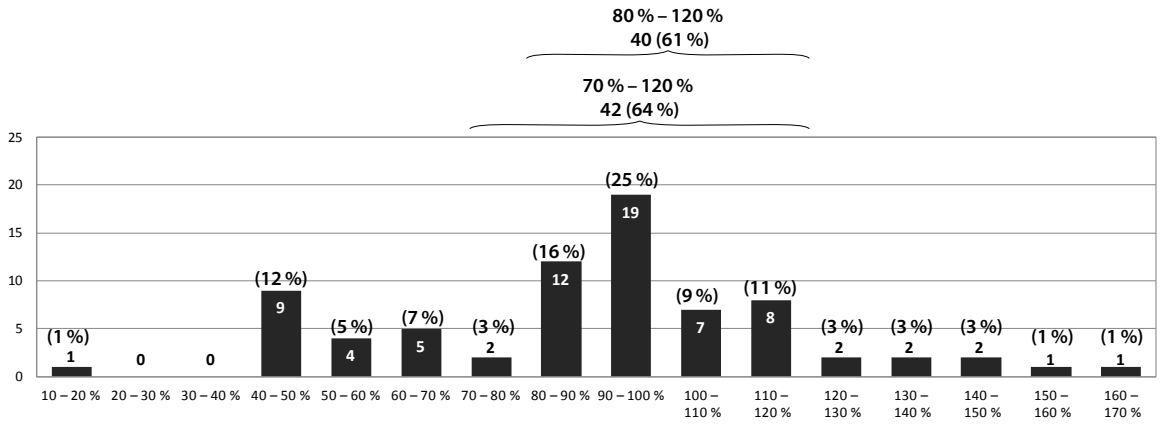


Figure 4-2: Distribution of recovery figures used for results correction for recovery. Four results using reported recovery rate of 100 % and one with reported recovery rate of 0 % were not taken into account.

Table 4-18: Compilation of results where RECOVERY FACTOR-BASED CORRECTION OF RESULTS was applied and influence on the AAZ-scores (average bias)

Compounds	LabCode SRM12-	Submitted Recovery rate [%] #	Recovery Replicates considered	Submitted Result [mg/kg]	z-Score derived from submitted result	z-Score (if non-corrected results were submitted)*
2,4-D | AV = 0.079 mg/kg	2	92.3	2	0.0692	**-0.5**	-0.8
	10	87	1	0.067	**-0.6**	-1.0
	70	96.6	1	0.056	**-1.2**	-1.3
	115	119	3	0.059	-1.0	-0.4
	130	127	3	0.101	**1.1**	2.5
Captan (parent) | AV = 0.085 mg/kg	2	92.5	2	0.0881	**0.1**	-0.2
	6	45.6	3	0.115	**1.4**	-1.5
	45	44	2	0.107	**1.0**	-1.8
	46	48	1	0.0748	**-0.5**	-2.3
	70	91.8	2	0.02	-3.1	-3.1
	115	92	3	0.09	0.2	-0.1
Chlorothalonil | AV = 0.125 mg/kg	2	88.1	2	0.12	**-0.2**	-0.6
	6	45.3	3	0.122	**-0.1**	-2.2
	10	116	1	0.29	**5.3**	6.7
	46	64	1	0.13	**0.2**	-1.3
	70	139	2	0.12	**-0.2**	1.3
	115	95	3	0.135	0.3	0.1
Dithiocarbamates | AV = 0.267 mg/kg	2	109	3	0.234	-0.5	-0.2
	10	85	1	0.382	1.7	0.9
	35	16	1	0.819	8.3	-2.0
	81	63	3	0.251	**-0.2**	-1.6
	103	61	1	0.263	**-0.1**	-1.6
Fenbutatin Oxide | AV = 0.086 mg/kg	70	92.1	1	0.065	**-1.0**	-1.2
	90	58	1	0.095	**0.4**	-1.4
	115	99	3	0.075	-0.5	-0.5
Folpet (parent) | AV = 0.334 mg/kg	2	99.3	2	0.349	0.2	0.1
	6	44.2	3	0.317	**-0.2**	-2.3
	45	54	1	0.462	1.5	-1.0
	46	48	1	0.326	**-0.1**	-2.1
	70	137.4	2	0.33	**-0.1**	1.4
	103	45	1	0.44	**1.3**	-1.6
	115	82	3	0.347	**0.2**	-0.6
Glyphosate | AV = 0.306 mg/kg	2	96.4	3	0.317	0.1	0.0
	10	86	1	0.312	**0.1**	-0.5
	14	104	2	0.311	**0.1**	0.2
	115	116	3	0.270	-0.5	0.1
Haloxyfop | AV = 0.07 mg/kg	2	81.4	2	0.058	**-0.7**	-1.3
	10	92	1	0.066	**-0.2**	-0.5
	70	148	1	0.066	**-0.2**	1.6
	115	101	3	0.071	**0.0**	0.1
	130	127	3	0.087	**1.0**	2.3
Bifenazate | AV = 0.27 mg/kg	2	78.5	2	0.198	**-1.1**	-1.7
	70	91	2	0.63	5.3	4.5
	72	155	2	0.187	-1.2	0.3
	115	119	3	0.25	**-0.3**	0.4
Bromide ion | AV = 19.1 mg/kg	2	97	5	17.3	**-0.4**	-0.5
Carbofuran | AV = 0.003 mg/kg	10	114	1	0.0067	**4.9**	6.2
	45	146	2	0.0023	-0.9	0.5
	67	54.9	1	0.0025	**-0.7**	-2.2
	70	112	2	0.0051	**2.8**	3.6
	92	60	2	0.0024	**-0.8**	-2.1
	115	86	3	0.0017	**-1.7**	-2.1
Chlorate | AV = 0.49 mg/kg	2	96.1	3	0.47	**-0.2**	-0.3
	10	97	1	0.546	0.5	0.3
	103	115	1	0.415	-0.6	-0.1

* Calculated using the current assigned values
# Four results with reported recovery rate of 100 % and one with reported recovery rate of 0 % were not taken into account.
AV = assigned value; AAZ = average of absolute z-score indicating average bias

Table 4-18 (cont.): Compilation of results where RECOVERY FACTOR-BASED CORRECTION OF RESULTS was applied and influence on the AAZ-scores (average bias)

Compounds	LabCode SRM12-	Submitted Recovery rate [%] #	Recovery Replicates considered	Submitted Result [mg/kg]	z-Score derived from submitted result	z-Score (if non-corrected results were submitted)*
	115	71	3	0.49	**0.0**	-1.2
Dithianon | AV = 0.294 mg/kg	2	87.9	2	0.268	**-0.3**	-0.8
	70	89	1	0.35	0.8	0.2
	103	107	1	0.323	**0.4**	0.7
	115	70	3	0.28	**-0.2**	-1.3
Phosphonic acid | AV = 19.3 mg/kg	2	101	2	17.9	-0.3	-0.2
	10	112	1	27.9	**1.8**	2.5
	115	95	3	29.0	2.0	1.7
N-Acetyl glyphosate | AV = 0.1 mg/kg	103	97	1	0.094	**-0.3**	-0.4
	115	108	3	0.071	-1.2	-0.9
Captan (sum) | AV = 0.302 mg/kg	115	106	3	0.183	-1.6	-1.4
Folpet (sum) | AV = 1.195 mg/kg	115	92	3	1.43	0.8	0.4
THPI | AV = 0.11 mg/kg	2	85.1	2	0.0946	**-0.6**	-1.1
	46	48	1	0.104	**-0.2**	-2.2
	70	62.4	2	0.064	**-1.7**	-2.6
	115	84	3	0.047	**-2.3**	-2.6
Phthalimide | AV = 0.446 mg/kg	2	95.1	2	0.382	**-0.6**	-0.7
	46	48	1	0.39	**-0.5**	-2.3
	70	161.5	2	0.31	-1.2	0.5
	115	85	3	0.5	0.5	-0.2
Overall	16 Labs	75 Cases	1 Repl.(26x) 2 Repl.(22x) 3 Repl.(26x) 4 Repl.(0x) 5 Repl.(1x)		AAZ(all) = 0.9 AAZ(80–120%) = 1.0 Acceptable: 68 Questionable: 2 Unacceptable: 5	AAZ(all) = 1.3 AAZ(80–120%) = 1.1 Acceptabel: 55 Questionable: 15 Unacceptable: 5

* Calculated using the current assigned values
# Four results with reported recovery rate of 100 % and one with reported recovery rate of 0 % were not taken into account.
AV = assigned value; AAZ = average of absolute z-score indicating average bias

Correction using a recovery factor will typically lead to a result that is closer to the assigned value compared to the result that would have been reported if no recovery correction had been applied (provided that the assigned value is not strongly distant from the real value itself due to a large number of laboratories using biased methods). Using the recovery factor rates submitted by the laboratories it is possible to calculate the original results of the laboratories and the z-scores that would have been achieved if these results were submitted. By applying a recovery factor to correct for recovery, participants' z-scores (in absolute terms) have improved in 47 cases and worsened in 23 cases. Excluding results associated with recovery rates between 80 – 120 %, the ratio of improved versus worsened z-scores shifts from approximately 2 (47 vs. 23) to 5 (25 vs. 5). This clarifies that recovery corrections based on recovery rates have little impact when recoveries are close to 100 %. In 12 cases the results moved from "questionable" to "acceptable" and in one case from "unacceptable" to "questionable". In further 5 cases (all associated with recovery rates between 80 % and 120 % where recovery correction has typically little impact) there was no change in the categorization (**Table 4-18**). In one case the result moved from "acceptable" to "unacceptable" when the recovery factor of 16 % (at n = 1) was applied. Comparing the AAZ of the recovery-corrected results with that of the results that would have been submitted if no recovery-based correction had been applied, a significant decline from 1.3 to 0.9 is observed. In previous EUPT-SRMs similar trends were observed, although these trends were typically stronger (AAZ shift from 1.3 to 0.7 in EUPT-SRM10; from 1.9 to 1.1 in EUPT-SRM9; from 1.7 to 1.1 in EUPT-SRM8 and from 2.2 to 1.1 in EUPT-SRM7). This is surely related to the fact that EUPT-SRM12 entailed only few compounds where low recoveries are typically achieved. Looking at the cases where correction was based on recoveries between 80 % and 120 % the AAZ improved from 1.1 to 1.0, whereas in all

other cases the improvement was from 1.3 to 0.9. Despite this positive effect, recovery correction based on recovery figures should be the last remedy as this approach is tricky and less accurate compared to other types of result correction such as the use of ILISs, procedural calibrations or standard addition to sample portions.

4.5.4 Comparison of Methods employed in the EUPT-SRM12

An overview of the methodological approaches mainly applied by the EU- and EFTA-Laboratories in the EUPT-SRM12 is shown in **Table 4-19**. Overall, QuEChERS (up to 92 % for dithianon) and in case of polar compounds QuPPe (up to 98 % for ***phosphonic acid***) were the most frequently used methods.

Transformation to Carbofuran: As shown in **Section 1.3 (p. 5)**, the concentration of ***carbofuran (part of sum)*** may be underestimated if no chemical transformation step to ***carbofuran*** is applied. Alternatively, ***carbofuran*** and carbosulfan may be determined separately and added up expressed as ***carbofuran***, but this adds to the uncertainty of analysis and increases the risk of false negative results. The influence of acidic hydrolysis is also reflected in the participants results (**Table 4-20**) with the robust mean of the population applying chemical transformation (0.0039 mg/kg) being by 33 % higher than that of the entire population. This value was closer to the mean concentration detected by the organizers in the homogeneity test (0.0043 mg/kg). However, due to the very low concentration and smaller population, the CV^* of this subpopulation was also very high.

Table 4-19: Comparison of methods mainly employed by the EU- and EFTA-laboratories in the EUPT-SRM12

	Compound	ACN based (various QuEChERS versions)	EtAc based (diff. SweEt versions	Acetone based (e.g. Mini-Luke/ S19/Dutch)	QuPPe	Involving Deriv./ Transf.	Liq-Liq Part./ Spectroph./ Head Space
Compulsory Compounds	2,4-D	87 %	2 %	4 %	3 %	1 %	
	Captan (parent)	76 %	11 %	12 %			
	Chlorothalonil	78 %	10 %	11 %			
	Dithiocarbamates						45 % / 21 % / 32 %
	Fenbutatin oxide	90 %	4 %	1 %	2 %	1 %	
	Folpet (parent)	78 %	10 %	11 %			
	Glyphosate	1 %			70 %	26 %	
	Haloxyfop	88 %	3 %	3 %	2 %	1 %	
Optional Compounds	Bifenazate	87 %	4 %	4 %	2 %		
	Bromide ion	2 %			21 %	52 %	
	Carbofuran (with chem. transformation)	82 % (20 %)	7 %	4 %	1 %	20 % (see QuEChERS)	
	Chlorate				100 %		
	Dithianon	92 %		2 %	2 %		
	Phosphonic acid	2 %			98 %		
	N-Acetyl glyphosate				94 %		
Additional Compounds	Captan (sum)	69 %	8 %	8 %			
	Folpet (sum)	68 %	9 %	8 %			
	THPI	82 %	9 %	7 %			
	Phthalimide	81 %	10 %	7 %			

Table 4-20: Impact of acidic transformation on the results of carbofuran

	Entire Population	With Chem. Transformation	Without Chem. Transformation	No Data
No. of Results (total)	74	14	38	22
No. of Results (nummerical)	58	13	31	14
No. of FN	3 + 13*	1*	5* + 2	1 + 7*
Robust Mean [mg/kg]	**0.0030**	**0.0039**	**0.0028**	**0.0027**
CV^*	47.1 %	50.2 %	49.7 %	28.9 %
* FN due to lab's RL higher than the assigned value				

Analysis of Bromide Ion: The QuPPe/LC-MS/MS method newly introduced by the EURL-SRM for the analysis of bromide ion was employed by 21 % of the participating laboratories. This method is much more simple compared to the traditional approach involving derivatisation. Compared with the other methods employed for the determination of ***bromide ion*** (**Table 4-21**), the robust mean of the LC-MS/MS method was overall comparable to that of the other methods but the distribution of the results overall broader. It should be noted, however, that several of the labs reporting biased results deviated from the procedure by not employing a large fragmentation energy to improve selectivity as indicated in the QuPPe protocol. Excluding these results, the CV^* decreased from 39.1 % to 17.7 %.

Compounds Used for Calibration: Table 4-22 (p. 86) shows an overview of compounds used for calibration for ***dithiocarbamates***, ***bifenazate (sum)***, ***carbofuran (part of sum)***, ***captan (parent and sum)*** and ***THPI*** as well as for ***folpet (parent and sum)*** and ***phthalimide***. Unfortunately, a substantial number of laboratories did not provide information this regarding. 69 out of total 82 (84 %) of the participants having submitted results for ***dithiocarbamates*** and giving an answer to this question, used CS_2 for calibration. 13 participants used thiram as calibration standard. With CS_2 being very volatile, its stock and working solutions are difficult to handle and store. On the other hand, the use of thiram for calibration requires its spiking at the beginning of the procedure in a procedural calibration approach.

In the case of ***bifenazate (sum)*** most labs used ***bifenazate*** for calibration, but there were also cases where ***bifenazate*** and bifenazate diazene were calibrated separately as well as one case where bifenazate diazene was used for calibration. In the case of ***carbofuran (part of sum)*** 25 laboratories reported the use of ***carbofuran*** for calibration with 10 laboratories thereof (40 %) using a procedure entailing a hydrolysis step. 7 laboratories indicated the separate calibration of ***carbofuran*** and carbosulfan, two of them reported concentrations at around the assigned value based on the entire population (0.0030 mg/kg), another two un-

Table 4-21: Comparison of results of bromide ion obtained from different methods

	Entire Population	LC-MS/MS (QuPPe)	LC-MS/MS (QuPPe) excl. 3 results *	Derivatisation-GC	IC-Conductivity	Other #
No. of Results (total)	52	11	8	27	8	6 *
No. of Results (nummerical)	52	11	8	27	8	6
No. of FN	0	0	0	0	0	0
Robust Mean [mg/kg]	**19.1**	**18.1**	**19.4**	**19.0**	**21.4**	**–**
CV^*	16.0 %	39.1 %	17.7 %	13.7 %	23.5 %	–

* Three results were obtained without using high collision energy and therefore excluded.
# Two results drived by extraction/UV-DAD, one by XRF, one by extraction/ICP-MS, one by ion-pair LC and one by QuEChERS/LC-Orbitrap

Table 4-22: Compounds used for calibration in the EUPT-SRM12

Analyte Compounds used for calibration	No. (%*)	AAZ
Dithiocarbamates		
CS_2	69 (61 %)	0.9
Thiram	13 (11 %)	0.8
No Data / None of those listed	32 (28 %)	0.8
Bifenazate (sum)		
Bifenazate	27 (48 %)	1.0
Bifenazate and Bifenazate diazene (separately)	6 (11 %)	0.7
Bifenazate diazene	1 (2 %)	1.6
No Data / None of those listed	22 (39 %)	0.6
Carbofuran (part of sum)		
Carbofuran	25	–
Carbofuran and Benfuracarb (separately)	1	–
Carbofuran and Carbosulfan (separately)	7	–
Carbofuran and Furathiocarb (separately)	1	–
No Data / None of those listed	43	–
Captan (parent)		
Captan	47 (48 %)	1.1
Captan and THPI (separately)	8 (8 %)	0.8
THPI	–	–
No Data / None of those listed	43 (44 %)	1.2
Captan (sum)		
Captan	8 (12 %)	1.4
Captan and THPI (separately)	30 (46 %)	0.8
THPI	3 (5 %)	0.7
No Data / None of those listed	24 (37 %)	0.6
THPI		
Captan	6 (9 %)	1.6
Captan and THPI (separately)	7 (10 %)	1.0
THPI	31 (46 %)	1.1
Phthalimide	1 (1 %)	1.5
No Data / None of those listed	23 (34 %)	1.0
Folpet (parent)		
Captan / Folpet	50 (49 %)	1.1
Folpet and Phthalimide (separately)	7 (7 %)	0.8
Phthalimide	–	–
No Data / None of those listed	46 (45 %)	1.2
Folpet (sum)		
Captan / Folpet	8 (12 %)	1.4
Folpet and Phthalimide (separately)	30 (45 %)	0.9
Phthalimide	3 (5 %)	0.5
No Data / None of those listed	25 (38 %)	0.6
Phthalimide		
Captan / Folpet	5 (7 %)	1.5
Folpet and Phthalimide (separately)	8 (12 %)	1.6
Phthalimide	32 (47 %)	0.9
No Data / None of those listed	23 (34 %)	0.7

* Percentages are based on the total number of laboratories submitting data as regards the compound(s) used for calibration.

derestimated (0.0018 and 0.0021 mg/kg), while the remaining three, all at or over 0.0046 mg/kg, were higher than the assigned value based on the population with chemical transformation (0.0039 mg/kg). As shown in **Section 1.3 (p. 5)**, the non-conduction of a hydrolysis step can lead to underestimated results.

Captan and Folpet: One of the biggest challenges in the present PT was surely the analysis of ***captan (parent)*** and ***folpet (parent)*** as well as their degradation products ***THPI*** and ***phthalimide***. Equally challenging is the determination of ***captan (sum)*** and ***folpet (sum)***, since the degradation products (***THPI*** and ***phthalimide***) may not only be present in the test material, but can be also generated from the respective parents during hot injection in the GC. Similar problems are faced in routine work. When analysing these compounds, it should be kept in mind that 1) the concentration of ***THPI*** or ***phthalimide*** detected by GC does not directly reflect the concentration of these compounds in the sample, as it includes those parts generated during injection; 2) when employing GC, the use of captan-ILIS and folpet-ILIS at the same time as the respective ILISs of the decomposition products can be critical as the latter is formed from the decomposition of the respective parents during injection and therefore introducing errors. At this point it should be noted that as strawberry is a strongly acidic commodity, the decomposition of these compounds (at any stage of analysis) was less pronounced compared to a basic commodity.

There are different approaches to determine ***captan (sum)*** and ***folpet (sum)***. A few days prior to submission deadline of the exercise the EURL-SRM has distributed two methods for the analysis of ***captan***, ***folpet*** and their degradation products (***THPI*** and ***phthalimide***) as well as the respective summed residues. The methods were accompanied by Excel-sheets to assist calculation. The distributed method makes the assumption that ***phthalimide*** and ***THPI*** are generated from their respective parents in the hot GC injection in a linear relationship and that these fractions can be calculated from the original levels of ***captan*** and ***folpet*** which can be determined using ILIS. The original levels of ***THPI*** and ***phthalimide*** are calculated by deducting the levels

of these degradation products generated during injection from the total levels determined via external calibration. ***Captan (sum)*** and ***folpet (sum)*** are finally calculated through addition, considering that the residue should be expressed as parent.

Hydrolysis of Acidic Pesticides: For some acidic pesticides in the Target Pesticides List the legal residue definition includes esters and conjugates. The organiser still decided to only ask for the analysis of the free acids to keep the PT more simple and avoid mixed populations of results. The impact of alkaline or acidic hydrolysis on the release of conjugated residues of acidic pesticides will be studied in future EUPTs and collaborative method validation tests. Although it was clearly defined in the target pesticides list that no hydrolysis step was to be applied, still, 3 laboratories have conducted a hydrolysis step for ***2,4-D***, ***haloxyfop***, and *fluazifop*. For ***2,4-D*** and ***haloxyfop***, which were spiked to the material in form of free acid, no significant influence of hydrolysis on the results could be observed by the organizers (as expected). However, the participants are urged to keep an eye on the residue definitions in the target pesticides list.

4.5.5 Coverage of Compounds in Routine Scope and Analytical Experience of Laboratories

As can be seen in **Figure 4-3 (p. 88)** the percentage of all participating laboratories (n = 138) that covered the various compounds in the EUPT-SRM12 Target Pesticides List varied greatly ranging from 59 % (*ethephon*) to 87 % (***chlorothalonil***) among the compulsory compounds and between 12 % (***N-acetyl glyphosate***) and 56 % (***carbofuran (part of sum)***) among the optional compounds. For the four additional compounds the analysis rate was around 50 % in all cases. For the four additional compounds the analysis rate was around 50 %. Although introduced several years ago in EUPT-schemes, and although included in the EU-coordinated monitoring, ***fenbutatin oxide***, *ethephon* and ***glyphosate*** are still analysed by the fewest laboratories on a routine basis, but a positive trend can be observed: The participants analysing for ***fenbutatin oxide***, *ethephon* and ***glyphosate*** rose from 59, 51, 49, respectively in 2013 (SRM8) to 60, 66 and 69, respectively in 2015 (SRM10) and further to 87, 81 and 92, respectively in the present PT.

In 40 cases the compounds within the labs' routine scope were not analysed for in this PT (**Table 4-23, p. 89**). Among them the participants reported in 15 cases that the analytes were accredited in their laboratories either via flexible scope or specifically for this commodity type and within routine scope. Except one case, participants reported the following reasons for not analysing those compounds: lack of personnel (19×), technical problem with instruments (12×), lab's reporting limit (RL) higher than the MRRL in the PT (5×) and lack of necessary analytical standards (3×).

In 458 cases the participating laboratories even analysed compounds not yet included in their routine scope, among them 256 cases concerning compulsory compounds, 115 cases concerning optional compounds and 87 cases concerning additional compounds. This indicates that many laboratories are in the position or in the process of expanding their scope with additional SRM-compounds. The compound most frequently analysed by laboratories but not yet included in their routine scope was ***glyphosate*** (28 laboratories).

Figure 4-4 (p. 89) gives an overview of the overall analytical experience of the participating laboratories for compulsory, optional and additional compounds separately. Overall the experience of the laboratories with compulsory compounds was clearly higher than that with optional and additional compounds, with the labs reporting > 2 years of experience with the analysis of the respective compounds in 80 %, 61 % and 51 % of the cases, respectively. **Table 4-24 (p. 90)** shows the experience reported for each single compound present in the test item as well as the average performance of the labs as reflected by the AAZ (average absolute bias). In general, laboratories with > 2 years of experience achieved on average better z-scores

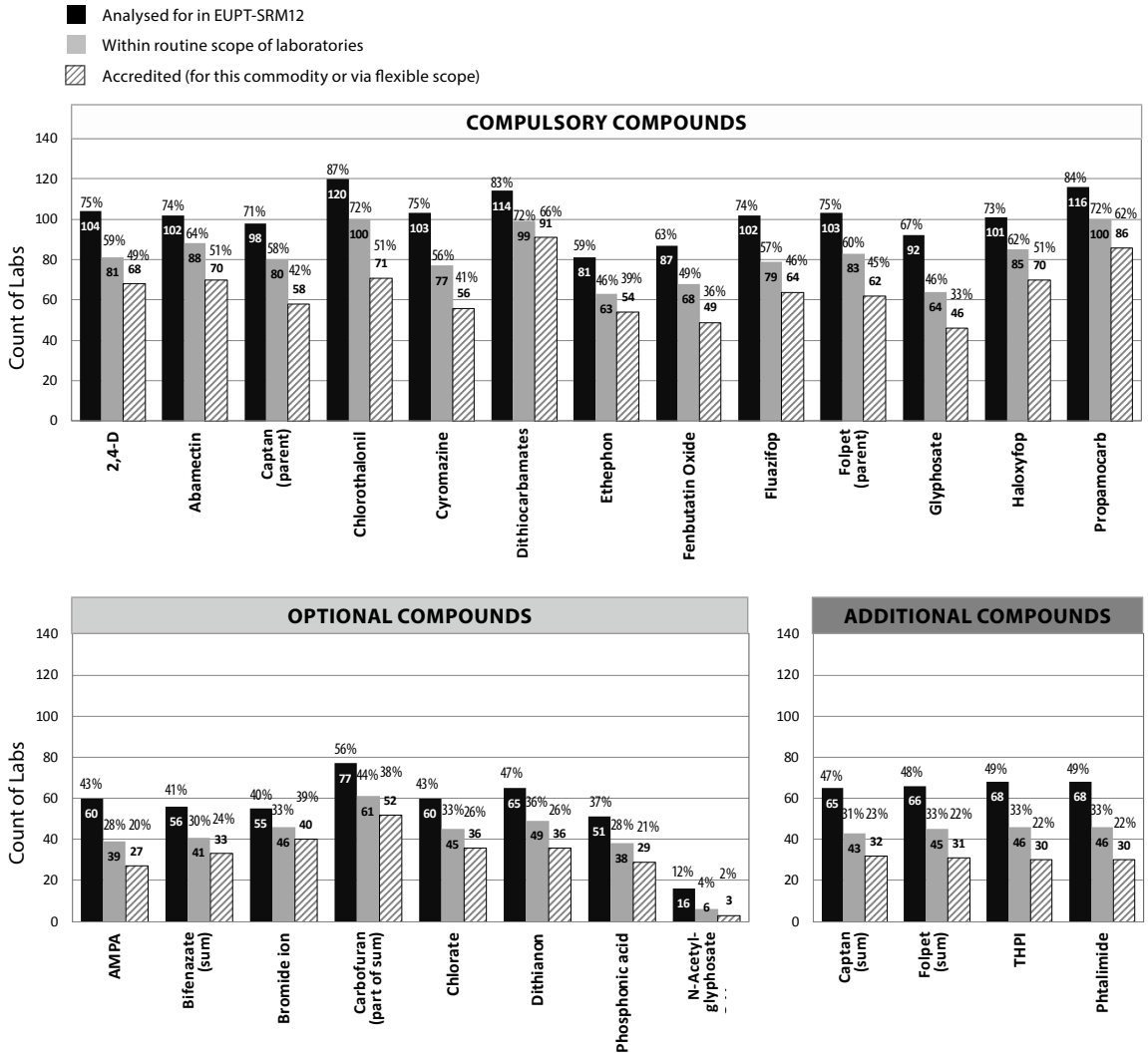


Figure 4-3: Number of laboratories targeting compounds within the framework of the EUPT-SRM12, within their routine scope and within the accredited scopes. Percentages are based on the total number of participating laboratories having submitted at least one result (n = 138).

than those having less experience. (**Figure 4-5, p. 91**) gives an overview of this correlation. Among the compulsory compounds present in the test item laboratories had the most experience with the analysis of ***dithiocarbamates***. 80 laboratories (90 %) indicated more than two years of experience with the analysis of ***dithiocarbamates***. The compulsory compound with which the laboratories had the least experience was ***glyphosate*** with 22 % of the laboratories reporting less than one year.

Bromide ion, ***chlorate*** and ***bifenazate (sum)*** were the optional analytes which the laboratories had the most experience with. 84 %, 67 % and 66 % of the laboratories indicated an experience of > 2 years with ***bromide ion***, ***chlorate*** and ***bifenazate (sum)***, respectively. ***N-Acetyl glyphosate*** was the compound which the participating laboratories had the least experience with: None of the participants had experience of more than two years and 88 % of the laboratories having submitted results reported experience of less than one year with the analysis of this compound.

Among laboratories having analysed for the additional compounds roughly every second laboratory had long experience (> 2 years), and roughly 40 % of the labs had experience of less than one year.

Table 4-23: Inclusion of EUPT-SRM12 compounds in the laboratories' routine scope (including data of laboratories from EU-candidate and third countries)

		within routine scope of lab		NOT within routine scope of lab	
		analysed for in this EUPT	not analysed for	analysed for in this EUPT	not analysed for
COMPULSORY COMPOUNDS	**2,4-D**	81 (98 %)	2	23 (42 %)	32
	Abamectin	88 (99 %)	1	14 (29 %)	35
	Captan (parent)	80 (99 %)	1	18 (32 %)	39
	Chlorothalonil	100 (98 %)	2	20 (56 %)	16
	Cyromazine	77 (99 %)	1	26 (43 %)	34
	Dithiocarbamates	99 (100 %)	0	15 (38 %)	24
	Ethephon	63 (97 %)	2	18 (25 %)	55
	Fenbutatin oxide	68 (99 %)	1	19 (28 %)	50
	Fluazifop	79 (98 %)	2	23 (40 %)	34
	Folpet (parent)	83 (99 %)	1	20 (37 %)	34
	Glyphosate	64 (97 %)	2	28 (39 %)	44
	Haloxyfop	85 (99 %)	1	16 (31 %)	36
	Propamocarb	100 (98 %)	2	16 (44 %)	20
	SUM	**1067 (98 %)**	**18 (1.7 %)**	**256 (36 %)**	**453 (64 %)**
OPTIONAL COMPOUNDS	**AMPA**	39 (100 %)	0	21 (21 %)	78
	Bifenazate (sum)	41 (98 %)	1	15 (16 %)	81
	Bromide ion	46 (98 %)	1	9 (10 %)	82
	Carbofuran	61 (94 %)	4	16 (22 %)	57
	Chlorate	45 (94 %)	3	15 (17 %)	75
	Dithianon	49 (96 %)	2	16 (18 %)	71
	Phosphonic acid	38 (100 %)	0	13 (13 %)	87
	N-Acetyl glyphosate	6 (86 %)	1	10 (8 %)	121
	SUM	**325 (96 %)**	**12 (3.6 %)**	**115 (15 %)**	**275 (76 %)**
ADDITIONAL COMPOUNDS	**Captan (sum)**	43 (96 %)	2	22 (24 %)	71
	Folpet (sum)	45 (96 %)	2	21 (23 %)	70
	THPI	46 (94 %)	3	22 (25 %)	67
	Phthalimide	46 (94 %)	3	22 (25 %)	67
	SUM	**180 (95 %)**	**10 (5.3 %)**	**87 (24 %)**	**652 (85 %)**
	Overall Sum	**1572 (98 %)**	**40 (2.5 %)**	**458 (25 %)**	**1380 (75 %)**

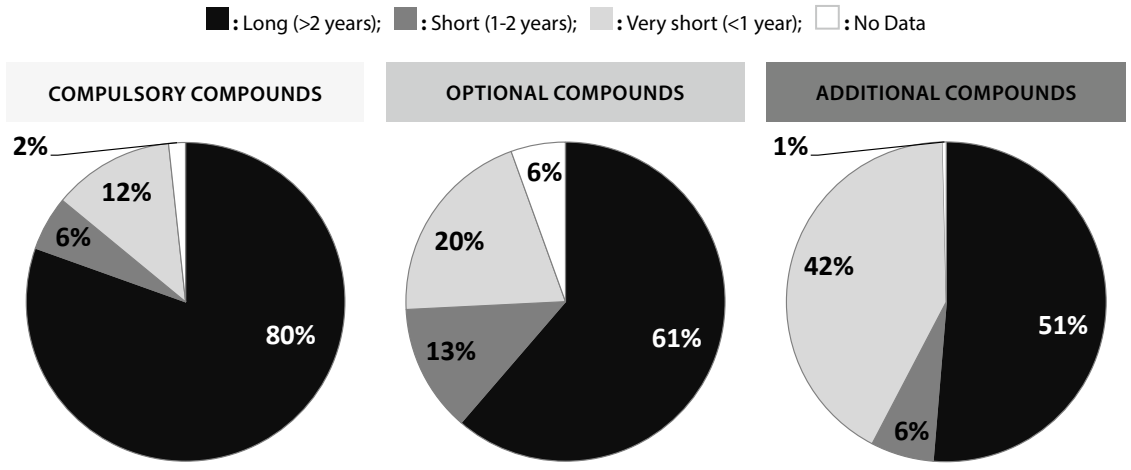


Figure 4-4: Overall experience of laboratories with the analysis of pesticides present in the test item (the shown figures refer to compounds present in the test item and analysed by the laboratories.)

Table 4-24: Laboratories' experience with the analysis of individual compounds present in the test item and correlation with AAZ reflecting the average deviation from the assigned value. AAZs and CV^* were calculated for populations with at least 5 laboratories.

