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 Soybean Flour

EUPT–SRM13

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Final Report

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cvua STUTTGART

EU PROFICIENCY TEST
EUPT-SRM13, 2018

Residues of Pesticides Requiring Single Residue Methods

Test Item: Soybean Flour

Final Report

Part One: Resutls Evaluation (excl. Phosphine)

Michelangelo Anastassiades
Pat Schreiter
Anja Barth
Hubert Zipper

approved by Michelangelo Anastassiades
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Organisers

EUPT-Coordinator:

Michelangelo Anastassiades

Head of EURL-SRM (Single Residue Methods)
CVUA Stuttgart
Department of Residues and Contaminants
Schaflandstrasse 3/2
D-70736 Fellbach

Phone: +49-711-3426-1124
Fax: +49-711-588176
E-Mail: Michelangelo.Anastassiades@cvuas.bwl.de

Organising Team:

EURL for Pesticides Requiring Single Residue Methods hosted by CVUA Stuttgart

Pat Schreiter, Senior Food Chemist
Anja Barth, Chemical Technician
Hubert Zipper, Senior Food Chemist
Carmen Wauschkuhn, Senior Food Chemist
Eric Eichhorn, Food Chemist
Nadine Kotte, Food Chemist
Giovanna Cerchia, Chemical Technician
Dorothea Mack, Chemical Technician
Sigrid Schüler, Chemical Technician
Sahra Söhnholz, Chemical Technician
Cristin Wildgrube, Chemical Technician

EURL for Pesticides in Cereals and Feedingstuff, National Food Institute, Technical University of Denmark
Jens-Ole Frimann, Software Programmer

Quality Control Group:

Antonio Valverde	University of Almería, ES
Paula Medina	European Food Safety Authority (EFSA)

Advisory Group:

Amadeo Fernández-Alba	EURL-FV, University of Almería (UAL), ES
Miguel Gamón	EURL-FV, Laboratorio Agroalimentario Generalitat Valenciana (LAGV), ES
Mette Erecius Poulsen	EURL-CF, National Food Institute (DTU), Søborg, DK
Ralf Lippold	EURL-AO, CVUA Freiburg, DE
Philippe Gros	Service Commun des Laboratoires (SCL) / Laboratoire de Montpellier, FR
Magnus Jezussek	Bavarian Health and Food Safety Authority (LGL), Erlangen, DE
André de Kok	Netherlands Food and Consumer Product Safety Authority (NVWA), Wageningen, NL
Sonja Masselter	Austrian Agency for Health and Food Safety (AGES), Innsbruck, AT
Finbarr O'Regan	Pesticide Control Laboratory (PCL), Department of Agriculture, Food and the Marine, IE
Tuija Pihlström	National Food Agency (Livsmedelsverket), Uppsala, SE
Patrizia Pelosi	National Institute of Health (ISS), Roma, IT
Carmelo Rodríguez	University of Almería (UAL), ES

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 thirteen 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 remaining four EUPT-SRMs were organized by the EURL-SRM unilaterally, two of them used commodities from plant origin with low fett content : milled dry lentils (EUPT-SRM7, 2012) and soybean slour (EUPT-SRM12, 2017). The EUPT-SRM9 was the only EUPT-SRM so far, in which a commodity of animal origin (cow's milk) was used. The current PT using flour of whole soybeans was the first one using a commodity with low water and high oil content (approx. 22 %).

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. In order to have sufficient results for evaluation of phosphine, a few private laboratories were exceptionally invided to participate in the present PT. These private labs were also allowed to report results also for any other pesticides in the target pesticides list, but those results was neither used to establish the assigned values nor be presented in this report.

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

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.

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

7. APPENDICES A-1

EUROPEAN COMMISSION –
EU-PROFICIENCY TEST ON RESIDUES OF PESTICIDES
REQUIRING SINGLE RESIDUE METHODS
TEST ITEM: SOYBEAN FLOUR
EUPT-SRM13, 2018

INTRODUCTION

On 23 Januar, 2018 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 13th European Commission's Proficiency Test Requiring Single Residue Methods (EUPT-SRM13). The EUPT-SRM13-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. 9 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 27 February to 16 March, 2018.

Based on its commodity scope (fruit and vegetables, feed, and commodities with high fett content) and its NRL-status (NRL-SRMs) a tentative status of a laboratory to be obliged to participate in the EUPT-SRM13 was stored in the DataPool, so that every participant can see it during the registration. 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 status of the laboratories 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 114 participating labs from EU and EFTA countries, 2 OfLs from an EU candidate country and 8 laboratories from third countries submitted results of at least one compound. Except 5 laboratories from EU and EFTA countries all other registered laboratories have submitted results.

The proficiency test EUPT-SRM13 was conducted using organic soybean flour originated from Austria and organic soybeans from EU countries. The test item was prepared by spiking the soybeans with 15 compounds highly concentrated dissolved in standard solutions, milling, filtering and mixing with the organic soybean flour. 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 soybean flour was chosen as commodity for the EUPT-SRM13.

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, one batch of organic soybean flour were purchased from a food processing company and checked for the absence of the analytes on the Target Pesticides List. None of the target pesticides was detected except ***phosphonic acid*** at 0.04 mg/kg and ***bromide ion*** at 1.3 mg/kg. More soybean flour of the same batch was purchased. This batch was finally used for the preparation of the blank material and for the preparation of the test item by spiking with 15 compounds (see **Section , p. 3**).

The minimum required reporting levels (MRRLs) were set at 0.003 mg/kg for ***haloxyfop***; at 0.005 mg/kg for *carbofuran* and ***phosphine***; at 0.01 mg/kg for *2,4-D*, *chlormequate-Cl*, ***cyromazine***, ***fluazifop***, ***mepiquat-Cl***, ***2,4-DB***, *chlorate*, *fenoxaprop*, ***perchlorate*** and ***quizalofop***; at 0.02 mg/kg for *ethephon*, *bentazone,* ***diquat***, ***glufosinate***, ***MPP***, ***N-acetyl glufosinate***, and ***N-acetyl glyphosate***, and *paraquat*; at 0.03 mg/kg for ***glyphosate***; at 0.05 mg/kg for *AMPA* and ***phosphonic acid*** and at 2.0 mg/kg for ***bromide ion***.

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

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.

1.2.1 Recovery Study

A recovery study (n = 5) for all analytes listed on the Target Pesticides List was conducted. Recoveries were found to be (65 %) 93 – 117 %.

1.2.2 Investigation on Homogeneity and on the Behavior of Analytes during Test Material Preparation using soybean flour in comparison with soybeans

The experiments consisted of spiking one portion (100 g) of soybean flour and one portion (100 g) of soybeans with a representative selection of pesticides listed on the Target Pesticides List as a mixture. The spiking was conducted in a metallic container. The spiked soybeans were dried and afterwards ground in a Thermomix® adding dried ice. The test materials both prepared with soybean flour and soybean were homogenized with blank soybean flour 1:10. These small scale test materials were analyzed for the yield of all spiked analytes using QuEChERS method and QuPPe PO method (n = 7).

Results showed that with the test material prepared with soybean flour acceptable yields (86 % – 109 %) could be obtained. However the results for homogeniety were not satisfying (RSD 22 % – 30 %)

Test material which was prepared with soybeans homogenized with soybean flour, obtained yields showed acceptable results for some analytes but also very low yields (37 % – 38 %) especially in case of highly polar pesticides. Here results for homogeniety were satisfying (RSD 4 % – 15 %).

It was concluded that another test is needed where the spiking of soybeans is conducted in a plastic container.

1.2.3 Investigation on the Behavior of Analytes during Test Material Preparation using Soybeans

1 kg soybeans was spiked with a representative selection of pesticides listed on the Target Pesticides List as a mixture. The spiking was conducted in a plastic container. The spiked soybeans were dried and afterwards ground in a Thermomix adding dried ice. This small scale test material was analyzed for the yield of all spiked analytes using QuEChERS method and QuPPe PO method (n = 4).

Yields obtained with the test material which was prepared in a plastic container showed satisfying results ((65 %) 90 – 119 %) and also satisfying homogeneity (RSD 2 % – 12 % (26 %)).

Using soybeans for spiking (in a plastic container) followed by dilution and homogenization with soybean flour was considered appropriate for the preparation of the test item.

1.3 Preparation and Bottling of the Blank Material

Treatment of blank soybeans was conducted to obtain the same conditions present in the sample material.

2.5 kg soybeans were spiked with a solvent mixture which corresponded both in volume and composition to the spiking solution (see below). These soybeans were dried and ground in a Thermomix® adding dry ice. Approximately 50 kg organic soybean flour from 5 packages, each containing 20 kg, were pooled in a large metal vessel, where it was layered with the processed blank soybeans. The blank material was mixed with a drum-hoop mixer over 10 h. Approximately 200 g portions of the well-mixed blank soybean flour 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.

1.4 Preparation and Bottling of the Blank Material

The test item was prepared in the same way as the blank material described above, but instead of adding pure solvent 150 ml of an equally composed mixture containing the target analytes was added. The mixture contained 14 different compounds and was prepared as described in **Table 1-1 (p. 3).**.

These two processed soybean flours and approximately 50 kg organic soybean flour from 5 packages were layered, and pooled in a large metal vessel. The spiked material was mixed with a drum-hoop mixer over 10 h. Approximately 200 g portions of the well-mixed spiked soybean flour 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.

Table 1-1: Analytes spiked into 50 kg soybean flour for the preparation of the test material

Analytes dissolved in 50 ml H_2O		Theor. Conc.	Analytes dissolved in 100 ml Aceton		Theor. Conc.
Compound	Amount	[mg/kg]	Compound	Amount	[mg/kg]
Bromide-K	1097.84 mg	14.742 [1)]	2,4-DB	10.238 mg	0.205
Cyromazine	5.64 mg	0.113			
Glyphosate	50.27 mg	1.005			
Mepiquat-Cl	6.8 mg	0.136			
Perchlorate-Na	6.473 mg	0.105 [2)]			
Diquat-dibromid hydrate	152.203 mg	1.549 [3)]	Spiking using Stock Solution (1 mg/ml ACN)		Theor. Conc.
Glufosinate-Al	11.098 mg	0.203 [4)]	Compound	Amount	[mg/kg]
MPPA	8.826 mg	0.177	Haloxyfop (free acid)	0.8 ml	0.016
N-Acetyl-glyphosate	30 mg	0.600	Fluazifop (free acid)	2.6 ml	0.052
Phosphonic acid	103.064 mg	2.061	Quizalofop (free acid)	3.0 ml	0.060

1) as bromide ion; 2) as perchlorate; 3) as diquate dication; 4) as glufosinate

1.5 Packaging and Delivery of PT Materials to Participants

Three days prior to the sample delivery, one bottle of test item and one of blank material, both deep frozen, as well as were packed into thermo-insulated polystyrene boxes, filled with four cooling elements and stored at –20 °C for three days, so that at the day of delivery the cooling elements were deep frozen. Once the parcel was picked up by DHL, the recipient received an e-mail from the shipping company entailing the individual online tracking number.

Among the 122 packages sent to laboratories in EU and EFTA countries, 106 (87 %) reached the participating labs within 24 hours and 11 packages within 48 hours. Due to the remote location of certain laboratories and holidays in certain countires, the remaining 5 packages took 3 days to arrive. The delivery to countries outside the EU and EFTA zones was accomplished within 48 hours in 3 cases, within 72 hours in 2 cases, within 4 days in 1 case, and more than 7 days in 4 cases. The latter was, however, due to delays at the customs. Overall, the EUPT-materials arrived at the laboratories very cold within two days. All material was accetped by the participants. Details on the shipment duration are shown in **Appendix 2**.

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 in case of opening time.

1.6 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 (p. 4)**. 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
2,4-DB	**Modified QuEChERS-method** [3] involving: weighing of 5 g soybean flour into a sealable vessel, addition of 10 ml H_2O, extraction with ACN (15 min), addition of partitioning salts (4 g $MgSO_4$, 1 g NaCl, 1 g trisodium citrate dihydrate and 0.5 g disodium hydrogencitrate sesquihydrate), 1 min shaking, centrifugation, addition of IS / ILISs to raw extract and direct determination by LC-MS/MS in the ESI (neg.) and ESI (pos) mode.	**BNPU**	**LC-MS/MS**	**ESI (neg)**	
Fluazifop		**BNPU**	**LC-MS/MS**	**ESI (neg)**	
Haloxyfop		**BNPU / Haloxyfop D_4**	**LC-MS/MS**	**ESI (neg)**	
Quizalofop		**BNPU / Quizalofop D_3**	**LC-MS/MS**	**ESI (neg)**	
*2,4-D**		*BNPU*	*LC-MS/MS*	*ESI (neg)*	
*Bentazone**		*BNPU*	*LC-MS/MS*	*ESI (neg)*	
*Carbofuran**		*Chlorpyrifos D_{10}*	*LC-MS/MS*	*ESI (pos)*	
*Fenoxaprop**		*BNPU*	*LC-MS/MS*	*ESI (neg)*	–
Bromide ion	**QuPPe-PO method** [5] involving: weighing of 5 g soybean flour into a sealable vessel, addition of ILISs, addition of methanol containing 1 % formic acid, shaking, centrifugation, Filtration, cleanup with C18 and possibly dilution with MeOH containing 1 % formic acid. Determination by LC-MS/MS in the ESI (neg.) or ESI (pos.) mode.	–	**LC-MS/MS**	**ESI (neg)**	**QuPPe M1.3**
Cyromazine		**Cyromazine D_4**	**LC-MS/MS**	**ESI (pos)**	**QuPPe M4.2**
Glufosinate		**Glufosinate D_3**	**LC-MS/MS**	**ESI (neg)**	**QuPPe M1.3**
Glyphosate		**Glyphosate ^{13}C ^{15}N**	**LC-MS/MS**	**ESI (neg)**	**QuPPe M1.3**
Mepiquat		**Mepiquat D_3**	**LC-MS/MS**	**ESI (pos)**	**QuPPe M4.2**
MPP		**MPP D_3**	**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 M1.3**
Perchlorate		**Perchlorate $^{18}O_4$**	**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*
*Chlorate**		*Chlorate $^{18}O_3$*	*LC-MS/MS*	*ESI (neg)*	*QuPPe M1.4*
*Chlormequat**		*Chlormequat D_4*	*LC-MS/MS*	*ESI (pos)*	*QuPPe M4.2*
*Ethephon**		*Ethephon D_4*	*LC-MS/MS*	*ESI (pos)*	*QuPPe M4.2*
*N-Acetyl-Glufosinate**		*N-Acetyl-Glufosinate D_3*	*LC-MS/MS*	*ESI (neg)*	*QuPPe M1.3*
*Paraquat**		*Paraquat D_6*	*LC-MS/MS*	*ESI (pos)*	*QuPPe M4.1*
Diquat	**QuPPe-PO method** [5] involving: weighing of 5 g soybean flour into a sealable vessel, addition of ILIS, addition a 1:1 mixture of methanol+ aqueous HCl 0.1 M, shaking, centrifugation, Filtration, cleanup with C18 and determination by LC-MS/MS in the ESI (pos.) mode.	**Diquat $^{13}C_2$**	**LC-MS/MS**	**ESI (pos)**	**QuPPe M4.1**
Phosphine	xxxxxxxxxxxxxxxxxx	**H_2S**	**GC-MS**	**EI (pos)**	

* : To check for absence in Blank Material

1.7 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 1 g for ***phosphine*** and 5 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-SRM13.

1.8 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

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

COMPULSORY COMPOUNDS										
	Bromide ion	Cyromazine	Fluazifop free acid	Glyphosate	Haloxyfop free acid	Mepiquat-Cl				
Analytical portion size [g]	5	5	5	5	5	5				
Mean [mg/kg]	14.6	0.107	0.049	0.924	0.016	0.126				
s_{sam}^2	2.34×10^{-1}	6.76×10^{-6}	2.73×10^{-6}	4.58×10^{-4}	4.34×10^{-8}	2.82×10^{-5}				
c	3.30×10^{0}	1.62×10^{-4}	2.89×10^{-5}	1.27×10^{-2}	3.27×10^{-6}	2.15×10^{-4}				
Passed/Failed	passed	passed	passed	passed	passed	passed				
OPTIONAL COMPOUNDS										
	2.4-DB free acid	Diquat dication	Glufosi-nate	MPP	N-Acetyl-Glyphosate	Perchlorate	Phosphonic acid	Quizalofop free acid	Phosphine (test Item)	Phosphine (PH3-Tube)
Analytical portion size [g]	5	5	5	5	5	5	5	5	1	1
Mean [mg/kg]	0.177	1.46	0.197	0.175	0.759	0.103	1.99	0.058	0.168	0.068
s_{sam}^2	1.44×10^{-5}	3.41×10^{-3}	2.25×10^{-4}	5.90×10^{-5}	1.79×10^{-3}	3.91×10^{-6}	7.33×10^{-3}	2.06×10^{-6}	2.34×10^{-7}	0×10^{-0}
c	4.11×10^{-4}	3.44×10^{-2}	8.71×10^{-4}	4.19×10^{-4}	8.58×10^{-3}	1.48×10^{-4}	5.46×10^{-2}	4.24×10^{-5}	8.71×10^{-8}	1.14×10^{-4}
Passed/Failed	passed	passed	passed	passed	passed	passed	passed	passed	passed	passed

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 1.6 (p. 3)**. 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):
03 May 2018 (analytes via QuPPe-Methods)
25 April 2018 (analytes via QuEChERS-Methods)
28 May 2018 (***phosphine (PH3-Tube)***)
20 June 2018 (***phosphine (test item)***)

Stability test 2 (extraction five weeks or more after shipment):
22 May 2018 (analytes via QuPPe-Methods)
17 May 2018 (analytes via QuEChERS-Methods)
19 June 2018 (***phosphine (PH3-Tube)***)
19 July 2018 (***phosphine (test item)***)

Stability test 3 (extraction at least four weeks after deadline for results submission):
18 June 2018 (analytes via QuPPe-Methods)
07 June 2018 (analytes via QuEChERS-Methods)
18 July 2018 (***phosphine (PH3-Tube)***)
24 Sept. 2018 (***phosphine (test item)***)

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 ***phosphine*** 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. 7**). 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.

In the case of ***phosphine (PH3-Tube)*** the determined concentration in day 3 was lower than that determined in day 1 by 20.0 %. This decline was significantly higher than the tolerance of 8.725 % (= $0.3\sigma_{pt}$ using FFP-RSD of 25 %). In case of ***phosphine (PT-Sample)***, compared with the concentration on day 1, the determined concentration in day 3 declined only 4.25 %. However, the determined concentration in day 2 deviated by 17.29 % that was higher than the tolerance of $0.3\sigma_{pt}$.

This fluctuation of the results of the stability test and the broad standard diviation reflected the fact that the analytical method was not robust. The analytical results of ***phosphine*** is thus associated with a considerable uncertainty. The determined deviations are probably related more to spurious analytical errors rather than to degradation. The higher concentration in day 2 compared to day 1 also points towards this direction. As analysis of ***phosphine*** is an optional compound in the PT, no further measures concerning its stability were deemed necessary, but report will be issued separately for its evaluation.

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

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										
	Bromide ion	Cyromazine	Fluazifop free acid	Glyphosate	Haloxyfop free acid	Mepiquat-Cl				
Storage at –18 °C (mean values in mg/kg)										
Analysis 1	13.729	0.105	0.050	0.913	0.016	0.123				
Analysis 2	14.054	0.105	0.050	0.964	0.016	0.128				
Analysis 3	14.110	0.111	0.049	0.933	0.017	0.131				
Deviation [mg/kg] ([%]) Analysis 3 vs. Analysis 1	0.381 (2.8 %)	0.006 (5.3 %)	0.001 (-1.7 %)	0.02 (2.2 %)	0.001 (7.1 %)	0.008 (6.2 %)				
$0.3 \times \sigma_{pt}$ [mg/kg]	1.152	0.007	0.004	0.068	0.001	0.009				
Passed/Failed	passed	passed	passed	passed	passed	passed				
COMPULSORY COMPOUNDS										
	2.4-DB free acid	Diquat dication	Glufosi-nate	MPP	N-Acetyl-Glyphosate	Perchlorate	Phosphonic acid	Quizalofop free acid	Phosphine (test Item)	Phosphine (PH3-Tube)
Storage at –18 °C (mean values in mg/kg)										
Analysis 1	0.177	1.423	0.186	0.170	0.734	0.101	1.922	0.058	0.180	0.063
Analysis 2	0.183	1.359	0.183	0.174	0.773	0.101	1.959	0.059	0.149	0.069
Analysis 3	0.165	1.422	0.186	0.165	0.742	0.108	1.939	0.058	0.188	0.080
Deviation [mg/kg] ([%]) Analysis 3 vs. Analysis 1	0.012 (-7.0 %)	0.001 (-0.1 %)	0 (0 %)	0.004 (-2.6 %)	0.008 (1.1 %)	0.007 (6.5 %)	0.016 (0.9 %)	0.001 (-1.2 %)	0.008 (4.25 %)	0.013 (20.0 %)
$0.3 \times \sigma_{pt}$ [mg/kg]	0.014	0.128	0.014	0.014	0.063	0.007	0.140	0.004	0.007	0.003
Passed/Failed	passed	passed	passed	passed	passed	passed	passed	passed	passed	failed

1.9 Transport Stability Test

With the exception of 7 laboratories with remote location or being on holiday and further 5 laboratories where the shipments were retarded due to customs clearance delays or remote location, all other 120 laboratories (91 %) received their test items within 48 hours. Among these 120 laboratories 106 received the parcels within one day. As no significantly negative influence of the long transport duration on the results from the 12 laboratories receiving the parcels more than 72 hours upon shipment was detected, the organisers decided not to conduct the transport stability test in the current PT.

1.10 Organisational Aspects

1.10.1 Laboratory Status as Mandatory to Participation

Based on available information on NRL-status and commodity scope as recorded in the EURL-DataPool, the EU and EFTA OfLs and NRLs are designated as "mandatory to participate in the current PT" or "participation on voluntary basis". The available information on the pesticide scope covered by the laboratories was not considered due to concerns that might not be up-to-date and/or not applicable to the present commodity (soybean). The OfLs can provide the organisers and their NRLs their reasons and ask for changing their status. The NRLs were reminded of their responsibility for their network and to ensure that all obliged OfLs within their network were informed of this EUPT. All NRLs and OfLs were informed that the status of obligation was tentative and 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-SRM13 were instructed to provide explanations for their non-participation.

1.10.2 Announcement / Invitation and EUPT-SRM13-Website

Within the EURL-Web-Portal an EUPT-SRM13-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-SRM13 was published on the EUPT-SRM13-Website in January 2018 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. Furthermore, in order to obtain sufficient data for statistical evaluation of ***phosphine***, a few private laboratories were exceptionally invited to participate in the current PT. These private laboratories were allowed to submitted also results for other analytes. However, their results for other analytes were neither be included in the establischmen of the assigned values nor present in the final report.

1.10.3 Registration and Confidentiality

Like in the previous PT in 2017 (EUPT-SRM12) the participants were able to register for this EUPT via a website connected to the EURL-DataPool. All laboratories 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. Three days before sample shipment, participating laboratories were provided via e-mail with a unique laboratory code as well as the 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**).

1.10.4 Distribution of the Test Items and the Blank Material

One bottle of test item (approx. 200 g), one bottle of blank material (approx. 200 g) were shipped on 23 April, 2018 to each participant in thermo-insulated polystyrene boxes with cooling pads. All the parcels hve been precooled at -20 °C for three days before shipment.

Two weeks 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**).

1.10.5 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 (30 May 2018). 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.

1.10.6 Actions following Results Submission and Distribution Preliminary Report

Where information on analytical methods or results was inconsistent, laboratories were contacted. Laboratories that had originally registered to participate in the current PT but finally did not submit any results, were asked to provide explanations. On 19 June, 2018, the preliminary report on the EUPT-SRM13 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 $|z\text{-score}| > 2$ 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, the proofreading version of this final report (part one: results evaluation) is accompanied by a survey on EUPT-SRM13. This survey contains 5 questions on the organisation (general, registration, information and instruction provided, shipment/delivery, test item, blank material and results submission pages), on the relevance of the used matrix (soybean flour) 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. The survey evaluation and compilation of comments will be published soon.

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:

$\lvert z\rvert \leq 2$	acceptable
$2 < \lvert z\rvert < 3$	questionable
$\lvert z\rvert \geq 3$	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

121 laboratories from 37 countries (28 EU-Member States, 2 EFTA- countries, 1 EU-candidate country and 6 third countries) originally registered for participation in the EUPT-SRM13. 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.

Out of the 109 EU OfLs having registered for participation in the current PT five laboratories from Three co-nutries, among them the NRL-SRM in Italy, failed to submit any results and reported after the PT "technical problems/problems with instruments" or "target pesticides out of routien analytical scope" as the reason for no results submitted.

All 12 laboratories from non-EU countries submitted results (4 from EFTA countries, 2 from one EU-candidate country and 6 from third countries). For the first time one OfL from Iceland has participated in an EUPT-SRM. The results submitted by the laboratories located in Serbia (EU candidate country) and by the 6 laboratories located in third countries were not taken into account when calculating the assigned values.

In total, 151 EU-OfLs, including NRL-SRMs, regardless of their commodity scope, as well as all EU-OfLs analysing for pesticide residues in food and feed with high oil content and very low water content, were originally considered as being obliged to participate in the present EUPT. These laboratories 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.

30 obliged laboratories explained their non-participation with the fact that the matrix (soybeans) or the SRM13 target pesticides or both were out of their routine scope, partly due to a lack of required instruments. One obliged laboratory was not able to participate in because of the relocation of this laboratory. Excluding those 31 laboratories that provided sufficient explanations, the number of EU-laboratories considered as being obliged decreased to 120. Out of the 92 obliged laboratories that have registered for this PT 88 laboratories finally submitted result. In addition, 17 OfLs registered for participation on voluntary basis, and 16 of them submitted results. Out of the 120 obliged OfLs 28 (23 %) did neither register for the PT nor provide any explanation for non-participation. These laboratories originated from 8 countries as follows: HR (1×), FR (2×), DE (3×), IT (8×), PL (4×), RO (4×), ES (5×), and UK (1×).

In order to have sufficient results for evaluation of phosphine, eleven private laboratories worldwide were exceptionally invided to participate in the present PT. These private laboratories were also allowed to report results for any other pesticides in the target pesticides list if they wish. Participants from private laboratories will receive the certificate and the final report, however, their results, except those for phosphine, were not considered in the establishment of assigned values and their participation will not be shown in the final report.

Table 3-1: Number of laboratories listed as being obliged to participate in the EUPT-SRM13, 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
		During Registration		All	NRL-SRMs	All	NRL-SRMs		
AL/ BE/ NL	1		1	1		1			
AT	1		1	1	1	1	1		
BE	6	1	5	5 + [1]	1	5 + [1]	1		
BE/ BG/ FR/ LU	1		1	1		1			
BG	2	1	1	1	1	1	1		
CY	2	1	1	1	1	1	1		HR has not yet established an NRL-SRM.
CZ	3		3	3	1	3	1		
DE	25	5	20	17 + [3]	1	16 + [3]	1	3	
DE/ MT	1		1	1		1			
DK	2	1	1	1	1	1	1		
EE	3	1	2	2	1	2	1		
FI	2		2	2	2	2	2		FI has appointed two NRL-SRMs.
FR	7		7	5 + [3]	1	5 + [3]	1	2	
GR	2		2	2 + [1]	2	2 + [1]	2		
HR	5	1	4	3		3		1	GR has appointed two NRL-SRMs.
HU	4		4	4 + [1]	1	4 + [1]	1		
IE	1		1	1	1	1	1		
IT	23	5	18	10	1	9	0	8	
IT/ MT	1		1	1		1			
LT	1		1	1 + [1]	1	1 + [1]	1		
LU	1		1	1	1	1	1		*MT-NRL-SRM represented by proxy by the UK-NRL-SRM; MT subcontracted routine analysis to an OfLs in DE and IT
LV	1		1	1	1	1	1		
MT	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		2	2	1	2	1		
PL	11	5	6	2 + [1]	1	2 + [1]	1	4	
PO	1		1	1 + [1]		1 + [1]			
PT	1		1	1	1	1	1		
RO	7	2	5	1		1		4	
SE	2		2	2	1	2	1		
SI	3	1	2	2	1	2	1		
SK	1		1	1	1	1	1		
ES	24	7	17	12 + [4]	2	10 + [3]	2	5	ES has appointed two NRL-SRMs
ES/ MT	1		1	1		1			
UK/MT	1		1	1	1	1	1		UK-NRL-SRM represents also MT
UK	2		2	1 + [1]		1 + [1]		1	
EU-total	**151**	**31**	**120**	**92 + [17]**	**28**	**88 + [16]**	**27**	**28**	

Table 3-1 (cont.): Number of laboratories listed as being obliged to participate in the EUPT-SRM13, 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		
CH					[3]	–	[3]	–		
EU+EFTA Total					92 + [21]	28	88 + [20]	27		
Third Countries / EU candidate country										
BR					1	–	1	–		
BY					1	–	1	–		
CR					1	–	1	–		
PE					1	–	1	–		
RS					2	–	2	–		
SG					1	–	1	–		
TH					1	–	1	–		
Third Countries / EU candidate country Total					8		8			
Overall Sum				120	121	28	116	27		

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. 20) 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. 36)** and **Table 4-9 (p. 42)** for compulsory and optional, respectively. Detailed information about the analytical methods used by the laboratories is shown on the web under "EUPT-SRM13 - Supplementary Information" accessible via the the link: http://www.eurl-pesticides.eu/library/docs/srm/EUPT-SRM13_Supplementary_Information.pdf.

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* = 109 [3])	No. [2]	% (based on *n* = 120 [4])
Compulsory Compounds	**2,4-D**	No	88	81 %	73	61 %
	Bromide ion	**Yes**	62	57 %	47	39 %
	Chlormequat-Cl	No	88	81 %	72	60 %
	Cyromazine	**Yes**	79	72 %	63	53 %
	Ethephon	No	75	69 %	60	50 %
	Fluazifop	**Yes**	86	79 %	70	58 %
	Glyphosate	**Yes**	83	76 %	68	57 %
	Haloxyfop	**Yes**	81	74 %	67	56 %
	Mepiquat-Cl	**Yes**	86	79 %	71	59 %
Optional Compounds	**2,4-DB**	**Yes**	52	48 %	43	36 %
	Bentazone	No	73	67 %	60	50 %
	Carbofuran	No	77	71 %	62	52 %
	Chlorate	No	56	51 %	42	35 %
	Diquat	**Yes**	30	28 %	21	18 %
	Fenoxaprop	No	41	38 %	33	28 %
	Glufosinate	**Yes**	44	40 %	33	28 %
	MPP	**Yes**	25	23 %	17	14 %
	N-Acetyl-Glufosinate	No	23	21 %	17	14 %
	AMPA	No	56	51 %	43	36 %
	N-Acetyl-Glyphosate	**Yes**	23	21 %	18	15 %
	Paraquat	No	29	27 %	21	18 %
	Perchlorate	**Yes**	55	50 %	41	34 %
	Phosphonic acid	**Yes**	45	41 %	32	27 %
	Quizalofop	**Yes**	62	57 %	50	42 %
	Phsophine (Test Item)	**Yes**	11	10 %	5	4 %
	Phosphine (PH3-Tube)	**Yes**	11	10 %	5	4 %

1) Including official laboratories participating on voluntary basis
2) Laboratories representing more than one country were counted only once.
3) 109 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) 120 is the number of OfLs (including NRLs) from EU countries, which were finally considered as obliged to participate in the EUPT-SRM13 (taking into account any explanations for non-participation).

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

			Compulsory Compounds										Optional Compounds			
Compulsory Compound listed in Target List			2,4-D	Bromide ion	Chlormequat-Cl	Cyromazine	Ethephon	Fluazifop	Glyphosate	Haloxyfop	Mepiquat-Cl	Analysed / correctly found among COMPULSORY compounds (max. 13 / 8)	2,4-DB	Bentazone	Carbofuran	Chlorate
within MACP [1]			Reg.	Reg.	Reg.	Reg.	Reg.	Reg.	Reg.	Reg.	Reg.		WD	–	Reg.	WD
present in Test Item			No	**Yes**	No	**Yes**	No	**Yes**	**Yes**	**Yes**	**Yes**		**Yes**	No	No	No
evaluated in this PT			No	**Yes**	No	**Yes**	No	**Yes**	**Yes**	**Yes**	**Yes**		**Yes**	No	No	No
Lab-Code SRM13-	NRL-SRM	Cat. [2]														
1		A	ND	V	ND		ND	V	V	V	V	8 / 5	V	ND	ND	
2		B	ND	V	ND	**FN**	ND	V	V	V	**FN**	9 / 4	V	ND	ND	ND
3		B	ND			V		V		V		4 / 3	V	ND	ND	
4	x	A	ND	V	ND	V	ND	V	V	V	V	9 / 6			ND	ND
5	x	B	ND		ND		ND	V	V	V	V	7 / 4		ND	ND	ND
6		A	ND	V	ND	V	ND	V	V	V	V	9 / 6		ND	ND	ND
7	x	B	ND			V		V		V		4 / 3	V	ND	ND	
8	x	A	ND	V	ND	V	ND	V	V	V	V	9 / 6	V	ND	ND	ND
10		A	ND	V	ND	V	ND	V	V	V	V	9 / 6		ND	ND	ND
11		A	ND	V	ND	V	ND	V	V	V	V	9 / 6	V	ND		ND
12	x	A	ND	V	ND	V	ND	V	V	V	V	9 / 6	V	ND	ND	
13	x	A	ND	V	ND	V	ND	V	V	V	V	9 / 6	V	ND		ND
14		A	ND	V	ND	V	ND	V	V	V	V	9 / 6	V	ND	ND	ND
15		A	ND	V	ND	V	ND	V	V	V	V	9 / 6		ND	ND	ND
16		A	ND	V	ND	V	ND	V	V	V	V	9 / 6	V	ND	ND	ND
17		B	ND		ND	V	ND	V	V	V		7 / 4			ND	ND
19	x	A	ND	V	ND	V	ND	V	V	V	V	9 / 6	V	ND	ND	
20	x	A	ND	V	ND	V	ND	V	V	V	V	9 / 6	V		ND	ND
21		A	ND	V	ND		ND	V	V	V	V	8 / 5		ND	ND	
22	x	A	ND	V	ND	V	ND	V	V	V	V	9 / 6	V	ND	ND	
23		B	ND		ND			V	V	V	V	6 / 4	V	ND		
24	x	A	ND		ND	V	ND	V	V	V	V	8 / 5	V	ND	ND	
25		B	ND	V	ND	V		V		V	V	7 / 5		ND	ND	
26		A	ND	V	ND	V	ND	V	V	V	V	9 / 6	V	ND	ND	ND
27		A	ND	V	ND	V	ND	V	V	V	V	9 / 6		ND	ND	ND
28		B	ND					V				2 / 1				
29	x	A	ND	V	ND	V	ND	V	V	V	V	9 / 6		ND	ND	

1) MACP = EU Multiannual Control Program; Reg.: MACP Regulation; WD: NCP Working Document SANCO/12745/2013, 21 –22 November 2017 rev. 9(1) Cont.: Contaminant

2) Category A/B classification (Cat A was assigned to laboratories that have analysed at least 8 out of the 9 compulsory compounds on the Target Pesticides List, correctly detected 5 or more out of the 6 compulsory compounds present in the test item and have not reported any false positive result, see **Section 4.4.4, p. 48**)

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)

			Optional Compounds													Total
Optional / Additional Compound listed in Target List			Diquat	Fenoxaprop	Glufosinate	MPP	N-Acetyl-Glufosinate	AMPA	N-Acetyl-Glyphosate	Paraquat	Perchlorate	Phosphonic acid	Quizalofop	Phsophine (Test Item)	Phosphine (PH3-Tube)	Analysed / correctly found among OPTIONAL compounds (max. 21 / 15)	Analysed / correctly found among COMPULSORY and OPTIONAL compounds (max. 21 / 15)
within MACP [1]			WD	–	WD	WD	WD	WD	WD	WD	Cont.	WD	WD	WD	WD		
present in Test Item			Yes	No	Yes	Yes	No	No	Yes	No	Yes	Yes	Yes	Yes	Yes		
evaluated in this PT			Yes	No	Yes	Yes	No	No	Yes	No	Yes	Yes	Yes	No	No		
Lab-Code SRM13-	NRL-SRM	Cat. [2]															
1		A	V	ND				ND		ND						7 / 2	15 / 7
2		B	V	ND	V			ND		ND	**FN**	V	V			12 / 5	21 / 9
3		B		ND												4 / 1	8 / 4
4	x	A			V		ND	ND	V		V	V	V			9 / 5	18 / 11
5	x	B									V					4 / 1	11 / 5
6		A									V	V	V			6 / 3	15 / 9
7	x	B		ND												4 / 1	8 / 4
8	x	A							V		V					6 / 3	15 / 9
10		A		ND							V	V	V			7 / 3	16 / 9
11		A		ND				ND			V	**FN**	**FN**	V	V	10 / 4	19 / 10
12	x	A		ND				ND	V							6 / 2	15 / 8
13	x	A	V	ND	V	V	ND	ND	V		V	V	V			13 / 8	22 / 14
14		A	V	ND	V			ND		ND	V	V	V	V	V	14 / 8	23 / 14
15		A			V	V	ND	ND	V		V	V	V			11 / 6	20 / 12
16		A		ND	V			ND			V	V	V	V	V	12 / 7	21 / 13
17		B			V			ND			V	V	V	V	V	9 / 6	16 / 10
19	x	A		ND												4 / 1	13 / 7
20	x	A									V					4 / 2	13 / 8
21		A	V	ND				ND	V	ND						7 / 2	15 / 7
22	x	A			V			ND								5 / 2	14 / 8
23		B		ND				ND								4 / 1	10 / 5
24	x	A														3 / 1	11 / 6
25		B														2 / 0	9 / 5
26		A			V						V	V	V			8 / 5	17 / 11
27		A	V	ND	V	V	ND	ND		ND	V	V	V			13 / 6	22 / 12
28		B														0 / 0	2 / 1
29	x	A														2 / 0	11 / 6

1) MACP = EU Multiannual Control Program; Reg.: MACP Regulation; WD: NCP Working Document SANCO/12745/2013, 21 –22 November 2017 rev. 9(1) Cont.: Contaminant

2) Category A/B classification (Cat A was assigned to laboratories that have analysed at least 8 out of the 9 compulsory compounds on the Target Pesticides List, correctly detected 5 or more out of the 6 compulsory compounds present in the test item and have not reported any false positive result, see **Section 4.4.4, p. 48**)

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										Optional Compounds			
Compulsory Compound listed in Target List			2,4-D	Bromide ion	Chlormequat-Cl	Cyromazine	Ethephon	Fluazifop	Glyphosate	Haloxyfop	Mepiquat-Cl	Analysed / correctly found among COMPULSORY compounds (max. 13 / 8)	2,4-DB	Bentazone	Carbofuran	Chlorate
within MACP [1]			Reg.	Reg.	Reg.	Reg.	Reg.	Reg.	Reg.	Reg.	Reg.		WD	–	Reg.	WD
present in Test Item			No	Yes	No	Yes	No	Yes	Yes	Yes	Yes		Yes	No	No	No
evaluated in this PT			No	Yes	No	Yes	No	Yes	Yes	Yes	Yes		Yes	No	No	No
Lab-Code SRM13-	NRL-SRM	Cat. [2]														
30	x	A	ND	V	ND	V	ND	V	V	V	V	9 / 6	V	ND	ND	ND
31		A	ND	V	ND	V	ND	V	V	V	V	9 / 6		ND	ND	ND
32	x	A	ND		ND	V	ND	V	V	V	V	8 / 5	V	ND	ND	ND
33		A	ND	V	ND	V	ND	V	V	V	V	9 / 6	V	ND	ND	ND
34		A	ND	V	ND	V	ND	V	V	V	V	9 / 6	V	ND	ND	ND
35		A	ND		ND	V	ND	V	V	V	V	8 / 5	V	ND	ND	ND
36	x	A	ND		ND	V	ND	V	V	V	V	8 / 5	V	ND	ND	ND
37		A	ND	V	ND	V	ND	V	V	V	V	9 / 6				ND
38		B	ND		ND		ND	V			V	5 / 2		ND		
39		B							V			1 / 1				
40		B							V			1 / 1				
41		B	ND				FP	V	V	V		5 / 3	V			
42		B	ND		ND	V		V		V	V	6 / 4		ND		
43		B							V			1 / 1				
44		B	ND						V			2 / 1		ND	ND	ND
45		B	ND	V	ND	V	ND	V	V	V	V	9 / 6	V	ND	ND	ND
46		A	ND	V	ND	V	ND	V	V	V	V	9 / 6	V	ND	ND	ND
47		B										0 / 0			ND	
48		A	ND	V	ND	V	ND	V	V	V	V	9 / 6		ND	ND	ND
49	x	A	ND	V	ND	V	ND	V	V	V	V	9 / 6	V	ND		
50		A	ND	V	ND	V	ND	V	V	V	V	9 / 6	V	ND	ND	ND
51	x	B	ND	V	ND	V	ND		V		V	7 / 4				
52		A	ND	V	ND	V	ND	V	V	V	V	9 / 6	V	ND	ND	ND
53		B	ND		ND	V	ND	V			V	6 / 3			ND	
54	x	A	ND	V	ND	V	ND	V	V	V	V	9 / 6		ND	ND	
55		A	ND	V	ND	V	ND	V	V	V	V	9 / 6	FN	ND	ND	
56		A	ND	V	ND	V	ND	V	V	V	V	9 / 6		ND	ND	ND

1) MACP = EU Multiannual Control Program; Reg.: MACP Regulation; WD: NCP Working Document SANCO/12745/2013, 21 –22 November 2017 rev. 9(1) Cont.: Contaminant

2) Category A/B classification (Cat A was assigned to laboratories that have analysed at least 8 out of the 9 compulsory compounds on the Target Pesticides List, correctly detected 5 or more out of the 6 compulsory compounds present in the test item and have not reported any false positive result, see **Section 4.4.4, p. 48**)

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)

			Optional Compounds													Total
Optional / Additional Compound listed in Target List			Diquat	Fenoxaprop	Glufosinate	MPP	N-Acetyl-Glufosinate	AMPA	N-Acetyl-Glyphosate	Paraquat	Perchlorate	Phosphonic acid	Quizalofop	Phsophine (Test Item)	Phosphine (PH3-Tube)	Analysed / correctly found among OPTIONAL compounds (max. 21 / 15)	Analysed / correctly found among COMPULSORY and OPTIONAL compounds (max. 21 / 15)
within MACP [1]			WD	–	WD	WD	WD	WD	WD	WD	Cont.	WD	WD	WD	WD		
present in Test Item			Yes	No	Yes	Yes	No	No	Yes	No	Yes	Yes	Yes	Yes	Yes		
evaluated in this PT			Yes	No	Yes	Yes	No	No	Yes	No	Yes	Yes	Yes	No	No		
Lab-Code SRM13-	NRL-SRM	Cat. [2]															
30	x	A	V	ND	V	V	ND	ND	V	ND	V	V	V			15 / 8	24 / 14
31		A	V	ND				ND		ND	V	V	V			10 / 4	19 / 10
32	x	A	V	ND				ND		ND	V	V	V	V	V	13 / 7	21 / 12
33		A		ND	V			ND			V	V	V			10 / 5	19 / 11
34		A	V	ND	V	V	ND	ND	V	ND	V	V	V	V	V	17 / 10	26 / 16
35		A	V		V	V	ND	ND			V	V	V			12 / 7	20 / 12
36	x	A	V		V	V		ND	V	ND	V	V	V			13 / 8	21 / 13
37		A			V	V	ND	ND			V	V	V			8 / 5	17 / 11
38		B		ND							V					3 / 1	8 / 3
39		B														0 / 0	1 / 1
40		B						ND								1 / 0	2 / 1
41		B			V	V	ND	**FP**	V			V	V			8 / 6	13 / 9
42		B		ND												2 / 0	8 / 4
43		B						ND								1 / 0	2 / 1
44		B			V			ND			V					6 / 2	8 / 3
45		B	V	ND	V	V	**FP**	ND	**FN**	ND	V	V	V			15 / 7	24 / 13
46		A	V	ND	V	V	ND	ND	V	ND	V	V	V			15 / 8	24 / 14
47		B														1 / 0	1 / 0
48		A		ND				ND			V	V	V			8 / 3	17 / 9
49	x	A														2 / 1	11 / 7
50		A		ND							V	V	V			8 / 4	17 / 10
51	x	B														0 / 0	7 / 4
52		A	V		V	V	ND	ND		ND	V	V	V			13 / 7	22 / 13
53		B														1 / 0	7 / 3
54	x	A		ND				ND								4 / 0	13 / 6
55		A			**FN**			ND	**FN**			V	V			8 / 2	17 / 8
56		A									V					4 / 1	13 / 7

1) MACP = EU Multiannual Control Program; Reg.: MACP Regulation; WD: NCP Working Document SANCO/12745/2013, 21 –22 November 2017 rev. 9(1) Cont.: Contaminant

2) Category A/B classification (Cat A was assigned to laboratories that have analysed at least 8 out of the 9 compulsory compounds on the Target Pesticides List, correctly detected 5 or more out of the 6 compulsory compounds present in the test item and have not reported any false positive result, see **Section 4.4.4, p. 48**)

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										Optional Compounds			
Compulsory Compound listed in Target List			2,4-D	Bromide ion	Chlormequat-Cl	Cyromazine	Ethephon	Fluazifop	Glyphosate	Haloxyfop	Mepiquat-Cl	Analysed / correctly found among COMPULSORY compounds (max. 13 / 8)	2,4-DB	Bentazone	Carbofuran	Chlorate
within MACP [1]			Reg.	Reg.	Reg.	Reg.	Reg.	Reg.	Reg.	Reg.	Reg.		WD	–	Reg.	WD
present in Test Item			No	Yes	No	Yes	No	Yes	Yes	Yes	Yes		Yes	No	No	No
evaluated in this PT			No	Yes	No	Yes	No	Yes	Yes	Yes	Yes		Yes	No	No	No
Lab-Code SRM13-	NRL-SRM	Cat. [2]														
57	x	B			ND	V					V	3 / 2			ND	ND
58		B							V			1 / 1				
59		B										0 / 0				
60		A	ND		ND	V	ND	V	V	V	V	8 / 5	V	ND		ND
61	x	A	ND		ND	V	ND	V	V	V	V	8 / 5		ND	ND	
62		B	ND	V	ND	V	ND	V		V		7 / 4			ND	ND
63		A	ND	V	ND	V	ND	V	V	V	V	9 / 6	V	ND	ND	ND
64		B			ND				V			2 / 1			ND	
65		A	ND	**FN**	ND	V		V	V	V	V	8 / 5		ND	ND	
66		B	ND		ND			V		V	**FN**	5 / 2		ND	ND	
67		B	ND	V	ND	V		V	V		V	7 / 5		ND	ND	ND
68	x	A	ND	V	ND	V	ND	V		V	V	8 / 5		ND	ND	
69		A	ND	V	ND	V	ND	V	V	V	V	9 / 6	V	ND	ND	ND
70		B			ND				V		V	3 / 2				
71		A	ND		ND	V	ND	V	V	V	V	8 / 5	V	ND	ND	ND
72		A	ND	V	ND	V	ND	V	V	V	V	9 / 6	V	ND	ND	ND
73		A	ND		ND	V	ND	V	V	V	V	8 / 5		ND	ND	
74	x	A	ND	V	ND	V	ND	V	V	V	V	9 / 6	V	ND	ND	
76	x	A	ND		ND	V	ND	V	V	V	V	8 / 5	V		ND	
77	x	B			ND	V					V	3 / 2			ND	
78	x	A	ND	V	ND	V	ND	V	V	V	V	9 / 6				
79	x	A	ND	V	ND	V	ND	V	V	V	V	9 / 6	V	ND	ND	ND
80	x	B	ND		ND			V	V	V	V	6 / 4			ND	
82		B	ND	V		V						3 / 2		ND	ND	
83		A	ND	V	ND	V	ND	V	V	V	V	9 / 6	V	ND	ND	ND
84		B	ND			V		V		V		4 / 3		ND		
85		A	ND	V	ND	V	ND	V		V	V	8 / 5	V	ND	ND	ND

1) MACP = EU Multiannual Control Program; Reg.: MACP Regulation; WD: NCP Working Document SANCO/12745/2013, 21 –22 November 2017 rev. 9(1) Cont.: Contaminant

2) Category A/B classification (Cat A was assigned to laboratories that have analysed at least 8 out of the 9 compulsory compounds on the Target Pesticides List, correctly detected 5 or more out of the 6 compulsory compounds present in the test item and have not reported any false positive result, see **Section 4.4.4, p. 48**)

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)

			Optional Compounds													Total
Optional / Additional Compound listed in Target List			Diquat	Fenoxaprop	Glufosinate	MPP	N-Acetyl-Glufosinate	AMPA	N-Acetyl-Glyphosate	Paraquat	Perchlorate	Phosphonic acid	Quizalofop	Phsophine (Test Item)	Phosphine (PH3-Tube)	Analysed / correctly found among OPTIONAL compounds (max. 21 / 15)	Analysed / correctly found among COMPULSORY and OPTIONAL compounds (max. 21 / 15)
within MACP [1]			WD	–	WD	WD	WD	WD	WD	WD	Cont.	WD	WD	WD	WD		
present in Test Item			Yes	No	Yes	Yes	No	No	Yes	No	Yes	Yes	Yes	Yes	Yes		
evaluated in this PT			Yes	No	Yes	Yes	No	No	Yes	No	Yes	Yes	Yes	No	No		
Lab-Code SRM13-	NRL-SRM	Cat. [2]															
57	x	B									V					3 / 1	6 / 3
58		B						ND								1 / 0	2 / 1
59		B								ND						1 / 0	1 / 0
60		A		ND	V	V	ND	ND			V					9 / 4	17 / 9
61	x	A														2 / 0	10 / 5
62		B	V		V			ND		ND	V	V	V			9 / 5	16 / 9
63		A	V	ND	V	V	ND	ND	V	ND	V	V	V			15 / 8	24 / 14
64		B						ND								2 / 0	4 / 1
65		A														2 / 0	10 / 5
66		B														2 / 0	7 / 2
67		B			V	V		ND			V	V	V			9 / 5	16 / 10
68	x	A														2 / 0	10 / 5
69		A		ND							V	V	V			8 / 4	17 / 10
70		B														0 / 0	3 / 2
71		A	V		V		ND	ND	V	ND	V					11 / 5	19 / 10
72		A		ND							V	V	V			8 / 4	17 / 10
73		A			V			ND								4 / 1	12 / 6
74	x	A														3 / 1	12 / 7
76	x	A	V	ND	V		ND	ND	**FN**	ND						9 / 3	17 / 8
77	x	B														1 / 0	4 / 2
78	x	A			V											1 / 1	10 / 7
79	x	A			V									V	V	7 / 4	16 / 10
80	x	B						ND								2 / 0	8 / 4
82		B														2 / 0	5 / 2
83		A	V	ND	V	V	ND	ND	V	ND	V	V	V			15 / 8	24 / 14
84		B														1 / 0	5 / 3
85		A									V	**FN**	**FN**			7 / 2	15 / 7

1) MACP = EU Multiannual Control Program; Reg.: MACP Regulation; WD: NCP Working Document SANCO/12745/2013, 21 –22 November 2017 rev. 9(1) Cont.: Contaminant

2) Category A/B classification (Cat A was assigned to laboratories that have analysed at least 8 out of the 9 compulsory compounds on the Target Pesticides List, correctly detected 5 or more out of the 6 compulsory compounds present in the test item and have not reported any false positive result, see **Section 4.4.4, p. 48**)

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										Optional Compounds			
Compulsory Compound listed in Target List			2,4-D	Bromide ion	Chlormequat-Cl	Cyromazine	Ethephon	Fluazifop	Glyphosate	Haloxyfop	Mepiquat-Cl	Analysed / correctly found among COMPULSORY compounds (max. 13 / 8)	2,4-DB	Bentazone	Carbofuran	Chlorate
within MACP [1]			Reg.	Reg.	Reg.	Reg.	Reg.	Reg.	Reg.	Reg.	Reg.		WD	–	Reg.	WD
present in Test Item			No	Yes	No	Yes	No	Yes	Yes	Yes	Yes		Yes	No	No	No
evaluated in this PT			No	Yes	No	Yes	No	Yes	Yes	Yes	Yes		Yes	No	No	No
Lab-Code SRM13-	NRL-SRM	Cat. [2]														
86		A	ND		ND	V	ND	V	V	V	V	8 / 5	V	ND	ND	ND
87		A	ND		ND	V	ND	V	V	V	V	8 / 5	V	ND	ND	ND
88		A	ND	V	ND	V	ND	V	V	V	V	9 / 6	V	ND	ND	ND
89		A	ND	V	ND	V	ND	V	V	V	V	9 / 6	V	ND	ND	ND
90		B						FN				1 / 0			ND	
91		A	ND	V	ND	V	ND	V	V	V	V	9 / 6	V	ND	ND	ND
92		A	ND	V	ND	V	ND	V	V	V	V	9 / 6				ND
93		A	ND	V	ND	V	ND	V	V	V	V	9 / 6	V	ND	ND	ND
94		B		V								1 / 1				
95		B									V	1 / 1				
96		B	ND		ND	V		V	V	V	V	7 / 5		ND		
97		A	ND	V	ND	V	ND	V	V	V	V	9 / 6	V	ND	ND	ND
98		A	ND	V	ND	V	ND	V	V	V	V	9 / 6	V	ND	ND	ND
99		B			ND						V	2 / 1			ND	
100	x	B	ND	V				V		V		4 / 3				
101		A	ND		ND	V	ND	V	V	V	V	8 / 5	V	ND	ND	ND
102		B		V								1 / 1				
103		B							V			1 / 1				
104		A	ND	V	ND	V	ND	V	V	V	V	9 / 6				
105		A	ND	V	ND	V	ND	V	V	V	V	9 / 6	V	ND	ND	ND
106		B			ND	V	ND		V		V	5 / 3				
107		B	ND		ND			V	V	V	V	6 / 4	V	ND	ND	
109		A	ND		ND	V	ND	V	V	V	V	8 / 5	FN	ND	ND	ND
110		B			ND		ND		V		V	4 / 2				ND
111		B		V	ND		ND				V	4 / 2				ND

1) MACP = EU Multiannual Control Program; Reg.: MACP Regulation; WD: NCP Working Document SANCO/12745/2013, 21 –22 November 2017 rev. 9(1) Cont.: Contaminant

2) Category A/B classification (Cat A was assigned to laboratories that have analysed at least 8 out of the 9 compulsory compounds on the Target Pesticides List, correctly detected 5 or more out of the 6 compulsory compounds present in the test item and have not reported any false positive result, see **Section 4.4.4, p. 48**)

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)

			Optional Compounds													Total
Optional / Additional Compound listed in Target List			Diquat	Fenoxaprop	Glufosinate	MPP	N-Acetyl-Glufosinate	AMPA	N-Acetyl-Glyphosate	Paraquat	Perchlorate	Phosphonic acid	Quizalofop	Phsophine (Test Item)	Phosphine (PH3-Tube)	Analysed / correctly found among OPTIONAL compounds (max. 21 / 15)	Analysed / correctly found among COMPULSORY and OPTIONAL compounds (max. 21 / 15)
within MACP [1]			WD	–	WD	WD	WD	WD	WD	WD	Cont.	WD	WD	WD	WD		
present in Test Item			Yes	No	Yes	Yes	No	No	Yes	No	Yes	Yes	Yes	Yes	Yes		
evaluated in this PT			Yes	No	Yes	Yes	No	No	Yes	No	Yes	Yes	Yes	No	No		
Lab-Code SRM13-	NRL-SRM	Cat. [2]															
86		A	V		V			ND	V	ND		V	V			11 / 6	19 / 11
87		A									V					5 / 2	13 / 7
88		A		ND	V	V					V	V	V			10 / 6	19 / 12
89		A	V	ND	**FN**	V	ND	ND	**FN**	ND	V	V	V			15 / 6	24 / 12
90		B														1 / 0	2 / 0
91		A		ND		V		ND	V		V	V	V			11 / 6	20 / 12
92		A	V		V	V	ND			ND	V					7 / 4	16 / 10
93		A	V	ND	V			ND		ND	V	V	V			12 / 6	21 / 12
94		B														0 / 0	1 / 1
95		B														0 / 0	1 / 1
96		B														1 / 0	8 / 5
97		A	V	ND	V	V		ND		ND	V	**FN**	**FN**			13 / 5	22 / 11
98		A		ND	V			ND			V	V	V	V	V	12 / 7	21 / 13
99		B														1 / 0	3 / 1
100	x	B														0 / 0	4 / 3
101		A	V	ND	V	V	ND	ND		ND	V	V	V			14 / 7	22 / 12
102		B														0 / 0	1 / 1
103		B														0 / 0	1 / 1
104		A														0 / 0	9 / 6
105		A		ND	**FN**	V	ND	ND	V		V	V	V			13 / 6	22 / 12
106		B						ND				V	V			3 / 2	8 / 5
107		B	V		V			ND		ND						7 / 3	13 / 7
109		A	**FN**		V	**FN**	ND	ND	**FN**	ND	**FN**					12 / 1	20 / 6
110		B	V					ND		ND	V					5 / 2	9 / 4
111		B									V					2 / 1	6 / 3

1) MACP = EU Multiannual Control Program; Reg.: MACP Regulation; WD: NCP Working Document SANCO/12745/2013, 21 –22 November 2017 rev. 9(1) Cont.: Contaminant

2) Category A/B classification (Cat A was assigned to laboratories that have analysed at least 8 out of the 9 compulsory compounds on the Target Pesticides List, correctly detected 5 or more out of the 6 compulsory compounds present in the test item and have not reported any false positive result, see **Section 4.4.4, p. 48**)

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												Optional Compounds				
Compulsory Compound listed in Target List			**2,4-D**	**Bromide ion**	**Chlormequat-Cl**	**Cyromazine**	**Ethephon**	**Fluazifop**	**Glyphosate**	**Haloxyfop**	**Mepiquat-Cl**	**Analysed / correctly found among COMPULSORY compounds (max. 13 / 8)**	**2,4-DB**	**Bentazone**	**Carbofuran**	**Chlorate**
within MACP [1]			**Reg.**	**Reg.**	**Reg.**	**Reg.**	**Reg.**	**Reg.**	**Reg.**	**Reg.**	**Reg.**		**WD**	**–**	**Reg.**	**WD**
present in Test Item			No	**Yes**	No	**Yes**	No	**Yes**	**Yes**	**Yes**	**Yes**		**Yes**	No	No	No
evaluated in this PT			No	**Yes**	No	**Yes**	No	**Yes**	**Yes**	**Yes**	**Yes**		**Yes**	No	No	No
Lab-Code SRM13-	**NRL-SRM**	**Cat.** [2]														
112		A	ND	V	ND	V	ND	V	V	V	V	9 / 6	V	ND	ND	ND
113		B										0 / 0				
114		B										0 / 0				
3rd-115		B	ND		ND	V			V	**FN**	V	6 / 3	**FN**	ND		
3rd-116		B	ND	V	ND	V	ND		V	V	V	8 / 5		ND	ND	
3rd-117		A	ND	V	ND	V	ND	V	V	V	V	9 / 6	V		ND	
3rd-118		B	ND	V		V	ND	**FN**	V			6 / 3		ND	ND	
3rd-119		B	ND					V				2 / 1	V	ND		
3rd-120		B	ND				ND		V			3 / 1			ND	
3rd-121		B					ND		V			1 / 1		ND	ND	
3rd-122		B	ND	V		V	ND		V	V	V	7 / 5				

1) MACP = EU Multiannual Control Program; Reg.: MACP Regulation; WD: NCP Working Document SANCO/12745/2013, 21 –22 November 2017 rev. 9(1) Cont.: Contaminant

2) Category A/B classification (Cat A was assigned to laboratories that have analysed at least 8 out of the 9 compulsory compounds on the Target Pesticides List, correctly detected 5 or more out of the 6 compulsory compounds present in the test item and have not reported any false positive result, see **Section 4.4.4, p. 48**)

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)

			Optional Compounds													Total
Optional / Additional Compound listed in Target List			Diquat	Fenoxaprop	Glufosinate	MPP	N-Acetyl-Glufosinate	AMPA	N-Acetyl-Glyphosate	Paraquat	Perchlorate	Phosphonic acid	Quizalofop	Phsophine (Test Item)	Phosphine (PH3-Tube)	Analysed / correctly found among OPTIONAL compounds (max. 21 / 15)	Analysed / correctly found among COMPULSORY and OPTIONAL compounds (max. 21 / 15)
within MACP [1]			WD	–	WD	WD	WD	WD	WD	WD	Cont.	WD	WD	WD	WD		
present in Test Item			Yes	No	Yes	Yes	No	No	Yes	No	Yes	Yes	Yes	Yes	Yes		
evaluated in this PT			Yes	No	Yes	Yes	No	No	Yes	No	Yes	Yes	Yes	No	No		
Lab-Code SRM13-	NRL-SRM	Cat. [2]															
112		A	V	ND	V	V	ND	ND	V	ND	V	V	V	V	V	17 / 10	26 / 16
113		B												V	V	2 / 2	2 / 2
114		B												V	V	2 / 2	2 / 2
3rd-115		B	V		V					ND						5 / 2	11 / 5
3rd-116		B	V		V											4 / 2	12 / 7
3rd-117		A			V			ND		ND						5 / 2	14 / 8
3rd-118		B	V	ND				**FP**		**FP**						6 / 1	12 / 4
3rd-119		B														2 / 1	4 / 2
3rd-120		B			V			ND								3 / 1	6 / 2
3rd-121		B			**FN***			ND								1 / 0	2 / 1
3rd-120		B			V											1 / 1	8 / 6

1) MACP = EU Multiannual Control Program; Reg.: MACP Regulation; WD: NCP Working Document SANCO/12745/2013, 21 –22 November 2017 rev. 9(1) Cont.: Contaminant

2) Category A/B classification (Cat A was assigned to laboratories that have analysed at least 8 out of the 9 compulsory compounds on the Target Pesticides List, correctly detected 5 or more out of the 6 compulsory compounds present in the test item and have not reported any false positive result, see **Section 4.4.4, p. 48**)

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.

4.2 Analysis of Blank Material

In 68 cases the laboratories reported detecting of target pesticides in the blank material under their reporting limit or MRRLs (data not shown). In further 22 cases (**Table 4-3)** the laboratories reported numerical results for the concentration of taget pesticides detected in the blank material, among them there were four cases (2× ***bromide ion***, 1× *paraquat* and 1× *AMPA*) where participants reported detections in the blank material at levels higher than both their own RLs and above the MRRL. All these four cases indicated contamination in the procedure or background value in the system. In the two cases of paraquat (MRRL = 0.02 mg/kg) at 0.497 mg/kg and *AMPA* (MRRL = 0.05 mg/kg) at 0.0923 mg/kg, both reported by SRM13-3rd-118, this laboratory has also detected these two analytes in the test item at the comparable levels although they were neither spiked to the test item nor detected by other laboratories. In the two cases of ***bromide ion*** (MRRL = 2 mg/kg) at 3.7 mg/kg (reported by SRM13-31) and 10.4 mg/kg (reported by SRM13-97) the participants would have achieved much better z-scores if the reported results would have been substrated from the concentration in the blank material. The affected laboratories are encouraged to find the reasons behind these contamination / background value.

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. Except the calculation of ***phosphine*** the results reported by private laboratories were also not taken into account. Based on these assigned values, z-scores were calculated for all submitted results using the FFP-approach

Table 4-3: Numerical values of analyte concentration in the blank material reported by the participating laboratories

Compound	MRRL [mg/kg]	Assigned Value [mg/kg]	Conc. in Blank Material [mg/kg]	Conc. in Test Item [mg/kg]	RL [mg/kg]	Reported by
Bromide ion	2	15.4	0.14	4.29	1	SRM13-74
			0.374	9.886	0.05	SRM13-34
			0.4	14.1	2	SRM13-72
			0.543	14.4	0.3	SRM13-3rd-118
			0.66	15.9	0.2	SRM13-45
			0.702	12.9	2	SRM13-92
			0.725	14.1	2	SRM13-67
			0.783	17.8	0.2	SRM13-13
			0.8	14.6	0.5	SRM13-6
			1.2	12	1	SRM13-112
			1.5	11.6	1	SRM13-98
			3.7	**18**	2	SRM13-31
			10.4	**27.9**	5	SRM13-97
Glyphosate	0.03	0.903	0.002	0.859	0.01	SRM13-98
AMPA	0.05	not present	**0.0923**	**0.0906 (= FP)**	0.05	SRM13-3rd-118
Paraquat	0.02	not present	**0.497**	**0.495 (= FP)**	0.05	SRM13-3rd-118
Perchlorate	0.01	0.100	0.0035	0.0825	0.005	SRM13-67
			0.004	0.102	0.01	SRM13-98
Phosphonic acid	0.05	1.87	0.02	1.59	0.01	SRM13-14
			0.039	2.45	0.05	SRM13-98
Phsophine (Test Item)	0.005	–	0.00001	0.21	0.0001	SRM13-113
Phosphine (PH3-Tube)	0.005	–	0.00001	0.106	0.0001	SRM13-113

(**Section 4.4.3, p. 34**), and a preliminary report was released on 19 June, 2018. The uncertainties ($u(x_{pt})$) of the assigned values were calculated as described under **Section 2.2, p. 11**.

In the case of ***diquat*** the very wide distribution of participants' results (CV^* 52.8 %) resulted in the robust mean being assosiated with a statistical uncertainty exceeding the tolerance (**Table 4-5, p. 32**). The Scientific Committee therefore decided to evaluate the robust mean and z-scores for ***diquat*** for informative purposes only.

In the case of ***phosphine*** (both in the test item and in the PH3-tube), althouth the results submitted from private laboratories routinely analysing this compound were taken into account, the total number of results (18) were low and the results distribution were extremely wide with a CV^*-value of 93.1 % for ***phosphine*** in the test item and 74.8 % for ***phosphine*** in the PH3-Tube. A reliable statistical evaluation for these two parameter was therefore not possible. A special report on phsophine with discussion about the analytical difficulties will be issued.

The CV^*-values of all other compulsory and optional analytes were lower than the FFP-RSD of 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 ***diquat***, ***phosphine (test tiem)*** and ***phosphine (PH-Tube)*** was 22.0 %. Both were clearly lower than the FFP-RSD of 25 %.