COMPULSORY COMPOUNDS				OPTIONAL COMPOUNDS			
Pesticides	**Experience**	**No. of Labs [2] (%)**	**AAZ/CV^* [2]**	**Pesticides**	**Experience**	**No. of Labs [2] (%)**	**AAZ/CV^* [2]**
2,4-D (free acid) AAZ [1]: 0.6 CV^* [1]: 13.3 %	> 2 years	88 (85 %)	0.5/13.1 %	**Bifenazate** AAZ [1]: 0.8 CV^* [1]: 22.1 %	> 2 years	37 (66 %)	0.7/20.1 %
	1 – 2 years	7 (7 %)	1.1/22.7 %		1 – 2 years	5 (9 %)	0.9/36.8 %
	< 1 year	8 (8 %)	0.8/8.6 %		< 1 year	13 (23 %)	1.1/33.1 %
	no data	1 (1 %)			no data	1 (2 %)	
Captan (parent) AAZ [1]: 1.1 CV^* [1]: 28.1 %	> 2 years	80 (82 %)	1.0/25.7 %	**Bromide ion** AAZ [1]: 0.7 CV^* [1]: 16.0 %	> 2 years	46 (84 %)	0.7/14.4 %
	1 – 2 years	3 (3 %)			1 – 2 years	3 (5 %)	
	< 1 year	13 (13 %)	1.6/41.8 %		< 1 year	6 (11 %)	1.1/46.4 %
	no data	2 (2 %)			no data	0 (0 %)	
Chlorothalonil AAZ [1]: 1.0 CV^* [1]: 25.2 %	> 2 years	100 (83 %)	0.9/23.9 %	**Carbofuran [3]** AAZ [1]: 1.8 CV^* [1]: 47.1 %	> 2 years	42 (55 %)	1.5/43.4 %
	1 – 2 years	4 (3 %)			1 – 2 years	2 (3 %)	
	< 1 year	14 (12 %)	1.8/54.8 %		< 1 year	15 (19 %)	2/55.7 %
	no data	2 (2 %)			no data	18 (23 %)	2.7/ -
Dithiocarbamates AAZ [1]: 0.8 CV^* [1]: 22.2 %	> 2 years	103 (90 %)	0.8/22.4 %	**Chlorate** AAZ [1]: 0.7 CV^* [1]: 16.2 %	> 2 years	40 (67 %)	0.7/11.5 %
	1 – 2 years	5 (4 %)	1.9/63.5 %		1 – 2 years	11 (18 %)	1/38.5 %
	< 1 year	6 (5 %)	0.4/12.3 %		< 1 year	9 (15 %)	0.8/20.5 %
	no data	0 (0 %)			no data	0 (0 %)	
Fenbutatin Oxide AAZ [1]: 0.9 CV^* [1]: 21.0 %	> 2 years	60 (69 %)	0.8/20.7 %	**Dithianon** AAZ [1]: 1.1 CV^* [1]: 25.3 %	> 2 years	40 (62 %)	1/19.8 %
	1 – 2 years	7 (8 %)	1.6/49.4 %		1 – 2 years	14 (22 %)	1/35.6 %
	< 1 year	16 (18 %)	0.6/17.5 %		< 1 year	10 (15 %)	1.4/36.8 %
	no data	4 (5 %)			no data	1 (2 %)	
Folpet (parent) AAZ [1]: 1.1 CV^* [1]: 25.0 %	> 2 years	84 (82 %)	0.9/22.1 %	**Phosphonic acid** AAZ [1]: 1.0 CV^* [1]: 27.0 %	> 2 years	28 (55 %)	1/26.9 %
	1 – 2 years	2 (2 %)			1 – 2 years	13 (25 %)	1/28.8 %
	< 1 year	15 (15 %)	2.2/69.4 %		< 1 year	10 (20 %)	1.2/28.4 %
	no data	2 (2 %)			no data	0 (0 %)	
Glyphosate AAZ [1]: 0.9 CV^* [1]: 20.9 %	> 2 years	57 (62 %)	0.6/15.7 %	**N-Acetyl glyphosate** AAZ [1]: 0.8 CV^* [1]: 23.2 %	> 2 years	0 (0 %)	
	1 – 2 years	12 (13 %)	1.7/53.4 %		1 – 2 years	1 (6 %)	
	< 1 year	22 (24 %)	1.0/33 %		< 1 year	14 (88 %)	0.7/24.2 %
	no data	1 (1 %)			no data	1 (6 %)	
Haloxyfop AAZ [1]: 0.7 CV^* [1]: 13.9 %	> 2 years	87 (86 %)	0.6/13.8 %				
	1 – 2 years	5 (5 %)	0.5/19.1 %				
	< 1 year	7 (7 %)	1.4/30.2 %				
	no data	2 (2 %)					

ADDITIONAL COMPOUNDS			
Pesticides	**Experience**	**No. of Labs [2] (%)**	**AAZ/CV^* [2]**
Captan (sum) AAZ [1]: 0.8 CV^* [1]: 25.2 %	> 2 years	35 (54 %)	0.9/27.5 %
	1 – 2 years	2 (3 %)	
	< 1 year	28 (43 %)	0.7/19.5 %
	no data	0 (0 %)	
Folpet (sum) AAZ [1]: 0.8 CV^* [1]: 21.1 %	> 2 years	33 (50 %)	0.8/17.2 %
	1 – 2 years	3 (5 %)	
	< 1 year	30 (45 %)	0.9/22.8 %
	no data	0 (0 %)	
THPI AAZ [1]: 1.1 CV^* [1]: 30.5 %	> 2 years	36 (53 %)	1.2/34 %
	1 – 2 years	5 (7 %)	0.6/21.7 %
	< 1 year	27 (40 %)	1.1/33.5 %
	no data	0 (0 %)	
Phthalimide AAZ [1]: 0.9 CV^* [1]: 21.6 %	> 2 years	33 (49 %)	0.9/19.6 %
	1 – 2 years	7 (10 %)	1.4/39 %
	< 1 year	27 (40 %)	0.9/25.6 %
	no data	1 (1 %)	

1) based on participants from EU and EFTA countries
2) based on all participants
3) Assigned value uncertain, AAZ for informative purpose only

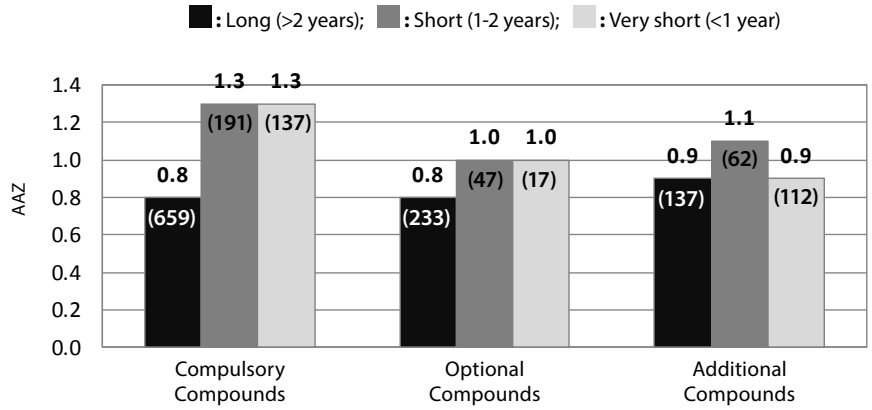


Figure 4-5: Correlation between the labs' experience with the analytes and the AAZ. (No. of data in each case in parentheses, excluding carbofuran and laboratories without data on experience with the analysis)

4.5.6 Size of Analytical Portions

Figure 4-6 (p. 92) gives an overview of the analytical sample sizes employed by the participants in this exercise. The majority of the laboratories (82 %) employed analytical portions equal or larger than 10 g, the size of the analytical portion used by the organisers in the homogeneity test for all compounds except dithiocarbamates where 20 g were used. In the case of ***dithiocarbamates*** 41 % laboratories used analytical portions smaller than 20 g (the sample weight used in the homogeneity test). In the case of ***bromide ion*** the share of the laboratories employing analytical portions < 10 g (the amount used in the homogeneity test) was even 47 %. The participating laboratories were informed in advance via the Specific Protocol about the sample sizes used in the homogeneity tests and that sufficient homogeneity cannot be always guaranteed where the analytical portions employed are significantly smaller than those used in the homogeneity test. To get an additional impression of the sub-sample variability of the test item when small analytical portions are used, an additional homogeneity test was conducted for some of the analytes contained in the test item (***2,4-D***, ***dithianon***, ***haloxyfop***, ***bifenazate***). For this analytical portions of 1 g and 10 g (10 each) were taken from one Test Item bottle. The relative standard deviation of results at 1 g were in general higher than those at 10 g sample size, but they did not exceed 10 %. This indicates sufficient homogeneity even in case of sample sizes of only 1 g.

4.5.7 Comparison of Reporting Limits, Assigned Values and MRRLs

Figure 4-7 (p. 93) shows the distribution of the reporting limits (RLs) reported by the participating laboratories for each of the compounds present in the test item.

Among the compulsory compounds present in the test item, the respective MRRLs were not met by the participating laboratories in 147 out of 819 cases (18 %). In one case of ***captan (parent)***, one case of ***fenbutatin oxide*** and two cases of ***dithiocarbamates***, the laboratories' RLs were even higher than the assigned values of the corresponding analytes. In two cases the participants were not able to analyse *abamectin* and *ethephon* due to RLs exceeding the MRRLs. Among the optional compounds the MRRLs were not met in 79 out of 380 cases (21 %). Due to RLs being higher than the MRRLs, 5 laboratories were not able to analyse for ***carbofuran (part of sum)*** (3×), ***N-acetyl glyphosate*** (1×), and *AMPA* (1×). Notably, 32 out of the 77 laboratories (42 %) having ana-

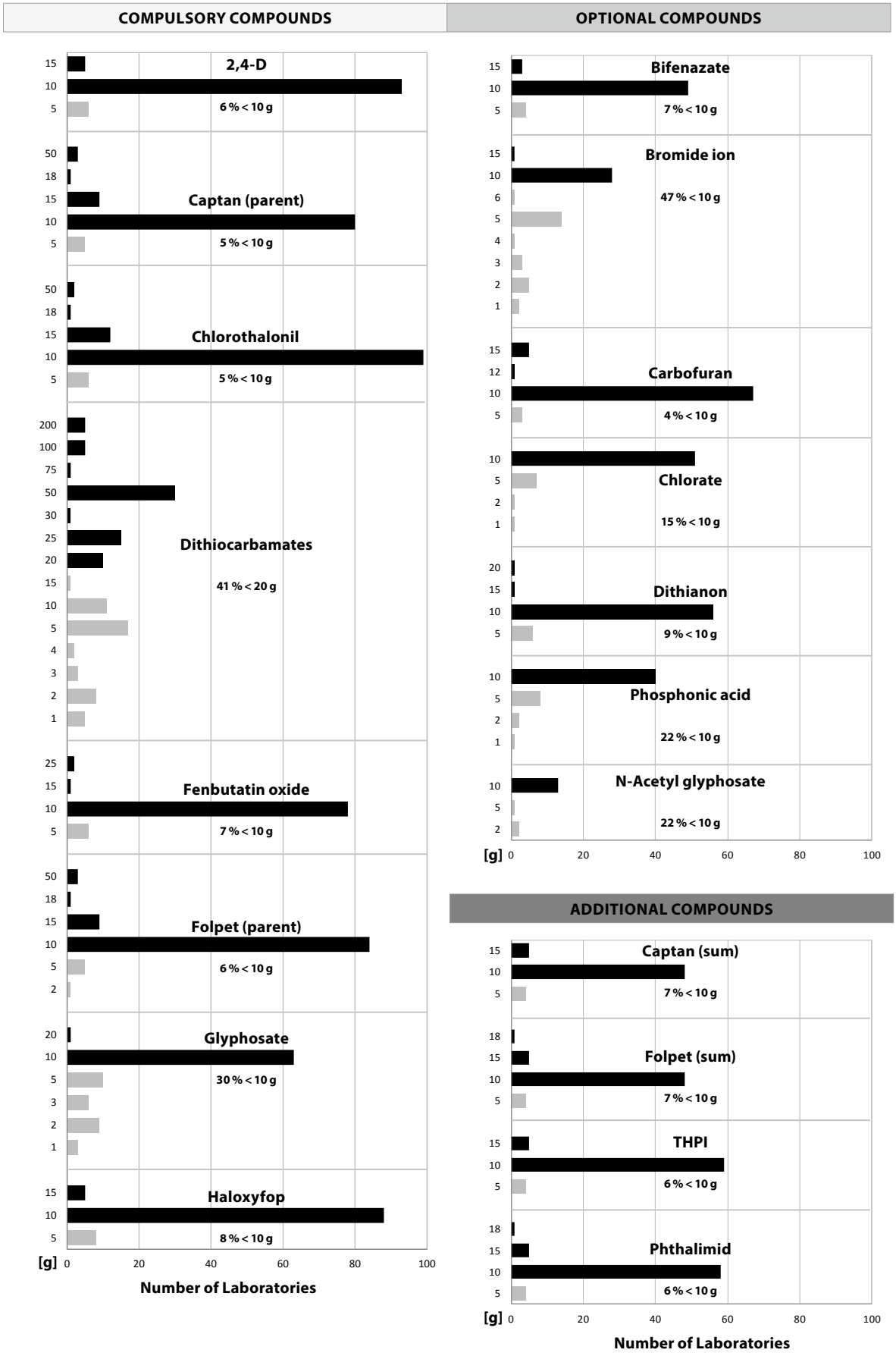


Figure 4-6: Size of analytical portions [g] employed by labs and percentage of cases where the analytical portions were smaller than those used to test homogeneity by the organiser

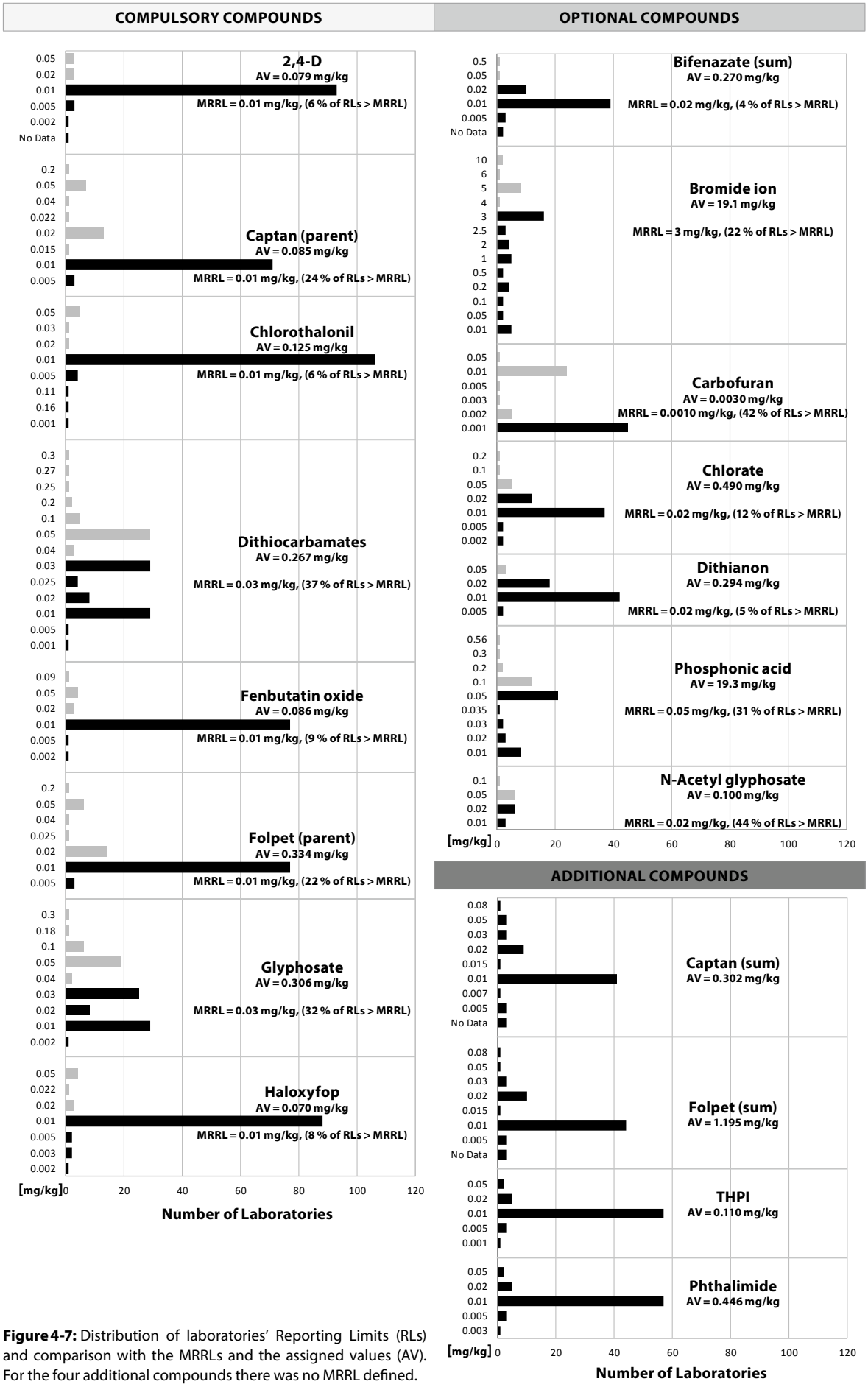


Figure 4-7: Distribution of laboratories' Reporting Limits (RLs) and comparison with the MRRLs and the assigned values (AV). For the four additional compounds there was no MRRL defined.

lysed for ***carbofuran (part of sum)*** were not able to meet the MRRL at 0.001 mg/kg, the RLs of 26 laboratories were even higher than the robust mean of the total population (0.0030 mg/kg) with 18 of them reporting not detected and receiving a "false negative" judgement. The organisers encourage the laboratories to improve their methods and are willing to assist them in this process.

4.6 Critical Points in this PT and Post-PT Advices to Participants

- To avoid bias it is important to compensate for strongly deviating recovery rates and matrix effects:
  - ◊ Strongly deviating recovery rates: Employ procedures that adjust results for recovery (e.g. ILIS added at the beginning of the procedure, standard addition to sample portions, procedural calibration); these approaches also correct for matrix effects.
  - ◊ Significant matrix effects: Use either the above mentioned procedures that also correct for recovery or procedures that compensate for matrix effects only (e.g. matrix-matched calibrations, ILISs added to the sample extract, standard addition to extract aliquots, analyte protectants in GC)
- When ***carbofuran (part of sum)*** is to be analysed, it is advised to perform the transformation of carbosulfan, benfuracarb and furanthiocarb into carbofuran. This reduces the number of different compounds to be analysed and also the possibility of false negatives. A simple method entailing an acidic hydrolysis with sulfuric acid directly in the final QuEChERS extract has been published in the EURL-SRM website (Method SRM-33),
- When ***bifenazate (sum)*** is to be analysed, it is advised to perform a transformation of bifenazate diazene into bifenazate. A method involving a reductive transformation with ascorbic acid directly in the QuEChERS final extract can be found in the EURL-SRM website (Method SRM-34)
- When analysing for ***captan (sum)*** and ***folpet (sum)*** or for ***THPI*** and ***phthalimide*** be aware of the decomposition of ***captan*** and ***folpet*** in the hot GC-injector and the formation of ***THPI*** and ***phthalimide***. Be careful with the calculations to avoid that the concentration of the degradation is overestimated. A possible procedure for the quantification of ***captan (sum)*** and ***folpet (sum)*** can be found in the EURL-SRM website (Method SRM-07).
- Make sure that the analytical portion is not too small as this increases portion-to-portion variability. If the methodology used requires the use of small analytical portions, repetitive analysis and averaging can reduce the influence of subsampling variability.
- Where possible, consider reducing the portion size of homogenized samples for ***dithiocarbamates*** analysis as the amount of Test Material that can be provided to the participants is limited.
- Consider improving the sensitivity and reducing the reporting limit of ***carbofuran***.
- Always refer to the analyte definition stated on the Target Pesticides List. For example, if the "free acid" without hydrolysis is asked, no hydrolysis step should be performed
- Always submit all methodological data requested and check their correctness and plausibility. Posterior corrections of missing or contradictory input is time consuming and delays the publication of the final report.
- Follow the instruction and details in the invoice for payment and indicate the complete invoice number as payee identification text. Otherwise it is not possible to identify the payer and the payment cannot be allocated.
- When analysing bromide by LC-MS/MS keep in mind to employ a very large fragmentation energy to reduce mass spectrometric interferences by other compounds. Detailed instructions in this regard can be found in the QuPPe procedure.
- The organisers would like to appeal to all laboratories to gradually expand their scope so that more SRM compounds are covered. Where possible and reasonable, specialized laboratories may be established to cover SRM compounds on a subcontract basis, both in commodities of animal and plant origin.

4.7 Survey to Collect the Participant's Feedback on EUPT-SRM12

In order to continuously improve the quality of the PTs and to better satisfy the participants' requirements, a survey on the EUPT-SRM12 was launched together with the release of the preliminary report on 11 May, 2017. The survey was conducted with the help of the "EU-Survey" platform with 127 of the 139 invited participants (91 %) taking part. Besides some critical points and some valuable suggestions that have to be considered in future EUPTs, lots of positive comments were received. A compilation of the results and the organiser's reactions were published on 27 July, 2017 and can be downloaded via the link: http://www.eurl-pesticides.eu/library/docs/srm/SRM12_Survey_Statistics_Evaluation.pdf

4.8 Summary, Conclusions, Retrospect and Prospect

The EUPT-SRM12 was the 12th scheduled EUPT focusing on pesticides requiring the use of "single" residue methods.

A total of 129 laboratories representing 28 EU and 3 EFTA countries registered for the EUPT-SRM12. In addition, two laboratories from one EU-candidate country and seven from third countries registered for participation. All of them submitted results. Croatia was the only EU-country not represented by an NRL-SRM. Malta was represented by its proxy-NRL-SRM based in the United Kingdom. For the first time one OfL from Iceland has participated in an EUPT-SRM.

Compared to the previous EUPT-SRMs using fruit and vegetables as commodity the number of laboratories that participated in this EUPT has increased significantly (**Table 4-25, p. 96**). It should be noted that participation in EUPTs mainly depends on the compounds included in the Target Pesticides List as well as the matrices concerned. The number of participants in EUPT-SRMs based on fruit or vegetables is generally higher compared to PTs using cereals or feeding stuff as matrix. EUPTs entailing target compounds which are included in the scope of many laboratories, such as ***dithiocarbamates***, also tend to show a higher number of participants (**Table 4-26, p. 98**).

The EUPT-SRM12 was the most successful one as regards the number of participants and the average number of compounds analysed per participant (**Table 4-25**). Also in terms of performance the EUPT-SRM12 was the most successful among the EUPT-SRMs so far, with the result distribution being in most cases quite narrow (average CV^* excluding carbofuran 21 %). The percentage of laboratories classified into Category A was also relatively high.

The Target Pesticides List of EUPT-SRM12 (**Appendix 10**) contained in total 25 SRM-compounds. 13 of them were compulsory, and 12 were optional for the laboratories in terms of scope, four of them were only for the purpose of data collection and were assigned as "additional compounds". All of the compulsory and additional compounds were relevant to the EU multiannual coordinated control program (MACP) for strawberry and listed in the MACP regulation. Four of the eight optional compounds were included in the MACP regulation and the other four were included in the MACP working document giving guidance to the EU Member States for designing the national monitoring programs.

8 of total 25 analytes in the Target Pesticides List were included for the first time in the EUPT-SRM with 7 of them being present in the test item: ***bifenazate (sum)***, ***carbofuran (part of sum)***, ***folpet (parent)***, ***N-acetyl glyphosate***, ***captan (sum)***, ***THPI***, ***folpet (sum)*** and ***phthalimide***. All these new compounds were analysed by a sufficient number of laboratories to allow proper statistical evaluation. Although only 15 laboratories reported a numerical result for ***N-acetyl glyphosate***, the CV^* of 23.2 % indicates the high analytical quality of these laboratories. Similar observations with compounds analysed by only a few, but obviously well performing, laboratories were also made in several past PTs.

Phosphonic acid and ***chlorate*** have been in the focus of pesticide residue laboratories for some years. It is pleasing to see that the number of laboratories covering these compounds in EUPTs has increased: from 46 in SRM11 to 60 in SRM12 for ***chlorate*** and from 40 in SRM11 to 50 SRM12 for ***phosphonic acid***. To enable simple and still accurate quantitative analysis the EURL-SRM synthesized and provided the ILISs of both compounds to all participants. As shown by the robust standard deviations (CV^*s), the distribution of results obtained by using ILIS was clearly narrower (***chlorate***: CV^* = 13.3 %; ***phosphonic acid***: CV^* = 19.3 %) than without using ILIS (***chlorate***: CV^* = 20.0 %; ***phosphonic acid***: CV^* = 40.7 %)

The robust relative standard deviation (CV^*) reflects the width of the result-distribution and was calculated for each target analyte. The average CV^*, which is calculated for informative purposes, was 21.2 % and 21.6 % for compulsory and optional compounds excluding carbofuran, respectively, and was thus clearly lower than the FFP-RSD of 25 % used to calculate the z-scores. The individual CV^* values of the compulsory

Table 4-25: Retrospective comparison of EUPT-SRMs (Statistical evaluation based on data from laboratories in EU and EFTA countries)

EUPT-	SRM1 (2007)	SRM2 (2008)	SRM3 (2009)	SRM4 (2009)	SRM5 (2010)	SRM6 (2011)
Test Item (Commodity)	Apple juice	Wheat flour	Carrot homogenate	Oat flour	Apple purée	Rice flour
Participants submitting results (EU/EFTA)	24	30	66	48	81	77
Participants submitting results (3rd and EU candidate countries)	–	–	–	–	2	2
Compounds in Target Pesticides List Compulsory / Optional	15 / –	8 / 3	8 / –	13 / 8	11 / –	13 / –
Compounds in test item Compulsory / Optional	3 [1] / –	3 / 2	5 / –	5 [2] / 2	5 [3] / –	7 / –
No. of results without false positives Compulsory / Optional	38 / –	56 / 22	193 / –	95 / 47	239 / –	291 / –
No. of false negative results Compulsory / Optional	0 / –	1 / 0	0 / –	3 / 2	5 / –	5 / –
Mean no. of results per lab Compulsory / Optional	1.58 / –	1.87 / 0.73	2.92 / –	1.97 / 0.98	2.95 / –	3.79 / –
Average of absolute z-scores (AAZ) Compulsory / Optional	0.57 / –	1.13 / 0.67	1.04 / –	0.98	1.11 / –	0.83 / –
Acceptable z-scores Compulsory / Optional	97 % / –	81 % / 100 %	87 % / –	89 % / 88 %	92 % / –	91 % / –
Questionable z-scores Compulsory / Optional	– / –	9 % / 0 %	7 % / –	5 % / 6 %	3 % / –	6 % / –
Unacceptable z-scores Compulsory / Optional (thereof false negatives)	3 % / –	10 % / 0 % (1.8 % / 0 %)	6 % / –	6 % / 6 % (3.7 % / 4 %)	5 % / – (0.6 % / –)	4 % / – (1.7 % / –)
Number of false positives Compulsory / Optional	0 / –	1 / –	0 / –	0 / –	3 / 3	0 / –
Category A laboratories [8]	–	–	–	31 %	19 %	25 %
CV^* (average) [9] Compulsory / Optional	25 % / –	37 % / 22 %	28 % / 24 %	27 %	22 % / –	23 % / –

1) One compound (fenbutatin oxide) was evaluated for information only due to insufficient number of participants.
2) Two compounds (ethephon and glyphosate) were evaluated for information only due to insufficient number of participants.
3) One compound (dithiocarbamates as CS_2) was evaluated for information only due to uncertain assigned value.
4) Three compounds (chlorothalonil, cyromazine and fenbutatin oxide) were evaluated for information only due to uncertain assigned value.
5) Two compounds (4-OH-chlorothalonil and trimesium) were evaluated for information only due to uncertain assigned value.
6) Three compounds (tolylfluanid, dithianon and pymethrozine) were evaluated for information only due to uncertain assigned value and excluded in the evaluation
7) One compound (carbofuran) was evaluated for information only due to uncertain assigned value and excluded in the evaluation.
8) The criteria applied to define Category A and B in EUPT-SRM4 and -SRM5 were different from those in EUPT-SRM6 – 10.
9) CV^* = robust relative standard dieviation, known as Qn-RSD in EUPT-SRM1 – 9 (calculated for informative purpose)

compounds were as follows: ***2,4-D*** 13.3 %, ***captan (parent)*** 28.1 %, ***chlorothalonil*** 25.2 %, ***dithiocarbamates*** 22.2 %, ***fenbutatin oxide*** 21.0 %, ***folpet (parent)*** 25.0 %, ***glyphosate*** 20.9 % and ***haloxyfop*** 13.9 %. The CV^* values of the optional compounds were as follows: ***bifenazate*** 22.1 %, ***bromide ion*** 16.0 %, ***carbofuran (part of sum)*** 47.1 % and excluded from the evaluation, ***chlorate*** 16.2 %, ***dithianon*** 25.3 %, ***phosphonic acid*** 27.0 % and ***N-acetyl glyphosate*** 18.6 %. The CV^* values for the four additional compounds ***captan (parent)***, ***folpet (parent)***, ***THPI*** and ***phthalimide*** were 25.2 %, 21.1 %, 30.5 %, 21.6 %, respectively.

Looking at the long-term CV^*s of selected individual compounds or compound groups (**Table 4-26**) acidic pesticides (***2,4-D***, *MCPA*, *bentazone*, ***haloxyfop***, *fluazifop*) showed an average CV^* of 22.9 %, chlormequat and mepiquat an average CV^* of 22.1 %, ***glyphosate***, and ***ethephon*** an average CV^* of 28.0 %, ***fenbutatin oxide*** an average CV^* of 25.6 % and ***bromide ion*** an average CV^* of 14.2 %. ***Dithiocarbamates*** with an average CV^* value of 34.0 % remains the most critical analyte. The positive trend in the laboratories' proficiency

EUPT-	SRM7 (2012)	SRM8 (2013)	SRM9 (2014)	SRM10 (2015)	SRM11 (2016)	SRM12 (2017)
Matrix of test item	Lentil flour	Potato homogenate	Cow's whole milk	Maize flour	Spinach homogenate	Strawberry purée
Participants submitting results (EU/EFTA)	110	110	62	104	120	129
Participants submitting results (3rd and EU candidate countries)	4	6	5	6	2	9
Compounds in Target Pesticide List Compulsory / Optional	16 / –	13 / 10	12 / 7	9 / 14	11 / 16	13 / 8
Compounds in test item Compulsory / Optional	8 [4] / –	8 [5] / 7	8 [5] / 6	8 / 5	6 [6] / 8 [6]	8 / 7 [7]
No. of results without false positives Compulsory / Optional	439 / –	604 / 212	361 / 132	461 / 135	479 / 411	772 / 370
No. of false negative results Compulsory / Optional	11 / –	14 / 8	3 / 4	4 / 2	8 / 20	14 / 18
Mean no. of results per lab Compulsory / Optional	4.12 / –	5.49 / 1.93	5.87 / 2.19	4.43 / 1.29	4.03 / 3.71	5.98 / 2.87
Average of absolute z-scores (AAZ) Compulsory / Optional	0.97 / –	0.98 / 1.06	0.75 / 0.80	0.9 / 0.7	1.0 [6] / 1.1 [6]	0.9 / 0.9 [7]
Acceptable z-scores Compulsory / Optional	90 % / –	88 % / 85 %	92 % / 71 %	87 % / 89 %	87 % [6] / 85 % [6]	89 % / 90 % [7]
Questionable z-scores Compulsory / Optional	3 % / –	6 % / 5 %	4 % / 5 %	8 % / 6 %	5 % [6] / 4 % [6]	4 % / 5 % [7]
Unacceptable z-scores Compulsory / Optional (thereof false negatives)	7 % / – (2.1 % / –)	6 % / 10 % (2.2 % / 3.6 %)	4 % / 3.5 % (0.8 % / 2.7 %)	5 % / 4 % (0.8 % / 2.9 %)	7 % [6] / 10 % [6] (1.0 % [6] / 4.8 % [6])	7 % / 6 % [7] (1.8 % / 0.7 % [7])
Number of false positives Compulsory / Optional	0 / –	2 / 0	6 / 0	0 / 4	4 / 4	1 / 0
Category A laboratories [8]	28 %	47 %	52 %	53 %	47 %	50 %
CV^* (average) [9] Compulsory / Optional	27 % / –	26 % / 26 %	20 % / 19 %	24 % / 19 %	28 % [6] / 30 % [6]	21 % / 22 % [7]

1) One compound (fenbutatin oxide) was evaluated for information only due to insufficient number of participants.
2) Two compounds (ethephon and glyphosate) were evaluated for information only due to insufficient number of participants.
3) One compound (dithiocarbamates as CS_2) was evaluated for information only due to uncertain assigned value.
4) Three compounds (chlorothalonil, cyromazine and fenbutatin oxide) were evaluated for information only due to uncertain assigned value.
5) Two compounds (4-OH-chlorothalonil and trimesium) were evaluated for information only due to uncertain assigned value.
6) Three compounds (tolylfluanid, dithianon and pymethrozine) were evaluated for information only due to uncertain assigned value and excluded in the evaluation
7) One compound (carbofuran) was evaluated for information only due to uncertain assigned value and excluded in the evaluation.
8) The criteria applied to define Category A and B in EUPT-SRM4 and -SRM5 were different from those in EUPT-SRM6 – 10.
9) CV^* = robust relative standard dieviation, known as Qn-RSD in EUPT-SRM1 – 9 (calculated for informative purpose)

Table 4-26: Overview of selected pesticides tested in the EUPT-SRMs 1 – 12 and analysed by the participating laboratories. ***n***: Number of laboratories having analysed selected pesticides present in the test items. The figures in brackets show the percentage of laboratories submitting numerical results for a compound out of the total number of laboratories submitting results (only EU and EFTA labs considered; CV^*, formerly known as Qn, was calculated for populations with at least 10 laboratories). Only CV^*s based on 15 or more labs were used to calculated the average CV^*s at the bottom.

EUPT	No. of laboratories	Commodity type[1]		Acidic pesticides					Requiring individual methods		Polar pesticides				Other
				2,4-D	MCPA	Bentazone	Haloxyfop	Fluazifop	Bromide	Dithio-carbamates	Chlormequat	Mepiquat	Ethephon	Glyphosate	Fenbutatin oxide
SRM1	24	FV	*n*		10 (42 %)						23 (96 %)				5 (21 %)
		HW	CV^*		**27.1 %**						**13.8 %**				**–**
SRM2	30	CF	*n*		13 (43 %)						25 (83 %)				
		D	CV^*		**45.8 %**						**29.1 %**				
SRM3	66	FV	*n*		38 (58 %)			35 (55 %)		59 (89 %)					
		HW	CV^*		**27.0 %**			**26.6 %**		**38.4 %**					
SRM4	48	CF	*n*	32 (66 %)							38 (83 %)		4 (8.3 %)	6 (13 %)	
		D	CV^*	**27.5 %**							**25.8 %**		**–**	**–**	
SRM5	81	FV	*n*					51 (64 %)		70 (86 %)			28 (35 %)		35 (43 %)
		HW	CV^*					**19.8 %**		**58.9 %**			**23.0 %**		**24.3 %**
SRM6	77	CF	*n*	57 (74 %)			49 (64 %)		34 (44 %)	64 (83 %)			28 (36 %)	34 (44 %)	
		D	CV^*	**22.1 %**			**17.7 %**		**8.6 %**	**24.2 %**			**29.7 %**	**40.6 %**	
SRM7	110	CF	*n*	70 (64 %)					44 (40 %)	83 (75 %)			32 (29 %)	39 (35 %)	
		D	CV^*	**27.9 %**					**18.0 %**	**23.1 %**			**25.2 %**	**34.5 %**	
SRM8	110	FV	*n*				81 (74 %)					71 (65 %)		45 (41 %)	59 (54 %)
		HW	CV^*				**20.2 %**					**22.2 %**		**24.5 %**	**31.4 %**
SRM9	62	AO	*n*	50 (81 %)				50 (81 %)			50 (81 %)	49 (79 %)			
		HW	CV^*	**18.7 %**				**26.0 %**			**29.8 %**	**19.6 %**			
SRM10	104	CF	*n*	82 (79 %)	79 (76 %)	69 (66 %)				85 (82 %)	75 (72 %)	76 (67 %)	61 (59 %)	62 (60 %)	
		D	CV^*	**18.2 %**	**18.9 %**	**18.5 %**				**36.9 %**	**18.2 %**	**18.5 %**	**30.8 %**	**22.8 %**	
SRM11	119	FV	*n*							95 (80 %)					
		HW	CV^*							**34.6 %**					
SRM12	129	FV	*n*	98 (76 %)			97 (65 %)		52 (50 %)	107 (83 %)				86 (67 %)	82 (64 %)
		HW	CV^*	**13.3 %**			**13.9 %**		**16.0 %**	**22.2 %**				**20.9 %**	**21.0 %**
Average CV^* EUPT-SRMs 1 – 12				**21.3 %**	**23.0 %**	**18.5 %**	**17.3 %**	**24.1 %**	**14.2 %**	**34.0 %**	**23.3 %**	**20.1 %**	**27.2 %**	**28.7 %**	**25.6 %**

Average CV^* of Group EUPT-SRMs 1 – 12	**Acidic pesticides**	**Br⁻**	**Dithiocarbamates**	**Chlormequat + Mepiquat**	**Ethephon + Glyphosate**	**FBO**
	21.1%	**14.2 %**	**34.0 %**	**22.1 %**	**28.0 %**	**25.6 %**

1) Commodity type:
HW: High water content; D: dry = high strach or high protein content and low water content

can be seen clearly in the analysis of ***glyphosate***: the number laboratories having analysed for ***glyphosate*** increased from 6 in EUPT-SRM4 to 34 in EUPT-SRM6 to 39 in EUPT-SRM7 to 45 in EUPT-SRM8 to 62 in EUPT-SRM10 and finally to 86 in the current PT with the CV^* decreasing from 40.6 % (EUPT-SRM6), 34.5 % (EUPT-SRM7), 24.5 % (EUPT-SRM8), 22.8 % (EUPT-SRM10) to 20.9 % (SRM12).