Table 4-4: 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	Bromide ion	1	61	15.3	+/-0.58988	1.1517	passed	24.0
	Cyromazine	1	78	0.097	+/-0.00323	0.0073	passed	23.5
	Fluazifop	1	85	0.049	+/-0.00136	0.0037	passed	20.3
	Glyphosate		83	0.903	+/-0.02798	0.0677	passed	22.6
	Haloxyfop		81	0.017	+/-0.00048	0.013	passed	20.4
	Mepiquat-Cl	2	84	0.124	+/-0.00387	0.0093	passed	22.9
	Average [3] CV^*							**21.2**
Optional Compounds	2,4-DB	2	50	0.183	+/-0.00669	0.0137	passed	20.7
	Diquat [4]	1	29	1.70	+/-0.20868	0.1267	**failed**	52.8 [4]
	Glufosinate	3	41	0.192	+/-0.01073	0.0144	passed	28.7
	MPP	1	24	0.188	+/-0.00908	0.0141	passed	18.9
	N-Acetyl-Glyphosate	5	18	0.835	+/-0.05185	0.0626	passed	21.1
	Perchlorate	2	53	0.100	+/-0.00289	0.0075	passed	16.9
	Phosphonic acid	3	42	1.86	+/-0.08723	0.1398	passed	24.3
	Quizalofop	2	60	0.052	+/-0.00198	0.0039	passed	23.6
	Phsophine (Test Item) [5]		11 + 7 [6]	0.092	+/-0.02517	0.0069	**failed**	93.1 [5]
	Phosphine (PH3-Tube) [5]		11 + 7 [6]	0.040	+/-0.00877	0.003	**failed**	74.8 [5]
	Average [3] CV^*							**22.0**

1: $u(x_{pt})$: Uncertainty of assigned value calculated as shown under **Section 2.2 (p. 38)**
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 as well as z-scores were calculated for informative purpose only.
5: Excluded from the calculation of the average CV^*s and from this report, will be evaluated and discussed in a separate report.
6: 7 results reported by private laboratories which were exceptionally invited to analyte phosphine

4.4 Assessment of Laboratory Performance

4.4.1 False Positives

Five results were preliminarily judged as false positives: *AMPA* (2x), *ethephon* (1x), *N-acetyl glufosinate* (1x) and *paraquat* (1x). In cases where the reported concentration of a compound not present in the Test Item was lower than the respective MRRL the results were preliminarily not judged as false positives. This concerned carbofuran, *chlorate* and *AMPA* in one case each.

Among EU- and EFTA-laboratories three laboratories reported in three cased numerical results for three analytes (*ethephon*, *N-Acetyl-Glufosinate* and *AMPA*) on the Target Pesticides List but not present in the test material. Two other false positive results (*AMLA* and *paraquat*) were reported by one laboratory from third countries. All these analytes were neither detected by the organisers nor by the overwhelming majority of the participants (**Table 4-5**). These five 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.

Three laboratories reported in three cases numerical results for *carbofuran* (0.0028 mg/kg), *chlorate* (0.0043 mg/kg), and *AMPA* (0.002 mg/kg), which were lower than the laboratories' RLs or MRRL. Following the rules in the General Protocol these two results were not judged as false positives, although they actually should not be reported.

4.4.2 False Negatives

Among the compulsory compounds there were 7 cases (2× ***fluazifop***, 2× ***Mepiquat-Cle-Cl***, 1× ***bromide ion,*** 1× ***cyromazine*** and 1× ***haloxyfop***) 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, p. 33**). Five of them were reported by laboratories from EU and EFTA countries and represented 1.0 % of the total 477 results reported by the EU/EFTA laboratories for compulsory target compounds present in the test item. The total 7 esults, including the other two reportet by laboratories from third countires, represented 1.4 % of the total 504 results from all participating laboratories. 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 ***cyromazine*** the "false negative" judgement resulted from the fact that the laboratory had a reporting limit at a similar level to the assigned value.

Table 4-5: 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
	Ethephon	SRM13-41	Yes	0.368	0.01	0.02	FP
Optional Compounds	Carbofuran	SRM13-7	Yes	0.0028	–	0.005	–
	Chlorate	SRM13-67	Yes	0.0043	0.005	0.01	–
	N-Acetyl-Glufosinate	SRM13-45	Yes	1.26	0.02	0.02	FP
	AMPA	SRM13-41	Yes	0.078	0.01	0.05	FP
		SRM13-98	Yes	0.002	0.01	0.05	–
		SRM13-3rd-118	Yes	0.0906	0.05	0.05	FP
	Paraquat	SRM13-3rd-118	Yes	0.495	0.05	0.02	FP

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	Bromide ion	SRM13-65	Yes	No	12.5	2	15.4	False Negative
	Cyromazine	SRM13-2	Yes	No	0.01	0.01	0.097	False Negative
	Fluazifop	SRM13-90	Yes	No	0.005	0.01	0.049	False Negative
		SRM13-3rd-118	Yes	No	0.01			False Negative
	Haloxyfop	SRM13-3rd-115	Yes	No	0.003	0.003	0.017	False Negative
	Mepiquat-Cl	SRM13-2	Yes	No	0.01	0.01	0.124	False Negative
		SRM13-66	Yes	No	0.01			False Negative
Optional Compounds	2,4-DB	SRM13-55	Yes	No	0.01	0.01	0.183	False Negative
		SRM13-109	Yes	No	0.01			False Negative
		SRM13-3rd-115	Yes	No	0.01			False Negative
	Diquat	SRM13-109	Yes	No	0.02	0.02	1.70	False Negative
	Glufosinate	SRM13-55	Yes	No	0.04	0.02	0.192	False Negative
		SRM13-89	Yes	No	0.02			False Negative
		SRM13-105	Yes	No	0.1			False Negative
		SRM13-3rd-121	Yes	No	**0.5**			False Negative*
	MPP	SRM13-109	Yes	No	0.02	0.02	0.188	False Negative
	N-Acetyl-Glyphosate	SRM13-45	Yes	No	0.02	0.02	0.835	False Negative
		SRM13-55	Yes	No	0.04			False Negative
		SRM13-76	Yes	No	mg/kg			False Negative
		SRM13-89	Yes	No	0.02			False Negative
		SRM13-109	Yes	No	0.02			False Negative
	Perchlorate	SRM13-2	Yes	No	0.01	0.01	0.100	False Negative
		SRM13-109	Yes	No	0.01			False Negative
	Phosphonic acid	SRM13-11	Yes	No	0.05	0.05	1.86	False Negative
		SRM13-85	Yes	No	0.1			False Negative
		SRM13-97	Yes	No	0.1			False Negative
	Quizalofop	SRM13-47	Yes	No	–	0.01	0.052	False Negative
		SRM13-66	Yes	No	0.01			False Negative
		SRM13-3rd-115	Yes	No	0.01			False Negative
		SRM13-3rd-118	Yes	No	0.01			False Negative

*: Laboratory's RL >> MRRL; in accordance with the General Protocole judged as false negative.

Among the optional compounds there were 23 cases (4× ***N-acetyl-glyphosate***, 4× ***Quizalofop***, 3× ***2,4-DB***, 4× ***glufosinate***, 3× ***phosphonic acid***, 2× ***perchlorate***, 1× ***diquat*** and 1× ***MPP***) 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**). In one case of ***glufosinate*** 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. The 19 false negative results reported by EU/EFTA laboratories accounted for 5.3 % of the total 358 results reported by the EU/EFTA laboratories for optional target compounds. The 23 false negative results reported in total represented 6.2 % of the 372 results reported by all participating labs for optional compounds.

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 and optional compounds. The respective rules are shown in **Section 2.4 (p. 12)**. Among the laboratories from EU and EFTA countries "Acceptable" z-scores were achieved by 82 – 97 % (91 % on average) of the labs in the case of compulsory compounds and by 74 – 92 % (85 % on average) in the case of optional compounds excluding ***diquat*** and ***phosphine***. Overall, 89 % of the results submitted by EU- and EFTA-countries were acceptable, 5 % questionable and 10 % unacceptable (including false negatives). The respective overall figures of 3rd country labs were 74 %, 5 % and 21 %.

A compilation of all individual results and z-scores for each laboratory is shown in **Table 4-8 (p. 36)** and **Table 4-9 (p. 42)** for compulsory and optional 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: 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	Bromide ion	62	56 (90 %)	2 (3 %)	4 (6 %)	1
	Cyromazine	79	65 (82 %)	8 (10 %)	6 (8 %)	1
	Fluazifop	86	83 (97 %)	2 (2 %)	1 (1 %)	1
	Glyphosate	83	78 (94 %)	2 (2 %)	3 (4 %)	0
	Haloxyfop	81	78 (96 %)	2 (2 %)	1 (1 %)	0
	Mepiquat-Cl	86	76 (88 %)	2 (2 %)	8 (9 %)	2
	Subtotal (average)	**477**	**436 (91 %)**	**18 (4 %)**	**23 (5 %)**	**5**
Optional Compounds [2]	2,4-DB	52	48 (92 %)	1 (2 %)	3 (6 %)	2
	Glufosinate	44	37 (84 %)	2 (5 %)	5 (11 %)	3
	MPP	25	20 (80 %)	2 (8 %)	3 (12 %)	1
	N-Acetyl-Glyphosate	23	17 (74 %)	(0 %)	6 (26 %)	5
	Perchlorate	55	47 (85 %)	3 (5 %)	5 (9 %)	2
	Phosphonic acid	45	36 (80 %)	2 (4 %)	7 (16 %)	3
	Quizalofop	62	54 (87 %)	5 (8 %)	3 (5 %)	2
	Subtotal (average)	**306**	**259 (85 %)**	**15 (5 %)**	**32 (10 %)**	**18**
Overall EU/EFTA (Average)		**783**	**695 (89 %)**	**33 (4 %)**	**55 (7 %)**	**23**
3rd country laboratories						
	Compound	No. of results [1]	Acceptable No. (%)	Questionable No. (%)	Unacceptable [1] No. (%)	FNs No.
Compulsory Compounds	Bromide ion	4	4 (100 %)		0 (0 %)	0
	Cyromazine	5	4 (80 %)	1 (20 %)	0 (0 %)	0
	Fluazifop	3	2 (67 %)		1 (33 %)	1
	Glyphosate	7	6 (86 %)		1 (14 %)	0
	Haloxyfop	4	2 (50 %)	1 (25 %)	1 (25 %)	1
	Mepiquat-Cl	4	3 (75 %)		1 (25 %)	0
	Subtotal (average)	**27**	**21 (78 %)**	**2 (7 %)**	**4 (15 %)**	**2**
Optional Compounds [2]	2,4-DB	3	2 (67 %)	(0 %)	1 (33 %)	1
	Glufosinate	6	5 (83 %)	(0 %)	1 (17 %)	1
	MPP					
	N-Acetyl-Glyphosate					
	Perchlorate					
	Phosphonic acid					
	Quizalofop	2	(0 %)	(0 %)	2 (100 %)	2
	Subtotal (average)	**11**	**7 (64 %)**	0 (0 %)	**4 (36 %)**	4
Overall 3rd country (Average)		**38**	**28 (74 %)**	**2 (5 %)**	**8 (21 %)**	**6**

1) including false negatives (FNs)
2) excluding diquat and phosphine

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

COMPULSORY Compound				Bromide ion		Cyromazine		Fluazifop	
MRRL [mg/kg]				2		0.01		0.01	
Assigned Value [mg/kg]				15.356		0.097		0.049	
*CV**				24.0 %		23.5 %		20.3 %	
Lab code SRM13-	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 %)
1		8 / 5	A	20.5	1.3			0.0534	0.3
2		9 / 4	B	15.5	0.0	FN	-3.6	0.052	0.2
3		4 / 3	B			0.0818	-0.6	0.0463	-0.2
4	x	9 / 6	A	18.5	0.8	0.1	0.1	0.0718	1.8
5	x	7 / 4	B					0.0473	-0.2
6		9 / 6	A	14.6	-0.2	0.107	0.4	0.0485	-0.1
7	x	4 / 3	B			0.113	0.7	0.0335	-1.3
8	x	9 / 6	A	16.4	0.3	0.111	0.6	0.0466	-0.2
10		9 / 6	A	14.8	-0.1	0.0464	-2.1	0.0213	-2.3
11		9 / 6	A	14.3	-0.3	0.102	0.2	0.0539	0.4
12	x	9 / 6	A	12.8	-0.7	0.1	0.1	0.052	0.2
13	x	9 / 6	A	17.8	0.6	0.23	5.5	0.038	-0.9
14		9 / 6	A	14.8	-0.1	0.112	0.6	0.0701	1.7
15		9 / 6	A	12.1	-0.8	0.0997	0.1	0.0632	1.1
16		9 / 6	A	19.3	1.0	0.072	-1.0	0.062	1.0
17		7 / 4	B			0.0848	-0.5	0.0426	-0.5
19	x	9 / 6	A	20.3	1.3	1.18	44.7	0.068	1.5
20	x	9 / 6	A	16.5	0.3	0.101	0.2	0.0422	-0.6
21		8 / 5	A	14.3	-0.3			0.048	-0.1
22	x	9 / 6	A	16.4	0.3	0.0772	-0.8	0.0406	-0.7
23		6 / 4	B					0.052	0.2
24	x	8 / 5	A			0.121	1.0	0.065	1.3
25		7 / 5	B	18.7	0.9	0.118	0.9	0.052	0.2
26		9 / 6	A	15.0	-0.1	0.085	-0.5	0.032	-1.4
27		9 / 6	A	17.3	0.5	0.084	-0.5	0.046	-0.3
28		2 / 1	B					0.0565	0.6
29	x	9 / 6	A	23.61	2.1	0.08	-0.7	0.072	1.8
30	x	9 / 6	A	12.9	-0.6	0.114	0.7	0.049	0.0
31		9 / 6	A	18.0	0.7	0.075	-0.9	0.049	0.0
32	x	8 / 5	A			0.0842	-0.5	0.0455	-0.3
33		9 / 6	A	11.3	-1.1	0.092	-0.2	0.0531	0.3
34		9 / 6	A	9.886	-1.4	0.105	0.3	0.047	-0.2
35		8 / 5	A			0.04	-2.3	0.037	-1.0
36	x	8 / 5	A			0.0786	-0.8	0.0496	0.0
37		9 / 6	A	16.4	0.3	0.087	-0.4	0.057	0.6
38		5 / 2	B					0.0483	-0.1
39		1 / 1	B						
40		1 / 1	B						
41		5 / 3	B					0.0686	1.6
42		6 / 4	B			0.075	-0.9	0.038	-0.9
43		1 / 1	B						
44		2 / 1	B						

* Category A/B classification (Cat A was assigned to laboratories that have correctly analysed at least 8 of 9 compulsory compounds on the Target Pesticides List, corretly detected 5 or more out of the 6 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			Glyphosate		Haloxyfop		Mepiquat-Cl	
	MRRL [mg/kg]			0.03		0.003		0.01	
	Assigned Value [mg/kg]			0.903		0.017		0.124	
	*CV**			22.6 %		20.4 %		22.9 %	
	Lab code SRM13-	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 %)
	1		8 / 5	A	0.922	0.1	0.0159	-0.2	0.123	0.0
	2		9 / 4	B	0.631	-1.2	0.014	-0.7	FN	-3.7
	3		4 / 3	B			0.0147	-0.5		
	4	x	9 / 6	A	0.926	0.1	0.016	-0.2	0.124	0.0
	5	x	7 / 4	B	0.4327	-2.1	0.0241	1.7	0.0829	-1.3
	6		9 / 6	A	0.95	0.2	0.0127	-1.0	0.115	-0.3
	7	x	4 / 3	B			0.0157	-0.3		
	8	x	9 / 6	A	0.924	0.1	0.0151	-0.4	0.12	-0.1
	10		9 / 6	A	0.999	0.4	0.0151	-0.4	0.11	-0.4
	11		9 / 6	A	1.12	1.0	0.0177	0.2	0.0959	-0.9
	12	x	9 / 6	A	1.04	0.6	0.017	0.0	0.154	1.0
	13	x	9 / 6	A	1.06	0.7	0.0139	-0.7	0.301	5.7
	14		9 / 6	A	0.821	-0.4	0.0181	0.3	0.12	-0.1
	15		9 / 6	A	0.733	-0.8	0.0181	0.3	0.128	0.1
	16		9 / 6	A	1.17	1.2	0.019	0.5	0.131	0.2
	17		7 / 4	B	0.615	-1.3	0.0182	0.3		
	19	x	9 / 6	A	1.22	1.4	0.017	0.0	1.33	39.0
	20	x	9 / 6	A	0.969	0.3	0.0144	-0.6	0.149	0.8
	21		8 / 5	A	0.839	-0.3	0.016	-0.2	0.147	0.7
	22	x	9 / 6	A	1.05	0.7	0.0147	-0.5	0.132	0.3
	23		6 / 4	B	1.02	0.5	0.017	0.0	0.138	0.5
	24	x	8 / 5	A	1	0.4	0.021	1.0	0.167	1.4
	25		7 / 5	B			0.019	0.5	0.125	0.0
	26		9 / 6	A	0.65	-1.1	0.011	-1.4	0.16	1.2
	27		9 / 6	A	1.02	0.5	0.01	-1.6	0.15	0.8
	28		2 / 1	B						
	29	x	9 / 6	A	0.893	0.0	0.015	-0.4	0.089	-1.1
	30	x	9 / 6	A	1.04	0.6	0.049	7.6	0.149	0.8
	31		9 / 6	A	0.959	0.2	0.016	-0.2	0.108	-0.5
	32	x	8 / 5	A	1.02	0.5	0.0157	-0.3	0.118	-0.2
	33		9 / 6	A	0.883	-0.1	0.017	0.0	0.118	-0.2
	34		9 / 6	A	0.779	-0.5	0.016	-0.2	0.088	-1.2
	35		8 / 5	A	1.23	1.4	0.011	-1.4	0.151	0.9
	36	x	8 / 5	A	0.721	-0.8	0.0171	0.1	0.0841	-1.3
	37		9 / 6	A	1.29	1.7	0.022	1.2	0.15	0.8
	38		5 / 2	B					0.131	0.2
	39		1 / 1	B	0.983	0.4				
	40		1 / 1	B	1.09	0.8				
	41		5 / 3	B	1.17	1.2	0.0226	1.4		
	42		6 / 4	B			0.014	-0.7	0.111	-0.4
	43		1 / 1	B	0.794	-0.5				
	44		2 / 1	B	1.989	4.8				

* Category A/B classification (Cat A was assigned to laboratories that have correctly analysed at least 8 of 9 compulsory compounds on the Target Pesticides List, corretly detected 5 or more out of the 6 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				Bromide ion		Cyromazine		Fluazifop	
MRRL [mg/kg]				2		0.01		0.01	
Assigned Value [mg/kg]				15.356		0.097		0.049	
*CV**				24.0 %		23.5 %		20.3 %	
Lab code SRM13-	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 %)
45		9 / 6	B	15.9	0.1	0.186	3.7	0.022	-2.2
46		9 / 6	A	15.12	-0.1	0.106	0.4	0.047	-0.2
47		0 / 0	B						
48		9 / 6	A	32.6	4.5	0.0792	-0.7	0.0538	0.4
49	x	9 / 6	A	21.85	1.7	0.058	-1.6	0.033	-1.3
50		9 / 6	A	17.1	0.5	0.0988	0.1	0.0472	-0.2
51	x	7 / 4	B	17.1	0.5	0.097	0.0		
52		9 / 6	A	16.2	0.2	0.101	0.2	0.052	0.2
53		6 / 3	B			0.1	0.1	0.039	-0.8
54	x	9 / 6	A	15.8	0.1	0.092	-0.2	0.044	-0.4
55		9 / 6	A	12.9	-0.6	0.11	0.5	0.052	0.2
56		9 / 6	A	13.1	-0.6	0.119	0.9	0.0613	1.0
57	x	3 / 2	B			0.104	0.3		
58		1 / 1	B						
59		0 / 0	B						
60		8 / 5	A			0.117	0.8	0.0507	0.1
61	x	8 / 5	A			0.116	0.8	0.052	0.2
62		7 / 4	B	20.0	1.2	0.0446	-2.2	0.0413	-0.6
63		9 / 6	A	13.63	-0.4	0.103	0.3	0.053	0.3
64		2 / 1	B						
65		8 / 5	A	FN	-3.5	0.073	-1.0	0.054	0.4
66		5 / 2	B					0.054	0.4
67		7 / 5	B	14.1	-0.3	0.0379	-2.4	0.0345	-1.2
68	x	8 / 5	A	13.7	-0.4	0.143	1.9	0.043	-0.5
69		9 / 6	A	13.7	-0.4	0.095	-0.1	0.047	-0.2
70		3 / 2	B						
71		8 / 5	A			0.0996	0.1	0.0516	0.2
72		9 / 6	A	14.1	-0.3	0.0894	-0.3	0.0431	-0.5
73		8 / 5	A			0.22	5.1	0.074	2.0
74	x	9 / 6	A	4.29	-2.9	0.0472	-2.1	0.0431	-0.5
76	x	8 / 5	A			0.0716	-1.0	0.0287	-1.7
77	x	3 / 2	B			0.084	-0.5		
78	x	9 / 6	A	12.4	-0.8	0.045	-2.1	0.055	0.5
79	x	9 / 6	A	12.5	-0.7	0.094	-0.1	0.05	0.1
80	x	6 / 4	B					0.062	1.0
82		3 / 2	B	17.0	0.4	0.096	0.0		
83		9 / 6	A	3.0	-3.2	0.104	0.3	0.047	-0.2
84		4 / 3	B			0.12	1.0	0.069	1.6
85		8 / 5	A	9.0	-1.7	0.132	1.4	0.038	-0.9
86		8 / 5	A			0.116	0.8	0.0538	0.4
87		8 / 5	A			0.0898	-0.3	0.0451	-0.3
88		9 / 6	A	16.9	0.4	0.0263	-2.9	0.048	-0.1
89		9 / 6	A	16.5	0.3	0.081	-0.7	0.051	0.1

* Category A/B classification (Cat A was assigned to laboratories that have correctly analysed at least 8 of 9 compulsory compounds on the Target Pesticides List, corretly detected 5 or more out of the 6 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			Glyphosate		Haloxyfop		Mepiquat-Cl	
	MRRL [mg/kg]			0.03		0.003		0.01	
	Assigned Value [mg/kg]			0.903		0.017		0.124	
	*CV**			22.6 %		20.4 %		22.9 %	
	Lab code SRM13-	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 %)
	45		9 / 6	B	0.736	-0.7	0.02	0.7	0.141	0.6
	46		9 / 6	A	0.912	0.0	0.016	-0.2	0.095	-0.9
	47		0 / 0	B						
	48		9 / 6	A	0.713	-0.8	0.0198	0.7	0.155	1.0
	49	x	9 / 6	A	0.756	-0.7	0.019	0.5	0.655	17.2
	50		9 / 6	A	1.31	1.8	0.0192	0.6	0.124	0.0
	51	x	7 / 4	B	0.97	0.3			0.122	-0.1
	52		9 / 6	A	0.794	-0.5	0.018	0.3	0.115	-0.3
	53		6 / 3	B					0.096	-0.9
	54	x	9 / 6	A	1.21	1.4	0.014	-0.7	0.126	0.1
	55		9 / 6	A	0.667	-1.0	0.019	0.5	0.133	0.3
	56		9 / 6	A	0.916	0.1	0.023	1.5	0.103	-0.7
	57	x	3 / 2	B					0.148	0.8
	58		1 / 1	B	0.867	-0.2				
	59		0 / 0	B						
	60		8 / 5	A	0.772	-0.6	0.0137	-0.7	0.12	-0.1
	61	x	8 / 5	A	0.987	0.4	0.017	0.0	0.142	0.6
	62		7 / 4	B			0.0117	-1.2		
	63		9 / 6	A	0.68	-1.0	0.0155	-0.3	0.021	-3.3
	64		2 / 1	B	0.751	-0.7				
	65		8 / 5	A	0.65	-1.1	0.018	0.3	0.115	-0.3
	66		5 / 2	B			0.023	1.5	FN	-3.7
	67		7 / 5	B	1.09	0.8			0.108	-0.5
	68	x	8 / 5	A			0.0058	-2.6	0.173	1.6
	69		9 / 6	A	0.849	-0.2	0.014	-0.7	0.107	-0.5
	70		3 / 2	B	0.962	0.3			0.128	0.1
	71		8 / 5	A	0.788	-0.5	0.014	-0.7	0.112	-0.4
	72		9 / 6	A	0.896	0.0	0.0164	-0.1	0.093	-1.0
	73		8 / 5	A	0.833	-0.3	0.022	1.2	0.127	0.1
	74	x	9 / 6	A	0.56	-1.5	0.0168	0.0	0.133	0.3
	76	x	8 / 5	A	0.69	-0.9	0.0166	-0.1	0.0657	-1.9
	77	x	3 / 2	B					0.122	-0.1
	78	x	9 / 6	A	0.722	-0.8	0.025	1.9	0.109	-0.5
	79	x	9 / 6	A	1.182	1.2	0.015	-0.4	0.11	-0.4
	80	x	6 / 4	B	2.3	6.2	0.025	1.9	0.131	0.2
	82		3 / 2	B						
	83		9 / 6	A	0.48	-1.9	0.016	-0.2	0.028	-3.1
	84		4 / 3	B			0.017	0.0		
	85		8 / 5	A			0.02	0.7	0.185	2.0
	86		8 / 5	A	0.896	0.0	0.0236	1.6	0.165	1.3
	87		8 / 5	A	0.692	-0.9	0.017	0.0	0.111	-0.4
	88		9 / 6	A	1.1	0.9	0.014	-0.7	0.104	-0.6
	89		9 / 6	A	0.931	0.1	0.015	-0.4	0.075	-1.6

* Category A/B classification (Cat A was assigned to laboratories that have correctly analysed at least 8 of 9 compulsory compounds on the Target Pesticides List, corretly detected 5 or more out of the 6 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				Bromide ion		Cyromazine		Fluazifop	
MRRL [mg/kg]				2		0.01		0.01	
Assigned Value [mg/kg]				15.356		0.097		0.049	
*CV**				24.0 %		23.5 %		20.3 %	
Lab code SRM13-	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 %)
90		1 / 0	B					FN	-3.6
91		9 / 6	A	12.4	-0.8	0.108	0.5	0.0579	0.7
92		9 / 6	A	12.9	-0.6	0.115	0.7	0.0403	-0.7
93		9 / 6	A	13.0	-0.6	0.111	0.6	0.055	0.5
94		1 / 1	B	18.473	0.8				
95		1 / 1	B						
96		7 / 5	B			0.0895	-0.3	0.058	0.7
97		9 / 6	A	27.9	3.3	0.102	0.2	0.048	-0.1
98		9 / 6	A	11.6	-1.0	0.084	-0.5	0.054	0.4
99		2 / 1	B						
100	x	4 / 3	B	16.3	0.2			0.049	0.0
101		8 / 5	A			0.101	0.2	0.025	-2.0
102		1 / 1	B	7.82	-2.0				
103		1 / 1	B						
104		9 / 6	A	23.0	2.0	0.075	-0.9	0.025	-2.0
105		9 / 6	A	10.3	-1.3	0.088	-0.4	0.041	-0.7
106		5 / 3	B			0.94	34.8		
107		6 / 4	B					0.06	0.9
109		8 / 5	A			0.168	2.9	0.0451	-0.3
110		4 / 2	B						
111		4 / 2	B	19.9	1.2				
112		9 / 6	A	12.0	-0.9	0.12	1.0	0.06	0.9
113		0 / 0	B						
114		0 / 0	B						
3rd-115		6 / 3	B			0.087	-0.4		
3rd-116		8 / 5	B	12.9	-0.6	0.07	-1.1		
3rd-117		9 / 6	A	15.8	0.1	0.0689	-1.2	0.0433	-0.5
3rd-118		6 / 3	B	14.4	-0.2	0.0831	-0.6	FN	-3.2
3rd-119		2 / 1	B					0.0407	-0.7
3rd-120		3 / 1	B						
3rd-121		1 / 1	B						
3rd-122		7 / 5	B	16.8	0.4	0.147	2.1		

* Category A/B classification (Cat A was assigned to laboratories that have correctly analysed at least 8 of 9 compulsory compounds on the Target Pesticides List, corretly detected 5 or more out of the 6 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				Glyphosate		Haloxyfop		Mepiquat-Cl	
MRRL [mg/kg]				0.03		0.003		0.01	
Assigned Value [mg/kg]				0.903		0.017		0.124	
*CV**				22.6 %		20.4 %		22.9 %	
Lab code SRM13-	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 %)
90		1 / 0	B						
91		9 / 6	A	1.01	0.5	0.0142	-0.6	0.149	0.8
92		9 / 6	A	0.967	0.3	0.014	-0.7	0.137	0.4
93		9 / 6	A	0.82	-0.4	0.018	0.3	0.15	0.8
94		1 / 1	B						
95		1 / 1	B					0.121	-0.1
96		7 / 5	B	0.954	0.2	0.0166	-0.1	0.0986	-0.8
97		9 / 6	A	0.832	-0.3	0.019	0.5	0.027	-3.1
98		9 / 6	A	0.859	-0.2	0.018	0.3	0.125	0.0
99		2 / 1	B					0.115	-0.3
100	x	4 / 3	B			0.019	0.5		
101		8 / 5	A	1.038	0.6	0.009	-1.9	0.198	2.4
102		1 / 1	B						
103		1 / 1	B	13.5	55.8				
104		9 / 6	A	0.678	-1.0	0.012	-1.2	0.11	-0.4
105		9 / 6	A	0.366	-2.4	0.0071	-2.3	0.19	2.1
106		5 / 3	B	0.93	0.1			0.1	-0.8
107		6 / 4	B	0.93	0.1	0.02	0.7	0.09	-1.1
109		8 / 5	A	0.824	-0.3	0.0199	0.7	0.122	-0.1
110		4 / 2	B	0.55	-1.6			0.143	0.6
111		4 / 2	B					0.089	-1.1
112		9 / 6	A	0.85	-0.2	0.02	0.7	0.117	-0.2
113		0 / 0	B						
114		0 / 0	B						
3rd-115		6 / 3	B	1.07	0.7	FN	-3.3	0.123	0.0
3rd-116		8 / 5	B	0.6	-1.3	0.007	-2.3	0.11	-0.4
3rd-117		9 / 6	A	0.175	-3.2	0.0119	-1.2	0.152	0.9
3rd-118		6 / 3	B	0.715	-0.8				
3rd-119		2 / 1	B						
3rd-120		3 / 1	B	0.9	0.0				
3rd-121		1 / 1	B	0.919	0.1				
3rd-122		7 / 5	B	0.903	0.0	0.019	0.5	0.27	4.7

* Category A/B classification (Cat A was assigned to laboratories that have correctly analysed at least 8 of 9 compulsory compounds on the Target Pesticides List, corretly detected 5 or more out of the 6 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

COMPULSORY Compound				2,4-DB		Glufosinate		MPP		N-Acetyl-Glyphosate	
MRRL [mg/kg]				0.01		0.02		0.02		0.02	
Assigned Value [mg/kg]				0.183		0.192		0.188		0.835	
*CV**				20.7 %		28.7 %		18.9 %		21.1 %	
Lab code SRM13-	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		7 / 2	A	0.231	1.0						
2		12 / 5	B	0.174	-0.2	0.137	-1.1				
3		4 / 1	B	0.146	-0.8						
4	x	9 / 5	A			0.187	-0.1			0.862	0.1
5	x	4 / 1	B								
6		6 / 3	A								
7	x	4 / 1	B	0.1557	-0.6						
8	x	6 / 3	A	0.156	-0.6					0.644	-0.9
10		7 / 3	A								
11		10 / 4	A	0.225	0.9						
12	x	6 / 2	A	0.16	-0.5					0.803	-0.2
13	x	13 / 8	A	0.285	2.2	0.194	0.1	0.215	0.6	0.784	-0.2
14		14 / 8	A	0.192	0.2	0.131	-1.3				
15		11 / 6	A			0.261	1.4	0.173	-0.3	0.613	-1.1
16		12 / 7	A	0.219	0.8	0.286	2.0				
17		9 / 6	B			0.0687	-2.6				
19	x	4 / 1	A	0.232	1.1						
20	x	4 / 2	A	0.157	-0.6						
21		7 / 2	A							0.863	0.1
22	x	5 / 2	A	0.2	0.4	0.198	0.1				
23		4 / 1	B	0.2	0.4						
24	x	3 / 1	A	0.19	0.2						
25		2 / 0	B								
26		8 / 5	A	0.14	-0.9	0.16	-0.7				
27		13 / 6	A			0.24	1.0	0.17	-0.4		
28		0 / 0	B								
29	x	2 / 0	A								
30	x	15 / 8	A	0.203	0.4	0.189	-0.1	0.189	0.0	1.18	1.7
31		10 / 4	A								
32	x	13 / 7	A	0.142	-0.9						
33		10 / 5	A	0.218	0.8	0.18	-0.2				
34		17 / 10	A	0.176	-0.2	0.201	0.2	0.181	-0.1	16.685	76.0
35		12 / 7	A	0.131	-1.1	0.183	-0.2	0.307	2.5		
36	x	13 / 8	A	0.197	0.3	0.154	-0.8	0.171	-0.4	0.733	-0.5
37		8 / 5	A			0.24	1.0	0.19	0.0		
38		3 / 1	B								
39		0 / 0	B								
40		1 / 0	B								
41		8 / 6	B	0.217	0.7	0.23	0.8	0.159	-0.6	0.743	-0.4
42		2 / 0	B								
43		1 / 0	B								

* Category A/B classification (Cat A was assigned to laboratories that have correctly analysed at least 8 of 9 compulsory compounds on the Target Pesticides List, corretly detected 5 or more out of the 6 compulsory compounds and that have not reported any false positive results)
FN* = analysed for a compound present in the test material and reported not detected due to lab's RL > assigned value, therefore judged as