In accordance with the definition in the General EUPT Protocol, z-scores based on the FFP-RSD of 25 % were calculated and classified into "acceptable", "questionable", and "unacceptable" for each laboratory/target-

analyte combination. Overall, the performance of the laboratories was very high. In the case of compulsory compounds 93 out of 98 laboratories (95 %) reported results within the acceptable z-score-range for ***2,4-D***, 77 out of 93 (83 %) for ***captan (parent)***, 97 out of 111 (87 %) for ***chlorothalonil***, 101 out of 107 (94 %) for ***dithiocarbamates***, 72 out of 87 (88 %) for ***fenbutatin oxide***, 82 out of 98 (84 %) for ***folpet (parent)***, 77 out of 86 (90 %) for ***glyphosate*** and 89 out of 97 (92 %) for ***haloxyfop***. In the case of optional compounds 52 out of 54 laboratories (96 %) submitted results within the acceptable z-score-range for ***bifenazate (sum)***, 47 out of 52 (90 %) for ***bromide ion***, 44 out of 74 (59 %) for ***carbofuran***, 54 out of 60 (90 %) for ***chlorate***, 54 out of 64 (84 %) for ***dithianon***, 43 out of 50 (86 %) for ***phosphonic acid*** and 15 out of 16 (94 %) for ***N-acetyl glyphosate***. In the case of additional compounds, where evaluation was only done for informative purposes, 61 out of 65 laboratories (94 %) submitted results within the acceptable z-score-range for ***captan (sum)***, 58 out of 66 (88 %) for ***folpet (sum)***, 58 out of 67 (87 %) for ***THPI*** and 61 out of 67 (97 %) for ***phthalimide***.

Considering results reported by all participating laboratories, false negative results were reported by EU/EFTA-laboratories in 14 cases among the compulsory compounds (4× ***fenbutatin oxide***, 2× ***captan (parent)***, 2× ***folpet (parent),*** 2× ***chlorothalonil,*** 2× ***haloxyfop,*** 1× ***glyphosate*** and 1× ***2,4-D***). Among the optional compounds false negative results were reported in 20 cases (18× ***carbofuran***, 1× ***dithianon***, 1× ***N-acetyl glyphosate***). Among the additional compounds there was one result for ***phthalimide*** judged as false negative. One numerical result for *propamocarb* was reported by an EU/EFTA-laboratory and judged as false positive. Two other false positive results (*abamectin* and *cyromazine*) were reported by the participants from third countries.

All participating laboratories were classified into category A and B following the rules in the General EUPT Protocol. Laboratories analysing at least 12 of the 13 the compulsory compounds and correctly detecting at least seven of the eight compulsory pesticides present in the test item without reporting any false positive result were classified into Category A. A total of 64 EU/EFTA-laboratories (50 %) were classified into Category A and the remaining 65 (50 %) laboratories into Category B. Among the 9 participating laboratories from third countries, three were classified into Category A and the other 6 into Category B.

16 of the 125 EU laboratories that registered for participation in this EUPT participated on a voluntary basis. The other 109 participating laboratories represent 74 % of the 147 laboratories that were finally considered as being obliged to participate in this exercise based on their function (NRL-SRM) or scope (routinely analysing official samples for pesticide residues in fruit and vegetable). 38 laboratories (26 %) that were considered as obliged to participate in the current PT had neither registered for non-participation nor stated any reason.

Post-PT measures and assistance to the laboratories: Following the distribution of the preliminary results all laboratories achieving questionable or unacceptable z-scores as well as false positive results were asked to investigate the reasons and report them to the organisers, as far as possible. 57 laboratories responded to the organisers with (possible) reasons for their poor performance in 123 cases. In 20 of those case the real reasons could not be clarified, in spite of intensive investigation. The most frequently reported error sources were “error in the concentration of analytical standards or calibration solutions” (24 cases), “lack of experience” (14 cases) and “transcription or administrative errors” (14 cases). “Use of inappropriate procedure”, “error in the evaluation or in the interpretation of measurement data” and “application of inappropriate calibration” were reported as reasons for the poor performance by in 9 cases each. “Technical problems with measurement instrumentation” like poor sensitivity (8 cases), “matrix effect was not properly compensated” (6 cases) and “result not corrected for low or high recovery” (5 cases) were other frequent error sources. The other reported reasons for the poor performance were: “procedure not properly conducted” (4 cases), “Strong chromatographic interferences” (3 cases), “detection signals strongly interfered by matrix components” and “degradation during sample preparation or measurement“ (2 cases each) as well as “misunderstanding of the residue definition of the analyte” (1 case). In 4 cases concerning ***captan (parent)*** and

THPI as well as ***folpet (parent)*** and ***phthalimide*** the laboratories reported problems with the conversion of the parent compounds into the metabolites which introduced an error in the final quantification. Even if in many cases the laboratories did not give any feedback to the organiser for their poor performance, the organisers hope that every participating laboratory has tried to find out the reason, as this will reduce errors in the future and improve analytical quality.

Expanding the scope and improving the overall performance of NRLs and OfLs in the area of pesticides and metabolites not amenable to multiresidue methods is one of the main aims of the EURL-SRM. The EURL-SRM is thus pleased to assist the laboratories via bilateral discussions, workshops and trainings and will continue developing, validating and distributing easy-to-use, fast and cost-efficient methodologies for such compounds. In future PTs, the selection of target analytes will continue to focus on those included in the scope of the EU coordinated control programs as well as on additional pesticides and metabolites of high relevance. Specific requests by NRLs and OfLs stated in the survey on the EUPT-SRM12 will be also taken into account.

5. ACKNOWLEDGEMENTS

The organisers wish to thank the members of the EUPT Scientific Committee (Quality Control Group and Advisory Group) for their valuable advice. Special thanks also go to Jens-Ole Frimann for his support in establishing the online result submission tool.

6. REFERENCES

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[5] http://quppe.eu/

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7. APPENDICES

Appendix 1 List of Laboratories Registered to Participate in the EUPT-SRM12

(a): participating labs of EU and EFTA Member States

Country (Location)	Analysed on behalf of	Institution	City	NRL*-SRM	Reported results
Austria	AT	AGES Innsbruck - Food Safety Institute	Innsbruck	x	Yes
Austria	AT	Lebensmitteluntersuchung Wien	Vienna		Yes
Austria	AT	LVA GmbH	Klosterneuburg		Yes
Belgium	BE	LOVAP NV	Geel		Yes
Belgium	BE	WIV-ISP (Scientific Institute of Public Health)	Brussels	x	Yes
Belgium	BE/BG/ FR/LU	Primoris Belgium	Gent - Zwijnaarde		Yes
Bulgaria	BG	CLCTC	Sofia	x	Yes
Croatia	HR	Bioinstitute Ltd.	Cakovec		Yes
Croatia	HR	CNIPH	Zagreb		Yes
Croatia	HR	Euroinspekt-Croatiakjontrola Laboratory	Zagreb		Yes
Croatia	HR	Inspecto d.o.o. Laboratorij	Osijek		Yes
Croatia	HR	Teaching Institute of Public Health, Dr. Andrija Štampar	Zagreb		Yes
Cyprus	CY	Laboratory of Pesticide Residues Analysis, State General Laboratory, Cyprus	Nicosia	x	Yes
Czech Republic	CZ	Central Institute for Supervising and Testing in Agriculture	Brno		Yes
Czech Republic	CZ	Czech Agriculture and Food Inspection Authority	Prague	x	Yes
Czech Republic	CZ	UCT Prague, Metrological and Testing laboratory	Praha 6		Yes
Denmark	DK	Danish Veterinary and Food Administration	Ringsted	x	Yes
Estonia	EE	Agricultural Research Centre, Laboratory for Residues and Contaminants	Saku		Yes
Estonia	EE	Tartu Laboratory of Health Board	Tartu	x	Yes
Finland	FI	Finnish Customs Laboratory	Espoo	x	Yes
Finland	FI	MetropoliLab Ltd	Helsinki		Yes
France	FR	Analysis Center Mediterranean Pyrenees (Perpignan)	Perpignan		Yes
France	FR	ANSES-PBM	Maisons-Alfort Cedex	x	Yes
France	FR	CAPINOV	Landerneau		Yes
France	FR	CERECO SUD	GARONS		Yes
France	FR	FREDON Pays de la Loire / GIRPA	BEAUCOUZE		Yes
France	FR	INOVALYS - Le MANS	Le Mans		Yes
France	FR	Laboratoire du SCL de Montpellier	Montpellier		Yes
France	FR	SCL Ile de France - Massy	Massy Cedex		Yes
France	BE	PHYTOCONTROL	NIMES		Yes
Germany	FR	Intertek Food Services GmbH Bremen	Bremen		Yes
Germany	BE	LUFA-ITL GmbH, Kiel	Kiel		Yes
Germany	DE	Amt für Verbraucherschutz Düsseldorf - Chemische and Lebensmitteluntersuchung	Duesseldorf		Yes
Germany	DE	Chemical and Veterinary Analytical Institute Rhine-Ruhr-Wupper	Krefeld		Yes
Germany	DE	Chemisches Labor Dr. Mang	Frankfurt am Main		Yes
Germany	DE	Chemisches and Veterinäruntersuchungsamt Münsterland Emscher-Lippe	Münster		Yes
Germany	DE	Federal Office of Consumer Protection and Food Safety, NRL for Pesticide Residues	Berlin-Marienfelde	x	Yes

* only for EU-Member States

Appendix 1-a (cont.): participating labs of EU and EFTA member states

Country (Location)	Analysed on behalf of	Institution	City	NRL*-SRM	Reported results
Germany	DE	Hessisches Landeslabor Kassel	Kassel		Yes
Germany	DE	Institut für Hygiene and Umwelt	Hamburg		Yes
Germany	DE	KWALIS Qualitätsforschung Fulda GmbH; D-36160 Dipperz	Dipperz		Yes
Germany	DE	Labor Friedle GmbH	Tegernheim		Yes
Germany	DE	Landesuntersuchungsamt Institut für Lebensmittelchemie Speyer	Speyer		Yes
Germany	DE	Landesuntersuchungsanstalt für das Gesundheits- und Veterinärwesen Sachsen, Standort Dresden	Dresden		Yes
Germany	DE	Landwirtschaftliches Technologiezentrum Augustenberg	Karlsruhe		Yes
Germany	DE	LAV Sachsen-Anhalt	Halle/Saale		Yes
Germany	DE	LGL Erlangen	Erlangen		Yes
Germany	DE	LLBB Frankfurt (Oder)	Frankfurt (Oder)		Yes
Germany	DE	Niedersächsisches Landesamt für Verbraucherschutz and Lebensmittelsicherheit, LVI Oldenburg	Oldenburg		Yes
Germany	DE	State Laboratory SH	Neumünster		Yes
Germany	DE	State Office for Agriculture, Food Safety and Fisheries - MV	Rostock		Yes
Germany	DE/MT	Eurofins Dr. Specht Laboratorien GmbH	Hamburg		Yes
Germany	LT	GALAB Laboratories GmbH	Hamburg		Yes
Greece	GR	Agrolab rds SA	Thessaloniki		Yes
Greece	GR	Benaki Phytopathological Institute, Pesticide Residue Laboratory.	Kifissia	x	Yes
Greece	GR	General Chemical State Laboratory	Athens	x	Yes
Hungary	HU	National Food Chain Safety Office, Food Chain Safety Centre Non-profit Ltd. Pesticide Residue Analytical Laboratory, Miskolc	Miskolc		Yes
Hungary	HU	National Food Chain Safety Office, Food Chain Safety Centre Non-profit Ltd., Pesticide Residue Analytical Laboratory, Hódmezővásárhely	Hódmezovásárhely		Yes
Hungary	HU	National Food Chain Safety Office, Pesticide Analytical Laboratory, Velence	Velence	x	Yes
Hungary	HU	National Food Chain Safety Office; Food Chain Safety Centre Non-profit Ltd.; Pesticide Residue Analytical Laboratory, Szolnok	Szolnok		Yes
Hungary	HU	WESSLING Hungary Ltd.	Budapest		Yes
Iceland	IS	Matis ltd.	Reykjavík		Yes
Ireland	IE	The Pesticide Control Laboratory	Co. Kildare	x	Yes
Italy	IT	APPA Bolzano	Bolzano		Yes
Italy	IT	ARPA Puglia - Polo alimenti Bari	Bari		Yes
Italy	IT	ARPAE Ferrara Laboratorio Tematico Fitofarmaci	Ferrara		Yes
Italy	IT	ARPAV Verona	Verona		Yes
Italy	IT	Departement Environmental and Health - Pesticide Section	Rome	x	Yes
Italy	IT	Istituto Zooprofilattico dell'Abruzzo e del Molise G.Caporale Teramo Italy	Teramo		Yes
Italy	IT	IZSLT department of Florence	Florence		Yes
Italy	IT	Laboratorio Contaminanti Ambientali - Istituto Zooprofilattico Sperimentale dell'Umbria e delle Marche - Perugia Italy.	Perugia		Yes
Italy	IT	Laboratorio di Sanità Pubblica USL Toscana centro	Firenze		Yes
Italy	IT/MT	IZSLER	Brescia		Yes
Latvia	LV	Research Institute BIOR	Riga	x	Yes
Lithuania	LT	National food and veterinary risk assessment institute	Vilnius	x	Yes
Luxembourg	LU	National Laboratory of Health - Food Laboratory	Dudelange	x	Yes

* only for EU-Member States

Appendix 1-a (cont.): participating labs of EU and EFTA member states

Country (Location)	Analysed on behalf of	Institution	City	NRL*-SRM	Reported results
The Netherlands	NL	NVWA - Netherlands Food and Consumer Product Safety Authority	Wageningen	x	Yes
The Netherlands	BE/NL	LZV20171043	Graauw		Yes
The Netherlands	BE	Dr. A. Verwey B.V.	Rotterdam		Yes
The Netherlands	BE	Groen Agro Control	Delfgauw		Yes
The Netherlands	BE	Nofalab B.V	Schiedam		Yes
Norway	NO	NIBIO, Biotechnology and Plant Health, Pesticides and Natural Products Chemistry	Aas		Yes
Poland	PL	Department of Pesticide Residue Research, Institute of Plant Protection - National Research Institute	Poznan		Yes
Poland	PL	Food Safety Laboratory/Research Institute of Horticulture	Skierniewice		Yes
Poland	PL	Institute of Plant Protection -National Research Institute, Branch Sosnicowice, Laboratory of Pesticide Residue Research	Sosnicowice		Yes
Poland	PL	Institute of Plant Protection-National Research Institute, Laboratory of Pesticide Residue Analysis, Bialystok	Bialystok		Yes
Poland	PL	Pesticide Residues Laboratory VSES in Warsaw	Warszawa	x	Yes
Poland	PL	Voievodship Sanitary - Epidemiological Station in Wroclaw	Wroclaw		Yes
Poland	PL	Wojewódzka Stacja Sanitarno-Epidemiologiczna w Opolu, Oddział Laboratoryjny w Kluczborku	Kluczbork		Yes
Poland	PL	WSSE LODZ	Lodz		Yes
Portugal	PT	INIAV – Laboratório de Resíduos de Pesticidas - Oeiras	Oeiras		Yes
Portugal	PT	LCCP / INIAV -VAIRAO	Vairão - Vila do Conde		Yes
Portugal	PT	Regional Laboratory of Veterinary and Food Safety	Funchal Madeira Island	x	Yes
Romania	RO	Central Laboratory for Pesticides Residues Control in Plants and Vegetable Products-Bucharest	Bucharest		Yes
Romania	RO	Institute for Hygiene and Veterinary Public Health	Bucharest	x	Yes
Romania	RO	Regional Laboratory for Pesticide Residues Control in Plant and Plant Products Mures	Tirgu Mures		Yes
Slovakia	SK	Veterinary and Food Institute in Bratislava	Bratislava	x	Yes
Slovenija	SI	Agricultural Institute of Slovenia	Ljubljana		Yes
Slovenija	SI	National Laboratory of Health, Environment and Food - Maribor (location Ljubljana)	Ljubljana		Yes
Slovenija	SI	National Laboratory of Health, Environment and Foodstuffs - Maribor, Pesticide Lab - Maribor (NLZOH)	Maribor	x	Yes
Spain	ES	Agricultural and Phytopathological Laboratory of Galicia	Abegondo. A Coruña		Yes
Spain	ES	AINIA	PATERNA, VALENCIA		Yes
Spain	ES	Analytica Alimentaria GmbH, sucursal en España	Almeria		Yes
Spain	ES	CNA (AECOSAN)	Majadahonda (Madrid)	x	Yes
Spain	ES	CNTA	San Adrián (Navarra)		Yes
Spain	ES	EURL-FV University of Almería	La Cañada de San Urbano-Almería		Yes
Spain	ES	EUROFINS SICA AGRIQ	VÍCAR (ALMERIA)		Yes
Spain	ES	Instituto Tecnológico de Canarias, S. A. Laboratorio de Residuos. Departamento de Análisis Ambiental	Agüimes, Las Palmas		Yes
Spain	ES	Lab. Agroalimentario y de Sanidad Animal	El Palmar-Murcia		Yes
Spain	ES	Laboratori Agència de Salut Pública de Barcelona	Barcelona		Yes

* only for EU-Member States

Appendix 1-a (cont.): participating labs of EU and EFTA member states

Country (Location)	Analysed on behalf of	Institution	City	NRL*-SRM	Reported results
Spain	ES	LABORATORIO AGRARIO REGIONAL. JUNTA DE CASTILLA Y LEON	Burgos		Yes
Spain	ES	Laboratorio Agroalimentario de Extremadura.	Cáceres		Yes
Spain	ES	Laboratorio Agroalimentario de Zaragoza	Zaragoza		Yes
Spain	ES	Laboratorio Agroalimentario Valencia	Burjassot-Valencia		Yes
Spain	ES	LABORATORIO ANALÍTICO BIOCLÍNICO SLU	ALMERÍA		Yes
Spain	ES	Laboratorio Arbitral Agroalimentario Madrid	Madrid	x	Yes
Spain	ES	Laboratorio de Producción y Sanidad Vegetal de Almería	La Mojonera (Almería)		Yes
Spain	ES	LABORATORIO DEL SERVICIO DE INSPECCION SOIVRE	Valencia		Yes
Spain	ES	LABORATORIO KUDAM	Pilar de la Horadada		Yes
Spain	ES	Laboratorio Salud Pública Ayuntamiento de Madrid. Madrid Salud	Madrid		Yes
Spain	ES	LABORATORIOS ECOSUR, S.A.	Lorquí (Murcia)		Yes
Spain	ES	Labs & Technological Services AGQ, S.L.	Burguillos (Sevilla)		Yes
Spain	ES	LPSV JAEN	Mengibar (Jaén)		Yes
Sweden	SE	Eurofins Food&Feed Testing Sweden AB	Lidkoping		Yes
Sweden	SE	National Food Agency	Uppsala	x	Yes
Switzerland	CH	Cantonal Office of Consumer Protection Aargau	Aarau		Yes
Switzerland	CH	Kantonales Labor Zürich	Zurich		Yes
Unied Kingdom	UK/MT	Fera Science Ltd	York	x	Yes
Unied Kingdom	UK	SASA	Edinburgh		Yes
Unied Kingdom	UK	Scientific Analysis Laboratories Ltd	Bar Hill		Yes

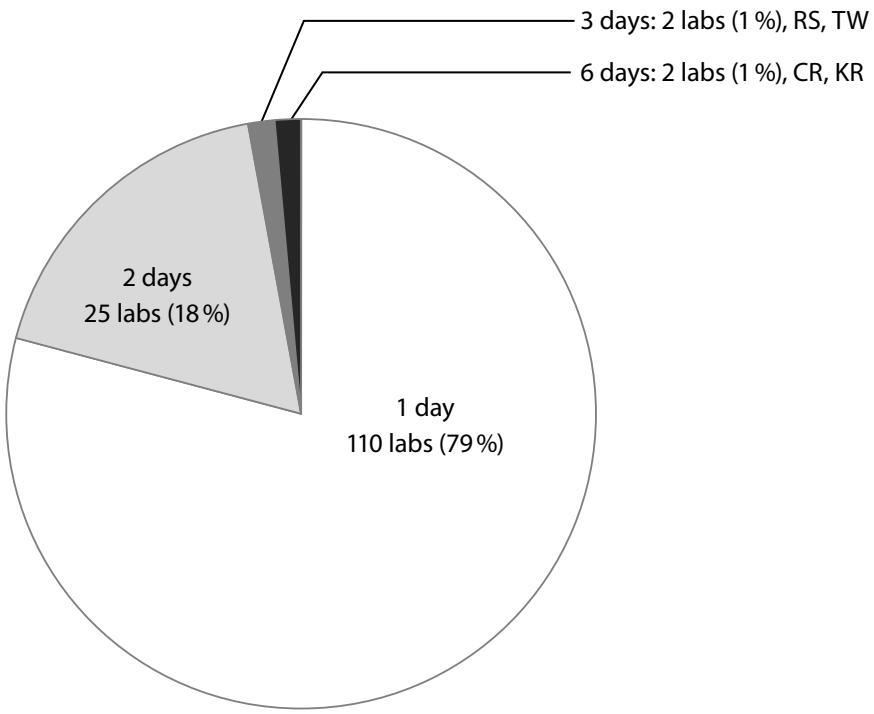
* only for EU-Member States

Appendix 1-b: Participating labs from EU candidate countries and third countries

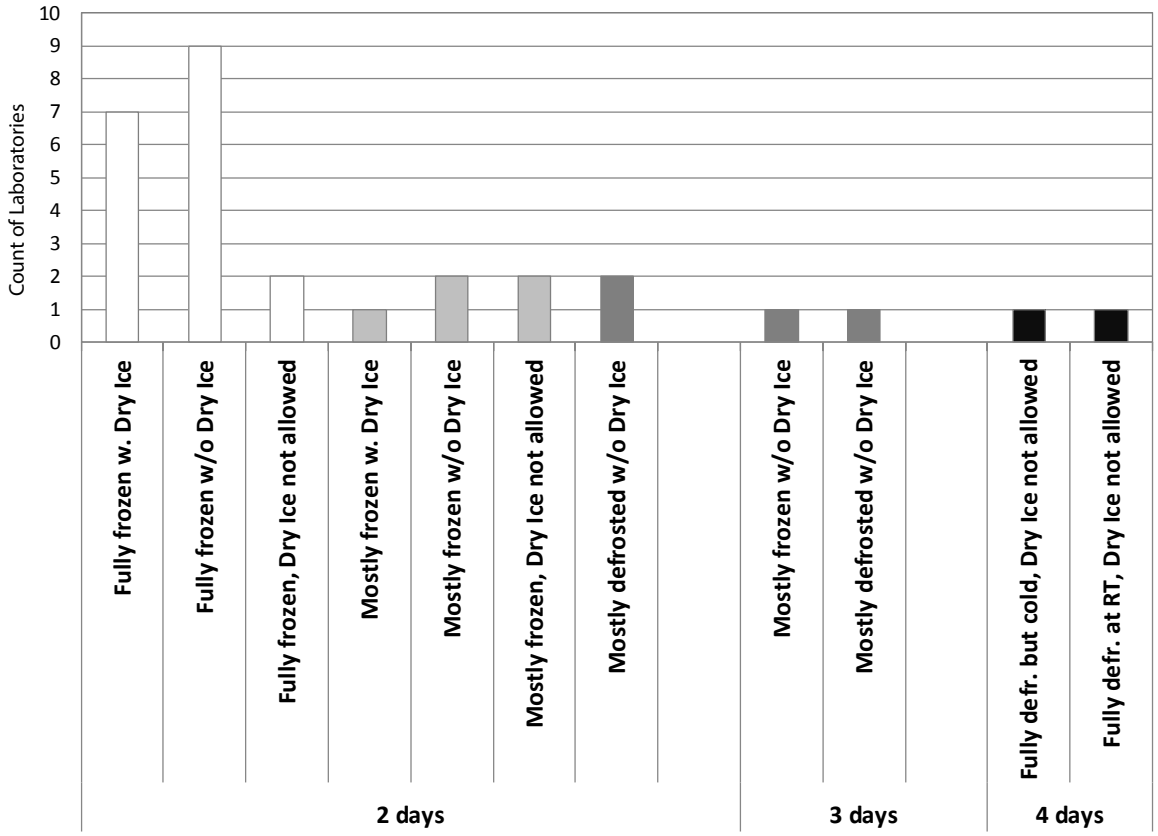
Country	Institution	City	Reported results
Canada	ISURA	Burnaby, BC	Yes
Costa Rica	Laboratorio de Análisis de Residuos de Agroquímicos	San José	Yes
Egypt	Central Lab of Residue Analysis of Pesticides and Heavy Metals in Foods	Giza	Yes
Hong Kong, People's Republic of China	Government Laboratory	Pok Fu Lam	Yes
Malaysia	Environmental Toxicology Research Center/Korea Institute of Toxicology	Petaling Jaya, Selangor	Yes
South Korea, Republic of Korea	Department of Chemistry Malaysia	JINJU-SI, GYEONG-SANGNAM-DO	Yes
Serbia	Institute of Public Health of Belgrade	Belgrade	Yes
Serbia	SP LABORATORIJA A.D.	BECEJ	Yes
Taiwan, Republic of China	ChiMei Inspection Technology Co., Ltd.	Taichung City	Yes

Appendix 2 Shipment Evaluation

(a): Compilation of shipment duration



(b): Condition of samples on arrival



Appendix 3 Data of Homogeneity Test

COMPULSORY COMPOUNDS											
2,4-D			Captan (parent)			Chlorothalonil			Dithiocarbamates		
Sample No.	Portion 1 [mg/kg]	Portion 2 [mg/kg]	Sample No.	Portion 1 [mg/kg]	Portion 2 [mg/kg]	Sample No.	Portion 1 [mg/kg]	Portion 2 [mg/kg]	Sample No.	Portion 1 [mg/kg]	Portion 2 [mg/kg]
No. 025	0.080	0.082	No. 011	0.097	0.092	No. 011	0.143	0.135	No. 011	0.253	0.287
No. 038	0.080	0.079	No. 025	0.089	0.092	No. 025	0.137	0.130	No. 025	0.276	0.246
No. 043	0.078	0.080	No. 038	0.097	0.096	No. 038	0.147	0.134	No. 038	0.300	0.225
No. 080	0.081	0.081	No. 043	0.092	0.096	No. 043	0.137	0.130	No. 043	0.254	0.282
No. 090	0.078	0.079	No. 080	0.092	0.097	No. 080	0.124	0.140	No. 080	0.270	0.262
No. 096	0.078	0.079	No. 090	0.094	0.099	No. 090	0.134	0.134	No. 090	0.260	0.238
No. 157	0.078	0.079	No. 096	0.098	0.091	No. 096	0.132	0.130	No. 096	0.232	0.262
No. 187	0.079	0.078	No. 157	0.093	0.092	No. 157	0.130	0.133	No. 157	0.262	0.261
No. 195	0.080	0.082	No. 187	0.096	0.097	No. 187	0.144	0.138	No. 187	0.255	0.259
No. 213	0.078	0.079	No. 195	0.093	0.096	No. 195	0.141	0.128	No. 195	0.276	0.262
mean / AV*	0.079 / 0.079		mean / AV*	0.094 / 0.085		mean / AV*	0.135 / 0.125		mean / AV*	0.261 / 0.267	

Fenbutatin Oxide			Folpet (parent)			Glyphosate			Haloxyfop		
Sample No.	Portion 1 [mg/kg]	Portion 2 [mg/kg]	Sample No.	Portion 1 [mg/kg]	Portion 2 [mg/kg]	Sample No.	Portion 1 [mg/kg]	Portion 2 [mg/kg]	Sample No.	Portion 1 [mg/kg]	Portion 2 [mg/kg]
No. 011	0.095	0.094	No. 011	0.402	0.385	No. 011	0.329	0.312	No. 011	0.076	0.075
No. 025	0.094	0.096	No. 025	0.383	0.394	No. 025	0.290	0.368	No. 025	0.072	0.074
No. 038	0.096	0.095	No. 038	0.398	0.406	No. 038	0.280	0.316	No. 038	0.075	0.073
No. 043	0.094	0.095	No. 043	0.407	0.388	No. 043	0.308	0.341	No. 043	0.072	0.073
No. 080	0.097	0.093	No. 080	0.391	0.402	No. 080	0.296	0.328	No. 080	0.072	0.078
No. 090	0.095	0.093	No. 090	0.384	0.391	No. 090	0.226	0.265	No. 090	0.073	0.077
No. 096	0.093	0.100	No. 096	0.398	0.404	No. 096	0.306	0.300	No. 096	0.074	0.074
No. 157	0.097	0.094	No. 157	0.383	0.393	No. 157	0.322	0.318	No. 157	0.076	0.071
No. 187	0.098	0.099	No. 187	0.394	0.396	No. 187	0.285	0.325	No. 195	0.070	0.070
No. 195	0.097	0.096	No. 195	0.406	0.391	No. 195	0.295	0.281	No. 213	0.076	0.075
mean / AV*	0.096 / 0.086		mean / AV*	0.395 / 0.334		mean / AV*	0.305 / 0.306		mean / AV*	0.074 / 0.070	

* mean / AV = Average value of the homogeneity test data [mg/kg] / Assigned value of PT [mg/kg] derived from the population of EU-/EFTA-Laboratories

OPTIONAL COMPOUNDS											
Bifenazate (sum)			Bromide ion			Carbofuran (part of sum)			Chlorate		
Sample No.	Portion 1 [mg/kg]	Portion 2 [mg/kg]	Sample No.	Portion 1 [mg/kg]	Portion 2 [mg/kg]	Sample No.	Portion 1 [mg/kg]	Portion 2 [mg/kg]	Sample No.	Portion 1 [mg/kg]	Portion 2 [mg/kg]
No. 011	0.282	0.274	No. 011	21.2	20.2	No. 011	0.0045	0.0044	No. 011	0.488	0.473
No. 025	0.282	0.282	No. 025	23.0	21.3	No. 025	0.0044	0.0043	No. 025	0.476	0.473
No. 038	0.277	0.279	No. 038	20.1	21.9	No. 038	0.0042	0.0044	No. 038	0.483	0.486
No. 043	0.270	0.276	No. 043	19.2	20.4	No. 043	0.0040	0.0044	No. 043	0.465	0.474
No. 080	0.278	0.277	No. 080	20.5	21.7	No. 080	0.0044	0.0043	No. 080	0.522	0.499
No. 090	0.282	0.255	No. 090	20.5	19.7	No. 090	0.0041	0.0042	No. 090	0.489	0.461
No. 096	0.248	0.270	No. 096	22.4	19.1	No. 096	0.0042	0.0043	No. 096	0.497	0.482
No. 157	0.277	0.280	No. 157	21.2	20.9	No. 157	0.0042	0.0042	No. 157	0.495	0.487
No. 187	0.268	0.288	No. 187	21.6	21.0	No. 187	0.0045	0.0042	No. 187	0.514	0.510
No. 195	0.281	0.274	No. 195	22.6	20.6	No. 195	0.0044	0.0044	No. 195	0.525	0.477
mean / AV*	0.275 / 0.270		mean / AV*	21.0 / 19.1		mean / AV*	0.0043 / 0.0030		mean / AV*	0.489 / 0.490	

Dithianon			Phosphonic acid			N-Acetyl glyphosate					
Sample No.	Portion 1 [mg/kg]	Portion 2 [mg/kg]	Sample No.	Portion 1 [mg/kg]	Portion 2 [mg/kg]	Sample No.	Portion 1 [mg/kg]	Portion 2 [mg/kg]			
No. 011	0.317	0.337	No. 011	19.7	18.9	No. 011	0.088	0.082			
No. 025	0.317	0.298	No. 025	18.1	19.0	No. 025	0.094	0.087			
No. 038	0.311	0.293	No. 038	18.0	17.8	No. 038	0.095	0.084			
No. 043	0.321	0.317	No. 043	17.8	19.5	No. 043	0.091	0.085			
No. 080	0.312	0.298	No. 080	19.0	17.7	No. 080	0.080	0.082			
No. 090	0.316	0.323	No. 090	17.8	18.6	No. 090	0.081	0.084			
No. 096	0.312	0.329	No. 096	18.7	17.9	No. 096	0.090	0.088			
No. 157	0.295	0.308	No. 157	18.2	18.4	No. 157	0.087	0.085			
No. 187	0.321	0.324	No. 187	18.3	17.9	No. 187	0.089	0.090			
No. 195	0.315	0.308	No. 195	18.6	19.3	No. 195	0.083	0.092			
mean / AV*	0.314 / 0.294		mean / AV*	18.5 / 19.3		mean / AV*	0.087 / 0.100				

ADDITIONAL COMPOUNDS											
Captan (sum)			Folpet (sum)			Phtalimid			THPI		
Sample No.	Portion 1 [mg/kg]	Portion 2 [mg/kg]	Sample No.	Portion 1 [mg/kg]	Portion 2 [mg/kg]	Sample No.	Portion 1 [mg/kg]	Portion 2 [mg/kg]	Sample No.	Portion 1 [mg/kg]	Portion 2 [mg/kg]
No. 011	0.300	0.280	No. 011	1.242	1.147	No. 011	0.416	0.377	No. 011	0.104	0.095
No. 025	0.285	0.281	No. 025	1.215	1.165	No. 025	0.412	0.382	No. 025	0.100	0.096
No. 038	0.291	0.275	No. 038	1.179	1.137	No. 038	0.386	0.361	No. 038	0.099	0.091
No. 043	0.296	0.306	No. 043	1.192	1.275	No. 043	0.388	0.439	No. 043	0.103	0.107
No. 080	0.300	0.282	No. 080	1.250	1.192	No. 080	0.425	0.391	No. 080	0.105	0.094
No. 090	0.278	0.286	No. 090	1.123	1.160	No. 090	0.366	0.380	No. 090	0.093	0.096
No. 096	0.279	0.290	No. 096	1.132	1.219	No. 096	0.363	0.402	No. 096	0.092	0.101
No. 157	0.293	0.275	No. 157	1.220	1.177	No. 157	0.414	0.388	No. 157	0.102	0.093
No. 187	0.288	0.295	No. 187	1.216	1.233	No. 187	0.407	0.414	No. 187	0.098	0.101
No. 195	0.287	0.294	No. 195	1.202	1.243	No. 195	0.393	0.422	No. 195	0.099	0.101
mean / AV*	0.288 / 0.302		mean / AV*	1.196 / 1.195		mean / AV*	0.396 / 0.446		mean / AV*	0.099 / 0.110	

* mean / AV = Average value of the homogeneity test data [mg/kg] / Assigned value of PT [mg/kg] derived from the population of EU-/EFTA-Laboratories

Appendix 4 Data of Stability Test / Compulsory Compounds

COMPULSORY COMPOUNDS													
2,4-D							**Captan (parent)**						
AV [mg/kg]	0.079						AV [mg/kg]	0.085					
Date	29.03.2017		19.04.2017		11.05.2017		Date	29.03.2017		19.04.2017		11.05.2017	
Sample	[mg/kg]		[mg/kg]		[mg/kg]		Sample	[mg/kg]		[mg/kg]		[mg/kg]	
Nr. 025	0.079	0.077	0.081	0.077	0.078	0.080	Nr. 025	0.094	0.087	0.097	0.095	0.087	0.089
Nr. 080	0.081	0.080	0.078	0.082	0.079	0.078	Nr. 080	0.085	0.094	0.092	0.089	0.091	0.093
Nr. 096	0.076	0.078	0.077	0.079	0.077	0.075	Nr. 187	0.074	0.090	0.091	0.093	0.086	0.085
Mean [mg/kg]	**0.079**		**0.079**		**0.078**		**Mean** [mg/kg]	**0.087**		**0.093**		**0.088**	
RSD* [%]	**2.30 %**		**1.27 %**		**2.07 %**		**RSD*** [%]	**5.41 %**		**2.79 %**		**3.55 %**	
Diviation [%] (ref. 1st Anaylsis)	**—**		**0.64 %**		**-0.85 %**		**Diviation** [%] (ref. 1st Anaylsis)	**—**		**6.29 %**		**1.32 %**	

Chlorothalonil							**Dithiocarbamates**						
AV [mg/kg]	0.125						AV [mg/kg]	0.267					
Date	29.03.2017		19.04.2017		11.05.2017		Date	06.04.2017		27.04.2017		18.05.2017	
Sample	[mg/kg]		[mg/kg]		[mg/kg]		Sample	[mg/kg]		[mg/kg]		[mg/kg]	
Nr. 025	0.146	0.145	0.137	0.135	0.143	0.142	Nr. 025	0.146	0.145	0.137	0.135	0.143	0.142
Nr. 080	0.141	0.138	0.139	0.139	0.131	0.136	Nr. 080	0.141	0.138	0.139	0.139	0.131	0.136
Nr. 096	0.138	0.140	0.125	0.129	0.132	0.135	Nr. 096	0.138	0.140	0.125	0.129	0.132	0.135
Mean [mg/kg]	**0.141**		**0.134**		**0.137**		**Mean** [mg/kg]	**0.141**		**0.134**		**0.137**	
RSD* [%]	**2.55 %**		**4.55 %**		**3.49 %**		**RSD*** [%]	**2.55 %**		**4.55 %**		**3.49 %**	
Diviation [%] (ref. 1st Anaylsis)	**—**		**-5.13 %**		**-3.43 %**		**Diviation** [%] (ref. 1st Anaylsis)	**—**		**-5.13 %**		**-3.43 %**	