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

	COMPULSORY Compound			Perchlorate		Phosphonic acid		Quizalofop		Diquat	
			MRRL [mg/kg]	0.01		0.05		0.01		0.02	
			Assigned Value [mg/kg]	0.100		1.864		0.052		*1.701 (uncertain)*	
			*CV**	16.9 %		24.3 %		23.6 %		52.8 %	
	Lab code SRM13-	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		7 / 2	A					0.0455	-0.5	1.2	*-1.2*
	2		12 / 5	B	FN	-3.6	0.142	-3.7	0.083	2.4	2.94	*2.9*
	3		4 / 1	B					0.054	0.2		
	4	x	9 / 5	A	0.101	0.1	2.38	1.1	0.0572	0.4		
	5	x	4 / 1	B	0.0842	-0.6						
	6		6 / 3	A	0.115	0.6	1.973	0.2				
	7	x	4 / 1	B					0.0395	-1.0		
	8	x	6 / 3	A	0.113	0.5			0.0377	-1.1		
	10		7 / 3	A	0.0996	0.0	0.902	-2.1				
	11		10 / 4	A	0.0915	-0.3	FN	-3.9				
	12	x	6 / 2	A					0.055	0.2		
	13	x	13 / 8	A	0.109	0.4	2.36	1.1	0.0557	0.3	7.59	*13.8*
	14		14 / 8	A	0.104	0.2	1.59	-0.6	0.0512	-0.1	1.39	*-0.7*
	15		11 / 6	A	0.0726	-1.1	2.01	0.3				
	16		12 / 7	A	0.093	-0.3	1.6	-0.6	0.066	1.1		
	17		9 / 6	B	0.0763	-0.9	1.94	0.2	0.0546	0.2		
	19	x	4 / 1	A					0.065	1.0		
	20	x	4 / 2	A	0.0968	-0.1			0.0437	-0.6		
	21		7 / 2	A					0.043	-0.7	0.529	*-2.8*
	22	x	5 / 2	A								
	23		4 / 1	B					0.056	0.3		
	24	x	3 / 1	A					0.059	0.5		
	25		2 / 0	B								
	26		8 / 5	A	0.16	2.4	2.2	0.7	0.023	-2.2		
	27		13 / 6	A	0.12	0.8	1.623	-0.5	0.03	-1.7	2.44	*1.7*
	28		0 / 0	B								
	29	x	2 / 0	A					0.071	1.5		
	30	x	15 / 8	A	0.093	-0.3	2.05	0.4	0.062	0.8	1.35	*-0.8*
	31		10 / 4	A	0.092	-0.3	1.47	-0.8			0.878	*-1.9*
	32	x	13 / 7	A	0.096	-0.1	1.98	0.2	0.0515	0.0	1.59	*-0.3*
	33		10 / 5	A	0.097	-0.1	1.91	0.1	0.061	0.7		
	34		17 / 10	A	0.089	-0.4	1.982	0.3	0.058	0.5	1.979	*0.7*
	35		12 / 7	A	0.048	-2.1	1.278	-1.3	0.037	-1.1	1.563	*-0.3*
	36	x	13 / 8	A	0.0728	-1.1	1.65	-0.5	0.0505	-0.1	0.742	*-2.3*
	37		8 / 5	A	0.11	0.4	2.04	0.4				
	38		3 / 1	B	0.102	0.1			0.0499	-0.2		
	39		0 / 0	B								
	40		1 / 0	B								
	41		8 / 6	B			7.5	12.1	0.0713	1.5		
	42		2 / 0	B					0.052	0.0		
	43		1 / 0	B								

* Category A/B classification (Cat A was assigned to laboratories that have correctly analysed at least 8 of 9 compulsory compounds on the Target Pesticides List, corretly detected 5 or more out of the 6 compulsory compounds and that have not reported any false positive results)
FN* = analysed for a compound present in the test material and reported not detected due to lab's RL > assigned value, therefore judged as

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

COMPULSORY Compound				2,4-DB		Glufosinate		MPP		N-Acetyl-Glyphosate	
MRRL [mg/kg]				0.01		0.02		0.02		0.02	
Assigned Value [mg/kg]				0.183		0.192		0.188		0.835	
*CV**				20.7 %		28.7 %		18.9 %		21.1 %	
Lab code SRM13-	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		6 / 2	B			0.319	2.7				
45		15 / 7	B	0.194	0.2	0.24	1.0	1.08	19.0	FN	-3.9
46		15 / 8	A	0.182	0.0	0.174	-0.4	0.17	-0.4	0.71	-0.6
47		1 / 0	B								
48		8 / 3	A								
49	x	2 / 1	A	0.164	-0.4						
50		8 / 4	A	0.183	0.0						
51	x	0 / 0	B								
52		13 / 7	A	0.216	0.7	0.205	0.3	0.225	0.8		
53		1 / 0	B								
54	x	4 / 0	A								
55		8 / 2	A	FN	-3.8	FN	-3.6			FN	-3.9
56		4 / 1	A								
57	x	3 / 1	B								
58		1 / 0	B								
59		1 / 0	B								
60		9 / 4	A	0.176	-0.2	0.172	-0.4	0.165	-0.5		
61	x	2 / 0	A								
62		9 / 5	B			0.371	3.7				
63		15 / 8	A	0.19	0.2	0.7	10.6	0.156	-0.7	0.769	-0.3
64		2 / 0	B								
65		2 / 0	A								
66		2 / 0	B								
67		9 / 5	B			0.177	-0.3	0.173	-0.3		
68	x	2 / 0	A								
69		8 / 4	A	0.234	1.1						
70		0 / 0	B								
71		11 / 5	A	0.155	-0.6	0.203	0.2			0.977	0.7
72		8 / 4	A	0.164	-0.4						
73		4 / 1	A			0.152	-0.8				
74	x	3 / 1	A	0.139	-1.0						
76	x	9 / 3	A	0.174	-0.2	0.166	-0.5			FN	-3.9
77	x	1 / 0	B								
78	x	1 / 1	A			0.133	-1.2				
79	x	7 / 4	A	0.132	-1.1	0.105	-1.8				
80	x	2 / 0	B								
82		2 / 0	B								
83		15 / 8	A	0.19	0.2	0.13	-1.3	0.15	-0.8	0.65	-0.9
84		1 / 0	B								
85		7 / 2	A	0.173	-0.2						
86		11 / 6	A	0.319	3.0	0.224	0.7			0.979	0.7

* Category A/B classification (Cat A was assigned to laboratories that have correctly analysed at least 8 of 9 compulsory compounds on the Target Pesticides List, corretly detected 5 or more out of the 6 compulsory compounds and that have not reported any false positive results)
FN* = analysed for a compound present in the test material and reported not detected due to lab's RL > assigned value, therefore judged as

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

	COMPULSORY Compound			Perchlorate		Phosphonic acid		Quizalofop		Diquat	
			MRRL [mg/kg]	0.01		0.05		0.01		0.02	
			Assigned Value [mg/kg]	0.100		1.864		0.052		*1.701 (uncertain)*	
			*CV**	16.9 %		24.3 %		23.6 %		52.8 %	
	Lab code SRM13-	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		6 / 2	B	0.138	1.5						
	45		15 / 7	B	0.143	1.7	1.17	-1.5	0.07	1.4	2.62	*2.2*
	46		15 / 8	A	0.102	0.1	1.93	0.1	0.049	-0.2	5.07	*7.9*
	47		1 / 0	B					FN	-4.0		
	48		8 / 3	A	0.0892	-0.4	2.07	0.4	0.0463	-0.4		
	49	x	2 / 1	A					0.057	0.4		
	50		8 / 4	A	0.101	0.1	2.29	0.9				
	51	x	0 / 0	B								
	52		13 / 7	A	0.11	0.4	1.84	-0.1	0.0504	-0.1	3.116	*3.3*
	53		1 / 0	B								
	54	x	4 / 0	A					0.04	-0.9		
	55		8 / 2	A			1.4	-1.0	0.056	0.3		
	56		4 / 1	A	0.098	-0.1			0.0596	0.6		
	57	x	3 / 1	B	0.102	0.1						
	58		1 / 0	B								
	59		1 / 0	B								
	60		9 / 4	A	0.0925	-0.3			0.0502	-0.1		
	61	x	2 / 0	A								
	62		9 / 5	B	0.115	0.6	1.96	0.2	0.0568	0.4	0.864	*-2.0*
	63		15 / 8	A	0.033	-2.7	0.387	-3.2	0.044	-0.6	0.43	*-3.0*
	64		2 / 0	B								
	65		2 / 0	A								
	66		2 / 0	B					FN	-3.2		
	67		9 / 5	B	0.0825	-0.7	1.95	0.2	0.0301	-1.7		
	68	x	2 / 0	A								
	69		8 / 4	A	0.193	3.7	1.86	0.0	0.046	-0.5		
	70		0 / 0	B								
	71		11 / 5	A	0.117	0.7					1.25	*-1.1*
	72		8 / 4	A	0.0921	-0.3	1.75	-0.2	0.0486	-0.3		
	73		4 / 1	A								
	74	x	3 / 1	A								
	76	x	9 / 3	A					0.0221	-2.3	3.48	*4.2*
	77	x	1 / 0	B								
	78	x	1 / 1	A								
	79	x	7 / 4	A					0.041	-0.8		
	80	x	2 / 0	B								
	82		2 / 0	B								
	83		15 / 8	A	0.023	-3.1	0.37	-3.2	0.055	0.2	1.5	*-0.5*
	84		1 / 0	B								
	85		7 / 2	A	0.102	0.1	FN	-3.9				
	86		11 / 6	A			2.13	0.6	0.0517	0.0	1.28	*-1.0*

* Category A/B classification (Cat A was assigned to laboratories that have correctly analysed at least 8 of 9 compulsory compounds on the Target Pesticides List, corretly detected 5 or more out of the 6 compulsory compounds and that have not reported any false positive results)
FN* = analysed for a compound present in the test material and reported not detected due to lab's RL > assigned value, therefore judged as

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

COMPULSORY Compound				2,4-DB		Glufosinate		MPP		N-Acetyl-Glyphosate	
MRRL [mg/kg]				0.01		0.02		0.02		0.02	
Assigned Value [mg/kg]				0.183		0.192		0.188		0.835	
*CV**				20.7 %		28.7 %		18.9 %		21.1 %	
Lab code SRM13-	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 %)
87		5 / 2	A	0.146	-0.8						
88		10 / 6	A	0.18	-0.1	0.247	1.2	0.233	1.0		
89		15 / 6	A	0.167	-0.3	FN	-3.6	0.162	-0.6	FN	-3.9
90		1 / 0	B								
91		11 / 6	A	0.134	-1.1			0.215	0.6	0.72	-0.5
92		7 / 4	A			0.0954	-2.0	0.19	0.0		
93		12 / 6	A	0.216	0.7	0.181	-0.2				
94		0 / 0	B								
95		0 / 0	B								
96		1 / 0	B								
97		13 / 5	A	0.234	1.1	0.227	0.7	0.169	-0.4		
98		12 / 7	A	0.147	-0.8	0.199	0.2				
99		1 / 0	B								
100	x	0 / 0	B								
101		14 / 7	A	0.104	-1.7	0.188	-0.1	0.617	9.1		
102		0 / 0	B								
103		0 / 0	B								
104		0 / 0	A								
105		13 / 6	A	0.193	0.2	FN	-3.6	0.066	-2.6	0.938	0.5
106		3 / 2	B								
107		7 / 3	B	0.16	-0.5	0.23	0.8				
109		12 / 1	A	FN	-3.8	0.114	-1.6	FN	-3.6	FN	-3.9
110		5 / 2	B								
111		2 / 1	B								
112		17 / 10	A	0.24	1.2	0.185	-0.1	0.198	0.2	1.04	1.0
113		2 / 2	B								
114		2 / 2	B								
3rd-115		5 / 2	B	FN	-3.8	0.183	-0.2				
3rd-116		4 / 2	B			0.166	-0.5				
3rd-117		5 / 2	A	0.131	-1.1	0.166	-0.5				
3rd-118		6 / 1	B								
3rd-119		2 / 1	B	0.188	0.1						
3rd-120		3 / 1	B			0.2	0.2				
3rd-121		1 / 0	B			FN	-3.6				
3rd-122		1 / 1	B			0.223	0.7				

* Category A/B classification (Cat A was assigned to laboratories that have correctly analysed at least 8 of 9 compulsory compounds on the Target Pesticides List, corretly detected 5 or more out of the 6 compulsory compounds and that have not reported any false positive results)
FN* = analysed for a compound present in the test material and reported not detected due to lab's RL > assigned value, therefore judged as

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

	COMPULSORY Compound			Perchlorate		Phosphonic acid		Quizalofop		Diquat	
	MRRL [mg/kg]			0.01		0.05		0.01		0.02	
	Assigned Value [mg/kg]			0.100		1.864		0.052		*1.701 (uncertain)*	
	*CV**			16.9 %		24.3 %		23.6 %		52.8 %	
	Lab code SRM13-	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 %)
	87		5 / 2	A	0.0679	-1.3			0.0508	-0.1		
	88		10 / 6	A	0.11	0.4	2.12	0.5	0.052	0.0		
	89		15 / 6	A	0.102	0.1	2.24	0.8	0.052	0.0	1.45	*-0.6*
	90		1 / 0	B								
	91		11 / 6	A	0.106	0.3	0.688	-2.5	0.0547	0.2		
	92		7 / 4	A	0.101	0.1			0.0332	-1.4	1.74	*0.1*
	93		12 / 6	A	0.14	1.6	2.4	1.1	0.068	1.2	1.8	*0.2*
	94		0 / 0	B								
	95		0 / 0	B								
	96		1 / 0	B					0.0613	0.7		
	97		13 / 5	A	0.099	0.0	FN	-3.9	0.063	0.9	1.9	*0.5*
	98		12 / 7	A	0.102	0.1	2.45	1.3	0.054	0.2		
	99		1 / 0	B								
	100	x	0 / 0	B								
	101		14 / 7	A	0.335	9.5	2.133	0.6	0.023	-2.2	1.922	*0.5*
	102		0 / 0	B								
	103		0 / 0	B								
	104		0 / 0	A								
	105		13 / 6	A	0.09	-0.4	1.67	-0.4	0.035	-1.3		
	106		3 / 2	B			2.4	1.1				
	107		7 / 3	B					0.1	3.7	1.41	*-0.7*
	109		12 / 1	A	FN	-3.6			0.045	-0.5	FN	*-4.0*
	110		5 / 2	B	0.087	-0.5					0.768	*-2.2*
	111		2 / 1	B	0.096	-0.1						
	112		17 / 10	A	0.106	0.3	2.02	0.3	0.081	2.2	1.6	*-0.2*
	113		2 / 2	B								
	114		2 / 2	B								
	3rd-115		5 / 2	B					FN	-3.6	1.11	*-1.4*
	3rd-116		4 / 2	B							0.27	*-3.4*
	3rd-117		5 / 2	A								
	3rd-118		6 / 1	B					FN	-3.6	0.913	*-1.9*
	3rd-119		2 / 1	B								
	3rd-120		3 / 1	B								
	3rd-121		1 / 0	B								
	3rd-122		1 / 1	B								

* Category A/B classification (Cat A was assigned to laboratories that have correctly analysed at least 8 of 9 compulsory compounds on the Target Pesticides List, corretly detected 5 or more out of the 6 compulsory compounds and that have not reported any false positive results)
FN* = analysed for a compound present in the test material and reported not detected due to lab's RL > assigned value, therefore judged as

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. 12)**. Following the rules defined in the General Protocol (8th 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 eight out of the nine compulsory pesticides on the Target Pesticides List; b) correct detection of at least five out of the six compulsory pesticides present in the test item, and c) no false positive results.

A total of 64 EU and EFTA laboratories (59 %) were classified into Category A and 45 (41 %) into Category B. One out of the 8 EU candidate and third-country laboratories was 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 = 362), whereas those classified into Category B achieved an overall AAZ of 1.1 (n = 110). When including laboratories from EU candidate and third countries, the AAZ for compsulsory compounds remains the same (n = 367) and for optional compounds decreased to 1.0 (n = 131).

Table 4-10 and **Table 4-11 (p. 50)** 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 4-10: Category A laboratories ordered by lab-codes

COMPULSORY Compounds			Bromide ion	Cyromazine	Fluazifop	Glyphosate	Haloxyfop	Mepiquat-Cl	
MRRL [mg/kg]			2	0.01	0.01	0.03	0.003	0.01	
Assigned Value [mg/kg]			15.356	0.097	0.049	0.903	0.017	0.124	
*CV**			24.0 %	23.5 %	20.3 %	22.6 %	20.4 %	22.9 %	
Lab code SRM13-	NRL-SRM	Analysed / corr. found [1]	z-Scores	z-Scores	z-Scores	z-Scores	z-Scores	z-Scores	AAZ [2]
1		8 / 5	1.3		0.3	0.1	-0.2	0.0	**0.4**
4	x	9 / 6	0.8	0.1	1.8	0.1	-0.2	0.0	**0.5**
6		9 / 6	-0.2	0.4	-0.1	0.2	-1.0	-0.3	**0.4**
8	x	9 / 6	0.3	0.6	-0.2	0.1	-0.4	-0.1	**0.3**
10		9 / 6	-0.1	-2.1	-2.3	0.4	-0.4	-0.4	**1.0**
11		9 / 6	-0.3	0.2	0.4	1.0	0.2	-0.9	**0.5**
12	x	9 / 6	-0.7	0.1	0.2	0.6	0.0	1.0	**0.4**
13	x	9 / 6	0.6	5.5	-0.9	0.7	-0.7	5.7	**2.2**
14		9 / 6	-0.1	0.6	1.7	-0.4	0.3	-0.1	**0.5**
15		9 / 6	-0.8	0.1	1.1	-0.8	0.3	0.1	**0.5**
16		9 / 6	1.0	-1.0	1.0	1.2	0.5	0.2	**0.8**
19	x	9 / 6	1.3	44.7	1.5	1.4	0.0	39.0	**2.4**
20	x	9 / 6	0.3	0.2	-0.6	0.3	-0.6	0.8	**0.5**
21		8 / 5	-0.3		-0.1	-0.3	-0.2	0.7	**0.3**
22	x	9 / 6	0.3	-0.8	-0.7	0.7	-0.5	0.3	**0.6**
24	x	8 / 5		1.0	1.3	0.4	1.0	1.4	**1.0**
26		9 / 6	-0.1	-0.5	-1.4	-1.1	-1.4	1.2	**1.0**
27		9 / 6	0.5	-0.5	-0.3	0.5	-1.6	0.8	**0.7**
29	x	9 / 6	2.1	-0.7	1.8	0.0	-0.4	-1.1	**1.0**
30	x	9 / 6	-0.6	0.7	0.0	0.6	7.6	0.8	**1.3**
31		9 / 6	0.7	-0.9	0.0	0.2	-0.2	-0.5	**0.4**

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			Bromide ion	Cyromazine	Fluazifop	Glyphosate	Haloxyfop	Mepiquat-Cl	
MRRL [mg/kg]			2	0.01	0.01	0.03	0.003	0.01	
Assigned Value [mg/kg]			15.356	0.097	0.049	0.903	0.017	0.124	
*CV**			24.0 %	23.5 %	20.3 %	22.6 %	20.4 %	22.9 %	
Lab code SRM13-	NRL-SRM	Analysed / corr. found [1]	z-Scores	z-Scores	z-Scores	z-Scores	z-Scores	z-Scores	AAZ [2]
32	x	8 / 5		-0.5	-0.3	0.5	-0.3	-0.2	**0.4**
33		9 / 6	-1.1	-0.2	0.3	-0.1	0.0	-0.2	**0.3**
34		9 / 6	-1.4	0.3	-0.2	-0.5	-0.2	-1.2	**0.6**
35		8 / 5		-2.3	-1.0	1.4	-1.4	0.9	**1.4**
36	x	8 / 5		-0.8	0.0	-0.8	0.1	-1.3	**0.6**
37		9 / 6	0.3	-0.4	0.6	1.7	1.2	0.8	**0.8**
46		9 / 6	-0.1	0.4	-0.2	0.0	-0.2	-0.9	**0.3**
48		9 / 6	4.5	-0.7	0.4	-0.8	0.7	1.0	**1.4**
49	x	9 / 6	1.7	-1.6	-1.3	-0.7	0.5	17.2	**1.8**
50		9 / 6	0.5	0.1	-0.2	1.8	0.6	0.0	**0.5**
52		9 / 6	0.2	0.2	0.2	-0.5	0.3	-0.3	**0.3**
54	x	9 / 6	0.1	-0.2	-0.4	1.4	-0.7	0.1	**0.5**
55		9 / 6	-0.6	0.5	0.2	-1.0	0.5	0.3	**0.5**
56		9 / 6	-0.6	0.9	1.0	0.1	1.5	-0.7	**0.8**
60		8 / 5		0.8	0.1	-0.6	-0.7	-0.1	**0.5**
61	x	8 / 5		0.8	0.2	0.4	0.0	0.6	**0.4**
63		9 / 6	-0.4	0.3	0.3	-1.0	-0.3	-3.3	**0.9**
65		8 / 5	-3.5 [FN]	-1.0	0.4	-1.1	0.3	-0.3	**1.1**
68	x	8 / 5	-0.4	1.9	-0.5		-2.6	1.6	**1.4**
69		9 / 6	-0.4	-0.1	-0.2	-0.2	-0.7	-0.5	**0.4**
71		8 / 5		0.1	0.2	-0.5	-0.7	-0.4	**0.4**
72		9 / 6	-0.3	-0.3	-0.5	0.0	-0.1	-1.0	**0.4**
73		8 / 5		5.1	2.0	-0.3	1.2	0.1	**1.7**
74	x	9 / 6	-2.9	-2.1	-0.5	-1.5	0.0	0.3	**1.2**
76	x	8 / 5		-1.0	-1.7	-0.9	-0.1	-1.9	**1.1**
78	x	9 / 6	-0.8	-2.1	0.5	-0.8	1.9	-0.5	**1.1**
79	x	9 / 6	-0.7	-0.1	0.1	1.2	-0.4	-0.4	**0.5**
83		9 / 6	-3.2	0.3	-0.2	-1.9	-0.2	-3.1	**1.5**
85		8 / 5	-1.7	1.4	-0.9		0.7	2.0	**1.3**
86		8 / 5		0.8	0.4	0.0	1.6	1.3	**0.8**
87		8 / 5		-0.3	-0.3	-0.9	0.0	-0.4	**0.4**
88		9 / 6	0.4	-2.9	-0.1	0.9	-0.7	-0.6	**0.9**
89		9 / 6	0.3	-0.7	0.1	0.1	-0.4	-1.6	**0.5**
91		9 / 6	-0.8	0.5	0.7	0.5	-0.6	0.8	**0.7**
92		9 / 6	-0.6	0.7	-0.7	0.3	-0.7	0.4	**0.6**
93		9 / 6	-0.6	0.6	0.5	-0.4	0.3	0.8	**0.5**
97		9 / 6	3.3	0.2	-0.1	-0.3	0.5	-3.1	**1.3**
98		9 / 6	-1.0	-0.5	0.4	-0.2	0.3	0.0	**0.4**
101		8 / 5		0.2	-2.0	0.6	-1.9	2.4	**1.4**
104		9 / 6	2.0	-0.9	-2.0	-1.0	-1.2	-0.4	**1.3**
105		9 / 6	-1.3	-0.4	-0.7	-2.4	-2.3	2.1	**1.5**
109		8 / 5		2.9	-0.3	-0.3	0.7	-0.1	**0.9**
112		9 / 6	-0.9	1.0	0.9	-0.2	0.7	-0.2	**0.7**
3rd-117		9 / 6	0.1	-1.2	-0.5	-3.2	-1.2	0.9	**1.2**

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: Category B laboratories ordered by lab-codes

COMPULSORY Compounds			Bromide ion	Cyromazine	Fluazifop	Glyphosate	Haloxyfop	Mepiquat-Cl	
MRRL [mg/kg]			2	0.01	0.01	0.03	0.003	0.01	
Assigned Value [mg/kg]			15.356	0.097	0.049	0.903	0.017	0.124	
*CV**			24.0 %	23.5 %	20.3 %	22.6 %	20.4 %	22.9 %	
Lab code SRM13-	NRL-SRM	Analysed / corr. found [1]	z-Scores	z-Scores	z-Scores	z-Scores	z-Scores	z-Scores	AAZ [2]
2		9 / 4	0.0	-3.6[FN]	0.2	-1.2	-0.7	-3.7[FN]	
3		4 / 3		-0.6	-0.2		-0.5		
5	x	7 / 4			-0.2	-2.1	1.7	-1.3	
7	x	4 / 3		0.7	-1.3		-0.3		
17		7 / 4		-0.5	-0.5	-1.3	0.3		
23		6 / 4			0.2	0.5	0.0	0.5	
25		7 / 5	0.9	0.9	0.2		0.5	0.0	**0.5**
28		2 / 1			0.6				
38		5 / 2			-0.1			0.2	
39		1 / 1				0.4			
40		1 / 1				0.8			
41		5 / 3			1.6	1.2	1.4		
42		6 / 4		-0.9	-0.9		-0.7	-0.4	
43		1 / 1				-0.5			
44		2 / 1				4.8			
45		9 / 6	0.1	3.7	-2.2	-0.7	0.7	0.6	**1.3**
47		0 / 0							
51	x	7 / 4	0.5	0.0		0.3		-0.1	
53		6 / 3		0.1	-0.8			-0.9	
57	x	3 / 2		0.3				0.8	
58		1 / 1				-0.2			
59		0 / 0							
62		7 / 4	1.2	-2.2	-0.6		-1.2		
64		2 / 1				-0.7			
66		5 / 2			0.4		1.5	-3.7[FN]	
67		7 / 5	-0.3	-2.4	-1.2	0.8		-0.5	**1**
70		3 / 2				0.3		0.1	
77	x	3 / 2		-0.5				-0.1	
80	x	6 / 4			1.0	6.2	1.9	0.2	
82		3 / 2	0.4	0.0					
84		4 / 3		1.0	1.6		0.0		
90		1 / 0			-3.6[FN]				
94		1 / 1	0.8						
95		1 / 1						-0.1	
96		7 / 5		-0.3	0.7	0.2	-0.1	-0.8	**0.4**
99		2 / 1						-0.3	
100	x	4 / 3	0.2		0.0		0.5		
102		1 / 1	-2.0						
103		1 / 1				55.8			
106		5 / 3		34.8		0.1		-0.8	
107		6 / 4			0.9	0.1	0.7	-1.1	

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

COMPULSORY Compounds			Bromide ion	Cyromazine	Fluazifop	Glyphosate	Haloxyfop	Mepiquat-Cl	
MRRL [mg/kg]			2	0.01	0.01	0.03	0.003	0.01	
Assigned Value [mg/kg]			15.356	0.097	0.049	0.903	0.017	0.124	
*CV**			24.0 %	23.5 %	20.3 %	22.6 %	20.4 %	22.9 %	
Lab code SRM13-	NRL-SRM	Analysed / corr. found [1]	z-Scores	z-Scores	z-Scores	z-Scores	z-Scores	z-Scores	AAZ [2]
110		4 / 2				-1.6		0.6	
111		4 / 2	1.2					-1.1	
113		0 / 0							
114		0 / 0							
3rd-115		6 / 3		-0.4		0.7	-3.3[FN]	0.0	
3rd-116		8 / 5	-0.6	-1.1		-1.3	-2.3	-0.4	**1.1**
3rd-118		6 / 3	-0.2	-0.6	-3.2[FN]	-0.8			
3rd-119		2 / 1			-0.7				
3rd-120		3 / 1				0.0			
3rd-121		1 / 1				0.1			
3rd-122		7 / 5	0.4	2.1		0.0	0.5	4.7	**1.5**

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

4.4.5 Feedback from Laboratories in Case of Poor Results

Like in the previous EUPT-SRMs, 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 ***diquat***, ***phosphine (Test Item)*** and ***phosphine (PH3-Tube)*** that showed an unacceptble uncertainty of their assigned values, in total 821 results for the analytes present in the test items and 5 false positive results were reported by 116 participants. 98 results by 51 laboratories were allocated with $|z| > 2$, and thereof 63 results by 40 laboratories with $|z| \geq 3$ (see **Section Table 4-7, p. 35**). Among EU and EFTA laboratories, $|z| > 2$ was assigned to 33 results by 23 laboratories, and $|z| \geq 3$ to 55 results by 33 laboratories. All these laboratories and those having obtained false positive results were asked to provide a feedback. Overall, 43 laboratories responced to the organisers with (possible) reasons for their poor performance in 86 cases. In 7 cases the real reasons for generating biased results could not be clarified, despite of intensive investigation. The most frequently reported error sources were "lack of experience" (28 cases) and often combined with "matrix effect not properly compensated" (20 cases) and/or "use of inappropriate procedure" (21 cases). The current matrix "soybean flour", a dry matrix with high content on fat (approx. 20 %) and even with starch and biological surfactant lecithin, was surely a difficult matrix and out of the routine analytical scope. Lacking of experience with soy beans, part of the participants applied in the PT

simply the the extraction solvents and procedures which work usually well for fruit and vegetable but not for soybeans. During the investigation a few participants observed that addition of water and soaking enhance the extraction yield (**Appendix 7**). Compared with the previouse EUPT-SRMs, the percentage of "transcription error" was high in this PT (14 cases). Most of the transcription errors occured during results submission, in particular if the submission tool didn't work correctly and one was under stress. Together with other EURLs, the organiser is working on a new, and hopefully more comfortable and stable submission tool. The transcription errors occured also during typing of the concentration of the calibration solution.

Other error sources commonly reported were: "error in concentration of analytical standard/calibration stock" (9 cases) or "procedure not properly conducted/error during sample preparation" (8 cases), "inappropriate calibration " (4 cases), "detection signals strongly interferred by matrix components/strong chromatographic interferences" (each 3 cases), "misunderstanding of the definition of the analyte", "sample weight too small, homogeneity not waranteed", "error in the evaluation/interpretation of measurement data", "technical problems/diffculties with measurement instrumentation" and "contamination" (each 2 cases) as well as "error in the conversion factor", "sample amount not sufficient for conducting confirmation analysis" and "reporting limit higher than the assigned value" (each one case). The responses from the other labratories are pending.

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

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

[2] Regulation (EC) N° 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.

[3] http://www.crl-pesticides.eu/userfiles/file/EurlSRM/EurlSrm_Observations_AcidicPesticides.pdf

[4] Thompson M., Ellison S.L.R. and Wood R., The International Harmonized Protocol for the Proficiency Testing of Analytical Chemistry Laboratories (IUPAC Technical Report). Pure Appl. Chem., Vol. 78, No. 1, pp. 145 – 196, 2006

[5] http://quppe.eu/

[6] ISO 13528:2015: Statistical methods for use in proficiency testing by interlaboratory comparisons.