Fenbutatin Oxide							**Folpet (parent)**						
AV [mg/kg]	0.086						AV [mg/kg]	0.334					
Date	29.03.2017		19.04.2017		11.05.2017		Date	29.03.2017		19.04.2017		11.05.2017	
Sample	[mg/kg]		[mg/kg]		[mg/kg]		Sample	[mg/kg]		[mg/kg]		[mg/kg]	
Nr. 025	0.095	0.093	0.100	0.100	0.102	0.104	Nr. 025	0.403	0.390	0.391	0.387	0.391	0.386
Nr. 080	0.096	0.100	0.098	0.099	0.099	0.092	Nr. 080	0.394	0.401	0.398	0.393	0.380	0.393
Nr. 096	0.097	0.101	0.094	0.098	0.099	0.099	Nr. 187	0.401	0.400	0.389	0.400	0.404	0.376
Mean [mg/kg]	**0.097**		**0.098**		**0.099**		**Mean** [mg/kg]	**0.398**		**0.393**		**0.388**	
RSD* [%]	**2.82 %**		**1.98 %**		**3.80 %**		**RSD*** [%]	**0.53 %**		**0.87 %**		**0.48 %**	
Diviation [%] (ref. 1st Anaylsis)	**—**		**1.28 %**		**1.96 %**		**Diviation** [%] (ref. 1st Anaylsis)	**—**		**-1.35 %**		**-2.53 %**	

Glyphosate							**Haloxyfop**						
AV [mg/kg]	0.306						AV [mg/kg]	0.070					
Date	24.03.2017		13.04.2017		12.05.2017		Date	29.03.2017		19.04.2017		11.05.2017	
Sample	[mg/kg]		[mg/kg]		[mg/kg]		Sample	[mg/kg]		[mg/kg]		[mg/kg]	
Nr. 025	0.302	0.279	0.300	0.259	0.233	0.325	Nr. 025	0.071	0.074	0.076	0.074	0.077	0.073
Nr. 080	0.318	0.336	0.311	0.288	0.248	0.288	Nr. 080	0.074	0.074	0.076	0.076	0.073	0.073
Nr. 096	0.293	0.309	0.290	0.301	0.341	0.287	Nr. 096	0.078	0.076	0.070	0.073	0.071	0.072
Mean [mg/kg]	**0.306**		**0.292**		**0.287**		**Mean** [mg/kg]	**0.075**		**0.074**		**0.073**	
RSD* [%]	**6.14 %**		**3.63 %**		**8.37 %**		**RSD*** [%]	**3.08 %**		**3.19 %**		**2.40 %**	
Diviation [%] (ref. 1st Anaylsis)	**—**		**-4.79 %**		**-6.26 %**		**Diviation** [%] (ref. 1st Anaylsis)	**—**		**-0.45 %**		**-1.79 %**	

***** RSD = relative standard diviation

Appendix 4 (cont.): Data of Stability Test / Optional Compounds

OPTIONAL COMPOUNDS													
Bifenazate (sum)							**Bromide Ion**						
AV [mg/kg]	0.270						AV [mg/kg]	19.1					
Date	29.03.2017		19.04.2017		11.05.2017		Date	24.03.2017		13.04.2017		12.05.2017	
Sample	[mg/kg]		[mg/kg]		[mg/kg]		Sample	[mg/kg]		[mg/kg]		[mg/kg]	
Nr. 025	0.291	0.294	0.283	0.287	0.288	0.283	Nr. 025	19.0	21.4	20.4	24.1	20.8	19.9
Nr. 080	0.285	0.289	0.285	0.283	0.282	0.290	Nr. 080	21.3	19.5	19.6	19.9	19.0	19.7
Nr. 096	0.295	0.287	0.278	0.286	0.276	0.281	Nr. 187	18.9	20.7	17.7	18.5	19.9	19.2
Mean [mg/kg]	**0.290**		**0.284**		**0.283**		**Mean** [mg/kg]	**20.1**		**20.0**		**19.8**	
RSD* [%]	**0.91 %**		**0.45 %**		**1.48 %**		**RSD*** [%]	**1.52 %**		**10.43 %**		**2.68 %**	
Diviation [%] (ref. 1st Anaylsis)	**—**		**-2.23 %**		**-2.39 %**		**Diviation** [%] (ref. 1st Anaylsis)	**—**		**-0.50 %**		**-1.90 %**	

Carbofuran (part of sum)							**Chlorate**						
AV [mg/kg]	0.0030						AV [mg/kg]	0.490					
Date	29.03.2017		19.04.2017		11.05.2017		Date	24.03.2017		13.04.2017		12.05.2017	
Sample	[mg/kg]		[mg/kg]		[mg/kg]		Sample	[mg/kg]		[mg/kg]		[mg/kg]	
Nr. 025	0.0046	0.0047	0.0041	0.0041	0.0044	0.0044	Nr. 025	0.432	0.457	0.455	0.453	0.441	0.449
Nr. 080	0.0047	0.0045	0.0043	0.0044	0.0045	0.0045	Nr. 080	0.445	0.425	0.443	0.473	0.442	0.437
Nr. 096	0.0045	0.0046	0.0043	0.0041	0.0045	0.0046	Nr. 096	0.412	0.416	0.439	0.443	0.401	0.431
Mean [mg/kg]	**0.0043**		**0.0042**		**0.0045**		**Mean** [mg/kg]	**0.431**		**0.451**		**0.434**	
RSD* [%]	**1.34 %**		**2.99 %**		**2.25 %**		**RSD*** [%]	**3.62 %**		**1.97 %**		**3.55 %**	
Diviation [%] (ref. 1st Anaylsis)	**—**		**-2.15 %**		**4.01 %**		**Diviation** [%] (ref. 1st Anaylsis)	**—**		**4.60 %**		**0.54 %**	

Dithianon							**Phosphonic acid**						
AV [mg/kg]	0.294						AV [mg/kg]	19.3					
Date	29.03.2017		19.04.2017		11.05.2017		Date	24.03.2017		13.04.2017		12.05.2017	
Sample	[mg/kg]		[mg/kg]		[mg/kg]		Sample	[mg/kg]		[mg/kg]		[mg/kg]	
Nr. 025	0.331	0.320	0.324	0.302	0.318	0.304	Nr. 025	17.5	21.0	19.1	22.6	19.4	17.9
Nr. 080	0.300	0.330	0.301	0.337	0.302	0.339	Nr. 080	18.4	16.2	18.5	18.8	18.7	17.4
Nr. 096	0.345	0.306	0.315	0.331	0.344	0.343	Nr. 096	17.5	18.8	17.1	18.0	18.0	18.8
Mean [mg/kg]	**0.322**		**0.318**		**0.325**		**Mean** [mg/kg]	**18.4**		**19.0**		**18.4**	
RSD* [%]	**1.88 %**		**1.58 %**		**5.14 %**		**RSD*** [%]	**0.77 %**		**8.84 %**		**1.64 %**	
Diviation [%] (ref. 1st Anaylsis)	**—**		**-1.14 %**		**0.93 %**		**Diviation** [%] (ref. 1st Anaylsis)	**—**		**3.22 %**		**-0.31 %**	

N-Acetyl glyphosate						
AV [mg/kg]	0.100					
Date	24.03.2017		13.04.2017		12.05.2017	
Sample	[mg/kg]		[mg/kg]		[mg/kg]	
Nr. 025	0.087	0.083	0.094	0.085	0.088	0.079
Nr. 080	0.087	0.089	0.088	0.088	0.084	0.089
Nr. 096	0.087	0.088	0.086	0.080	0.082	0.090
Mean [mg/kg]	**0.087**		**0.087**		**0.085**	
RSD* [%]	**1.85 %**		**3.92 %**		**1.88 %**	
Diviation [%] (ref. 1st Anaylsis)	**—**		**0.00 %**		**-1.73 %**	

Appendix 4 (cont.): Data of Stability Test / Additional Compounds

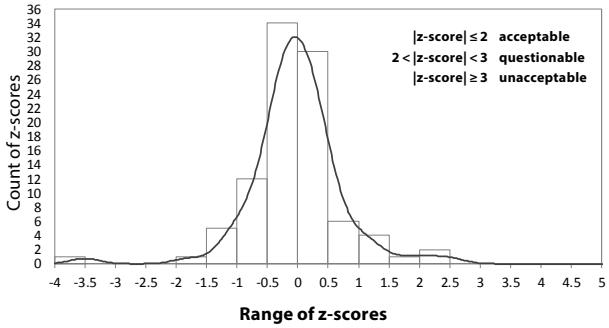
ADDITIONAL COMPOUNDS													
Captan (sum)							**Folpet (sum)**						
AV [mg/kg]	0.302						AV [mg/kg]	1.195					
Date	29.03.2017		19.04.2017		11.05.2017		Date	29.03.2017		19.04.2017		11.05.2017	
Sample	[mg/kg]		[mg/kg]		[mg/kg]		Sample	[mg/kg]		[mg/kg]		[mg/kg]	
Nr. 025	0.282	0.273	0.298	0.292	0.250	0.274	Nr. 025	1.148	1.118	1.217	1.180	1.007	1.106
Nr. 080	0.277	0.308	0.288	0.304	0.279	0.273	Nr. 080	1.140	1.264	1.186	1.259	1.137	1.112
Nr. 187	0.291	0.286	0.288	0.288	0.261	0.271	Nr. 187	1.217	1.203	1.195	1.158	1.057	1.068
Mean [mg/kg]	**0.286**		**0.293**		**0.268**		**Mean** [mg/kg]	**1.182**		**1.199**		**1.081**	
RSD* [%]	**2.70 %**		**1.44 %**		**2.72 %**		**RSD*** [%]	**3.59 %**		**1.91 %**		**3.50 %**	
Diviation [%] (ref. 1st Anaylsis)	**—**		**2.34 %**		**-6.33 %**		**Diviation** [%] (ref. 1st Anaylsis)	**—**		**1.47 %**		**-8.53 %**	

Phthalimide							**THPI**						
AV [mg/kg]	0.446						AV [mg/kg]	0.110					
Date	29.03.2017		19.04.2017		11.05.2017		Date	29.03.2017		19.04.2017		11.05.2017	
Sample	[mg/kg]		[mg/kg]		[mg/kg]		Sample	[mg/kg]		[mg/kg]		[mg/kg]	
Nr. 025	0.370	0.361	0.410	0.393	0.306	0.357	Nr. 025	0.094	0.094	0.101	0.099	0.082	0.093
Nr. 080	0.370	0.429	0.391	0.430	0.376	0.357	Nr. 080	0.096	0.108	0.098	0.108	0.095	0.091
Nr. 187	0.405	0.399	0.400	0.376	0.324	0.343	Nr. 187	0.109	0.099	0.099	0.098	0.088	0.094
Mean [mg/kg]	**0.389**		**0.400**		**0.344**		**Mean** [mg/kg]	**0.100**		**0.101**		**0.090**	
RSD* [%]	**5.23 %**		**2.78 %**		**5.69 %**		**RSD*** [%]	**5.30 %**		**2.32 %**		**2.91 %**	
Diviation [%] (ref. 1st Anaylsis)	**—**		**2.90 %**		**-11.58 %**		**Diviation** [%] (ref. 1st Anaylsis)	**—**		**0.61 %**		**-9.69 %**	

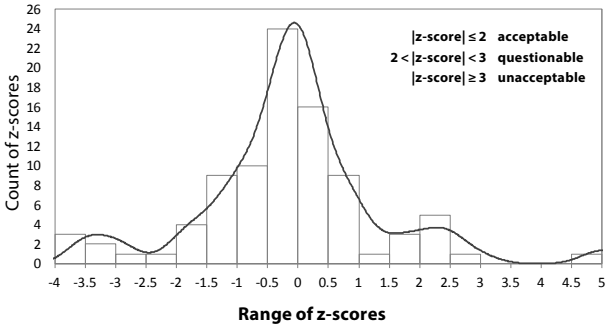
Appendix 5 Histograms and Kernel Density Estimates of z-score* Distributions
(Results from EU and EFTA Laboratories only)

Compulsory Compounds

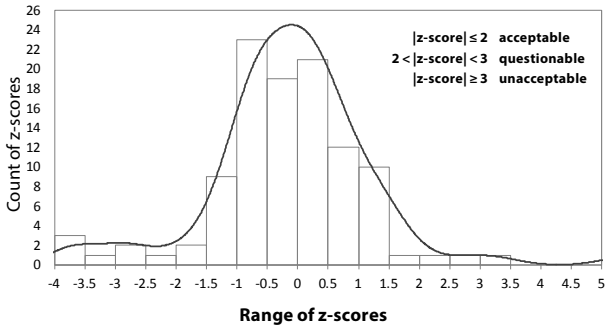
2,4-D
(AV derived from entire population)



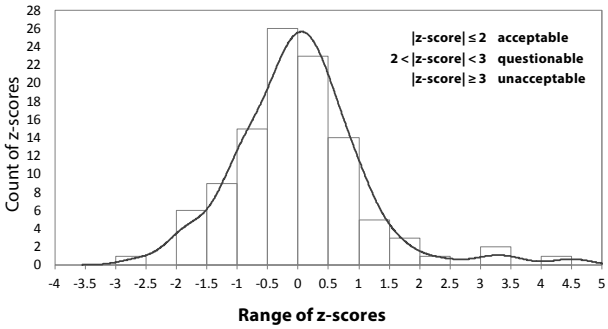
Captan (parent)
(AV derived from entire population)



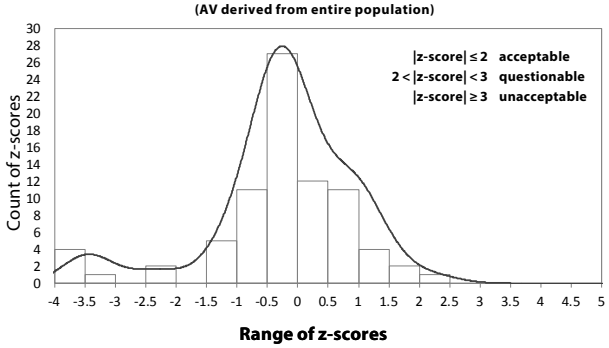
Chlorothalonil
(AV derived from entire population)



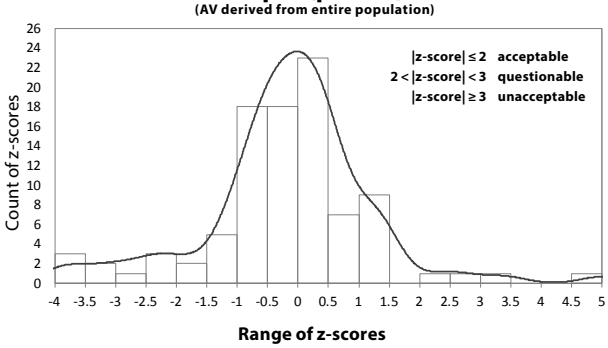
Dithiocarbamates
(AV derived from entire population)



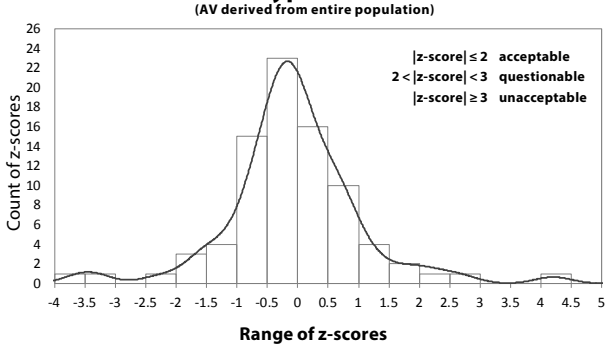
Fenbutatin oxide
(AV derived from entire population)



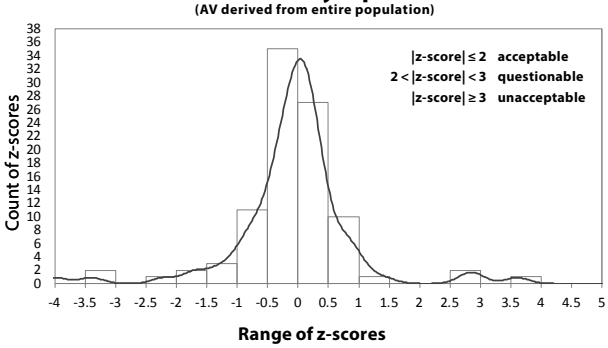
Folpet (parent)
(AV derived from entire population)



Glyphosate
(AV derived from entire population)



Haloxyfop
(AV derived from entire population)



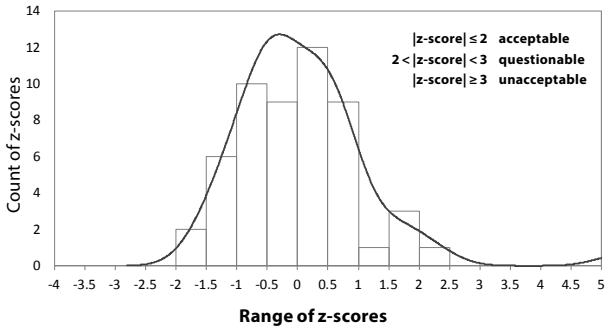
* Cut-off at z-score = 5;

Appendix 5 (cont.) Histograms and Kernel Density Estimates of z-score* Distributions

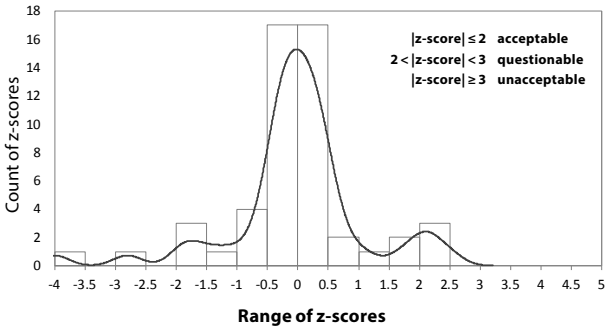
(Results from EU and EFTA Laboratories only)

Optional Compounds

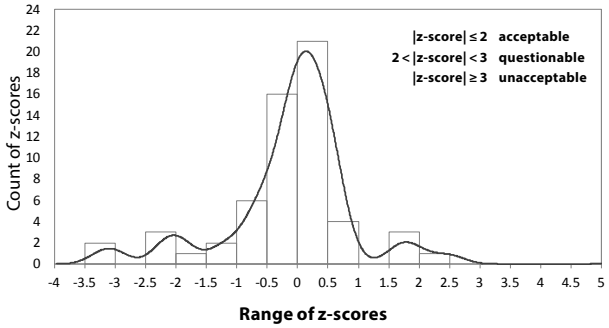
Bifenazate (P)
(AV derived from entire population)



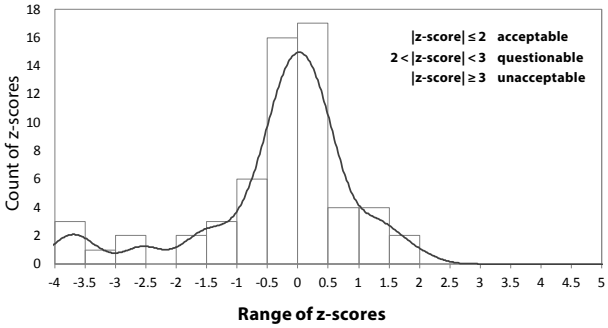
Bromide ion
(AV derived from entire population)



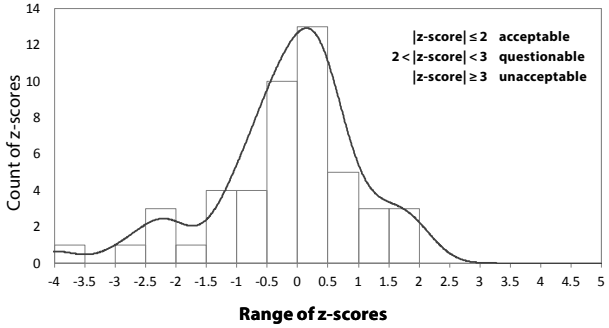
Chlorate
(AV derived from entire population)



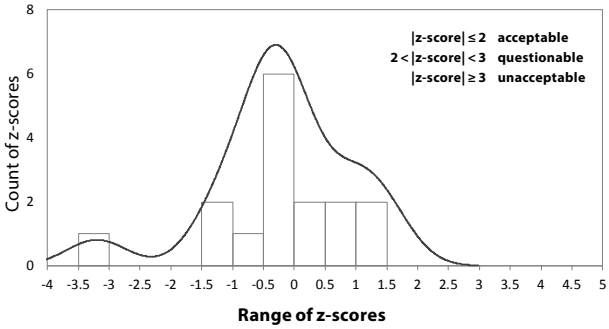
Dithianon
(AV derived from entire population)



Phosphonic acid
(AV# derived from entire population)



N-Acetyl glyphosate
(AV derived from entire population)



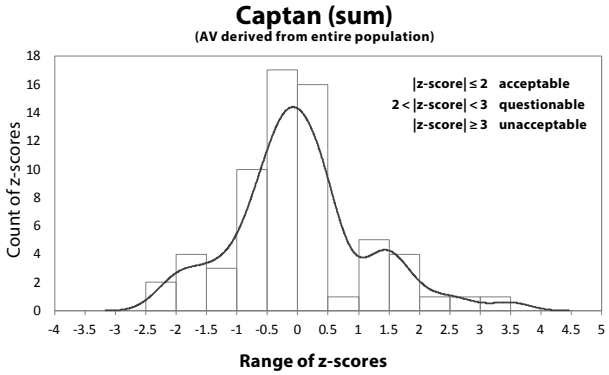
***** Cut-off at z-score = 5

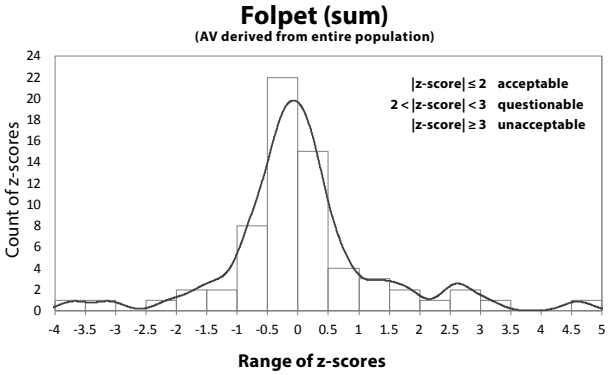
excluding carbofuran due to high uncertainty of its assigned value

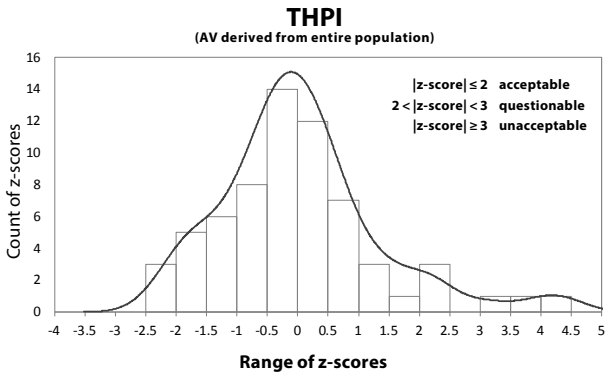
Appendix 5 (cont.) Histograms and Kernel Density Estimates of z-score* Distributions

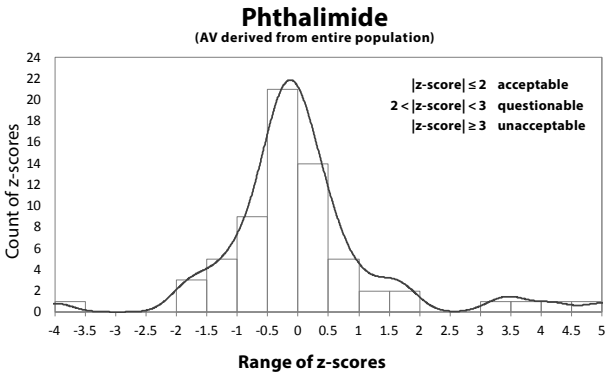
(Results from EU and EFTA Laboratories only)

Additional Compounds



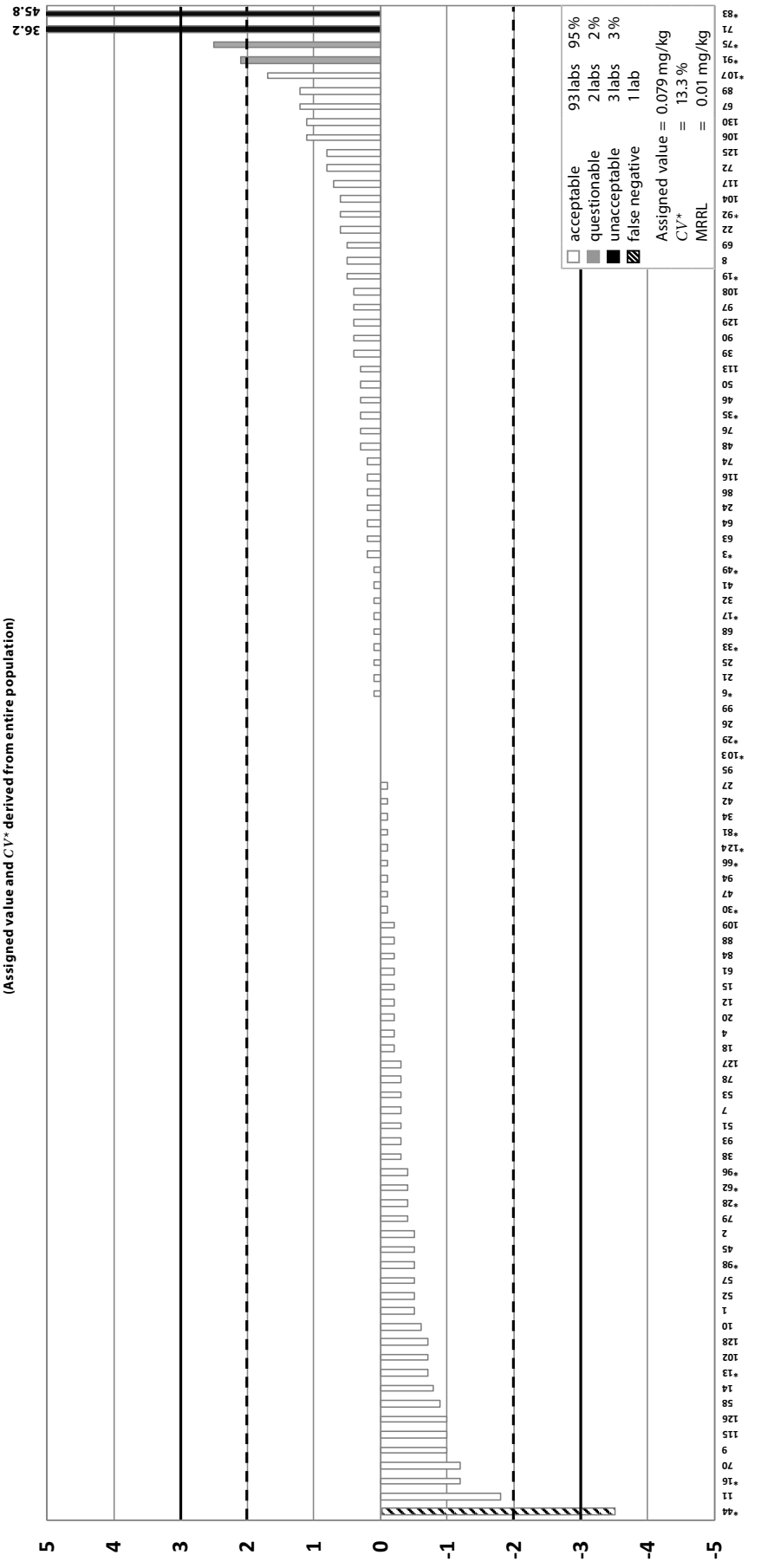




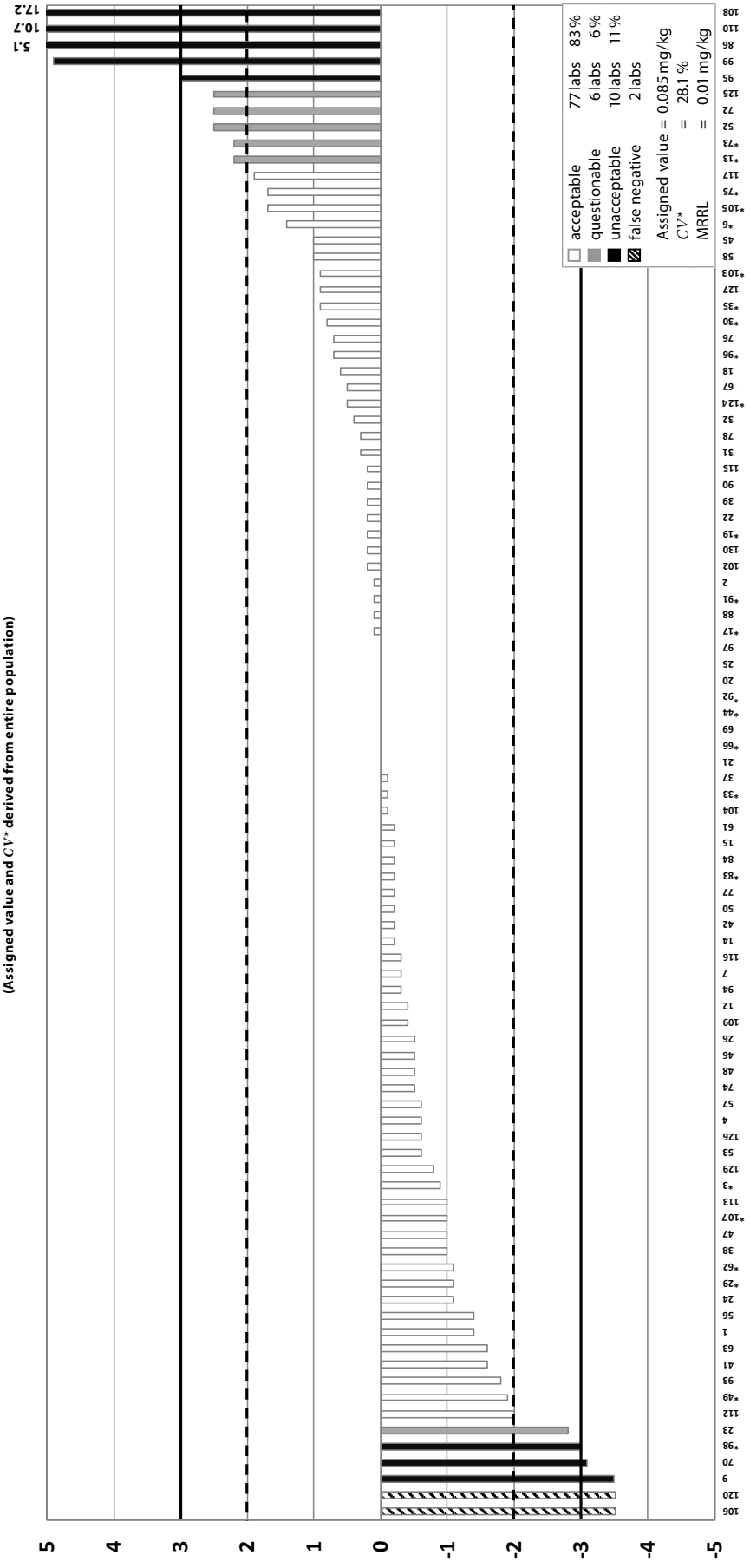


* Cut-off at z-score = 5;

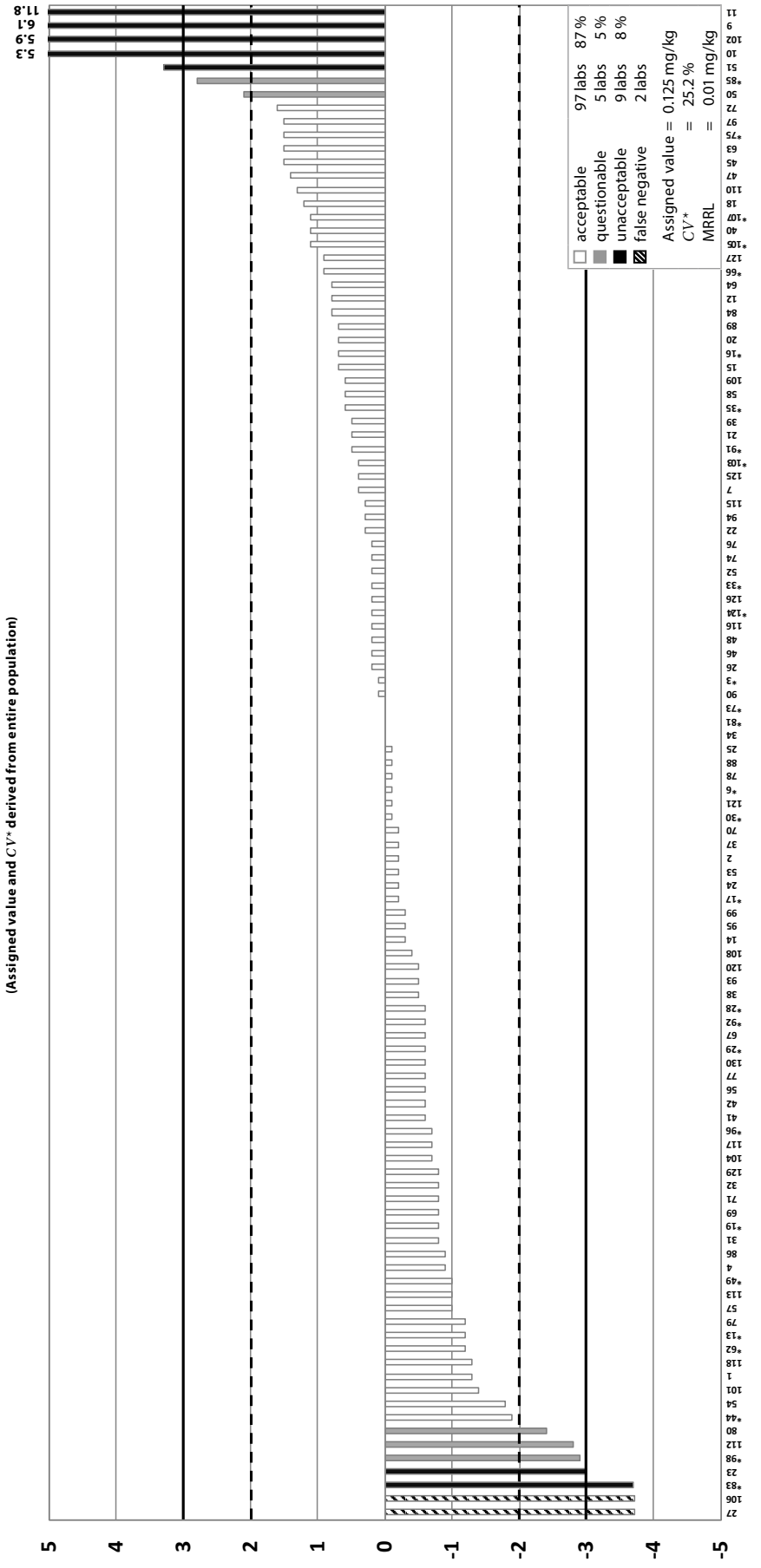
Appendix 6 Graphic Presentation of z-Scores: Compulsory Compounds(Results from EU and EFTA Laboratories only, * = NRL)



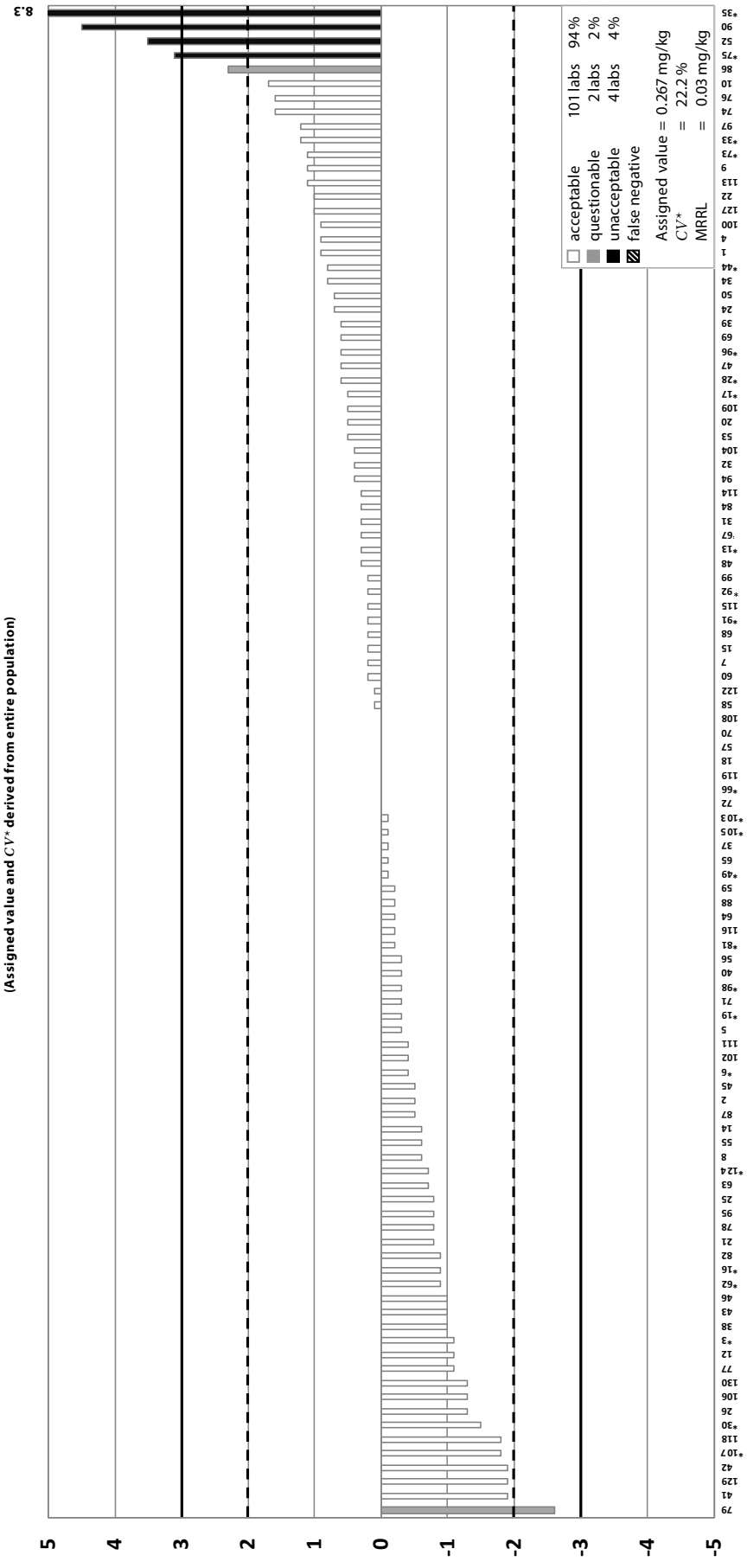
Appendix 6 (cont.) **Graphic Presentation of z-Scores: Compulsory Compounds** (Results from EU and EFTA Laboratories only, * = NRL)



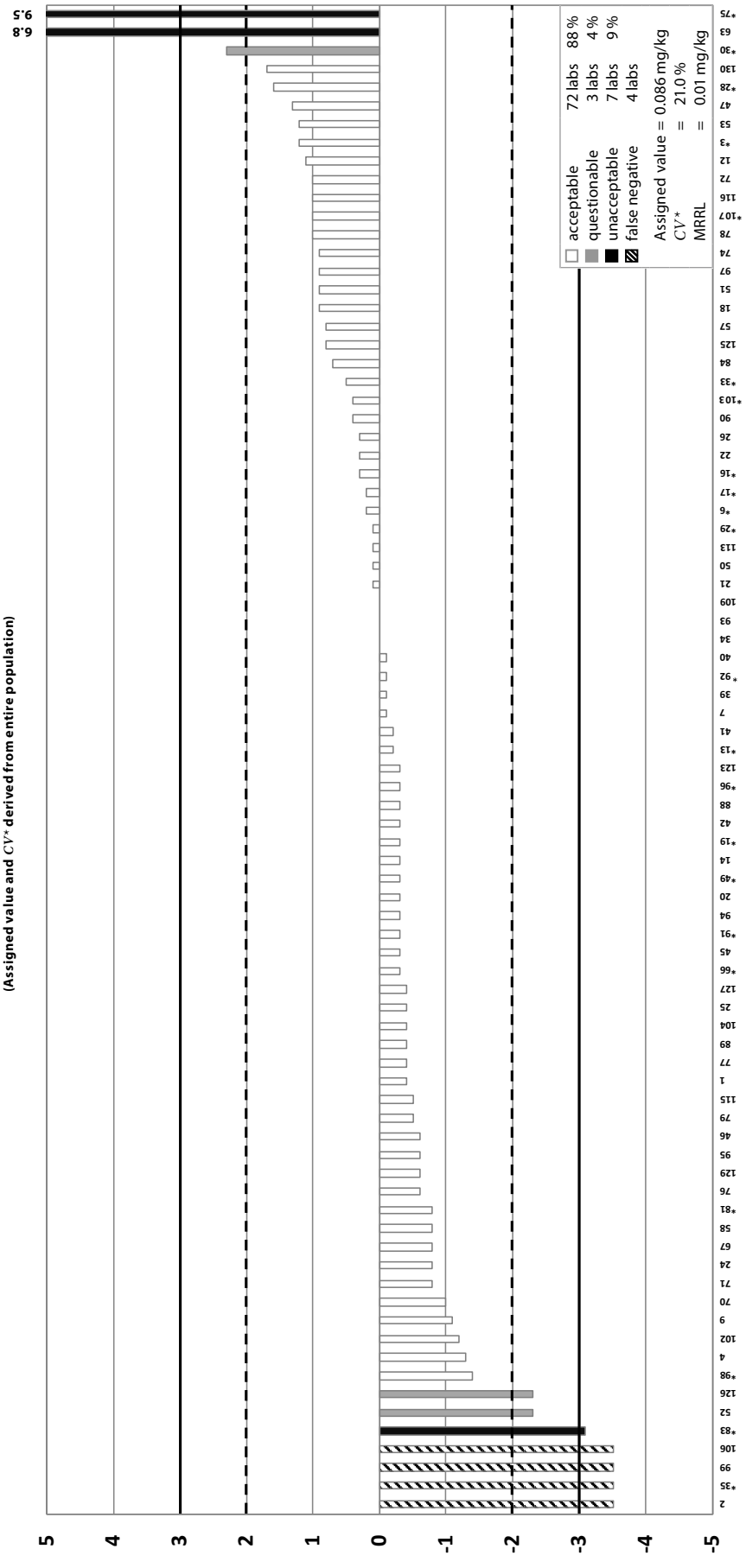
Appendix 6 (cont.) **Graphic Presentation of z-Scores: Compulsory Compounds** (Results from EU and EFTA Laboratories only, * = NRL)



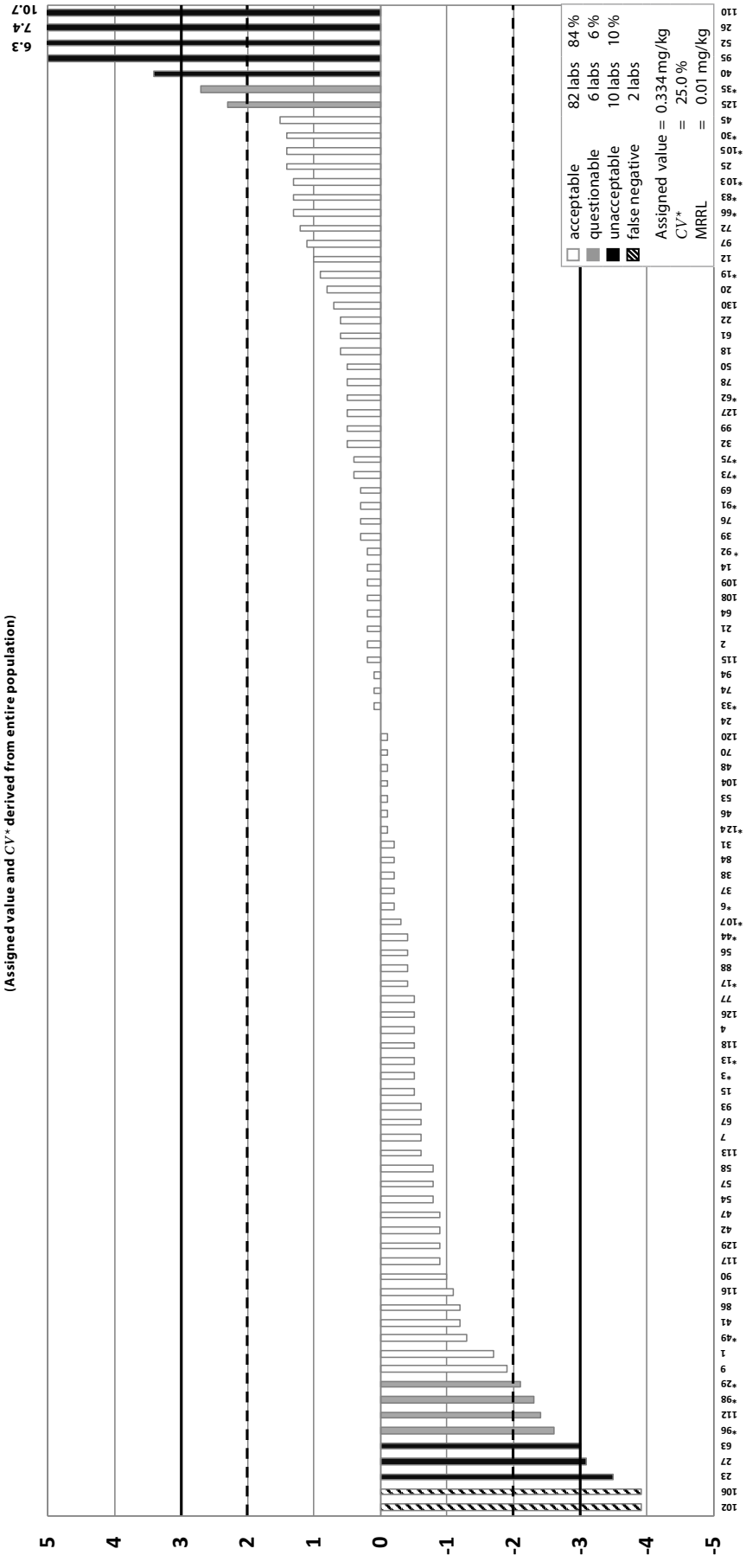
Appendix 6 (cont.) Graphic Presentation of z-Scores: Compulsory Compounds (Results from EU and EFTA Laboratories only, * = NRL)



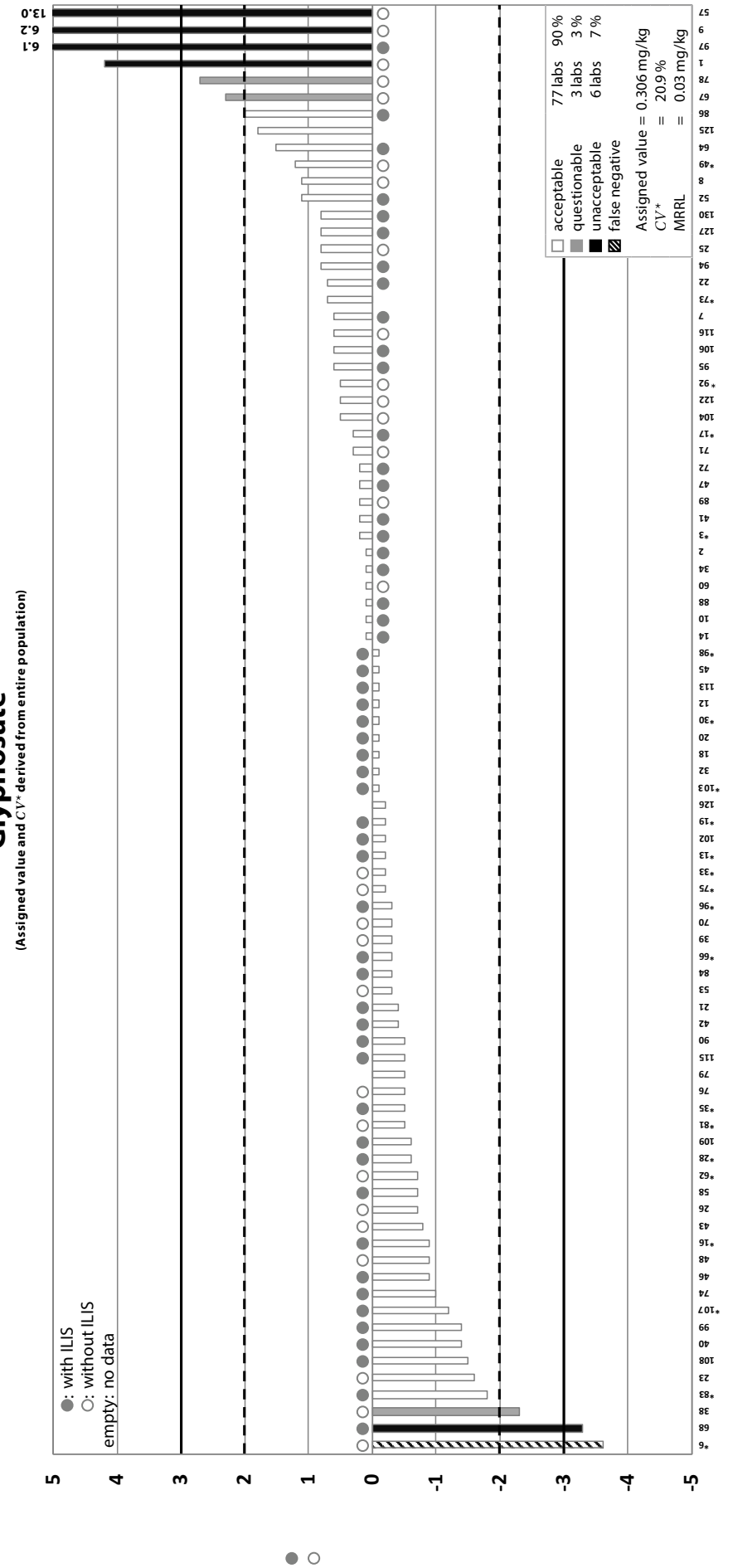
Appendix 6 (cont.) Graphic Presentation of z-Scores: Compulsory Compounds (Results from EU and EFTA Laboratories only, * = NRL)



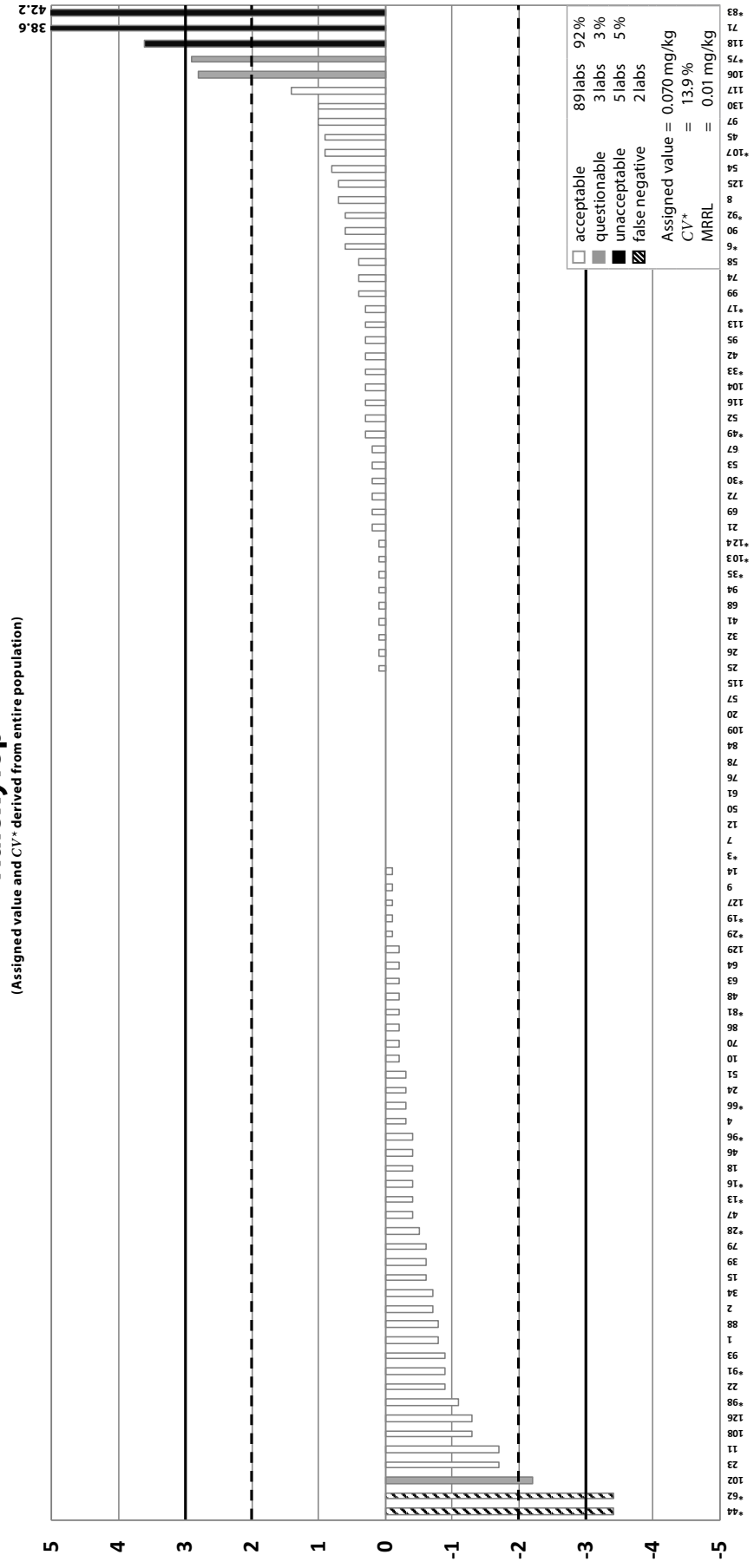
Appendix 6 (cont.) **Graphic Presentation of z-Scores: Compulsory Compounds** (Results from EU and EFTA Laboratories only, * = NRL)



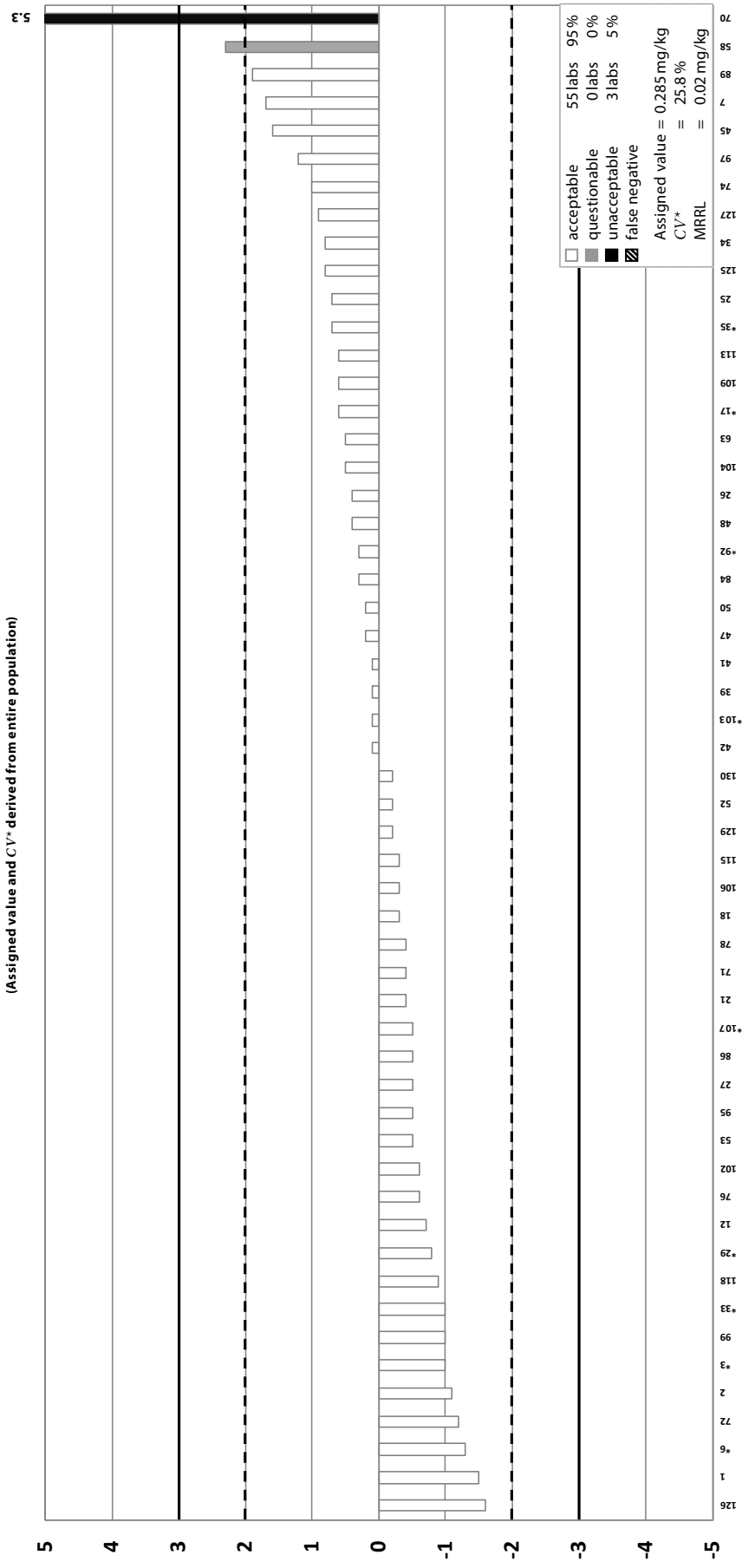
Appendix 6 (cont.) Graphic Presentation of z-Scores: Compulsory Compounds (Results from EU and EFTA Laboratories only, * = NRL)



Appendix 6 (cont.) Graphic Presentation of z-Scores: Compulsory Compounds (Results from EU and EFTA Laboratories only, * = NRL)



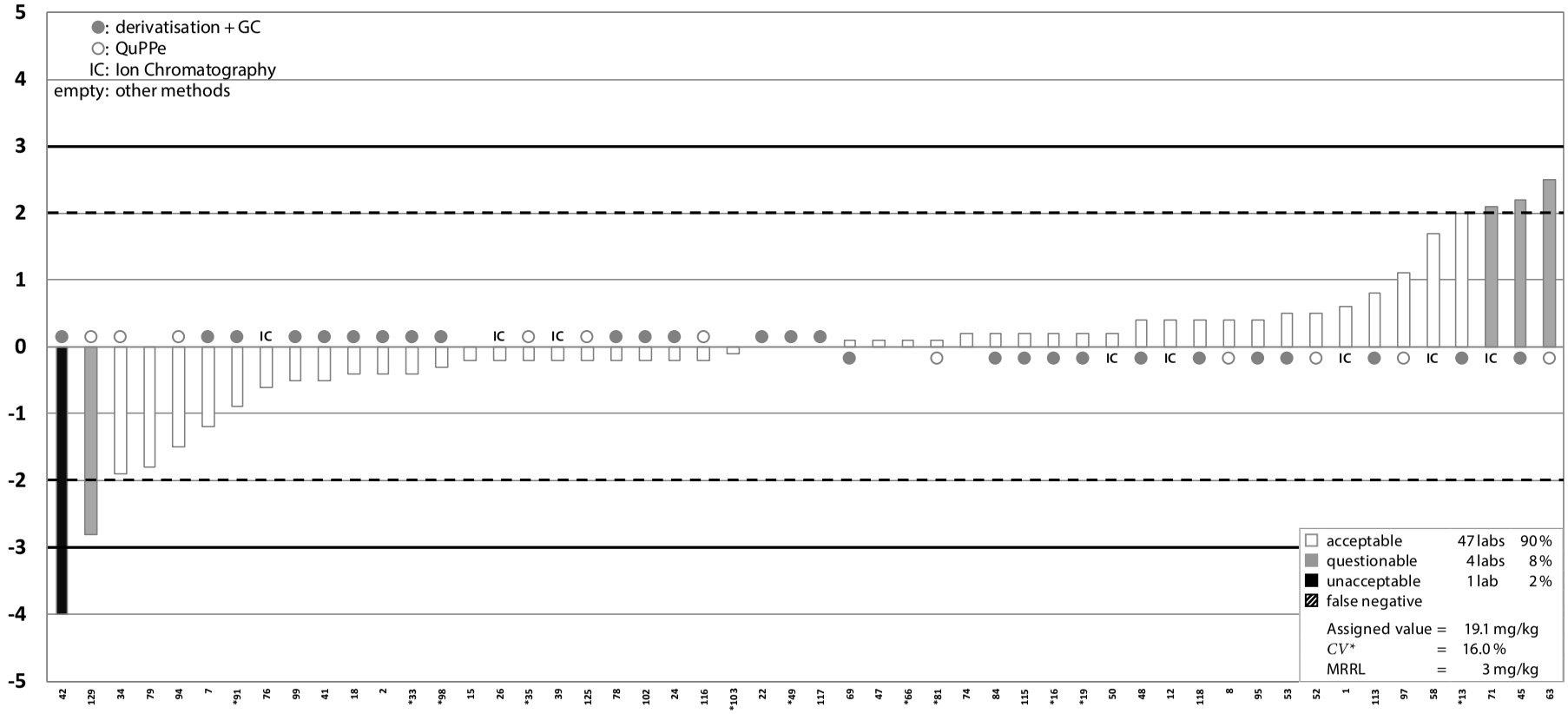
Appendix 6 (cont.) Graphic Presentation of z-Scores: Optional Compounds (Results from EU and EFTA Laboratories only, * = NRL)



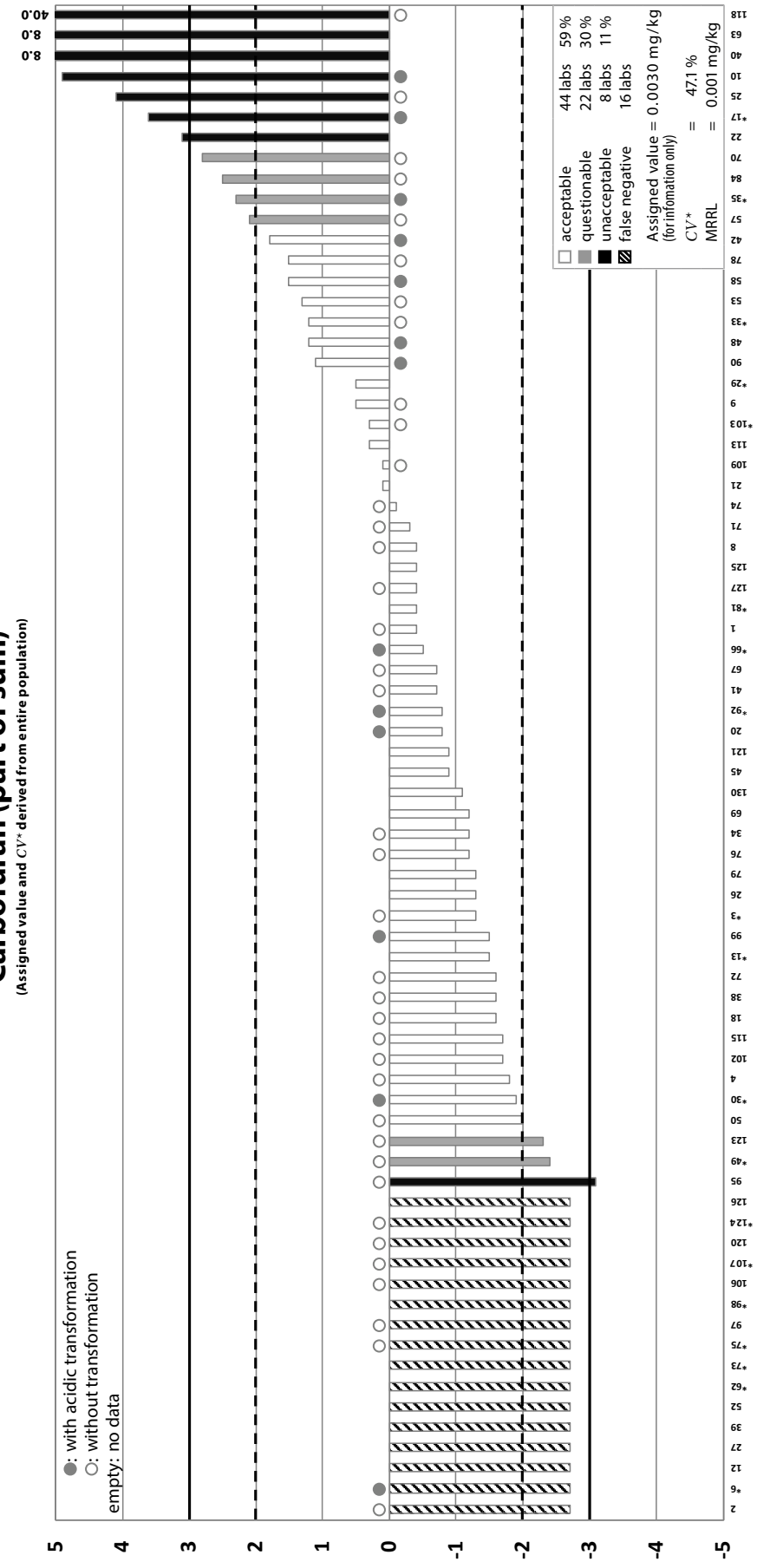
Appendix 6 (cont.) **Graphic Presentation of z-Scores: Optional Compounds** (Results from EU and EFTA Laboratories only, * = NRL)

Bromide Ion

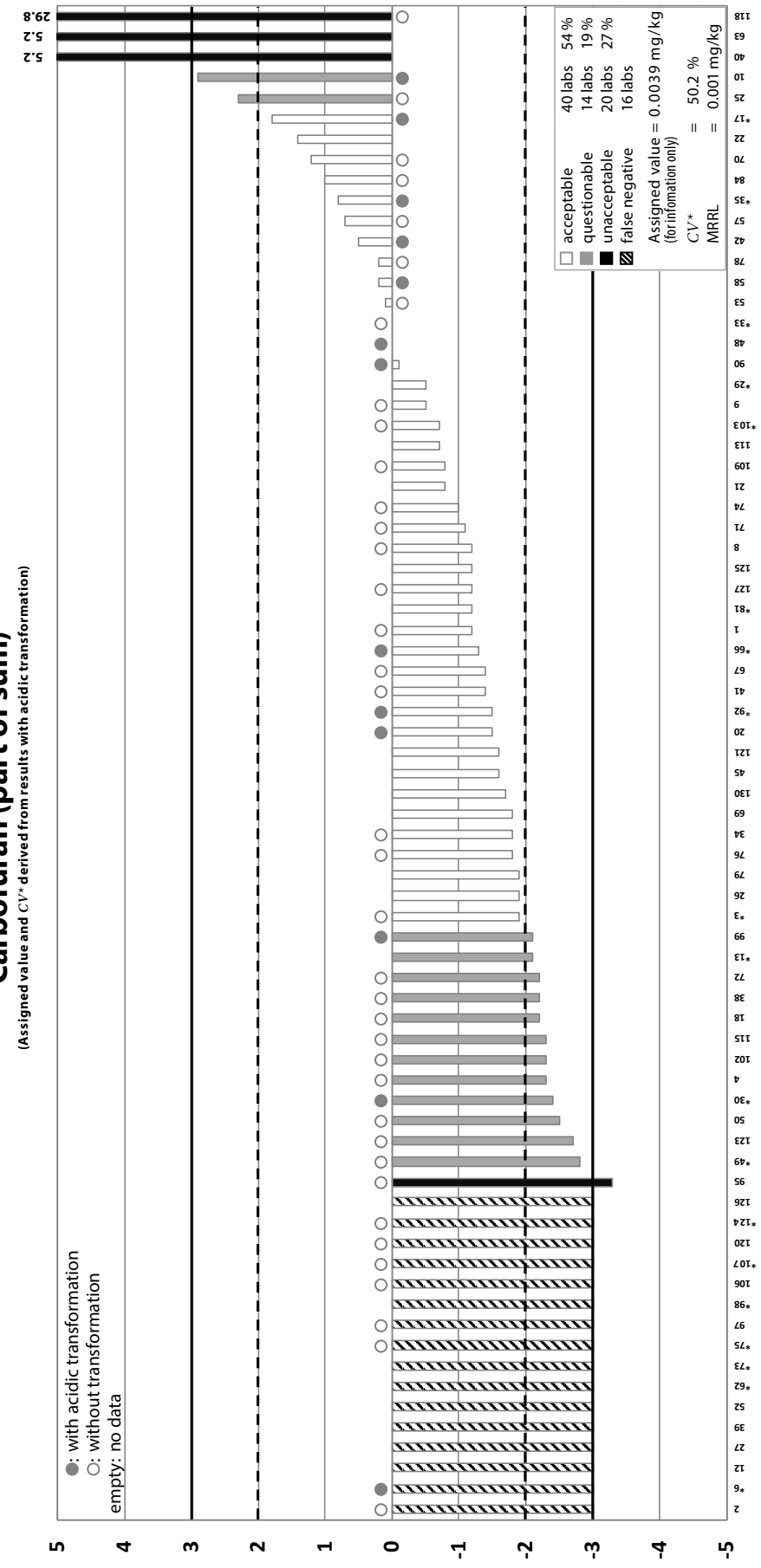
(Assigned value and CV^* derived from entire population; assigned value and z-scores for informative purpose only)



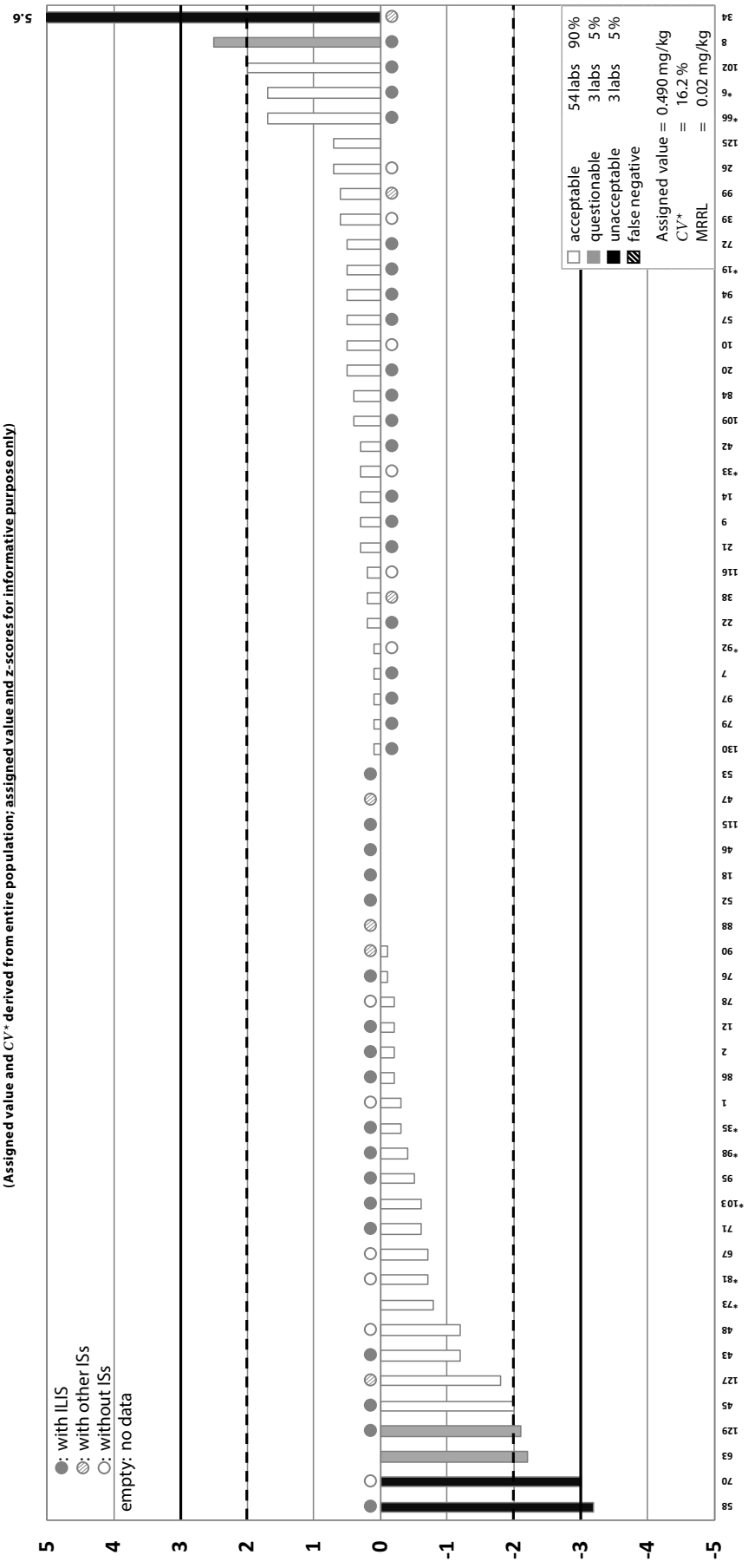
Appendix 6 (cont.) Graphic Presentation of z-Scores: Optional Compounds (Results from EU and EFTA Laboratories only, * = NRL)