7. APPENDICES

Appendix 1 List of Laboratories Registered to Participate in the EUPT-SRM13

(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 (LSI-PLMA)	Innsbruck	x	Yes
Belgium	BE	WIV-ISP (Belgian 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	Euroinspekt - Croatiakontrola	Zagreb		Yes
Croatia	HR	Inspecto d.o.o. Laboratorij	Osijek (Industrijska zona Nemetin)		Yes
Croatia	HR	NZJZ dr.A.Štampar	Zagreb		Yes
Cyprus	CY	FP lab of S.G.L	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	VSCHT Praha, Analyza potravin	Praha 6		Yes
Denmark	DK	Fodevarestyrelsen	Ringsted	x	Yes
Estonia	EE	PMK, JSL	Saku		Yes
Estonia	EE	Tartu Laboratory of Health Boa	Tartu	x	Yes
Finland	FI	Finnish Customs Laboratory	Espoo	x	Yes
Finland	FI	Finnish Food Safety Authority	Helsinki	x	Yes
France	FR	ANSES-LSAL	Maisons-Alfort Cedex	x	Yes
France	FR	CAMP 66	perpignan		Yes
France	FR	CAPINOV	Landerneau		Yes
France	FR	CERECO	GARONS		Yes
France	FR	GIRPA	BEAUCOUZE		Yes
France	FR	INOVALYS	Le Mans		Yes
France	FR	Laboratoire du SCL	Montpellier		Yes
France	FR	SCL Laboratoire de Paris	Massy Cedex		Yes
France	BE	PHYTOCONTROL	NIMES		Yes
Germany	BE	LUFA-ITL GmbH	Kiel		Yes
Germany	DE	BfUL FB 42	Nossen		Yes
Germany	DE	Bundesamt für Verbraucherschutz und Lebensmittelsicherheit	Berlin-Marienfelde	x	Yes
Germany	DE	CVUA Münsterland Emscher-Lippe	Münster		Yes
Germany	DE	CVUA RRW	Krefeld		Yes
Germany	DE	Eurofins SOFIA GmbH	Berlin		Yes
Germany	DE	Institut für Hygiene und Umwel	Hamburg		No
Germany	DE	Kwalis Qualitätsforschung Fulda	Dipperz		Yes
Germany	DE	Labor Friedle GmbH	Tegernheim		Yes
Germany	DE	Landesamt für Landwirtschaft, Lebensmittelsicherheit und Fischerei	Rostock		Yes
Germany	DE	Landesamt für Verbraucherschut	Halle/Saale		Yes
Germany	DE	Landeslabor Berlin-Brandenburg	Potsdam		Yes
Germany	DE	Landesuntersuchungsamt - ILC Speyer	Speyer		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	Landesuntersuchungsanstalt für das Gesundheits- und Veterinärwesen Sachsen Reichenbachstr. 71/73 01217 Dresden Germany	Dresden		Yes
Germany	DE	LAVES Futtermittelinstitut Stade	Stade		Yes
Germany	DE	LAVES, LVI Oldenburg	Oldenburg		Yes
Germany	DE	LGL	Erlangen		Yes
Germany	DE	LLG Halle	Halle/Saale		Yes
Germany	DE	LTZ Augustenberg	Karlsruhe		Yes
Germany	DE	LUFA Speyer	Speyer		Yes
Germany	DE	Stadt Duesseldorf - Abteilung	Duesseldorf		Yes
Germany	DE	State Laboratory Schleswig-Holstein	Neumünster		Yes
Germany	DE; MT	Eurofins Dr. Specht Laboratorien GmbH	Hamburg		Yes
Germany	LT	GALAB Laboratories GmbH	Hamburg		Yes
Greece	GR	AGROLAB-RDS	Thessaloniki		Yes
Greece	GR	Benaki Pesticide Residue Labor	Kifissia	x	Yes
Greece	GR	General Chemical State Laboratory, A Chemical Division, Pesticide Residues Laboratory	Athens	x	Yes
Hungary	HU	Food Chain Safety Centre Non-profit Ltd. Pesticide Residue Analytical Laboratory, Hódmezővásárhely	Hódmezovásárhely		Yes
Hungary	HU	Food Chain Safety Centre Nonprofit Ltd. Pesticide Residue Analytical Laboratory, Miskolc	Miskolc		Yes
Hungary	HU	Food Chain Safety Centre Non-profit Ltd., Pesticide Residue Analytical Laboratory, Szolnok	Szolnok		Yes
Hungary	HU	National Food Chain Safety Office, Directorate of Plant Protection, Soil Conservation and Agri-environment - Pesticide Analytical Laboratory, Velence	Velence	x	Yes
Hungary	HU	Wessling Hungary Ltd. Food Testing Laboratory	Budapest		Yes
Ireland	IE	PCL, Backweston Lab Complex	Co. Kildare	x	Yes
Italy	IT	APPA Bolzano	Bolzano		Yes
Italy	IT	ARPA Puglia	Bari		Yes
Italy	IT	ARPAE Ferrara Laboratorio Tema	Ferrara		Yes
Italy	IT	ARPAV Verona	Verona		Yes
Italy	IT	Contaminanti Ambientali	Perugia		Yes
Italy	IT	Istituto Superiore di Sanità	Rome	x	No
Italy	IT	Istituto Zooprofilattico Sperimentale Abruzzo e Molise	Teramo		Yes
Italy	IT	IZS Sardegna - Lab Chimica Ambientale e Tossicologia	Sassari		Yes
Italy	IT	IZS Sicilia - Pesticide Lab	Palermo		Yes
Italy	IT	USL Toscana centro - Lab. Sani	Firenze		Yes
Italy	IT; MT	Istituto Zooprofilattico Sperimentale Lombardia ed Emilia Romagna	Brescia		Yes
Italy	IT	IZSLT	Roma		Yes
Latvia	LV	Research Institute BIOR	Riga	x	Yes
Lithuania	LT	NMVRVI	Vilnius	x	Yes
Luxembourg	LU	LNS-ALI	Dudelange	x	Yes
Norway	NO	NIBIO, Biotechnology and Plant Health, Pesticides and Natural Products Chemistry	Aas		Yes
Poland	PL	IPP-NRI, Bialystok	Bialystok		Yes
Poland	PL	Wojewódzka Stacja Sanitarno-Epidemiologiczna w Opolu	Opole		Yes
Poland	PL	WSSE w Warszawie	Warszawa	x	Yes
Poland	PO	J.S. Hamilton Poland S.A.	Gdynia		Yes
Poland	PO	UO-Technologia	Grójec		Yes
Portugal	PT	Laboratório Regional de Veterinária e Segurança Alimentar	Funchal Madeira Island	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
Slovakia	SK	Veterinary and Food Institute in Bratislava	Bratislava	x	Yes
Slovenija	SI	National Laboratory for Health, Environment and Food - Maribor (location Ljubljana)	Ljubljana		Yes
Slovenija	SI	NLZOH-Maribor	Maribor	x	Yes
Spain	ES	AINIA	PATERNA, VALENCIA		Yes
Spain	ES	Analytica Alimentaria	Almeria		Yes
Spain	ES	CNA (AECOSAN)	Majadahonda (Madrid)	x	Yes
Spain	ES	CNTA	San Adrián (Navarra)		Yes
Spain	ES	DIRECCION TERRITORIAL COMERCIO	Valencia		No
Spain	ES	EUROFINS SICA AGRIQ, S.L.	VÍCAR (ALMERIA)		Yes
Spain	ES	Lab Agroalim. de Extremadura	Cáceres		Yes
Spain	ES	Lab Agroamb Zaragoza	Zaragoza		Yes
Spain	ES	Lab. Agroalimentario y de Sani	El Palmar-Murcia		No
Spain	ES	Laboratori Agència de Salut Pública de Barcelona	Barcelona		Yes
Spain	ES	Laboratorio Agrario	Abegondo. A Coruña		No
Spain	ES	Laboratorio Agrario Regional,	Burgos		Yes
Spain	ES	Laboratorio Arbitral Agroalimentario	Madrid	x	Yes
Spain	ES	Laboratorio SOIVRE Tenerife	Santa Cruz de Tenerife. Canay Island		Yes
Spain	ES	LABORATORIOS ECOSUR, S.A.	Lorquí (Murcia)		Yes
Spain	ES	LPSVJAEN	Mengibar (Jaén)		Yes
Spain	ES; MT	LAGV	Burjassot-Valencia		Yes
Sweden	SE	Eurofins Food&Feed Sweden AB	Lidkoping		Yes
Sweden	SE	Livsmedelsverket, Dep of Chemi	Uppsala	x	Yes
Switzerland	CH	Kantonales Labor Zürich	Zurich		Yes
Switzerland	CH	Kantonales Labor, Gesundheitsdepartement Basel-Stadt	Basel		Yes
Switzerland	CH	LABORATORIUM DER URKANTONE	BRUNNEN		Yes
The Netherlands	AL; BE; NL	Eurofins Lab Zeeuws-Vlaanderen	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
The Netherlands	NL	NVWA - NRL for Pesticide Residues in Food and Feed	Wageningen	x	Yes
The Netherlands	NL	RIKILT - Wageningen University	Wageningen		Yes
United Kingdom	UK; MT	Fera Science Ltd	York	x	Yes
United Kingdom	UK	Concept Life Sciences Ltd	Bar Hill		Yes
United Kingdom	UK	Eurofins Food Testing UK	Wolverhampton		Yes

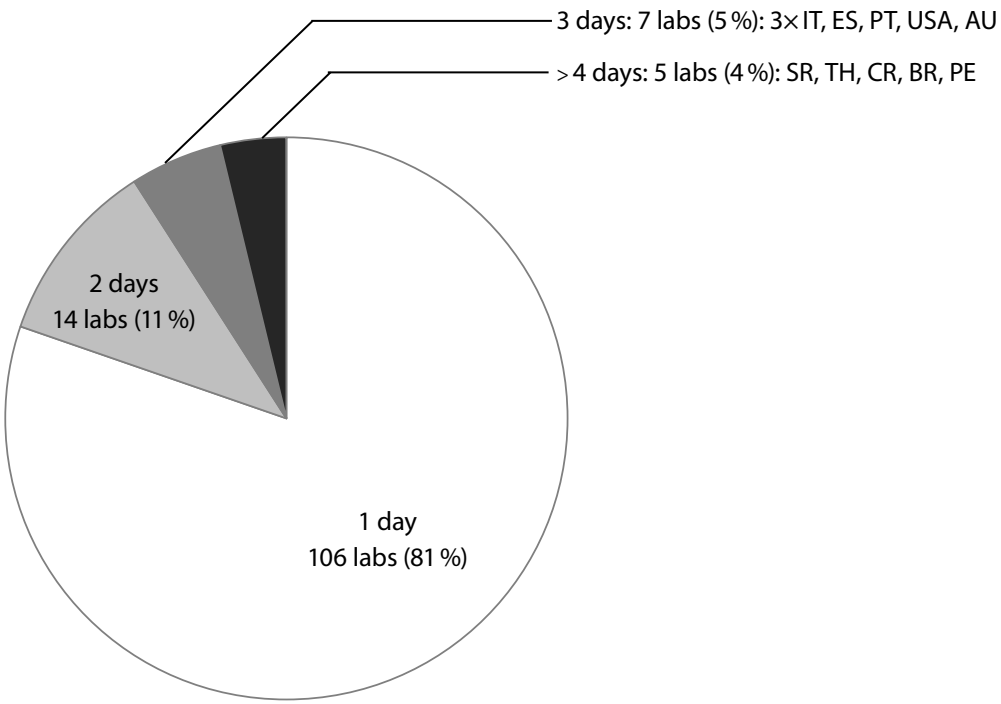
* only for EU-Member States

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

Country	Institution	City	Reported results
Belarus	Food testing laboratory/BelGIM	Minsk	Yes
Brazil	LANAGRO/MG	Pedro Leopoldo	Yes
Costa Rica	LABORATORIO DE ANÁLISIS DE RESIDUOS DE AGROQUÍMICOS	San José	Yes
Peru	Unidad del Centro de Control de Insumos y Residuos Tóxicos - SENASA	Lima	Yes
Serbia	GZZJZ Belgrade	Belgrade	Yes
Serbia	SP LABORATORIJA A.D.	BECEJ	Yes
Singapore	Agri-Food and Veterinary Authority Veterinary Public Health Laboratory Pesticide Residue Section	Singapore	Yes
Thailand	Central Laboratory	Bangkok	Yes

Appendix 2 Shipment Evaluation

Compilation of shipment duration



Appendix 3 Data of Homogeneity Test

COMPULSORY COMPOUNDS								
Bromide ion			**Cyromazine**			**Fluazifop**		
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. 024	14.2	15.3	No. 024	0.098	0.116	No. 024	0.053	0.050
No. 036	14.3	16.5	No. 036	0.112	0.121	No. 036	0.050	0.051
No. 047	14.8	14.0	No. 047	0.113	0.106	No. 047	0.045	0.048
No. 066	15.9	14.6	No. 066	0.099	0.109	No. 066	0.054	0.049
No. 077	14.4	14.9	No. 077	0.108	0.108	No. 077	0.052	0.053
No. 090	15.1	15.7	No. 090	0.105	0.108	No. 090	0.050	0.047
No. 110	12.2	13.0	No. 110	0.101	0.094	No. 110	0.048	0.048
No. 122	13.7	15.3	No. 122	0.100	0.104	No. 122	0.049	0.051
No. 142	14.2	13.6	No. 142	0.113	0.106	No. 142	0.049	0.047
No. 167	13.9	16.8	No. 167	0.105	0.116	No. 167	0.049	0.048
mean / AV*	14.6 / 15.4		mean / AV*	0.107 / 0.097		mean / AV*	0.049 / 0.049	

Glyphosate			**Haloxyfop**			**Mepiquat-Cl**		
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. 024	0.840	0.938	No. 024	0.017	0.017	No. 024	0.121	0.136
No. 036	0.923	0.973	No. 036	0.016	0.018	No. 036	0.130	0.135
No. 047	1.017	0.973	No. 047	0.016	0.015	No. 047	0.131	0.124
No. 066	0.885	0.891	No. 066	0.017	0.016	No. 066	0.115	0.128
No. 077	0.914	0.999	No. 077	0.017	0.017	No. 077	0.128	0.134
No. 090	0.866	0.936	No. 090	0.016	0.016	No. 090	0.125	0.132
No. 110	0.842	0.814	No. 110	0.017	0.016	No. 110	0.105	0.113
No. 122	0.825	1.029	No. 122	0.016	0.017	No. 122	0.121	0.125
No. 142	0.969	0.938	No. 142	0.016	0.015	No. 142	0.132	0.128
No. 167	0.922	0.976	No. 167	0.016	0.016	No. 167	0.125	0.141
mean / AV*	0.924 / 0.903		mean / AV*	0.016 / 0.017		mean / AV*	0.126 / 0.124	

* 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											
2,4-DB			Diquat			Glufosinate			MPP		
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. 024	0.187	0.192	No. 024	1.59	1.24	No. 024	0.195	0.211	No. 024	0.164	0.189
No. 036	0.181	0.187	No. 036	1.52	1.64	No. 036	0.211	0.255	No. 036	0.187	0.197
No. 047	0.163	0.167	No. 047	1.29	1.35	No. 047	0.181	0.192	No. 047	0.179	0.178
No. 066	0.194	0.164	No. 066	1.46	1.46	No. 066	0.212	0.202	No. 066	0.171	0.177
No. 077	0.183	0.185	No. 077	1.42	1.61	No. 077	0.216	0.174	No. 077	0.172	0.188
No. 090	0.179	0.169	No. 090	1.39	1.41	No. 090	0.213	0.207	No. 090	0.174	0.179
No. 110	0.172	0.179	No. 110	1.31	1.44	No. 110	0.160	0.167	No. 110	0.155	0.151
No. 122	0.173	0.184	No. 122	1.67	1.57	No. 122	0.149	0.180	No. 122	0.157	0.175
No. 142	0.166	0.180	No. 142	1.47	1.29	No. 142	0.213	0.184	No. 142	0.179	0.173
No. 167	0.165	0.176	No. 167	1.52	1.49	No. 167	0.182	0.237	No. 167	0.171	0.192
mean / AV*	0.177 / 0.183		mean / AV*	1.46 / *1.70*‡		mean / AV*	0.197 / 0.192		mean / AV*	0.489 / 0.188	

N-Acetyl-Glyphosate			Perchlorate			Phosphonic acid			Quizalofop		
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. 024	0.696	0.769	No. 024	0.099	0.109	No. 024	1.874	2.082	No. 024	0.061	0.061
No. 036	0.832	0.880	No. 036	0.104	0.117	No. 036	2.161	2.197	No. 036	0.056	0.062
No. 047	0.772	0.817	No. 047	0.108	0.100	No. 047	2.079	2.025	No. 047	0.057	0.056
No. 066	0.725	0.792	No. 066	0.103	0.106	No. 066	1.931	2.011	No. 066	0.063	0.060
No. 077	0.739	0.821	No. 077	0.096	0.111	No. 077	1.902	2.094	No. 077	0.062	0.061
No. 090	0.739	0.750	No. 090	0.104	0.106	No. 090	1.947	2.055	No. 090	0.060	0.054
No. 110	0.654	0.633	No. 110	0.095	0.092	No. 110	1.774	1.668	No. 110	0.056	0.057
No. 122	0.666	0.779	No. 122	0.100	0.100	No. 122	1.866	2.013	No. 122	0.056	0.059
No. 142	0.794	0.762	No. 142	0.110	0.104	No. 142	2.086	2.011	No. 142	0.059	0.055
No. 167	0.716	0.835	No. 167	0.095	0.106	No. 167	1.882	2.209	No. 167	0.057	0.055
mean / AV*	0.759 / 0.835		mean / AV*	0.103 / 0.100		mean / AV*	1.99 / 1.86		mean / AV*	0.058 / 0.052	

OPTIONAL COMPOUND (PHOSPHINE)					
Phsophine (Test Item)			Phosphine (PH3-Tube)		
Sample No.	Portion 1 [mg/kg]	Portion 2 [mg/kg]	Sample No.	Portion 1 [mg/kg]	Portion 2 [mg/kg]
No. 001	0.187	0.189	No. 001	0.068	0.082
No. 006	0.170	0.191	No. 005	0.059	0.068
No. 013	0.178	0.185	No. 008	0.077	0.060
No. 016	0.175	0.192	No. 011	0.079	0.064
No. 031	0.167	0.183	No. 017	0.064	0.078
No. 100	0.184	0.180	No. 021	0.063	0.067
No. 114	0.177	0.197	No. 026	0.073	0.059
No. 140	0.170	0.185	No. 041	0.057	0.060
No. 154	0.176	0.192	No. 048	0.062	0.069
No. 172	0.186	0.191	No. 051	0.073	0.076
mean / AV*	0.183 / –		mean / AV*	0.068 / –	

* 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

‡: statistically uncertain

Appendix 4 Data of Stability Test / Compulsory Compounds

COMPULSORY COMPOUNDS												
Bormide Ion							**Cyromazine**					
AV [mg/kg]	15.4						AV [mg/kg]	0.097				
Date	03.05.2018		22.05.2018		18.06.2018		Date	03.05.2018		22.05.2018		18.06.2018
Sample	[mg/kg]		[mg/kg]		[mg/kg]		Sample	[mg/kg]		[mg/kg]		[mg/kg]
No. 077	14.4	14.9	13.3	13.8	14.3	13.9	No. 077	0.108	0.108	0.099	0.100	0.116	0.108
No. 110	12.2	13.0	13.8	14.0	14.1	13.8	No. 110	0.101	0.094	0.108	0.104	0.109	0.108
No. 142	14.2	13.6	14.0	15.5	14.6	13.9	No. 142	0.113	0.106	0.103	0.113	0.109	0.114
Mean [mg/kg]	**13.73**		**14.05**		**14.11**		**Mean** [mg/kg]	**0.105**		**0.105**		**0.111**	
RSD* [%]	**7.5 %**		**4.3 %**		**0.9 %**		**RSD*** [%]	**6.2 %**		**4.2 %**		**1.5 %**	
Diviation [%] (ref. 1st Anaylsis)	**—**		**2.4 %**		**2.8 %**			**—**		**-0.4 %**		**5.3 %**	

Fluazifop							**Glyphosate**					
AV [mg/kg]	0.049						AV [mg/kg]	0.903				
Date	25.04.2018		17.05.2018		07.06.2018		Date	03.05.2018		22.05.2018		18.06.2018
Sample	[mg/kg]		[mg/kg]		[mg/kg]		Sample	[mg/kg]		[mg/kg]		[mg/kg]
No. 077	0.052	0.053	0.051	0.046	0.048	0.051	No. 077	0.914	0.999	0.938	0.942	0.998	0.909
No. 110	0.048	0.048	0.050	0.051	0.052	0.047	No. 110	0.842	0.814	1.017	0.905	0.919	0.819
No. 142	0.049	0.047	0.051	0.050	0.047	0.047	No. 142	0.969	0.938	0.951	1.030	0.922	1.031
Mean [mg/kg]	**0.050**		**0.050**		**0.049**		**Mean** [mg/kg]	**0.913**		**0.964**		**0.933**	
RSD* [%]	**5.5 %**		**2.6 %**		**3.0 %**		**RSD*** [%]	**8.0 %**		**2.7 %**		**6.1 %**	
Diviation [%] (ref. 1st Anaylsis)	**—**		**0.4 %**		**-1.7 %**			**—**		**5.6 %**		**2.2 %**	

Haloxyfop							**Mepiquat-Cl**					
AV [mg/kg]	0.017						AV [mg/kg]	0.124				
Date	25.04.2018		17.05.2018		07.06.2018		Date	03.05.2018		22.05.2018		18.06.2018
Sample	[mg/kg]		[mg/kg]		[mg/kg]		Sample	[mg/kg]		[mg/kg]		[mg/kg]
No. 077	0.017	0.017	0.016	0.019	0.018	0.015	No. 077	0.128	0.134	0.121	0.123	0.139	0.128
No. 110	0.017	0.016	0.015	0.016	0.017	0.018	No. 110	0.105	0.113	0.140	0.128	0.123	0.135
No. 142	0.016	0.015	0.016	0.015	0.019	0.017	No. 142	0.132	0.128	0.126	0.131	0.129	0.133
Mean [mg/kg]	**0.016**		**0.016**		**0.017**		**Mean** [mg/kg]	**0.123**		**0.128**		**0.131**	
RSD* [%]	**3.7 %**		**6.6 %**		**4.4 %**		**RSD*** [%]	**10.0 %**		**4.7 %**		**1.7 %**	
Diviation [%] (ref. 1st Anaylsis)	**—**		**0.1 %**		**7.1 %**			**—**		**3.8 %**		**6.2 %**	

***** RSD = relative standard diviation

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

OPTIONAL COMPOUNDS													
2,4-DB							**Diquat**						
AV [mg/kg]	0.183						AV [mg/kg]	*1.70‡*					
Date	25.04.2018		17.05.2018		07.06.2018		Date	03.05.2018		22.05.2018		18.06.2018	
Sample	[mg/kg]		[mg/kg]		[mg/kg]		Sample	[mg/kg]		[mg/kg]		[mg/kg]	
No. 077	0.181	0.187	0.188	0.169	0.157	0.170	No. 077	1.42	1.61	1.24	1.45	1.63	1.23
No. 110	0.172	0.179	0.190	0.184	0.161	0.175	No. 110	1.31	1.44	1.43	1.27	1.48	1.45
No. 142	0.166	0.180	0.184	0.182	0.165	0.162	No. 142	1.47	1.29	1.42	1.35	1.34	1.40
Mean [mg/kg]	**0.177**		**0.183**		**0.165**		**Mean** [mg/kg]	**1.42**		**1.36**		**1.42**	
RSD* [%]	**3.3 %**		**2.3 %**		**1.6 %**		**RSD*** [%]	**5.7 %**		**1.5 %**		**3.5 %**	
Diviation [%] (ref. 1st Anaylsis)	**—**		**3.1 %**		**-7.0 %**			**—**		**-4.5 %**		**-0.1 %**	

Glufosinate							**MPP**						
AV [mg/kg]	0.192						AV [mg/kg]	0.188					
Date	03.05.2018		22.05.2018		18.06.2018		Date	03.05.2018		22.05.2018		18.06.2018	
Sample	[mg/kg]		[mg/kg]		[mg/kg]		Sample	[mg/kg]		[mg/kg]		[mg/kg]	
No. 077	0.216	0.174	0.192	0.201	0.202	0.218	No. 077	0.172	0.188	0.163	0.180	0.175	0.171
No. 110	0.160	0.167	0.166	0.174	0.175	0.159	No. 110	0.155	0.151	0.171	0.174	0.164	0.154
No. 142	0.213	0.184	0.191	0.176	0.149	0.211	No. 142	0.179	0.173	0.172	0.185	0.155	0.172
Mean [mg/kg]	**0.186**		**0.183**		**0.186**		**Mean** [mg/kg]	**0.170**		**0.174**		**0.165**	
RSD* [%]	**10.4 %**		**7.2 %**		**11.9 %**		**RSD*** [%]	**8.7 %**		**2.2 %**		**4.3 %**	
Diviation [%] (ref. 1st Anaylsis)	**—**		**-1.3 %**		**0.0 %**			**—**		**2.7 %**		**-2.6 %**	

N-Acetyl-Glyphosate							**Perchlorate**						
AV [mg/kg]	0.835						AV [mg/kg]	0.100					
Date	03.05.2018		22.05.2018		18.06.2018		Date	03.05.2018		22.05.2018		18.06.2018	
Sample	[mg/kg]		[mg/kg]		[mg/kg]		Sample	[mg/kg]		[mg/kg]		[mg/kg]	
No. 077	0.739	0.821	0.705	0.798	0.823	0.800	No. 077	0.096	0.111	0.094	0.097	0.109	0.105
No. 110	0.654	0.633	0.773	0.771	0.735	0.659	No. 110	0.095	0.092	0.106	0.100	0.109	0.105
No. 142	0.794	0.762	0.753	0.840	0.648	0.785	No. 142	0.110	0.104	0.101	0.107	0.109	0.110
Mean [mg/kg]	**0.734**		**0.773**		**0.742**		**Mean** [mg/kg]	**0.101**		**0.101**		**0.108**	
RSD* [%]	**10.6 %**		**2.9 %**		**8.3 %**		**RSD*** [%]	**7.0 %**		**4.8 %**		**1.3 %**	
Diviation [%] (ref. 1st Anaylsis)	**—**		**5.4 %**		**1.1 %**			**—**		**-0.4 %**		**6.5 %**	

Phosphonic acid							**Quizalofop**						
AV [mg/kg]	1.86						AV [mg/kg]	0.052					
Date	03.05.2018		22.05.2018		18.06.2018		Date	25.04.2018		17.05.2018		07.06.2018	
Sample	[mg/kg]		[mg/kg]		[mg/kg]		Sample	[mg/kg]		[mg/kg]		[mg/kg]	
No. 077	1.90	2.09	1.84	1.94	2.09	1.94	No. 077	0.062	0.061	0.064	0.060	0.053	0.057
No. 110	1.77	1.67	2.01	1.97	1.94	1.81	No. 110	0.056	0.057	0.057	0.057	0.056	0.063
No. 142	2.09	2.01	1.90	2.09	1.86	1.99	No. 142	0.059	0.055	0.056	0.057	0.059	0.058
Mean [mg/kg]	**1.92**		**1.96**		**1.94**		**Mean** [mg/kg]	**0.058**		**0.059**		**0.058**	
RSD* [%]	**9.2 %**		**3.1 %**		**3.6 %**		**RSD*** [%]	**4.7 %**		**5.4 %**		**4.1 %**	
Diviation [%] (ref. 1st Anaylsis)	**—**		**1.9 %**		**0.9 %**			**—**		**0.3 %**		**-1.2 %**	

***** RSD = relative standard diviation; ‡: statistically uncertain

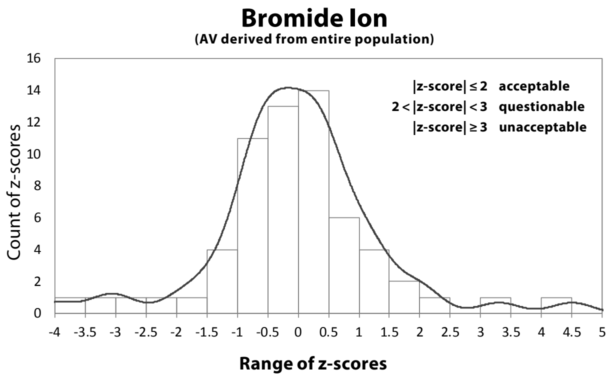
Appendix 4 (cont.): Data of Stability Test / Optional Compounds

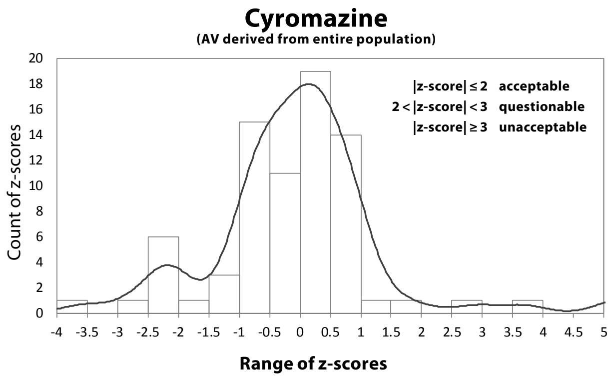
OPTIONAL COMPOUNDS (PHOSPHINE)												
Phosphine (Test Item)							**Phosphine (PH3-Tube)**					
AV [mg/kg]	–						AV [mg/kg]	–				
Date	20.06.2018		19.07.2018		24.09.2018		Date	28.05.2018		19.06.2018		18.07.2018	
Sample	[mg/kg]		[mg/kg]		[mg/kg]		Sample	[mg/kg]		[mg/kg]		[mg/kg]	
No. 006	0.170	0.191	0.147	0.156	0.194	0.182	No. 005	0.059	0.068	0.073	0.065	0.075	0.076
No. 016	0.175	0.192	0.149	0.150	0.184	0.189	No. 017	0.064	0.078	0.064	0.067	0.090	0.083
No. 140	0.170	0.185	0.142	0.152	0.194	0.186	No. 026	0.073	0.059	0.075	0.072	0.082	0.078
Mean [mg/kg]	**0.180**		**0.149**		**0.188**		**Mean** [mg/kg]	**0.067**		**0.069**		**0.080**	
RSD* [%]	**1.69 %**		**1.58 %**		**0.90 %**		**RSD*** [%]	**5.61 %**		**6.11 %**		**6.73 %**	
Diviation [%] (ref. 1st Anaylsis)	**—**		**-17.29 %**		**4.25 %**		**Diviation** [%] (ref. 1st Anaylsis)	**—**		**3.8 %**		**20.0 %**	

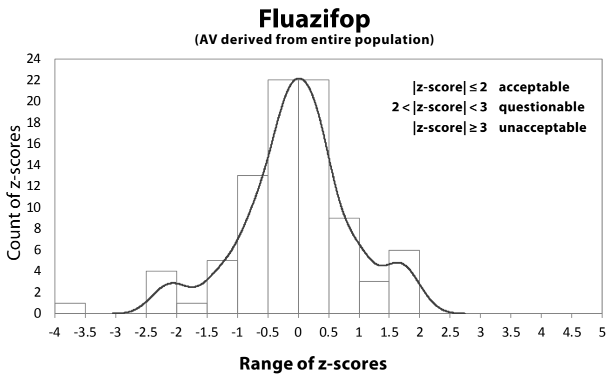
***** RSD = relative standard diviation

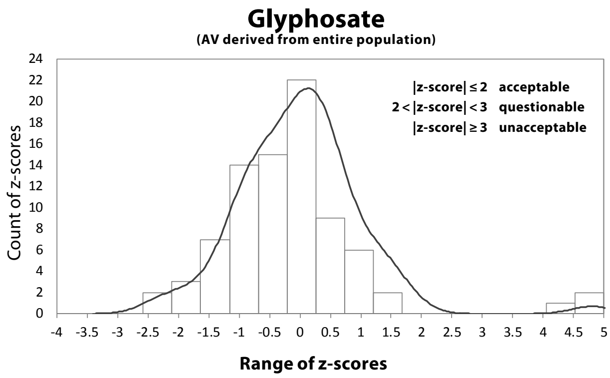
Appendix 5 Histograms and Kernel Density Estimates of z-score* Distributions
(Results from EU and EFTA Laboratories only)

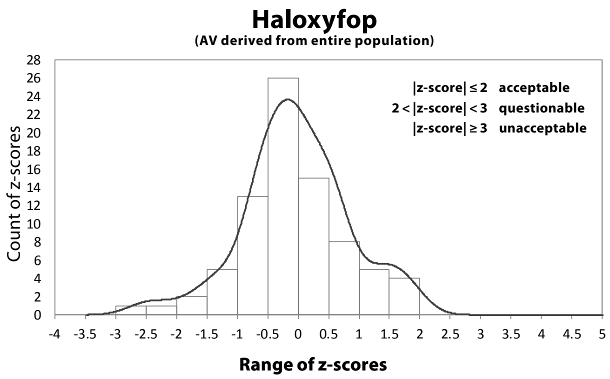
Compulsory Compounds

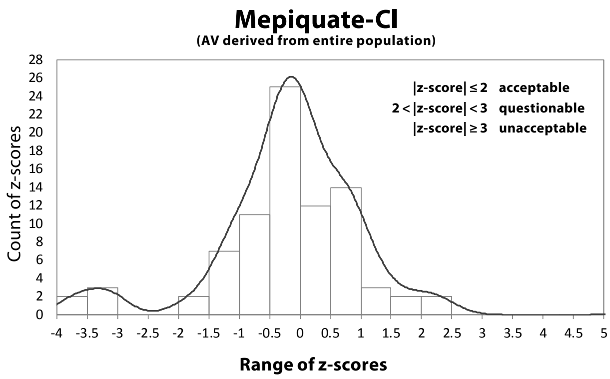












|z-score| ≤ 2 acceptable
2 < |z-score| < 3 questionable
|z-score| ≥ 3 unacceptable

Count of z-scores

Range of z-scores

|z-score| ≤ 2 acceptable
2 < |z-score| < 3 questionable
|z-score| ≥ 3 unacceptable

Count of z-scores

Range of z-scores

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

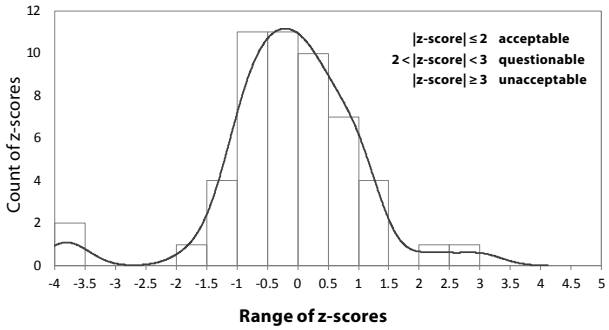
A5 HISTOGRAMM

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

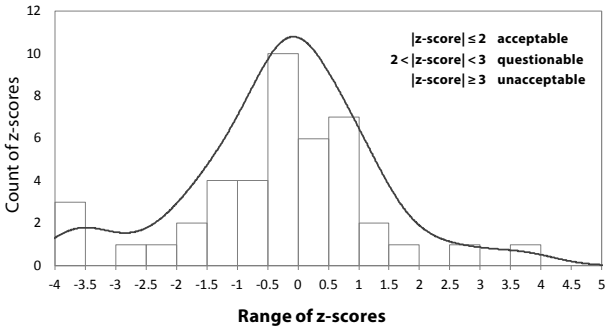
(Results from EU and EFTA Laboratories only)

Optional Compounds

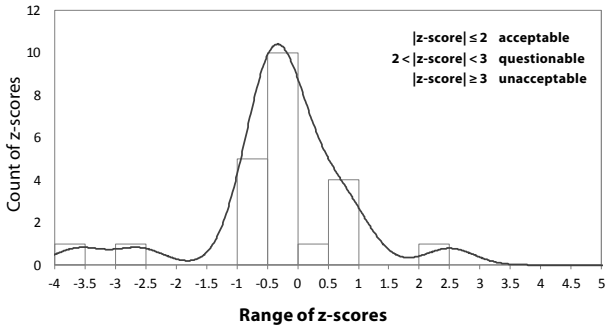
2,4-DB
(AV derived from entire population)