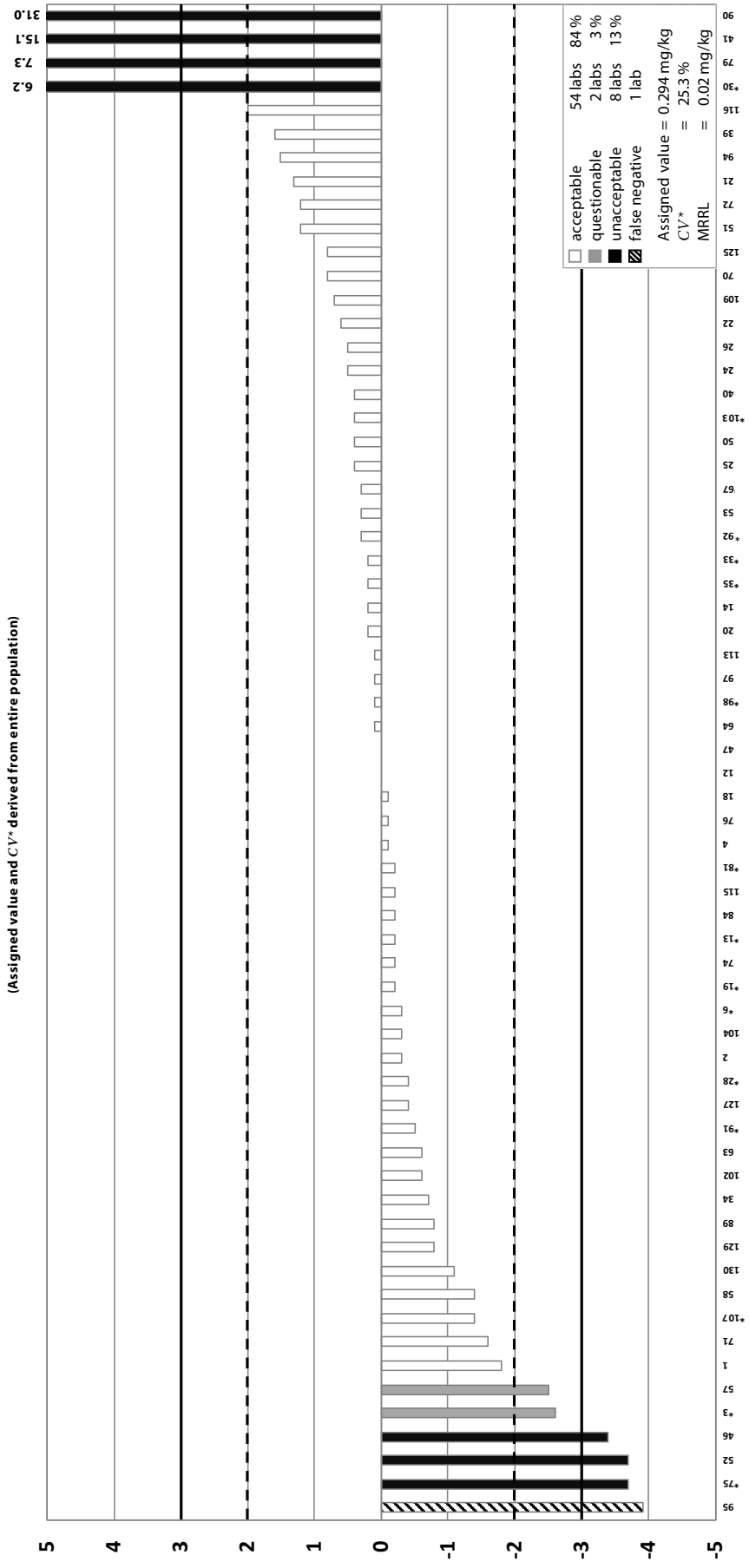
Appendix 6 (cont.) Graphic Presentation of z-Scores: Optional Compounds (Results from EU and EFTA Laboratories only, * = NRL)



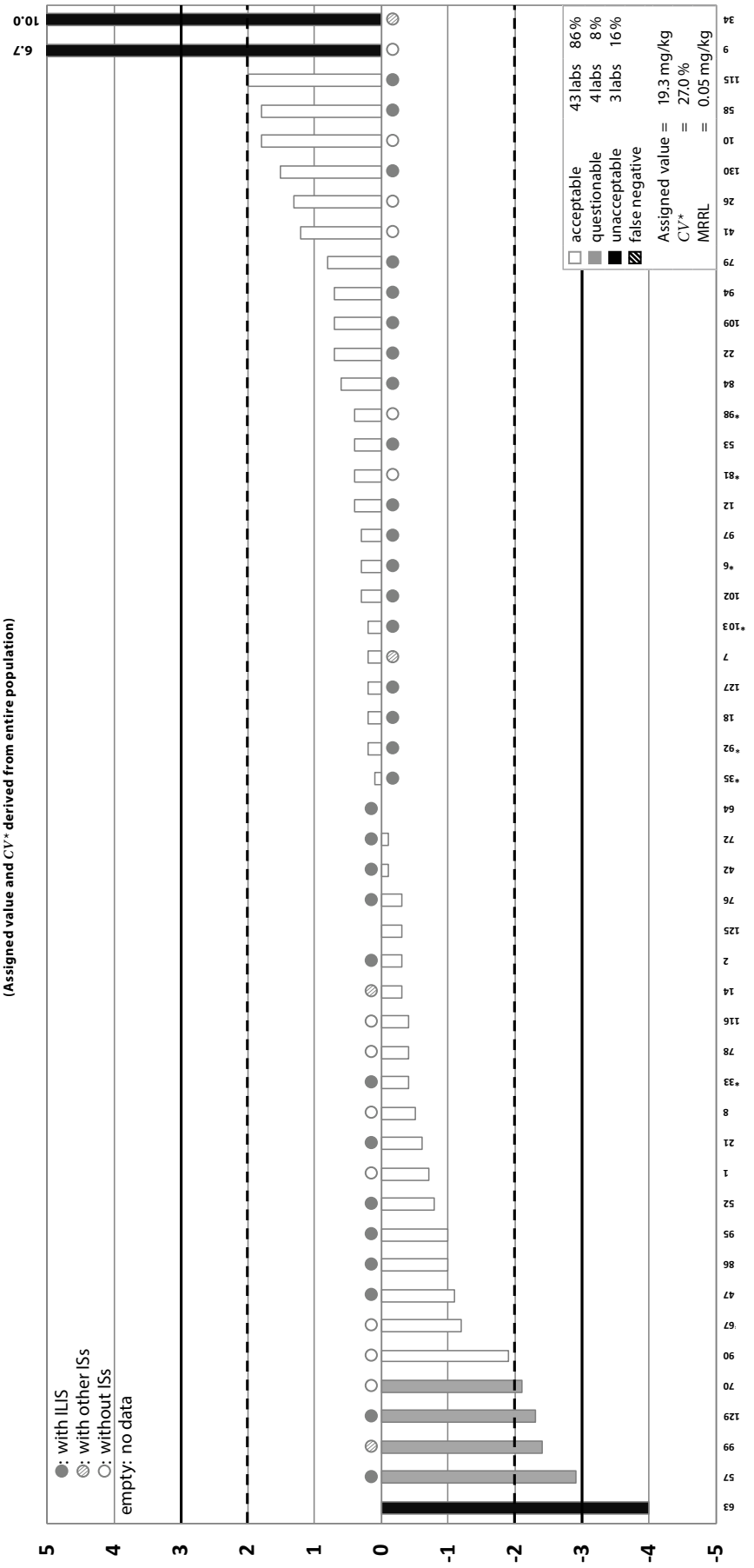
Appendix 6 (cont.) Graphic Presentation of z-Scores: Optional Compounds (Results from EU and EFTA Laboratories only, * = NRL)



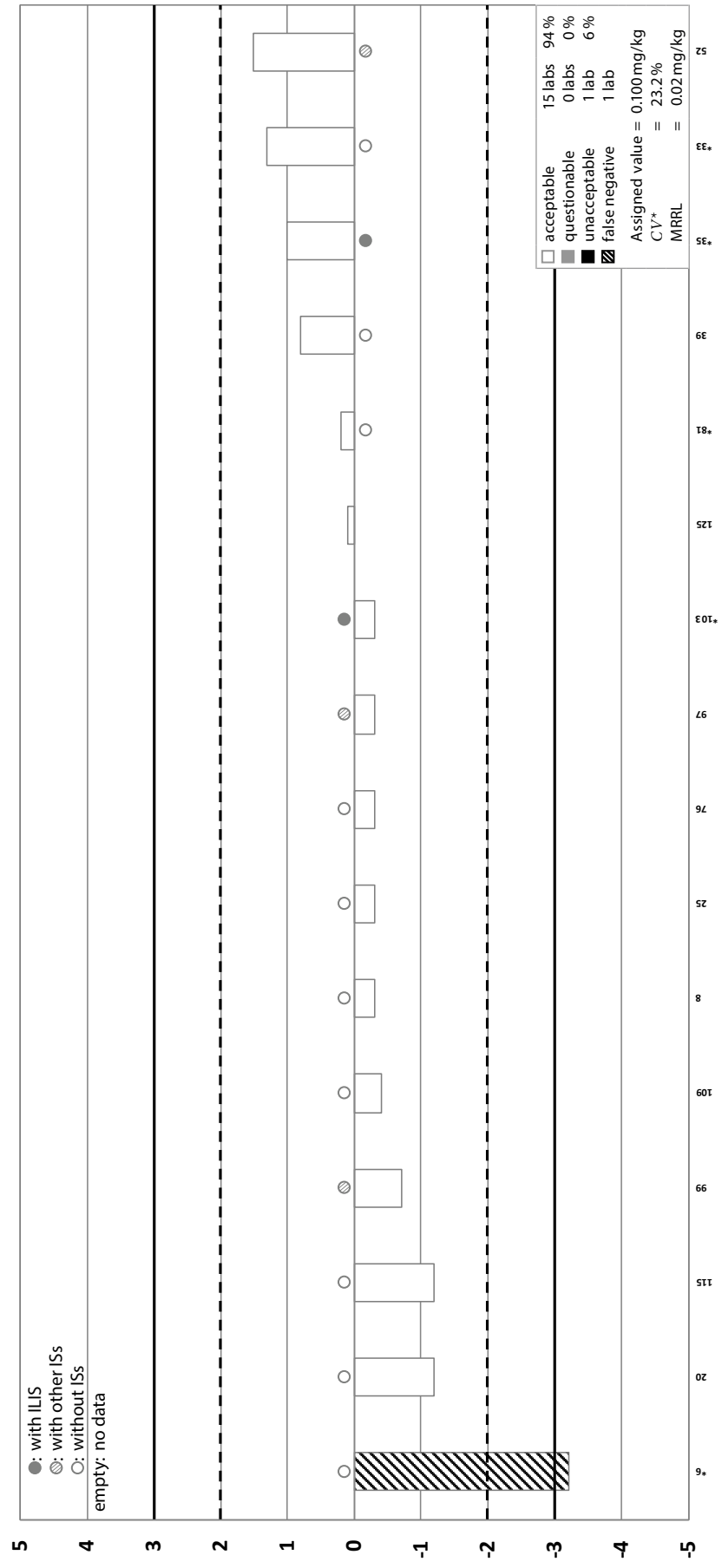
Appendix 6 (cont.) Graphic Presentation of z-Scores: Optional Compounds (Results from EU and EFTA Laboratories only, * = NRL)



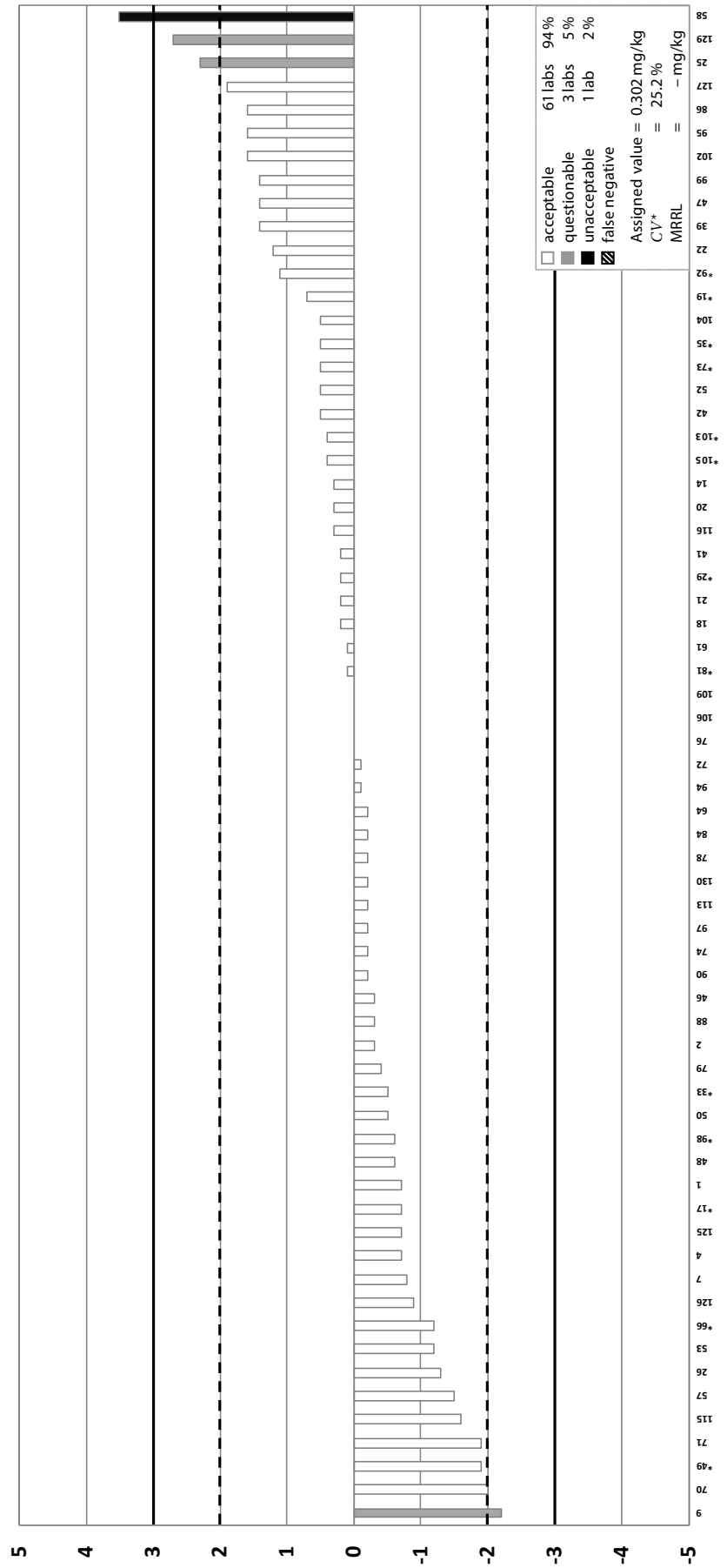
Appendix 6 (cont.) Graphic Presentation of z-Scores: Optional Compounds (Results from EU and EFTA Laboratories only, * = NRL)



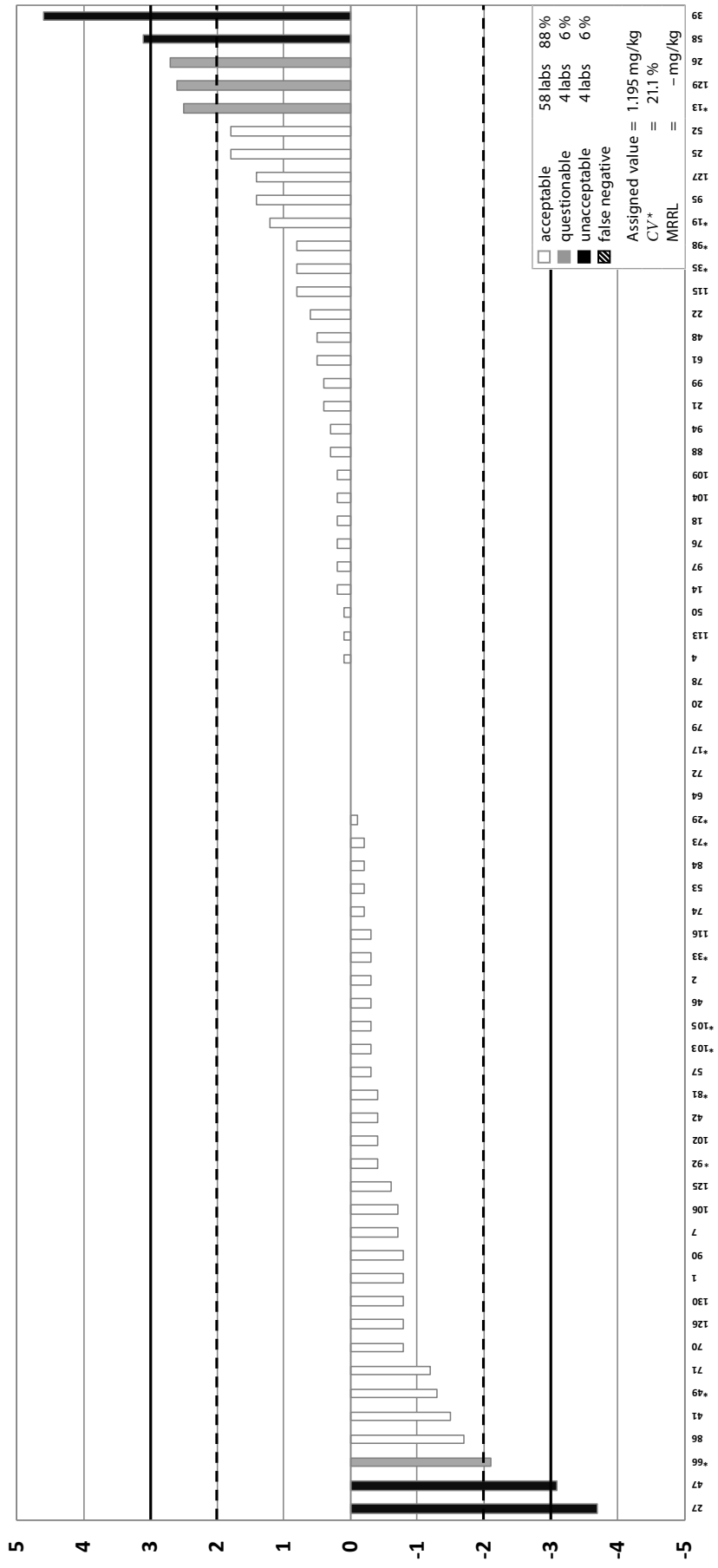
Appendix 6 (cont.) Graphic Presentation of z-Scores: Optional compounds (Results from EU and EFTA Laboratories only, * = NRL)



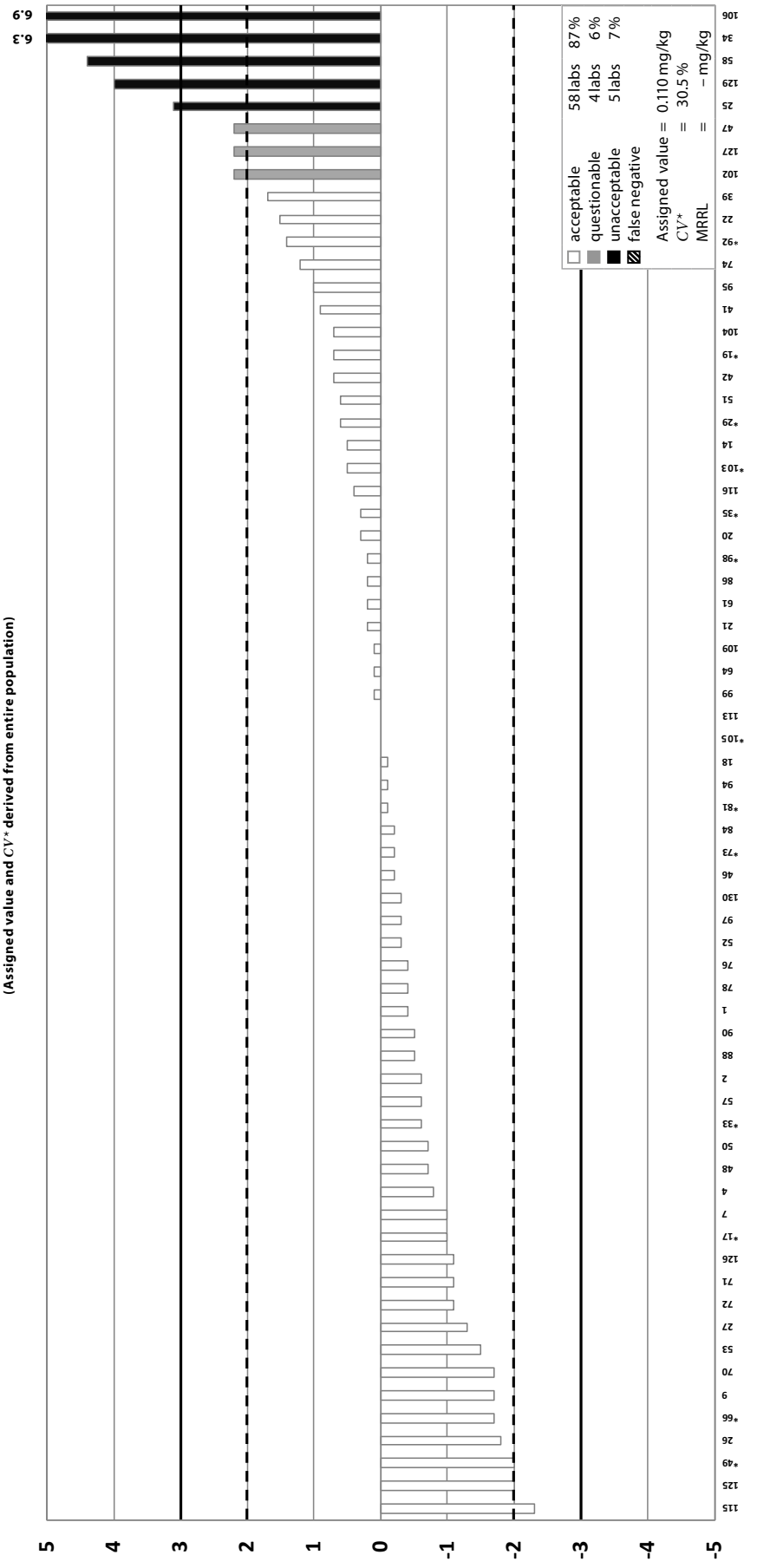
Appendix 6 (cont.) **Graphic Presentation of z-Scores: Additional compounds (Assigned Value and z-score for informativ purpose only)**
(Results from EU and EFTA Laboratories only, * = NRL))



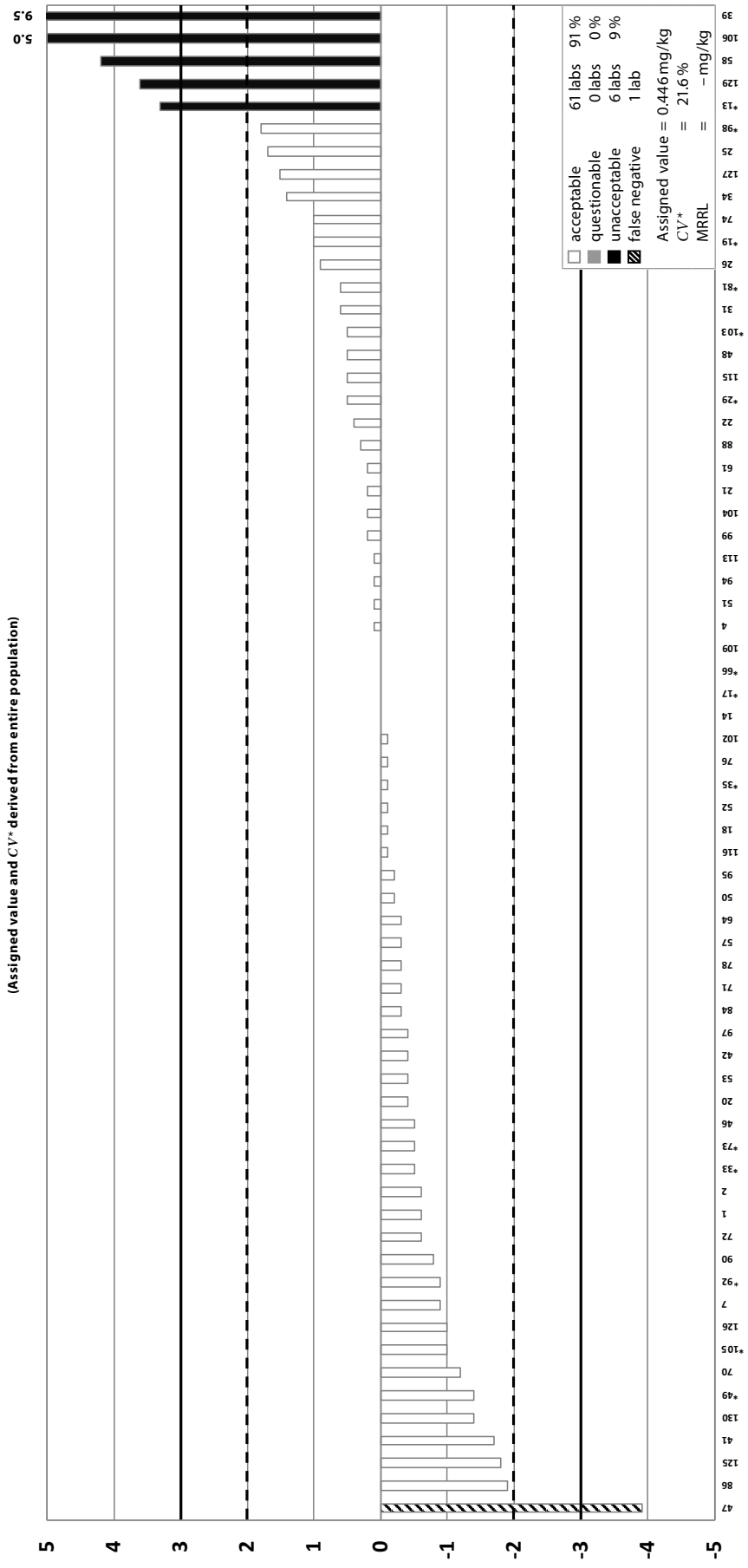
Appendix 6 (cont.) **Graphic Presentation of z-Scores: Additional compounds (Assigned Value and z-score for informativ purpose only)**
(Results from EU and EFTA Laboratories only, * = NRL)



Appendix 6 (cont.) **Graphic Presentation of z-Scores: Additional compounds (Assigned Value and z-score for informativ purpose only)**
(Results from EU and EFTA Laboratories only, * = NRL))



Appendix 6 (cont.) **Graphic Presentation of z-Scores: Additional compounds (Assigned Value and z-score for informativ purpose only)**
(Results from EU and EFTA Laboratories only, * = NRL)



Appendix 7 Possible Reasons Reported for Poor Performance (ordered by z-scores)

A: Technical problems with measurement instrumentation
B: Procedure not properly conducted
C: Matrix effect not properly compensated
D: Lack of experience
E: Error in concentration of analytical standard
F: Error in the evaluation/interpretation of measurement data
G: Use of inappropriate procedure
H: Reporting limit higher than the assigned value
I: Sample weight too small, homogeneity insufficient
J: Transcription error
K: Result not corrected for low recovery
L: Inappropriate calibration
M: Detection signals strongly interfered by matrix components/Strong chromatographic interferences
N: Misunderstanding of the definition of the analyte
O: Degradation during sample preparation or measurement
P: Sample amount not sufficient for quantitative analysis
Q: Consecutive error (e.g. parent too low, degraded product too high, but sum o.k.)
U: Undefined

2,4-D (free acid) Assigned value: 0.079 mg/kg

LabCode	z-Score	Source of error localized?	Reason / Remarks	
44	-3.5 (FN)	(Yes)	We have checked our method (calibration curve, recovery and SRM), we think that the most likely cause of the poor results is the used derivatisation reagent (trimethylsulfonium hydroxide (0.2 mol/L in MeOH). Unfortunately, there is not an expiry date in the certificate we have. To avoid getting bad results in the future, we ordered a new production reagent and will review our results.	B
75	2.5	Yes	The Concentration was determined directly without dilution. The determined concentration was not far from the highest calibration points. The repeat analysis was performed and the sample was diluted to the concentration at almost the same level as the average calibration points. It has been shown that the measured concentration for 2,4-D was then 0,094 mg/kg with an acceptable z-score.	C, L
3rd-139	6.6	(Yes)	2,4-D, fenbutatin oxide, fluazifop, haloxyfop standard are mixed together, standard may have degraded when we mixed together.	C
71	36.2	Yes	Transcription error. It should be 0.0791 mg/kg	J

Captan (parent) Assigned value: 0.085 mg/kg

LabCode	z-Score	Source of error localized?	Reason / Remarks	
70	-3.1	Yes	Captan is degraded in metabolite tetrahydrophthalimide (THPI) and Captan is underestimated.	O
98	-3.0	Yes	We used QuEChERS method (EN) without acidification for routine analysis and was used for this work. This method converts more of the parent into tetrahydrophthamide (THPI) and therefore the value for captan is underestimated and value for THPI is overestimated. However these effects cancel each other out for sum of two components, as required for MRL residue definition. Our result for full residue definition (sum) was within the acceptable range. We have re-analysed the PT sample using acidic extraction and our results are 0.079 mg/kg for captan and 0.141 mg/kg for THPI. These results would have achieved acceptable z-scores. I have introduced a corrective action to use acidic extraction method for positive samples.	G, Q

Appendix 7 (cont.) Possible Reasons for Poor Performance (ordered by z-scores)

Captan (parent) Assigned value: 0.085 mg/kg				
LabCode	z-Score	Source of error localized?	Reason / Remarks	
23	-2.8	No	error source not detected	
13	2.2	Yes	It was a stupid mistake by overlooking the result columns and reported result for mg/l not mg/kg. Our result is actually 0,087 mg/kg, so absolutely in line.	J
73	2.2	(Yes)	Probably due to a less degradation than other labs of this pesticide in the injector (GC-ECD)	O
52	2.5	No	Actually no idea, maybe analytical protectant was fault/old, Sum for captan was OK.	U
72	2.5	Yes	The high z-score for captan (parent) was due to a transcription error when averaging duplicate results. The correct value should have been 0.126 mg/kg, achieving a z-score of 1.9. There would have been a minor change to the captan (sum) result to 0.283 mg/kg and a z-score of -0.2.	J
125	2.5	–	We know that we got that values higher than the assigned value, but it is due to the analysis of captan, folpet and their degradation products by cool on column injection (GC-COC-MS/MS). So the degradation produced because of the temperature set in "normal" injection is not produced. Also we have a value for their degradation products lower than the assigned value, but the z score for the sum is ok (-0.6 and -0.7 for captan and folpet, respectively).	U
95	3.0	No	Analysis was performed using QuEChERS-Extract with SPE cleanup, not acidified by formic acid, but with ascorbic acid (5 µg/ml extract) as analytical protectant. Calculation was down by standard addition to sample extract (since the sample amount was not sufficient for standard addition to sample portion), and internal Standards Captan-D6 was added. Results of investigation: Standard solution was o.k., results from a new sample extract and standard addition was the same as that submitted. Conversion factor was correctly involved. No error source could be found. The degradation product THPI with an acceptable z-score (1.0) was analysed in the same way with the exception, that no internal standard was used. It is not explainable to us, that the captan (parent) was clearly overestimated, the degradation product THPI was not underestimate, and the sum of captan (parent) and THPI was again within acceptable range (|z-score|<2).	U
99	4.9	No	The submitted result was calculated using a calibration mix in the routine and strawberry as matrix. For the investigation we repeated the analysis with 4 sample portions, a freshly prepared calibration stock and strawberry as well as kiwi/cucumber as matrix. Captan-D6 was used as internal standard. The result: 0.056 mg/kg using strawberry as matrix; 0.106 mg/kg using kiwi/cucumber as matrix. No error source was found. No improvement, especially for the quantification, can be observed.	U
108	17.2	Yes	Transcription error: Laboratory has obtained results for captan 0.045 mg/kg, however the wrong concentration has been entered into the form (0.45 mg/kg).	J

Chlorothalonil Assigned value: 0.125 mg/kg				
LabCode	z-Score	Source of error localized?	Reason / Remarks	
27	-3.7 (FN)	No	We had poor calibration, poor recovery on matrix and unrepeatable results, too. Now, our GCMSMS is out of use because of poor sensitivity. After it will be fixed, we will repeat analyses of test sample strawberry puree.	U
23	-3.0	No	error source not detected	
98	-2.9	Yes	We used QuEChERS method (EN) without acidification for routine analysis and applied it in this work. This approach can lead to breakdown of chlorothalonil and results in underestimation and poor z-score. We have re-analysed the PT sample using acidic extraction and our results are 0.125 mg/kg for chlorothalonil. This would have achieved a very good z-score. I have introduced a corrective action to use acidic extraction method for positive samples.	G
112	-2.8	Yes	Instrument not yes evaluated for its capacity for quantification and maybe not sensitive enough. Result from the other instrument: 0.136 mg/kg	A

Appendix 7 (cont.) Possible Reasons for Poor Performance (ordered by z-scores)

A: Technical problems with measurement instrumentation
B: Procedure not properly conducted
C: Matrix effect not properly compensated
D: Lack of experience
E: Error in concentration of analytical standard
F: Error in the evaluation/interpretation of measurement data
G: Use of inappropriate procedure
H: Reporting limit higher than the assigned value
I: Sample weight too small, homogeneity insufficient
J: Transcription error
K: Result not corrected for low recovery
L: Inappropriate calibration
M: Detection signals strongly interfered by matrix components/Strong chromatographic interferences
N: Misunderstanding of the definition of the analyte
O: Degradation during sample preparation or measurement
P: Sample amount not sufficient for quantitative analysis
Q: Consecutive error (e.g. parent too low, degraded product too high, but sum o.k.)
U: Undefined

Chlorothalonil Assigned value: 0.125 mg/kg

LabCode	z-Score	Source of error localized?	Reason / Remarks	
80	-2.4	Yes	1) Chlorothalonil is not included in our scope (high acid content group). We had bad results in our in house validation, so this analyte was excluded from our accreditation scope for this kind of matrix with high acid content. 2) The aim of our participation in this trial was to check which method would be useful for this analyte, taking into account that we were testing two methods. 3) One of this methods was acidifying the extract with sulfuric acid, as described in http://www.eurl-pesticides.eu/docs/public/tmplt_article.asp?LabID=200&CntID=802&Theme_ID=1&Pdf=False&Lang=EN. The results obtained with this method was sent as participant in the SRM12 test. 4) In a parallel way we had tried another options, as matrix calibration (not calibration using a blank matrix extract, but making addition into a blank sample from beginning for calibration). This option leads to better results: chlorothalonil of 0.117 and 0.141 mg/kg, with a mean of 0.129 mg/kg that was close to the assigned value of 0.125 mg/kg. (The results were approximate because the area was over the highest calibration point.) In order to include this analyte in our scope, our effort will be directed to this way.	D, C
85	2.8	Yes	This compound is during implementation and the validation was not yet completed.	D
51	3.3	Yes	Analysing the data, we conclude that in problematic cases such as chlorothalonil, where many factors may influence in the study, the best method of quantification would be the standard addition. We didn't report the data of standard addition because we had only one value with Method M2.	L
3rd-137	5.1	Yes	Extraction method employed is not optimal, the QuEChERS method for multiresidues is used while it is recommended using a single method to analyse chlorothalonil. Besides, the conditions of the equipment were not the best for the analysis.	G
10	5.3	Yes	We tried to extent the number of analytes potentially present in the target pesticides list incl. analysis of chlorothalonil, but we had an expired standard in our lab. Probably the bad performance is related to degradation of standard (we used the same standard for spiking the positive control sample and recovery results were good, this means, in my opinion, that analytical method should be ok).	E
11	11.8	(Yes)	We analyse this pesticide with an evaporation to dry and finally a re-dissolving with isooctane. We suspect that we lose part of the internal standard in the evaporation process, this causes errors in the calculation of the final concentration.	G