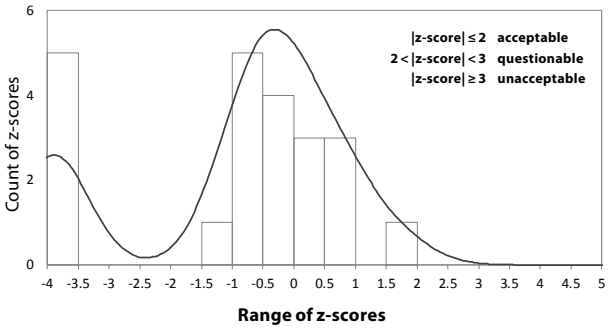
Glufosinate
(AV derived from entire population)



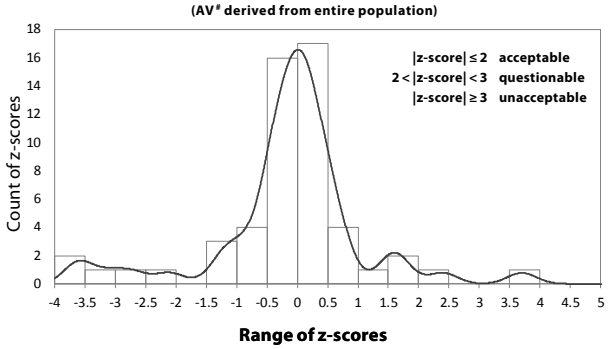
MPP
(AV derived from entire population)



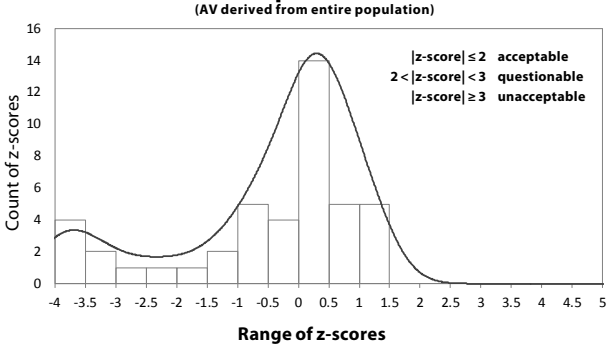
N-Acetyl-Glyphosate
(AV derived from entire population)



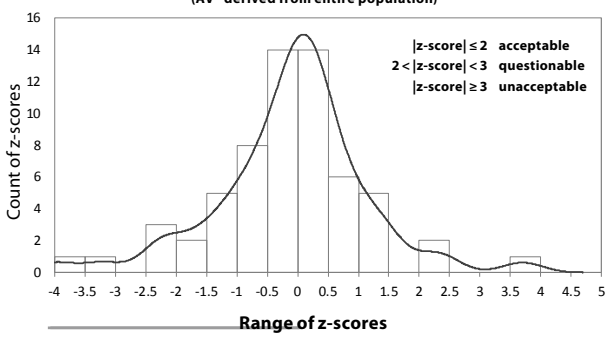
Perchlorate
(AV# derived from entire population)



Phosphonic acid
(AV derived from entire population)

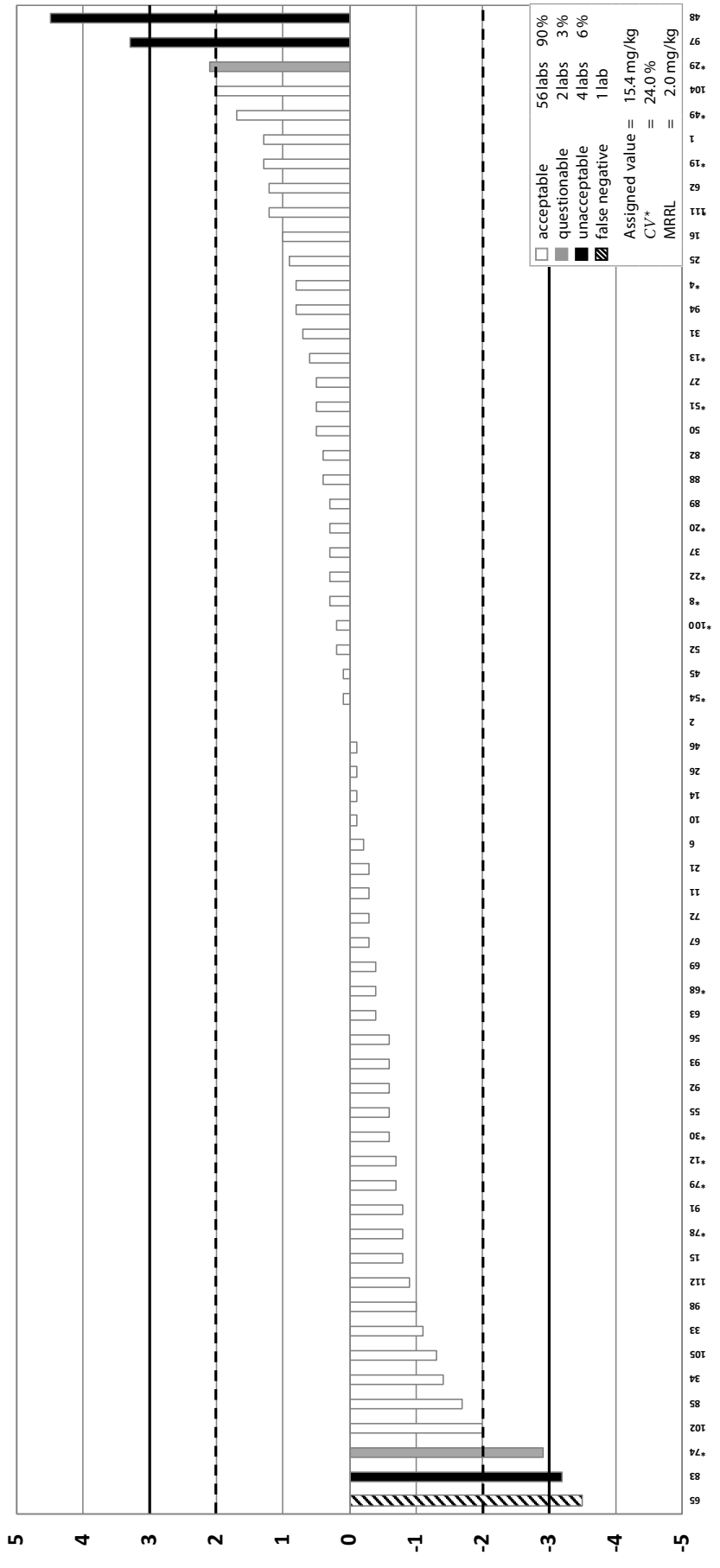


Quizalofop
(AV# derived from entire population)

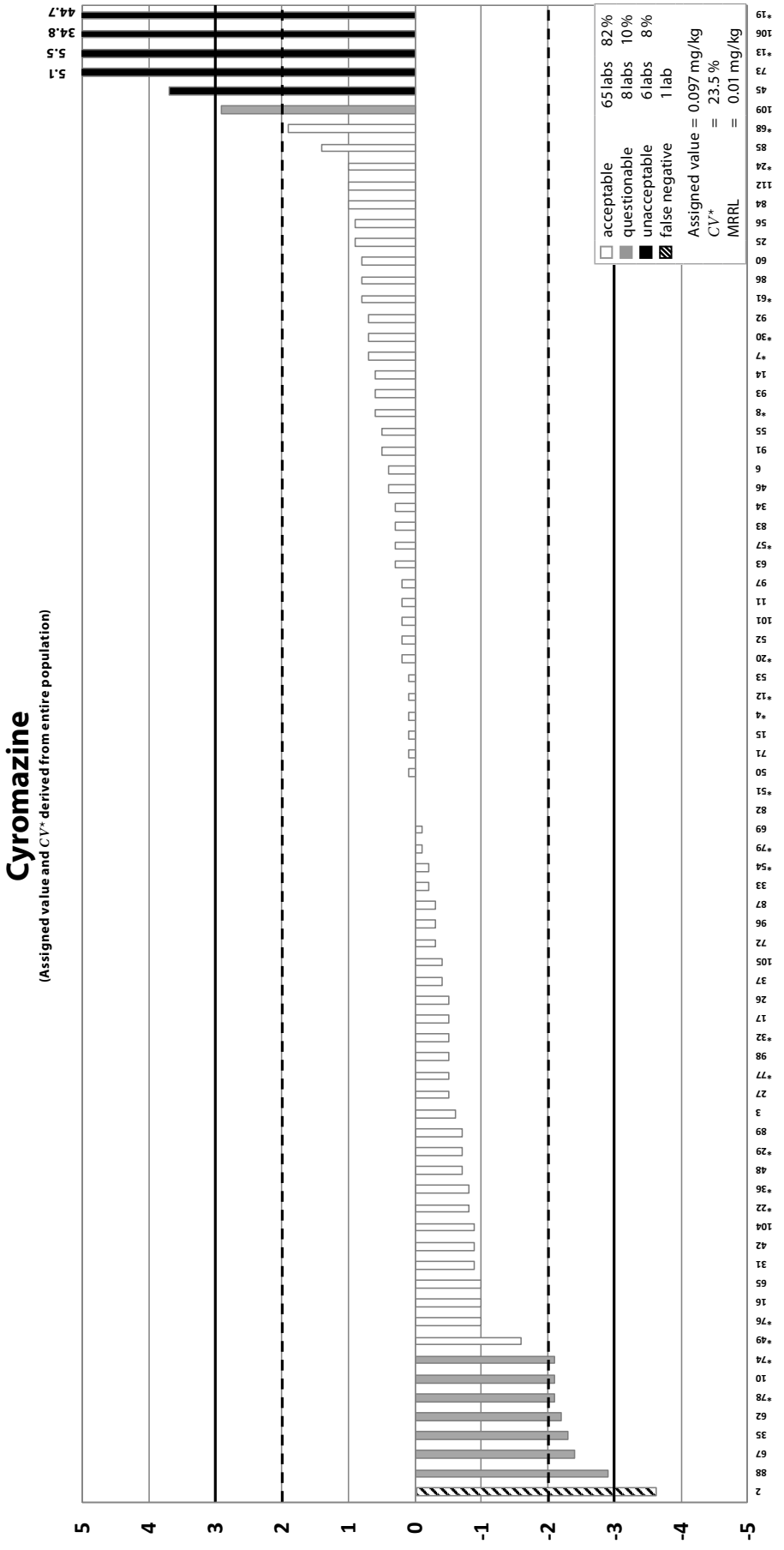


***** Cut-off at z-score = 5;
# excluding diquat and phosphine due to high uncertainty of their assigned values

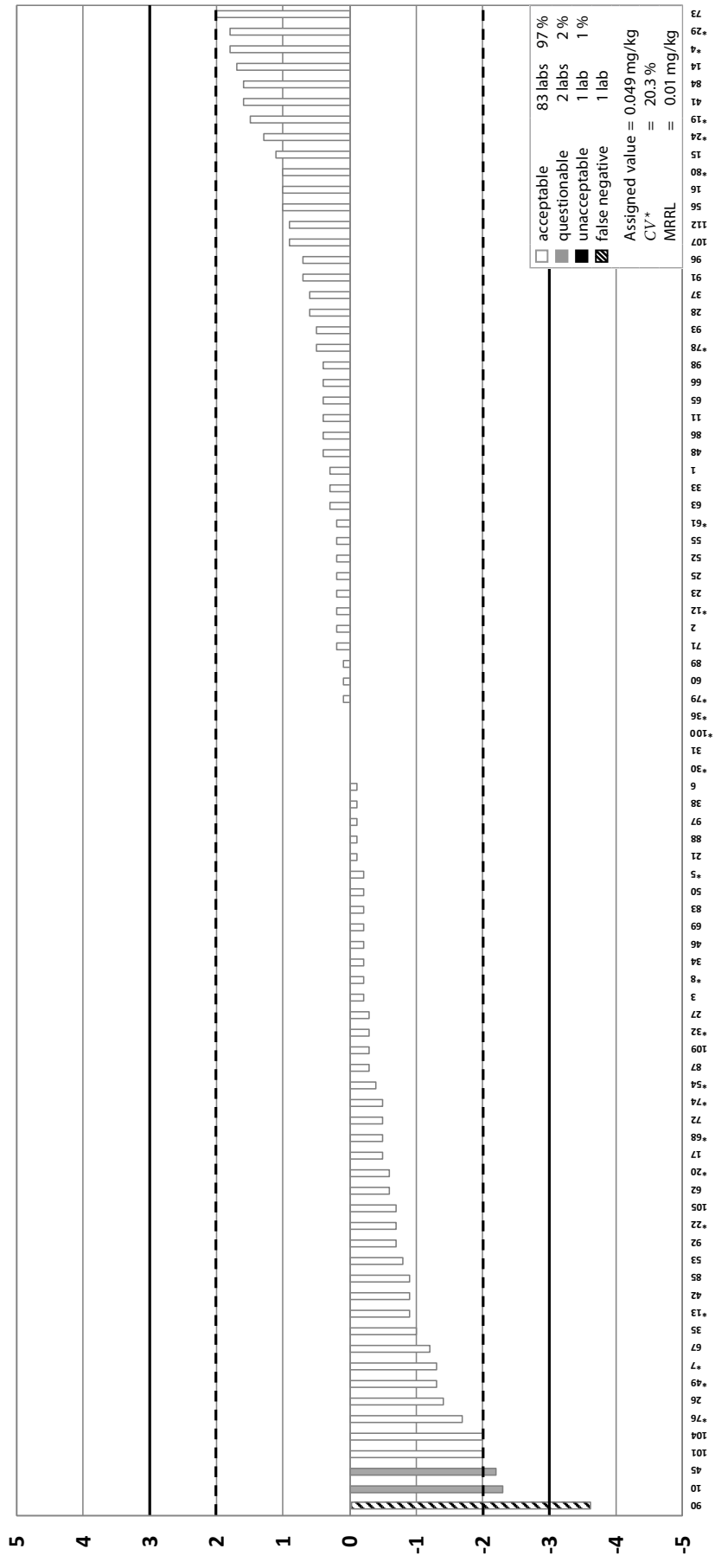
Appendix 6 Graphic Presentation of z-Scores: Compulsory Compounds(Results from EU and EFTA Laboratories only, * = NRL)



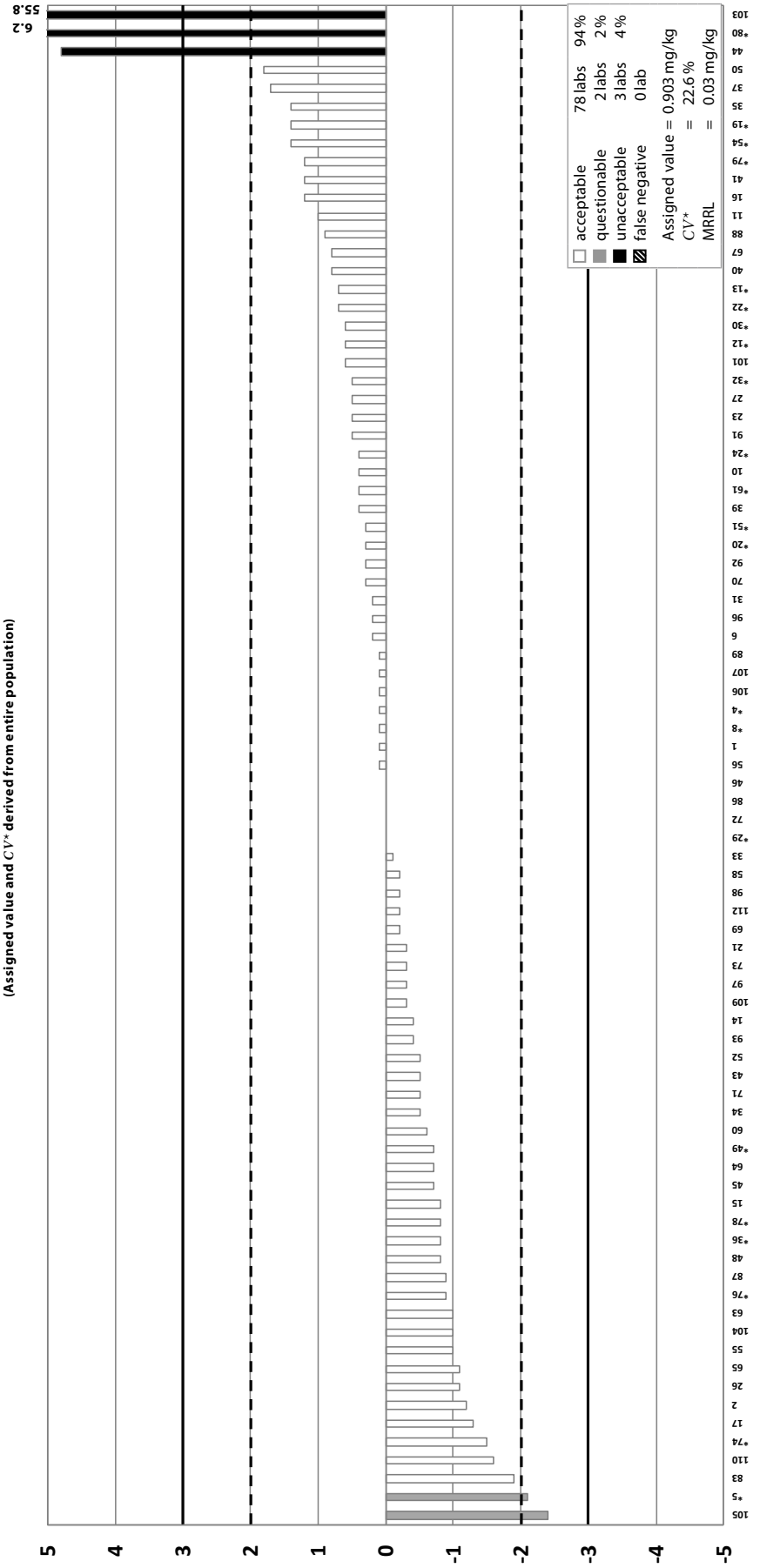
Appendix 6 (cont.) Graphic Presentation of z-Scores: Compulsory Compounds (Results from EU and EFTA Laboratories only, * = NRL)



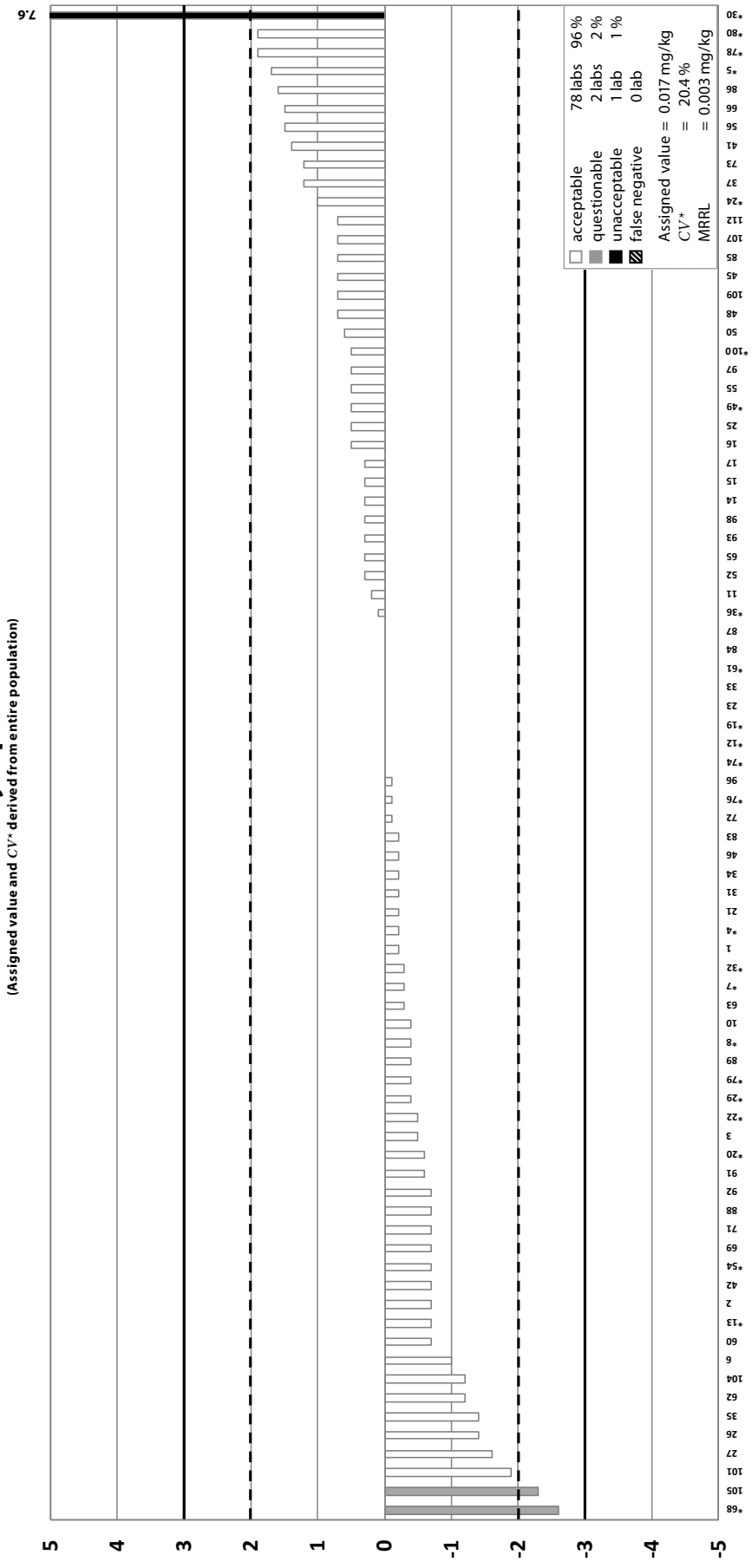
Appendix 6 (cont.) Graphic Presentation of z-Scores: Compulsory Compounds (Results from EU and EFTA Laboratories only, * = NRL)



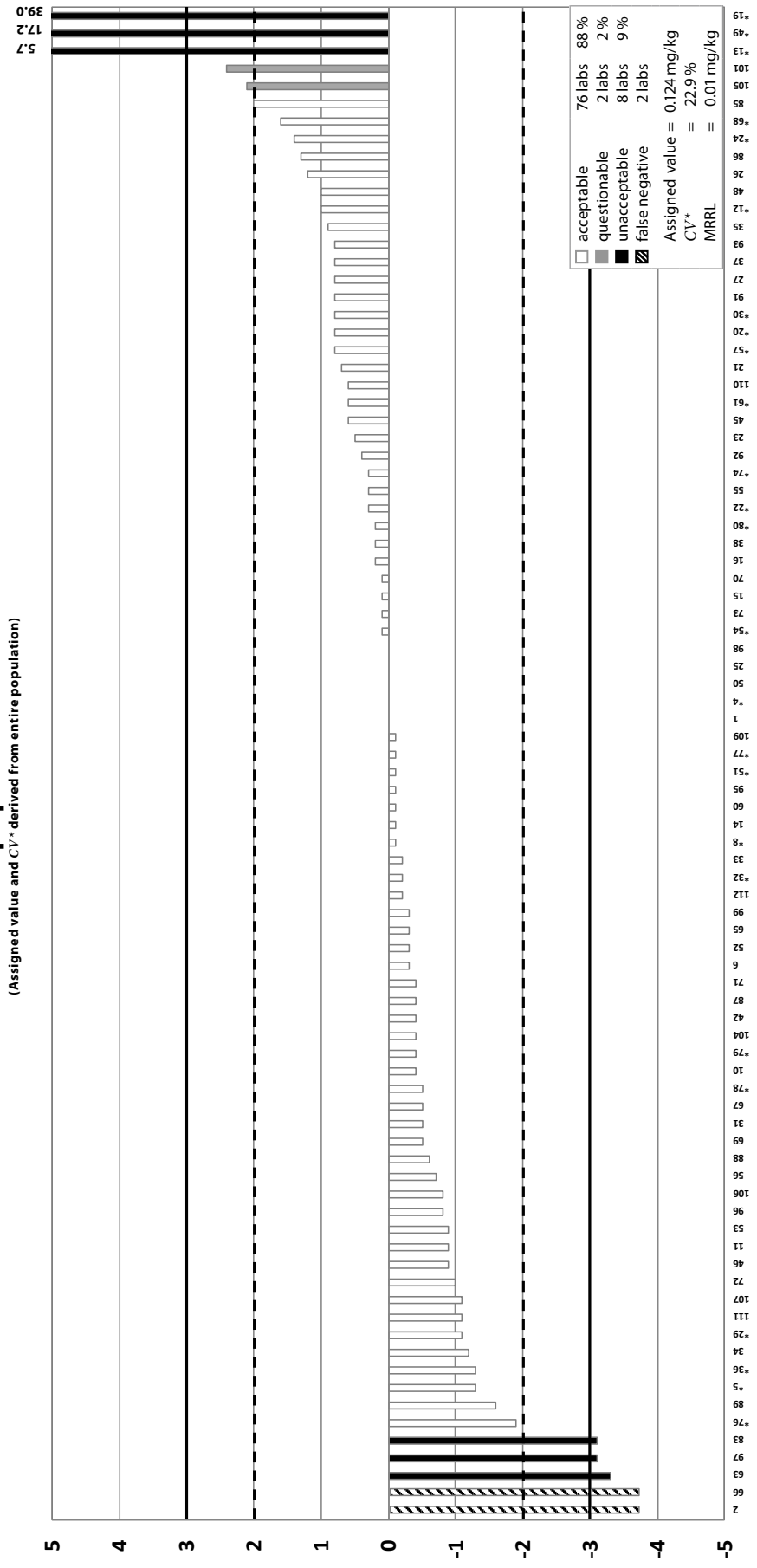
Appendix 6 (cont.) Graphic Presentation of z-Scores: Compulsory Compounds (Results from EU and EFTA Laboratories only, * = NRL)



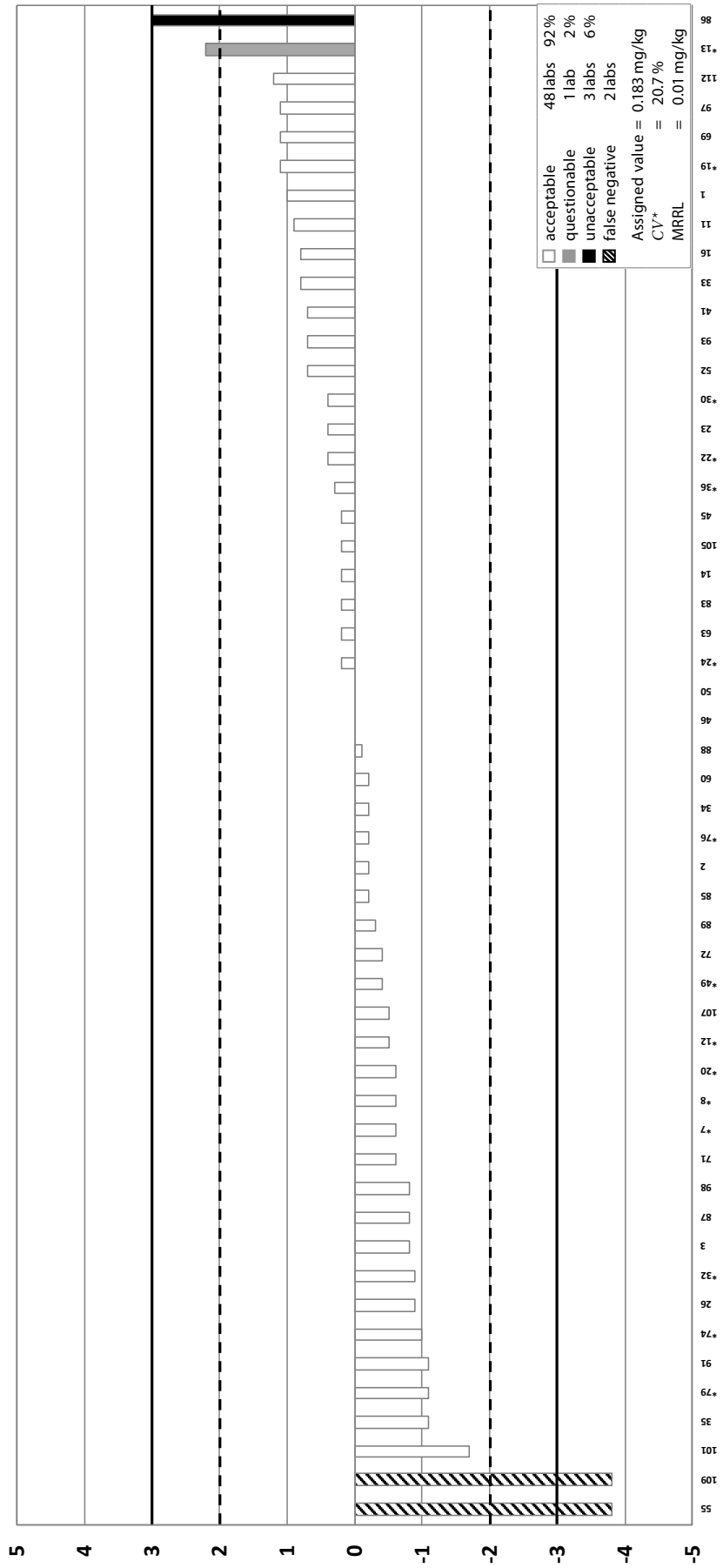
Appendix 6 (cont.) Graphic Presentation of z-Scores: Compulsory Compounds (Results from EU and EFTA Laboratories only, * = NRL)



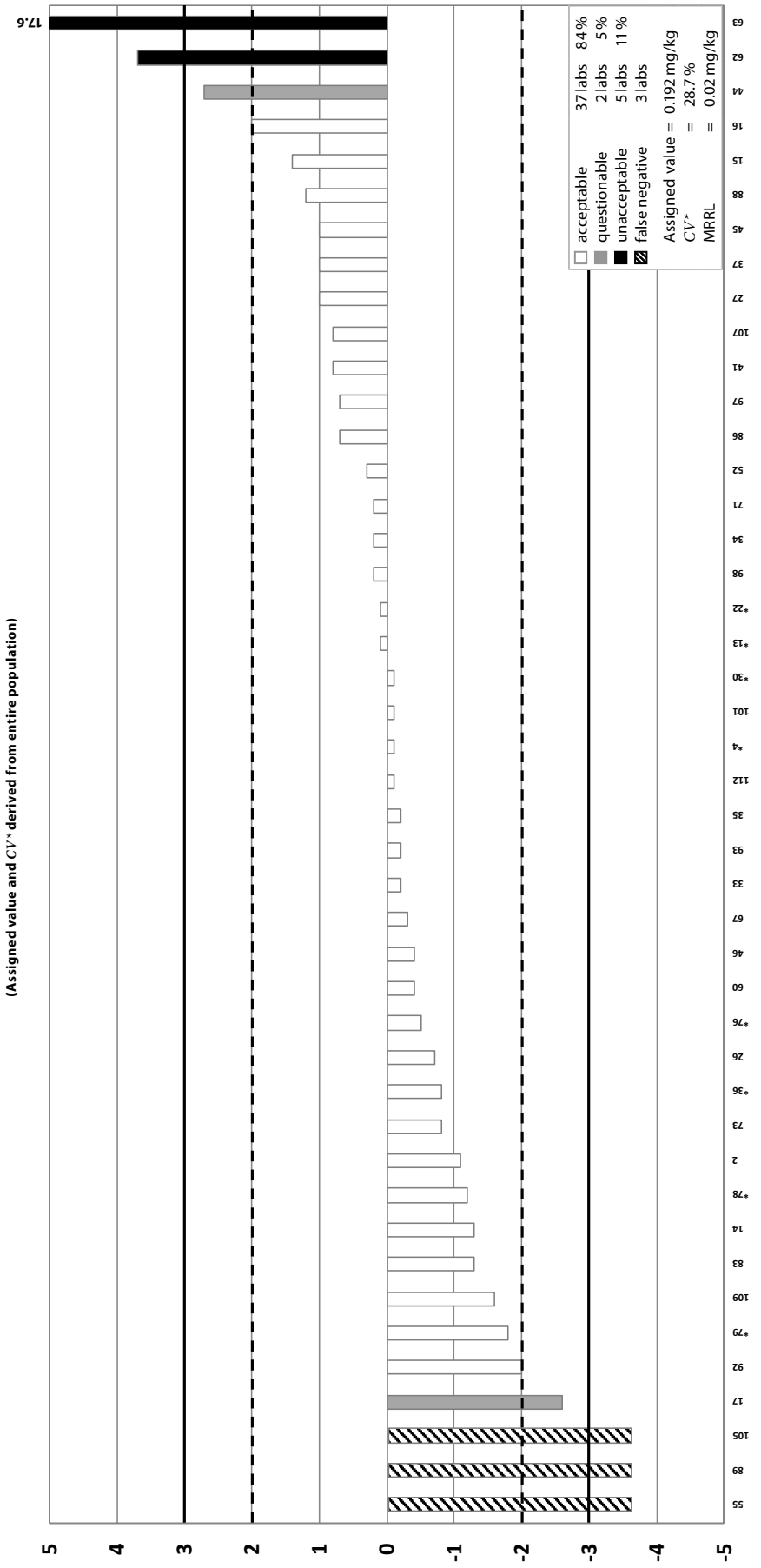
Appendix 6 (cont.) Graphic Presentation of z-Scores: Compulsory Compounds (Results from EU and EFTA Laboratories only, * = NRL)



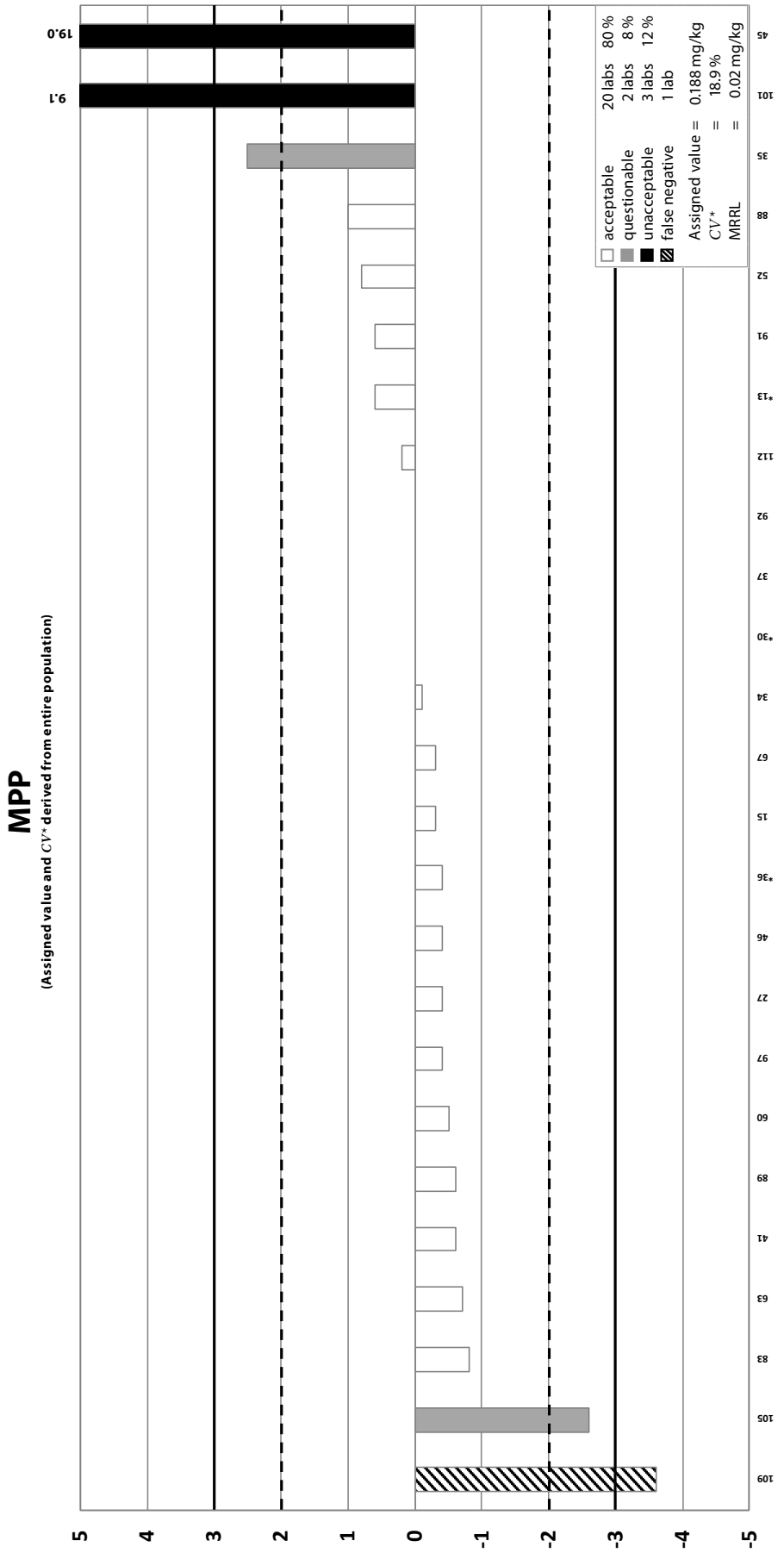
Appendix 6 (cont.) Graphic Presentation of z-Scores: Optional Compounds (Results from EU and EFTA Laboratories only, * = NRL)



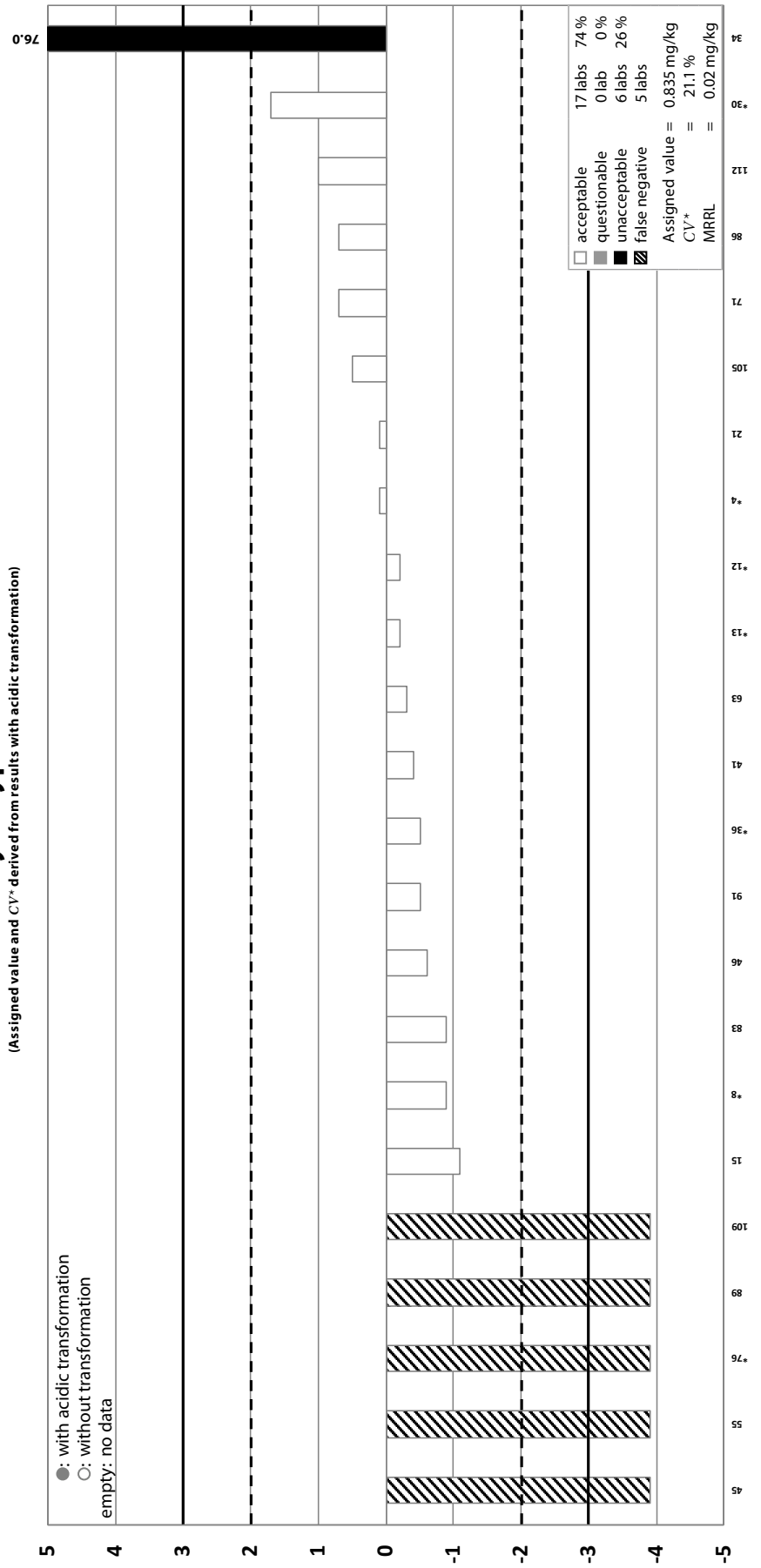
Appendix 6 (cont.) Graphic Presentation of z-Scores: Optional Compounds (Results from EU and EFTA Laboratories only, * = NRL)



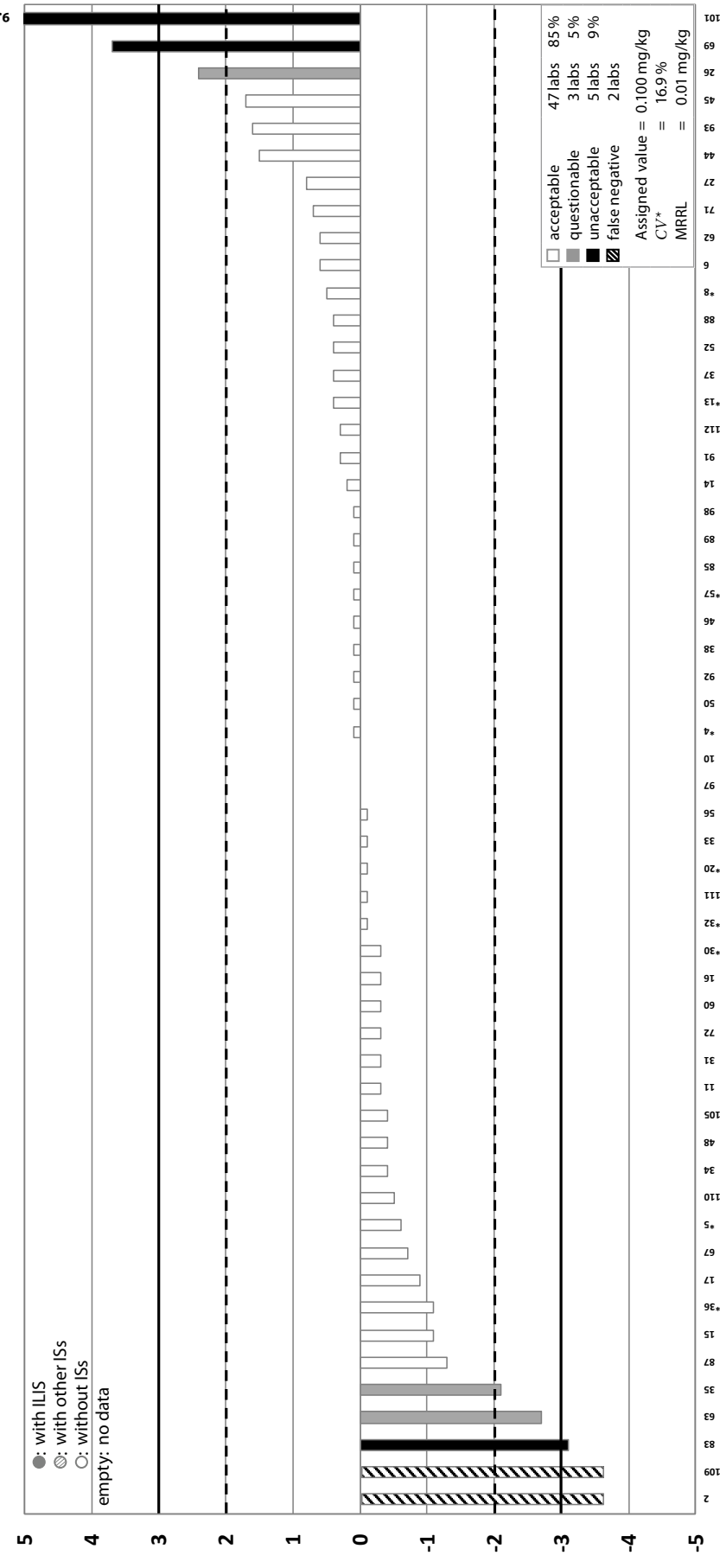
Appendix 6 (cont.) Graphic Presentation of z-Scores: Optional Compounds (Results from EU and EFTA Laboratories only, * = NRL)



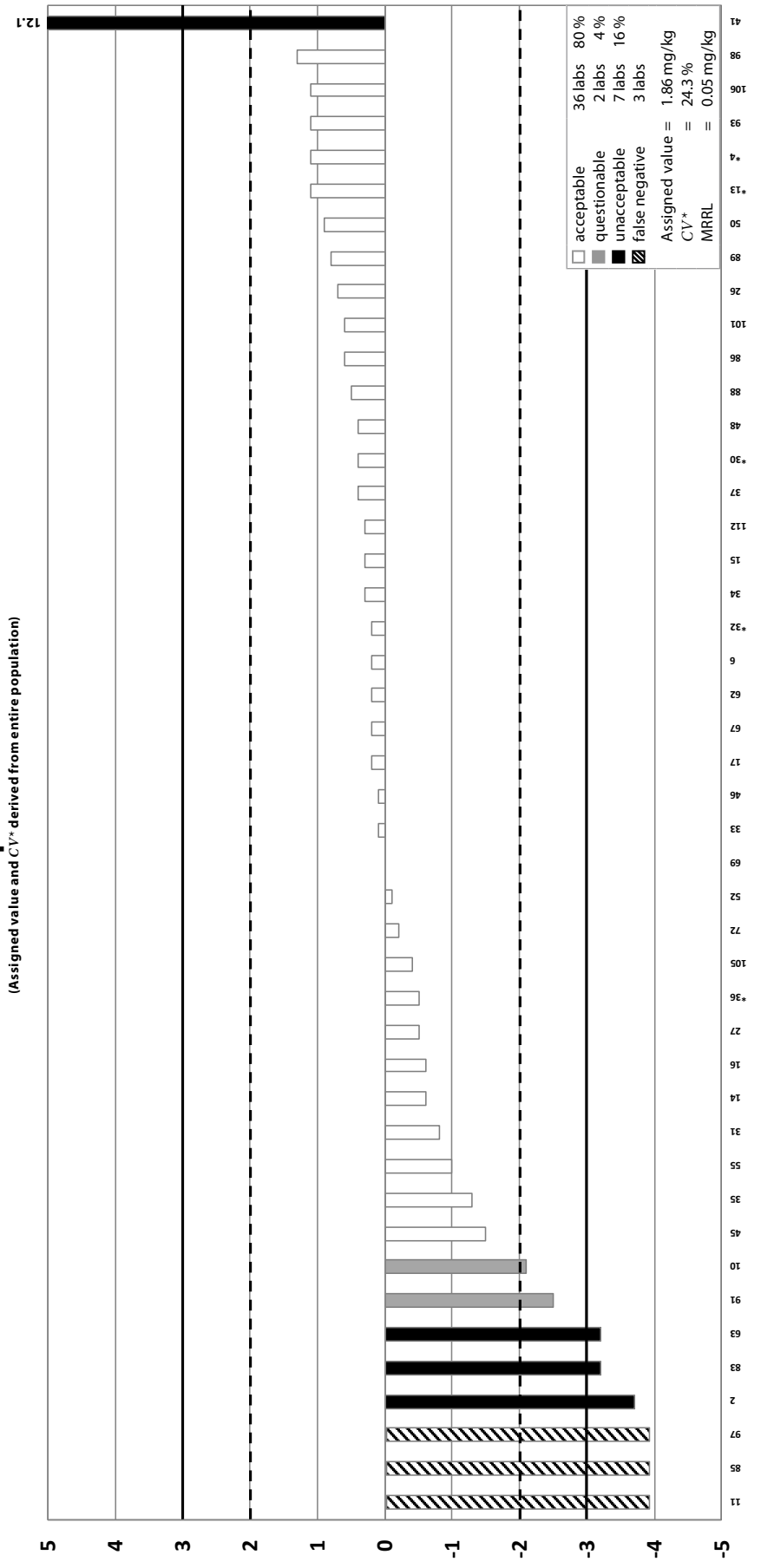
Appendix 6 (cont.) Graphic Presentation of z-Scores: Optional Compounds (Results from EU and EFTA Laboratories only, * = NRL)



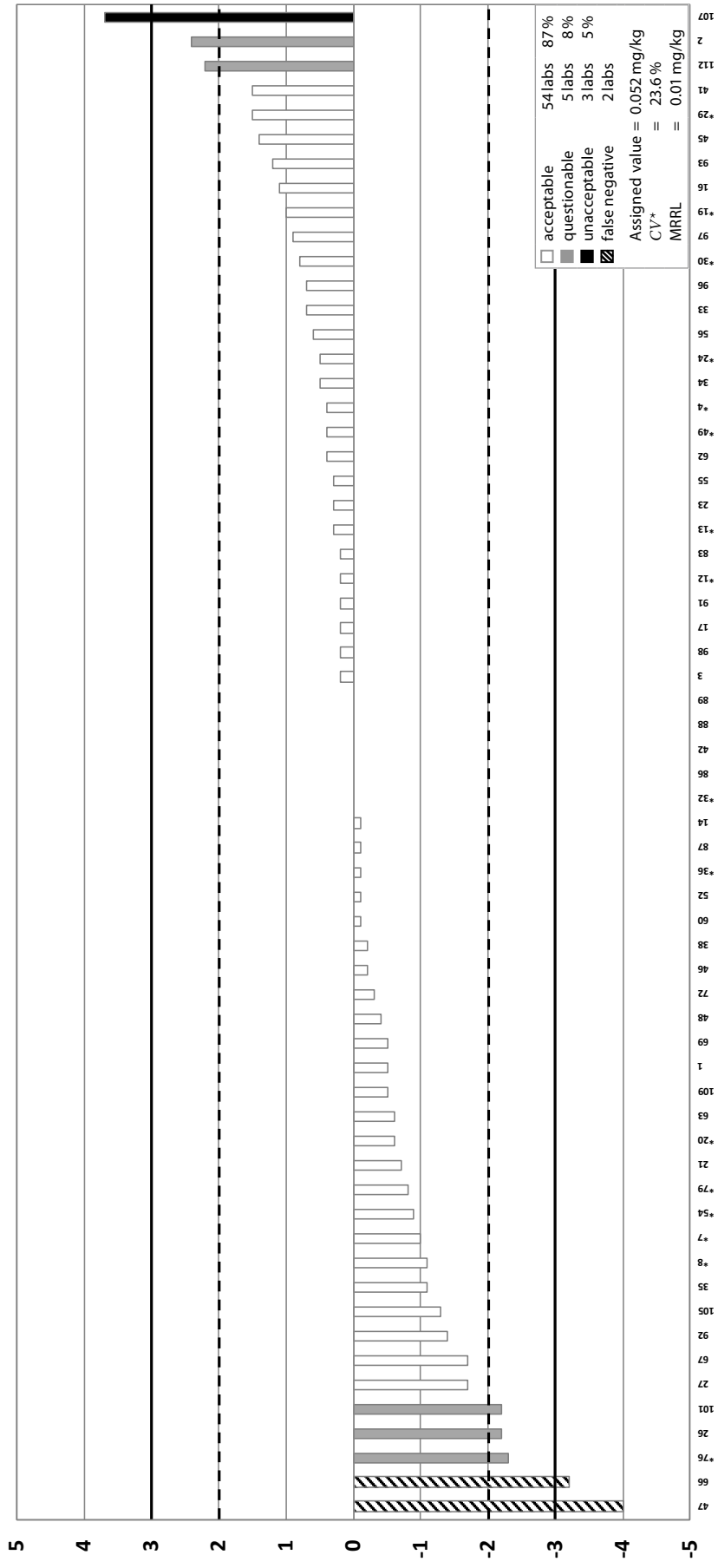
Appendix 6 (cont.) Graphic Presentation of z-Scores: Optional Compounds (Results from EU and EFTA Laboratories only, * = NRL)



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: Optional Compounds (Results from EU and EFTA Laboratories only, * = NRL)



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

A: Technical problems/diffculties with measurement instrumentation
B: Procedure not properly conducted / Error during sample preparation
C: Matrix effect not properly compensated
D: Lack of experience
E: Error in concentration of analytical standard / calibration stock
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 not waranteed
J: Transcription error / Adminitrative fault
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: Contamination
P: Sample amount not sufficient for quantitative investigation
Q: Error in the conversion factor
U: Undefined

Bromide ion Assigned value: 15.4 mg/kg

LabCode	z-Score	Source of error localized?	Reason / Remarks	
65	-3.5 (FN)	No	Our conclution is that this method ""EURL (CRL) Single Residue Method Ver.1, 2009 for Bromide ion"" is not suitable analysis for this type matrices as Soybean Flour. ***Comment from the Organizer:*** Looking at the results of the participants employing methods of this type, I must say that the result distribution was remarkably narrow with the median being close to the assigned value. So, the method seems to work, although it is surely not recommendable to inject many extracts of this type of commodities (high fat content), to avoid fat accumulation in the injector. We ourselves have used the QuPPe method for the homogeneity and stability tests of bromide. I was personally concerned whether the unsaturated fatty acids would be brominated thus consuming bromide, but a cross-check with the derivatization method resulted in similar results. If you like to study this issue a bit more I would recommend conducting a standard addition experiment and maybe comparing the slope of the standard addition experiment with the slope of the normal calibration curve (in solvent).	B?, G?
83	-3.2	Yes	no experience at all with analysis of bromide using LC-MS/MS; not appropriate extraction procedure (use of only MeOH without hydration)	D, G
74	-2.9	Yes	significant matrix effect. We have not much experience with this matrix	C, D
29	2.1	No	Reason not found. We may consider to attribute the overestimated result to the procedural standard calibration that we applied; there is the possibility of increased values due to the inherent correction with recovery of this kind of calibration	L?, U
97	3.3	No	The samples blank (042) and SRM sample (130) have been analyzed in the same sequence including a reference sample containing 10 mg/kg bromide ion. In the SRM-blank sample (042) 10 mg/kg bromide are detected. While the detected concentration in the SRM sample (bottle 130) is 27.9 mg/kg, this calculated using a slope obtained by standard addition method on the blank 042 sample and undiluted samples measurements. The extracts are analyzed both undiluted and 5 times diluted. The results of the 5 times diluted extract have been calculated using a slope with a result of 18.4 mg/kg. The peak of the bromide ion does not show a good shape and the measured amounts in the extract are outside the calibration range. Because the results of the 5 times diluted extract are outside the calibration range and the poor peak shape of bromide, the results of the undiluted measurements have been reported. The used method are optimized and validated for fresh product. The reason for poor result is not clear. Further investigation for dried product is needed.	U

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

Bromide ion Assigned value: 15.4 mg/kg				
LabCode	z-Score	Source of error localized?	Reason / Remarks	
48	4.5	Yes	wir haben unsere Bromid-Analyse in dem Soja-Mehl des SRM13 noch einmal näher untersucht. Wir verwenden die Methode DIN EN 13191-2 (2000-10) mit GC-ECD-Detektion. Wir verwenden sie für Gemüse, hauptsächlich Salat und bisher schien die Methode sehr robust zu sein - wir haben bisher alle Bromid-Ringversuche bestanden. Ein Problem ist die geringe vorgeschriebene Einwaagemenge für trockene Proben. Da die Konzentration an Bromid im Sojamehl ebenfalls recht niedrig war, mussten wir knapp an der Bestimmungsgrenze messen. Wir haben die Analyse des Ringversuchs wiederholt, parallel auch einen alten Ringversuch in Brokkoli. Das Ergebnis der Wiederholung in Sojamehl war zwar etwas niedriger als vorher, aber immer noch zu hoch, während die Konzentration in Brokkoli mit dem Referenzwert übereinstimmte. Wir ziehen daraus den Schluss, dass unsere Analysenmethode für das Sojamehl (und vermutlich andere trockene Proben) nicht geeignet ist. Wir wollten aber ohnehin die Bromid-Bestimmung auf die QuPPe-Methode und LC-MS/MS-Detektion umstellen.	C, G

Cyromazine Assigned value: 0.097 mg/kg				
LabCode	z-Score	Source of error localized?	Reason / Remarks	
2	-3.6 (FN)	Yes	Although we generally add water in extraction of dry cereal- and cereal based products, we did not add water to this 'dry oil-containing soybean' PT- sample. After internal investigation it is clear that we should have added water to this matrix-type in order to achieve a good extraction efficiency. We have retested the sample with water addition and would have obtained a good result for the poor performing compounds. Comment from Organizer: For oily seeds and nuts we use a Variant of the QuOil Method, in which we use as extraction solvent ACN containing 5% Wasser. Without the addition of water we have also observed severe losses of certain pesticides with polar groups, that tend to interact with surfaces. For the analytes relevant to the EUPT-SRM13, QuEChERS for dry products (involving addition of 10 mL water) also works well. For acidic pesticides the use of PSA sorbent in dSPE-cleanup is critical of course. In absence of water, interactions of pesticides with PSA are generally stronger and more pesticides are negatively affected than when dSPE cleanup is conducted on QuEChERS extracts of fruits and vegetables. The 5% water mentioned above is thus helpful in the cleanup step as well. We intend to soon launch an interalboratory validation round for the abovementioned Variant of the QuOil Method with the focus being on nuts and oily seeds. If you are interested to take part, just tell me.	B, G
88	-2.9	Yes	"we analyzed cyromazine with two methods in our lab; the QuEChERS method and the QuPPe method. We detected 0,09 mg/kg cyromazine with the QuEPP method and 0,026 mg/kg cyromazine with the QuEChERS method. Only the QuEChERS method is validated for cyromazine and we have no experience in the analysis of cyromazine with QuPPe. For these reasons we trusted the results of the QuEChERS method more than the results of the QuEPP method. Because of the results of the EUPT-SRM13 we will validate the QuPPe method for cyromazine and use the QuEChERS method only for screening."	D, G
67	-2.4	Yes	Matrix effect (result from the analysis with the Sweet-Method which usually works well, even for difficult matrix, but obviousely not for soy flour in this PT. Using QuPPe and a HILIC column the result was 0.0094 mg/kg but with high Ionensuppression of approx. 50%.	C
35	-2.3	Yes	on classical vegetables, we've not problems with cyromazine analysed by Quechers methodology. But we have no experience with soybean flour. Low recovery can be explained by a matrix effect and difficulties on the integration of the cyromazine peak. Quppe as recommended by EURL with specific chromatographic conditions should be better.	C, D

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

A: Technical problems/diffculties with measurement instrumentation
B: Procedure not properly conducted / Error during sample preparation
C: Matrix effect not properly compensated
D: Lack of experience
E: Error in concentration of analytical standard / calibration stock
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 not waranteed
J: Transcription error / Adminitrative fault
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: Contamination
P: Sample amount not sufficient for quantitative investigation
Q: Error in the conversion factor
U: Undefined

Cyromazine Assigned value: 0.097 mg/kg

LabCode	z-Score	Source of error localized?	Reason / Remarks	
62	-2.2	Yes	The recovery obtained in the spiked sample of the blank material was 67 %. It complains with the acceptance criteria for recovery in routinary analysis (60-140%), so recovery factor was not applied to correct sample concentration. The low recovery obtained would explain the low concentration submitted for ciromazine (0.0446 mg/kg).	K
78	-2.1	(Yes)	No experience; The reason for the poor performance is probably due to problems with extraction efficiency (human error or matrix effect). Investigations in progress.	C, D
74	-2.1	Yes	significant matrix effect. We have not much experience with this matrix	C, D
3rd-122	2.1	Yes	"Error calculation, because we used a factor of two during the calculation of the results for these pesticides. The implementation of the analytical method for theses pesticide is in process in our laboratory; but we consider to send our result of these pesticide to SRM13 as an opportunity in order to have a preliminary evaluation of our analytical performance. Currently we don't include these pesticide on the scope our analytical method for routine analysis. "	D, F
109	2.9	(Yes)	It's probably a standard problem (ordered 7 years ago). A new one will be ordered and a comparison made	E?
45	3.7	Yes	Matrix effect (Area of ILIS of cyromazine in the sample extract was half so big as that in the calitration solutions); Einen Pipettierfehler der Lösung des Internen Standards schließe ich aus, da wir alle Internen Standards in einer Dotierlösung haben. Was ich unter den gegebenen Umständen nicht sagen kann ist, ob beim Ansetzen der Lösung ein Pipettierfehler vorgelegen hat. Den Extrakt haben wir zusätzlich verdünnt gemessen. Allerdings habe wir bei der Verdünnung den Internen Standard aufgestockt (Verdünnung mit Blankextrakt dem interner Standard für Aufarbeitung zugesetzt wurde). Hier bei erhalten wir einen Gehalt von 0,081mg/kg. Allerdings ist die Fläche des IS im verd. Extrakt auch etwas niedriger als in den Kalibrierlösungen. Aktuell setzen wir gerade unsere Stamm- und Arbeitslösungen neu an. Es ist geplant die LVU nach Austausch der Lösungen erneut aufzuarbeiten und die Parameter zu bestimmen. Im Zuge der Nachanalyse der LVU werden wir ggf. die Bestimmung auch gegen eine über das Gesamtverfahren aufgearbeitete Kalibrierung durchführen.	E
106	34.8	Yes	Transcription error resulting from difficulties of the submission tool	J
19	44.7	Yes	Concentration of calibration standard not correct (too low)	E

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

Fluazifop Assigned value: 0.049 mg/kg

LabCode	z-Score	Source of error localized?	Reason / Remarks	
3rd-118	-3.2 (FN)	Yes	Our standard solution was Fluazifop-butyl, not free acid	N
45	-2.2	No	Reason not found	U

Glyphoaste Assigned value: 0.903 mg/kg

LabCode	z-Score	Source of error localized?	Reason / Remarks	
105	-2.4	Yes	Out of lab routine scope, we don't have the experience yet.	D
5	-2.1	(Yes)	Our lab don`t consider the result of glyphosate as poor performance. The reasons are: our 2 analysts have made overall 6 replacates of the sample SRM13, 6 replicates of recovery (addition to blank SRM13). All above mentioned replicates were analysed with two analytical chormatographic columns: ACCLAIM Trinity and Hypercarb. We have made several dilutions to explore dependance between concentration and dilution factor: 4 graphs each has 6 points=> 24 dilutions. Our LC/MS/MS piping is metallic, so we have to passive the piping. We can`t change pipes into plastic, because plastic pipes don`t tolerate pressure very well. It was the first time to analyse soya matrix for us and Quppe method didn`t perform very well. At the moment we don`t use IS, because it is very expensive to buy it just for PT. If we start analyse samples then we consider to buy it. Perhaps IS facilitates the analysis.	A, D
44	4.8	Yes	few experience (less than one year), matrix effect and too less sample used for analysis. The result reported was from a sample weight of 0.2 g with clean extract. With a sample weight of 0.5 g, the extract was dirty and, therefore, the result (0.821 mg/kg) was not submitted.	C, D, I
80	6.2	Yes	Very likely due to matrice - we never analyse such heavy matrice (20% fat). We do quantification with Standard addition for glyphosate, this seems not appropriate for difficult matrices. Our first results using external calibration gave concentrations of 1.25 mg/kg. With our second extern calibration with more calibartion points we got 1.08 mg Glyphosate/kg. Comment from Organizer: Standard addition may indeed be associated with an error if the function is curved. If the lower part of the curve is still linear you may skip the upper points and just do a one-point standard addition. Otherwise dilution will help you to reach the linear range. If you use isotope labelled standard it should also behave similarly to the native one in terms of non-linearity, so the ratio should be expectedly still close to linear even if you work within a non-linear range.	C, D; L
103	55.8	(Yes)	our method is a screening method used only to determinate if glyphosate is more then 0,1 mg/kg we checked our result and we observed we made a mistake with the amount of internal standard but also in this case we have a lower z-score > 3. we think that the only way to solve our problems is use ionic chromatography.	