Appendix 7 (cont.) Possible Reasons for Poor Performance (ordered by z-scores)

Dithiocarbamates Assigned value: 0.267 mg/kg

LabCode	z-Score	Source of error localized?	Reason / Remarks	
86	2.3	Yes	The calibration standards used during the first analysis were not properly prepared for the compound CS_2. We've re-analysed the sample using newly prepared calibration standards and found the concentration of the CS_2 was 0.339 mg/kg. This is equal to a z-score of 1.08"	E
75	3.1	Yes	This almost double-overestimation was caused by mistake in making calibration solutions and building calibration curve from them.	E
3rd-139	3.1	Yes	CS_2 standard was degraded on the date we preformed the test; we have recalculated result using standard ran 2 weeks before: new result is 0.290 ppm.	E
52	3.5	No	No idea, strong matrix interference	M
90	4.5	(Yes)	We usually quantify residue level with a matrix-matched standard solution instead of standard solutions in solvent as soon as a matrix affect is shown. For this analysis (assigned value at 0.267 mg/kg), residue levels were different according to the quantification and the matrix-matched quantification seemed to be the most accurate: - with standard solutions in solvent :0.157 mg/kg (closer to the assigned value) - with a matrix-matched standard solution : 0.566 mg/kg (z-score 4.5). If the spiking level is close to the assigned value, it doesn't seem to be relevant to quantify with a matrix-matched standard solution: the quantification has to be performed in solvent.	
35	8.3	Yes	This compound is not in our scope. The method used is the EURL method (Analysis of Dithiocarbamate Residues in Foods of Plant Origin involving Cleavage into Carbon Disulfide, Partitioning into Isooctane and Determinative Analysis by GC-ECD, 2nd Version) which is not optimised in our lab.	D
3rd-135	25.4	Yes	Root Cause : Methods that has not been properly documented has been used. Analyst has made an error in the preparation of standards. Corrective action : Test method for the above analysis has been documented.	E

Fenbutatin oxide Assigned value: 0.086 mg/kg

LabCode	z-Score	Source of error localized?	Reason / Remarks	
2	-3.5 (FN)	Yes	Correctly found and quantified (0.087 mg/kg), but the system setting for specific parameters was not correct, so that the result was not highlighted and not reported. This problem was already solved by the participant.	J
35	-3.5 (FN)	Yes	This compound is not in our scope. The extraction method used is the EURL method (QuEChERS with sulfuric acid, without dSPE) which is not optimised in our lab. We observed a very poor sensitivity for the detection of this compound, which comes from our chromatographic conditions that need to be optimised. We had a look at the chromatogram and there was no presence of fenbutatin oxide compared to the standard and to the recovery test, that is the reason why we reported as not detected.	D, A
99	-3.5 (FN)	Yes	For the PT a special measurement program containing only the SRM12 analytes was developed for analysis via LC-MS/MS. Unfortunately, the transition of fenbutatin oxide was by mistake not included.	J
52	-2.3	Yes	Recovery of only 60 %. If we had stated the for recovery corrected result, we would have achieved a z-score about 0.8.	K
30	2.3	(Yes)	The reason for higher concentration compared to the AV could be due to the matrix effect. The matrix calibration (procedural) were prepared on the other matrix (pears) than strawberry because high signal at the retention time of fenbutatin oxide in strawberry blank matrix was observed.	C
63	6.8	Yes	For this PT our lab staff prepared a fresh stock solution. The stock solution was prepared with acetonitrile. Unfortunately, fenbutatin oxide wasn't completely dissolved, however, the lab staff didn't notice that during the tests. To secure best possible performance for the future we decided to always buy fenbutatin oxide as a liquid 100ng/µL.	E
3rd-139	7.6	(Yes)	2,4-D, fenbutatin oxide, fluazifop, haloxyfop standard are mixed together, standard may have degraded when we mixed together.	E
75	9.5	Yes	In case of fenbutatin oxide we used several dilutions for the determination of the concentration, but the compound was overlapping. For fenbutatin oxide analysis we also used different analytical columns: sun fire columns and kinetex biphenyl column, but the overlapping persisted. In repeat analysis we used direct determination concentration without dilution the concentration was 0.118 mg/kg.	M

Appendix 7 (cont.) Possible Reasons for Poor Performance (ordered by z-scores)

A: Technical problems with measurement instrumentation
B: Procedure not properly conducted
C: Matrix effect not properly compensated
D: Lack of experience
E: Error in concentration of analytical standard
F: Error in the evaluation/interpretation of measurement data
G: Use of inappropriate procedure
H: Reporting limit higher than the assigned value
I: Sample weight too small, homogeneity insufficient
J: Transcription error
K: Result not corrected for low recovery
L: Inappropriate calibration
M: Detection signals strongly interfered by matrix components/Strong chromatographic interferences
N: Misunderstanding of the definition of the analyte
O: Degradation during sample preparation or measurement
P: Sample amount not sufficient for quantitative analysis
Q: Consecutive error (e.g. parent too low, degraded product too high, but sum o.k.)
U: Undefined

Glyphosate Assigned value: 0.306 mg/kg				
LabCode	z-Score	Source of error localized?	Reason / Remarks	
6	-3.6 (FN)	(Yes)	Reason not yet found, probably human error on extraction step, investigation in progress	B
3rd-132	-2.7	No	We couldn't find the problem. Standards and LC conditions were ok, recovery (87 %) was ok and we didn't make any calculation error. During the analysis, we couldn't repeat analysis a few times more because all the sample was used for GC determination. Plastic bottles were used for the sample preparation, sample extract was filled in the glass vials. **Note of organiser:** Plastic vials should be used for the analysis of glyphosate.	U
38	-2.3	(Yes)	We have less experience with this compound. We have analysed this compound only since this year using Quick Polar Pesticides Method and Obelisc N column. Due to this questionable result and the instability and poor reproducibility of this column, we will test a new analytical method (first step, extraction using the QuPPE method and the second step, derivatization using FMOC).	D
67	2.3	(Yes)	The situation was the following: The average of first 3 parallel samples was about 0.354 mg/kg but due to the high RSD% (= 30 %) and poor recovery (55.1 %) the procedure was repeated with second 3 parallel samples. Although the average result was similar to the previous one (0.339 mg/kg) and the recovery (76 %) and RSD% (24.5 %) better due to the result of phosphonic acid analysed from same sample the injection/analyses was repeated on next day. The 3rd compound analysed in this sample was the chlorate. During the repeating, all parameters remained the same: st. dilutions, sample extracts, spiked sample. But the result was quite convincing: RSD% = 1.6 %, recovery 101.4 %, however the average 0.485 mg/kg for glyphosate. We suspected, the sample was concentrated (all the time was on the autosampler) but due to lower average result of phosphonic acid with better recovery (104 %) and lower average result of chlorate with same recovery (87 %) we rejected this suspect. There was no any possibility to repeat the process because the test sample was run out.	F
78	2.7	Yes	Probably due to degradation of glyphosate stock solution in glass bottles. We have changed it and use now only plastic bottles for glyphosate stock solution.	E

Appendix 7 (cont.) Possible Reasons for Poor Performance (ordered by z-scores)

Glyphosate Assigned value: 0.306 mg/kg				
LabCode	z-Score	Source of error localized?	Reason / Remarks	
1	4.2	Yes	Degradation of the stock solution (Factor: 1.715. Applying this factor, it would be 0.367 mg/kg, corresponding to a z-score of 0.8)	E
97	6.1	Yes	Freshly prepared calibration and fortification solutions contained only half the concentration. Short of time to verify in another assay.	E
57	13.0	Yes	Inappropriate calibration. The 1: 4 dilution factor was not applied during the quantification of the result obtained. After its correct application a concentration of 0.343 mg/kg	F

Folpet (parent) Assigned value: 0.334 mg/kg				
LabCode	z-Score	Source of error localized?	Reason / Remarks	
23	-3.5	Yes	Transcription error (it was 0.400 mg/kg)	J
27	-3.1	No	We had poor calibration, poor recovery on matrix and unrepeatable results, too. Now, our GC-MS/MS is out of use because of poor sensitivity. After it will be fixed, we will repeat analyses of test sample strawberry puree.	U
63	-3.0	Yes	Phthalimide was not included in the result.	F
3rd-139	-2.9	No	Folpet was tested before last update of method: We didn't skip the dSPE step, and we didn't account for degradation product Phthalimide.	U
96	-2.6	Yes	Due to technical problems we were under enormous time pressure. At the beginning we integrated the interfering peak instead of folpet, but we had the time to correct this fault. Unfortunately, we also integrated an interfering peak instead of the ILIS for folpet and didn't correct this fault. After correcting this mistake we determine 0.251 mg/kg by standard addition which leads to a z-score of ~ -1.0.	F, M
112	-2.4	Yes	Lacking of reference material not yet checked if it is due to insufficient sensitivity of the instrument (s. also chlorothalonil)	A
98	-2.3	Yes	We used QuEChERS method (EN) without acidification for routine analysis and was used for this work. This method converts more of the parent to phthalimide and therefore the value for folpet is underestimated and value for phthalimide is overestimated. However these effects cancel each other out for sum of two components, as required for MRL residue definition. Our result for full residue definition (sum) was within the acceptable range. We have re-analysed the PT sample using acidic extraction and our results are 0.346 mg/kg for folpet and 0.57 mg/kg for phthalimide. These results would have achieved acceptable z-scores. I have introduced a corrective action to use acidic extraction method for positive samples.	G, Q
29	-2.1	Yes	The reason for the poor performance is the unsatisfactory method used for the quantification of folpet in our lab, which does not take the inter-injection variable breakdown of folpet to phthalimide into account. Analysis on GC-ECD yielded a result of about 0.25 mg/kg (low but not questionable/ unacceptable). We've looked on the results of folpet over the last 3 years (in total approx. 350 samples of fruits and vegetables) using GC-MS/MS and found one finding (SANTE MS/ MS identification criteria fulfilled) of phthalimide at a conc. above 0.01 mg/kg (folpet <0.01). The false negative rate to date has thus not been alarmingly high and the sum folpet calculated from this single actual finding (of PI) is below 0.03 mg/kg (current default MRL in apples). The method for folpet will be revised this autumn to cover the full residue definition. The method for captan will be revised as well. We will use the methodology presented on the EURL-SRM webpage as a starting point for the re-validation of these two active substances.	F

Appendix 7 (cont.) Possible Reasons for Poor Performance (ordered by z-scores)

A: Technical problems with measurement instrumentation
B: Procedure not properly conducted
C: Matrix effect not properly compensated
D: Lack of experience
E: Error in concentration of analytical standard
F: Error in the evaluation/interpretation of measurement data
G: Use of inappropriate procedure
H: Reporting limit higher than the assigned value
I: Sample weight too small, homogeneity insufficient
J: Transcription error
K: Result not corrected for low recovery
L: Inappropriate calibration
M: Detection signals strongly interfered by matrix components/Strong chromatographic interferences
N: Misunderstanding of the definition of the analyte
O: Degradation during sample preparation or measurement
P: Sample amount not sufficient for quantitative analysis
Q: Consecutive error (e.g. parent too low, degraded product too high, but sum o.k.)
U: Undefined

Folpet (parent) Assigned value: 0.334 mg/kg

LabCode	z-Score	Source of error localized?	Reason / Remarks	
125	2.3	–	We know that we got that values higher than the assigned value, but it is due to the analysis of captan, folpet and their degradation products by cool on column injection (GC-COC-MS/MS). So the degradation produced because of the temperature set in "normal" injection is not produced. Also we have a value for their degradation products lower than the assigned value, but the z score for the sum is ok (-0.6 and -0.7 for captan and folpet, respectively).	U
35	2.7	No	This compound is not in our scope . The method (QuEChERS citrate buffered, without dSPE) is not optimised in our lab. We haven't managed yet to identify the error source, as we used folpet D4 combined with standard addition for quantifying this compound. We obtained the same amount without correcting the result by standard addition. We obtained a concentration of 0.375 mg/kg with a recovery of only 64 % using the calculation sheet published lately by the EURL, that's why we preferred to provide the result obtained by standard additions with folpet D4.	A
40	3.4	(Yes)	We assume that the problem is in the stability of our standard and we expect a new standard to determine the problem	G
95	5.0	No	Analysis was performed using QuEChERS-Extract with SPE cleanup, not acidified by formic acid, but with ascorbic acid (5 µg/ml extract) as analytical protectant. Calculation was down by one standard addition to sample extract (since the sample amount was not sufficient for standard addition to sample portion), and internal standards Folpet-D4 was added. Results of investigation: Standard solution was o.k., results from a new sample preparation and standard addition was the same as that submitted. Conversion factor was correctly involved. No error source could be found. The degradation product PI with an acceptable z-score (-0.2) was analysed in the same way with the exception, that no internal standard was used. It is not explainable to us, that the folpet (parent) was clearly overestimated, the degradation product PI was not underestimate, and the sum of folpet (parent) and PI was again within acceptable range (|z-score|<2).	U
52	6.3	No	Actually no idea, maybe analytical protectant was fault/old, Sum for Folpet was OK	U

Appendix 7 (cont.) Possible Reasons for Poor Performance (ordered by z-scores)

Haloxyfop Assigned value: 0.070 mg/kg

LabCode	z-Score	Source of error localized?	Reason / Remarks	
62	-4.0 (FN)	Yes	It was my fault, that we missed haloxyfop. I put it to the GC pesticides but we screen it with LC! We found it in the LC screens and we quantified it: 0.054 mg/kg.	J
44	-3.4 (FN)	(Yes)	We have checked our method (calibration curve, recovery and SRM), we think that the most likely cause of the poor results is the used derivatization reagent (trimethylsulfonium hydroxide (0.2 mol/L in methanol). Unfortunately, there is not an expiry date in the certificate we have. To avoid getting bad results in the future, we ordered a new reagent and will review our results.	B
3rd-139	-3.1	(Yes)	2,4-D, fenbutatin oxide, fluazifop, haloxyfop standard are mixed together, standard may have degraded when we mixed together.	E
3rd-132	-2.2	No	We couldn't find the problem. Standards and LC conditions were ok, recovery (85 %) was ok and we didn't make any calculation error. During the analysis, we couldn't repeat analysis a few more times because all material was used for GC determination.	
75	2.9	Yes	The concentrations were determined directly without dilution. The determined concentration was not far from the highest calibration points. The repeat analysis was performed and the sample was diluted to the concentration at almost the same level as the average calibration points. It has been shown that the measured concentration for haloxyfop was then 0.084 mg/kg with an acceptable z-score.	C, L
71	38.6	Yes	Transcription error. It should be 0.0748 mg/kg	J

Bifenazate (sum) Assigned value: 0.267 mg/kg

LabCode	z-Score	Source of error localized?	Reason / Remarks	
58	2.3	Yes	a standard with an incorrect concentration was used (factor 1.81, applying the factor: concentration=0.234 mg/kg, new z-score:-0.53)	E
70	5.3	Yes	Bifenazate is degraded in standard solution. In the calibration, the peak corresponding to bifenazate was smaller than normal. So bifenazate concentration in the strawberry sample was overestimated.	E
3rd-134	3.4	(Yes)	We are focusing on the dithiocarbamate this time. After numerous of testing, we are pretty sure the result for dithiocarbamate is correct. Unfortunately, the remaining sample in good condition is not enough for regular pesticides analysis. We can only do a rough trial once and submit our data. The pesticide data is apparently not reliable. Many points, like sample condition and quality of pesticide standard, can be wrong in this rough trial.	P

Bromide ion Assigned value: 19.1 mg/kg

LabCode	z-Score	Source of error localized?	Reason / Remarks	
42	-4.0	Yes	It was a calculation error. Recalculation give an amount of 16.0 mg/kg.	F
129	-2.8	Yes	The instrument we used is 11 years old and in case of some compounds we have some problems with quantification. In May we bought a new one LC-MS/MS (Thermo-Quantiva), I run the PT again with this instrument and the results was perfect.	A
71	2.1	Yes	Sample weight 1 mg too small, resulting in loss of homogeneity. We repeated the analysis by weighting 5 grams of sample and we obtained a concentration of 23.8 mg/kg (Z-score of around 1). The original method was initially performed for aromatic herbs, so by weighting 1 gram we avoid some interferences in the ionic chromatograph. As a corrective measure, we are going to amend this procedure and we will establish a weight of 5 grams for high water content commodities such as fruits and vegetables, in which less interferences are expected.	I
45	2.2	Yes	The result was not corrected for the high recovery of 130 %. If we would have corrected it for recovery, the z-score would have been fine.	K
63	2.5	Yes	In this PT we used the QuPPe method that we want to use in the near future. Our accredited method uses GC after derivatisation.	D

Appendix 7 (cont.) Possible Reasons for Poor Performance (ordered by z-scores)

A: Technical problems with measurement instrumentation
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K: Result not corrected for low recovery
L: Inappropriate calibration
M: Detection signals strongly interfered by matrix components/Strong chromatographic interferences
N: Misunderstanding of the definition of the analyte
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U: Undefined

Chlorate Assigned value: 0.490 mg/kg

LabCode	z-Score	Source of error localized?	Reason / Remarks	
58	-3.2	Yes	A standard with an incorrect concentration was used (factor 4.71, applying this factor, concentration=0.480 mg/kg, new z-score: -0.08)	E
70	-3.0	Yes	In the calibration solution chlorate concentration is bigger than theoretical concentration. The peak corresponding to chlorate was bigger than normal. So chlorate concentration in the Strawberry sample was underestimated.	E
63	-2.2	Yes	The ISTD correction wasn't applied and will soon be used for the analysis.	K
129	-2.1	Yes	The instrument we used is 11 years old and in case of some compounds we have some problems with quantification. In May we bought a new one LC-MS/MS (Thermo-Quantiva), I run the PT again with this instrument and the results was perfect.	A
8	2.5	Yes	we have found a terrible error in the expression of the results for having given the chlorate value as potassium chlorate without having applied the conversion factor. Applying the factor we would have given: Chlorate: 0.537 mg / kg instead of .0.790 mg/kg	N
34	5.6	Yes	The standard was degraded.	E

Dithianon Assigned value: 0.294 mg/kg

LabCode	z-Score	Source of error localized?	Reason / Remarks	
95	-3.9 (FN)	Yes	Based on our validation data, dithianon in acid and sugar rich matrix can be determined using QuEChERS-Extract without clean up using PSA at a concentration around 0.02 mg/kg and the recovery rate of 10 – 20 %. If dithianon was detected in such a sample matrix, the quantification is followed using a single method based on SRM-12 (Extraction with acetonitrile, acidified by sulfuric acid, without citrate buffer.) This process was also applied in the PT. Since there was no signal in the chromatogram of the QuEChERS-Extract, no single method was applied. Obviously, this decision was not appropriate here. We re-analysed the sample but using the single method and got the concentration of dithianon at 0.286 mg/kg corresponding a z-score of -0.1.	G

Appendix 7 (cont.) Possible Reasons for Poor Performance (ordered by z-scores)

Dithianon Assigned value: 0.294 mg/kg				
LabCode	z-Score	Source of error localized?	Reason / Remarks	
52	-3.7	Yes	Transcription error (I put in the wrong number, measurement values: 0.219; 0.243; 0.235)	J
75	-3.7	Yes	Dithianon is not within laboratory's routine scope. The analysis were performed without acidifying the sample with 1 % HCOOH or H_2SO_4. The standard solution wasn't acidified, too. These factors affect the recovery crucially.	D
46	-3.4	(Yes)	Three possible explanations for the nonconforming dithianon results, namely: 1: We corrected the result by the doping efficiency: gross result = 0.122 mg/kg (300 % doping yield), 2: We introduced dithianon D4 before injection and not before extraction (to save the amount of solution used) 3: The calibration is done in the solvent and not in presence of matrix, there is perhaps a matrix effect: "red fruit" not yet checked. We regularly perform doping on peaches and apricots (dithianon regularly requested on this type of matrix) and our yields are in conformity. This is an analysis that is never asked for red fruits.	C
3	-2.6	(Yes)	We don't use dithianon D4 and we don't use procedural calibration in our routine procedure, it could be an explanation for the difference with labs who use one of this 2 kind of recovery corrections (our recovery is 71 %). As our method is not accredited for this analyt, we didn't do further investigation.	L
57	-2.5	Yes	The recovery obtained was only 51 %.	K
30	6.2	No	Unfortunately, I couldn't find any reasons associated with analytical detection.	
90	31.0	Yes	The technician made mistake in the preparation of the standard solution (factor 10). The result is indeed 0.257 mg/kg instead of 2.57 mg/kg and good (assigned value at 0.294 mg/kg).	E

Phosphonic acid Assigned value: 19.3 mg/kg				
LabCode	z-Score	Source of error localized?	Reason / Remarks	
63	-4	Yes	A dilution factor of 10 wasn't applied, therefore, the result was wrong of this factor.	F
57	-2.9	Yes	Procedure not properly conducted. Error in the preparation of the working solution of ILIS-P (WS1), water was used instead of acetonitrile. Analysis using our standard gave an average concentration of 5382.45 mg/kg.	E, B
99	-2.4	–	In the last two PTs, our lab also has submitted results for phosphonic acid lower than the assigned values. Usually, we analysed phosphonic acid together with glyphosate with a hypercarb column. So far we have not adapted the special chromatographic condition given in Method 1.4, since we modified the chromatographic condition using QuPPe M1.3 and the recovery rate was usually within 70 % and 120 %. Therefore, we have not ruled out a significant discrimination between phosphonic acid and phosphoric acid. For matrix with a lot of interference, e.g. Tea, the extract was diluted before measurement to reduce the interference.	U
129	-2.3	Yes	The instrument we used is 11 years old and in some compounds we have some problems with quantification. In May we buy a new one LC-MSMS (Thermo-Quantiva) and I run the PT's again in this instrument and the results was perfect	A
70	-2.1	Yes	Integration error. With the new integration, the result is 13,2 mg/kg (Z-score =-1,26) which is a correct result.	F
34	10.0	Yes	The standard was degraded.	E

N-Acetyl glyphosate Assigned value: 0.100 mg/kg				
LabCode	z-Score	Source of error localized?	Reason / Remarks	
6	-3.2 (FN)	(Yes)	Reason not yet found, probably low sensitivity of the equipment on ESI neg. mode. LOD for NAG too high on ESI neg. mode, investigation in progress	A

Appendix 7 (cont.) Possible Reasons for Poor Performance (ordered by z-scores)

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Q: Consecutive error (e.g. parent too low, degraded product too high, but sum o.k.)
U: Undefined

Captan (sum) Assigned value: 0.302 mg/kg

LabCode	z-Score	Source of error localized?	Reason / Remarks	
25	2.3	Yes	New result calculated with the new THPI result: 0.265 mg/kg, estimated z-score: -0.5)	
129	2.7	Yes	We don't have internal standard for this compounds. We order this. We used a chemical ionization, but it is something new for us and because of this we didn't had good quantification for this compounds	D
58	3.5	Yes	We did not use and follow the SRM method "Quantification of Residues of Folpet and Captan in QuEChERS Extracts" Version 3.1 (update 6/4/17).	L

Folpet (sum) Assigned value: 1.195 mg/kg

LabCode	z-Score	Source of error localized?	Reason / Remarks	
47	-3.1	Yes	Transcription error (please see phthalimide)	J
66	-2.1	Yes	Transcription error (Our folpet (parent) result was 0.440 mg/kg (z-score = 1.3) and phthalimide result was 0.444 mg/kg (z-score = 0.0). So our folpet sum (sum of folpet and phthalimide expressed as folpet) should be 1,337 mg/kg. But we had reported by mistake 0.579 mg/kg.	J
13	2.5	No	We used 1) ECD for detection, correction using ILIS was not possible; 2) just the folpet for calibration and quantified both compounds phthalimide and folpet as a sum; 3) calculation Excel-sheet provided by the organisers	U
129	2.6	Yes	We don't have internal standard for this compounds. We order this. We used a chemical ionization, but it is something new for us and because of this we didn't had good quantification for this compounds	D
58	3.1	Yes	We did not use and follow the SRM method "Quantification of Residues of Folpet and Captan in QuEChERS Extracts" Version 3.1 (update 6/4/17).	L
39	4.6	Yes	Phthalimide too high, consequently folpet too high	Q

Appendix 7 (cont.) Possible Reasons for Poor Performance (ordered by z-scores)

THPI Assigned value: 0.110 mg/kg				
LabCode	z-Score	Source of error localized?	Reason / Remarks	
47	2.2	Yes	We have reported an elevated z-score for THPI (z-score = 2.2) but the result of captan is slightly lowered (z-score = -1.1). Since THPI is a degradation compound of captan, and the result of captan is lowered, it is logic to find an elevated result for THPI. When assessing the sum of both compounds (in accordance with the residue definition), the z-score is 1.4, which is a good result. It is clear that there is no structural method problem.	Q
127	2.2	(Yes)	We have investigated the causes of the result of THPI, and the explication is we used isotopically labelled standards (ILIS) of the parent (captan D6) instead of other internal standard like chlorpyrifos D10. Captan D6 presented low sensibility and resolution. We think that was the reason we obtained a slightly higher result. **Note of organiser:** Captan D6 will be degraded during hot injection into GC, its amount varies from vial to vial and, therefore, not suitable as internal standards.	G
25	3.1	Yes	New calibration standard solution realized with another batch of reference standard (new result: 0.0907 mg/kg, estimated z-score: -0.7)	E
129	4.0	Yes	We used a chemical ionization, but it is something new for us and because of this we didn't had good quantification for this compounds.	D
58	4.4	Yes	We did not use and follow the SRM method "Quantification of Residues of Folpet and Captan in QuEChERS Extracts" Version 3.1 (update 6/4/17). For phthalimide and THPI, however, we did not calculate the concentration against the standards phthalimide and THPI (non degraded) but against phthalimide and THPI coming from degradation of standards folpet and captan. This causes a overestimation of the concentration leading to the high, positive z-scores. Conclusion: For a correct quantification the SRM method should be followed using also the ILIS.	L
34	6.3	Yes	The standard was degraded.	E
3rd-132	21.7	Yes	THPI and phthalimide are in implementation process and we hadn't experience with that analysis. We couldn't get a good results by GC-MS/MS and used GC-MS without confirmation. We submitted by mistake this result.	D

Phthalimide Assigned value: 0.446 mg/kg				
LabCode	z-Score	Source of error localized?	Reason / Remarks	
47	-4.0	Yes	Transcription error (The result of phthalimide was 0.518 mg/kg, but not reported due to an administrative fault. This result would have given a good z-score for phthalimide and folpet (sum).	J
13	3.3	No	unclear, no calculation error	
129	3.6	Yes	We used a chemical ionization, but it is something new for us and because of this we didn't had good quantification for this compounds.	D
58	4.2	Yes	We did not use and follow the SRM method "Quantification of Residues of Folpet and Captan in QuEChERS Extracts" Version 3.1 (update 6/4/17). For phthalimide and THPI, however, we did not calculate the concentration against the standards phthalimide and THPI (non degraded) but against phthalimide and THPI coming from degradation of standards folpet and captan. This causes a overestimation of the concentration leading to the high, positive z-scores. Conclusion: For a correct quantification the SRM method should be followed using also the ILIS.	L
39	9.5	Yes	Bad concentration standard of phthalimide which was degraded and increased the result of the sum.	E
3rd-132	10.0	Yes	THPI and phthalimide are in implementation process and we hadn't experience with that analysis. We couldn't get a good results by GC-MS/MS and used GC-MS without confirmation. We submitted by mistake this result.	D

Appendix 7 (cont.) Possible Reasons for Poor Performance (ordered by z-scores)

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Q: Consecutive error (e.g. parent too low, degraded product too high, but sum o.k.)
U: Undefined

False Positive Results

Analyte	LabCode	Reason / Remarks	
Abamectin	3rd-132	We made a transcription error, because the result for Abamectin was 0,0023 mg/kg.	J
Cyromazine	3rd-135	Misinterpretation of GC-MS/MS data by the analyst. Corrective action : Retraining on the interpretation of GC-MS/MS data to the analyst and all officers in Pesticide Laboratory."	F

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GENERAL PROTOCOL

for EU Proficiency Tests on Pesticide Residues in Food and Feed

Introduction

This protocol contains general procedures valid for all European Union Proficiency Tests (EUPTs) organised on behalf of the European Commission, DG-SANTE[1] by the four European Union Reference Laboratories (EURLs) responsible for pesticide residues in food and feed. These EUPTs are directed at laboratories belonging to the Network[2] of National Reference Laboratories (NRLs) and Official Laboratories (OfLs) of the EU Member States. OfLs from EFTA countries and EU-Candidate countries are also welcome to participate in the EUPTs. OfLs from Third countries may be permitted to participate on a case-by-case basis.

The following four EURLs for pesticide residues were appointed by DG-SANTE based on regulation 882/2004/EC[3]:

- EURL for Fruits and Vegetables (EURL-FV),
- EURL for Cereals and Feedingstuffs (EURL-CF),
- EURL for Food of Animal Origin and Commodities with High Fat Content (EURL-AO) and
- EURL for pesticides requiring Single Residue Methods (EURL-SRM).

The aim of these EUPTs is to obtain information regarding the quality, accuracy and comparability of pesticide residue data in food and feed reported to the European Union within the framework of the national control programmes and the EU multiannual co-ordinated control programme[4]. Participating laboratories will be provided with an assessment of their analytical performance that

[1] DG-SANTÉ = European Commission, Health and Food Safety Directorate-General

[2] For more information about the EURL/NRL/OfL-Network please refer to the EURL-Web-portal under: http://www.eurl-pesticides.eu

[3] 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

[4] European Commission Proficiency Tests for Pesticide Residues in Fruits and Vegetables, Trends in Analytical Chemistry, 2010, 29 (1), 70 – 83.

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they can use to demonstrate their analytical performance and compare themselves with other participating laboratories.

EUPT-Organisers and Scientific Committee

EUPTs are organised by individual EURLs, or by more than one EURL, in joint collaboration.

An **Organising Team** is appointed by the EURL(s) in charge. This team is responsible for all administrative and technical matters concerning the organisation of the PT, e.g. the PT-announcement, production of Test Item and Blank Material, the undertaking of homogeneity and stability tests, packing and shipment of the Test Item and Blank Material, handling and evaluation of the results and method information submitted by the participants and the drafting of the preliminary and final reports.

To complement the internal expertise of the EURLs, a group of external consultants that form the **EUPT-Scientific Committee** (EUPT-SC)[5] has been established and approved by DG-SANTE. The EUPT-SC consists of expert scientists with many years of experience in PTs and/or pesticide residue analysis. The actual composition of the EUPT-SC, the affiliation of each member is shown on the EURL-Website. The members of the EUPT-SC will also be listed in the Specific Protocol and the Final Report of each EUPT.

The EUPT-SC is made up of the following two subgroups:

a) An independent **Quality Control Group** (EUPT-QCG) and

b) An **Advisory Group** (EUPT-AG).

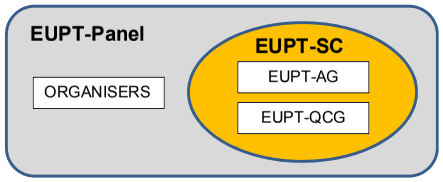
The EUPT-SC's role is to help the Organisers make decisions regarding the EUPT design: the selection of the commodity, the selection of pesticides to be included in the Target Pesticide List (see below), the establishment of the Minimum Required Reporting Levels (MRRLs), the statistical treatment and evaluation of participants results (in anonymous form), and the drafting and updating of documents such as the General and Specific PT Protocols and the Final EUPT-Reports.

The EUPT-QCG has the additional function of supervising the quality of EUPTs and of assisting the EURLs in confidential aspects such as the choice of the pesticides to be present in the Test Item and the concentrations at which they should be present.

[5] Link to the List of current members of the EUPT Scientific Committee: http://www.eurl-pesticides.eu/library/docs/allcrl/EUPT-SC.pdf

The EUPT-SC typically meets once a year, after the EUPTs of all four pesticide EURLs have been conducted, to discuss the evaluation of the EUPT-results and to consult with the EURLs in their decision making. Upcoming EUPTs are also planned during these meetings.

The EUPT-Organising Team and the EUPT-SC together form the **EUPT-Panel**.



The decisions of the EUPT-Panel will be documented.

This present EUPT General Protocol was jointly drafted by the EUPT-SC and the EURLs and was approved by DG-SANTE.

EUPT Participants

Within the European Union all NRLs operating in the same area as the organising EURL, as well as all OfLs whose scope overlaps with that of the EUPT, are legally obliged to participate in EUPTs. The legal obligation of NRLs and OfLs to participate in EUPTs arises from:

- Art. 28 of Reg. 396/2005/EC[6] (for all OfLs analysing for pesticide residues within the framework of official controls[7] of food or feed)
- Art. 33 of Reg. 882/2004/EC (for all NRLs)

The four EURLs will annually issue and distribute, via the EURL-website, a joint list of all OfLs that must participate in each of the EUPTs to be conducted within a given year. The list of obliged labs will be updated every year to take account of any changes in the lab profiles. Interim updates will be issued to eliminate any possible errors.

[6] 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.

[7] Official controls in the sense of Reg. 882/2004/EC, This includes labs involved in controls within the framework of national and/or EU-controlled programmes as well as labs involved in import controls according to Regulation 669/2009/EC.

EU REFERENCE LABORATORIES FOR RESIDUES OF PESTICIDES

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NRLs are responsible for checking whether all relevant OfLs within their network are included in the list of obligated laboratories and whether the contact information and commodity-scopes are correct.

OfLs are furthermore urged to keep their own profiles within the EURL-DataPool up-to-date, especially their commodity and pesticide scopes and their contact information.

Labs that are obliged to participate in a given EUPT, and that are not able to participate, must provide the reasons for their non-participation without prejudice of any legal action taken against them for not participating. This also applies to any participating laboratories that then fail to report results.

Confidentiality and Communication

The proprietor of all EUPT data is DG-SANTE and as such has access to all information.

For each EUPT, the laboratories are given a unique code (lab code), initially only known to themselves and the Organisers. In the final EUPT-Report, the names of participating laboratories will not be linked to their laboratory codes. It should be noted, however, that the Organisers, at the request by DG-SANTE, may present the EUPT-results on a country-by-country basis. It may therefore be possible that a link between codes and laboratories could be made, especially for those countries where only one laboratory has participated. Furthermore, the EURLs reserve the right to share EUPT results and codes amongst themselves: for example, for the purpose of evaluating overall lab or country performance as requested by DG-SANTE.

As laid down in Regulation 882/2004, NRLs are responsible for evaluating and improving their own OfL-Network. On request from the NRLs, the EURLs will provide them with the PT-codes of the participating OfLs belonging to their OfL-Network. This will allow NRLs to follow the participation and performance of the laboratories within their network.

Communication between participating laboratories during the test on matters concerning a PT exercise is not permitted from the start of the PT exercise until the distribution of the preliminary report.

For each EUPT the organising EURL prepares a specific EUPT-Website where all relevant documents in their latest version are linked.

The official language used in all EUPTs is English.

www.eurl-pesticides.eu

Announcement / Invitation Letter

At least 3 months before the distribution of the Test Item the EURLs will publish an Announcement/Invitation letter on the EURL-web-portal and distribute it via e-mail to the NRL/OfL mailing list available to the EURLs. This letter will inform about the commodity to be used as Test Item, as well as links to the tentative EUPT-Target Pesticide List and the tentative EUPT-Calendar.