Haloxyfop Assigned value: 0.017 mg/kg

LabCode	z-Score	Source of error localized?	Reason / Remarks	
3rd-115	-3.3 (FN)	Yes	No IS was used and therefore the matrix effect not properly compensated. After using IS (Bentazon-D7) the result was 0.173 mg/kg.	C
3rd-116	-2.3	Yes	For Haloxyfop, we had 2 group of results, in interval from 0,004mg/kg to 0,009mg/kg, and group with 2 results of 0,014mg/kg and 0,015mg/kg. We calculated the average of first group of results without last two and it was a mistake. If we had calculated the average of all results, z-score would have been satisfactory.	F
105	-2.3	Yes	Matrix in not within our routine scope.	D

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

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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 not waranteed
J: Transcription error / Adminitrative fault
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: Contamination
P: Sample amount not sufficient for quantitative investigation
Q: Error in the conversion factor
U: Undefined

Mepiquat Assigned value: 0.124 mg/kg

LabCode	z-Score	Source of error localized?	Reason / Remarks	
2	-3.7 (FN)	Yes	Although we generally add water in extraction of dry cereal- and cereal based products, we did not add water to this 'dry oil-containing soybean' PT- sample. After internal investigation it is clear that we should have added water to this matrix-type in order to achieve a good extraction efficiency. We have retested the sample with water addition and would have obtained a good result for the poor performing compounds. Comment from Organizer: For oily seeds and nuts we use a Variant of the QuOil Method, in which we use as extraction solvent ACN containing 5% Wasser. Without the addition of water we have also observed severe losses of certain pesticides with polar groups, that tend to interact with surfaces. For the analytes relevant to the EUPT-SRM13, QuEChERS for dry products (involving addition of 10 mL water) also works well. For acidic pesticides the use of PSA sorbent in dSPE-cleanup is critical of course. In absence of water, interactions of pesticides with PSA are generally stronger and more pesticides are negatively affected than when dSPE cleanup is conducted on QuEChERS extracts of fruits and vegetables. The 5% water mentioned above is thus helpful in the cleanup step as well. We intend to soon launch an interalboratory validation round for the abovementioned Variant of the QuOil Method with the focus being on nuts and oily seeds. If you are interested to take part, just tell me.	B, G
66	-3.7 (FN)	Yes	Transcription error under stress caused by technical problem with LC-MS/MS (actually 0.175 mg/kg)	J
63	-3.3	Yes	We have checked the standars solutions involved in the test, some of them with reference material and after repeating the extractions, we would need more time to perform other extraction methods because we believe that it could be the problem to obtain a correct quantification.	C, G

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

Mepiquat Assigned value: 0.124 mg/kg				
LabCode	z-Score	Source of error localized?	Reason / Remarks	
97	-3.1	Yew	"The sample has been hydrolyzed by adding water to the weighted fraction of sample. After 10 minutes the extraction is started by acidified methanol extraction. The calculations have been checked and it was OK. The normal used method applies for fruits and vegetables. Because of the sample are an dried product an additional step of hydrolyzation has been applied for the analysis of the SRM material. An investigation has been carried out for the hydrolyzation time by adding water to different fractions of the SRM sample and proceeding to the extraction step after 10 min and over two nights. The results are below: - Starting with the extraction after 10 minutes hydrolyzation time: concentration of mepiquat is 0.026 mg/kg. The results of the first and reported results is 0.027 mg/kg and the waiting time was 15 min. - Starting with the extraction after 2 nights hydrolyzation time: concentration of mepiquat is 0.077 mg/kg . The reason for poor results is a non optimal extraction time of the dried product. This will be further investigated.	D, G
83	-3.1	Yes	we have never analyzed Mepiquat in cereals or oilseeds. We believe that the unsatisfactory results are due to the fact that we used 5 grams of sample and did not hydrate with water before extraction. After seeing the results in the preliminary report, we repeated the extraction using 2 grams of sample and hydrating with 5 mL of water for 1 hour before extraction. The result obtained now is 0,120 mg/kg	C, D, G
105	2.1	Yes	Matrix in not in our routine scope.	D
49	17.2	Yes	conversion factor for mepiquat ion to mepiquate chlorate was incorrect	Q
101	2.4	No	The result of the repeat is 0.076 mg / kg. This gives a Z-score of -1.56, which satisfies, which satisfies. No deviations in integration, calibration, standards and control sample were detected. No apparent cause was found for the abnormality. The deviation for MPP is very high. The original z-score was just above 2. This in combination with the good scores of the reanalysis is the conclusion that the deviation only applies to the sequence in which the ring test was measured.	U
3rd-122	4.7	Yes	Error calculation, because we used a factor of two during the calculation of the results for these pesticides. The implementation of the analytical method for theses pesticide is in process in our laboratory; but we consider to send our result of these pesticide to SRM13 as an opportunity in order to have a preliminary evaluation of our analytical performance. Currently we don't include these pesticide on the scope our analytical method for routine analysis.	D, F
19	39	Yes	Concentration of calibration standard not correct (too low)	E

2,4-DB Assigned value: 0.183 mg/kg				
LabCode	z-Score	Source of error localized?	Reason / Remarks	
55	-3.8 (FN)	Yes	Transcritption error (actually not analysed), not routinely analysed	J
109	-3.8 (FN)	Yes	transcriptional error, actually not analysed	J
86	3.0	Yes	This poor performance was probably due to the use of an old calibration solution. We did not detect this problem as our 2,4-DB recovery was correct. New standard ordered, investigation ongoing.	E

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

A: Technical problems/diffculties with measurement instrumentation
B: Procedure not properly conducted / Error during sample preparation
C: Matrix effect not properly compensated
D: Lack of experience
E: Error in concentration of analytical standard / calibration stock
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 not waranteed
J: Transcription error / Adminitrative fault
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: Contamination
P: Sample amount not sufficient for quantitative investigation
Q: Error in the conversion factor
U: Undefined

Glufosinate Assigned value: 0.192 mg/kg

LabCode	z-Score	Source of error localized?	Reason / Remarks	
55	-3.6 (FN)	Yes	Transcritption error (actually not analysed), not routinely analysed	J
89	-3.6 (FN)	Yes	No experience with this analyte, re-analysed and evaluated: 0.981 mg/kg, z-score = 0.7	D
105	-3.6 (FN)	Yes	Out of lab routine scope, we don't have the experience yet.	D
3rd-121	-3.6 (FN)	Yes	detected concentration 0.182 mg/kg < RL (0.5 mg/kg), therefore no numerical result reported.	H
17	-2.6	Yes	two analysis and two other for recovery were conducted; errors occured during sample preparation and lack of material no repeatation was possible; no information about the recovery was available, and the result was not corrected by the recovery.	B, R, P
44	2.7	Yes	few experience (less than one year), matrix effect and too less sample used for analysis. The result reported was from a sample weight of 0.2 g with clean extract. With a sample weight of 0.5 g, the extract was dirty and, therefore, the result (0.19 mg/kg) was not submitted.	C, D, I
62	3.7	Yes	The recovery obtained in the spiked sample of the blank material was 42 %. It does not complain with the acceptance criteria for recovery in routinary analysis (60-140%), so recovery factor was applied to correct sample concentration. However, we should have demonstrate good reproducibility before apply this recovery factor	K
63	10.6	Yes	We have checked the standars solutions involved in the test, some of them with reference material and after repeating the extractions, we would need more time to perform other extraction methods because we believe that it could be the problem to obtain a correct quantification.	C, G

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

MPP Assigned value: 0.188 mg/kg

LabCode	z-Score	Source of error localized?	Reason / Remarks	
109	-3.6 (FN)	Yes	transcriptional error, actually not analysed	J
105	-2.6	Yes	Out of lab routine scope, we don't have the experience yet.	D
35	2.5	Yes	we have no experience with this compound (not analysed till today in the laboratory). Analysed to test us. A correction with glyphosate C13 was done but MPP D3 for recovery correction should be better	D, L
101	9.1	No	The result of the repeat is 0.268 mg / kg. This gives a Z-score of 1.7, which satisfies. No deviations in integration, calibration, standards and control sample were detected. No apparent cause was found for the abnormality. The deviation for MPP is very high. This component is kept in the control card and there are no deviations in the chard. Because the re-analysis is good, this deviation also applies only to the sequence in which it was measured.	U
45	19	Yes	bad chromatography (small and wide peak, perhaps column not sufficiently preconditioned)	B?, M

N-Acetyl glyphosate Assigned value: 0.835 mg/kg

LabCode	z-Score	Source of error localized?	Reason / Remarks	
45	-3.9 (FN)	Yes	transcrpition errer (confusing between n-acetyl-glyphosate and n-ycetyl-glufosinate); ich muss gestehen, dass ich mir bei dem N-Acetyl-Glyphosat nicht die Abweichung zum assigned value angesehen hatte. Wir haben das N-Acetyl-Glyphosat gegen das isotopenmarkierte Glyphosat als Internem Standard ausgewertet. Bei der erneuten Durchsicht der Daten ist mir aufgefallen, dass die Widerfindung für das N-Acetylglyphosat um den Faktor 2 zu hoch ist, während die Wiederfindung für Glyphosat i.O. ist (98%). Die Fläche des Internen Standards ist im LVU-Probenextrakt geringer als in den Kalibrierlösungen, was den Überbefund erklären könnte (für die Kalibrierung dotieren wir den Blank-Extrakt an, keine Kalibrierung über Gesamtverfahren). Auswertung ohne internen Standard gegen die externe Kalibrierung liefert etwas geringere Werte von ~1,0 mg/kg. Aktuell setzen wir gerade unsere Stamm- und Arbeitslösungen neu an. Es ist geplant die LVU nach Austausch der Lösungen erneut aufzuarbeiten und die Parameter zu bestimmen. Im Zuge der Nachanalyse der LVU werden wir ggf. die Bestimmung auch gegen eine über das Gesamtverfahren aufgearbeitete Kalibrierung durchführen.	J
55	-3.9 (FN)	Yes	Transcritption error (actually not analysed) The N acetylglyphosate is not analyzed because the Hypercarb column is too tricky to maintain in routine flow. So we let it for an ionic column and it's impossible to get it	J
76	-3.9 (FN)	Yes	Transcription error. (This was a result submission error. This compound is not part of the laboratory's scope. It is not a true false negative result, the laboratory does not currently analyse for n-acetyl-glyphosate.)	J
89	-3.9 (FN)	Yes	No experience with this analyte	D
109	-3.9 (FN)	Yes	transcriptional error, actually not analysed	J
34	76	Yes	Standard used (pure substance) for calculation was probably degradeted (cp. with a new standard resulted to a factor of 25.)	E

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

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D: Lack of experience
E: Error in concentration of analytical standard / calibration stock
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 not waranteed
J: Transcription error / Adminitrative fault
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: Contamination
P: Sample amount not sufficient for quantitative investigation
Q: Error in the conversion factor
U: Undefined

Perchlorate Assigned value: 0.100 mg/kg

LabCode	z-Score	Source of error localized?	Reason / Remarks	
2	-3.6 (FN)	Yes	Although we generally add water in extraction of dry cereal- and cereal based products, we did not add water to this 'dry oil-containing soybean' PT- sample. After internal investigation it is clear that we should have added water to this matrix-type in order to achieve a good extraction efficiency. We have retested the sample with water addition and would have obtained a good result for the poor performing compounds. Comment from Organizer: For oily seeds and nuts we use a Variant of the QuOil Method, in which we use as extraction solvent ACN containing 5% Wasser. Without the addition of water we have also observed severe losses of certain pesticides with polar groups, that tend to interact with surfaces. For the analytes relevant to the EUPT-SRM13, QuEChERS for dry products (involving addition of 10 mL water) also works well. For acidic pesticides the use of PSA sorbent in dSPE-cleanup is critical of course. In absence of water, interactions of pesticides with PSA are generally stronger and more pesticides are negatively affected than when dSPE cleanup is conducted on QuEChERS extracts of fruits and vegetables. The 5% water mentioned above is thus helpful in the cleanup step as well. We intend to soon launch an interalboratory validation round for the abovementioned Variant of the QuOil Method with the focus being on nuts and oily seeds. If you are interested to take part, just tell me.	B, G
109	-3.6 (FN)	Yes	transcriptional error, actually not analysed	J
83	-3.1	Yes	we have never analyzed perchlorate in cereals or oilseeds. We believe that the unsatisfactory results are due to the fact that we used 5 grams of sample and did not hydrate with water before extraction. After seeing the results in the preliminary report, we repeated the extraction using 2 grams of sample and hydrating with 5 mL of water for 1 hour before extraction. The result obtained now is 0,090 mg/kg.	C, D, G
63	-2.7	(Yes)	We have checked the standars solutions involved in the test, some of them with reference material and after repeating the extractions, we would need more time to perform other extraction methods because we believe that it could be the problem to obtain a correct quantification.	C, G

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

Perchlorate Assigned value: 0.100 mg/kg

LabCode	z-Score	Source of error localized?	Reason / Remarks	
35	-2.1	Yes	we have no experience with soybean flour. A correction with fosetyl D15 is done and usually give us good results. Perchlorate O18 for recovery correction should be better.	D, L
26	2.4	Yes	Soy matrix little analyzed by the laboratory - essentially water-rich matrix - no accreditation on perchlorate; Intégration large sur la 1ère analyse et ajouts dosé trop importants par rapport à la concentration cible. Relance avec ajouts dosé plus faibles (0,2 et 0,4) qui permettent d'obtenir un résultat conforme. Suivi de ce paramètre sur les essais ultérieurs.	D
69	3.7	Yes	Precipitation of stock solution (1mg/ml MeOH with 5% FA) stored at -20 °C that was not redisolved at ambient temperature. New stock prepared in 50% MeOH/50% Water with 5% FA. No ppt even stored at low temperature. With new stock: conc. determined at 0,077 mg/kg, corresponding to a z.score of -0.9; Recovery: 77%	E
101	9.5	No	The cause was probably the concentration of the blank that was forgotten to take of the result. The result after blank correction is (0.355-0.214) 0.121 mg / kg. This yields a Z score of 0.85. This does meet. The deviation of perchlorate applies, however, only to this sequence. In "regular" samples no blank is included and therefore no blank deduction is possible.	U

Phosphonic acid Assigned value: 1.86 mg/kg

LabCode	z-Score	Source of error localized?	Reason / Remarks	
11	-3.9 (FN)	Yes	Transcription error resulting from difficulties of the submision tool (actually not analysed at all)	J
97	-3.9 (FN)	Yes	The peak of phosphonic acid detected in the sample fulfills the criteria of retention time and MRM ratio, (see the chromatograms 1 and 2). By adding a known amount of phosphonic acid to a second fraction of the sample, by applying the standard addition method, the detected peak splits in to two peaks that are not totally separated. The detected peak of phosphonic acid in the first fraction without addition is considered as false positive and the results are not reported. Because of a poor z-score the extracts analyses performed on the SRM material are repeated with a 20 times extra dilution of the extracts compared to the initial measurements performed by the first analyses of the SRM samples. The obtained peaks shape was good and the splitting in the spiked extract was not observed, (see the chromatograms 3 and 4). The reason for poor results is an insufficient chromatography method application. A new method will be developed using a different column with more separation power.	F, G
2	-3.7	Yes	Although we generally add water in extraction of dry cereal- and cereal based products, we did not add water to this 'dry oil-containing soybean' PT- sample. After internal investigation it is clear that we should have added water to this matrix-type in order to achieve a good extraction efficiency. We have retested the sample with water addition and would have obtained a good result for the poor performing compounds. Comment from Organizer: For oily seeds and nuts we use a Variant of the QuOil Method, in which we use as extraction solvent ACN containing 5% Wasser. Without the addition of water we have also observed severe losses of certain pesticides with polar groups, that tend to interact with surfaces. For the analytes relevant to the EUPT-SRM13, QuEChERS for dry products (involving addition of 10 mL water) also works well. For acidic pesticides the use of PSA sorbent in dSPE-cleanup is critical of course. In absence of water, interactions of pesticides with PSA are generally stronger and more pesticides are negatively affected than when dSPE cleanup is conducted on QuEChERS extracts of fruits and vegetables. The 5% water mentioned above is thus helpful in the cleanup step as well. We intend to soon launch an interalboratory validation round for the abovementioned Variant of the QuOil Method with the focus being on nuts and oily seeds. If you are interested to take part, just tell me.	B, G

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

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H: Reporting limit higher than the assigned value
I: Sample weight too small, homogeneity not waranteed
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K: Result not corrected for low recovery
L: Inappropriate calibration
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N: Misunderstanding of the definition of the analyte
O: Contamination
P: Sample amount not sufficient for quantitative investigation
Q: Error in the conversion factor
U: Undefined

Phosphonic acid Assigned value: 1.86 mg/kg

LabCode	z-Score	Source of error localized?	Reason / Remarks	
83	-3.2	Yes	Until now, we have never analyzed Phosphonic acid in cereals or oilseeds. We believe that the unsatisfactory results are due to the fact that we used 5 grams of sample and did not hydrate with water before extraction. After seeing the results in the preliminary report, we repeated the extraction using 2 grams of sample and hydrating with 5 mL of water for 1 hour before extraction. The result obtained now is 2,90 mg/kg.	C, D, G
63	-3.2	Yes	We have checked the standars solutions involved in the test, some of them with reference material and after repeating the extractions, we would need more time to perform other extraction methods because we believe that it could be the problem to obtain a correct quantification.	C, G
91	-2.5	Yes	Despite the fact, that the isotopically labelled phosphorus acid standard has been used and the prepared spike in the blank matrix has provided a satisfactory value, these measures did not provide effective help in case of the correct determination of phosphonic acid as incurred residues. After re-extraction of sample, when 2.5 g was weighted and soaking time was extended to 30 min (instead of 10 min in original procedure), we obtained the concentration of phosphonic acid 1.871 mg/kg. Considering this fact the soaking time seems to be the key parameter for our originally reported concentration level of phosphonic acid.	G
41	12.1	(Yes)	in the same analytical batch we processed the EUPT_SRM12 material and we obtained the acceptable result for this molecule. So we believed that EUPT_SRM13 results was good. Now we are testing our standard pool and single Phosphonic acid solution to verify the retention time and possible degradation of other pesticides in pool to Phosphonic acid.	E?

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

Quizalofop Assigned value: 0.052 mg/kg				
LabCode	z-Score	Source of error localized?	Reason / Remarks	
47	-4 (FN)	Yes	The active substance that we analyzed is Quizalofop-ethyl CAS No. 76578. (The analysis was carried out by extraction with acetonitrile, cleaning with PSA and evaporation of the extract in a stream of nitrogen. The addition made to the white sample at the level of 0.01 mg / kg gives a correct recovery of 68%.)	U
66	-3.2 (FN)	Yes	Transcription error under stress caused by technical problem with LC-MS/MS (actually 0.034 mg/kg)	J
3rd-118	-3.2 (FN)	Yes	Our standard solution was Quizalofop-ethyl, not free acid	N
76	-2.3	Yes	Ne experience and special matrix effect of soybean flour. (Soybean flour is not tested routinely in our laboratory. There was a poor recovery of quizalofop in the lab soybean flour spike (56%) analysed with this PT scheme. This poor recovery is unique to soybean flour and not other cereals analysed by the laboratory. The lab did not correct for recovery. If the lab had corrected for recovery in the spike then the z-score would have been acceptable.)	C, D
26	-2.2	Yes	Soy matrix little analyzed by the laboratory - essentially water-rich matrix - no accreditation on quizalofop; Sous dosage lors de la 1ère analyse, rendement pris en compte lors du rendu. Relance conforme. Problème ponctuel d'extraction	D
101	-2.2	No	The result of the repeat is 0.030 mg / kg. This gives a Z-score of -1.7, which satisfies. No deviations in integration, calibration, standards and control sample were detected. No apparent cause was found for the abnormality. The deviation for MPP is very high. The original z-score was just below -2. This in combination with the good scores of the reanalysis is the conclusion that the deviation only applies to the sequence in which the ring test was measured.	U
112	2.2	Yes	the evaluation of this non-conformity showed that the standard used for Quizalofop was 20% too low, presumably due to precipitation. When correcting for this factor, we would have found a value of 0,064 mg/kg, thus well within the acceptable range	U
2	2.4	Yes	Although we generally add water in extraction of dry cereal- and cereal based products, we did not add water to this 'dry oil-containing soybean' PT- sample. After internal investigation it is clear that we should have added water to this matrix-type in order to achieve a good extraction efficiency. We have retested the sample with water addition and would have obtained a good result for the poor performing compounds. Comment from Organizer: For oily seeds and nuts we use a Variant of the QuOil Method, in which we use as extraction solvent ACN containing 5% Wasser. Without the addition of water we have also observed severe losses of certain pesticides with polar groups, that tend to interact with surfaces. For the analytes relevant to the EUPT-SRM13, QuEChERS for dry products (involving addition of 10 mL water) also works well. For acidic pesticides the use of PSA sorbent in dSPE-cleanup is critical of course. In absence of water, interactions of pesticides with PSA are generally stronger and more pesticides are negatively affected than when dSPE cleanup is conducted on QuEChERS extracts of fruits and vegetables. The 5% water mentioned above is thus helpful in the cleanup step as well. We intend to soon launch an interalboratory validation round for the abovementioned Variant of the QuOil Method with the focus being on nuts and oily seeds. If you are interested to take part, just tell me.	B, G

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

A: Technical problems/diffculties with measurement instrumentation
B: Procedure not properly conducted / Error during sample preparation
C: Matrix effect not properly compensated
D: Lack of experience
E: Error in concentration of analytical standard / calibration stock
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 not waranteed
J: Transcription error / Adminitrative fault
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: Contamination
P: Sample amount not sufficient for quantitative investigation
Q: Error in the conversion factor
U: Undefined

False Positive Results

Analyte	LabCode	Reason / Remarks	
Ethephon	41	we determined the pesticide using only one transition and now we are trying to use a second qualifier transition to be compliant to SANTE regulation	G
N-Acetyl glufosinate	45	transcrition errer (confusing between n-acetyl-glyphosate and n-ycetyl-glufosinate)	J
AMPA	41	we quantified an interfering peak, so we tried to change the quantifier ion	F, M
AMPA	3rd-118	For Glyphosate we use HPLC with post column derivertizer and analysis Glyphosate and AMPA (N-Acetyl-Glyphosate not analysis), we found AMPA in blank. Our LC condition had the wrong separation due to dry soybean matrix or N-Acetyl-Glyphosate . We will purchase N-Acetyl-Glyphosate standard and check again. Comment form Organizer: Contamination	C, M, O
Paraquat	3rd-118	Paraquat we use LC-MS, Idenitfication not enough for dry soybeans matrix due to we found paraquat in sample blank cannot separate paraquat with matrix in soybeans Comment form Organizer: Contamination	C, O

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 (EC) 625/2017[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-SANTE = 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 (EU) 2017/625 of the European Parliament and of the Council on official controls and other official activities performed to ensure the application of food and feed law, rules on animal health and welfare, plant health and plant protection products.. Published at OJ of the EU L95 of 07.04.2017

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

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).

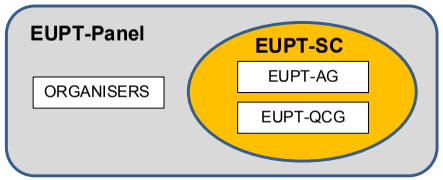
The EUPT-SC's role is to help the Organisers make decisions regarding the EUPT design: the selection of the commodity, the selection of pesticides to be included in the Target Pesticide List (see below), the establishment of the Minimum Required Reporting Levels (MRRLs), the statistical treatment and 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. 101 (1)(a) of Reg. (EC) 625/2017 (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. (EC) 625/2017. 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.

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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.

Based on Reg. (EC) 625/2017, OfLs not paying the EUPT sample delivery fee will be initially warned that their participation in subsequent EUPTs could be denied. In case of a repetitive non-payment, the EUPT organisers will inform the competent authority to take action.

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 625/2017, NRLs are responsible for supporting 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.

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 choose 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

[8] Document N° SANTE/11813/2017; Method Validation and Quality Control Procedures for Pesticide Residues Analysis in Food and Feed

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of the target analytes as Internal Standards (ILISs), the 'procedural calibration' approach as well as 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

[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).

data processing (e.g. integration of wrong peak), major deviations from the analytical procedure, 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 $\text{FFP-}\sigma_{pt}$ 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]:

$\lvert z \rvert \leq 2.0$	Acceptable
$2.0 < \lvert z \rvert < 3.0$	Questionable
$\lvert z \rvert \geq 3.0$	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.

[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

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– ***Category A and B classification***

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:

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

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.

[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.

EURL EU REFERENCE LABORATORIES FOR RESIDUES OF PESTICIDES

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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.

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).

[14] Article 101 of Regulation (EC) 625/2017

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- 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.

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.

SPECIFIC PROTOCOL

for the 13th EU Proficiency Test
on Pesticides requiring Single Residue Methods
EUPT – SRM13 (2018)
(released on 09.April, 2018)

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-SRM13 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-SRM13 deals with the analysis of SRM-pesticides in dry soybeans 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-SRM13. This includes laboratories involved in import control within the frame of Reg. 669/2009/EC. A special **EUPT-SRM13-Website** containing links to the most important documents of relevance was constructed.

A preliminary selection of the laboratories considered to be obliged to participate in the present was done 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 was shown on the registration page. As far as the EUPT-SRM13 is concerned, all OfLs analysing pesticides of food and feed in the commodity group "high oil content and very low water content" were considered as obliged to participate in this PT. OfLs listed as "obliged to participate in the EUPT-SRM13" but not intending to participate had to state their reasons for non-participation during the online registration of the EUPT-SRM13, which lasted from 26 February till 16 March, 2018. The feedback received during registration is considered in the final list of obliged laboratories.

Test Item and Blank Material

This EUPT deals with the analysis of pesticide residues in **Soybean Flour**.

Participants will receive two bottles containing:

1) ca. 200 g **Test Item with a selection of incurred and spiked analytes** from the **Target Pesticides List**.
2) ca. 200 g **Blank Material**, that can be used for recovery experiments as well as for the preparation of matrix-matched calibration standards

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

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-SRM13 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-SRM13 Target Pesticides List before starting with analysis and result reporting**.

Please note, the definitions of the following target pesticides was modified:

- "Chlormequat cation" and "mepiquat cation" were changed to "chlormequat chloride" and "mepiquat chloride" respectively, to reflect the new wording of the MRL-regulation
- "Phosphane" was changed to "Phophane (phosphine, incl. phosphane released from phosphide salts)", to reflect the new wording of the MRL-regulation

It should not be assumed that only pesticides registered for use in Soybeans are present in the Test Item.

Shipment of Test Item

Test item and Blank Material are planned to be shipped on 23 April, 2018.
Cooled Test Item and Blank Material will be packed in thermo-boxes together with cool gel packs and shipped from Germany via DHL express parcel 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 in advance 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.

After the packages were picked up and stored in the DHL delivery system, each participant will be informed by DHL about the tracking number of his package. The participants can follow the delivery state of their own packages online.

Once a package has arrived the recipient country and an unexpected delay is notices, the participant himself is strongly encouraged to contact his local DHL Express or the customs in order to accelerate the clearance and delivery procedures.

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Instructions on handling the Test Item

Both Test Item and Blank material are provided as milled flour. Once arrived, the Test Item should be stored deep frozen (at -18°C or lower) until analysis in order to avoid any possible deterioration/spoilage and to minimize pesticide degradation.
Before analytical portions are taken for analysis, the Test Item should be mixed thoroughly in its entirety. During mixing, try to keep temperatures low to avoid losses of susceptible 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 analytes in the routine scope should be indicated in the EUPT-SRM13 Result Submission Website.

The homogeneity tests will be conducted using 5 g for QuEChERS and QuPPe amenable pesticides. As sub-sampling variability increases with decreasing analytical portion size, sufficient homogeneity can be guaranteed only for sample portions equal to or bigger than the used portion size in the homogeneity test.

Results submission website

Sample receipt acknowledgement, analytical results and method information are to be submitted via the following website: EUPT-SRM13 result submission website.

- **Sub-Page 0 (Sample receipt acknowledgement), accessible from 25 April, 2018.**
- **Sub-Pages 1-3 (analytical results and method information) accessible from 30 April, till 22 May, 2018.**
- **The deadline for result submission is 22 May, 2018 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-SRM13 lab-codes 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 package it must report to the organiser via the EUPT-SRM13 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 27 April, 2018. 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 27 April at noon, 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.

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

To report their results, laboratories must access the **EUPT-SRM13 Result Submission Website**.
All results must be reported on this website by 22 May, 2018 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-SRM13 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 Guide on Result Submission (http://www.eurl-pesticides.eu/library/docs/srm/EUPT-SRM13_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-SRM13 result submission website

Follow-up actions

After the distribution of the EUPT-SRM13 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-SRM13-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–SRM13 can be found in the **EURL-Document Repository (CIRCA-BC)**. Links to the documents can also be found in the **EUPT-SRM13 Website**.
For further information please contact the organizers **EURL-SRM@cvuas.bwl.de**
Please check the **EUPT-SRM13 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: 200 €
- Labs based in third countries: 350 €

An invoice issued to the "invoice address" stated in the registration form will be sent to the invoice e-mail address 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 unless special information was provided by the participant during registration and/or otherwise agreed between participant and organiser.

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 its 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 are given in the invoices.

Calendar of EUPT-SRM13

(please see http://www.eurl-pesticides.eu/library/docs/srm/EUPT-SRM13_Calendar.pdf)

Target Pesticides List of EUPT-SRM13

(please see http://www.eurl-pesticides.eu/library/docs/srm/EUPT-SRM13_TargetPesticideList.pdf)

Contact information

EU Reference Laboratory for Single Residue Methods (EURL-SRM)
Chemisches und Veterinäruntersuchunsamt Stuttgart
Schaflandstr. 3/2,
D-70736 Fellbach
Germany

e-mail: EURL-SRM@cvuas.bwl.de
Fax: +49 3426 1124

Organising Group at the EURL-SRM (Stuttgart)

Michelangelo Anastassiades	phone: +49 3426 1124
Pat Schreiter	phone: +49 3426 1029
Carmen Wauschkuhn	phone: +49 3426 1159
Hubert Zipper	phone: +49 3426 1141
Anja Barth	phone: +49 3426 1935
Giovanna Cerchia	phone: +49 3426 1114

Advisory Group

Amadeo Fernández-Alba	EURL-FV, University of Almería (UAL), ES
Miguel Gamón	EURL-FV, Laboratorio Agroalimentario Generalitat Valenciana (LAGV), ES
Mette Erecius Poulsen	EURL-CF, National Food Institute (DTU), Søborg, DK
Ralf Lippold	EURL-AO, CVUA Freiburg, DE
Philippe Gros	Service Commun des Laboratoires (SCL) / Laboratoire de Montpellier, FR
Magnus Jezussek	Bavarian Health and Food Safety Authority (LGL), Erlangen, DE
André de Kok	Netherlands Food and Consumer Product Safety Authority (NVWA), Amsterdam, NL
Sonja Masselter	Austrian Agency for Health and Food Safety (AGES), Innsbruck, AT
Finbarr O'Regan	Pesticide Control Laboratory (PCL), Dept. of Agriculture, Food and the Marine (DAFM) IR
Tuija Pihlström	Swedish National Food Agency (SNFA-Livsmedelsverket), Uppsala, SE
Carmelo Rodríguez	University of Almería (UAL), Spain

Quality Control Group

Antonio Valverde	University of Almería (UAL), ES
Paula Medina	European Food Safety Authority (EFSA)

CALENDAR for the EUPT – SRM13

Soybeans

(released on 23 January, 2018)

Activity	Who?	Dates
Opening of the **EUPT-SRM13 Website** with links to all relevant documents (**Calendar**, **Target Pesticides List**, **General Protocol**)	EURL-SRM	23 Jan., 2018
Registration via "**EUPT-Registration Website**" (Note: Obliged Laboratories MUST enter this Website and either register or give **explanations for non-participation**)	**All Laboratories interested in participation and all obliged labs even if not interested**	**26 Feb. – 16 March 2018**
Dispatch of **EUPT-SRM13-Specific Protocol**	EURL-SRM	April 2018
Preparation of EUPT-SRM13-Test Item (preliminary tests Spiking / Homogenization)	EURL-SRM	March 2018 – April 2018
Homogeneity Tests	EURL-SRM	March 2018 – April 2018
Stability Tests	EURL-SRM	April – June 2018
Shipment of EUPT-SRM13 Test Item (+reminder of upcoming parcel arrival)	EURL-SRM	**23** April**, 2018**
Confirmation of sample Receipt and acceptance via "**EUPT-SRM13 Result Submission Website**", (Sub-Page 0)	**Participating Labs**	**within 48 h of receipt**
Result Submission (Pesticide scope, Results, Method Info) in "**EUPT-SRM13 Result Submission Website**", (Sub-Pages 1 – 3)	**Participating Labs**	**30 April – 22 May 2018**
Preliminary Report (only compilation of results and preliminary assigned values)	EURL-SRM	June 2018
EUPT Evaluation Meeting	EUPT-SC, DG-SANTE	–
Survey to collect reasons for underperformance and missing information on methods	EURL-SRM / **Participating Labs**	June / July 2018
Final Report	EURL-SRM	Dec. 2018

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

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 – SRM13 2018, Dry Soybeans

update on 14.03.2018

Compounds Potentially Present in Test Item	Listed in	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
Bromide ion	MACP-Reg.	2
Chlormequat chloride	MACP-Reg.	0.01
Cyromazine	MACP-Reg.	0.01
Ethephon	MACP-Reg.	0.02
Fluazifop (free acid, no hydrolysis step to be applied)	MACP-Reg.	0.01
Glyphosate (parent only)	MACP-Reg.	0.03
Haloxyfop (free acid, no hydrolysis step to be applied)	MACP-Reg.	0.003
Mepiquat chloride	MACP-Reg.	0.01
Optional Compounds (will NOT be considered in Category A/B classification)		
2,4-DB (free acid, no hydrolysis step to be applied)	NCP-WD	0.01
Bentazone (free acid, no hydrolysis step to be applied)	–	0.02
Carbofuran (sum, except 3-OH-CF)	MACP-Reg.	0.005
Chlorate (anion)	NCP-WD	0.01
Diquat (dication)	NCP-WD	0.02
Fenoxaprop (free acid, no hydrolysis step to be applied)	–	0.01
Glufosinate	NCP-WD	0.02
MPP (metabolite of glufosinate)	NCP-WD	0.02
N-Acetyl-Glufosinate (metabolite of glufosinate)	NCP-WD	0.02
AMPA (metabolite of glyphosate)	NCP-WD	0.05
N-Acetyl-Glyphosate (metabolite of glyphosate)	NCP-WD	0.02
Paraquat (dication)	NCP-WD	0.02
Perchlorate (anion)	Contaminant	0.01
Phosphane (phosphine, incl. Phosphane released from phosphide salts)	NCP-WD	0.005
Phosphonic acid	NCP-WD	0.05
Quizalofop (free acid, no hydrolysis step to be applied)	NCP-WD	0.01

MACP-Reg.: MACP Regulation
NCP-WD: Working Document SANCO/12745/2013, 21 –22 November 2017 rev. 9(1)

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-SRM13 Organising Team

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

Appendix 10. Calendar and Target Pesticides List of EUPT-SRM13

European Union Reference Laboratory
for pesticides requiring Single Residue Methods (EURL – SRM)
hosted at Chemisches Veterinäruntersuchungsamt Stuttgart (CVUA Stuttgart)

Schaflandstr. 3/2
70736 Fellbach
Germany

Tel: + 49 711 3426 1124
Fax: + 49 711 58 81 76

http://www.srm.eurl-pesticides.eu
e-mail: eurl-srm@cvuas.bwl.de