Target Pesticide List

This list contains all analytes (pesticides and metabolites) to be sought, along with the Minimum Required Reporting Levels (MRRLs) valid for the specific EUPT. The MRRLs are typically based upon the lowest MRLs found either in Regulation 396/2005/EC or Commission Directive 2006/125/EC (Baby Food Directive).

Labs must express their results as stated in the Target Pesticides List.

Specific Protocol

For each EUPT the organizing EURL will publish a Specific Protocol at least 2 weeks before the Test Item is distributed to the participating laboratories. The Specific Protocol will contain all the information previously included in the Invitation Letter but in its final version, information on payment and delivery, instructions on how to handle the Test Item upon receipt and on how to submit results, as well as any other relevant information.

Homogeneity of the Test Item

The Test Item will be tested for homogeneity typically before distribution to participants. The homogeneity tests usually involve the analysis of two replicate analytical portions, taken from at least ten randomly chosen units of treated Test Item. Both, sample preparation and measurements should be conducted in random order.

The homogeneity test data are statistically evaluated according to ISO 13528, Annex B or to the International Harmonized Protocols jointly published by ISO, AOAC and IUPAC. The results of all homogeneity tests are presented to the EUPT-SC. In special cases, where the above homogeneity test criteria are not met, the EUPT-SC considering all relevant aspects (e.g. the homogeneity results of other pesticides spiked at the same time, the overall distribution of the participants' results, the analytical difficulties faced during the test, knowledge of the analytical behaviour of the

pesticide question) may decide to overrule the test. The reasons of this overruling have to be transparently explained in the Final EUPT-Report.

Stability of the analytes contained in the Test Item

The Test Items will also be tested for stability - according to ISO 13528, Annex B. The time delay between the first and the last stability test must exceed the period of the EUPT-exercise. Typically the first analysis is carried out shortly before the shipment of the Test Items and the last one shortly after the deadline for submission of results. To better recognise trends and gain additional certainty one or more additional tests may be conducted by the Organisers. At least 6 sub-samples (analytical portions) should be analysed on each test day (e.g. 2 analytical portions withdrawn from three randomly chosen containers OR 6 portions withdrawn from a single container). In principle all pesticides contained in the Test Item should be checked for stability. However, in individual cases, where sufficient knowledge exists that the stability of a certain analyte is very unlikely to be significantly affected during storage (e.g. based on experience from past stability tests or knowledge of its physicochemical properties), the Organisers, after consultation with the EUPT-QCG, may decide to omit a specific stability test. The EUPT-SC will finally decide whether analytes for which the stability test was not undertaken will be included in the final report, considering all relevant aspects such as the distribution of the participant's results (CV^*).

A pesticide is considered to be adequately stable if $|y_i - y| \leq 0.3 \times \sigma_{pt}$, where y_i the mean value of the last period of the stability test, y is the mean value of the first period of the stability test and σ_{pt} the standard deviation used for proficiency assessment (typically 25% of the assigned value).

The results of all stability tests are presented to the EUPT-SC. In special cases where the above stability test criteria are not met, the EUPT-SC considering all relevant aspects (e.g. the past experience with the stability of the compound, the overall distribution the participants' results, the measurement variability, analytical difficulties faced during the test and knowledge about the analytical behaviour of the pesticide question) may decide to overrule the test. The reasons of this overruling will be transparently explained in the Final EUPT-Report.

The Organisers may also decide to conduct additional stability tests at different storage conditions than those recommended to the participants e.g. at ambient temperature.

Considering knowledge about the expected susceptibility of pesticides in the Test Item to possible losses, the Organisers will chose the shipment conditions to be such that pesticide losses are minimised (e.g. shipment of frozen samples, addition of dry ice). As shipment time can differ between labs/countries it is recommended that the Organisers conduct additional stability tests at

conditions simulating shipment. Should critical losses be detected for certain pesticides the EUPT-SC will be informed (or the EUPT-QCG before or during the test). Case-by-case decisions may be taken considering all relevant aspects including the shipment time of the samples to each laboratory.

Methodologies to be used by the participants

Participating laboratories are instructed to use the analytical procedure(s) that they would routinely employ in official control activities (monitoring etc.). Where an analytical method has not yet been established routinely this should be stated.

General procedures for reporting results

Participating laboratories are responsible for reporting their own quantitative results to the Organiser within the stipulated deadline. Any pesticide that was targeted by a participating laboratory should be reported as "analysed". Each laboratory will be able to report only one result for each analyte detected in the Test Item. The concentrations of the pesticides detected should be expressed in 'mg/kg' unless indicated otherwise in the specific protocol.

The Test Item is intentionally treated with pesticides whereas the Blank Material is analysed to ensure that it does not contain any of the pesticides in the Target Pesticides List, at or above, the specified MRRLs. Both the Test Item and Blank Material have to be analysed by the participating laboratories and any pesticide detected in them must be reported.

Correction of results for recovery

According to the Method Validation and Quality Control Procedures for Pesticide Residues Analysis in Food and Feed[8], it is common practice that pesticide analysis results are not corrected for recovery if the recovery rates range between 70 and 120 %. Correction of results for recovery is recommended if the average recovery is significantly different from 100 % (typically if outside the 70 – 120 % range). Approaches for recovery correction explicitly stated in the DG-SANTE document are the use of recovery correction factors, the use of stable isotope labelled analogues of the target analytes as Internal Standards (ILISs), the 'procedural calibration' approach as well as

[8] Document N° SANTE/11945/2015; Method Validation and Quality Control Procedures for Pesticide Residues Analysis in Food and Feed

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the approach of 'standard addition' with additions of analyte(s) being made to analytical portions. Results may be corrected for recovery only in cases where this correction is applied in routine practice (including cases of MRL-violations). Laboratories are required to report whether their results were adjusted for recovery and, if a recovery factor was used, the recovery rate (in percentage) must also be reported. No recovery data are required where correction for recovery is automatic by adding amounts of analytes to the test portion for using the 'standard addition' approach, or isotopically-labelled internal standards (in both cases with spiking into the Test Item at the beginning of the extraction procedures) or procedural calibration. In these cases, the laboratories should report the actual approach that was followed.

Methodology information

All laboratories are requested to provide information on the analytical method(s) they have used. A compilation of the methodology information submitted by all participants is presented in an Annex of the final report or in a separate report. Where necessary the methods are evaluated and discussed, especially in those cases where the result distribution is not unimodal or very broad (e.g. CV* > 35 %). If no sufficient information on the methodology used is provided, the Organiser reserves the right not to accept the analytical results reported by the participants concerned or even refuse participation in the following PT.

Results evaluation

The procedures used for the treatment and assessment of results are described below.

- ***False Positive results***

These are results of pesticides from the Target Pesticides List, that are reported, at or above, their respective MRRL although they were: (i) not detected by the Organiser, even after repeated analyses, and/or (ii) not detected by the overwhelming majority (e.g. > 95 %) of the participating laboratories that had targeted the specific pesticides. In certain instances, case-by-case decisions by the EUPT-Panel may be necessary.

Any results reported lower than the MRRL will not be considered as false positives, even though these results should not have been reported.

– ***False Negative results***

These are results for pesticides reported by the laboratories as 'analysed' but without reporting numerical values although they were: a) used by the Organiser to treat the Test Item and b) detected by the Organiser as well as the majority of the participants that had targeted these specific pesticides at or above the respective MRRLs. Results reported as '< RL' (RL= Reporting Limit of the laboratory) will be considered as not detected and will be judged as false negatives. In certain instances, case-by-case decisions by the EUPT-Panel may be necessary.

In cases of the assigned value being less than a factor of 3 times the MRRL, false negatives will typically not be assigned. The EUPT-Panel may decide to take case-by-case decisions in this respect after considering all relevant factors such as the result distribution and the reporting limits of the affected labs.

– ***Estimation of the assigned value*** (x_{pt})

In order to minimise the influence of out-lying results on the statistical evaluation, the assigned value x_{pt} (= consensus concentration) will typically be estimated using robust estimate of the participant's mean (x^*) as described in ISO 13528:2015[9], taking into account the results reported by EU and EFTA countries laboratories only. In special justifiable cases, the EUPT-Panel may decide to eliminate certain results traceably associated with gross errors (see "Omission or Exclusion of results" below) or to use only the results of a subgroup consisting of laboratories that have repeatedly demonstrated good performance for the specific compound in the past.

– ***Omission or Exclusion of results***

Before estimating the assigned value results associated with obvious mistakes have to be examined to decide whether they should be removed from the population. Such gross errors may include incorrect recording (e.g. due to transcription errors by the participant, decimal point faults or transposed digits, incorrect unit), calculation errors (e.g. missing factors), analysis of a wrong sample/extract (e.g. a spiked blank), use of wrong concentrations of standard solutions, incorrect data processing (e.g. integration of wrong peak), major deviations from the analytical procedure,

[9] DIN ISO 13528:2015, Statistical methods for use in proficiency testing by interlaboratory comparisons, International Organization for Standardization. Therein a specific robust method for determination of the consensus mean and standard deviation without the need for removal of deviating results is described (Algorithm A in Annex C).

inappropriate storage or transport conditions (in case of susceptible compounds), and the use of inappropriate procedures that demonstrably lead to significantly biased results (e.g. due to degradation or incomplete extraction). Where the Organisers (e.g. after the publication of the preliminary report) receive information of such gross errors, having a significant impact on a generated result, the affected results will be examined on a case-by-case basis to decide whether, or not, they should be excluded from the population used for robust statistics. Results may also be omitted e.g. if an inappropriate method has been used even if they are not outliers. All decisions to omit/exclude results will be discussed with the EUPT-SC and the reasoning for the omission of each result clearly stated in the final EUPT-Report. However, z scores will be calculated for all results irrespective of the fact that they were omitted from the calculation of the assigned value.

Omitted results might be interesting as they might give indications about possible source(s) of errors. The Organisers will thus ask the relevant lab(s) to provide feedback on possible sources of errors (see also "follow-up activities").

Uncertainty of the assigned value

The uncertainty of the assigned values $u(x_{pt})$ is calculated according to ISO 13528:2015 as:

$$u\left(x_{pt}\right) = 1{,}25 \times \frac{s^*}{\sqrt{p}}$$

where s^* is the robust standard deviation and p is the number of results.

In certain cases and considering all relevant factors (e.g. the result distribution, multimodality), the number of submitted results, information regarding analyte homogeneity/stability, information regarding the use of methodologies that might produce a bias that were used by the participants), the EUPT-Panel may consider the assigned value of a specific analyte to be too uncertain and decide that the results should not be evaluated, or only evaluated for informative purposes. The provisions of ISO 13528:2015 concerning the uncertainty of the assigned value will be taken into account.

– ***Standard deviation of the assigned value (target standard deviation)***

The target standard deviation of the assigned value (FFP-σ_{pt}) will be calculated using a Fit-For-Purpose approach with a fixed Relative Standard Deviation (FFP-RSD) of 25% as follows:

$$\text{FFP-}\sigma_{pt} = 0.25 \times x_{pt}$$

The percentage FFP-RSD is set at 25% based on experience from results of previous EUPTs[10]. The EUPT-Panel reserves the right to also employ other approaches on a case-by-case basis considering analytical difficulties and experience gained from previous proficiency tests.

For informative purposes the robust relative standard deviation (CV^*) is calculated according to ISO 13528:2015; Chapter 7.7 (Consensus value from participant results) following Algorithm A in Annex C.

- ***z scores***

This parameter is calculated using the following formula:

$$z_i = \frac{(x_i - x_{pt})}{FFP\text{-}\sigma_{pt}}$$

where x_i is the value reported by the laboratory, x_{pt} is the assigned value, and $FFP\text{-}\sigma_{pt}$ is the standard deviation using FFP approach. Z scores will be rounded to one decimal place. For the calculation of combined z scores (see below) the original z scores will be used and rounded to one decimal place after calculation.

Any z scores > 5 will be typically reported as '> 5' and a value of '5' will be used to calculate combined z scores (see below).

Z scores will be interpreted in the following way, as is set in the ISO 17043:2010[11]:

$\|z\| \leq 2.0$	Acceptable
$2.0 < \|z\| < 3.0$	Questionable
$\|z\| \geq 3.0$	Unacceptable

For results considered as false negatives, z scores will be calculated using the MRRL or RL (the laboratory's Reporting Limit) if the RL < MRRL. The EUPT-Panel will decide whether, or not, these values should appear in the z score histograms.

- ***Category A and B classification***

[10] Comparative Study of the Main Top-down Approaches for the Estimation of Measurement Uncertainty in Multiresidue Analysis of Pesticides in Fruits and Vegetables. J. Agric. Food Chem., 2011, 59(14), 7609-7619.

[11] ISO/IEC 17043:2010. Conformity assessment – General requirements for proficiency testing

The EUPT-Panel will decide if and how to classify the laboratories into two categories - A or B. Currently, laboratories that are able to analyse at least 90% of the compulsory pesticides in the target pesticides list, have correctly detected and quantified a sufficiently high percentage of the pesticides present in the Test Item (at least 90 %) and reported no false positives will have demonstrated 'sufficient scope' and can therefore be classified into Category A. For the 90% criterion the number of pesticides needed to be correctly analysed to have sufficient scope will be calculated by multiplying the number of compulsory pesticides from the Target Pesticides List by 0.9 and rounding to the nearest full number with 0.5 decimals being rounded downwards (see some examples in Table 1).

Table 1. No. of pesticides from the Target Pesticides List needed to be targeted or pesticides present in the Test Item that need to be correctly detected and quantified to have sufficient scope.

No. of compulsory pesticides present in the Test Item / Target Pesticides List (N)	90 %	No. of pesticides needed to be correctly detected and quantified / targeted to have sufficient scope (n)	n
3	2.7	3	N
4	3.6	4	
5	4.5	4	N - 1
6	5.4	5	
7	6.3	6	
8	7.2	7	
9	8.1	8	
10	9.0	9	
11	9.9	10	
12	10.8	11	
13	11.7	12	
14	12.6	13	
15	13.5	13	N - 2
16	14.4	14	
17	15.3	15	
18	16.2	16	
19	17.1	17	
20	18	18	
21	18.9	19	
22	19.8	20	
23	20.7	21	
24	21.6	22	
25	22.5	22	N - 3
26	23.4	23	

– ***Overall performance of laboratories - combined z scores***

For evaluation of the overall performance of laboratories within Category A, the Average of the Squared z score (AZ^2)[12,13] (see below) will be used. The AZ^2 is calculated as follows:

$$AZ^2 = \frac{\sum_{i=1}^{n} z_i^2}{n}$$

[12] Formerly named "Sum of squared z scores (SZ^2)"

[13] Laboratory assessment by combined z score values in proficiency tests: experience gained through the EUPT for pesticide residues in fruits and vegetables. Anal. Bioanal. Chem., 2010, 397, 3061–3070.

Where n is the number of z scores to be considered in the calculation. In the calculation of the AZ^2, z scores higher than 5 will be set as 5. Based on the AZ^2 achieved, the laboratories are classified as follows:

$AZ^2 \leq 2.0$	Good
$2.0 < AZ^2 < 3.0$	Satisfactory
$AZ^2 \geq 3.0$	Unsatisfactory

Combined z scores are considered to be of lesser importance than the individual z scores. The EUPT-Panel retains the right not to calculate AZ^2 if it is considered as not being useful or if the number of results reported by any participant is considered to be too low.

In the case of EUPT-SRMs, where only a few results per lab may be available, the Average of the Absolute z scores (AAZ) may be calculated for informative purposes, but only for labs that have reported enough results to obtain 5 or more z scores. For the calculation of the AAZ, z scores higher than 5 will also be set as 5.

Laboratories within Category B will be ranked according to the total number of pesticides that they correctly reported to be present in the Test Item. The number of acceptable z scores achieved will be presented, too. The EURL-Panel retains the right to calculate combined z scores (see above) also for labs within Category B, e.g. for informative purposes, provided that a minimum number of results (z scores) have been reported.

Publication of results

The EURLs will publish a preliminary report, containing tentative assigned values and z score values for all pesticides present in the Test Item, within 2 months of the deadline for result submission.

The Final EUPT Report will be published after the EUPT-Panel has discussed the results. Taking into account that the EUPT-Panel meets normally only once a year (typically in late summer or autumn) to discuss the results of all EUPTs organised by the EURLs earlier in the year, the final report may be published up to 10 months after the deadline for results submission. Results submitted by non-EU/EFTA laboratories might not always be used in the tables or figures in the final report.

Certificates of participation

Together with the Final EUPT-Report, the EURL Organiser will deliver a Certificate of Participation to each participating laboratory showing the z scores achieved for each individual pesticide, the combined z scores calculated (if any), and the classification into Category A or B.

Feedback

At any time before, during or after the PT participants have the possibility to contact the Organisers and make suggestions or indicate errors. After the distribution of the Final EUPT-Report, participating laboratories will be given the opportunity to give their feedback to the Organisers and make suggestions for future improvements.

Correction of errors

Should errors be discovered in any of the documents issued prior to the EUPT (Calendar, Target Pesticides List, Specific Protocol, General Protocol) the corrected documents will be uploaded onto the website and in the case of substantial errors the participants will be informed. **Before starting the exercise participants should make sure to download the latest version of these documents.**

If substantial errors are discovered in the Preliminary EUPT-Report the Organisers will distribute a new corrected version, where it will be stated that the previous version is no longer valid.

Where substantial errors are discovered in the Final EUPT-Report the EUPT-Panel will decide whether a corrigendum will be issued and how this should look. The online version of the final report will be replaced by the new one and all affected labs will be contacted.

Where errors are discovered in EUPT-Certificates the relevant laboratories will be sent new corrected ones. Where necessary the laboratories will be asked to return the old ones.

Follow-up activities

Laboratories are expected to undertake follow-up activities to trace back the sources of erroneous or strongly deviating results (typically those with $|z| > 2.0$) - including all false positives. Even results within $|z| \leq 2.0$ may have to be checked if there is indications of a significant positive or negative bias.

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Upon request, the laboratory's corresponding NRL and EURL are to be informed of the outcome of any investigative activities for false positives, false negatives and for results with $|z| \geq 3.0$. Concerning z scores between 2.0 and 3.0 the communication of the outcome of follow-up activities is optional but highly encouraged where the source of deviation could be identified and could be of interest to other labs.

According to instructions from DG-SANTE, the "Protocol for management of underperformance in comparative testing and/or lack of collaboration of National Reference Laboratories (NRLs) with EU Reference Laboratories (EURLs) activities" is to be followed.

NRLs will be considered as **underperforming in relation to scope** if in at least two of the last four EUPTs falling within their responsibility area if they: a) haven't participated, or b) targeted less than 90% of the compulsory pesticides in the target lists (80% for SRM-compounds), or c) detected less than 90% of the compulsory compounds present in the test items (80% for SRM-compounds). Additionally, NRLs that obtained AZ^2 higher than 3 in two consecutive EUPTs of the last four EUPTs, will be considered as **underperforming in accuracy**. A two-step protocol established by DG-SANTE will be applied as soon as underperformance of an NRL is detected[14]:

Phase 1:

- Identifying the origin of the bad results (failure in EUPTs).
- Actions: On the spot visits and training if necessary and repetition of the comparative test if feasible and close the assessment of results by the EURL.

Phase 2:

- If the results still reveal underperformance the Commission shall be informed officially by the EURL including a report of the main findings and corrective actions.
- The Commission shall inform the Competent Authority and require that appropriate actions are taken.

Underperformance rules for the OfLs will be established at a later stage.

[14] Article 32 of the Regulation 882/2004

Appendix 8 (cont.) General EUPT Protocol (7th Ed.)

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Disclaimer

The EUPT-Panel retains the right to change any parts of this EUPT – General Protocol based on new scientific or technical information. Any changes will be communicated in due course.

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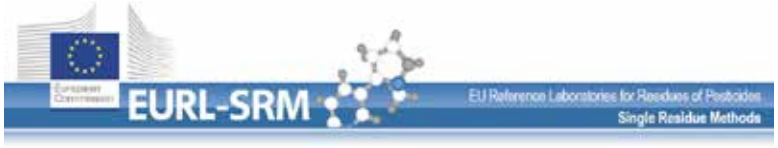

SPECIFIC PROTOCOL

for the 12th EU Proficiency Test
on Pesticides requiring Single Residue Methods
EUPT – SRM12 (2017)
(update on 27 February, 2017)

Introduction

This protocol is complementary to the valid version of the "**General Protocol for EU Proficiency Tests for Pesticide Residues in Food and Feed**" covering all EUPTs.
The EUPT-SRM12 is organised by the EU Reference Laboratory for pesticides requiring Single Residue Methods (EURL–SRM) which is **accredited according to ISO 17043** as providers of proficiency tests.
The EUPT-SRM12 deals with the analysis of SRM-pesticides in strawberry purée and is to be performed by all National Reference Laboratories for Single Residue Methods (NRL-SRMs) as well as by all official EU laboratories (OfLs) involved in official pesticide residue controls as far as their scope overlaps with that of the EUPT-SRM12. This includes laboratories involved in import control within the frame of Reg. 669/2009/EC. A special **EUPT-SRM12-Website** containing links to the most important documents of relevance was constructed.

Based on the information in the official Lab-Network-database hosted in the EURL DataPool and considering the commodity scope only (not the pesticide scope), the status of the OfLs concerning their participation in the current PT is shown on the registration page. As far as the EUPT-SRM12 is concerned, all OfLs analysing pesticides in fruits and vegetables were considered as obliged. OfLs listed as "obliged to participate in the EUPT-SRM12" but not intending to participate had to state their reasons for non-participation during the online registration of the EUPT-SRM12, which lasted from 17 January till 20 February, 2017.

Test Item and Blank Material

This EUPT deals with the analysis of pesticide residues in **Strawberry Purée**.

Participants will receive two bottles containing:

1) ca. 400 g **Test Item (with incurred or spiked analytes)**, containing pesticides from the **Target Pesticides List.**
2) ca. 400 g **Blank Material**, that can be used for recovery experiments as well as for the preparation of matrix-matched calibration standards

Using randomly chosen bottles, the Organizers will check the Test Item for sufficient homogeneity and for the stability of the pesticides contained over the period of the exercise. The Blank Material will be also checked to prove that none of the pesticides on the Target pesticides List is contained at relevant levels.

Target Analytes and MRRLs

The Test Item will contain several pesticides from the **EUPT-SRM12 Target Pesticides List.** Laboratories should read this list carefully, as it shows how the residues are expected to be reported as well as the **Minimum Required Reporting Levels (MRRLs)**. The MRRL values will be used to help identify false positive and false negative results and for the calculation of z-scores for false negatives. **Make sure to download the latest version of the EUPT-SRM12 Target Pesticides List before starting with analysis and result reporting.**
It should not be assumed that only pesticides registered for use in strawberry are present in the Test Item.

Shipment of Test Item

Test item and Blank Material are planned to be shipped on 13 March, 2017.
Frozen Test Item and Blank Material will be packed in thermo-boxes together with dry ice and shipped to the participants. Prior to shipment a reminder will be sent to the participating laboratories by e-mail.

Laboratories must make their own arrangements for the receipt of the package. They should **inform the Organisers of any public holidays in their country/city during the week of the shipment, and must make the necessary arrangements to receive the shipment, even if the laboratory is closed.**
Should any complications during shipment, delivery or the customs be expected, the participating laboratories should provide the Organizers with contact information of possible contact persons of the lab (e.g. mobile phone numbers) as well as instructions in local language explaining the need to keep the package in freezer during delay in transit and delivery. This information will be attached to the package.

Instructions on handling the Test Item

Once received, the Test Item should be stored deep frozen (at -18°C or lower) until analysis in order to minimize pesticide degradation and avoid any possible deterioration/spoilage.

Before analytical portions are taken for analysis, it is recommended to mix the material thoroughly in its entirety. While mixing, try to keep temperatures as low as possible to avoid the loss of unstable pesticides.

Participating laboratories should use their routine standard operating procedures for extraction, clean-up and analytical measurement as well as their own reference standards for identification and quantification purposes. Laboratories may also employ methods not yet implemented routinely, for example if they are in the test-phase of implementing them. In this case the limited experience and the non-inclusion of the analyte in the routine scope should be indicated in the result submission website.

The homogeneity tests will be conducted using 10 g analytical portions of Test Item for all analytes except for dithiocarbamates where expectedly 20 g will be used. Please note: Sub-sampling variability increases with decreasing analytical portion size, and sufficient homogeneity can only be guaranteed for sample portions ≥ 10 g.

Results submission website

Sample receipt acknowledgement, analytical results and method information are to be submitted via the following web-site: **EUPT-SRM12 result submission website**.

- **Sub-Page 0 (Sample receipt acknowledgement), accessible from 14 March, 2017.**
- **Sub-Pages 1-3 (analytical results and method information) accessible from 20 March, till 10 April, 2017.**
- **The deadline for result submission is 10 April, 2017 at 16 h (CEST).**

- Login Credential and Lab-Code

To access the data-submission forms participants must use their unique login credentials (username and password). The login credential together with the EUPT-SRM12 lab-codes the will be provided to each of the participating laboratories on the shipment day.

- Sample Receipt and Acceptance (Sub-Page 0)

Once the laboratory has received the Test Items it must report to the organiser via the **EUPT-SRM12 Result Submission Website** (sub-page 0) the date of receipt, the condition of the Test Item, and its acceptance. For laboratories in the EU- and EFTA countries and EU candidate countries, the deadline for acceptance is 17 March, 2017. If a laboratory does not respond by this deadline, the Organisers will assume that Test Item and Blank Material have been received and accepted.
Any participants that have not received the Test Items by the 17 March in the afternoon, they must inform the Organiser via e-mail (EURL-SRM@cvuas.bwl.de). The Organiser will consult the shipping company to localize the package and decide on further actions including new shipment, if necessary.
Selected participants might be asked to provide information on the condition of the Test Item upon receipt (e.g. core temperature of Test Item etc.).

- Reporting qualitative and quantitative Results (Sub-Page 1 and 2)

To report their results, laboratories must access the **EUPT-SRM12 Result Submission Website**.
All results must be reported on this website by 10 April, 2017 at 16 h (CEST). The website will not be accessible after this deadline, and all results submitted afterwards will not be accepted.

Before entering the results, please study the Target Pesticide List carefully, in particular the residue definitions that apply to the EUPT, which are not necessarily given in full on the Result Submission Website.

The following fields will be available for reporting the quantitative results:

- "**Concentration in mg/kg":** the pesticide concentrations that would be reported in routine work. Results should not be reported where a pesticide was not detected, or was detected below the RL (Reporting Limit) of the laboratory or the MRRL. Results reported as "< RL" or "< #,# mg/kg" will be considered as „Not Detected".

  The residue levels of the pesticides must be reported in mg/kg using the following **significant figures**:
  - Levels <0.010 mg/kg to be expressed to 2 significant figures, e.g. 0.0058 mg/kg;
  - Levels ≥ 0.010 mg/kg to be expressed to 3 significant figures, e.g. 0.156, 1.64, 10.3 mg/kg

  Recovery-corrected results should be reported only where this reflects the routine lab's procedure; otherwise the non-recovery-corrected result should be reported. Where a **result was corrected for recovery** the approach(es) followed to achieve this correction (e.g. standard additions to sample portions, procedural calibration, recovery must be reported in the respective fields in **Subpage 3** factor, use of ILIS).

- **"Conc. in blank in mg/kg":** concentration values of any pesticides from the **Target Pesticide List** determined in the Blank Material (even at levels below the MRRL).

- **"Experience with this compound":** Use the dropdown-menu to indicate for how many years you have been analysing for each compound using the method applied in this EUPT.

- Reporting Information on Analytical Methodology (Sub-Page 3)

On **sub-page 3 of** the "**EUPT-SRM12 Result Submission Website"** the participating laboratories must provide **COMPLETE** information on the analytical method(s) applied to **all pesticides which were analysed, irrespective of whether they were detected or not**.

The participating laboratories are urged to thoroughly fill-in all requested information and control it carefully in order to minimize the administrative burden of collecting and correcting it a posteriori.
If no sufficient information on the methodology used is provided, the Organisers reserve the right not to accept the analytical results reported by the participant or to refuse participation is future EUPT-SRMs.

For detailed information on the columns on sub-page 3 please refer to the detailed Guide on Result Submission (http://www.eurl-pesticides.eu/library/docs/srm/EUPT-SRM12_Short_Guide_SubPages.pdf).

Once your results are completely submitted, please export them via the function on the submission Main Page as a csv-format. This file can be viewed via Excel, and you can easily check your entries stored in the database.

Subcontracting

The following task was subcontracted to the EURL-CF, Søborg, Denmark:

a) Generation of the login credentials

b) Administration of EUPT-SRM12 result submission website

Follow-up actions

After the distribution of the EUPT-SRM12 Preliminary Report , laboratories with poor results (high absolute z-scores, false negatives or false positives) will be asked to provide information concerning the reasons for this and possible corrective actions. This information will be forwarded to the corresponding NRL-SRMs upon request. All EUPT-SRM12-participants are welcome to ask the EURL-SRM for technical assistance.
The Organiser might ask laboratories to provide missing methodology information that is important for the evaluation and interpretation of the PT.
According to instructions by DG-SANTE, the "**Protocol for management of underperformance in comparative testing and/or lack of collaboration of National Reference Laboratories (NRLs) with Community reference laboratories (CRLs) activities**" will be followed by NRLs.

Documents

All documents related to the EUPT–SRM12 can be found in the **EURL-Document Repository (CIRCA-BC).** Links to the documents can also be found in the **EUPT-SRM12 Website.**
For further information please contact the organizers **EURL-SRM@cvuas.bwl.de**
Please check the **EUPT-SRM12 Website** before starting with the analysis to **make sure that you have the latest version of all documents available**. In case of major changes the participants will be informed via e-mail.

Participation fees and payment details

To cover the costs of production, handling and shipment of the PT-Materials the following fees will be charged for one unit of the PT-Material to the participating laboratories:

- OfLs (including NRLs) from EU countries, EU-candidate countries and EFTA countries: 250 €
- Labs based in third countries: 350 €

An invoice issued to the "invoice address" stated in the registration form will be sent to the e-mail address of the invoice recipient stated during registration. Should the payment being taken care of by another department/institution, the recipient of the invoice is requested to forward the invoice accordingly. Details of payment will be given in the invoices.

Payment is expected to be made within 30 days upon the date of shipment.
If for any reason payment cannot be carried out before this date, please contact the Organizer to give explanations.
If no payment or no proof of payment is received and no explanation is given to the Organizers, the Organizers reserve the right to exclude the results of the concerned laboratories from the Final EUPT-Report or to refuse participation in future EUPT-SRMs.

Bank Details:

Bank account holder:	**Landesoberkasse Baden Wuerttemberg**
Bank Name :	**Baden Wuerttembergische Bank**
IBAN:	**DE 02 6005 0101 7495 5301 02**
BIC/SWIFT:	**SOLADESTXXX**
Payee identification text:	***See invoice (important and MUST be indicated!)***
VAT of CVUA Stuttgart	**DE 811 600 510**

To facilitate tracking of money transfer the special **payee identification text (= invoice number) as shown in the invoice MUST be indicated in the remittance.**

More details for bank-remittance will be given in the invoices.

Calendar of EUPT-SRM12

(please see: http://www.eurl-pesticides.eu/userfiles/file//EUPT-SRM12_Calendar.pdf)

Target Pesticides List of EUPT-SRM12

(please see: http://www.eurl-pesticides.eu/userfiles/file//EUPT-SRM12_TargetPesticideList.pdf)

Contact information

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Carmelo Rodríguez	University of Almería, Spain

Quality Control Group

Antonio Valverde	University of Almería, ES
Paula Medina	EFSA, Parma, EU

CALENDAR for the EUPT – SRM12

Strawberry Purée

(update on 17 January, 2017)

Activity	Who?	Dates
Opening of the **EUPT-SRM12 Website** with links to all relevant documents (List of obliged labs, **Calendar**, **Target Pesticides List**, **General Protocol**)	EURL-SRM	16 Dec., 2016
Registration via "**EUPT-Registration Website**" (Note: obliged OfLs MUST enter this Website and either register or give **explanations for non-participation**)	All Laboratories interested to participate and all obliged labs[1] even if not interested	**17 Jan. – 10 Feb. 2017**
Dispatch of EUPT-SRM12-Specific Protocol	EURL-SRM	Feb. 2017
Preparation of EUPT-SRM12-Test Item (preliminary tests Spiking / Homogenization)	EURL-SRM	Nov. 2016 – Feb. 2017
Homogeneity Tests	EURL-SRM	Jan. – Feb. 2017
Stability Tests	EURL-SRM	March – May 2017
Shipment of EUPT-SRM12 Test Item (+reminder of upcoming parcel arrival)	EURL-SRM	**13 March, 2017**
Confirmation of Sample Receipt and Acceptance via "EUPT-SRM12 Result Submission Website", (Sub-Page 0)	**Participating Labs**	**within 48 h of receipt**
Result Submission (Pesticide scope, Results, Method Info) in "EUPT-SRM12 Result Submission Website", (Sub-Pages 1 – 3)	**Participating Labs**	**20 March – 10 Apr. 2017**
Preliminary Report (only compilation of results)	EURL-SRM	May 2017
EUPT Evaluation Meeting	EUPT-SC, DG-SANTE	–
Survey to collect reasons for underperformance and missing information on methods	EURL-SRM / **Participating Labs**	May/June 2017
Final Report	EURL-SRM	Dec. 2017

REMARK: Please note that the dates mentioned above may be subject to minor **changes**. In the case of changes the participants will be informed via e-mail. **But please, still check periodically our website for possible updates** in case the email does not get through to you.
Contact: eurl-srm@cvuas.bwl.de

The EUPT-SRM Team

[1] These are OfLs of EU MS that are considered obliged to participate (please refer to the invitation letter for more details)

EU Reference Laboratory Requiring Single Residue Methods (EURL-SRM)
CVUA Stuttgart, Schaflandstr. 3/2, DE-70736 Fellbach, Germany
Website: www.eurl-pesticides.eu, E-Mail: EURL-SRM@cvuas.bwl.de

TARGET PESTICIDE LIST

for the EUPT – SRM12 2017, Strawberry Purée

released on 16.12.2016

Compounds Potentially Present in Test Item	In MACP	MRRL (mg/kg)
Compulsory Compounds (will be considered in Category A/B classification)		
2,4-D (free acid, no hydrolysis step to be applied)	MACP-Reg.	0.01
Abamectin (avermectin B1a)	MACP-Reg.	0.01
Captan (parent only)	MACP-Reg.	0.01
Chlorothalonil	MACP-Reg.	0.01
Cyromazine	MACP-Reg.	0.01
Dithiocarbamates (expressed as CS2)	MACP-Reg.	0.03
Ethephon	MACP-Reg.	0.02
Fenbutatin Oxide	MACP-Reg.	0.01
Fluazifop (free acid, no hydrolysis step to be applied)	MACP-Reg.	0.01
Folpet (parent only)	MACP-Reg.	0.01
Glyphosate (parent only)	MACP-Reg.	0.03
Haloxyfop (free acid, no hydrolysis step to be applied)	MACP-Reg.	0.01
Propamocarb	MACP-Reg.	0.01
Additional Data Collection for informative purposes (not for scoring)		
Captan (sum) (sum of captan and THPI, expressed as captan)	MACP-Reg.	
Folpet (sum) (sum of folpet and phtalimide, expressed as folpet)	MACP-Reg.	
THPI	MACP-Reg.	
Phtalimide	MACP-Reg.	
Optional Compounds (will NOT be considered in Category A/B classification)		
AMPA	MACP-WD	0.03
Bifenazate (sum) (sum of bifenazate plus bifenazate-diazene expressed as bifenazate)	MACP-WD	0.02
Bromide ion	MACP-Reg.	3.0
Carbofuran (part of sum) (sum of carbofuran, carbosulfan, benfuracarb or furathiocarb expressed as carbofuran); 3-OH-carbofuran is not to be considered within this exercize!	MACP-Reg.	0.001
Chlorate	MACP-WD	0.02
Dithianon	MACP-Reg.	0.02
Phosphonic acid	MACP-Reg.	0.05
N-Acetyl-Glyphosate	MACP-WD	0.02

MACP = EU Multi-Annual Coordinated Control Program;
MACP-Reg.: MACP Regulation; MACP-WD: MACP Working Document

Note: This document may be subject to minor changes. In case of significant changes the organizers will send e-mails. In any case please check our website periodically to make sure you are using the latest available version.

For any further clarification don't hesitate to contact us under eurl-srm@cvuas.bwl.de

The EUPT-SRM12 Organising Team

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